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Advances in Materials Technology: MONITOR

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POWDER METALLURGY

Dear Reader,

This is number 22 of UNIDO's state-of-the-art series in the field of materials entitled Advances in Materials Technology: Monitor. In this issue we return to the subject of Powder Metallurgy. As many of our readers will recall, Issue No. 4 of the Monitor had already covered this subject. This issue will hopefully bring us up to date on the subject.

Some of the main articles for this Monitor were provided by Prof. Roland Stickler, University of Vienna.

We apologize for the order of articles in the table of contents due to technical difficulties.

We invite our readers also to share with us their experience related to any aspect of production and utilization of materials. Due to paucity of space and other reasons, we reserve the right to abridge the presentation or not publish them at all. We also would be happy to publish your forthcoming meetings, which have to reach us at least 6 months prior to the meeting.

For the interest of those of our readers who may not know, UNIDO also publishes two other Monitors: Microelectronics Monitor and Genetic Engineering and Biotechnology Monitor. For those who would like to receive them please write to the Editor of those Monitors.

Industrial Technology Development Division

CONTENTS

1. DEFECTS LIMITING THE POTENTIAL USE OF PM-MATERIALS SUBJECTED TO CYCLIC LOADING

(Brigitte Weiss and Roland Stickler,
University of Vienna, and
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Reutte, Austria)

Introduction

It is known that even pm-materials of highest quality may contain small amounts of microstructural defects (pores, inclusions) which may affect the mechanical properties of such materials, in particular the high cycle fatigue behaviour.

In view of the inherent risk involved with unexpected failures of components made of high-strength pm-alloys it appears mandatory to provide the designer with sufficient information on the reliability of property data. At the present state of art it seems that the presence of small amounts of inhomogeneities cannot be avoided, however, a control of maximum sizes of such defects and their distribution should be feasible. For quality control a prediction of the detrimental effects of microstructural defects as function of their size, distribution, geometry and nature is required. For economic processing and to achieve mininum guaranteed properties it is essential to define realistic limits of tolerable defects.

Several attemps to describe the high-cycle fatigue (HCF) behaviour of defect-containing pm-materials have been published. Amongst the first to suggest an explanation of the effects of defects on the fatigue properties of pm-alloys by a fracture mechanical approach were Betz and Track. (1) They assumed that fatigue crack initiation occurs during the first few loading cycles and that it is possible to calculate fatigue crack growth to overload failure by fracture mechanic (FM) relationships. The authors could demonstrate that the fatigue strength of pm-materials is related to the size and location of inclusions.

The fact that superficial pits or scratches may affect the HCF-properties is known. While numerous publications exist about the influence of macroscopic notches on the fatigue behaviour based on FM considerations (2, 3, 4, 5), only recently a qualitative explanation for the existence of small non-damaging surface notches and a quantitative derivation of their critical sizes were presented. (6, 7) The proposed analysis requires only the knowledge of geometrical factors (stress concentration factor, notch geometry and size) and intrinsic material parameters (fatigue limit of the unnotched material, effective threshold stress intensity). According to this analysis the radius of a non-damaging semicircular circumferential notch is given by

$$r \leq 4.5 l_0/(K_t^{\,2}-1) \qquad (1)$$

with

$$l_0 = (dK_{theff}/S_u \cdot F)^2 \qquad (2)$$

The fatigue strength of a notched specimen can then be deduced from the relationship

$$S_n = S_u/K_t \cdot (1+4.5 l_0/r)^{1/2} \qquad (3)$$

The proposed relations were found to predict the limiting size of semicircular notches (edge notches, circumferential notches) and to give a reasonable estimate of the reduction of the fatigue strength of specimens as function of notch size for recrystallized Cu, Al-alloys and steels. The results for Cu in excellent agreement with experimental findings are shown in figure 1 which contains also the data calculated for recrystallized Mo-specimens.

Recently, several investigators studied the effects of small artificial surface defects (micropits) on the fatigue limit. Yamada et al. (8) and Murakami et al. (9) found in high-strength steels during cyclic loading close to the HCF-limit that NPCs were formed around such micropits. The problem of defining the fatigue limit was then related to a description of the condition for propagation of such NPCs either based on conventional S-N data or fatigue threshold conditions.

To overcome the difficulties of correlating the size and shape of various defects Murakami (9) proposed that the parameter describing best different geometrical shapes is the square root of the defect area projected onto a plane normal to the stress direction, but neglecting the contribution of NPCs. The value of dK is then described by the following equation

$$dK/2 = 0.65 \cdot S \cdot \sqrt{\pi} \cdot \sqrt{area} \qquad (4)$$

In this relationship the influence of dK_{theff} in describing the growth conditions of such NPC is neglected.

Recently we have proposed another approach to treat the effects of artificial micropits on the fatigue behaviour. (10) This model is based on the experimental observation that NPCs are associated and comparable in size with such micropits in specimens cyclically loaded close to the fatigue limit. The size of the NPCs was found to correlate well with the extent of the Kt-field around the micropit, as computed by FEM methods for various hemispherical and cylindrical closed-bottom holes. In combination with the appropriate Kitagawa-diagram these considerations were found appropriate to predict the size of non-damaging micropits and of the decrease in fatigue strength resulting from larger holes. This model is demonstrated schematically in figure 2 by means of a Kitagawa diagram for the cases of a non-damaging and a damaging micropit. The influence of closure which is build-up during microcrack growth (11) and which results in a reduction of the NPC-size can be calculated by equation 5 (valid for R = -1).

$$dK_{eff} = dK - (1-e^{-k \cdot c}) \cdot K_{opmax} \qquad (5)$$

with dK being calculated for the effective crack length (c+r) and the second term resembling the closure contribution increasing with the crack length.

In present investigation these considerations were applied to evaluate the effect of micropits on the fatigue properties of specimens of recrystallized molybdenum. It was the aim of this study to predict non-damaging defect sizes and the reduction of the fatigue strength by larger pit-shaped defects.

Experimental details

For the investigations, bars of recrystallized technically pure Mo produced and processed by conventional pm-procedures were used as specimen material. Fatigue test specimens were cut from the cylindrical rods of 5 mm diameter.

The specimens were mechanically polished in longitudinal direction and subjected to a brief electropolishing procedure. Hemispherical micropits and cylindrical holes with hemispherical bottoms were produced by electro-discharge machining. Electrodes were prepared by fine tungsten wires embedded in stainless steel capillaries. It was found that the diameter of the holes was related essentially to the diameter of the tungsten wire whereas the depth was controlled by the number of individual discharges.

After the electro-discharge machining the specimens were thoroughly cleaned and heat treated (recrystallization at 1350C/4h). The dimensions of each surface hole were measured by light microscopy (hole diameter by a travelling microscope, accuracy +/-5 μm, hole depth by a focusing method, accuracy +/-2 μm). The actual sizes of hemispherical pits ranged between 60 and 125 μm radius, for the cyclindrical holes with hemispherical bottoms for a radius of 125 μm of depth between 60 and 250 μm.

The fatigue tests were performed with a resonance system operated at 20 kHz at room temperature and at a stress ratio of R = -1.

Details of this test procedure have been described earlier. (12) In present investigation the fatigue loading was extended to 1×10^9 cycles to obtain reliable data on the existence of a fatigue limit. At least eight specimens of the defect-free material and of each hole geometry were tested to determine the fatigue limit. Each run-out specimen was examined by LM and SEM to reveal the presence and length of microcracks in the vicinity of the holes. Subsequently the specimens were fractured by cyclic loading at an amplitude slightly above the fatigue limit for a quantitative evaluation of the fracture surfaces. The extent and the shape of the NPCs around the holes were determined.

Measurements of the threshold stress intensity for fatigue crack growth were carried out by a high-frequency test technique. (12) The value of dk_{theff} was computed from closure measurements by a strain gauge method. (13) Specimens were prepared from the cylindrical rods by matching flat centre portions of approximately 3 mm thickness. A small center notch (0.4 mm diameter and 0.18 mm depth) was introduced on one of the flat sides by electro-discharge machining for the initiation of the fatigue crack. The growth behaviour during cyclic loading was observed under a high-power light microscope, the threshold values were determined in accordance with ASTM recommendations. (14) A summary of the mechanical property data is given in table 1.

The K_t-values for the various sizes and geometries of micropits were taken from FEM computations (10) carried out for cylindrical specimens.

Experimental results

Fatigue endurance data for semicircular circumferential notches:

The calculations of the non-damaging notch size and the reduction of the fatigue limit according to Lukas et al. (6) were shown to be in good agreement with experimental data for Cu, Al-alloys and steels. Therefore, no extensive experiments were considered necessary for Mo. The calculated data shown in figure 1 indicate that for the semicircular circumferential notch geometry the critical size for the non-damaging notch is approximately 30 μm radius. The reduction in fatigue strength with increasing notch size is considerable, with a minimum at a notch of approximately 300 μm radius.

Fatigue endurance data for micropit-shaped notches:

The values of the fatigue limit (for $N = 1\times10^9$) of specimens containing hemispherical micropits are plotted as function of the defect radius in figure 3a. This diagram clearly reveals that already micropits with a radius of 60 μm appear to affect the fatigue limit of the defect-free specimens. With increasing defect-radius a gradual reduction of the fatigue limit can be noticed. The scatter in the experimental data is indicated by the bars in the diagram.

In figure 3b the influence of increasing depth for a defect radius of approximately 125 μm is shown. A gradual decrease of the fatigue limit with increasing depth of the hole can be noticed. Although the non-damaging hole dimensions were not established we may assume from an extrapolation of the curve that the critical depth of a defect of 125 μm radius would amount only to approximately 20 μm.

Fractography:

The LM investigation of run-out specimens revealed only in some specimens the presence of NPCs, however, it should be mentioned that in this material it is extremely difficult to identify such microcracks because of their predominant nature of grain boundary failure associated with virtually no residual crack opening. In figure 4a a NPC is shown initiated at the edge of the hole. The fracture surface is shown in figure 4b, from which the extent of the NPC can be determined.

The results of the fractographic evaluation are plotted in figures 5a and 5b. For the hemispherical micropits the NPCs follow the perimeter of the hole in a semicircular shape. A comparison of these findings with data from FEM-computation on the extent of the K_t-field (to a level of K_t = 1.05) shows good agreement.

The NPCs around the cylindrical blind-bottom holes exhibited an approximate parabolic shape. The length of the NPCs at the surface, "c", increased with increasing depth of the hole while the depth below the bottom of the hole, "a", increased with the hole depth at a much smaller rate. The drop-off of Kt along the specimen surface, "K_{tc}", and below the bottom of the hole, "K_{ta}", is also plotted in figure 5b.

K_t values computed by FEM:

The values of the K_t values at the perimeter of the micropits and at the deepest point of the pit computed by FEM are listed in table 2 which also contains values of the distance from the hole at which K_t has decreased to 1.05.

The maximum of K_t occurs in a plane normal to the stress axis. For the hemispherical holes K_t is essentially the same at the specimen surface and at the bottom of the hole. For cylindrical holes the maximum in K_t occurs at the specimen surface with lower values for increasing hole-depth.

Discussion of results

The experimental work was carried out in an attempt to simulate the effects of micropits or of small pores intersecting the specimen surface of typical pm-materials.

The analysis of the influence of circumferential surface notches according to the approach shown to yield engineering data for other alloys resulted for recrystallized Mo in values for a critical notch radius of about 30 μm. These calculations were based on the measured dK_{theff}, fatigue limit, and a value of l_o = 53 μm. It is obvious that small surface notches or scratches already affect the fatigue limit. The limiting size of non-damaging notches can be predicted, as well as the fatigue limit of specimens containing larger notches.

An attempt to apply the same reasoning to micropits and hole-shaped surface notches failed to give reasonable predictions. This is probably due to the fact that the approach by Lukas (7) is based on deductions valid only for a notch the lateral extension of which is large compared to its radius.

However, results in agreement with observations were obtained by using the recently proposed model (10) based on an experimentally determined Kitagawa Diagram, with the fatigue limit of unnotched specimens, the dK_{theff}, the K_t values and the extension of the stress field around the hole as parameters (see figure 2). The results for semicircular surface cracks with F = 1.24 are plotted in the Kitagawa Diagram in figure 6. The accuracy of the presentation is limited by the scatter in the experimental data on the fatigue limit as well as by the uncertainties in determining the dK_{theff} values. Under the assumption of a closure-free material a crack nucleated at the edge of a hole under a cyclic stress $S_u \times K_t$ should grow up to a distance where K_t approaches unity. As revealed by the FEM computations the stress field around hemispherical pits extend for a distance of approximately twice the radius of the notch. If this total length ("effective length" notch plus NPC) is shorter than the critical length c_0 the crack around the notch should cease to grow. If the length is larger than c_0 the value of dK at the crack tip is larger than the limiting dK_{theff} at the fatigue limit of the unnotched specimen. Therefore, the crack should continue to grow up to failure of the specimen. To obtain a fatigue limit for this notched specimen the cyclic stress amplitude must be lowered until the effective defect length becomes smaller than the corresponding point on the dK_{theff}-line. The results obtained by this reasoning are in reasonable agreement with the experimental findings, figure 7.

Closure effects would reduce the effective length of the NPC, however, in view of the predominant intergranular fracture mode associated with the NPC in the recrystallized Mo material we may assume little or negligible closure contributions. A similar analysis performed for cylindrical holes yields also results in fair agreement with experimental observations.

For comparison the tolerable micropit sizes were evaluated following the method proposed by Murakami. (9) Under the assumption of the effective defect size (cross-section of hole plus NPC) the obtained results slightly underestimate the experimental findings, table 3.

Conclusions

From the experimental results on the effects of small superficial defects (grooves, pits) on the HCF-fatigue strength of recrystallized pm-Mo we arrive at the following conclusions:

- The analysis for surface grooves as shown to predict non-damaging notch radii in specimens for Cu and steels also appears applicable to recrystallized Mo. The critical radius of a circumferential hemispherical notch not affecting the fatigue limit of unnotched specimens (for R = -1) is about 30 μm;
- For pit-shaped surface holes the experimentally verified non-damaging size of less than 30 μm can be predicted using a model based on experimental data of dK_{theff}, S_u, the K_t and its extent into the interior of the specimen which approximately equals the length of the NPCs. The effective crack size in the Mo-specimens corresponded to about 2.5 times the notch radius;
- Under the applied experimental conditions closure build-up was found to be negligible probably due to the predominant intergranular growth of the short fatigue cracks from the holes;
- The proposed model was found also suitable to approximate the fatigue strength of specimens containing superficial holes;
- It may be speculated that the proposed model can be applied in general to predict the fatigue behaviour of metals and alloys containing isolated defects (notches, voids, inclusions, etc.).

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List of symbols used

a	crack depth below notch
c	crack length on specimen surface
c_o	critical crack length on specimen surface
c_{eff}	effective crack length at specimen surface, r+c
d	depth of notch
r	radius of notch
k	materials constant in 1/mm units
l_o	critical crack size associated with circumferential notch
F	geometrical correction factor
NPC	non-propagating crack
R	stress ratio
S	cyclic stress
S_u	fatigue limit of unnotched specimen
S_n	fatigue limit of notched specimen
K_t	stress concentration factor
dK	range of stress intensity factor
K	stress intensity factor
dK_{eff}	range of effective stress intensity factor
dK_{theff}	range of effective threshold stress intensity factor
K_{opmax}	opening level for macroscopic crack

Table 1: Property data of specimen material

technically pure pm-Mo
cyclindrical rods, 5mm diam, recrystallized 1350C/4h in vacuum
grain size: 80-100 µm average grain diameter
tensile: R_m = 675 MPa, A = 35%
dynamic Young's modulus: Edyn = 340 GPa
fatigue limit (N = $1x10^9$, RT, R= -1): S_u = 290 +/-5% MPa
threshold stress intensity for fatigue crack growth (RT, R= -1,
da/dN≤ $1x10^{-11}$ m/c, semi-elliptical surface crack):

dK_{th} = 5.6+/-10% MPa.$m^{1/2}$, dK_{theff} = 4.2+/-10% MPa.$m^{1/2}$

Table 2: FEM results of K_t for single micropits at the surface of long cylindrical bars:

Hole shape	Dimensions radius/depth µm	K_t at surface/bottom	distance from hole surface to K_t = 1.05, µm c(surface)/a(bottom)
hemispherical	60/60	2.0/2.0	60/60
	80/80	2.05/2.05	80/80
	125/125	2.10/2.10	120/120
cylindrical	125/250	2.54/2.25	250/120

Table 3: Analysis of effects of micropits on fatigue strength of recrystallized Mo by a modified Murakami procedure (9)

micropit size radius/depths µm	effective size radius/depths µm	dK (calc.) MPa.$m^{1/2}$	effect on S_u (dK_{theff} = 4.2 MPa.$m^{1/2}$) *)
60/60	120/120	4.09	non damaging
80/80	160/160	4.72	damaging
125/125	240/240	5.8	damaging
125/60	250/120	4.9	damaging
125/250	370/350	7.1	damaging

*) damaging for dK ≥ dK_{theff}

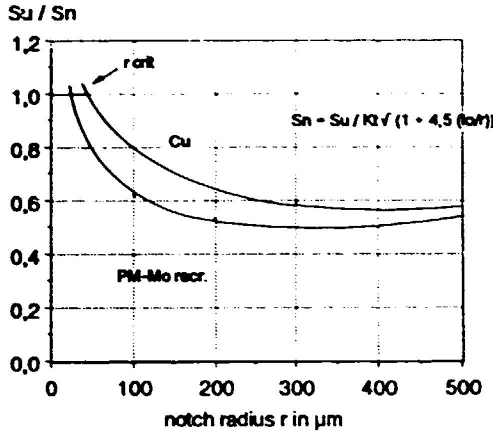


Fig. 1: Influence of the radius of a circumferential semicircular notch on the fatigue strength of Cu and recrystallized pm-Mo

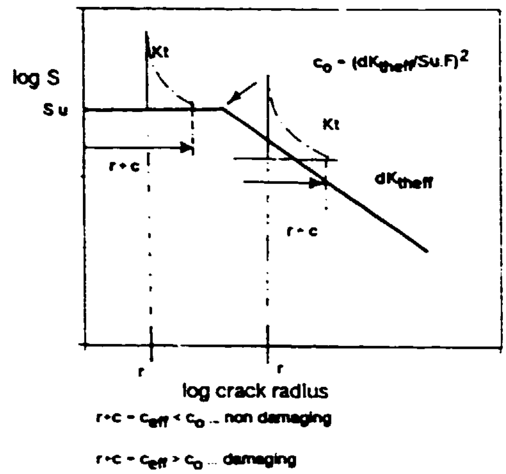


Fig. 2: Kitagawa diagram for the evaluation of the critical size of superficial holes on the fatigue limit, schematic. The values of Kt at the edge of the hole and the extent of the stress concentration into material surrounding the hole are indicated for hemispherical pits.

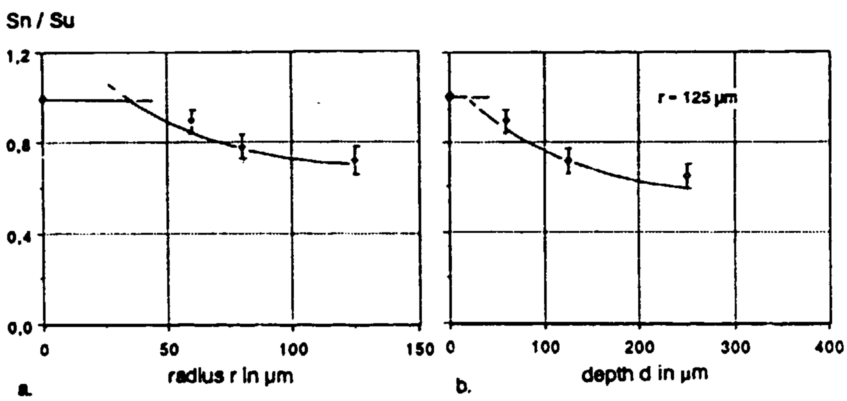


Fig. 3: Effect of the dimension of superficial holes on the high-cycle fatigue strength ($N = 2x10^8$):

a. hemispherical micropits of radius r

b. cyclindrical holes with hemispherical bottoms of 125µm radius and various depth

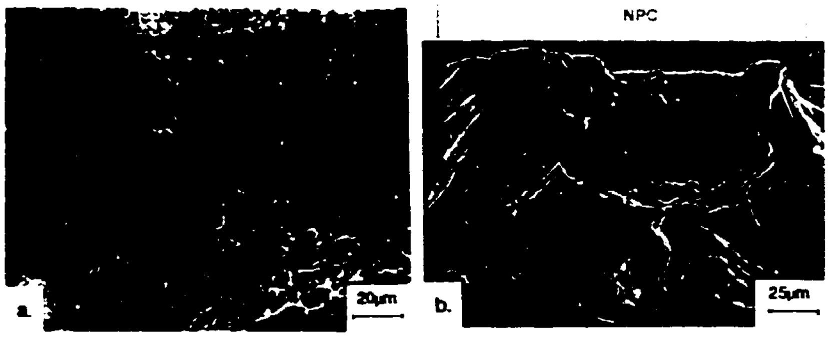
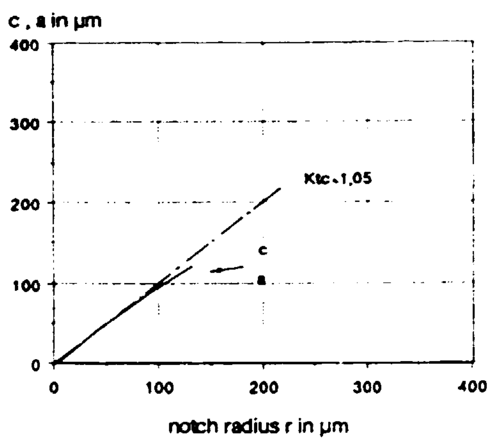
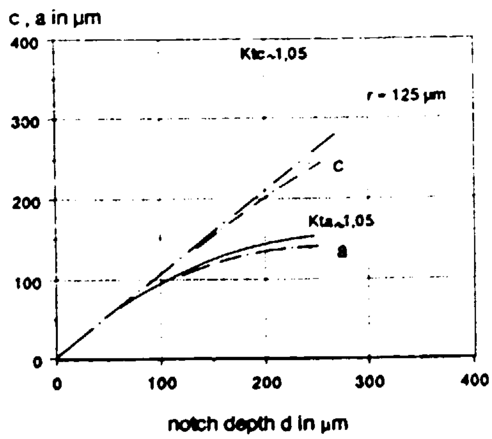
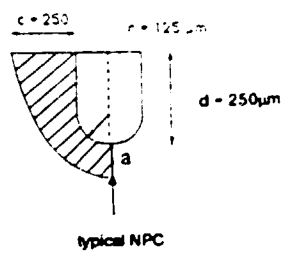


Fig. [illegible]: Appearance of NPC in a fatigue exp[illegible] specimen of recrystallized [illegible] = [illegible], N = [illegible] ^{9}:
a. Specimen surface near hemispherical pit, light micrograph
b. Fracture surface of same specimen exhibiting extent of NPC around pit



a. for hemispherical pits





b. for cylindrical holes of 125µm radius with increasing depth

Fig. [illegible]: Dependence of NPC size (crack length from edge of hole at surface, "c", and crack depth below bottom of hole, "a", experimental data

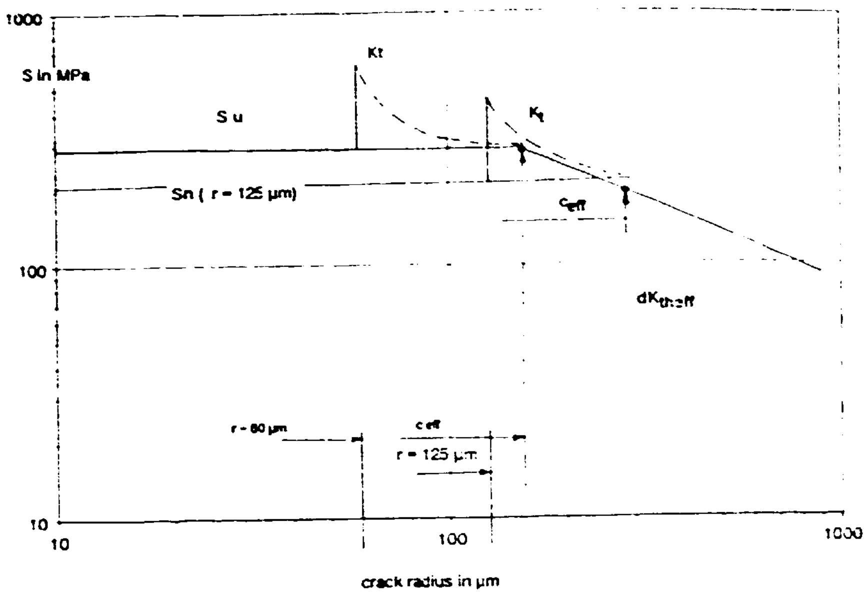


Fig. 6: Kitagawa diagram for the evaluation of the critical radius of a non-damaging hemispherical micropit and the effect of larger pits on the high-cycle fatigue limit (c_{eff} about 0.5r)

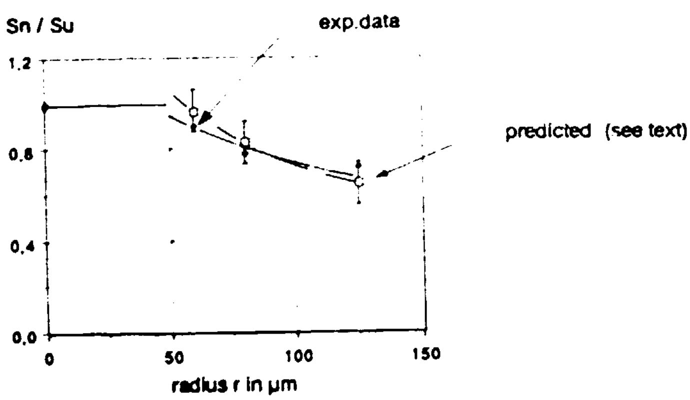


Fig. 7: Comparison of experimental and predicted data on the fatigue strength of recrystallized pm-Mo rods containing hemispherical micropits

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2. DEVELOPMENTS IN POWDER METALLURGY

1. Introduction

The art of making metal articles using powder as the raw material began in ancient times (2000-3000 BC). It was eventually superseded by melting procedures but a revival began at the turn of the century with the powder metallurgy (PM) method being used to fabricate metals with very high melting points, such as tungsten electric light filaments and platinum ware. Later developments included cemented carbides, electrical contact materials and brushes, bronze self-lubricating bearings, powdered iron cores for electrical components, friction materials for brakes and clutches and automobile gear pumps.

There has been rapid growth in the use of iron and steel powder in recent years for the mass production of precision mechanical components. The main consumer has been the motor industry; the average Japanese car now contains ~ 8 kg of PM parts. The past 10 years has also seen the development of new types of PM process and new uses for metal powders: the industry is moving quite rapidly towards becoming more sophisticated and "high tech", making products with a high added value.

2. Traditional PM process

PM is often referred to in current terminology as a near net shape forming process. All of these processes, such as casting, forging, PM and spray forming, aim to produce a part with a complex shape in as near to the final required shape and dimensions as possible. In this way expensive finishing operations, such as machining, can be minimized. All of the processes use a mould or die to duplicate the required shape and dimensions; the metal must be in a form in which it will flow into the mould or die, that is, liquid metal, plastically deforming metal, metal powder, or liquid metal particles arriving at a surface.

The PM process consists essentially of (i) filling a die with metal powder; (ii) compacting the powder in the die; (iii) removing the "green" compact from the die; and (iv) sintering the "green" compact at high temperature to produce a dense part. PM is usually taken to mean the pressing and sintering process; there are more complex processes, for example, hot pressing and powder forging, but the simple press and sinter route is the most widely used. The metal powder is not usually made by the PM parts manufacturer, but is supplied by a separate powder manufacturer.

Fabrication of parts from powders has the following distinct advantages:

(1) The parts produced require very little, if any, machining or other finishing operations. There is, therefore, virtually no scrap loss. Losses by the conventional melting, casting and fabrication route can be, by comparison, substantial, for example, ingot cropping, rolling losses, extrusion discard, etc.

(2) Parts can be mass produced.

(3) Complicated shapes are possible.

(4) Very uniform parts are produced provided good control is kept over the process.

(5) The process is particularly suited to reactive metals and/or metals with high melting point such as beryllium and tungsten. To make parts by any other way with such metals would be uneconomical or impractical.

(6) PM techniques permit a much greater range of compositions and microstructures to be produced compared with the casting route. Structures are generally more isotropic with a consequent improvement in the isotropy of mechanical properties.

PM also has a number of disadvantages:

(1) PM parts produced by conventional methods have lower impact strengths and lower elongation than forged metal parts because of the difficulty of obtaining full 100 per cent density by the sintering route.

(2) There are no established specifications for powders and, as powders produced in different places by different methods tend to differ considerably, this produces control problems. Powder from a different supplier might require changes to the pressing and sintering process to achieve the same final specification.

(3) Die costs are high for parts with complex shape. The process is more economically suited, therefore, to long production runs.

(4) The degree of compressibility of powder varies across large cross-sections, creating problems in the sintering behaviour of large parts. The process is suited best, therefore, to the manufacture of relatively small components.

(5) Due to their different mechanical properties, long-term service trials are usually necessary before a PM part can replace a forged or cast part. These trials can be expensive.

In advanced countries, the largest consumer of PM components is the automotive industry (60-70 per cent of total tonnage of iron and steel powder). Typical automotive applications include oil pump gears, self-lubricating bearings, fan hubs, camshafts, connecting rods, thrust bearings, carburettor cams, front suspension ball joint bearings, door striker racks, steering support yokes, valve seats, gear-box synchronizing struts, timing chain sprockets and shock absorber parts such as pistons and rod guides.

The total weight of PM parts used in a car is not large compared with the total weight of the car, but these parts are mostly small and serve some special or critical function. They are "high value" parts. In addition to the automotive industry, ferrous PM parts are being used increasingly in a wide range of other machinery and domestic appliances. In Japan, a large market has grown up for small PM parts in business machines, computer peripherals such as printers and plotters, and audio and video recorders. A particular use of iron and Fe-Si alloy PM parts is in stepper motors for printers and disc drives. The market for stepper motors in Japan is estimated at 20 million units/year. PM parts made of special steels such as stainless steel and high speed steel (HSS) are used in special applications; HSS, in particular, made by PM, is finding a market in various tooling applications.

Of the non-ferrous alloys, copper and copper alloys made by PM have been used for many years. The parts include bearings and porous filters. The use of PM in the aerospace industry has grown, particularly for certain critical engine parts such as discs made from superalloys (Ni and Co base). Aluminium alloys made by PM also appear to be finding increasing use. Another specialist area where PM has inherent advantage is for the manufacture of magnet materials such as samarium-cobalt and neodymium-iron-boron permanent magnets and Nb_3Sn superconductors. The manufacture of refractory metal parts by PM is well established. Tantalum sheet is used for electronic capacitors as well as for many high-temperature furnace applications. Perhaps the best known materials made by the powder route are the refractory metal carbides, used as cutting tools. These are usually based on tungsten carbide but most cutting tools contain alloying additions of TiC, TaC, NbC and VC. These materials are a perfect example of a material that can be made in no other way; they are quite small and yet are indispensable to nearly all engineering manufacturing operations.

3. Powder production

Metal powders are used in a wide variety of other applications apart from PM. Examples are ferrous alloy powders for welding consumables, aluminium powder as a rocket fuel, and lead powder for oil drilling muds. The type of powder and particle size and shape required tends to be different for each application and, therefore, the methods of making powders differ widely depending upon the application. However, about 75 per cent of all powder produced is iron and steel powder, and of this, about 85 per cent is used to make PM parts. By far the greatest tonnage of powder produced, therefore, is ferrous powder made specifically for the PM industry.

3.1 Production of iron and steel powder

For PM applications, the major requirements of the powder are: (i) high apparent density to reduce filling height and improve flowability; (ii) good compressibility to produce a high green density; and (iii) a suitable and consistent chemical analysis to achieve rapid sintering and consistent final properties.

The two main methods of producing iron and steel powders for PM are (i) direct reduction of magnetite (Fe_3O_4) and (ii) atomization of liquid iron and steel.

Various other processes exist, such as the electrolytic process and the carbonyl process, and these are used in special circumstances, such as a requirement for high purity iron powder. However, they do not appear to have the technical or economic potential of either the direct reduction or atomization processes for producing powder suitable for PM.

The direct reduction process for producing iron powder has largely been pioneered by the Swedish firm, Höganäs, which now controls a major share of ferrous powder production in the world ($\sim$100,000 t/year). The process consists of reducting magnetic iron ore with coke to produce sponge iron. This sponge iron is subsequently broken up and milled to powder.

Perhaps the major achievement of Höganäs is the steady improvement in the properties of their powder for PM use, in relation to both compressibility and consistency. They have managed to control the ore beneficiation and the reduction process to achieve this. The main disadvantage of the use of pure iron powder for making steel parts is that the additional elements have to be added as elemental powders, which can give rise to segregation during the powder handling procedures, die filling, etc., and hence give rise to heterogenous structures. Höganäs has developed a process that largely overcomes this problem by partially pre-alloying the powder by subjecting the mixture to a low-temperature anneal. This allows some diffusion and bonding to occur.

This difficulty does not occur with water atomized powders which generally have greater uniformity of composition and fewer inclusions. The main disadvantage of water atomized powder is that the powder particles do not have the ductility of sponge iron powder, particularly if it is heavily alloyed, and hence the powder does not have good compressibility. This can be overcome to a large degree by annealing the powder subsequently. The process has now been improved so that it is competitive in price with sponge iron powder. The trend in PM generally towards higher density and greater strength has meant growing use of water atomized powder and about half of the total production of iron and steel powder is now produced by this route.

Powders can also be manufactured by air or gas atomization rather than water atomization. This method is used where roughly spherically shaped particles are required. Generally air or gas atomized powders are not favoured for PM because they give low compressibility and poor green strength; the more ragged and flake-like particles produced by water atomization deform more readily and therefore give better green strength.

The major use of iron and steel powders other than in PMs is in the manufacture of welding consumables; currently about seven per cent of iron and steel powder is used for this purpose. Substantial development has taken place in developing powders with the appropriate particle size and shape to achieve high apparent density and with the appropriate carbon and oxygen contents. Iron powder is also used in a variety of other applications; examples include photocopying powders, reducing agents in the chemical industry, fillers in plastics and iron enrichment of flour.

3.2 Production and use of non-ferrous powders

The production of powders of non-ferrous metals is, in many cases, carried out by a chemical process rather than atomization. Examples are electrolysis for producing copper powder, the carbonyl process for nickel powder and the Sherritt-Gordon process for cobalt powder. However the increasing use of alloys for PM has meant that atomizing of the liquid alloy is becoming the preferred method in many cases.

Powders of metals and alloys with melting points much above 1,600°C are difficult to produce by atomization and, generally, chemical reduction methods are adopted in these cases. The best known example is that of tungsten powder for lamp filaments and tungsten carbide tool manufacture. In this case, the starting point is tungstic oxide powder which is reduced with hydrogen. Tungsten carbide powder is produced by carburization of tungsten powder.

Although developments in the manufacture of powder have been gradual rather than sudden, two new processes have appeared recently that are of interest. Both of these relate to the manufacture of powders for the aerospace industry. The first is a method of making powder of very reactive metals such as titanium by centrifugal spraying. An arc or a plasma is struck between the end of a titanium rod and a water-cooled copper cup or disc which is spinning rapidly. Metal is melted from the end of the rod and is subsequently flung from the periphery of the cup to solidify in flight and form a powder. The rod is fed down continuously and the operation is carried out in a partial pressure of argon gas. An alternative method of heating is to use a tungsten electrode or an electron beam focused onto the end of the rod; in this case the rod itself is rotated rapidly and metal from the molten pool is flung out radially to form the powder (figure 1, see page 22).

The second new development is not really a method of making powder but is a novel means of mixing powders to obtain improved properties in the final product. It is known as mechanical alloying and consists essentially of ball milling powder mixtures containing constituents such as yttria for a very long period (several days). The effect is to distribute the oxide additions on an extremely fine scale (35 nm particles); this very fine distribution is preserved during the consolidation procedure with the result that the final product has exceptional strength, especially at elevated temperature (figure 2, see page 22). Mechanically alloyed nickel alloys (such as Ni-Cr-Al-Y) are now being used for turbine discs in high-performance jet engines. The process was developed by INCO in the US about 10 years ago. Recently, a series of Al-Mg-Li alloys, made by mechanical alloying, has also been produced.

The variety of uses of non-ferrous metal powders is very great, some of the better known uses are given below.

3.2.1 Copper and copper alloy powders

The main use of copper and copper alloy powders is in PM parts. Electrolysis was once the dominant method of copper powder production, but with an energy usage of 3,000 kWh/t it has become uneconomic. Atomization is now the major method of production. Uses of pure copper powder include chemicals and conductive inks. Copper alloy powders are used for brazing and as decorative "gold" flake powders.

3.2.2 Nickel and cobalt alloy powders

These powders find three major fields of application - hard facing, brazing and PM superalloys. Elemental nickel is also used in the chemical industry.

3.2.3 Aluminium powder

Aluminium powder is used in large tonnages - ~100,000 t/year, world wide. Applications include rocket fuel, chemicals, paints, plastics and explosives. The US Challenger space vehicle uses ~200 tons of aluminium powder per launch. It is mostly produced by air atomization. Aluminium alloy powder is being used in increasing quantities for PM. In the aerospace industry, powder for this purpose is made by inert gas atomizing.

3.2.4 Zinc powder

This is used on a very large scale, with the paint industry dominating the usage. Zinc powder is also used in the refining of zinc; all electrolytic refineries use about five per cent of their output as powder in the range 30-200 μm. Atomizing with air is normally used; finer powders (1-10 μ) can be made by vaporization and condensation but this consumes ~1,100 kWh/t and is therefore very expensive.

3.2.5 Lead and tin powder

Lead powder is used in some paints and for special oil drilling muds. Tin powder is used in chemical processes and for mixing with bronze powder for bearing manufacture. Pb/Sn alloy powders are used as solder pastes.

3.2.6 Magnesium powder

This is used in small quantities in the chemical industry and for pyrotechnics. It is also used for special military applications. In the latter application, spherical particle shape and small size (20-100 μm) is essential and the powder is made by gas atomization. The usual method of production is by rasping or milling ingots.

An overall picture of the relative consumption of different metal powders in the US during 1983 and 1984 is given in table 1, page 22.

3.3 Growth in the use of powders

Growth in the PM industry over the past 20 years has been steady rather than spectacular (figure 3, see page 22). The consumption of iron and steel powder, which accounts for ~75 per cent of total powder production, has been growing at a rate of ~13,000 t/year over this time. This is in the context of a total world consumption of ferrous powder of ~400,000 t year in 1984 (excluding USSR and China). Total world steel production is ~600 Mt/year; thus less than 0.1 per cent is used in the form of powder.

It is perhaps more meaningful to compare ferrous powder usage with the tonnages used in steel forging and steel casting. Powder accounts for about 10 per cent of steel used in either forging or casting, but what is more significant is that powder consumption is increasing while steel usage for forging and casting is falling.

There is evidence that the use of components manufactured from powder is now increasing more rapidly, particularly in the high technology end of the market. Japan appears to be leading the way: in the years 1982-1984, ferrous powder consumption increased from 54,000 to 70,000 tons, an increase of ~13 per cent per annum. The greatest part of this tonnage is used in the automotive industry and although there was an increase in the total number of cars produced at that time, the total weight of PM parts per car also increased rapidly and is now ~8 kg/vehicle in Japan.

Growth would also appear to be occurring in the use of non-ferrous powder both for PM parts and for other purposes. The major area of expansion for PM parts is in the aerospace industry. The nickel-based superalloys have been produced by PM for some years; indeed, for certain very highly stressed parts, such as turbine discs, no other way exists for making parts with the requisite strength. The use of PM for making advanced aluminium alloys for the aerospace industry also appears to be increasing. Aluminium-based alloys containing substantial additions of lithium for light weight and of iron and molybdenum for high strength are now being developed. Another specialized area where powder usage is likely to increase is in the manufacture of permanent magnets. There has been substantial development in recent years in producing new rare earth-containing magnet alloys; for example, samarium-cobalt and neodymium-iron-boron. All of these alloys are produced by the powder route.

Other than PM, there would also appear to be expanding needs for non-ferrous metal powders. Usage of nickel powder for Ni/Cd batteries is increasing, and there is steady growth in the use of Ni, Cr, Mo and Co powders in welding consumables and for plasma spraying.

3.4 Powder characterization

Specification of the properties of powders is a matter of great importance to the PM industry and one that has not received a great deal of attention. The two most important properties of a powder particle are its size and shape; other properties that can also be important are chemical composition, structure and surface condition, for example, state of oxidation. Comparative measurements of particle size and size distribution are relatively easily made and these parameters are most often used to describe a powder. Particle size measurement, however, is convoluted with particle shape; the different physical techniques used for size measurement only agree exactly when the particles are spherical. Sieving is perhaps the oldest and cheapest method of measuring particle size and size distribution down to 20 μm. Below this size more physical techniques must be used such as sedimentation (for non-ferromagnetic powders), gas permeability or light scattering.

Particle shape is much more difficult to measure and quantify, although with the availability of computers, quantitative particle shape analysis is becoming readily available. The usual method of describing shape is based on the appearance of the powder particles under a microscope. On this basis, a series of typical powder types can be specified. One such series divides powders into seven types, based largely on the appearance of different metal powders made in different ways.

(1) Spherical powder: typical examples are, Ag, Cu and Cu-Sn powders made by gas atomizing.

(2) Nodular powder: nearly spherical or drip-shaped powder; Sn and Pb powders made by gas atomization are typical examples.

(3) Dendritic powder: electrolytic copper powder deposited on the cathode is an example of a dendritic powder.

(4) Flaky powder: very thin flakes, Al, Cu and Cu-Zn powders made by chemical milling are examples.

(5) Angular powder: mechanically crushed Fe, Cr and Si powders are of this shape.

(6) Spongy powder: porous powder: Fe powder manufactured by the reduction method is an example.

(7) Irregular powder: asymmetrical powder, typical examples are mechanically pulverized Zn and Pb powders, and atomized Cu powder.

Quantitative shape analysis is a subject in its own right, and it is undergoing quite rapid development with the advent of rapid computing and automatic vision systems. Several different approaches have been taken such as (i) conventional shape factors; (ii) stereological characterization of shape; (iii) morphological analysis; and (iv) the use of fractals. Most of these methods use a series of mathematically defined parameters with names such as elongation, compactness and ruggedness. None of these systems of defining shape has yet been incorporated into any of the standards. However, the increasing use of metal powders, not only for conventional PM, but also for a range of new applications, will increase the need for a better means of specifying powers. Currently, with relatively few large powder manufacturers in the world, the problem is not acute because the properties of a particular grade are carefully controlled by the manufacturer. The need to be able to specify an "equivalent" powder will become increasingly necessary.

From a practical point of view, the most useful parameters for describing a powder relate to its bulk properties. The most important of these are apparent density, flow rate and compressibility. Apparent density is usually measured with a Hall flowmeter in which a 25 cm^3 cup is filled with powder at a constant rate, the excess powder is levelled off with a spatula and the contents of the cup weighed. Flow rate is also measured with a Hall flowmeter or a Carney funnel. Compressibility is measured by pressing powder in a die with pressure applied simultaneously from top and bottom. The pressure required to achieve a specified density is a measure of compressibility. Alternatively, it can be specified as the density achievable at a given pressure. By plotting the density obtained at a series of increasing pressures, a compressibility curve is developed.

A further standard test is used to measure the dimensional change on sintering. In this test a standard sized test bar is pressed and sintered and the shrinkage (or growth) calculated from the difference between the die cavity dimensions and the final dimensions.

All of these tests demonstrate the empirical nature of powder testing for conventional PM. Yet these tests are essential for obtaining the information necessary for making a die, for controlling the consistency of the powder, and for the overall control of the process.

4. New PM processes

4.1 Powder forging

The conventional cold press and sinter method of producing parts by PM suffers from the difficulty of obtaining 100 per cent density in the final product. This has been, and remains, a substantial obstacle to the wider use of PM for highly stressed parts. One way of overcoming this problem is to carry out the final shaping operation by forging in a closed die. This not only produces a product of high density and hence high strength, but it is also a product with high dimensional accuracy. The cost of subsequent machining is therefore largely eliminated.

Interest in the powder forging process dates back to the mid-1960s. An excellent review of the history and development of powder forging has been written by G. Brown of Guest Keen & Nettlefolds (GKN). At that time it was judged that there was a large market in powder forging and significant research and development exercises were started, notably by GKN in the United Kingdom, Federal Mogul in the United States and Sintermetallwerk Krebsöge in the Federal Republic of Germany. Gears and connecting rods were seen as obvious initial candidates extending to certain automatic transmission components. By the late 1960s, it was apparent that components could be made successfully and were capable of giving good performance.

The process that was developed involves two stages. In the first, a preform is produced by conventional pressing and sintering to a relatively low density of 70-80 per cent of the theoretical. This preform is then either cooled and reheated (1,050-1,125°C), or transferred directly to a set of closed dies where it is forged to final size. To maintain dimensional tolerances, the temperature of the work, the dwell time and the die temperature must be controlled carefully.

The type of powder required for powder forging is quite different from that required for conventional pressing and sintering; a soft powder with high compressibility is not necessary because the compaction is performed hot. Fully pre-alloyed powders made by water atomization can therefore be used. Non-metallic inclusion structure and content is very important in powder forged components and considerable development has taken place to try to produce alloy powders which give manageable inclusion content while at the same time maintaining a reasonable cost. A balance must be struck between the expensive elements, nickel and molybdenum and the powerful hardenability promoters, manganese and chromium. Currently, there are three or four primary alloy specifications in use, and these are capable of generating, with varying carbon content by graphite addition, mechanical properties to suit the majority of components likely to be produced by the powder forging process.

The first mass-produced component produced by powder forging was a new design of connecting rod for Porsche in the mid-1970s. This was fully tested and was found to out-perform the best forged component: the endurance limit was raised by at least 25 p cent. The development attracted considerable attention at the time. However, it is only in the last few years that powder forging has become an accepted production process. Toyota in Japan now has two full-scale production lines to produce connecting rods and Ford has advanced plans for producing connecting rods by this route. A wide variety of other components is currently being made by about eight companies around the world; these include components for transmissions, drive trains and engines.

It is interesting that the full potential of powder forging is only now being realized, some 20 years later. There would appear to have been several reasons for this. First, the process is not inherently cheap because two die sets must be used, one for the preform and one for the final product. Second, technical difficulties were encountered, particularly with regard to internal oxidation in the heating and transfer stages. Third, developments in other materials were also taking place, particularly in SG iron, and it was possible for component manufacturers to lower their price sufficiently to prevent powder forging from becoming a serious challenge. However, probably the most important reason for the slow acceptance of powder forging was (and still is) the very large investment in existing manufacturing methods and machining lines, coupled with a downturn in economic activity. The inertia of a large-scale manufacturing operation is very considerable and a 15-20 year interval for the introduction of a radically new process is probably not unusual, a point that many enthusiastic research and development workers do not always appreciate.

Powder forging is now becoming an accepted manufacturing technique and, although the amount of powder being forged is still relatively small ($\sim$20,000 t/year), it appears to be growing. The powder producers appear to be anticipating this with reports of planned increases in production capacity of water atomized powders. A factor that is helping the move to powder forging is the increasing cost of energy; it has been estimated that powder forging energy requirements are about 50 per cent of conventional hot forging.

4.2 Injection moulding of metal powders

There has been considerable interest in the past 5-10 years in the possibility of producing metal (and ceramic) components by injection moulding technology. As yet, production of parts by this route is quite small (an estimated use of 45 tons of carbonyl iron powder in 1985), but the potential of the process is considered to be very great.

The process was invented by Raymond E. Weich in 1972, who founded a company, Parmatech, in California. Most of the development has taken place in the United States, where there are now three major companies who have developed the technology. These companies have licensed their technology to a range of other companies (including IBM) in the US, Switzerland and Japan. As in many modern technological developments, in-depth information about the process is difficult to find; there are no published papers that discuss the process in detail. The technological "know-how" appears to reside in the United States.

Essentially, powder injection moulding consists of four steps.

(1) Metal powder or ceramic powder (or a mixture of both) is mixed with a polymeric binder to make a feedstock material that acts as if it were a more or less conventional thermoplastic during the moulding cycle. The size of the particles is usually under 20 μm and preferably under 10 μm.

(2) The feedstock is moulded to the desired (oversize) shape in more or less conventional dies. All plastic forming techniques, such as extrusion, vacuum forming, roto moulding, etc., can be used to shape the feedstock.

(3) The binder is removed from the green shape, leaving behind a matrix of powder. This is carried out either by (i) dissolving out the binder or (ii) heating in a controlled manner to burn it out. The space between the particles is now empty (or full of gas) but the shape of the matrix is the same as the formed green part.

(4) The matrix is sintered at a high temperature in a well-defined atmosphere and the part shrinks to the final dimensions. The shrinkage is substantial, about 15-25 per cent; it is also predictable, controllable and substantially isotropic. The final product will have a density $>$95 per cent of the theoretical density.

The process differs significantly from conventional PM. The main difference lies in the particle size of the powder used. The conventional press and sinter process typically uses iron powder with a size of $\sim$100 μm; at this size the powder flows freely and automatic die-filling to give a uniform loose powder density is relatively easily accomplished. The properties and shape of the powder particles are also carefully chosen and controlled to give good compressibility and uniform compaction in the cold pressing operation. Typically, the density is raised from 2.4-2.8 g/cm (apparent density) to 6.5-6.8 g/cm in this operation. The final sintering at 1,100-1,150°C produces a further densification to 6.5-7.2 g/cm with an associated shrinkage of 1-5 per cent. This shrinkage is not very large but the final density is only 83-92 per cent of full theoretical density.

Very fine powders (10-20 μm), by comparison, sinter at a much higher rate because of the larger surface:volume ratio and, therefore, much higher final densities are achievable, >95 per cent, in shorter times at the same sintering temperature. Thus, there would appear to be significant advantage in using powders with a small particle size. Why then are they not used? Apart from the cost factor in producing fine powders, the main reasons are that they do not flow freely and do not compress uniformly. The powder density is, therefore, not uniform in the die after filling and even if it were, it would not remain so after cold pressing. This gives rise to non-uniform shrinkage, distortion and poor homogeneity in the final product. The main advantage of PM therefore as a near net shape forming process is largely lost.

The advantages of using fine powders, namely, near theoretical density and relatively low sintering temperatures, can be utilized if the powder is first mixed with a polymer to provide uniform flow in the shaping process. In this way, a high density product can be produced with higher strength and more uniform structure than can be produced by conventional PM.

The disadvantages are that 15-20 per cent shrinkage must be contended with, and the time necessary for removal of the polymer, prior to final sintering, can be long (several days). There is also a limitation on the size of parts that can be made by this technique; the upper limit on thickness, to permit easy binder removal, is ~10 mm. Nevertheless, despite these limitations, the process has the great advantage of being able to produce parts of more complex shape than the conventional press and sinter route, and it is this factor, together with the high production rates that are achievable with injection moulding, that gives this new method of near net shape forming its great potential.

4.3 Continuous strip production from powder

Conventional PM is a near net shape forming process: the aim is to produce a complex shape with a minimum of further processing. The powder route can also be used to produce a continuous product such as strip or sheet. In this case, the advantages of the process are that either it produces a cheaper product than the conventional manufacturing route, or, more usually, that it produces a technically superior product. In particular, the fine grain structure and near zero segregation obtained by particle technology frequently make it possible to fabricate alloys that could not be made using conventional ingot technology.

There are three different ways that are being used to make continuous strip from powder: (i) powder rolling, (ii) prebonded powder rolling, and (iii) hot powder and pellet rolling. Each process has its own niche created by its particular combination of benefits and disadvantages, although the final product in each case is remarkably similar.

4.3.1 Powder rolling

Powder rolling is the oldest and best-established process and, in principle, is the simplest. Powder is poured into the nip of a rolling mill which is usually positioned with the roll axes in a horizontal plane. It is compacted and emerges as a "green" strip which must subsequently be sintered and/or hot rolled to give an acceptable product. The apparent simplicity of the process hides a number of technical complexities which have a profound effect on its operation in practice.

The most important of these is the inability to control the size of the feed and compaction zones as the powder moves to the centre of the rolling nip: this controls the density of the green strip. The process therefore has limited flexibility as regards the thickness and degree of compaction of the green strip. A further problem is that a considerable quantity of air is eliminated from the interstices between the powder grains as they move through the feed and compaction zones. The effluxed air is vented upwards, disturbing the powder in the feed zone. The effect is greater at high rolling speeds, and this limits the speed at which green strip can be made.

Notwithstanding these shortcomings, the process is used, particularly in the United States, for making nickel and cobalt strip. It is also being tried for making titanium and the superalloys. The Sherritt-Gordon process for producing nickel strip uses powder rolling; the strip is used for making coinage blanks for the Canadian mint. It is claimed that roll compacted nickel strip has a work hardening rate 25 per cent less than for wrought nickel strip and it has a higher electrical conductivity.

4.3.2 Prebonded powder rolling

The purpose of prebonding is to enable the powder to be metered accurately into the rolls, especially in relation to distribution across the width. The process consists of (i) mixing the powder with water and a binder (methyl cellulose) to form a slurry; (ii) coating a moving belt substrate with the slurry to the desired depth (in much the same way as in paper making); (iii) drying the slurry and peeling it away from the moving belt; (iv) rolling to ~95 per cent density with the binder still present; (v) sintering in reducing atmosphere - the product is now ~80 per cent dense and quite brittle; and (vi) cold rolling and resintering followed by a final cold rolling operation.

The main advantages of the process are (i) that much higher rolling speeds are possible (~100 m/min) because there is no powder disturbance and (ii) that wide strips (1 m or more) may be made. At the same time very thin strips can be processed because it is possible to use unsaturated feed. The main disadvantages are the cost of the binder, which could be significant in a large-scale operation, and that more than one roll/sinter stage is often necessary to achieve 100 per cent density. This limits the maximum thickness of the strip that can be produced.

The process was initially developed at the BISRA Laboratories in the United Kingdom in the 1960s. It was taken up by the British Steel Corporation in the 1970s with the intention of manufacturing stainless steel strip and strip for tinplate; a pilot plant was built with an associated water atomizing plant to produce steel powder. Economic analysis had apparently shown that this route for producing strip was viable; certainly, from an energy point of view, the powder route would appear to be more economic. However, the scheme was aborted in 1980, following updating of existing rolling mill plant and the downturn in the world steel industry.

The technology was not lost, however, and a small independent company was set up in 1981 to produce special alloys that are difficult or impossible to produce by conventional methods. The company, Mixalloy Ltd., specializes in producing strips in a wide range of iron nickel and cobalt alloys. These are used mainly for welding consumables, electrical resistance alloys, and specialist alloys for the chemical industry. One of their successful products is Invar/NiCr bimetal and trimetal strip for use in thermostats, etc. This is very easily produced using the powder route: the green strip is passed through the coating stage a second time to have a layer of the second alloy deposited on top, the double layer (or triple layer) is then passed through the rest of the line. Examples of some of the alloys being produced are Co/Fe, Ni/Fe, Ni/Cr and Fe/Cr as well as pure Ni and pure Fe. Alloys produced in this way are ductile even with high amounts of alloying addition.

4.3.3 Hot powder or pellet rolling

Hot rolling powder has an immediate technological appeal because it seems to combine all of the virtues of powder processing for the manufacture of strip. By using temperatures in the region of sintering, it holds the possibility of hot compacting with a corresponding reduction in the flow stress and simultaneously sintering the product in order to gain relatively strong strip in one operation. The obstacle to this "all-in-one" concept is that metal powders at sintering temperature aggregate and stick together to form lumps. This constitutes a major problem because hot powder is prevented from flowing freely into the nip of the rolling mill.

One way of overcoming this obstacle is to use very large particles in the form of granules or pellets. The sticking tendency is a surface phenomenon and if the surface:volume ratio is reduced there will be a lower tendency to stick and agglomerate.

The first successful use of this technique was by Reynolds Metals who carried out extensive development work on the hot rolling of aluminium granules and pellets. The pellets were the size of rice grains. The use of aluminium had the major advantage in that the granules readily formed a surface oxide skin and this helped to ensure free flow and prevent sticking. However, this also created a problem in that these oxide films were necessarily incorporated into the product. More recently pellet rolling of iron has been tried by Singer. In this work, pellets of reduced very high purity iron ore, approximately 1 cm in diameter, were poured at a temperature of 1,100°C into the nip of a horizontal rolling mill having rolls 1 m in diameter and a face width of 125 mm. During preheating and during their travel to the roll nip, the pellets were retained in a reducing atmosphere and kept in constant motion. The large size of the pellets offset any tendency to sticking because gravitational and inertial forces were large compared with adhesion between individual pellets. In this case, the pellets were entirely free from oxide so that, as soon as they entered the compaction zone, they plastically deformed and sintered together completely. In the hot band their individual identity had been lost.

An essential part of this process was the need to use a roll diameter related to the pellet size such that a sufficiently large number of pellets would always be present across the entry plane of the compaction zone to ensure that the interstices of the pellet mass were completely filled during rolling. With this precaution a relatively ductile and dense hot band was produced in a single rolling pass. Subsequent hot and cold rolling could be carried out with conventional hot band.

It would appear from this work that the process of hot pellet rolling has some promise for medium scale industrial use. The economics of a direct reduction process to form granules followed by hot roll compaction would appear to be favourable, but in-depth studies might prove otherwise.

4.4 Hot isostatic pressing and hot extrusion

Hot isostatic pressing (HIP) and hot extrusion are techniques for the consolidation of powders to full density. They are mainly, but not exclusively, used in the high technology and aerospace areas of PM.

HIP is a process whereby high isostatic pressure is applied to a presintered part, or a containerized loose powder, at high temperature. The pressure is applied equally on all sides of the object being pressed using argon gas in a specially constructed pressure vessel. Pressures range from 20 to 300 MPa and temperatures from 480°C for aluminium alloy powders to ~1,700°C for tungsten powder pressing. The effect of the high pressure is to squeeze the component uniformly to close up the porosity with simultaneous sintering. Press sizes vary considerably but the largest units can process billets up to 60 cm in diameter and 300 cm long.

The first use of HIP was in the 1960s for diffusion bonding of clad nuclear fuel elements. Consolidation of beryllium metal powder "to shape" followed shortly afterwards. Since then, the size of units and use of the process has extended very greatly. Uses now include the production of titanium and superalloys for the aerospace industry, net shapes in PM beryllium and niobium alloys and other refractory metals, the production of fibre strengthened aluminium alloys, the consolidation of HSS steel parts and rare earth magnets, and the manufacture of tungsten carbide/cobalt and other carbide compositions. Nickel-based PM aircraft engine applications represent the highest technology level of the method; the highest production tonnages are obtained on tool steel and sintered carbide parts.

The production of net shape parts, such as aircraft engine turbine discs, is achieved by first making a container of the required shape and dimensions (with the necessary allowance for densification shrinkage, machining, etc.), filling with powder, evacuating and sealing, and finally HIP. The powder would normally be made by atomizing the liquid superalloy with argon gas, with all subsequent powder handling carried out under argon. The can material must be chosen so that there is no interaction with the powder. Alternatively, an encapsulated powder may be hot isostatically pressed at a low temperature to a closed porosity condition and repressed at a higher temperature after removal from the container to produce the fully dense part.

Some other materials, such as HSS, are usually made in billet form by HIP. Typical processing conditions use 1,100°C at 100 MPa for 1 hour. Tungsten carbide/cobalt tools and dies are usually first made in the conventional way by cold isostatic pressing and sintering; this is followed by containerless HIP to close up any residual porosity.

Hot extrusion combines hot compaction with hot mechanical working to yield a fully dense product. A unidirectional force component is superimposed on a large hydrostatic compressive force to make the metal flow through a die. The resulting product usually has a circular or rectangular cross-section.

There are three different ways in which a powder can be hot extruded. In the first, loose powder is placed without preheating into the heated extrusion container and extruded directly through the die. This method is used for the extrusion of certain magnesium alloy powders. No atmospheric protection is provided, and the heat of the container is used to raise the temperature of the powder sufficiently to allow extrusion.

In the second method, used for the hot extrusion of aluminium alloy powder, the powder is first cold compacted and then heated and extruded. Cold isostatically pressed components of molybdenum powder, preheated to the extrusion temperature, can also be extruded this way, without canning. Some data obtained during the development of high-temperature aluminium alloys are shown (table 2, see page 22). It can be seen that the extruded powder product is stronger than the conventionally forged material at room temperature.

The third and most widely used method is to place the powder in a can and extrude the heated can. In some cases the powder may have to be cold pressed into the can before evacuation, sealing, heating and extrusion.

Hot extrusion of powders encapsulated in cans was first developed as a method for hot consolidation of powders that are toxic, radioactive, pyrophoric or easily contaminated by atmosphere, for example, beryllium and uranium. The method is almost universally used for making copper and nickel dispersion-strengthened alloys; these include copper dispersion strengthened with aluminium oxide, and TD nickel containing thorium oxide. The yttria dispersion-strengthened nickel alloys produced by mechanical alloying can be produced in this way. A hot extrusion process for producing seamless tubing from stainless steel powder was developed in Sweden. In this process, an argon or nitrogen atomized spherical stainless steel powder is filled into preformed moulds of low carbon steel. These moulds, weighing up to 120 kg, are cold isostatically pressed at pressures of 400-500 MPa and then hot extruded at temperatures near 1,200°C using a glass lubricant. With extrusion ratios of 4:1 or higher, completely dense tubing is produced.

4.5 Some other metal powder consolidation techniques

4.5.1 Ceracon process

The term "ceracon" is derived from ceramic granular consolidation. It is a proprietary process which uses a granular ceramic medium to transmit pressure to a powder compact rather than a fluid (liquid or gas), as in a true hydrostatic pressing situation.

The processing sequence is, first, to make a preform (typically 80 per cent of full density) by any suitable PM technique, such as cold pressing, injection moulding or slip casting. This preform is then heated in a controlled atmosphere to the consolidation temperature. Simultaneously, a granular ceramic medium is heated to the same or a higher temperature. The heated ceramic and the preform are inserted into a cylindrical die so that the preform is completely surrounded by the ceramic which acts as a pressure transmitting medium. The die is then transferred to a large press where an axial force is applied. Cycle time under pressure is a matter of seconds with the ram being retracted once the desired load has been achieved. After consolidation, the ceramic granular medium and now fully dense part are separated by ejecting the contents of the die. A shaker table serves to separate the ceramic grains from the part (or parts).

The selection of the correct ceramic granular material is the most critical part of the process. Alumina-based materials with a particle size of ~140 µm (a powder) appear to be the most suitable for the manufacture of steel parts. Preheat temperatures of ~1,050°C are used and consolidation pressures of 400 MPa.

4.5.2 Rapid omnidirectional compaction

This is another proprietary pseudo-isostatic compaction process, using, in this case, a metal or glass composite as the pressure transmitting medium. The main advantage claimed is that it is fast compared with HIP.

The process consists essentially of (i) making a closed, cylindrically shaped die of the pressure transmitting medium (low carbon steel, Cu - 10 per cent Ni, or a filled glass ceramic); (ii) filling the cavity in the die (which has the shape of the part required) with powder; (iii) evacuating and sealing the die; (iv) preheating the filled cylindrical die to the compaction temperature; and (v) transferring the heated die to a forging press and compressing it axially in a pot die (the outside diameter of the cylindrical die corresponding with the internal diameter of the pot die). After removal from the forging press, the material of the die must be removed to reveal the PM part. This is achieved by machining in the case of the low carbon steel, by melting away the Cu-Ni alloy, or by mechanically breaking away the ceramic-filled glass. The process is probably better suited to parts with a complex shape; the main disadvantage is the difficulty of making the cavity in the die with the shape of the required part. In the case of the mild steel die, it is split into two halves and the cavity is machined out to the required shape; with the copper alloy and glass dies, a thin-walled can of the required shape must first be made by spinning, stamping, etc., and the copper alloy or glass is then cast around the powder-filled can.

4.5.3 Consolidation by atmosphere pressure

The consolidation by atmosphere pressure (CAP) process is a simpler version of the pseudo-isostatic compaction process. It uses glass as the pressure transmitting medium. The process consists very simply of filling an inexpensive borosilicate glass mould with powder, evacuating and sealing the mould and then placing it in a standard air atmosphere furnace used for heat treating. The glass mould softens and contracts as densification takes place under the action of the atmospheric pressure. The mould must be supported during sintering to maintain part shape; simple placement in a sand medium provides sufficient support. When sintering is complete, the moulds are removed from the furnace and air-cooled. The glass moulds are self-stripping and spall from the consolidated parts at around 315°C.

The process has clear limitations regarding the metal and alloys that can be sintered in this way; the technique is apparently used successfully for the densification of PM tool steels and nickel-based alloys.

4.5.4 Conform process

This process, invented by the UK Atomic Energy Authority in 1972, was developed initially for the extrusion of non-ferrous metals and alloys such as aluminium, copper, zinc, silver and magnesium. More recently, it has been shown that it can be used for the consolidation and extrusion of metal powders. Particular attention is currently being paid to the use of Conform for densifying rapidly solidified powders and extruding cermets such as aluminium/boron carbide (ceramic content 2.5-20 per cent).

Material fed to a Conform machine can be in the form of rod, or particulates such as powder or swarf, or some combination of rod and particulate. The material is fed by a rotating wheel having a circumferential groove to a stationary shoe containing the extrusion die (figure 4, see page 22). High pressures generated between the wheel and shoe and an associated increase in temperature cause the particles to be pressure-welded together. Bulk stress in the compaction zone is as high as 1,000 N/mm with some materials, which ensures that the material fills the groove section completely.

During extrusion through the die, large-scale deformation occurs to produce a fully dense, homogeneous product. Extrusion ratios can be up to 100:1 with soft metals, while expansion ratios through the die of up to 12:1 have also been demonstrated. Machines that are currently in use range in power from 70 kW to 450 kW, with a production capacity for materials such as aluminium being up to 2 t/h. For most applications, the flow of material through the die is radial to the wheel, but in some applications, such as in the production of long-fibre ceramic composites, the die is in the tangential mode.

In addition to producing a range of continuous product shapes by changing dies, tooling can be designed to produce individual components in cavities machined into the bore or sides of the groove in the wheel. After passing through the shoe zone, the components are ejected automatically. Typical components for manufacture in this way are electrical contacts.

5. Other new developments

5.1 Liquid phase sintering

The ideal PM process is one following the cold press and sinter route (cheaper than hot pressing) in which the sintering takes place rapidly, with zero shrinkage, at a low temperature. This ideal, of course, can never be realized in practice; to achieve it would require a powder with an infinitely small particle size. Present practice in ferrous PM is quite a long way short of this goal; there is room for improvement, even though there has been steady development in this direction over the past 20 years.

Sintered structural steel components that are being made currently have a density in the range 6.5-7.2 g/cm^3 (~83-92 per cent of full density). Shrinkage is typically 1-5 per cent during sintering. Strength and associated properties are directly related to density and, therefore, there is a strong incentive to increase final density to 7.2-8.6 g/cm^3. If this could be achieved, it would open up a large new market currently being held by forged and cast parts. The powder forging process produces parts to full density which compete with more traditionally wrought products but this process is more expensive than the cold press and sinter route. Other methods also exist for densification such as double pressing and sintering, cold forging after sintering and high-temperature sintering, but all are more expensive and most involve an additional operation.

One way to achieve increased density relatively easily without recourse to an additional densifying operation is to control the composition of the alloy to allow liquid phase sintering. Sintering in the presence of a liquid phase not only enhances densification by shrinkage, but also enhances the sintering mechanism by promoting grain growth.

The process is not new - it has been used for about 30 years in the field of cemented carbides. Tungsten-silver and tungsten-copper alloys also have long been known to undergo liquid phase sintering. Of the ferrous alloys, the Fe-Cu system has undergone extensive study and Fe-Cu alloys made by PM are used quite widely. The major problem with these alloys is that it is very difficult to control the shrinkage (or with some compositions, growth) of the compact during sintering. The mechanisms of this change in volume are not understood well.

Another way of introducing the liquid phase is by infiltration. In this method the alloy is sintered only until it forms a rigid compact containing interconnected porosity. A small block of the sintering aid, for example, copper, is placed in contact with the compact; this melts and is drawn into the compact by capillary action. The sintering aid must, of course, wet the surface of the compact. Good dimensional control and elimination of porosity are achieved using this method.

There has been considerable activity in recent years in developing alloys that undergo liquid phase sintering. The majority of these developments has been in alloys undergoing transient liquid phase sintering; in this process, the activating liquid remains for a limited time only before it reacts or is absorbed by the solid matrix. During the time that it exists as a liquid, it is able to promote grain rearrangement and densification. Examples of the type of alloy where this occurs are where the two constituent metals form a low melting point alloy or eutectic at the point where grains of the two constituents touch. The liquid formed initially at the point of contact will remain only until equilibrium is established. It has been found that iron alloys that contain phosphorus undergo transient liquid phase sintering. Probably the most successful alloy so far developed, incorporating both phosphorus and copper, is the Toyota cam lobe material. This material, which has very high wear resistance, has the composition 5 per cent Cr, 1 per cent Mo, 0.5 per cent P, 2 per cent Cu, 2.5 per cent C. It has formed the basis of the highly successful manufacture of camshafts by this company using the PM route.

Another commercially successful application of liquid phase sintering is the use of master alloys of complex carbides by companies such as Krebsöge Sintermetallwerke of the Federal Republic of Germany.

The alloy melts during sintering at 1,280°C, wets the iron particles and greatly enhances the diffusion of the alloying elements into the iron particles. A more recent study has found that titanium additions can promote transient liquid phase sintering in iron alloys; about 3 per cent appears to have the optimum effect. Successful applications of transient liquid phase sintering depend upon striking an optimal balance between the processing parameters - rate of heating, final temperature, etc. - to obtain optimum properties.

Another way in which sintering may be promoted by the presence of a liquid phase is by supersolidus sintering. This method relies quite simply on raising the temperature above the solidus to produce incipient melting. A fully alloyed powder is used. The main application has been with tool steels, nickel-based superalloys and cobalt-based wear-resisting alloys. The key process control parameters are temperature and powder composition, since these control the volume fraction of liquid present. For example, in the vacuum sintering of HSS powders for cutting tools, the temperature control has to be maintained to within $\sim$2.5°C at 1,200°C to obtain a good product. If the temperature is too low, less than full density is obtained, and if the temperature is too high, carbide formation occurs at triple points and the steel becomes brittle. When the conditions are correct, the equiaxed structure of the powder compact has a greater strength compared with the forged product tested in the transverse direction (figure 5, see page 23). This method of promoting liquid phase sintering has the strong practical limitation that the temperatures required can be outside the range of normal sintering furnaces. The upper temperature limitation on practical furnaces used in production is often $\sim$1,200°C. For sintering temperatures above this, special furnaces have to be used, vacuum furnaces or special atmosphere furnaces, etc., which are more expensive and can only be operated on a batch basis.

Another type of liquid phase-assisted sintering is reactive sintering. This is similar to transient liquid phase sintering, and is characterized by a large heat liberation due to reaction between the constituent powders. Rapid sintering results from the formation of a liquid and the self-heating due to the exothermic reaction.

A variation on liquid phase sintering is "activated" sintering. This can apply to solid or liquid phase sintering. The concept is to supply an activating agent that covers the surface and speeds up diffusion. Metal is more quickly redistributed from high energy points in the region of interparticle contact to low energy points outside the contact area. Quite large changes in surface energy and surface diffusion rate are known to occur in studies of surface diffusion, for example, the thermal etching of silver surfaces in the presence of oxygen, but there do not appear to have been any developments using activated sintering that have found practical application.

5.2 Composite materials

A composite material is made up of two quite different chemical constituents, for example, metal-ceramic, polymer-ceramic or polymer-metal. The two constituents can be considered as two separate phases, a continuous or matrix phase and a discontinuous or disperse phase; the particles in the disperse phase can have any shape ranging from spherical to long continuous fibres.

Metal powders may be used either as continuous or disperse phase in a composite. The most obvious use of PM is to use the powder as the continuous phase and to incorporate the non-metallic disperse phase into the powder before it is pressed and sintered. This approach has been adopted particularly for the manufacture of high-strength components such as used in the aerospace industry and increasingly in the automotive industry. Examples are alumina, silica and boron fibres for strengthening aluminium alloys, and the yttria dispersion strengthening of superalloys (mechanical alloying). Of course, the powder route is not the only one by which ceramic fibres can be introduced into a metal matrix; considerable effort is being devoted to producing these materials by squeeze casting and squeeze infiltration. Fibre-reinforced aluminium parts, such as pistons and connecting rods, are currently being manufactured in Japan.

Another novel PM process, developed in the UK, which can incorporate fibres or other strengthening particles into the final product, is known as COMPASS (consolidation of mixed powders as synthetic slurry). In this process, two powders of different composition are premixed, compacted, heated into the semi-solid régime and hot pressed to yield a billet with a microstructure suitable for diecasting as a semisolid. The process has not been adopted commercially as far as is known. Other specific examples where the powder route is used to incorporate a non-metallic material are bronze-polytetrafluoroethylene bearings and copper-graphite for electrical brushes.

Metal powder is being used increasingly as an addition to polymeric materials. The purpose of the metal powder is not only to act as a filler but also to improve certain properties to the plastic material. For example, considerable effort is being expended, particularly in Japan, into making electrically conducting plastic materials that may be injection moulded. This requires a particular size and shape of metal particle. The applications are for electro-magnetic interference shielding and as a surface heater. Another novel application is the filling of an elastomer with a conducting metal powder; if the correct composition, type of powder, etc., is chosen, the elastomer can act as a switch. When it is stretched, contact is lost between the particles and the resistance is high; when it is relaxed or compressed, the particles touch and the resistance is low.

Dense metal powders, for example, lead and iron, are often added to resins where dense parts are required. These are used for sound applications or for rotary parts, for example, a flywheel. Heavy metal powders are added to polyvinylchloride or to polyamide to make sound-proof sheets. The sound insulation performance of a non-porous uniform material depends upon the weight of the material. Flake powders are added to resins in small quantities ($\sim$2-3 per cent) to give a metallic colour; aluminium for example, produces a silver appearance, or brass added to aluminium powder gives a brighter appearance. Another novel use of a resin-metal composite is the manufacture of a bacteria-proof material. Copper-filled resins have a pharmacological function in killing bacteria; one example is the manufacture of copper-filled yarns for making fishing nets.

The majority of newly developed composites materials has the disperse phase in the form of fibres; the large aspect ratio gives a proportionally larger strengthening effect. Several processes have been developed recently to produce metal fibres with varying lengths and thicknesses. The methods used to produce these fibres have generally evolved from the world-wide effort to produce rapidly solidified materials. Very small diameter fibres will cool very rapidly and produce non-equilibrium structures. These fibres can either be incorporated as strengthening agents into another material or they can be chopped up and consolidated into a product with the properties of the rapidly solidified material.

The best known method of producing fibres is the melt extraction technique developed by Battelle in the United States in the mid-1970s and now licensed around the world. This process is used mainly for the production of stainless steel fibres; it has a water-cooled wheel with a multi-edged profile which is just allowed to touch the surface of a bath of molten steel (or other metal). Filaments are extracted which solidify on the wheel prior to being thrown clear (figure 6, see page 23). The length of the fibres is controlled by cross-cuts machined into the wheel, and can be varied from 1 mm to several metres. In the normal production process for stainless steel fibres, the length is typically 25 mm with a diameter of 0.1-1 mm. The current major use of these fibres is for reinforcement of concrete and castable refractories. At the plant in the UK at Fibre Technology Ltd., the production rate is up to 10 kg/min and world-wide production is estimated to be $\sim$3,500 t/year.

Several variants of this process have been developed. Battelle have a melt spinning process to produce rapidly solidified flake, about 1 mm^2 and 50-100 μm thick, which operates by squirting a very small diameter ($\sim$20 μm) stream of molten metal onto a rotating drum with a notched surface. The production facility is capable of making $\sim$4 kg/min of aluminium flake; this is used for mixing with a polymer (polypropylene in amounts up to 40 per cent) and moulded into shapes for housings that shield electronic equipment. A second application is for adding to tar in the manufacture of tar paper used for heat insulation under roofs, etc.

Another similar process, developed by Fibre Technology Ltd., is the melt overflow process (figure 7, see page 23). This operates by allowing the melt to overflow the crucible, to flow across a flat lip and onto the periphery of a rotating wheel; the rate at which the melt is fed onto the wheel is more controlled than in the melt extraction process and finer fibres can be produced with a higher cooling rate. Metal fibres produced in this way can also be incorporated into metal matrices, either by mixing the fibres with the metal powder or by incorporating the fibres into the liquid metal. There is considerable interest in producing stainless steel fibre-reinforced aluminium alloys; in Japan, Honda has developed a stainless steel fibre-reinforced aluminium connecting rod.

5.3 Rapid solidification processing

Since 1960, when Duwez first recognized rapid solidification processing (RSP) as a means of controlling microstructure, there has been a major research effort to explore this phenomenon and to develop it into a useful practical process. A large part of this effort has been at the research end of the research and development spectrum, to develop ways of obtaining extremely high cooling rates, $>10^6$°C/s, and to investigate the range of completely new structures thus obtained. It has always been recognized that it can be difficult or impossible to make materials cooled at these very high rates in significant quantities and to consolidate into a useful solid product without destroying the highly metastable structures obtained by the rapid cooling. To obtain the rapid cooling, the section of the material must be small ($<100\,\mu$m) and therefore all rapid solidification materials are either in the form of thin ribbon, thin fibre or powder. Most consolidation processes rely on heating to a greater or lesser degree to achieve bonding between particles and this will tend to anneal out the metastable structure.

Despite these difficulties, considerable progress has been made in making alloys with unique properties by this route. A major breakthrough was made by Pratt and Whitney, in 1977, when they developed a centrifugal atomizing process for making very fine aluminium alloy powder. The metal was poured onto a water-cooled horizontal disc spinning at very high speed; the metal was flung off the periphery of the disc to form fine droplets which were quenched in a stream of helium gas. The alloys made by this route (Al-Fe-Mo alloys) were found to be stronger at high temperatures than the same alloy made by the conventional route. The method used for consolidation was the conventional one (for the aero industry) of placing the powder in an aluminium can, evacuating and sealing the can and hot extruding. This method of atomization is also being used for making superalloys, with, it is claimed, much better high-temperature strength and creep resistance.

Considerable effort is currently directed, particularly in the United States, to try to make PM aluminium alloys using rapid solidification powder. The objectives are to obtain (i) low-density high-strength and high stiffness alloys based on aluminium-lithium; (ii) improved toughness and stress corrosion-resistant alloys; and (iii) improved elevated temperature alloys.

In the United Kingdom, there is a substantial research effort to produce rapidly solidified powder by gas atomizing and to consolidate the powder by various techniques such as the Conform process and dynamic compaction. In a recent report it is claimed that magnesium alloy rapidly solidified material (in the form of chopped ribbon) can be consolidated successfully by extrusion at 200-250°C. The normal temperature required for extrusion of magnesium alloys is 300-400°C.

The alternative route to gas atomizing for making rapidly solidified materials is melt spinning, planar flow casting or melt extraction. This type of process, using a water-cooled spinning disc to produce a thin ribbon or fibre, was described previously. The usual method of handling this material is to chop it up and to grind it into a powder. It can then be consolidated using the conventional methods of hot extrusion and/or HIP, or one of the newer methods that are being developed. The most interesting of these is dynamic compaction or explosive compaction. The principle is that the shock waves produced by the impact of a high-speed punch or explosive can consolidate the powder in a time of a few milliseconds. This rate of compaction allows particle interface melting and welding without allowing sufficient time for significant longer range microstructural change to take place. The rapidly solidified structure is, in large measure, preserved, while a strong, dense compact is produced simultaneously. The major disadvantage is the size of the billet that can be compacted by this means - gas guns with a diameter of 70 mm have been used but the length of the billet is usually less than its diameter. Larger sytems would be very costly.

5.4 Magnetic materials

Two classes of magnetic materials are made by PM: soft magnetic materials with a high permeability and low coercive force used for pole pieces, armatures, relay cores, etc., and hard magnetic materials used for making permanent magnets. Using PM has the usual advantages of production of final shape with a minimum of subsequent machining, and finer equiaxed structures; and, in the case of magnetic materials, the additional ability of producing desirable magnetic properties. In the case of hard ferrites and the new rare earth magnet materials based on a single phase of high magnetocrystalline anisotropy, there is no alternative to the PM route.

5.4.1 Soft magnetic materials

The soft magnetic materials made by PM are pure iron, Fe – 3 per cent Si, Fe – 0.5 per cent P, P and Fe – 50 per cent Ni. These materials can, of course, be made by conventional casting and machining and/or other formed sections (strip etc.), but for small, intricate shapes, the PM route is often preferable. With many of these alloys the magnetic properties are not much improved by making them in this way, but for pure iron parts there is an advantage.

Atomized powder is generally used; iron parts made from carbonyl iron powders have superior magnetic properties but the additional cost is not unusually justified. Magnetic properties are a function of sintered density, sintering temperature and particle size of the powder. Sintering temperatures are usually high, 1,250-1,350°C, and sintering has to be carried out in a reducing atmosphere or in vacuum.

In many cases, the use of soft magnetic alloys has been replaced by soft ferrites. These are generally based on the cubic (oxide) compound $Mo.Fe_2O_3$ (where M = Mg, Zn, Mn, Cu or mixtures of these elements) and possess a typical spinel structure. They can be made with a wide range of magnetic properties. However, they are more expensive than pure iron. They have the advantage of having a high electrical resistance and can virtually eliminate eddy current losses.

While the principal requirement for direct current applications of soft ferromagnetic materials is low hysteresis loss, eddy current losses are of primary importance for alternating current applications. One method of decreasing eddy current losses is to increase the electrical resistivity of the material. For power frequency applications, high resistance is obtained by dividing the Fe-Si alloy cores of a.c. motors, generators or transformers into laminations, each separated from the next by a thin insulating layer. The sheet for these laminations is made conventionally from silicon steel (Fe – 3 per cent Si) made by casting and hot and cold rolling. World consumption of electrical steels is ~2 Mt/year. The alloy is often subjected to a further grain orientation process to improve its magnetic properties prior to use.

It is interesting that there does not appear to have been any major effort to try to make strip for this application via the powder rolling route, although there would appear to be major technical advantages from doing so. The limiting silicon content in alloys made by casting and rolling is ~3 per cent; above this, the alloy cracks on hot rolling. Magnetic properties and electrical resistance are improved by increasing the silicon content to ~6 per cent. There is considerable interest in the possibility of using rapidly solidified amorphous alloys for this purpose. This idea has been promoted by Allied Corporation in the United States who have developed a proprietary alloy "Metglas" (92% Fe : 5% Si : 3% B) made by a melt spinning route. The ribbon has a thickness of 50-100 μm and can be laid together to form laminations. The claims for this material are spectacular, with core losses reducing from 1.5 to 0.44 Wkg. The implications of this are very considerable, with an estimated saving of ~$200 million/year in the United States. There are difficulties in processing the ribbon into insulated sheets because of the thinness and brittleness of the amorphous ribbon. It has also been pointed out that, if conventional silicon-iron sheet could be made as thin as metallic glasses without loss of preferred orientation, core losses could be as low as for metallic glasses. Another way of achieving the same result is to manufacture strips with twice the silicon content by spray deposition. The much finer equiaxed structure obtained by spray deposition can be hot rolled at the higher silicon content.

These processes of spray deposition and melt spinning are not PM processes. Powders of ferro-magnetic materials are used as core materials in applications where the frequencies are higher than power frequencies. In this case, the powder particles are insulated from one another. Cores for radiofrequency applications are usually made from carbonyl iron powder (3-9 μm); they are either treated with phosphoric acid to produce a non-conducting oxide film on the particles or a thermosetting phenolic resin is used. Iron powder cores have been largely superseded by soft ferrites. Permalloy powder cores are used at audio frequencies. These are made of 81% Ni : 17% Fe : 2% Si by a complex route involving sulphur addition to promote brittleness, comminution, coating with a mixture of sodium silicate, magnesium oxide, colloidal clay and kaolin, followed by pressing and heat treatment.

5.4.2 Permanent magnets

Since the Second World War, the most widely used permanent magnet alloy has been Alnico, which is essentially a quaternary alloy of iron, nickel, aluminium and cobalt with small additions of copper and titanium. The high coercive force of these alloys is connected with the spinodal decomposition into a duplex interconnected structure of elongated single domain particles of a strongly magnetic iron-cobalt rich phase (α_1) and a weakly magnetic nickel-aluminium rich phase (α_2).

Alnico magnets are usually made by casting into a sand mould. The alloy is hard and brittle and therefore does not lend itself to machining other than diamond grinding; for this reason, the production of small, more intricately shaped magnets is best carried out by PM.

The powder mixture used consists of about 50 per cent of the soft elemental powders (Fe, Ni, Co and Cu), mixed with a master alloy powder made by grinding an Al-containing precast alloy. The master alloy has a melting point below the sintering temperature, ~1,200°C, and provides transient liquid phase sintering. The alloy is pressed and sintered in the normal way, with sintering carried out in dry hydrogen or vacuum at ~1,300°C.

Sintered magnets have certain advantages over cast magnets; because of their fine grain size, they are stronger than coarse-grained magnets and they can be produced to closer dimensional tolerance. However, because of their slight porosity, the magnetic characteristics (of induction and maximum energy product) are not generally as good as those of cast magnets. High cobalt-containing Alnico 8 grade magnets are the most widely used sintered Alnico magnets.

The dominant position of Alnico magnets was challenged in the late 1950s by the appearance of hard ceramic ferrites such as $BaO.6Fe_2O_3$ or $SrO.6Fe_2O_3$. These materials have, in some respects, inferior magnetic properties to Alnico, for example, reduced induction and maximum energy product and poorer temperature stability, but they have the great advantage that they are cheaper and have superior resistance to demagnetization, that is, higher coercive force.

The second major change took place in the early 1970s when the first generation of rare earth (RE) - transition metal (TM) magnets were produced and immediately set new standards for high class magnets. The first commercial RE-TM magnet had the composition $SmCo_5$. Its magnetic properties were outstanding; for Alnico 8, barium ferrite and $SmCo_5$, the coercive forces were 127,300, 262,600 and $>636,600$ A m^{-1}, respectively. One of the first applications of the new material was stepping motors of analog wrist watches.

The manufacturing process of samarium-cobalt magnets is via the powder route; no other commercial method of manufacture exists. The alloy which possesses a high magnetocrystalline anisotropy, is first made up by melting and casting, and it is then crushed and milled to the required size, 6-8 μm, under an inert liquid (cyclohexane). The powder particles are aligned and compacted to a low density in the presence of a very strong magnetic field and then placed in a bag and isostatically pressed so as not to disturb the magnetic particle alignment. Sintering is carried out in purified argon at 1,150°C and this locks in the hexagonal crystal anisotropy of the particles. This is followed by a thermal treatment at 900°C to increase the coercive force, before final magnetization in a strong field of $\sim$4,774 MA/m. Following the development of $SmCo_5$ magnetic material, a number of other Co-RE compositions have been developed, based on the intermetallic compound $(RE)_2Co_5$. The development has been driven by the high cost of the raw material, particularly samarium.

In the 1980s, massive work started on the development of the second generation of RE magnets - neodymium-iron alloys. This work originated from cost considerations; the fraction of neodymium in rare earth ores, bastnasite (USA and China) and monazite (Australia, Brazil, India, Malaysia and USA), is much higher than that of samarium. Iron is also much cheaper than cobalt.

The major difficulty with Nd-Fe compounds encountered by the workers at General Motors (GM), where this work started, was the fact that Nd-Fe alloys only had good magnetic properties when in a metastable state. This led them to pursue rapid solidification by melt spinning as a means of producing the alloy. Subsequently, they had the problem of processing melt spun ribbon into magnets. These problems appear to have been largely overcome and good magnets can be made by double pressing the crushed ribbon and bonding with a resin having a high glass transition temperature.

Another approach, adopted mainly by the Japanese Sumitomo company, was based on a discovery, by Ukrainian researchers, that the magnetic phase of Nd-Fe could be stabilized by an addition of boron. The development effort in this case was aimed at finding a composition that could produce a thermally stable magnet material when processed by the conventional powder route. Current Nd-Fe materials produced commercially by both GM and Sumitomo contain boron and material compositions are centred around the magnetically highly anisotropic $Fe_{14}Nd_2B$ tetragonal phase.

The processing of Nd-B-Fe magnets is very similar to samarium-cobalt, but further stringent precautionary measures are required at all stages when handling the powder because it is extremely pyrophoric.

Nd-Fe magnets are now being used quite widely for a range of applications where their high energy can be effectively utilized. Examples are motors, computer disc drives, sensors and medical imaging equipment. The rate of introduction however is not as fast as had been predicted because of major redesign problems in changing to a new magnet material, and because the price has not fallen as predicted.

6. Conclusion

The traditional view of PM, that it is a cheap way to produce non-load-bearing components, is changing, albeit slowly. It is being established that the powder route is an economic alternative method of manufacture for many highly stressed engineering parts. Perhaps the best example of this change is the move by most of the large automotive manufacturers to producing connecting rods by powder forging. Substantial advances have been made in recent years in what may be termed this "low tech", mass-production end of the PM spectrum. Considerable scope still exists, however, for the future development of the PM process to achieve higher densities and optimum properties at reasonable cost.

At the "hi tech" end of the spectrum, PM is used to produce artefacts with critical properties for specialized application. These are usually associated with a high cost. Examples are high strength turbine discs for aero engines and high power magnets. Only some of these developments have been covered in this review; there are many others that have not been touched upon, such as superconductors, cemented carbides, refractory alloys and precious metals. It is easy to view these "hi tech" developments as the only interesting ones, where research is closest to the frontiers of knowledge and where the aim is to produce a unique material with exceptional strength, or some other critical property. This view is incorrect and is an oversimplification. Both ends of the PM spectrum, high value/small number and low value/mass production, depend on an understanding of the basic scientific and engineering principles underlying powder production, characterization, compaction and properties. Technical advances will only occur in both spheres with better understanding of these principles. (Source: Materials FORUM (1989/13), article written by N. Gane, CSIRO Division of Manufacturing Technology, Melbourne Laboratory, Cnr. Albert and Raglan Streets, Preston, Vic. 3072, Australia)

TABLE 1

North American metal powder shipments

Metal	1983 (tons)	1984 (tons)
Iron and steel	184 000	216 000
Stainless steel	2 400	2 500
Copper and copper base	19 400	21 600
Aluminium	28 000	37 000
Molybdenum	1 900	2 600
Tungsten	1 500	2 100
Nickel	12 700	15 800
Tin	800	1 000
Total	252 500	300 700

TABLE 2

Mechanical properties of an Al-10% Fe-V-Si alloy

Temperature (°C)	Yield stress (MPa)	UTS (MPa)	Elongation (%)
Extrusion consolidated powder			
25	529	561	8 3
177	438	453	5 0
202	404	416	5 0
288	336	351	8 7
343	268	286	7 6
Forged			
25	452	500	7 6

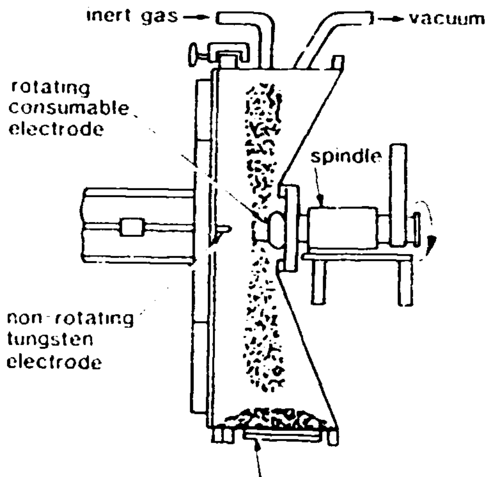


Fig. 1 Rotating electrode process for the production of powder of reactive metals such as titanium.

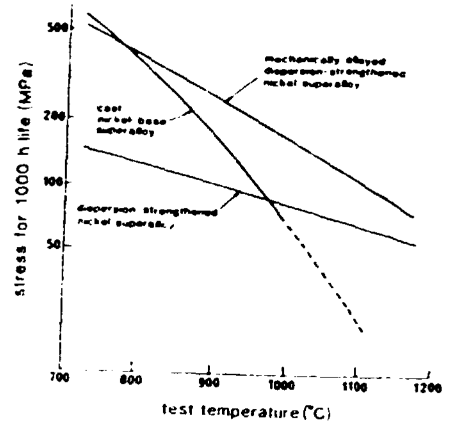


Fig 2 Stress rupture strength of mechanically alloyed nickel superalloy compared with conventionally made superalloy (Courtesy of Huntingdon Alloys)

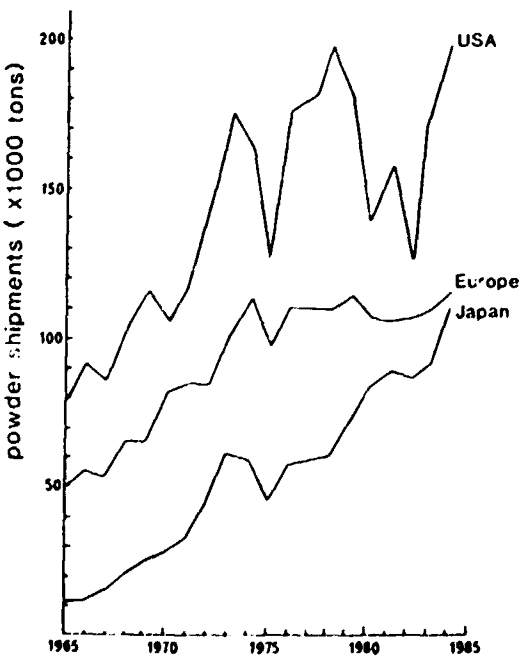


Fig. 3 Total iron powder shipments in North America, Europe (except USSR) and Japan (After Lindskey and Arbstedt)

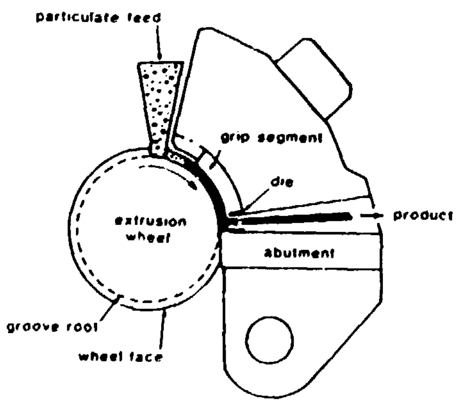


Fig 4 The Conform continuous extrusion process

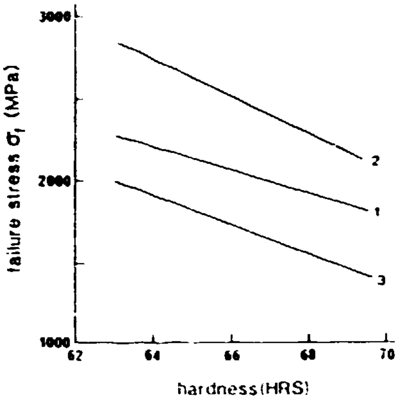


Fig. 5 Four-point bend test data for forged and fully dense T15 HSS powder compacts [11] 1: T15 sintered, 2. T15 wrought longitudinal, 3. T15 wrought transverse (Data courtesy of Powdrex Ltd.)

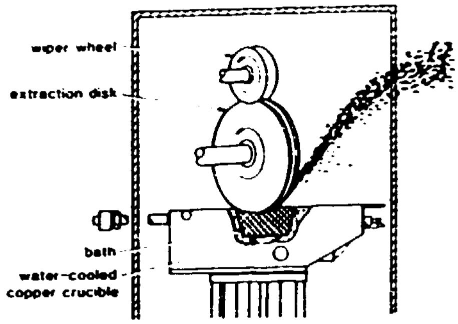


Fig 6 Schematic diagram of Battelle's crucible melt extraction process

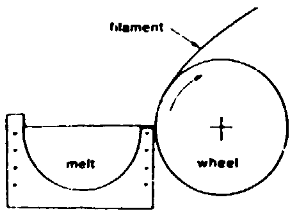


Fig. 7 Melt overflow process. Molten alloy is displaced over the lip onto a multi-edged wheel. Fine filament and fibres down to 25–30 µm thick can be made by this process.

* * * * *

3. COMPLEX COMPONENTS VIA "NEW" TECHNIQUES

Powder metallurgy is not a new process. It owes its origins to the invention, in the early 1900s, of tungsten lamp filaments. But recently there has been a growth in the versatility of the process as a result of new ways of compacting the powder forms. Several of the methods are adaptations of techniques in use with other manufacturing processes.

Powder metallurgy is a production process on the move.

Among new processes being applied are a number developed for other uses - in particular rotary forging, conforming, explosive compaction and, perhaps most significant, injection moulding. There are also advances in isostatic pressing, in one case by adapting the core blowing techniques used in making sand casting cores to pre-forming parts. One result is that new composites and ceramics are being moulded.

The basic powder metallurgy process involves pressing metal powders into a wide range of near-net finish shapes followed by sintering. In the first main stage (see diagram from a booklet produced by the British Powder Metal Federation) (see page 25) a mixture of metal or non-metal particles is compacted into a precision mould. The "green" moulding is then sintered in a controlled atmosphere furnace to bond the particles.

Powder metallurgy is mostly used in large batch or volume production for components weighing less than 2.5 kg, although larger parts have been produced. The complexity of shape has increased greatly, and the process enables production of parts in materials such as carbides which are difficult to process by other methods. It is also a viable alternative to processes such as machining, casting and forging.

Chief among its benefits are: the elimination, or minimizing, of machining by the production of near-net-size parts; the achievement of close tolerances; and the possibility for combining features that would otherwise require a sub-assembly. Its suitability for making parts impregnated with lubricant or low melting point alloys, and for being sealed with resins, is well known.

By the conventional route, powder metallurgy achieves 9? to 97 theoretical density, depending on the powder material. Improving on this figure to achieve maximum density and freedom from porosity is, along with the production of more complex parts, one of the main aims of most of the metallurgy developments taking place. Some are still in the laboratory. Others are making production components.

The new process attracting most interest is injection moulding. Limited batch production is currently being carried out in about 30 companies in the United States, also by Degussa in the Federal Republic of Germany. Development work is being done in the United Kingdom at the BNF Metals Technology Centre and at the Loughborough University of Technology. The process uses both the principles and the machinery of plastics injection moulding, producing small precision components (up to 200 g) to the same level of complexity.

Those who are working on the application of injection moulding see it as having enormous potential for markets in metallic and non-metallic (ceramics and cermets) precision components. One estimate is that the market in 1995 will be worth between half and one billion United States dollars world-wide. Such predictions are put into perspective by the findings of a United Kingdom Department of Trade and Industry-sponsored mission sent last year to the United States.

With the injection moulding in standard injection moulding machines, "green" components of metal, ceramic or carbide are formed from a mixture of the powder plus lubricant, thermoplastic plasticizer and binder. The resultant plastisol is able to flow like plastic when injected into a closed cavity. The pressurized injection of this plastic-like material provides greater freedom in the design of shapes which can be formed.

After compaction, the binder is removed from the green component either by solvent extraction or thermal debinding (depending on the type of binder) and is carried out in the same furnace as the sintering process which then follows. The process requires finer powder than normal for powder metallurgy if the sintering is to be done at regular sintering temperature; also the debinding process can be lengthy, anything up to 20 hours.

Successful production by injection moulding, to achieve uniform shrinkage, depends on a suitable powder mixture and a carefully controlled production process, particularly debinding. Shrinkage with injection moulding is high, in the region of 20 to 25 per cent but there need be little distortion, and densities approaching 100 per cent are possible. Claims regarding accuracy vary between ± 0.3 to ± 0.75 per cent and possibly 0.1 per cent with care.

Another production process being adapted is rotary forging. As a metalforming process, rotary or orbital forging depends for its forming action on an inclined cone being rolled round the surface of the workpiece to cause its progressive deformation. The relatively small and continuous changing area of contact using this process means the forces required to form metal are much less than with direct hit forging and stamping processes.

It is this benefit of rotary forging which is directly transferred to the process of compacting powders in powder metallurgy. The forces needed during compacting to achieve high densities are one-tenth those needed for normal single-axis compaction.

The process is already being exploited commercially in the rest of Europe for densifying preformed green components. But it is now also being applied to compacting loose powders as a result of SERC-funded work which has been going on at Nottingham University for the past two years. The result has been the invention of a method for consolidating loose powder that is significantly faster than with sintered preforms. Densities of better than 96 per cent have been achieved in a range of non-ferrous powders.

Nottingham has also done work on rapidly compacting preforms to full density using rotary forging. For example, compacts such as spiral bevel gears have been produced from preforms in three to five seconds. Using preforms, the total process is more lengthy because there is preforming and additional sintering. The benefits are in the high density achieved, ease of handling and higher strength of the component during forming.

Another compaction process which is widening the possibilities for powder metallurgy is the Conform process. This is an extrusion technique invented in 1971 by the UK Atomic Energy Authority for forming continuous lengths of simple high quality solids and hollow sections in non-ferrous metals from rod. More recently it has shown possibilities for handling more complex materials, including composities. For powder metallurgy, its attraction is that it also accepts metal particles, granules and powders as raw material.

The principles of the Conform process depend on heat-generating friction between the enclosed billet or powder material and a rotating wheel, as together they move towards a fixed abutment and extrusion die. In the case of powder materials, the resultant temperature rise is enough both to flow the material through the aperture of the die and to pressure weld together the particles to form a dense and homogeneous extrusion.

As a powder metallurgy process, Conform is beginning to show advantages over other powder metallurgy processes as an alternative route for handling new generations of difficult-to-process advanced materials which are themselves the results of developments in powder metallurgy. According to work which continues at the UKAEA, these centre around rapid solidification processing, mechanical alloying and metal matrix composite reinforcement.

Rapid solidification processing produces metal powders having improved structural and metallurgical properties as a result of rapid cooling. The resultant powders are very small (a few microns). To have any practical use they must be consolidated by powder metallurgy. But with the temperatures generated by conventional methods, the benefits gained by rapid cooling could be lost. The relatively low temperatures of the Conform process, together with the high mechanical deformation which takes place, make it a better option.

A method to benefit from Conform is mechanical alloying. In this the composite powders are formed by a mechanical process of repeated fracturing and cold welding. Each resultant particle has a lamellar structure containing all the alloying constituents in correct proportion. It is a commercially acceptable method of mixing refractory oxide powders into nickel-based super alloys. Experiments in using Conform as a means of consolidating the powders into usable forms are proving encouraging.

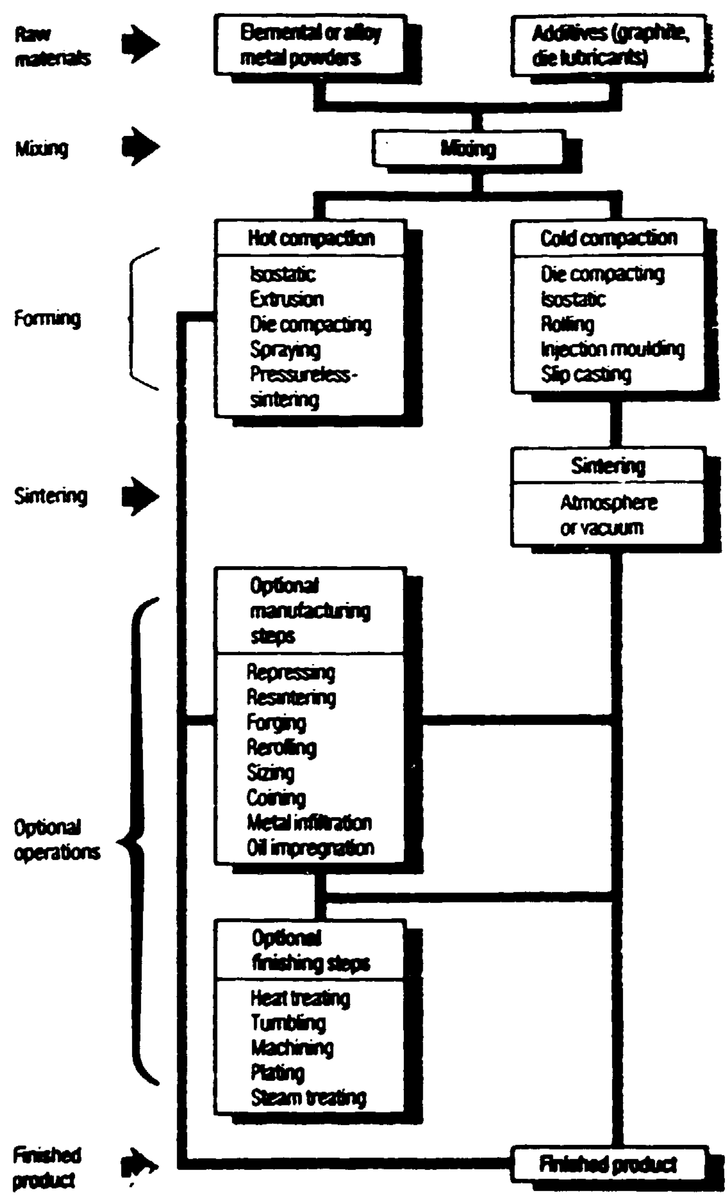
Powder metallurgy is being used today to obtain components with wrought properties. The basic approach is to re-compact preformed green to the final shape and density by isostatic pressing either at room temperature or elevated temperature. With isostatic pressing, a flexible membrane or hermetically-sealed container surrounds the powder mass and is subject to uniform external pressure from a surrounding fluid to achieve isotropic compaction of the contents.

A new method developed by the Technological Institute of Copenhagen for producing isostatic pressing preforms that are more complex than those formed by normal pressing, is based on the core blowing process used for producing sand cores in the foundry industry. The steps involved are: mixing of the powder with three per cent by weight of binder; core blowing the mixture into a core box using a strong jet of air; curing the binder in the mould and releasing the preform; isostatic pressing of the form followed by sintering.

Explosive compaction of metal and ceramic powders has been experimented with since the 1960s. Industrial applications include the production of synthetic diamonds and the compaction of heavy metals. One of the aimed-for benefits of the process is maximum density of green component. The Nobel Explosives (NEB) Research Centre in Belgium has achieved compaction of 100 per cent for aluminium.

Such high density minimizes shrinkage during sintering, and this opens up possibilites for machining difficult materials in their green state.

A further attraction of explosive compaction is its ability to compact powder that has low compatibility, for the formation of cermets (combinations of ceramics and metals) for example. There have also been experiments at the NEB with the explosive compaction of metal matrix composites. (Extracted from Machinery and Production Engineering, 20 April 1988)



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4. PERFECTING POWDER METALLURGY

Powder metallurgy techniques always have provided a means to produce cost-effective parts with good microstructural homogeneity and material integrity - excellent advantages over other metal-working methods. Although low cost still is an important criterion in the selection of PM parts over other materials, customers also want higher performance and improved quality.

Higher, more uniform properties relate to higher density parts, which require improved powders and better process control. Powder producers are providing more than a stable powder supply: today's powders are much cleaner from a metallurgical standpoint than ever before. They have greater lot-to-lot consistency, and properties in general are improved. This is especially true of machinability.

Providing what the customer wants

For years PM manufacturers have been telling customers what their industry is capable of providing. Now, customers are telling the PM industry what they must provide to sell their parts.

For example, auto manufacturers are currently creating new procurement procedures that will change the role of the buyer and increase the involvement of the supplier of PM parts. Buyers will have extensive upfront supplier plus engineering involvement. In addition, contracts will be awarded on a quality/delivery/target-cost basis instead of a cost/competitive-bid basis, and problem prevention will be more important than problem solving.

On the supplier side, PM manufacturers must meet the challenge of obtaining vendor approval via engineering expertise (providing assistance to PM parts designers and solving problems promptly) and consistent product reliability. Consistent material quality is paramount: improved product integrity (elimination of cracks), improved dynamic properties, better carbon control through improved furnace regulation, and better lot-to-lot consistency for improved machinability. PM manufacturers must begin thinking in terms of producing low-cost, high-integrity parts rather than low-cost or high-integrity parts.

There is an increasing need by design engineers for dynamic-property data on pressed and sintered ferrous materials for applications involving more highly stressed components. The impact energy of porous steels is much lower than that of fully dense (wrought) materials, which precludes a direct comparison of the two. Thus, impact-energy values of porous steels cannot be used in part design the same way wrought impact values are used. They can, however, be used to compare different porous steels, and also can be used as a benchmark with other materials that are performing satisfactorily in similar parts.

Work at Hoeganaes Corp., Riverton, N.J., characterizes some commonly used commercial PM alloys with respect to impact and fatigue properties. Test results are from materials tested under laboratory conditions, and their application to part design must take into account the effect of potential flaws in PM production parts such as low-density areas and ejection cracks.

Test results show the most important factor affecting fatigue and impact properties for any PM material, heat treated or not, is sintered density. Properties at any density are virtually unaffected by sintering between 1,120 and 1,260°C (2,050 and 2,300°F), and impact energy is not affected by a double pressing/double sintering process if the process is not used to increase the final sintered density.

Of the materials tested, only carbon-free phosphorous steel had an impact-energy transition temperature between room temperature and 0°C (32°F), which suggests the need for caution in applying this material at "normal" density levels. Increasing density by double pressing lowers the transition temperature to between -10 and -20°C (15 and -4°F). By contrast, the impact energy of Distaloy 4800A (< 0.01C-4Ni-0.5Mo-1.6Cu) with 0.4 per cent graphite, at either the single or double-pressed density level, is insensitive to test temperatures down to -50°C (-58°F). Because the transition temperature of phosphorus steels can be a critical factor in PM part design, low-temperature impact testing should be one of the criteria for PM materials selection.

Unnotched impact energy of heat-treated steels is less than 13 J (10 ft/lbf). Heat treatment of Distaloy 4600A (< 0.01C-1.8Ni-0.5Mo-1.6Cu) and 4800A with varying graphite additions consisted of austenitizing between 800 and 840°C (1,475 and 1,550°F), quenching, and tempering beween 180 and 230°C (350 and 450°F). Whereas lowering the graphite addition from 0.8 to 0.4 per cent in as-sintered material produces modest improvements in impact energy, lowering the graphite from 0.6 to 0.2 per cent does not improve the impact energy of heat-treated steels. Based on these results, going to higher carbon contents that provide higher tensile and fatigue strength would be advantageous.

The highest fatigue strength (380 MPa, 55,000 psi) was obtained from quenched and tempered Ancorsteel 4600V (0.02C-1.8Ni-0.5Mo) with 0.6 per cent graphite, double pressed/double sintered to 7.25 g/cm^3 (0.26 lb/in.^{3}) density. Single-pressed, as-sintered Distaloy 4800A with 0.4 per cent graphite produced the best combination of toughness and fatigue endurance limit, 21 J and 255 MPa (16 ft/lbf and 37,000 psi), respectively.

Air-hardening alloys based on Ancorsteel 4600V and 2000V with two per cent Cu and one per cent graphite have good tensile strength, but only modest fatigue and impact properties in the sintered and stress-relieved condition. The shape of the S/N curves indicate these materials are more suitable in applications involving low-cycle fatigue. Shot peening the fatigue-specimen surface of these steels increases the endurance limit.

Rotating bending fatigue tests generally yield higher endurance-limit values than uniaxial fatigue tests on the same material. Researchers are not certain which test method is more applicable to part design. Although uniaxial tests are more flexible in stress ratio and give more conservative results than rotating bending fatigue tests, the latter often closely simulates operating conditions of rotating parts. Porous materials probably should be tested to 5×10^7 cycles rather than the standard 10^7 cycles since some samples that were considered "runouts" ($N=10^7$ cycles) contained stable fatigue cracks. Also, researchers say stress intervals for fatigue testing should be chosen in terms of absolute stress values rather than a fraction of the ultimate tensile strength.

Selecting the right heat treatment

Hardening treatments for ferrous PM parts are difficult to control and can result in a considerable amount of scrap parts; this diminishes the cost-saving advantages of using PM parts. Most heat-treating problems are said to arise due to oversight during PM design and purchasing stages. Density and carbon content are key factors to control if increased toughness and fatigue strength are required in heat-treated parts. At density levels over 92 per cent of theoretical, these properties increase exponentially, however, they decrease with increasing carbon content. Optimum carbon content should be lower in the as-sintered condition if the part will be heat treated to obtain maximum strength. Also, fatigue ratios for as-sintered materials should not be used if parts are subsequently heat treated.

Porous steels are sensitive to cyclic stressing and the morphology of the porosity is a limiting factor to improvements in fatigue strength. Fatigue cracks usually are initiated at sharp fissures in the pore network. Density is not as important as surface carbon content and interconnected surface porosity if wear resistance is the primary requirement.

The most common hardening treatment for PM parts is carbonitriding, which is recommended for lower density parts with low alloy content. Nitrogen and carbon diffusion reduce the critical cooling rate, allowing a more uniform martensite transformation to occur. Carbonitriding usually provides the most uniform hardness and wear resistance. Excessive nitrogen diffusion must be prevented since this can lead to erratic changes in size and part distortion.

Large parts (relatively massive cross-sections), repressed to densities over 7 g/cm^3 (0.25 lb/in.^{3}), generally are carburized above 870°C (1,600°F) for one hour minimum to stabilize surface-carbon content. As-sintered carbon content should be low for repressibility, and the material should have some alloy content for hardenability. A diffusion cycle is recommended for case depths over 0.8 mm (0.030 in.).

Induction hardening requires a very uniform chemical composition (especially at surfaces to be hardened) and a uniform density. Uniform composition is difficult to control since surface-carbon content can vary with sintering conditions, and with the condition of the heat-treating equipment. Porosity tends to insulate the part due to air gaps, which requires the use of higher frequencies and more energy consumption than for wrought materials.

Will intermetallic compounds ever fly?

Iron, nickel, and titanium aluminides are touted as the much needed next generation of high-temperature, oxidation-resistant materials for turbine applications. According to some researchers, the great interest in aluminides stems from the failure, so far, of ceramics to live up to their promise, and from the fact that superalloys appear to have reached their upper limits of performance.

Intermetallic compounds based on aluminium have attractive characteristics including relatively high melting points, low density, high strength, good corrosion and oxidation resistance, non-strategic element composition, and relatively low cost. In some cases, these materials have a unique characteristic of increasing strength with increasing temperature. These are ideal properties for a material used in high-temperature applications.

Unfortunately, major roadblocks to the commercialization of aluminides are poor processibility and low ductility; processibility is said to present a major challenge to make these materials viable for any application. Conventional casting results in chemical inhomogeneities, which leads to more severe problems than normally exist with conventional alloys since aluminides are very sensitive to compositional modifications. Importantly, recent research shows some intermetallic systems have some ductility, which has generated even more interest in these materials.

Powder metallurgy techniques - always an attractive processing route for materials difficult to process by conventional methods - are ideal for fabricating complex shaped, intermetallic compound alloys. PM methods under evaluation include hot isostatic pressing (HIP), vacuum hot pressing (VHP), injection molding, transient liquid-phase sintering, reactive sintering, and hot extrusion.

Research at Rensselaer Polytechnic Institute, Troy, N.Y., is aimed at using nickel-aluminide (Ni_3Al) intermetallic compound as a matrix for high-temperature composites. Concerned with processing effects on microstructure and properties, researchers are using reactive sintering, a novel low-temperature process, to fabricate the intermetallic matrix at temperatures as low as 500°C (930°F). This is the first step in a program, supported by Defense Advanced Research Project Agency (DARPA) through the Center for Design, Analysis and Fabrication of Innovative High Temperature Structural Composites, with the goal of fabricating a metal-matrix composite ceramic fibres in an intermetallic matrix. Since processing difficulties are expected from thermal expansion mismatches and interface interactions between the reinforcement and matrix, low processing temperatures are advantageous.

Reactive sintering involves a transient liquid phase; the reaction occurs above the lowest eutectic temperature in the system, yet at a temperature where the compound is solid. A transient liquid forms at the lowest eutectic temperature and spreads through the compact during heating. The reaction is nearly spontaneous since heat is liberated due to the thermodynamic stability of the compound's high melting temperature.

A temperature over 550°C (1,020°F) is required to optimize the reaction of nickel and aluminium powders. Higher temperatures are not necessary because the reaction is nearly complete around 600°C (1,110°F); maximum heating (150 K) from the exothermic reaction is sufficient to reach the lower eutectic temperature of 640°C (1,184°F). The reaction is spontaneous during heating and is independent of final temperature and holding time.

Processing time is short - about one-half hour total. Densities over 97 per cent of theoretical are obtained, and the product has good strength and some ductility despite containing residual porosity. Properties also are retained after high-temperature exposure.

Hot extrusion was studied in joint research by Case Western Reserve University, Cleveland, and NASA Lewis Research Center to evaluate the potential of this process for hot consolidation of prealloyed aluminide powders (FeAl, NiAl, and Ni_3Al). The process consists of canning the powder and extruding at a temperature and area-reduction ratio that are high enough to produce adequate material flow and efficient filling of interparticle spaces. This eliminates porosity and causes grains to recrystallize dynamically.

Air atomization produces a wide size distribution of particles with a very tenacious aluminium-rich oxide layer on particle surfaces. The oxide layer plays an important role in the evolution of the microstructure during and after hot consolidation. Since oxide particles act as barriers to grain growth, recrystallized grains can only grow to the extent that the oxide barriers allow. The oxide envelope seems to be very sensitive to extrusion temperature and ratio and to alloy composition.

Overall grain size is slightly larger and size distribution is more uniform for a 16:1 extrusion ratio than for an 8:1 ratio. This possibly is due to a more effective break-up of oxide at the higher reduction ratio. Grain size also increases at higher extrusion temperatures, which may be due to aluminium-oxide dispersion dissolution/coarsening. Microstructures of aluminides containing alloying elements, e.g. NiAl-1 at %Ta, are typically finer grained than those of binary alloys since extrusion breaks up the second-phase interdendritic segregation and the second-phase particles act as additional grain-growth inhibitors.

Compared with extruded castings of similar compositions, much finer grain sizes are obtained in extruded powders. For example, the grain size of extruded cast Fe-40 at %Al (extruded at 980°C, 1,790°F at a 16:1 reduction ratio) is about 103 μm compared with 20 μm for extruded powder.

Grain growth in re-extruded samples is similar to that occurring in initial extrusions: lower extrusion temperatures produce finer grain sizes. For example, when samples (initially extruded (8:1) at 980°C, 1,800°F) are re-extruded (6:1) at 800°C (1,470°F), they have a grain size of 12 μm, while re-extrusion at 1,080°C (1,980°F) produces a grain size of 25 μm.

Some of these extruded alloys also have texture in the dynamically recrystallized grains. Powder processed B2 alloys (Ni-45 at %Al and Fe-40 at %Al) have a strong $\{111\}$ and $\{110\}$ texture, while Ni_3Al has only a mild texture of the same type. The $\{110\}$ texture of cast plus extruded Fe-40 at %Al is much stronger relative to the $\{111\}$ texture in the extruded powder alloy. Extruded texture is due to preferred orientation for grain nucleation and growth during dynamic recrystallization.

Grain growth during heat treatment of extrusions is complicated and is sensitive to the same factors that control extruded-grain size. Abnormal and directional grain growth often occurs at high temperatures, while little or no grain growth occurs at low temperatures.

Although not widely used in the United States, gaseous ferritic nitrocarburizing is considered to yield the most reproducible and uniform properties. The process consists of nitrogen and carbon diffusion into the surface at a temperature below the martensite start (M_s) temperature - volume changes resulting from this transformation cause most of the PM-part distortion problems. Process temperatures in the range of 570 to 650°C (1,050 to 1,200°F) are below those recommended for safe furnace operating control, which require very tight adherence to process control and equipment preventive maintenance. The hard epsilon-nitride white layer provides maximum wear resistance compared with other treatments; however, the hard thin layer cannot support heavy cyclic or impact loading.

High sulphur boosts machinability

A key advantage to making parts by powder metallurgy techniques is minimization or elimination of machining; however, finish machining operations, such as threading, turning, drilling, tapping, or surface grinding, are sometimes necessary. The machinability of sintered PM steels, especially high-strength steels like Mn-Cr-Mo or Ni-Mo steels, is poor due to porosity, hardness, and low thermal conductivity. Although beneficial for self-lubrication, porosity has a detrimental effect on machinability. It causes a constantly interrupted cut and accelerates tool wear; this results in excessive tool costs and poor surface finish.

Free-cutting steels (wrought) were developed to obtain higher metal-removal rates and longer tool life. These steels have a high sulphur content with a manganese content high enough to ensure that all the sulphur is tied up in the form of manganese-sulphide (MnS) inclusions. Low-carbon grades of these ingot-metallurgy steels typically contain 1.2 per cent Mn, and have a Mn/S ratio of 3.0 to 4.0. This approach has been used to a limited extent in powder metallurgy, but until recently, the Mn/S ratio of commercially produced iron powders was considerably less than that of free-cutting steels.

Powder metallurgy techniques allow the incorporation of machinability-enhancing additives either by prealloying or blending with the powder mix (admixing) prior to pressing and sintering. It is important that the additive(s) do not affect mechanical properties or PM part growth during sintering.

Several studies show that increased sulphur content significantly improves the machinability of PM parts. Research at Elkem Metals Co., Niagara Falls, N.Y., shows as low as 0.5 per cent high-purity MnS addition to the blended iron-powder mix prior to pressing and sintering improves machinability without any loss in mechanical properties or stability during sintering. Machinability, based on drill tests, is increased by 4 to 16 times compared with unresulphurized steel, depending on throughput and drilling parameters.

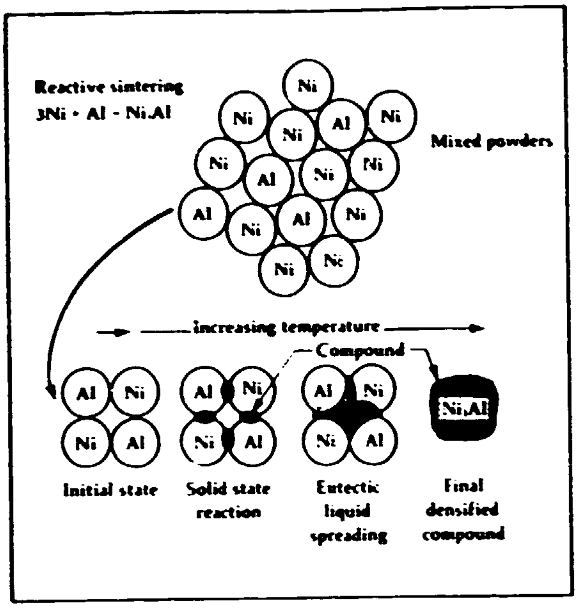
MnS helps reduce tool chip friction and lowers the cutting temperature and cutting forces, producing less tool wear. Another advantage is the presence of MnS particles near the part surface, which can aid surface finishing. In addition, MnS is stable in furnace atmospheres at the sintering temperatures; it has no detrimental effect on the muffle, belt, or furnace walls.

Researchers at Sumitomo Metal Industries Ltd., evaluated the machinability of high-strength 4100 sintered steel (0.89Mn-1Cr-0.2Mo) with different amounts of sulphur added by prealloying or admixing with MnS powder. Tool life of tungsten carbide in turning tests increases with increasing sulphur up to 0.2 per cent S content regardless of the method of sulphur addition. High-speed tool steel life also is better at higher sulphur contents; the best life is obtained when sulphur is added by prealloying.

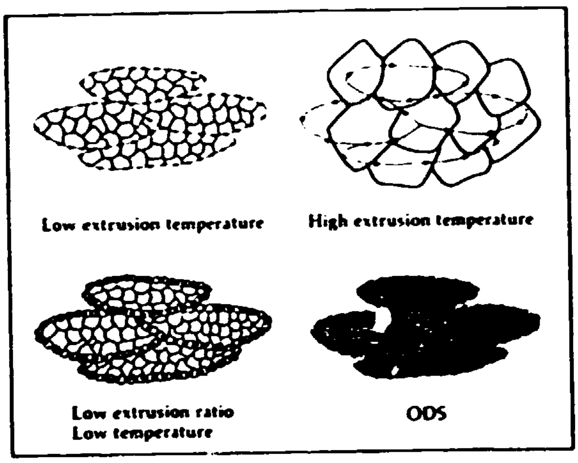
Machinability also is affected by MnS inclusion size, and by the hardness, microstructure, and Mn content of the sintered part - all of which depend on the method of addition. Hardness remains constant in prealloyed and admixed-powder parts regardless of sulphur content when Mn is increased proportionally to increasing sulphur. Hardness decreases with increasing sulphur content in prealloyed material when Mn content is kept constant due to decreasing hardenability. Tensile strength decreases with increasing sulphur content over 0.05 per cent. Neither increasing sulphur content nor method of addition has any effect on the hardness of quenched and tempered parts, but the tensile strength of admixed material is lowest.

Joint research at Domfer Metal Powders Ltd. and Genie Metallurgique, Ecole Polytechnique, Montreal, Canada, and Powdertech Associates Inc., North Andover, Mass. shows the machinability of prealloyed iron powder with a higher Mn/S ratio (4 versus 1) is improved when mixes are adjusted to compensate for different strength levels. Material with a Mn/S ratio of 4 (0.94Mn/0.236S) has significantly higher transverse rupture strength than material with a Mn/S ratio of 1 (0.38Mn/0.38S), which is due to improved sintering characteristics, i.e. more extensive closure of initial particle boundaries. Reasons for the different behaviour of powder surfaces during sintering is being evaluated.

The higher Mn/S ratio produces a much larger number of manganese-sulphide and manganese-oxide inclusions (inclusion aggregates larger than 10 μm), whereas at a lower ratio, more hard silicate inclusions are present. In this work, the material with an Mn/S ratio of 1 had slightly better machinability, which may be due to weaker interparticle bonding after sintering. This is consistent with its lower mechanical strength, and therefore would require lower cutting force for material removal; hence, better machinability. In spite of this, when adjusted to compensate for the difference in strength, iron-powder mixes with an Mn/S ratio of 4 are expected to provide an improved powder for the industry. (Source: Advanced Materials & Processes, September 1987)



In the reactive sintering process, solid-state interdiffusion generates intermetallic compound phases and some self-heating as the powder mix is heated to the first eutectic temperature. At this point, a eutectic liquid forms and spreads rapidly throughout the structure, consuming the elemental powders. Solid Ni_3Al, precipitated behind the advancing liquid interface, is nearly fully densified and is suitable for containerless HIP to full density.



Schematic illustration of the possible effects processing parameters have on hot-extruded microstructures. As-extruded grain size is related to powder size, extrusion temperature and ratio, prior particle-boundary oxides, and to the presence of dispersoids, such as in ODS alloys.

* * * * *

5. CLEAN PM SUPERALLOYS

High strength and near-net-shape capability make powder metallurgy (PM) processing an attractive option for producing superalloy components.

First applied to superalloys in the 1970s, powder metallurgy (PM) processing techniques are now routinely used to produce high-strength alloys with large amounts of strengthening elements. Severe macrosegregation and ingot cracking would make such compositions impossible to produce using conventional ingot metallurgy (IM) techniques. Other advantages of PM processing for superalloys include improved microstructural and chemical homogeneity, and the potential for cost savings resulting from greater materials utilization and near-net-shape parts processing. Contamination during processing, however, is a problem for PM superalloys. Driven by the need for components with enhanced strength and reliability, improvements in cleanness in all phases of PM superalloy production are continuing. Much current research is focused on production of clean, defect-free powders, processes have been developed for production of clean starting stock, and atomization and defect detection and removal are becoming increasingly important. Powder consolidation processes also are being optimized to introduce minimal contamination into superalloy components.

A delicate balance

With their complex, multi-component compositions and equally complicated microstructures, PM superalloys are especially susceptible to contamination. Defects and inclusions serve as crack initiation sites in service, eventually leading to failure. Defects are particularly damaging to low-cycle fatigue properties and toughness. The most common defects that occur in PM superalloy components are voids and pores, metallic inclusions, ceramic particles or agglomerations, and prior particle boundary (PPB) segregation.

Cleanness begins with the starting material. Vacuum induction melting (VIM) is the most common melting method, although other techniques, including electroslag remelting (ESR) and electron beam cold-hearth refining (EBCHR) also are used. VIM has the advantages of minimal loss of alloying elements due to oxidation and volatilization, very close composition control, accurate temperature control, and removal of tramp elements and dissolved gases. Superalloy ingots melted using EBCHR also are nearly free of non-metallic inclusions and trace elements. Regardless of the melting method used, choice of refractory is important in minimizing ceramic inclusions.

Refractory inclusions are completely eliminated in "ceramicless" melting, in which melting occurs in a water-cooled copper crucible. Plasma arc melting usually is used with this method. After melting, the starting material pours over a spout in the crucible and is atomized by inert gas.

Inert-gas atomization is the most widely used powder production method. Argon atomization results in nearly spherical powder particles with smaller "satellite" particles attached to their surfaces. Particle size depends on melt superheat, the melt head in the tundish, gas velocity, and gas-nozzle and jet configuration. Particle size, size distribution, and cooling rate are controlled by adjusting atomization parameters.

Regardless of the atomization process used, emphasis is placed on obtaining the highest possible yields of fine (50 μm) powder. A technique recently developed to try to maximize production of fines is ultrasonic gas atomization (USGA). A variation of conventional inert-gas atomization, USGA uses the basic gas atomization nozzle configuration. Shock-wave resonators fitted into the inert-gas nozzle are used to generate pulsing gas jets at an ultrasonic frequency of 60 to 120 Hz. The pulsating gas jets break up the molten metal stream more rapidly and into smaller droplets than conventional inert-gas atomization, resulting in production of finer, more uniform powders. Morphology of powders produced using USGA is very similar to conventional inert-gas atomized powders. After several years of development, the technique is nearing commercial application.

Results obtained using USGA have been mixed, with some studies indicating a higher yield of fines than conventional inert-gas atomization. Fine-particle yields as high as 95 per cent have been reported, although other investigations indicate a conventional inert-gas atomization system optimized for production of fine superalloy powders yields particle size distributions virtually identical to USGA. Gas pressure and specific gas consumption seem to be critical to success of the USGA process. In one report, the percentage of fines obtained in USGA was directly proportional to specific gas consumption.

Yet another approach to attaining fine powders is close-coupled gas atomization, which uses very high gas-jet velocities to increase the yields of fines. Gas velocities above mach 1 at the nozzle exit result in very high yields of fine powders; close-coupled gas atomization has produced yields of fine powder three times greater than conventional gas atomization.

Centrifugal atomization techniques also are used to produce superalloy powders. Centrifugal atomization generally results in a narrower particle-size distribution than gas atomization processes. Rapid solidification rate (RSR) processing, which has received much attention over the past few years, is a centrifugal method. In RSR processing, the melt is dispersed by a rapidly rotating disk, and the melt droplets are cooled by inert-gas jets at rates of 10^5 to 10^7 °C/sec. In addition to narrower particle-size distribution than gas-atomized powders, RSR-processed powders contain relatively few satellites and pores.

Another centrifugal atomization technique used to produce superalloy powders is the rotating electrode process (REP). The process is used commercially for production of rapidly solidified titanium powders, but is beginning to be used more frequently for superalloys. In REP, the tip of a rapidly spinning alloy rod is melted in an inert-gas atmosphere by a plasma arc or electron beam. The molten metal droplets are then flung tangentially from the rod and solidify in flight. Process variables controlling particle size and solidification rate are electrode diameter, rate of melting, and rotational speed of the consumable electrode.

The major advantage of REP is the elimination of ceramics, and thus, the complete absence of ceramic inclusions in REP powders. A problem with superalloys is obtaining a sound electrode and one that is dynamically balanced to withstand the high rotational speeds needed for the process.

Vacuum atomization also is used in commercial production of superalloy powders. Vacuum-atomized powders are slightly less spherical than gas-atomized materials, and satellite particles are more common. The presence of satellites increases apparent particle size and changes the rheological properties of the powder. Particle size and size distribution are more difficult to control in vacuum atomization than in gas atomization.

Putting it all together

Hot extrusion and hot isostatic pressing (HIP) have long been the workhorse consolidation processes for PM superalloys. Hot extrusion produces fully dense billets that are then subjected to further processing to near-net shape. Extrusion also is used sometimes after an initial consolidation step such as HIP or hot pressing. The large amounts of deformation that occur during extrusion promote interparticle shearing and breakup of prior particle boundaries, resulting in a homogeneous, fully dense product. Microstructural control is accomplished by varying reduction ratios as well as extrusion rate and temperature.

Although the name implies purely isostatic pressure, HIP is similar to hot extrusion in that particle-to-particle contacts under isostatic pressure result in formation of shear stresses. The primary difference between the two processes is the degree of powder particle deformation; hot extrusion results in much more macrodeformation than HIP.

Both hot extrusion and HIP are reliable consolidation methods. Recently, however, several new consolidation techniques have begun making inroads in PM superalloy processing. All these techniques offer reduced costs and/or a reduction in powder handling, which translates into cleaner parts with fewer defects and enhanced mechanical properties. Two of the most promising are metal injection moulding (MIM) and spray deposition.

MIM, in which a paste of fine metal powder and an organic binder at moderate temperature is injected under moderate pressure into shaped mould, is just beginning to be applied to production of PM superalloy components. One problem with using MIM for processing superalloys is their sensitivity to organic comtaminates. Therefore, removal of the binder after moulding is the most critical step in producing clean PM superalloy parts by this consolidation technique.

Another process waiting to take off is spray deposition. The most well-known of the spray deposition processes is Osprey, although other similar processes also are being developed. Spray deposition offers both economical and time-saving benefits by eliminating powder handling. The basic Osprey apparatus consists of an induction furnace, a gas-atomizer, and a spray chamber. After atomization, the hot powder collects on a cooled preform mould. Solidification and particle welding take place as the powder cools. The resulting preforms are 99 to 99.9 per cent dense and have a fine grain size. Mechanical properties of spray-formed superalloys are comparable to or better than wrought materials. Spray-deposited preforms are sometimes subjected to post-consolidation forging or other treatments to produce full dense components.

In one investigation, spray deposited disk preforms of five commercial PM superalloys demonstrated good high-temperature strength and stress-rupture properties in testing at 650 and 760°C (1,200 and 1,400°F). The intermediate grain size of the spray-deposited alloys - larger than grains produced by other PM techniques, but smaller than a cast superalloy - resulted in a combination of good workability and high-temperature capability.

Characterizing defects

Identifying and characterizing defects has become extremely important as cleanness becomes a critical concern in the PM superalloys industry. Typical specifications might allow about one part per million of ceramic particles. This translates to about 20 particles per kilogram of alloy, of which perhaps three are allowed to be larger than 400 μm in diameter. Some cleanness goals are much stricter than this standard, however.

As a result of the push for cleanness, inclusion contents in some PM superalloys have been reduced to such low levels that conventional metallographic techniques cannot adequately characterize cleanness. The need for more quantitative evaluation of inclusions also is making conventional metallographic analysis a less viable option for PM superalloys.

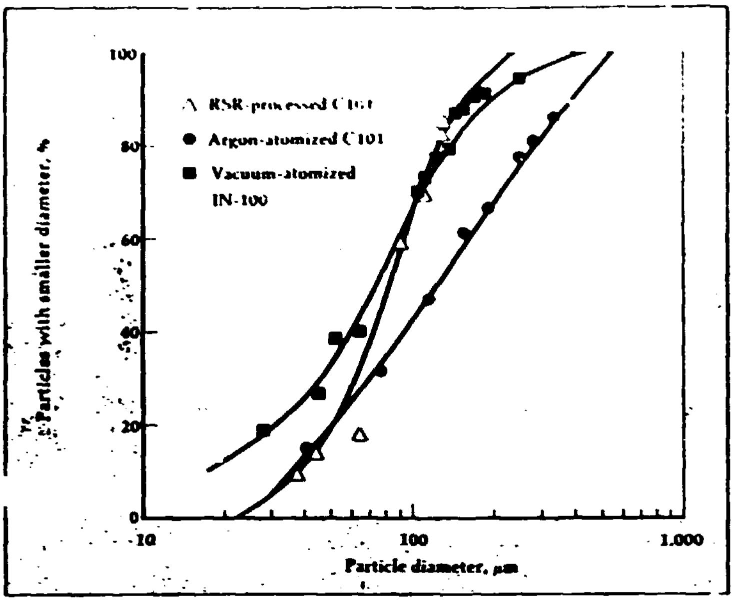
Methods used to characterize ceramic defects in PM superalloys include water elutriation and electron beam (EB) button melting. The tried-and-true former technique uses a density gradient in a column of water to separate relatively light organic and ceramic inclusions from a screened powder sample. The low-density ceramic and organic particles are lifted above the relatively heavy metal particles by the rising water column and filtered for counting and measuring.

EB button melting separates ceramic inclusions from the melt by fusing a small sample ("button") with an electron beam. The low-density ceramic rises to the surface of the melted button and forms an inclusion "raft". After cooling, the raft is examined *in situ* or the button is electrochemically dissolved, leaving the residual ceramic inclusions for counting and weighing. Analysis is facilitated because the inclusion particles are concentrated in a small area.

EB button melting works well for low-density oxide inclusions, but nitrides and other relatively heavy particles may not raft as well as oxides. One investigation into the effectiveness of the technique indicated that only about 10 per cent of nitrides were collected in the raft. Collection efficiency also is influenced by the solidifcation mechanism, operating within the button.

Success of EB button melting depends on achieving reproducible conditions during button melting and solidification. Tests indicate reproducibility is good enough to allow quantitative evaluation of inclusion content.

Maintaining a low gas content also is important to minimize voids and pores in consolidated components. A common test for excessive gas content is thermally induced porosity, in which the density of the material is measured before and after exposure to temperature for a specified length of time. If density decreases more than a predetermined amount, typically 0.3 per cent, the material is considered to contain excessive amounts of entrapped gas. Sources of excessive gas in PM superalloys include hollow argon-atomized powder particles, coalescence of absorbed gases, and container leakage before consolidation. (Source: Advanced Materials & Processes, September 1989)



Depending on process parameters, vacuum and centrifugal atomization processes generally result in slightly higher yields of fines and narrower particle size-distributions than inert-gas atomization. Gas atomization is still the most commonly used powder production method for superalloys.

Minimizing common defects in PM superalloys

Defect	Minimized by
Ceramic inclusions	• Screening • "Rafting" and removal of low-density ceramic defects during melting • Cyclone separation and other techniques that take advantage of density differences
Metallic inclusions	• Adequate cleaning of atomization facility at startup and during changeover of alloy compositions
Voids and pores	• Removal of hollow powder particles • Hot or cold outgassing of powder during can filling • Leak testing of containers
Prior particle boundary contamination	• Ultralow-carbon compositions • Addition of strong carbide formers, *e.g.*, Hf, Nb, and Ta • Modification of heating schedules before and during consolidation • Maximizing deformation of powder particles during consolidation • Post-consolidation working, *e.g.*, isostatic forging

Compiled from information in Superalloys, Supercomposites, and Superceramics, *J.K. Tien and T. Caulfield, Ed., Academic Press, New York, 1989.*

Properties of spray-deposited PM superalloys[1]

Alloy	TS, MPa	YS, MPa	Elongation, %
At 650°C			
René 95	1,471	1,040	12
AF115	1,464	1,080	15
Merl 76	1,327	1,007	21
Astroloy	1,347	903	27
At 760°C			
René 95	1,152	1,008	26
AF115	1,242	1,049	30
Merl 76	1,102	1,007	30
Astroloy	1,040	887	30

[1]*All alloys were supersolvus annealed after spray deposition and forging of the disk preforms. Data from K.-M. Chang and H.C. Fiedler, Spray-Formed High-Strength Superalloys, in* Superalloys '88, *The Metallurgical Society, Warrendale, Pa., p 485-493, 1988.*

* * * * *

6. JOINING OF RAPIDLY SOLIDIFIED POWDER METALLURGY AL-FE-CE ALLOYS

Abstract

The joining of rapidly-solidified powder metallurgy (RS/PM) Al-Fe-Ce alloys by capacitor-discharge and inertia-friction welding has been investigated from a metallurgical perspective. Capacitor-discharge welding was effective in the joining of 6.4 mm diameter Al-8Fe-4Ce rod. The application of pressure simultaneously with fusion zone solidification suppressed the formation of fusion zone porosity in this high hydrogen PM product. In addition, extremely rapid cooling rates promoted RS fusion zone microstructures and prevented heat-affected zone (HAZ) formation, thereby providing joint efficiencies up to 100 per cent. High joint efficiencies were also obtained in 12.5 mm diameter Al-9Fe-4Ce and Al-9Fe-7Ce rod using the solid-state inertia-friction welding process. Welds produced at low axial force were characterized by defects along the weld interface and a relatively large heat and deformation zone (HDZ). Increased axial force significantly reduced HDZ width and minimized defect formation, resulting in optimum joint efficiencies exceeding 90 per cent with satisfactory ductilities.

1. Introduction

Al-Fe-Ce alloys offer significant advantages over conventional aluminium alloys for elevated-temperature applications up to 350°C. These dispersion-strengthened alloys are basically hypereutectic Al-Fe compositions with ternary additions of Ce for enhanced dispersoid stability. Through air or flue-gas atomization, extremely fine, irregular-shaped powder particles are produced which vary in size from about 5 to 40 microns in diameter. The rapid cooling rates (10^3 to 10^6°C/s) experienced by these particles suppresses formation of the equilibrium primary intermetallic structure and instead promotes solidification to supersaturated alpha aluminium.

Subsequent powder consolidation and thermomechanical processing results in a unique microstructure comprised of fine dispersoids in a matrix of submicron alpha aluminium grains.

The efficient utilization of Al-Fe-Ce alloys in structural applications will require their joining. Unfortunately, the application of conventional welding processes has been only marginally successful with the Al-Fe-Ce alloys. Gas tungsten-arc (GTA) welding studies by Metzger on thin-sheet Al-10Fe-5Ce found poor weld quality to result principally from the high inherent hydrogen content of these flue-gas atomized alloys (1-10 ml/100 gm) and the associated evolution of gross fusion zone porosity. In addition, the moderate cooling rates experienced by the GTA weld fusion zone allowed the formation of a coarse, primary intermetallic solidification structure. Although the addition of filler metal was ineffective in reducing porosity, the combination of a preweld vacuum heat treatment (400°C/100 hrs), a direct current, electrode negative welding arc and the addition of 5,356 filler metal significantly improved weld integrity. However, poor filler metal/base metal mixing prevented elimination of the coarse, primary intermetallic solidification structure near the weld fusion boundary. This region served as the preferential fracture site in transverse tensile testing which determined a 55 per cent joint efficiency and negligible ductility.

Considering the physical metallurgy of the Al-Fe-Ce alloy system, it is apparent that achieving high joint efficiencies requires the application of joining techniques which can either recreate or retain the metastable base metal microstructure across the joint while precluding the formation of defects. In this work, capacitor-discharge and inertia-friction welding processes were evaluated in an attempt to meet these requirements.

2. Capacitor-discharge welding

Recent work by Devletian has demonstrated the effectiveness of capacitor-discharge (CD) welding in producing defect-free joints in SiC/Al metal-matrix composites. In addition to suppressing the formation of fusion zone porosity in these high hydrogen, PM materials, the process also provided rapid solidification rates in the melt zone and minimized HAZ formation. Based on these advantages, the CD welding process was employed to join 6.4 mm diameter Al-8.4Fe-3.6Ce (Al-8Fe-4Ce) extruded rod.

The "initial-gap" CD welding process involves the impacting of specimen surfaces under gravity with the simultaneous discharge of a capacitor bank, causing arcing and subsequent melting. Upon impact, the arc is extinguished, molten metal is expelled and rapid solidification occurs. In the present work, welding parameters (table 1, page 35) were controlled to provide both high and moderate cooling rate welds (about 10^7 and 10^5°C/s respectively). Representative welds were sectioned axially and either mounted for metallographic characterization and DPH hardness testing, or three-point bend tested in both the as-welded condition and after removal of the flash/notch at the outer weld periphery. SEM was used for fractographic analysis.

Visual examination of the CD welds revealed minimal flash at the outer weld periphery, with a somewhat greater flash observed in the moderate cooling rate welds. Due to a slight axial misalignment of the specimens during welding, the welds exhibited a notch or ledge at the outer weld periphery.

Metallographic characterization of the CD welds revealed narrow melt zones about 50 to 100 microns in width. In the high cooling rate welds, the fusion zone width was relatively constant across the entire specimen, tapering slightly towards the outer periphery. In contrast, the width of the moderate cooling rate weld fusion zone varied continually across the specimen cross-section.

Three-point bend tests performed on the axially-sectioned specimens showed a significant reduction in bend ductility versus the base metal (table 1).

In summary, CD welding was found to be effective in producing high-quality welds in the Al-8Fe-4Ce alloy. Based on hardness and bend test results, it is anticipated that welding parameter optimization and improved specimen alignment could readily provide 100 per cent joint efficiencies in the as-welded condition.

3. Inertia-friction welding

Inertia-friction (IF) welding is a solid-state welding process which offers an important alternative to fusion welding for joining materials in which melting and solidification must be avoided. The process involves a combination of frictional heating at faying surfaces and the application of axial pressure to produce a metallurgical bond. The absence of melting during IF welding can alleviate weldability problems including the formation of porosity and solidification cracking. Also, the expulsion of contaminated surface layers and heat-and-deformation affected metal at the weld interface during the final forging and upset stage of the process allows final bonding between nascent subsurface materials. Based on these inherent characteristics, this process offers the capability to "retain" to a large degree the base metal microstructure across the joint, making it highly suitable for the joining of RS/PM Al-Fe-Ce alloys with a high residual gas content.

In the present study, 12.5 mm diameter rod of Al-8.6Fe-3.8Ce (Al-9Fe-4Ce) and Al-8.6Fe-6.9Ce (Al-9Fe-7Ce) were joined axially using a conventional IF welding system. Through preliminary testing, welding parameters including the moment of inertia, rotational speed and axial force were optimized. In addition to joints produced using these optimized parameters, welds were also produced at lower and higher axial forces for comparative purposes (table 2, page 35). Following visual analysis, representative welds were sectioned axially and metallographically prepared for microstructure analysis using light and electron microscopy and DPH hardness testing.

In addition, round tensile specimens were machined from the centre of the welded specimens (4 mm gauge diameter, 16.5 mm gauge length) and tested at an extension rate of 0.0042 mm/s. Fractography of the fractured specimens was performed using SEM.

As expected, an increase in axial force promoted an increase in axial displacement (table 2) and the quantity of flash. A consistently greater axial displacement in the Al-9Fe-4Ce versus the Al-9Fe-7Ce alloy was attributed to the lower compressive yield strength of the Al-9Fe-4Ce.

Macroscopic characterization of the as-polished specimens revealed two basic defect types: (1) voids which resulted from the non-uniform deformation and insufficient extrusion of metal out of the weld interface region and (2) fine, linear lack-of-bonding defects which may have been associated with dispersed oxides. The number and size of these defects decreased with increased axial force, and were only occasionally observed in welds produced at the highest axial force. Although the base alloys exhibited high hydrogen contents of 2-3 ml/100 gm, no evidence of porosity or blistering was observed. Macroscopic examination of the welds after etching with Ke ler's reagent revealed heat-and-deformation zones (HDZs) which were observed to decrease in width with increased axial force. This decrease was attributed to the greater extrusion of heat-and-deformation affected metal out of the weld interface as flash.

Figure 1 (see page 35) illustrates microstructures of IF welds produced at low (a) and high (b) axial forces. Evidence of a radially outward flow of metal which is characteristic of IF welding is apparent in both microstructures, with the original base metal texture changing 90°C to an orientation parallel to the weld interface. In the vicinity of the weld interface, elevated temperatures promoted coarsening of the dispersoid structure. The use of high axial force essentially eliminated the most severely coarsened light-etching region and also reduced the width of the dark-etching transition region. The dispersoid-coarsened, light-etching structure in the low-axial force weld exhibited a significant decrease in hardness versus the base metal, with the dark-etching region showing a range of hardnesses. Elimination of the light-etching region in the high axial force weld and a reduction in the width of the transition region resulted in a minimal drop in hardness across the weld zone.

Tensile properties for the welded specimens are shown in table 2. Increased axial force was found to promote a significant increase in tensile strength, with an increase in joint efficiency from 56 per cent for welds produced at low axial force to over 90 per cent for high axial force welds. Tensile ductilities of the specimens also increased with increased axial force, although elongation values were still well below those of the unaffected base metal.

Fractographic analysis of the tensile specimen surfaces showed fracture in low and medium axial force welds to occur along the weld interface through the low hardness, dispersoid-coarsened structure. The present of voids along the weld interface may also have contributed to property degradation in these welds. Higher weld strengths associated with welds produced at increased axial force promoted a shear mode of failure across the weld interface into the unaffected base metal. All of the weld fracture surfaces exhibited a distinct spiral appearance emanating from the centre of the rod, which reflected the effects of combined torsional and axial forging forces on metal flow experienced at the weld interface during IF welding. In addition, on a microscopic scale all fracture was observed to involve microvoid formation around the fine dispersoids and the associated ductile failure of the alpha aluminium matrix.

In summary, the IF welding process has been shown to be effective for the joining of RS/PM Al-Fe-Ce alloys. Increased axial force was found to improve weld integrity and tensile properties by eliminating weld interface defects and minimizing microstructural coarsening across the weld zone. (Extracted from the proceedings of the <u>2nd International SAMPE Metals Conference</u>, 2-4 August 1988, Dayton, Ohio, USA. Paper submitted by: W.A. Baeslack III, Department of Welding Engineering, The Ohio State University, Columbus, Ohio 43210)

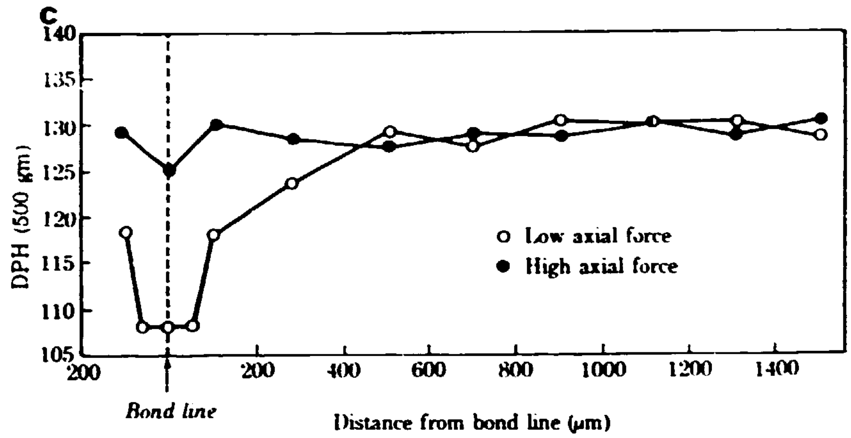


Figure 1
Microstructures (a,b) and DPH hardness traverse (c) near center of inertia-friction welds in Al-9Fe-4Ce produced at (a) low and (b) high axial force. Arrows indicate weld interface.

Table 1. Hardness and bend ductility of Al-8Fe-4Ce base metal and capacitor-discharge weld specimens.

Specimen*	DPH** (50 gm load)	Bend Ductility***	Fracture Initiation Site
Base Metal	132-137	>20%	-
HC Weld/AW	286-341	3.3%	outer periphery notch
HC Weld/Mach.	"	>16%	FZ weld defect
MC Weld/AW	148-161	3.7%	outer periphery notch
MC Weld/Mach.	"	>17%	base metal

*HC weld: 0.4 mm tip dia., 0.5 mm tip length, 60° conical tip, 80 μF, 100V, head + 5.68 kg drop wt., 50 mm drop ht.
MC weld: 0.8 mm tip dia., 1.0 mm tip length, 60° conical tip, 80 μF, 100V, head + 1.93 kg drop wt., 25 mm drop ht.
**Hardness at center of fusion zone
***Flat surface in tension

Table 2. Inertia-friction weld axial displacements and tensile properties

Alloy	Axial Force kN	Axial Displacement mm	Tensile Strength MPa	Joint Efficiency %	% Elong.	Fracture Location*
Al-9Fe-4Ce	4.4	2.5	234	56	1.9	IR
"	6.5	5.8	262	62	2.9	IR
"	8.7	9.6	386	92	3.0	IR/HDZ
Al-9Fe-7Ce	4.4	1.8	262	56	1.9	IR
"	6.5	4.6	386	82	2.7	IR
"	8.7	8.9	427	91	3.0	IR/HDZ

*IR - Interface Region, HDZ - Heat-and-Deformation Zone

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7. POWDER METALLURGY COMPOSITE PRODUCTS USING HIP METHOD

Report by Nobuyasu Kawai and Hiroshi Takigawa, Materials Research Institute of Kobe Steel, Ltd: "Development of Powder Metallurgy Composite Products Using HIP Method".

1. Introduction

To meet the demand for improved and more complex performance and for advanced qualities of components in the various industries, the manufacture of composite materials based on diffusion connection has been highlighted recently. What has been attracting most attention is a moulding method by which powder materials can be solidified based on HIP (hot isostatic processing). This method, whereby powder materials can be solidified and moulded based on the HIP method and at the same time joined with dissimilar materials, can be applied to products of complex configuration with ease, covering wide areas of application. Examples of concrete development include a valve body for an oil well, a roll for a cold work mill, and a cylinder for an injection moulding machine.

2. Method

At Kobe Steel, a Co-based metallurgy powder had been produced that is excellent in terms of corrosion and abrasion resistance by means of the Ar gas atomizing method, as illustrated in figure 1 (see page 37). This powder was filled into the clearance between an SCM 440 steel cylinder and a mild steel capsule in a vacuum state and sealed. Then, HIP processing was carried out at a temperature of 950°C and a pressure of 1,000 kg/cm^2; machining was performed to remove the internal capsule, and a cylinder lined with a 2 mm thick layer was produced. Tests to confirm the physical properties involved measuring the strength of the connected members, the microscopic organization, mechanical properties, high temperature, corrosion resistance, abrasion resistance, and expansion coefficient of the alloy. As a practical equipment test, an effort was made to obtain the lifetime of the equipment by moulding various kinds of engineering plastics with an injection moulding machine. Co-based and Ni-based centrifugal casting materials and nitrided steel, which are very popular, were used as reference materials. Table 1 on page 37 shows the composition of these alloys.

3. Results

3.1 The diffusion layer of the connection member was about 50 μm in thickness; the deposit of an abnormal layer was not observed. The tensile strength in the connection section was found to be 40.9 kg/mm^2. This value is satisfactory in relation to the calculated values (12 to 24 kg/mm^2) of tensile strength produced under ordinary injection moulding conditions in the peripheral direction of the connection member.

3.2 The abrasion resistance of the HIP alloys evaluated by the Ohgoshi method-based abrasion test exceeded the values of the reference material over the entire sliding speed range, as illustrated in figure 2 (see page 37). This is considered attributable to the uniform and microscopic distribution of the hard particles of fine B (Cr, W).

3.3 The result of the practical equipment test, shown in table 2 (see page 37), indicates that the injection moulding of engineering plastics, including 30-45 per cent glass fibre, can provide a maximum lifetime exceeding 14 months - a value five to six times that of conventional nitrided steel.

As stated, it has been ascertained that the use of the HIP method is capable of producing the cylinder of an injection moulding machine for plastics engineering which is excellent in corrosion resistance and abrasion resistance. Based on these achievements, further attempts are being made to apply the HIP method to the manufacture of a non-magnetic cylinder for "plamag" [phonetic] using high Mn non-magnetic steel, a superabrasion resistant cylinder including WC powder, or a bi-axial cylinder which is excellent for kneading. This technology can also be applied to the cylinder of an injection and moulding machine for ceramics and metal powder, for which high expectations are harboured for future demand. (Source: Tokyo NIHON FUNMATSU FUNTAI YAKIN KYOKAI, 16-19 May 1988)

(wt.%)

Materials	Symbols	Co	Cr	Ni	W	Mo	B	Si	C
HIPed Lining Alloy	HA	Bal	25.0	16.0	10.0	–	3.0	3.0	0.80
Co Based Centrifugal Cast Alloy	CC	Bal	20.0	13.6	16.0	–	3.5	3.5	1.10
Ni Based Centrifugal Cast Alloy	NC	–	16.8	Bal	–	1.8	3.0	5.0	0.60
Nitrided Steel	NS	Fe Bal	1.4	–	–	0.2	Al 0.9	0.2	0.46

Table 1. Composition of HIP-Based Alloy and of Reference Materials

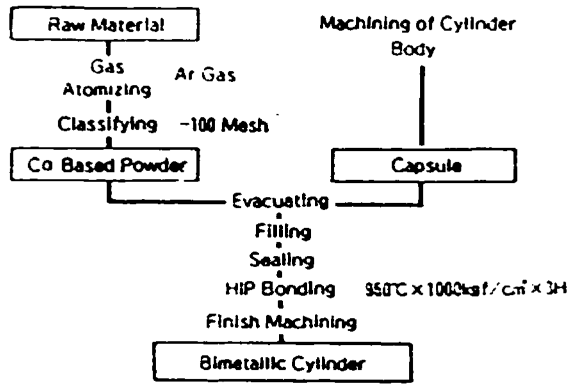

Figure 1. Manufacturing of complex cylinder according to HIP method

Compound Resin	Injection Temp. ℃	Injection Pressure kgf/cm²	Injection Cycle sec	Life	
				HIPed Cylinder	Conventional
PC+G30%	300	1500	30-40	18 months, continue	- - -
PC+G30%	300	1500	30-40	13 months, 45μm worn, continue	5~8 months (nitrided)
PBT+G30%	250	800	25-30	17 months, continue	- - -
POM+G20%	270	1750	25-30	20 months, continue	8 months (nitrided)
POM+G45%	300	1750	25-30	18 months, continue	15 months (Centrifugal cast)
PES+G30%	350	2700	46	12 months, continue	- - -
PC+G30%	- -	- - -	- - -	6310hr 38μm worn, continue	- - -

Table 2. Practical Equipment Test Result for Injection and Molding Machine

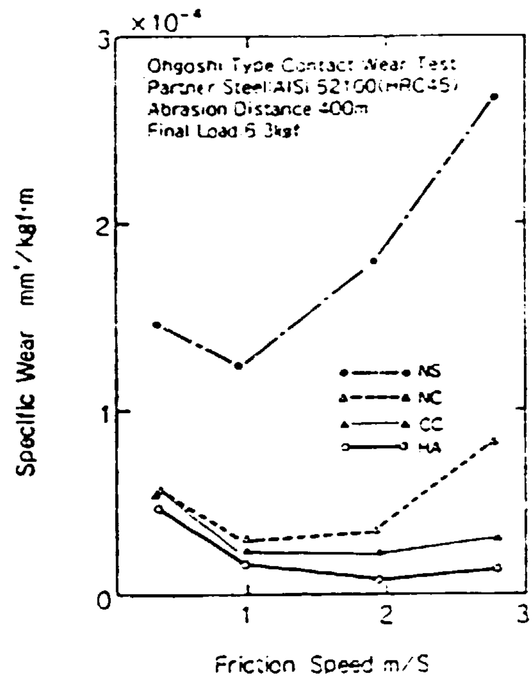

Figure 2. Abrasion test result

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8. NEW PRODUCTS AND THEIR APPLICATIONS

Applications

Fibre metallurgy has many commercial applications

Any machinable metal can be converted into short fibres economically by a chatter machining process developed at Tokyo University. Metals such as aluminium, copper, brass, iron, stainless steel, nickel, titanium and the like can be cut into fibres from 0.2 to 100 mm long with diameters of 0.02 to 0.5 mm. And experimental silver fibres have been made that are only 0.12 mm long and 20 μm in diameter. These fibres, which can be treated much like metal powders in that they can be mixed with other powders, compacted, and sintered using conventional powder metallurgy technology, can be fabricated into a number of commercial products.

The metal fibre fabrication process takes positive advantage of chattering, which when it occurs during machining, is deleterious to the finished machined product's surface. As shown in figure 1 (see page 46), an elastic cutting tool set-up for a conventional turning operation is used. By selecting the appropriate machining conditions, the cutting tool can be made to vibrate. The fibres are produced one at a time per vibration. The vibration frequency, which depends on the natural frequency of the cutting tool, is about 4,000 Hz. Thus 4,000 fibres per second are produced by each cutting edge. The set-up presently used in Japan turns out 320,000 fibres per second per machine by using 20 cutting tools, each with four cutting edges. About 300 tons of fibres were produced in 1987 by the 30 machines presently in operation in Japan.

Some of the fibre-containing materials under development are cast iron fibres compacted with 30 per cent graphite for self-lubricated bearings and wear-resistant seals, compacted with diamond powder for an exceptionally strong and wear-resistant grinding wheel for machining ceramics, and compacted with sub-micron abrasives for a lapping tool; 10 vol. per cent brass fibres mixed with a polymer such as nylon for electromagnetic interference shielding; steel fibres mixed with cement for plastic and sheet metal stamping tools, mixed with sintered refractory material for durable nozzles for continuous steel casting and blast furnace plugs, and mixed and sintered with ceramic powder for porous moulds that improve the surface quality of the moulded product; and metal matrix composites made by squeeze casting molten aluminium alloys into steel fibres.

In addition, sintering titanium and nickel short fibres produces porous sheets for catalysts, and sintering stainless steel fibres produces porous sheets for filters. Using textile techniques, a longer nickel fibre, approximately 30 mm, can be fabricated into a continuous sheet for battery electrodes. And an experimental printed circuit board made by "planting" numerous short metal fibres on the substrate material by electrostatic attraction, then soldering to obtain adequate conductivity, is almost as electrically conductive as a copper-plated circuit board made by electroplating or etching. The developers believe that fibre metallurgy's potential applications have only begun to be explored. [Source: Professor Takeo Nakagawa, Head of Research Center for The Development of Advanced Materials, Institute of Industrial Science, University of Tokyo, Roppongi, Minato-ku, Tokyo 106, Japan. Telephone: (81) 03-402-6231. Fax: (81) 03-402-5078.] (Source: Materials and Processing Report, October 1988)

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Fine powders produced by an improved microatomization process

An improved version of the patented microatomization process developed at GTE can potentially produce high yields of ultrafine powders of a variety of metals and alloys. The GTE microatomization process uses a DC plasma jet to simultaneously melt and accelerate metal or alloyed powders to high velocities. The stream of molten particles impacts a substrate and fragments into smaller droplets. The molten fragments of the droplets, after bouncing off the substrate, are solidified in flight, producing fine spherical powder particles that are much smaller in size than the starting powder. The median size of the product powder is typically less than 10 μm.

The original version of the microatomization process developed by GTE uses a cold substrate. To minimize the build-up of an uneven rough surface of solidified material, which has a deleterious effect on microatomization efficiency, the cold substrate is usually rotated to constantly change the area contacted by the impacting molten droplets. Recent work has led to the development of a different mode of process operation - hot substrate microatomization - shown schematically in figure 3 (see page 46). Here the substrate is maintained at a temperature above the melting point of the material to be microatomized. The plasma jet is used to melt and accelerate the powder particles to high velocity as well as to heat the substrate to the appropriate temperature. The substrate does not need to be rotated because the plasma jet is also used to melt and blow off any unmelted particles adhering to the substrate. Hot substrate microatomization has been found to improve the efficiency of the microatomization process by producing higher yields of finer powder.

Higher process efficiency and ease of operation are facilitated by operating the plasma gun at higher power levels and gas flow rates (gases used include argon, helium, hydrogen, nitrogen or their combinations) in a reduced pressure environment (a controlled vacuum chamber). These operating conditions result in long, diffuse, high-velocity supersonic plasma jets (Mach 2-3), which help to achieve higher particle velocities and to heat the substrate more uniformly. The particle size of the MICROATOMIZED™ powder can be varied by appropriately selecting both the plasma gun operating conditions and the size of the starting powder.

Process considerations dictate that the substrate should retain sufficient strength and erosion resistance to withstand the continuous impact of the high-velocity molten droplets when heated to temperatures above the melting point of the material to be microatomized. A further consideration, particularly for highly reactive metals and alloys, is the degree of contamination that could result from their alloying or reacting with the substrate. Possible substrate materials include high-temperature metals and alloys, intermetallics, and ceramics such as borides, carbides, nitrides, oxides and silicides.

The hot substrate microatomization process has been tested using copper as the model material. Figure 4 (see page 46) shows that 68 wt per cent of the yield of the MICROATOMIZED[TM] copper powder with a median particle size of 6.7 µm (curve A) is less than 10 µm, and 87 wt per cent is less than 20 µm. For powder with a 3.6 µm median particle size (curve B) these numbers are 88 wt per cent and 98 wt per cent respectively.

The GTE researchers are now working on optimizing the process to yield finer powders with tighter size distributions. Several patents covering this technology have been issued (some are pending) to GTE. The company is interested in discussing licensing and joint development programmes. [Sources: For the technology, Muktesh Paliwal, Advanced Research Engineer, GTE Products Corp., Hawes St., Towanda, PA 18848. Telephone: (717) 265-2121. Fax: (717) 265-1450. For licensing: Robert J. Dobbs, New Business Manager (same address, telephone and fax numbers).] (Source: Materials and Processing Report, October 1989)

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Bulk amorphous metal parts and amorphous coatings by powder metallurgy

Amorphous nickel niobium ($Ni_{60}Nb_{40}$) powder produced by mechanical alloying has been compacted into a semifinished part at the Fraunhofer Institute in Bremen, Federal Republic of Germany. The powder geometry of the amorphous, mechanically alloyed powder was found to be more suitable for compaction than that produced from melt-spun ribbons. X-ray analysis after explosive compaction showed that the bulk material was still amorphous.

In mechanical alloying, the elemental metal powders are mixed together and dry milled for a number of hours (in this case 60 h) in a high energy ball mill, in an inert gas such as argon. During the course of the milling the powder particles, which have become cold-welded together, become finer and are alloyed via a solid state reaction. X-ray diffraction patterns indicate decreasing intensities of crystallinity as the milling proceeds, until no crystalline reflection is observed. And optical micrographs show that the microstructure has become increasingly refined. In the case of the nickel/niobium material, the microhardness of the amorphous powder increased throughout the milling to a maximum of 850 HV 0.025 N/mm^2. Further, during the mechanical alloying the particles became globular in shape and layered, with a particle size of approximately 50 µm.

The researchers consolidated the powders by explosive compaction in a copper tube, forming a machinable, semifinished product in which the powder's amorphous properties, hardness and crystallization temperature were retained. They also suggest that an amorphous sheet could be fabricated by hot rolling the powder at temperatures just below the crystallization temperature (678°C), because it becomes quite ductile and flows freely at its glass-point temperature. Further, they note that recent experiments indicate the possibility of producing amorphous coatings by thermal spraying.

The researchers believe that because amorphous powders are more easily made by mechanical alloying than from rapidly quenched 50 µm thick melt-spun ribbons, it is the more likely process for producing these powders commercially. In addition, while the physical properties of the powders are similar, the morphology of mechanically alloyed powder appears to be more suitable for the fabrication of bulk amorphous material. They anticipate that their work will spark new interest and applications for amorphous materials. (Source: Materials and Processing Report, October 1988)

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Metal powder production by ultrasonic atomization

Potentially cost-effective ultrasonic techniques to atomize liquid metals for the production of metal powders are under development in the Federal Republic of Germany at Leybold AG, Hanau and the Battelle Institute, Frankfurt. The economic advantage of this approach, once scaled up, is that it does not require the large atomization towers (up to 8 m high and 3 m in diameter; 26.3 ft x 9.8 ft) or consume the quantities of gas during atomization of the melt (approximately 30 Nm^2/min), typical of inert gas atomization.

There are two ultrasonic techniques being investigated. In one, ultrasonic capillary wave atomization, a thin layer of liquid metal covers the surface of an active solid resonator that vibrates ultrasonically. At certain vibrational frequencies, i.e. , when the correct amplitude of oscillation is reached, microdroplets fly off from the antinodes, solidifying into spherical metal powder as they cool down. Several precautions must be observed, however. To avoid solidification of the melt on the resonator's surface, it has to be heated to the temperature of the melt. Further, to achieve good wetting of the resonator's surface, it must be compatible with the melted alloy without itself dissolving. Because this problem increases with higher melting temperatures, this technique is probably limited to metals with melting points between about 800°C and 1,000°C. However, this process consumes very little energy, and because the microdroplets leave the resonator at relatively slow speeds, the atomization chamber can be compact.

In the other technique, standing wave atomization, illustrated in figure 5 (see page 46), the automization takes place in the velocity knot of an ultrasonic standing wave between a 20 kHz transmitter and a reflector. The size of the droplets is controlled by the sound intensity of the standing wave. As the gas pressure is increased in the knot region, the increased sound intensity decreases the droplet size. The flight distance of the molten droplets is also markedly reduced. Thus the size of the atomization chamber can be kept to a minimum by increasing the gas pressure in the chamber. However, a high gas pressure must be maintained, which can be disadvantageous. In tests with a tin alloy, atomization rates were 2 to 4 kg/min, and powder particles with an average diameter of 40 to 60 µm and particle size distribution similar to that obtained in conventional processes were produced. Coatings are being developed to avoid the chief problem of particle accumulation on the reflector system.

The developers are working on scaling up to production quantities, and on increasing the melt temperatures so that superalloys can be atomized. However, they believe that the present systems could prove useful to researchers for producing powders in the laboratory with a reasonably sized device. [Powder Technology, Leybold, AC, Wilhelm-Rohn Strasse 25, D-6450 Hanau 1, Federal Republic of Germany. Fax: (49) 61-81-34-1690. Telex: 4-15-206-0 LHD.] (Source: Materials and Processing Report, October 1988)

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Method for producing an amorphous material in powder form by performing a milling process

The properties of amorphous metals prepared by mechanical alloying correspond in general to those which are produced by melt spinning methods. However, the range of concentrations which form glasslike structures can be far larger than with melt spinning. Metal/metalloid alloys containing boron or boron compounds have special properties including greater hardness than metal/metal alloys and special magnetic and corrosion resistant properties. These properties occur with a composition of $(Fe_{1-x}Zr_x)_{1-y}B_y$ (in atom percents) where $20 \leq x \leq 80$ and $4 \leq y \leq 30$, for example $Fe_{60}Zr_{20}B_{20}$. The metal powders are in a size range of 50 μm to 0.5 mm but the boron powder should be below 1 μm. The powder mixture is ball milled with hardened steel balls for 10 to 30 hours in an argon atmosphere which embeds the boron in the metal particles. Finally the milled mixture is annealed at 600°C for 4 hours, at a temperature below the crystallization temperature of the alloy component. The amorphous state is confirmed by X-ray examination. [Siemens Aktiengesellschaft, Munich, Federal Republic of Germany.] (See following article.) (Source: Materials and Processing Report, August 1989)

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Amorphous alloys produced by mechanical alloying

It has recently been shown that mechanical alloying can form amorphous metallic powders in a large number of alloy systems, particularly alloys of the transition metals. In many cases, compositions can be made amorphous that cannot be made by melt spinning. This has renewed interest in this solid-state technique for combining metals, which was developed about 10 years ago.

In mechanical alloying, the elemental metal powders are mixed together and dry milled for several hours in a high-energy ball mill in an inert gas. In the early stage of the milling the collisions between the steel balls and the elemental powders cold weld the particles together. As the milling proceeds the particles get finer and finer, becoming quite regularly arranged in a layered microstructure. Finally, true alloying occurs via a solid-state reaction, and an amorphous phase is formed that increases with continued milling. Figure 6 (see page 46) shows the X-ray diffraction patterns of the iron-zirconium alloy, $Fe_{50}Zr_{50}$, after milling times from a half hour to 60 hours. As can be seen, the amorphous peak grows with increased milling time as a larger amount of the amorphous phase in the powder is being formed, until the powder is completely amorphous at 60 hours. A conclusion from property studies of both nickel zirconium and iron zirconium is that the final composition of the amorphous phase depends on the milling conditions - i.e., the intensity of the milling and also probably the temperature.

The researchers have found that under their experimental conditions they can produce a large number of amorphous alloys of the 3d transition metals (Ni, Co, Fe, Cu, Mn) with Zr and Ti. While these alloys appear to be quite similar to those produced by melt spinning, they have some advantageous differences with commercial potential. For example, mechanically alloyed $Fe_{91}Zr_9$ appears to be a much better catalyst for the production of ammonia than the melt spun or high vacuum crystallized material. The higher reactivity of the mechanically alloyed powders could be due to their very large surface area.

The researchers have also found that they can form an almost unlimited number of boron-containing transition metal alloys. They have produced magnetically hard $Nd_{15}Fe_{77}B_8$ with a coercivity as high as 13 kOe by a combination of mechanical alloying followed by annealing for a relatively short time. The temperature dependence of the coercivity for the mechanically alloyed material is comparable to the rapidly quenched material and superior to that made by standard powder metallurgy. Further, by adding a small amount of Dy (dysprosium) to form a $Nd_{13}Dy_2Fe_{77}B_8$ alloy, the room temperature coercivity is increased to 20 kOe (10 kOe at 150°C), values that compare favourably with standard preparation processes. Because of the potentially large number of amorphous metal powders that can be formed which cannot be made by melt spinning, the researchers believe that mechanical alloying is an attractive technique for making novel amorphous metals that could be of interest both technologically and commercially. [Source: Ludwig Schultz, Siemens AG, Research Laboratory, Günther Scharowsky Strasse 2, D-8520 Erlangen, Federal Republic of Germany. Telephone: (49) 09131-722727.] (Source: Materials and Processing Report, March 1987)

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Powder-metallurgical production of a work-piece from a heat-resistant aluminium alloy

Some heat resistant aluminium alloys produced by gas jet atomization with rapid cooling are deficient in toughness and ductility. Improved products are obtained by atomization of a composition composed of 10 per cent Fe, 1 per cent V, 0.5 per cent Mn, and the remainder Al in a gas jet (nitrogen), with a cooling rate of at least 10^5°C/sec. The average particle size is approximately 20 μm with a maximum of 40 μm. The particles have no micro-eutectic zone. The powder is pressed at 2,000 to 6,000 bar at 350° to 450°C. The intermetallic Al_6Fe phase, stabilized by Mn, is formed and the Al_3Fe phase is largely suppressed. A moulded slug (40 mm diameter) placed in an extrusion press was reduced to 13 mm diameter (reduction ratio 9:1). The yield strength of a test specimen was approximately 450 MPa at room temperature. [BBC Brown Boveri AG, Baden, Switzerland.] (Source: Materials and Processing Report, May 1989)

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Swedish company claims that it is breaking new ground by using powder metallurgy in conjunction with hot isostatic pressing

With the world's largest hot isostatic pressing (HIPping) facility at its disposal, ASEA Powdermet in Sweden is claiming to have pushed powder metallurgy into new fields - for the alloys that can be made and the types of components produced.

Powdermet uses an ASEA Quintus wire-wound hot isostatic press having a 1,470 mm diameter chamber and a furnace volume of 5,000 litres. It can operate at a temperature of 1,25°C and a pressure of 140 MPa (1,400 bar).

Powder compacting produces components to near nett shapes, thus minimizing subsequent machining. But in some instances the process can also create shapes that are impossible by traditional machining.

This large diameter, thick-walled tube features an internal, circular-section, helical duct passing from one end to the other, making three complete turns on its way. At each end, the duct emerges at right angles to the face All welds provide gas-tight joints.

During HIPping, the capsule is subjected to high isostatic argon gas pressure and sintering heat, and "shrinks" from 10 to 12 per cent in all directions (for which allowance is made in the initial design). At the end of the process, the capsule is removed from the press and the sheet steel is cut and stripped from the component.

The only other practical production method would be precision casting or investment (lost wax) casting, neither of which could probably give the high homogeneity and material integrity yielded by HIPping.

As far as alloying is concerned, ASEA Powdermet claims that HIPping opens the way for new families of materials, based on combining powders or combining powder with a conventionally-manufactured alloy. By these means it is possible to produce a component having layers of materials with different properties - a composite/compound.

For example, a composite of stainless steel and a case-hardening steel has been produced, and micro examination shows that metal-to-metal bonding is achieved. Many of the new alloys produced by HIPping lack counterparts among conventional special steels as regards certain properties. And it is suggested they would be extremely difficult to produce by conventional means.

For instance, a high-strength stainless ferritic-austenitic steel (APM 2389), produced by HIPping has a resistance to stress corrosion and pitting equal to the best of its conventional counterparts but its tensile yield strength is 600 N/mm^2 compared with around 450 N/mm^2 for SS 2377 and SS 2324. The reason is that HIPping allows the steel to have a high nitrogen content. APM 2389 has been patented and is already being manufactured on a large scale for a variety of applications: 50 products, weighing from 30 to 500 kg, have been produced.

Another new material made by HIPping is APM 2390-93, a 12 per cent chromium steel. It has found immediate use in the turbine industry for high-temperature applications, and tests show that it has better ductility than conventionally-manufactured alloys.

ASEA Powdermet manufactures its own powders and has an atomizing plant with a melting capacity of 2.5 tons. Molten metal is tapped into the atomizing chamber where a jet of an inert gas impinges at right angles to the stream, to break it into small droplets. Atomization occurs in a very low oxygen atmosphere, and the droplets cool at about 1,000°C/sec. As a result, the normal reactions occurring during solidification are limited, compared with the slow cooling rate of an ingot, for example.

Each particle has the same chemical composition as the parent melt, and a fine micro-crystalline structure. In addition to possessing excellent homogeneity, materials made from powder are free from macro-segregation and have the same mechanical properties in all directions. (Source: Machinery and Production Engineering, 1 January 1988)

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Ultra-fine, ultra-pure oxide powders

Increased use of ceramics as electronic and structural materials has led to increased demands for ultra-fine and ultra-pure ceramic powders, as the end product properties are critically dependent on precursor quality. Researchers at Penn State have developed a new method of producing fine and ultra-fine powders for such specialty applications as nanocomposite components, based on work begun by Faraday in the last century. The technique, called reactive electrode submerged arc (RESA) processing, uses two submerged metal or otherwise conducting electrodes that arc in a dielectric fluid that reacts with the metal. The extremely high temperature generated by the brief spark vaporizes the electrode material and the surrounding fluid, creating a reactive bubble for an instant. The reaction product of the combined vapours is quickly quenched by the surrounding dielectric fluid, yielding a colloidal sol of spherically shaped particles.

The process can be readily demonstrated with normal line voltages (230V) across thin metal foils immersed in water or mineral oil. Using somewhat more sophisticated equipment, the researchers generated gamma-aluminium oxide, a variety of titanium oxides, zirconias, and oxides of tin, tungsten, and iron, all using metal rods and such common dielectrics as water. Control of voltage, amperage and liquid composition allows the production of fine sols of aluminium, titanium and zirconium anhydrous oxides, and even some phase control of variable valence metals such as chromium. Oxide particles ranged in size from 10 to 1,000 nm. [Amitabh Kumar and Rustum Roy, Materials Research Laboratory, Pennsylvania State University, University Park, PA 16802, USA. Tel.: (814) 8651174.] (Source: New Materials World, March 1989)

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Explosion compaction improves powder metallurgy structures

Compressing of temperature-sensitive metal powders by explosion methods offers a little-used but very promising method or alternative to other methods of reducing brittleness in the products. The explosion-compression makes an extremely high pressure effective for a short period. The impact wave may be produced by igniting an explosive that has been poured over the material to be compacted. This process has the advantage that systems sensitive to recrystallization can be compressed without altering the microstructure because of the process taking only microseconds.

The bodies thus formed can be further hot-worked or cold-worked. In addition, in some systems, this process can achieve synthesis of inter-metallic phases. The prospect is of interest for future applications for preparing special inter-metallic phases of interest that cannot be produced by other methods. [Battelle-Institut, Postfach 90 01 60, D-6000 Frankfurt 90, Federal Republic of Germany. Tel.: 069/79 08-0. Telex: 411 966. Fax: 069/79 08 -80. Dr. Inken Nielsen. Tel.: 069/79 08-2426.] (Source: New Materials World, March 1989)

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Ceramic fibres and CMCs from readily-available precursors

Ceramic fibres and ceramic matrix composites (CMCs) have been successfully produced from polymeric precursors including polysilazane, polycarbosilane and polysilane, yielding continuous filaments of high strength, stiffness and thermal stability. These polymers require somewhat complex synthesis, however, and the stoichiometry of the resulting ceramic is difficult to control. Recently, researchers at NASA Lewis have successfully produced ceramic fibres and CMCs from silsesquioxanes, an easily-synthesized polymer already used commercially for such applications as paint surfactants. Silsesquioxanes have the general formula RSiO, where R can be a methyl, ethyl, propyl, vinly, phenyl or alkyl group.

In the NASA process, which is available for commercial licensing, silsesquioxane powders are blended to achieve the desired silicon/carbon ratio. This blend is heated, causing excess silanol groups to condense out with the evolution of water. When the melt reaches a suitable viscosity, it may be extruded or drawn into fibres. These, in turn, are cured and then fired to yield ceramic fibres. Melt stability is good, so very narrow filaments may be spun. Composites may be produced by first winding the reinforcement fibre onto a mandrel, and then spraying it with the silsesquioxane melt. Alternatively, reinforcement preforms may be melt-infiltrated. After cooling, the pre-impregnated fibres may be cut into plies and stacked in the desired configuration in a metal mould, where they are heated under pressure according to a sequence designed to eliminate any residual void-causing volatiles. The laminate is then cured and heat treated. This new process avoids the problems of solvent removal, shrinkage and void formation that have accompanied conventional ceramic matrix composite fabrication. [Ref.: EW-14566/TN. Daniel G. Soltis, Technology Utilization Officer, Mail Stop 7-3, 21000 Brookpark Road, Cleveland, OH 44135, USA. Tel.: (216) 433-5567. Fax: (216) 433-8000.] (Source: New Materials World, August 1989)

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Beta-SIALON as fine powder

To prepare beta-SIALON ceramics with their high-temperature strength and high oxidation resistance "reaction sintering" has been used. This is a method in which powders like Si_3N_4, AlN and Al_2O_3 are combined so that the mixture has the β-SIALON composition beforehand and is then calcined. However, since it is difficult to combine uniformly raw material powders, the finished ceramic composition prepared by this reaction sintering method becomes non-uniform and a part of the ceramic becomes glass. The new development is purely a fine powder of β-SIALON, and the manufacturing method solves the above problems.

The synthesizing method of β-SIALON fine powder starts by hydrolyzing aluminium alkoxide in solution while adding it to silica powder and carbon powder. This results in a uniformly mixed powder of metal oxides and carbon. The oxides are reduced by heating this powder in nitrogen and grinding to a fine powder. The average particle size of the β-SIALON fine powder thus produced is 0.6 μm, the specific surface 1.6 m^2/g and the content of impurities is under 0.05 per cent.

β-SIALON ceramics synthesized by calcining this fine powder at normal pressure have flexural strength at 1,200°C of 60 kgf/mm^2, and when heated at 1,300°C for 50 hours, the gain in weight due to oxidation is 0.5 mg/cm^2.

Compared with β-SIALON ceramics synthesized by the reaction sintering method, the high-temperature flexural strength of the newly developed material improved 6 kgf/mm^2 and the weight gain due to oxidation was reduced 0.2 mg/cm^2. [Nihon Cement Co. Ltd., Public Relations Dept., 1-6-1, Otemachi, Chiyoda-Ku, Tokyo 100, Japan. Tel.: (03) 201-1731. Telex: 23972. Fax: (03) 284-1728.] (Source: New Materials World, April 1989)

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Cleaner fines powders by gas atomization

Fulmer Research has commissioned a high energy gas atomization facility for the production of special powders and for R&D into new alloys. This process produces fine powders with substantial fractions below 15 μm, which enhances sinterability and provides finer strutures after consolidation. Using inert gas, clean powders are provided. The resulting powder surfaces free of contamination are less likely to produce defects in compacts. Rapidly solidified structures are achieved, leading to enhanced compact properties. The method is claimed to be capable of producing alloys not feasible by conventional methods. It can melt metals up to temperatures of 1,800°C.

In early trials with the equipment Fulmer have produced powders in aluminium, aluminium and magnesium-based alloys, copper and its alloys, nickel-based alloys and intermetallics, iron-neodynium-boron and other ferrous-based alloys. Over the coming months work for clients will include development of novel alloys using the rapid solidification capabilities to produce both low and high melting point matrices for metal matrix composites, and to develop novel brazing alloys. [George Yiasemides, Fulmer Research Ltd., Hollybush Hill, Stoke Poges, Slough, Berkshire SL2 4QQ, UK. Tel.: (02816) 2181. Telex: 849374. Fax: (02816) 3178.] (Source: New Materials World, June 1989)

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Powder metallurgy has become a quickly-growing $2.5 billion/year segment of the steel industry

It involves production of steel dust which then is packed and shaped into detailed small parts. Several small firms are making moves to take advantage of the technology, which produces durable items such as bushings, gears and hand wrenches. The products are said to be as strong as steel parts that are made by conventional machining. Most of the growth in the powder metallurgy market has been due to gradual acceptance by auto-makers. (Source: Forbes, 26 June 1989)

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Impregnation by improving final quality, permits manufacturers to use casting and powdered methods

Benefits of impregnation include preventing "leakers" to supply pressure-tight castings and increased capacity to plate or anodize the impregnated parts. Casting impregnation can be achieved successfully using several different techniques that use various sealant materials. Each technique, kind of sealant, and method/sealant mix has certain advantages and disadvantages. The most fitting combination of system, sealant, casting recovery and cost may depend on the particular customer's preference or the amount of porosity found in the castings. Manufacturers generally will try to combine the impregnation system and sealant supplying the best recovery rate at the lowest feasible cost. (Source: Metal Fin, June 1988)

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Powder metallurgy is becoming an increasingly pleasing alternative to conventionally formed metal parts, and the developing area of metal injection moulding may get back some metal markets lost to plastics. Rapid solidification technology, high-temperature sintering, hot forging, hot isostatic pressing, and such new materials as nickel aluminides continue being developed. Industry experts expect more expansion into the aerospace market and other areas needing complex forming and/or unique alloy properties. While 75 per cent of all powders now produced are iron or steel, the ways the powders are being blended and the higher-property alloys being produced show that P/M may be the key to better future metal parts. Due to the microstructural homogeneity and material integrity existing in P/M parts, designers increasingly see them as a reliable, cost-effective alternative to ingot metallurgy parts. (Source: Material Engineering, November 1987)

Ultrapure silicon nitride

An expanded line of advanced non-oxide ceramic powders from Superior Graphite now includes ultra-pure silicon nitride. Originally produced by KemaNord of Sweden, Siconide U powders have less than 150 ppm of iron. They are particularly well suited for gas turbines, high-temperature bearings, and other high-temperature applications for which absolute purity is critical.

Produced by computer-controlled nitridation of a proprietary high-quality silicon (KemaNord Sicomill Grade 5), all chemical processing is completed before nitriding, eliminating most production problems (such as difficulty in dispersing silicon carbide whiskers throughout the powder). The resulting silicon nitride has less than 50 ppm aluminium, 30 ppm calcium, 0.3 per cent carbon, and less than 0.2 per cent elemental silicon. [Dr. Read Stewart, Superior Graphite Co., 120 S. Riverside Plaza, Chicago, IL 60606, USA.] (Source: New Materials World, March 1989)

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Making film-sintered body of mono-dispersed fine-particle ceramic

A new method has been developed for producing a sintered film of "mono-dispersed" ceramic fine particles, that is ceramic with uniform particle size. In the case of ordinary ceramics, as the raw material particle size gets smaller and more uniform, the material becomes stronger and more stable. However, in the case of mono-dispersed ceramic fine particles, two major problems arose: (1) only bulk-sintered body could be prepared; and (2) while sintering, the fine particles would cluster and grow into particles of five times larger diameter than the original size, thus spoiling the original merit of the raw material being a fine particle.

The mono-dispersed ceramic fine particles used in this development for sintering are TiO_2, synthesized from metal alkoxide, with particle size 0.45 µm. In order to mould it, add alcohol and different kinds of binder (the composition of the binders has not been made public) to the TiO_2 powder and make a slurry. Pour this slurry into a gypsum mould and let the gypsum mould absorb the alcohol contained in the slurry. Then part-dry the material, remove it from the mould and calcine at 1,060°C for 120 minutes. The sintered body produced by this method becomes a film with density of nearly 100 per cent and thickness of 5 mm. The average particle diameter of the sintered body is 0.99 µm, which means that the growth of the particle was only 2.2 times the raw material.

Research is in progress on synthesizing mono-dispersed ceramic films with different characteristics to develop new complex ceramics by lamination. [Professor Seiki Kato, Tokyo Institute of Technology, Faculty of Engineering Inorganic Materials, 2-12-1 Ookayama, Meguro-Ku, Tokyo 152, Japan. Tel: (03) 726-111.] (Source: New Materials World, March 1989)

* * * * *

Ultra-fine powders through spark erosion

Spark erosion has been used to produce research quantities of very-rapidly-solidified micropowders, and for production of such specialty powders as MnAlC permanent magnet powders. Researchers at the General Electric Corporate R&D centre have demonstrated that spark erosion can be a versatile and economical method for producing a wide range of metal, alloy and semi-conductor powders.

The process involves the use of repetitive spark discharges among chunks of material immersed in a dielectric liquid. Whenever two electrodes are in close proximity in such a dielectric, the spark discharge between them can produce highly localized heating of spots on the electrodes. If that heating is over the melting temperature of the electrode (or materials suspended in the dielectric fluid), molten droplets or vaporized material can be evolved. These particles can be condensed out or frozen by the dielectric fluid to produce powders. Because the particles are evolved in a quenching fluid, cooling can be extremely rapid. Particles from 5 nanometers to 75 micrometers have been produced.

The production rate and size distribution depend on the pulse power characteristics, the dielectric fluid, and the electrode materials. Generally, the rate of production increases with voltage and capacitance. Small units, it was shown, are quite capable of producing substantial amounts of useful powders: a 450 V oscillator produced about 77 gram/h of powders with diameters less than 20 micrometers; total power consumption was 20 kWh per kilogram of acceptable powder.

In the case of alloys, the composition of the powder produced may differ somewhat from that of the electrode due to microstructure - that is, if the grain size of different phases is larger than the spark-eroded particle size, compositional inhomogeneity is to be expected. Although it is difficult to predict, homogenization is favoured by the use of grain-refined materials and higher-energy sparks. [J. L. Walter, General Electric Corporate Research & Development, P.O. Box 8, Schenectady, NY 12301, USA. Tel: (518) 387-7574. Fax: (518) 387-7597.] (Source: New Materials World, June 1989)

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Powder remains a dream material

Producing alloys by powder metallurgy for the aerospace industry represents an unfulfilled dream for many engineers. The potential of this process for manufacturing materials remains largely untapped.

Currently, the use of powder metallurgy has been limited to special segments of gas turbine engines that cannot be produced by any other process.

The situation can be depicted as a love-hate relationship that began in the early 1970s. Great expectations were projected by the love side of the relationship, whose advocates pointed out all the advantages of powder metallurgy materials.

The advantages listed by proponents include the ability to make complex alloys not possible with conventional ingot techniques, freedom from segregation, fine grain sizes, and super-plastic forming and hot consolidation processes to produce near net shapes.

All of this was coupled with lower projected costs.

Factual results, on the other hand, led to the hate side of the relationship. Costs turned out to be higher, not lower, than material produced by other processes. Quality failures further exacerbated the situation, with the subsequent development of serious reservations concerning powder metallurgy product usage.

The most serious of the problems involved poor cleanliness, inadequate bonding of powder particles, contamination with organic compounds that detrimentally affected grain boundaries and distortion of near net shapes in some consolidation processes.

Hindsight shows that the powder metallurgy industry should have been less stilted in its views and more careful in adapting and tapping the potential of this process.

Powder metallurgy alloys such as Rene 95, IN100 and AF2-IDA are being used today in both large and small gas turbine engines. These complex alloys, which cannot be made satisfactorily any other way, provide higher strength or higher temperature capabilities that allow engines to develop more thrust and/or operate with greater efficiencies while at the same time offering increased reliability.

Powder manufacturing processes - gas atomization, spinning disk and vacuum atomization - have been developed that now provide the cleanliness and freedom from organic compounds that initially caused quality problems.

Additionally, consolidation techniques - extrusion, consolidation at atmospheric pressure and hot isostatic pressure - have been refined to provide quality billet and preforms that give consistently high properties.

Today's powder metallurgy materials are the cleanest, most structurally uniform and reliable materials available. Not one failure has been reported in thousands of turbine disks used in critical rotating-part applications.

Faster growth of powder metallurgy usage is being hindered by a number of factors, most of which centre around the fear of change and some myths based on past history.

We all recognize that progress can only occur with change, but we are reluctant to make changes unless a major problem arises. This human trait is reinforced by aerospace specifications that lock in material and processes in the name of quality and safety.

Basically, the specifications are a crutch for not doing our jobs of adapting new technology in a manner that will ensure quality and safety while still making technical progress. The situation is further complicated by the NIH (not invented here) syndrome, which is very much alive in the aerospace industry.

The myths about aerospace powder metallurgy materials are many and varied.

Myth No. 1 is that these materials exhibit porosity, lack ductility and show inconsistent properties. The fact is that these materials are fully dense (100 per cent) and exhibit, at the very least, properties equal to or better than cast and wrought counterparts.

They are used for the most critical of rotating parts in gas turbine engines with unparalleled performance, so why not use them for static structural parts?

Myth No. 2 is that the powder metallurgy process can be applied only to advanced materials such as Rene 95 and IN100 specifically designed for that purpose.

The fact is that the technology can be applied equally well to high-volume nickel-base alloys used in the gas turbine industry, which include Alloy 718, Waspaloy and Rene 41.

Myth No. 3 is that the cleanliness of powder metallurgy products is inferior, based on past quality problems associated with inter-metallic and non-metallic inclusions as well as undesired precipitates decorating grain boundaries as a result of organic contamination.

While it is not possible to make perfect material, the industry as a whole has made great strides in powder handling techniques. Major suppliers such as Special Metals Corp., the Cytemp Specialty Steel division of Cyclops Corp., Cameron Iron Works and Crucible Materials Corp. all have developed and implemented improved processing and handling techniques to produce material that exceeds the sonic quality of any cast and wrought alloy.

This improvement has resulted from the control of melting operations through the final consolidation operation. Changes include complete inert handling of powder, pneumatic conveyance of powder through the entire process and clean room environments.

Myth No. 4 is that the cost of aerospace powder metallurgy materials prevents use in all but the most critical applications, which cannot be satisfied any other way.

While it is true that current costs are high in comparison to some cast and wrought alloys, and that there is a low end of the price range in which this technology cannot compete, one must look at the reasons for this disparity. The fact is that raw material costs are similar and that the major cost differences are associated with strict processing requirements, limited capacity and small market volume.

Solving past problems has resulted in quality and testing requirements that far exceed any cast and wrought material used for the same application.

Restrictive powder particle size lowers yields and significantly raises costs. Limited consolidation sources raise costs, and low volumes do not allow economy of scale.

Suppliers, however, are eager to solve these problems by putting in more capacity and using advanced technology to improve productivity. Efforts already are being made to introduce computerization of the powder metallurgy process and to use robots to do some of the more expensive and routine manual labour.

The trials and tribulations of the aerospace powder metallurgy materials will be overcome. The technical problems have been or are being solved. The cost problem will be solved and the process will ultimately allow a net cost saving to end-users.

Furthermore, the powder metallurgy process will become more viable as raw material costs increase.

The greatest deterrent to progress in this field is a psychological one. Our inherent fear of change, the NIH factor and arbitrary rejection of these materials or processes by managers may be the insolvable problems.

If we do not overcome these barriers, this advanced technology, in which the United States currently enjoys a distinct advantage, will flow to other parts of the world by default. (Source: American Metal Market, 9 May 1989)

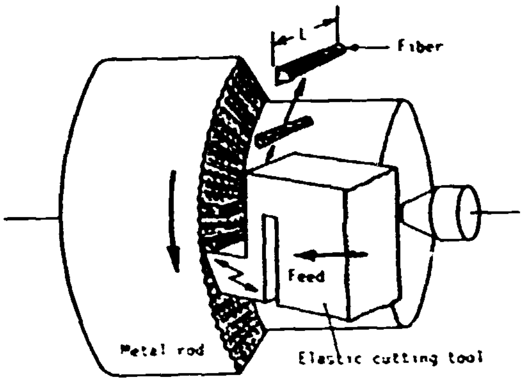

Fig. 1 Schematic of the manufacturing method for fine short metal fibers by chatter machining.

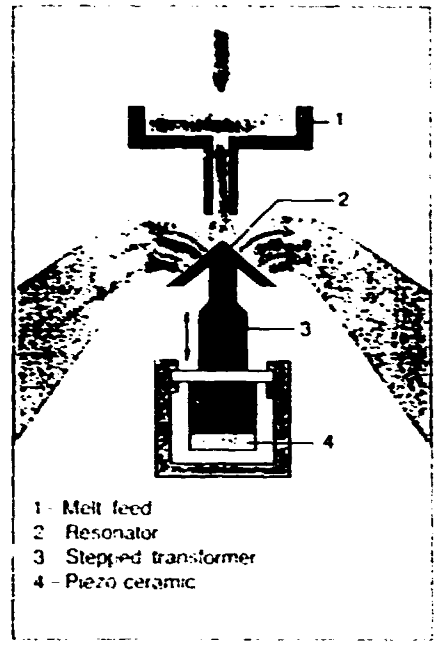

Fig. 2 Schematic design of ultrasonic capillary wave atomization.

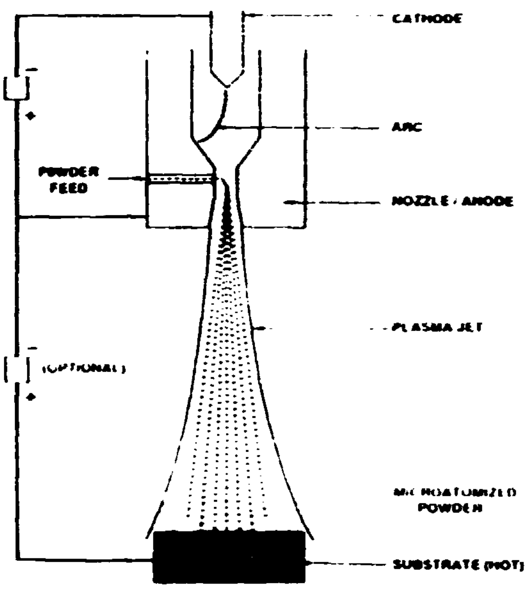

Fig. 3 Hot substrate microatomization.

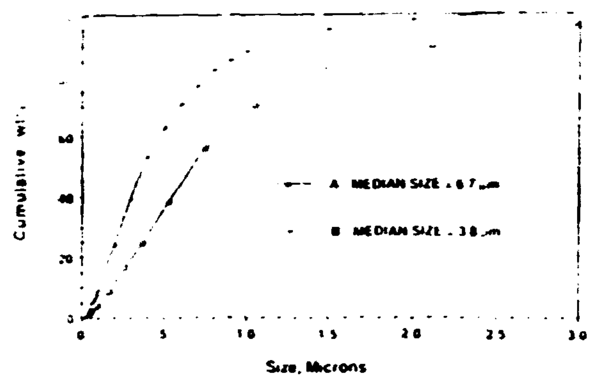

Fig. 4 Particle size distribution of MICROATOMIZED™ copper powder.

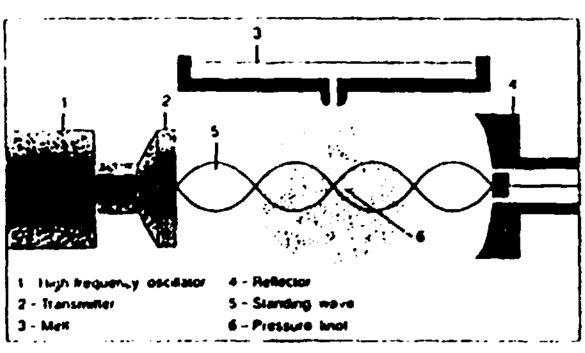

Fig 5 Schematic design of ultrasonic standing wave atomization.

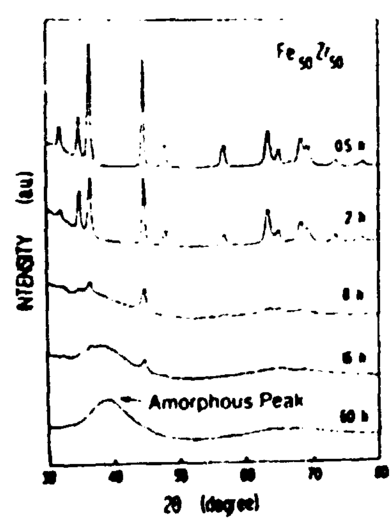

Fig. 6 X-ray Diffraction Patterns of $Fe_{50}Zr_{50}$ Powder Particles After Different Milling Times

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9. NEW R&D CENTRES IN POWDER METALLURGY

Powder metallurgy R&D

Indo-Soviet Powder Metallurgy Centre in Hyderabad.

A general agreement has been arrived at for the establishment in India of a new joint Indo-Soviet Advanced Powder Metallurgy Research and Development Centre. This was stated by Academician Professor G.I. Marchuk, President of the USSR Academy of Sciences, at a press conference to mark the conclusion of the first meeting of the Indo-Soviet Joint Council for the Integrated Long-Term Programme (ILTP) of Co-operation in Science and Technology.

The setting up of the Powder Metallurgy Centre, Prof. Marchuk said, would help broaden the already existing collaborative research programme between the Defense Metallurgical Research Laboratory (DMRL), Hyderabad, and the Byelorussian Academy of Sciences. The centre is to be set up in Hyderabad, next to the DMRL campus itself.

Professor Rao (leader of Indian team) said 65-70 specific projects had been clearly identified in this meeting. Seventy Indian laboratories and research institutions will collaborate with about an equal number of Soviet institutions. Over 200 Indian scientists will visit the Soviet Union under this programme over the next two years and a similar number of Soviet scientists will visit India. In fact, nearly 70 Soviet scientists have already come to India for initiating several joint projects since the ILTP was signed last year.

"Over a dozen really outstanding results have already come out of projects undertaken under this collaborative programme", Professor Marchuk said. He added that these would not have been possible if the two groups of scientists had worked independently. Among the projects Professor Marchuk highlighted was electron accelerator-based technology, being actively carried out between the Bhabha Atomic Research Centre and the Institute of Nuclear Physics of the Siberian Academy of Sciences at Novosibirsk. This co-operative project, he said, was now operative being used for irradiating materials and food. In this field the other relates to the design and installation of synchrotron radiation source to study interaction of radiation with matter which will be useful in sub-micron microelectronic technologies and a new cycle of biology research.

Apart from powder metallurgy, production of basalt fibres for the first time have also been achieved by Indian scientists. These fibres will be soon sent to the Institute of Organic Chemistry of the Siberian Academy of Sciences.

Collaboration in the area of catalysis between the National Chemical Laboratory, Pune, and the Institute of Catalysis of the Siberian Academy of Sciences was described by Professor Marchuk as "unrivalled anywhere in the world". According to Dr. L.K. Doraiswamy, Director, NCL, this process is altogether new and quite different from the NCL's process for the several zeolite catalysts it had earlier developed.

Basic research: According to Professor Rao, about 50 per cent of the projects taken up relate to basic research while about 30 per cent are product-oriented technologies like very special lasers, superhard materials, diamond film coating of materials, special catalysts, vaccine production, etc. The remaining 20 per cent would constitute areas where "we would like to develop technologies where both the countries will benefit". (Extracted from The Hindu, 1 April 1988)

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Powder metallurgy R&D centre proposed

A plan to bolster the international competitiveness of the United States powder metals industry by creating a research and development centre was awaiting the approval - and the appropriations - of Congress. The proposal, presented by the Industry to a group of legislators, argued that government support is needed to establish a powder metallurgy R&D centre as a key way of keeping the domestic industry ahead of the global pack.

A group of legislators with powder metallurgy firms in their districts or states has shown support for the idea; they were expected to seek funding for it as part of the fiscal 1990 appropriations process under way on Capitol Hill.

The programme is necessary because the US is in danger of losing its leadership position in powder metallurgy, according to the Center for Powder Metallurgy Technology, the non-profit, industry-supported foundation which produced the proposal.

Though the US is the world leader in powder metals production and powder metals technology, major European countries and Japan have targeted the growth of the powder metals industry world-wide.

There are a number of warning signals, says the proposal. The Finnish Government and industry have launched an $11 million, three-year programme for powder metallurgy research. Taiwan and Japan reportedly have efforts under way to upgrade their domestic industries.

"In addition", the proposal notes, "the European Economic Community countries and several non-community countries are sponsoring a co-operative international research effort called COST, short for European Co-operation in the Field of Scientific and Technical Research".

There is reason for concern about Eastern Bloc activities, too: Powder metallurgy has been "prominently mentioned" in several Soviet five-year plans, according to the technology centre. "In fact, the Soviet Union probably has more people working in powder metallurgy research than any other country in the world. Some Soviet powder metallurgy research institutes have staffs of over 2,000 people".

What would be the agenda for R&D projects? Efforts to develop a complete structure of standards for the manufacture and testing of materials would have high priority. One of the projects outlined in the proposal would seek to "develop and understand powder flow under pressure, tooling stress, design and dynamics of forming operations using analytic techniques such as finite element analysis and soil mechanics".

The other projects, as outlined in the proposal, would include these:

- Develop a data base and network for exchange of new and existing technologies;
- Develop methods to eliminate harmful effects of admixed lubricants using non-harmful substitutes, die coatings and die materials;
- Develop alternate sintering methods or technologies;
- Develop techniques such as robotics, automated adjustment systems, automation applied to short runs, automated inspection systems and automated material handling systems;
- Monitor marketability of powder metallurgy versus competitive technologies and off-shore products;
- Study the means of further penetrating and expanding off-shore markets. (Extracted from American Metal Market, 13 June 1989)

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10. TRENDS IN MARKETING

Changing demands remold the p/m industry's make-up

Powder metallurgy was developed largely by post-World War II entrepreneurs who imagined the possibilities of a new technique and set up mom-and-pop-style shops to make it a working technology.

Now, four decades later, many of the original moms and pops are eying retirement, and those who remain are imagining a far different business when powder metallurgy celebrates its fifth or sixth decade as a full-fledged industry.

Realities of the marketplace and the demands of hard-driving customers are pushing the more than $1 billion North American p/m industry towards a new sophistication, and towards an industry consolidation that could compress the number of parts-making companies in the United States to 50 in 1999 from 150 now, according to some estimates.

Today, about 70 per cent of the p/m companies take in less than $20 million from annual sales, while the more successful companies of the future probably will have sales of more than $100 million, according to some estimates.

Bulk suppliers of metal powder already are quite large. But they face demands for ever-better materials because of pressures on parts makers.

A complex of factors is at work to force powder metallurgy into a new era.

The changes are looming at the same time that the technology seems to have gained a new level of acceptance among customers.

"I am begining to sense that powder metallurgy in the past couple of years has matured in the sense that it is now truly a technology in its own right, a technology that is very often now a preferred solution", said the president of the Metal Powder Industries Federation (MPIF).

In the past, powder metallurgy was the stepchild. It was very often an alternative, and not the choice at the top of a customer's list.

Users increasingly are recognizing possibilities for gains in strength and complexity of parts, as well as in costs of producing them.

Contributing to the new view are major gains in the performance of parts made by powder metallurgy.

For example, Ford Motor Co., Dearborn, Mich., is using a p/m forged connecting rod in 1.9 liter engines for its Lynx and Escort models. Early this year it announced plans to install p/m connecting rods in its new modular engine, scheduled to go into production in 1991. The rod is said to be stronger than conventional forged parts.

Such significant gains in the automotive sector are particularly important, because the car industry accounts for some 60 per cent of p/m parts consumption, industry executives said.

For the same reason, the policies adopted in recent years by the big automakers, led by Ford, have spurred some of the fundamental changes under way in the p/m industry.

Powder metallurgy companies have been in a heated competition to win coveted preferred-supplier among the auto companies, as the carmakers pursue tough quality standards in line with their plans to develop long-term relationships with a smaller number of suppliers.

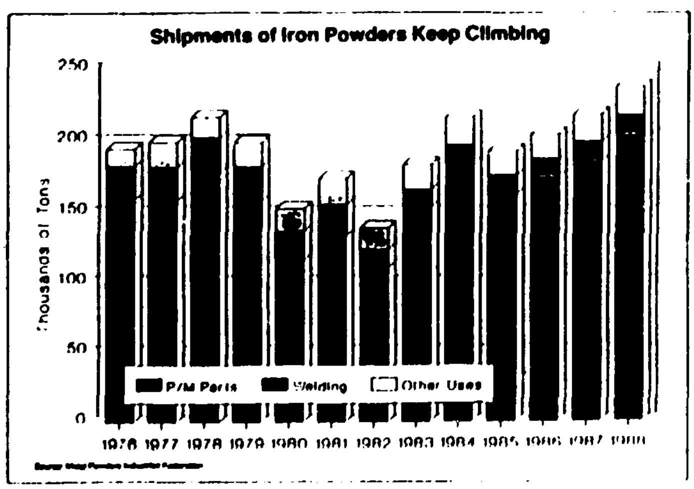
Because p/m is so heavily dependent upon the auto industry as a market for its goods, the automakers' requirements alone are spurring significant changes in powder metals. Carmakers' moves to reduce their supplier base probably will be the cause of some industry consolidation, according to industry executives and observers.

The US p/m industry is the largest and most advanced in the world, accounting for about 60 per cent of p/m output.

In 1988, shipments of iron and steel powders reached a record of more than 235,000 short tons, up about 9 per cent from 1987, according to MPIF figures.

North American powder makers are entering a period of stiff competition, partly because of the entry of a Japanese-owned competitor, Kobeico, a joint venture involving Kobe Steel Ltd.

Where shipments of iron and steel powder rose to about 235,000 tons in 1988, they are expected to decline slightly from that record in 1989. But with Kobelco's addition, North American capacity would hit 360,000 tons, according to MPIF calculations.



(Extracted from American Metal Market, 13 June 1989)

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11. CONCERTED EFFORTS IN POWDER METALLURGY WITHIN THE EUROPEAN COMMUNITY

Osvaldo Morocutti*) and Roland Stickler**)

*) Secretary, COST Activities, Materials Sciences, CEC-Brussels, B

**) Chairman, Management Committee COST-503 Powder Metallurgy, University of Vienna, A.

ABSTRACT:

A joint European policy on research in science and technology of materials appears necessary for Europe's economic independence and to secure Europe's industrial and scientific competitiveness. For this, joint multinational R&D policies, coordination of national policies, and definition of projects of interest to the European Community are required. Such research activities can be carried out as Community funded programs, for which participation is open only to Community member states, or in the form of concerted research actions at which all OECD states, and also Yugoslavia, may take part. These COST-actions are operating in various fields of materials research, the action COST-503 is solely devoted to concerted research work in the field of powder metallurgy. The European activities in powder metallurgy will be discussed in detail.

INTRODUCTION:

The changing face of world politics, staggering technological developments, the oil and energy crises, and new awareness of environment and resources emphasised the key role of research and technology for future developments. It became evident that a joint policy on science and technology is absolutely necessary for Europe's independence and that European industrial and scientific competitiveness could only be secured in the long term by the

* creation of a joint multinational R&D policy for the European Community,
* coordination of national policies in Europe,
* definition of projects in the fields of science and technology of interest to the Community States +).

Based on these considerations the Council of the European Community adopted already 1974 a resolution calling for a framework program for Community research, development and demonstration activities. According to this action-programmes are established for specific scientific and technical objectives. In each case a decision has to be arrived at to which extent the objectives can be attained by the Community's own research capacity (e.g. at the Joint Research Center), or in form of contractual research (preferably involving financial contributions by the contractors), or by coordination of national activities (e.g. in form of concerted actions).

Community actions must satisfy following criteria:

* research on a large scale for which individual Community states could not provide the necessary means,

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+) At present, the European Community consists of the following member states: B, D, DK, E, F, GR, I, IRL, L, NL, P, and the UK.

* research the joint execution of which would offer obvious financial benefits (even after taking into account the extra costs inherent in all international cooperation),
* research which, because of the complementary nature of work being done nationally in a given field, enables significant results to be obtained in the Community for the case of problems whose solution requires research on a large scale,
* research which helps to strengthen the cohesion of the Community, reduce the technological gaps, to unify European scientific and technical areas, and
* research leading to the establishment of uniform standards.

COMMUNITY FUNDED MATERIALS RESEARCH PROGRAMS:

The research programs cited in the following are carried out with partial Community funding and should serve as typical examples for "Community programs". Participation in these programs is in general limited to Community member states.
The BRITE program (Basic Research in Industrial Technologies for Europe) was adopted in 1985 with Community funding of 125 Mio ECU for the period 1985-1988, to which industry will add an identical sum. This program aims to stimulate the development of a solid foundation of advanced technologies to support traditional Community industries. Industry and institutions are encouraged to work together on certain projects. The BRITE program covers pre-competitive research as an intermediate stage between fundamental research and development work preceeding marketing. The research topics include new materials and powder metallurgy.
The EURAM program (European Research on Advanced Materials) aims to combine basic materials research with the engineering development of advanced materials in order to help to raise the technological level of industrial products and to compete better on world markets. The program includes creation, development and use of new materials as well as upgrading of more conventional materials to higher levels of performance. The EURAM program takes into account the existence of national programs in order to

provide cohesion between complimentary activities. The main topics of EURAM program are listed in the Table 1, projects on powder metallurgy of Al-, Mg- and Ti-alloys as well as advanced ceramic and composite materials are part of the EURAM program (1).

Table 1: Research Topics of the EURAM Program

a. Metallic Materials:

Al-, Mg-, Ti-alloys
Electrical contact materials
Magnetic materials
Coating materials
Thin-walled castings

b. Ceramic Materials:

Optimization of technical ceramics
Compatibility of metals and ceramics
Cermets
Properties of ceramics at elevated temperatures

c. Composite Materials:

Organic composites
Metal-matrix composites

d. Advanced Materials for Special Applications:

Shape-memory alloys
Metallic glasses
High-damping materials, etc.

RESEARCH PROJECTS BY COORDINATION OF NATIONAL ACTIONS:

It was soon recognized that the geographic framework of cooperation should not be limited by the Community's frontiers but that neighbouring European states with an equivalent level of technical development were also interested to take part in the common endeavor. It was anticipated that this extension of cooperative efforts would lead to quicker success at lower costs. The framework and forum for international research cooperation was termed COST, i.e. European Cooperation in the field of Scientific and Technical Research (2). Participation in COST is open to all OECD Member States which signed the agreement at the Ministerial Conference on European Cooperation in Scientific Research, i.e. the EEC-Countries and the non-EEC Countries A, CH, N, S, SF, TR, and, in particular, also Yugoslavia, Fig. 1.
COST is closely bound up with the creation of important sections of the European community research policy and constitutes a framework for cooperation between the European community and European non-community states in the field of research and development. Multiannual research programs are decided by the Council of the European Communities. Joint research planning is carried out through a concerted action system which means that financing is provided by individual states, only results are exchanged across national boundaries. It is important to note that all participating states enjoy the same rights whether they are Community members or not.
The COST-Action has no legal personality but possesses its own particular institutions and its own jointly managed financial resources, adopted in a joint resolution of the European Research Ministers in 1971. The Community-COST Concerted Agreement can be considered as a unique model for formal international agreements which since has been copied many times. Memoranda of Understanding are the expression of the will of the signatories to coordinate projects on the basis of national laws, that duplication can be avoided and results can be exchanged without infringing industrial property rights.

There are four possible categories of cooperation, i.e.:

Category I: Community programs with which interested COST States which are not members of the Community may be associated,

Category II: COST projects which alsc form the subject of a Community program,

Category III: COST agreements where there is parallel participation by Community states, the Community itself, and by COST states which are not members of the Community, and

Category IV: COST projects where there is no participation by the Community as sucn.

In most cases the Commission of the European Communities provides for the secretarial services to avoid states having to carry out their own finance authorization procedures. The commission is also kept informed of the progress of the projects.

PREPARATION OF COST-PROJECTS

The preparation of COST projects starts with a submission of proposals to the COST-Committee of Senior Officials which decides whether a COST-project in this particular field should be undertaken. If so, a working party is entrusted with further planning. Next, the states representing COST consider whether they wish to participate in the proposed joint research project. With signing of a Memorandum of Understanding by the Signatory Countries an implementation phase is initiated. All COST states are invited by the Commision to an initial meeting of a project committee, which selects a chairman and co-chairman, agrees on rules of procedures and decides on the Secretariate. This is shown schematically in Fig. 2.
Sub-committees may be appointed to ensure that research topics and the research contributions made by the parties are exploited

to the best. Regular coordination meetings or subgroup meetings may be organized to review the status and progress of individual projects. Annual reports and a final report at the end of the working period (in most cases after 3 years) must be issued to all partners of the collaborative projects to document the achievements of the COST action.

CONCERTED ACTIONS IN THE FIELDS OF METALLURGY:

In the fields of metallurgy several concerted actions have been initiated. A program on gas turbine materials (COST-50) arose from the recognition that Europe needed collaborative efforts to maintain future competitive positions of the relevant European industries. The initial first round of three years was followed by two additional three-years working periods, participating were major companies involving building of gas turbines, suppliers of components and alloys, as well as users, research institutions and universities. Powder metallurgical projects formed part of this program with the major objectives in the selection of fabrication parameters to achieve optimum mechanical properties of gas turbine components. Grain size control and properties of dispersion strengthened alloys were of particular interest. Results of this Action were presented in accordance with the prescribed reporting procedures and in addition summarized in two international conferences organized by the COST management committee (3, 4, 5).

It soon became apparent that the concerted activities should be expanded into materials for broader areas of energy production and conversion. This need resulted in COST 50i, which included powder metallurgy in research projects on creep and fatigue properties of oxide-dispersion strengthened superalloys for blading application. Alloys have been produced and effects of microstructure on mechanical properties determined. Further research and development efforts were devoted to improve disk materials prepared from powders produced by rotating electrode

processes. In summary it can be stated that the Actions COST-50 and 501 have provided the participants with information by a combined use of resources which would not have been available to a single institution. The Actions COST-50 and COST-501 have created significant savings for the European gas turbine industry and associated firms.
In view of the success of these concerted actions in metallurgy further COST-programs were initiated, i.e.

COST-502, Corrosion in the construction industry (32 projects)
COST-503, Powder metallurgy (58 projects)
COST-504, Advanced casting technologies (36 projects)
COST-505, Materials for steam turbines (54 projects), and
COST-506, Light alloys for transport systems (in preparation).

To date more than 340 professional man-years have been invested in the COST-actions 501 to 505. The participants are associated by 60% with industry, by 40% with research institutions or universities. It is interesting to note that while the guide lines of COST limit public funding to 50% of total costs, approximately 8% of the industrial participation is self-supporting (zero public funding). Apparantly the value of information received in the course of collaboration is sufficient to justify corporate fundings.
A more detailed description of the Action COST-503 "Powder metallurgy" is given in the following.

COST-ACTION 503 ON POWDER METALLURGY:

In view of the advantages of the powder metallurgical processes (savings in energy, materials, taylor-made properties) this particular industry has been considered as one with a high growth potential. Experience has shown that the lead time between R&D and transition into production can be relatively short, so that under appropriate support-measures technological progress can be

achieved rapidly.
These considerations resulted in a project proposal and a Memorandum of understanding for a COST-Action on Powder Metallurgy, COST-503, which was signed by A, B, CH, DK, FRG, SF, S and the UK in 1984, Fig.3. The basic objectives of COST-503 include:

* to provide cooperation between the industrial and research organizations in Europe (of which more than 100 are engaged in pm-processing, technical development and research work)
* to support and coordinate these activities and to improve the competitive position by work-sharing
* to provide collaborative research on a wide range of topics of particular relevance to industry.

The main areas of this pm-research effort are listed in the following:

* Powder metallurgy of light metals and alloys, including projects on:
  - powder production by inert gas atomisation
  - powder compaction and extrusion, evaluation of mechanical properties, microstructural investigations
  - fatigue testing of ODS and RS alloys, extruded and forged parts
  - investigations on alloy TiAl6V4 with respect to different powder sources and techniques of consolidation

* Powder metallurgy of hard materials, including projects on:
  - ceramic materials (evaluation of boron carbide, cemented carbides, fine grained aluminum oxides)
  hard metals and heavy alloys (cemented carbides from recycled powders, impuritiy effects, characterization of carbide powders, effects of trace elements)

* Powder metallurgy of Fe-base alloys, including projects on:
  - HIP of corrosion resistant alloys
  - HIP processing
  - fatigue design of connecting rods of pm-materials
  - optimization of properties of pm-products by heat treatments
  - processing with binder materials
  - production of complex shaped parts by joining
  - heat resistant parts by joining
  - high performance gears
  - near net shape high speed steel parts.

For the first round of COST-503 a total of more than 50 projects have been positively evaluated by an Expert Team and recommended for national funding by the Management Committee. Table 1 gives a listing of the titles of projects presently in progress. As an average, the size of the projects is one professional man-year per year. This approximates a total value of all efforts in COST-503 to 7,5 Million ECU (European Units of Account).

An extension of COST-503 for a second round starting from 1988 appears feasible, procedures and topics will be decided upon by the Management Committee.

Work in most of the projects has been started during 1985 and some of the first annual progress reports (classified) have been completed and distributed amongst project partners. Annual Reports of the Management Committee for 1984 and 1985 have been issued (6, 7) and are available on request from the COST-Secretariat, Brussels (EUROP-COST, Secretariat, Rue de la Loi 200, B-1049 Brussels, B).

Table 1: Summary of titles of projects carried out in the concerted action COST-503

1. Powder Metallurgy of Light Metals and Alloys:

Country: no.	Project title:
A 2	Preparation of high-class Ti-parts by powder metallurgical technique
CH 1	High performance P/M Al alloys, produced via extrusion
CH 2	Mechanical properties & microstructure of high-temperature powder metallurgy Al alloys
D 1	Process development for rapid solidification of titanium alloy powders by inert gas atomization
D 22	Mechanical properties and microstructure of high-temperature powder metallurgy Al alloys
D 41	Process development for rapid solidification of Al alloy powders by inert gas atomization
D 42	Mechanical properties and microstructure of high temperature powder metallurgy Al alloys
S 9	Production and assessment of rapidly solidified temperature aluminium alloy
UK 5	Production and assessment of forgings made from mechanically alloyed aluminium alloys

2. Powder Metallurgy of Hard Materials:

Country: no.	Project title:
A 3	Development of methods for trace element analysis for PM-materials
A 9	Characterization of powders in view of their application in hard metal manufacturing and their influence on the mechanical and technological properties of cemented carbides
A 10/13	Investigation of thermophysical properties of compacted and of sintered parts
A 11	Effect of trace elements on metallurgical and microstructural features of sintered & deformed heavy metals
B 2	Preparation and characterization of cemented carbide parts from recycled powders
CH 6	Impact and crack propagation testing of sintered products
CH 7	Morphological description of sintered polycrystals
D 7	Densification of boron at relatively low temperatures by hot pressing & hot isostatic pressing
D 32	Investigation of the influence of powders, especially TaC, NbC, HfC and WC, on the performance of cemented carbide milling grade, based on WC-TiC-TaC-Co and WC-Co

D 37 Characterization of carbide powders in view of their application in hard metal manufacturing & their influence on the mechanical & technological properties of these hard metals, in particular based on WC-TiC-TaC-NbC mixed carbides

DK 2 Quality examination of powder metallurgical products by use of non-destructive control methods

S 4 Densification of fine grained Boron Carbide by hot isostatic pressing

S 5 The characterization of impurities in cemented carbides and their influence on technological properties

S 11 Effect of trace elements on metallurgical and microstructural features of sintered and deformed heavy metals

SF 1 Preparation & characterization of WC and cemented carbide powders for hard metal production

3. Powder Metallurgy of Iron-based Alloys:

Country: no.	Project title:
A 1	Preparation of complex PM-Iron parts by joining methods and evaluation of the mechanical properties of the joints
A 5	Preparation and evaluation of PM composite materials for application as wear resistant components
A 12	Fatigue and fracture behavior of PM-components
D 5	Hot isostatic pressing of highly corrosion resistant P/M alloys
D 16	Preparation of complex & multifunctional sintered steel parts by joining two or more different PM parts. Evaluation of mechanical properties of the joints
D 19	Powder metallurgical production of near net shape parts of high speed steel by direct sintering
D 20/36	Fatigue design of PM-connecting rods using sintered steels
D 21	Use of powder metallurgy for load bearing applications such as gears
D 27	Non-destructive evaluation of residual stresses in hardmetals and ceramics
D 30	Preparation of sintered, wear resistant parts by joining and evaluation of their properties

D 34	Influence of segregation behavior of trace elements on the mechanical properties of P/M-materials
D 35/33	Improvement of fatigue & toughness properties under constant & variable amplitude loading for alloyed sintered steel by optimizing heat treatments
D 39	Preparation & evaluation of P/M composite materials for application as wear-resistant components
D 40	Influence of inclusions on the mechanical properties of powder metallurgical products
D 43	Use of powder metallurgy for load bearing applications such as gears
D 45	Influence of segregation behavior of trace elements on the mechanical properties of PM materials (sintered steel)
DK 1	Influence of inclusions on the mechanical properties in powder metallurgical products
S 1	Fatigue design of PM-connecting rods using sintered steels
S 2	Heating of powder compacts
S 3/6	Improvement of mechanical properties of alloyed sintered steels by optimising heat treatments
S 8	Hot isostatic pressing of highly corrosion resistant PM alloys
S 10	The use of powder metallurgy for load bearing applications such as gears

UK 4 The development of high density, high performance sintered powder metals components using Hot Isostatic Pressing

UK 9 Development of powders for the production of high speed steel bars & preformed components by a new process

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7. COST-503, 2nd Annual report of the Managment Committe on its Activities in 1985, EUCO/PM/01/86, DOK.XII/335/86 of April 1986

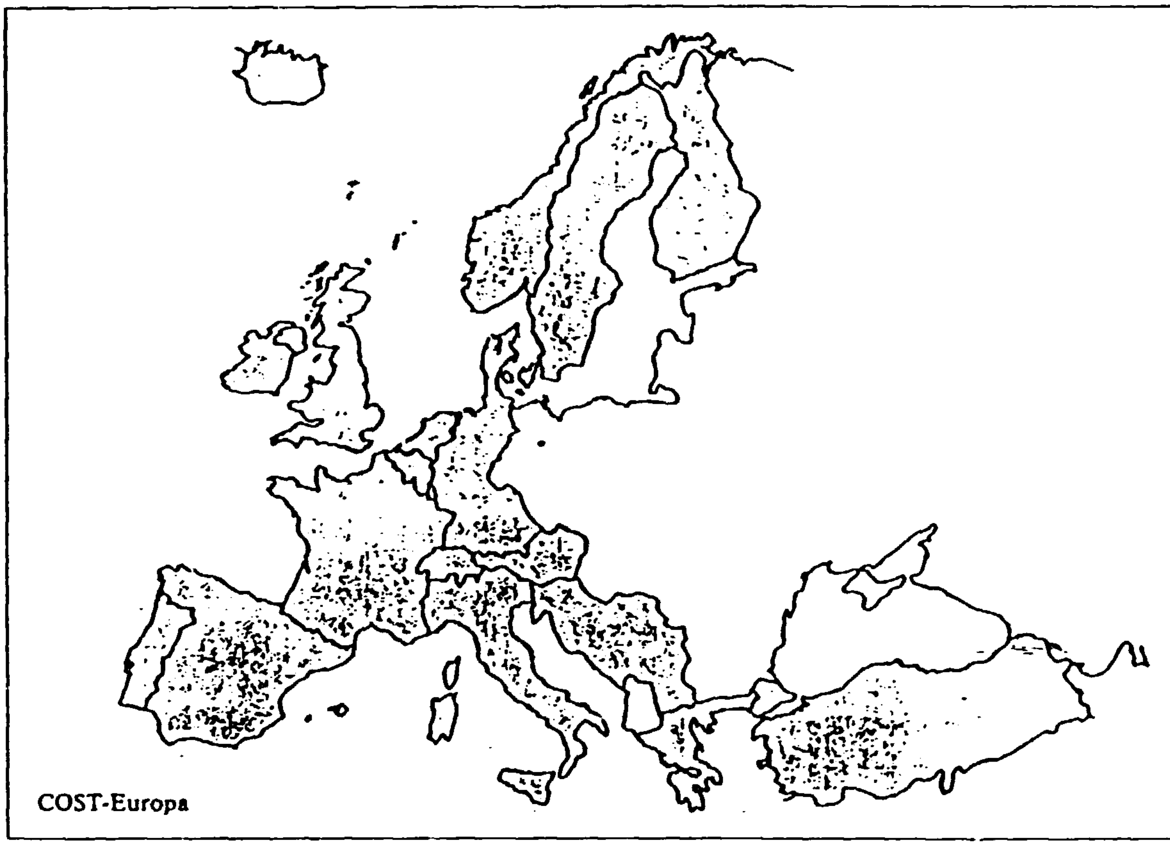

Fig.1: Countries participating in COST-Actions

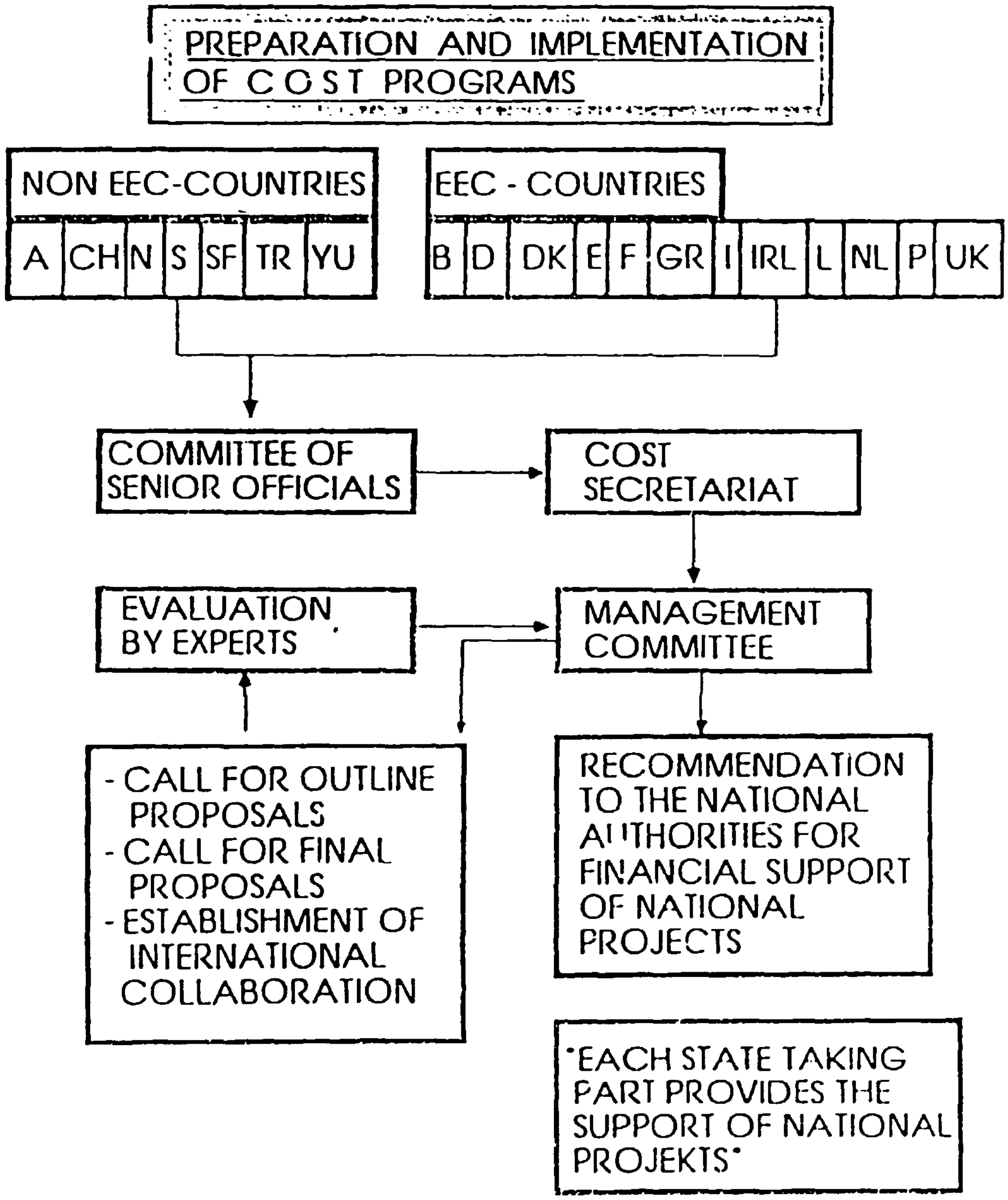

Fig.2: Preparation and implementation of COST-programs

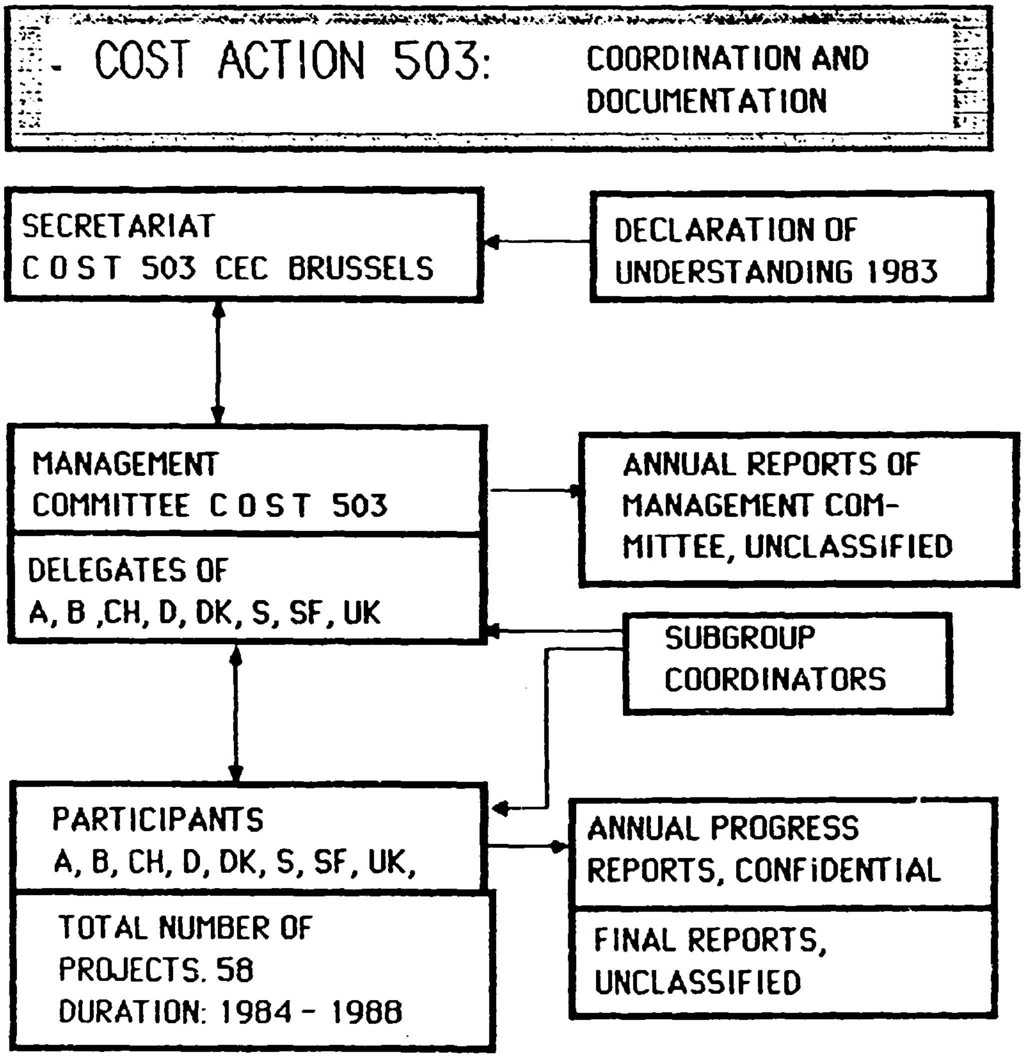

Fig. 3: Coordination and documentation
in COST - Action 503

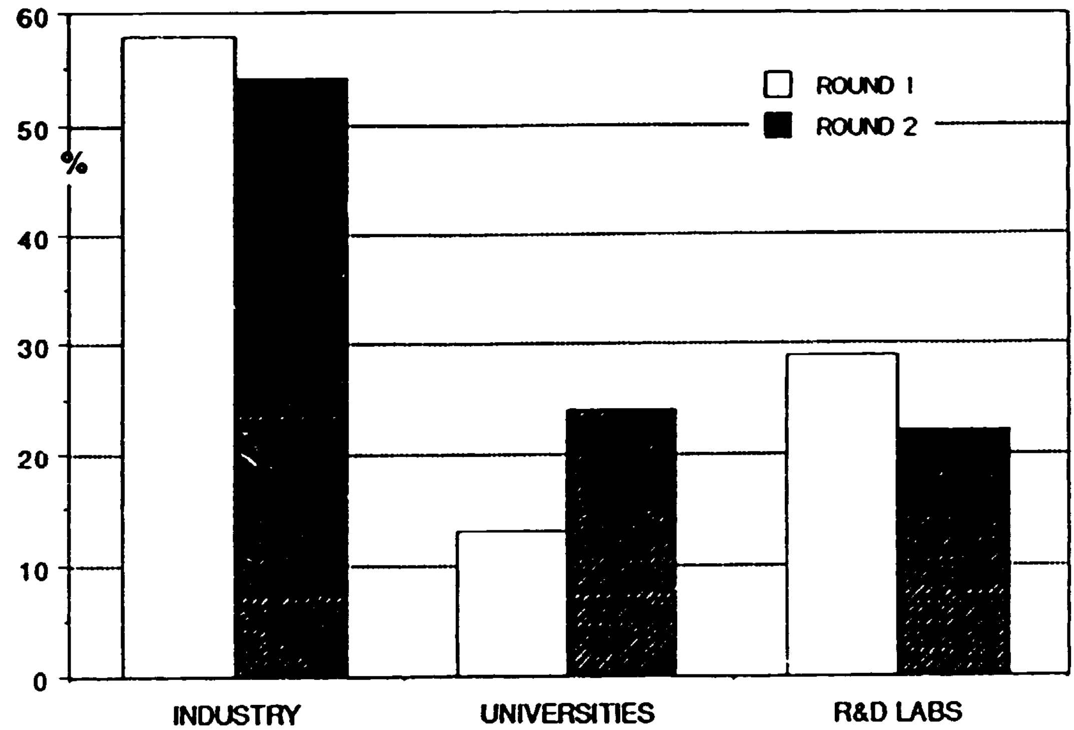

Fig.4: Institutions participating in COST-503

COUNTRY	ROUND	ROUND II
AUSTRIA	8	7
BELGIUM	2	2
DENMARK	2	1
FINLAND	2	3
FRANCE	-	3
GERMANY	20	10
LUXEMBOURG	-	2
SWEDEN	10	4
SWITZERLAND	4	2
UNITED KINGDOM	5	3
TOTAL:	53	37
STARTING DATE:	1985	1988
FINISHING DATE:	1988	1991

Table 1: COST-503 NUMBER OF PROJECTS

SUBGROUPS	COORDINATORS ROUND I	ROUND II
PM of light metals	G.Höllriegl (CH) P.Schwellinger (CH)	M.J.Couper (CH) R.Schäfer (D)
PM of hard materials	A.Mocellin (CH) H.Ortner (A)	K.Weiss (FL) H.Ortner (A)
PM of Fe-based materials	W.Paton (UK)	W.Paton (UK)

Table 2: Concerted action COST-503 Powder Metallurgy

Processing, manufacturing economics, materials and product performance, quality control, etc.

SUBGROUP 1:	LIGHT METALS pm-Al alloys: RSP, ODS, Al-Li pm-Ti alloys: blend and prealloyed
SUBGROUP 2:	Fe-BASED MATERIALS CIP and HIP processing of components, special property alloys fatigue resistant components, joining of dissimilar pm-components, heavy-duty components (gears, connecting-rods)
SUBGROUP 3:	HARD MATERIALS hard metals hard materials and ceramics high speed steels heavy metals

Table 3: COST-503 Powder Metallurgy I.Round

Optimization of processing, manufacturing; quality control; product characterization, NDT evaluation; testing, service performance; data bank, specifications

SUBGROUP 1:	LIGHT METALS high-temperature alloys from RS-powders processing, properties, evaluation precision forging, effects of sintering defects
SUBGROUP 2:	Fe-BASED MATERIALS components (roller bearings, connecting rods) near-net-shape processing dynamic/static properties effects of surface treatments
SUBGROUP 3:	HARD MATERIALS AND REFRACTORY METALS processing/microstructure/properties/performance effects of impurities T(C, N) and B-carbide based hard materials Zr-oxide ceramics ultrapure sputter targets high-purity refractory metal silicides

Table 4: COST-503 Powder Metallurgy, II.Round

12. METHODS FOR PREDICTING THE FATIGUE STRENGTH OF pm-MATERIALS

Brigitte Weiss and Roland Stickler
University of Vienna, Austria

Abstract

Experimental data on the high-cycle fatigue properties of defect-containing pm-materials showed that fatigue failure may occur after high numbers of loading cycles at stress amplitudes much below the fatigue limit deduced from conventional test results. It was the objective of present investigation to develop a procedure which permits a prediction of the high-cycle fatigue limit and of the influence of microdefects (pores, inclusions, precipitates) on the basis of geometrical and intrinsic material properties. As demonstrated with several examples, this task can be accomplished with the aid of experimentally determined modified Kitagawa-diagrams with the effective threshold stress intensity as the critical parameter and a quantitative metallographic characterization of the size and location of the largest defects.

Introduction

Powder metallurgical materials are known to contain various degrees of porosity, inclusions and microstructural defects which may affect their mechanical properties. The magnitude of the decrease of properties depends on amount, nature, size, shape and distribution of these inhomogeneities within the material.

At present there seems to exist no general model to explain on a quantitative basis the interrelationship between defects/porosity and the mechanical properties of pm-materials. While these materials usually exhibit outstanding tensile properties, the high-cycle fatigue strength may be affected drastically. More or less empirical models have been described in the literature correlating the detrimental effects of inclusions or voids on fatigue properties with the stress concentration around defects, differences in elastic properties and strength between inclusions and matrix, etc. The experimental verification of these models requires involved testing efforts for each material under consideration. It appears therefore of interest to find methods for the deduction of the high-cycle fatigue properties of defect-containing materials from basic principles, i.e. on the basis of intrinsic material constants and microstructural parameters.

Why predictive methods?

In earlier work (1) on the fatigue limit of high-strength pm-Al alloys fatigue failures were encountered in specimens after exposures to 10^8 to 10^9 loading cycles, at stress amplitudes considerably below the endurance limit found at the conventional run-out limit of $N = 10^7$. We may surmise that the endurance limit at more than 10^8 cycles approaches the true fatigue limit of the material (19). On the other hand, technical components in rotating machinery may well experience such numbers of loading cycles over their life time (2).

Fractography of the specimens failed after extensive cyclic loading invariably revealed that nucleation of the fatigue cracks occurred at internal sites, e.g. at inclusions, voids or microstructural segregation zones. The large scatter in experimental data for the pm-Al alloy IN 9052 shown in the S-N diagram in Fig.1 is caused mainly by the non-uniform

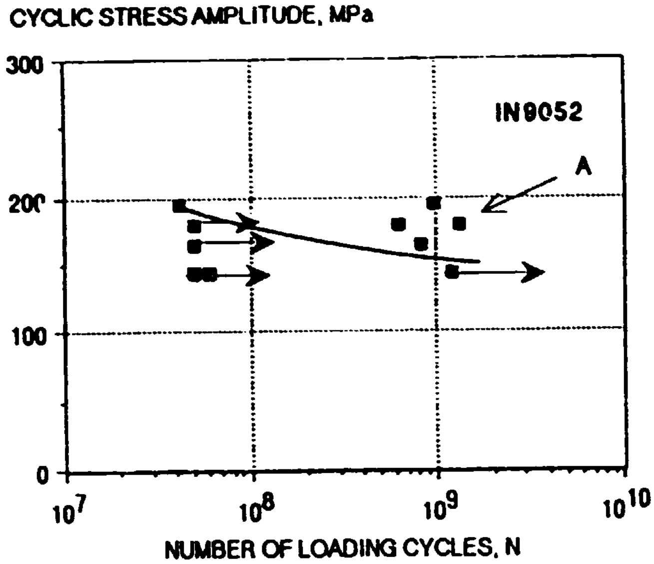

Fig.1: S-N diagram for the high-cycle region of the pm-Al alloy IN-9052 tested at R = -1

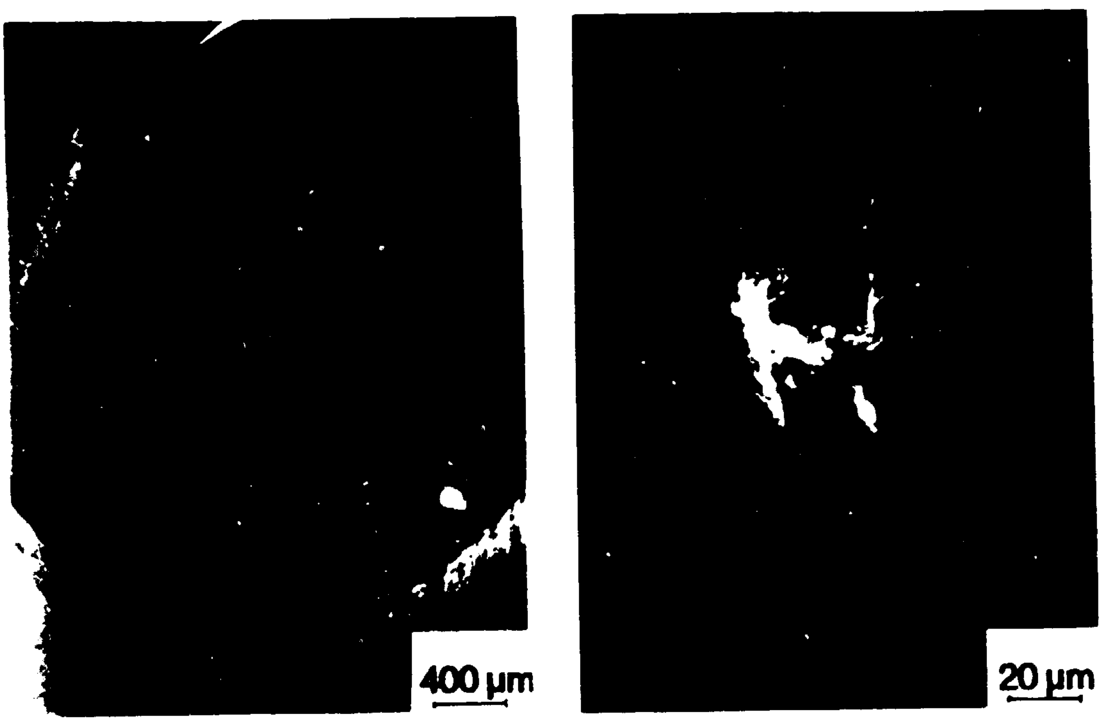

Fig.2: Scanning electron micrograph of the fracture surface of the IN-9052 specimen marked A in Fig. 1

distribution of internal defects. The characteristic appearance of such an internal nucleation site, the fatigue fracture zone and the overload failure is shown in Fig.2.

In view of the inherent risk involved with the unexpected failure of components made of high-strength alloys under high-cycle loading it seems mandatory to provide the designer with sufficient information on the reliability of such materials. At the present state of the art it appears that the occasional presence of inclusions, foreign particles and microstructural inhomogeneities cannot be avoided. A control of the maximum sizes of such defects and their distribution, however, may be feasible. Therefore, experimental procedures are required to predict the magnitude and criticalness of the detrimental effects on the high-cycle fatigue behavior as function of the size, distribution, location, geometry and nature of the defects.

Since such detrimental effects cannot be deduced from tensile data and are only in severe cases indicated in fatigue data within the conventional range of loading cycles, fatigue tests need to be extended to 10^9 cycles. This evaluation requires extreme long testing times and large numbers of specimens to permit the required statistical evaluation. Predictive methods which provide information on the degree of degradation due to the presence of defects in pm-components appear therefore highly desirable from a standpoint of both reliability and economics.

Proposed procedures for the prediction of the high-cycle fatigue behavior of defect-containing pm-materials

Amongst the first to suggest an explanation of the effects of defects on the fatigue behavior of pm Ni-base alloys by a fracture mechanical approach were Betz&Track (3). In their study the authors assumed that fatigue crack initiation occurs during the first few loading cycles and calculated the fatigue crack growth to overload failure by fracture mechanics relationships. The authors showed that the fatigue strength of pm-materials is related to the number, the size and the location of inclusions. Three cases of locations of defects were considered, i.e. internal, near sub-surface (with the fatigue microcrack intersecting the specimen already after short growth episodes) and tangential.

Depending on the position of this crack nucleating defect different relationships for calculating the stress intensity factors at the crack tip (edge of the circular crack) may be applied (3). The pertinent equations are inserted in Fig.3. Based on these equations and the knowledge of the position of detrimental defects the fatigue life of components may be calculated. It is obvious from above equations that a defect positioned tangentially (at the specimen surface) is the most critical of the three cases considered.

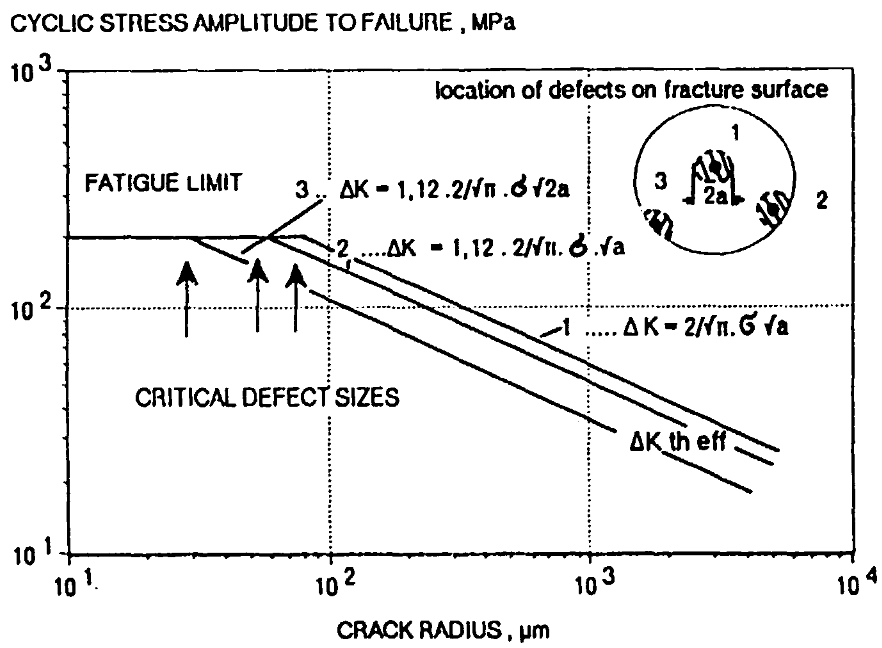

Fig.3: Kitagawa-diagram for the pm-Al alloy IN-905XL showing the effect of defect location (calculated according to Ref.3)

A dependence of the fatigue limit on the size of a fatigue crack was noticed already by Frost (4). Kitagawa&Takahashi (5) found that the similitude concept of fracture mechanics appears to break down for small crack sizes, as revealed in a diagram of cyclic stress amplitude vs. crack length ("Kitagawa-diagram"). In this diagram the amplitude for resumption of growth of long fatigue cracks can be revealed by a line corresponding to the threshold stress intensity for fatigue crack growth. For shorter fatigue cracks no explicit expression for the threshold was found, a cut-off value is given by the fatigue limit. Thus, a critical crack size can be deduced which, when exceeded, causes a reduction in fatigue strength. The significance of the various regions of the Kitagawa-diagram was discussed in detail by Miller (6).

Pineau (7) in studying the fatigue behavior of nodular cast iron found that shrinkage pores acted as potent crack nuclei. Critical defect sizes could be determined from a Kitagawa-diagram by using an effective value of the threshold stress intensity as the critical parameter. This effective threshold stress intensity is supposed to reflect an intrinsic materials property independent of testing parameters or experimental configurations. Fathulla et al (8) could demonstrate that fatigue cracks initiate at inclusions or precipitates in a number of commercial grade cast, wrought and pm-alloys. The typical short crack behavior could be correlated with the critical sizes revealed in modified Kitagawa diagrams based on the effective threshold values (assumed to correspond to the stress intensity value at a crack growth rate smaller than 10^{-11} m/cycle).

The effects of porosity on the fatigue crack initiation in pm-steels were studied by Holmes&Queeney (9). While for low porosities the reduction of the load bearing cross-section was found negligible, the effect of pores was explained by acting as crack precursors and as sites of local stress concentration in a plane normal to the stress axis (a stress concentration factor for pores was given as K_t = 2, with the region of stress concentration extending up to 5 times the pore radius). The initiation endurance was proposed to be a function of the quotient interpore-spacing/pore-size. Thus, one should be able to calculate the initiation endurance of a porous materials based on results from quantitative metallography. Unfortunately, the authors were not able to predict the fatigue behavior when the porosity approaches small values.

The effect of small artificial surface defects on the fatigue limit of high-strength steels was studied extensively by Murakami and coworkers (10, 11). In agreement with others these authors found that the effect of small surface notches cannot be described sufficiently by a stress-concentration factor. It could be shown that during initial cyclic loading a small fatigue crack is formed around an artificial surface defect (small drilled holes). Quantitative relationships for computing the values of the threshold stress intensity and the fatigue limit were proposed using the fatigue limit (deduced from hardness measurements) and the square-root of the area of the initial fatigue crack (projected on a plane normal to the stress axis) as parameters. In a similar fashion the "fatal" size of inclusions or internal pores were calculated, whereby size and location of the defects have to be taken into consideration. Murakami concludes (in agreement with the definition of the fatigue limit by Lukas,(12)) that the fatigue limit of unnotched specimens corresponds to the threshold stress for non-propagating fatigue cracks nucleated at holes, pores, inclusions etc. Thus the fatigue problem of defects and inclusions should be considered as a problem of the growth conditions for small superficial or internal cracks. It is unfortunate that Murakami did not correlate the results with the effective threshold values more appropriate to describe the growth conditions for small cracks.

Numerous papers have been published about the influence of macroscopic notches on the fatigue behavior. The fact that microscopic notches (cavities, holes, scratches) may affect the fatigue limit of materials has also been noted. However, only recently a qualitative explanation for the existence of non-damaging notches and a quantitative derivation of their critical sizes were presented by Lukas et al (13). This analysis requires the knowledge of only geometrical factors describing the notch (stress concentration factor) and intrinsic materials parameters (fatigue limit of the unnotched materials and the value of the threshold stress intensity). This proposed relation was shown to be valuable for the prediction of the limiting size of non-damaging notches in engineering application of metals and alloys. The analysis of notches was subsequently extended to a prediction of the size of non-damaging surface holes (hemispherical or shallow cylindrical) under fatigue loading (14). Results to date indicate that the critical hole geometry can be predicted also on the basis of material properties (fatigue limit of the unnotched material, effective threshold stress intensity) and parameters pertaining to the distribution of the stress concentration around the hole. These considerations may eventually be extended for a quantitative prediction of the effects of internal pores on the high cycle fatigue limit of porous pm-materials.

As a consequence of the review of all pertinent possibilities we considered the use of modified Kitagawa-diagrams as the most suitable and practical method for the prediction of fatigue limits of defect-containing pm-materials. The procedure for the experimental determination of such diagrams is outlined in the following.

Experimental technique to establish Kitagawa-type diagrams

To establish a Kitagawa-diagram of a particular material it is necessary to determine the high-cycle S-N data, in particular the fatigue limit for high numbers of loading cycles, e.g. $N = 10^9$, and the threshold stress intensity for fatigue crack growth, ΔK_{th}. It should be pointed out that this ΔK_{th} is not a material constant and depends on loading conditions, crack closure, etc (15). As an intrinsic material parameter the effective threshold stress intensity, $\Delta K_{th,eff}$, must be applied. In fact this parameter was found independent over a wide range of loading conditions (mean load) for a number of technical alloys (16) and should represent the actual stress condition at the crack tip. Only this value governs the crack propagation behavior. To obtain the $\Delta K_{th,eff}$-value, the magnitude of the crack opening stress intensity is determined, and $\Delta K_{th,eff}$ computed (16).

The Kitagawa diagram (log σ_c vs.log a; σ_c .. cyclic stress amplitude, a .. crack length or crack depth) is then established by drawing the fatigue limit as a horizontal line, and a slanting line according to

$$\sigma_c \text{ proportional to } \Delta K_{th,eff}/a^{1/2},$$

in addition a factor related to the geometrical conditions has to be applied (17).
Both lines intersect at a point assumed to resemble the critical size below which defects will not affect the fatigue limit. Since the ΔK_{theff} value is independent of mean stress, the same diagram is representative for a wide range of loading conditions if for the fatigue limit (depending on mean stress) the appropriate maximum stress is plotted (18).

Experimental approach

Specimen materials:

To demonstrate the applicability of the predictive methodology the experimental results of various powder-metallurgical Fe-, Al- and Ti-based materials are presented in the following. The composition and properties of these materials are summarized in Table 1.

Test methods:

For the S-N measurements dumbbell shaped specimens with a cylindrical gauge section of 4mm diam. and 15mm length were used. Constant amplitude fatigue tests were carried out in a high-frequency resonance system, operated at 20 kHz to achieve the desired numbers of loading cycles of up to 10^9 within reasonable test times. All fatigue tests were carried out at room temperature at a stress ratio of R = -1. A detailed description of this test procedure is given in Ref.19.

Table 1: PM-MATERIALS REFERRED TO IN THIS PAPER

Alloy:	Composition, condition and heat treatment, properties
pm-Fe *)	pure Fe-powder (Höganäs ASC 100.29) compacted and sintered or hipped, R_m: 140-320, density: 6.8-7.8, Porosity 0-12,
DISTALOY-AB **)	1.5Cu-1.75Ni-0.5Mo-0.01C-rem.Fe compacted and sintered or hipped, porosity 0-9, R_m: 700-900
Al IN-905XL ***)	4Mg-1.5Li-1.1C-0.8O_2-rem.Al mechanically alloyed and extruded, R_m: 565, density: 2.57
Al IN-9021 ***)	4Cu-1.5Mg-1.1C-0.8O_2-rem.Al mechanically alloyed and extruded, R_m: 625, density 2.78
Al IN-9052 ***)	4Mg-1.1C-0.8O_2-rem.Al mechanically alloyed and extruded, R_m: 515 density 2.66
TiAl6V4 +)	prealloyed powder (6Al-4.1V-0.01O_2-rem.Ti), compacted and heat treated or hipped, R_m: 880 density 4.43

(R_m in MPa, density in g/cm^3, porosity in %)

Suppliers: *) Prof.G.Jangg, TU-Vienna
**) HIP-Ltd. Chesterfield UK
***) INCO Engineered Products Ltd., Birmingham, UK
+) Metallwerk Plansee GmbH, Reutte, A

For the determination of the fatigue crack growth behavior near threshold a servohydraulic system operated between 10 Hz and 50 Hz at room temperature was available. For the experiments center-surface-notched specimens (20) with cross sections of 5mm x 10mm or 6mm x 15mm were used. The tests were carried out in accordance with ASTM-recommendations (21). The stress intensity values for such cracks were computed as given in Ref. (22). Details of the test procedure to determine the crack opening stress intensity by a strain gauge method are described in Ref. (16).

After completion of the crack growth measurements the specimens were ruptured in tension and the shape and the extent of the fatigue crack determined by SEM-microfractography. This information is needed for the computation of the stress-intensity values. Particular attention was given to the identification of the region of crack nucleation, and the nature, size and magnitude of the crack-initiating defect. This information is compared with the value of the critical defect size deduced from the Kitagawa-diagram.

Examples of the application of Kitagawa-diagrams to predict the high-cycle fatigue properties of pm-materials

Effect of the location of defects:

In the diagram shown in Fig.3 for the alloy IN-905XL the effect of the location of the initiating defect is revealed. Assuming that there is no drastic change in the fatigue limit, the reasoning of Betz&Track (3) may be applied for the calculation of ΔK_{th} for defects in various locations of the specimens (see section 3). The defect may be considered as an inclusion surrounded by a short circular crack formed early during the fatigue loading of the specimen.

As shown in Fig.3 the maximum size of a critical crack not affecting the fatigue limit is 30 µm depths for a semi-circular tangential crack while for an internal penny-shaped crack the critical radius is about 80 µm, applying experimental values of the fatigue limit at $N = 1.10^9$ and a value of $\Delta K_{th,eff} = 2\ MPa.m^{1/2}$.

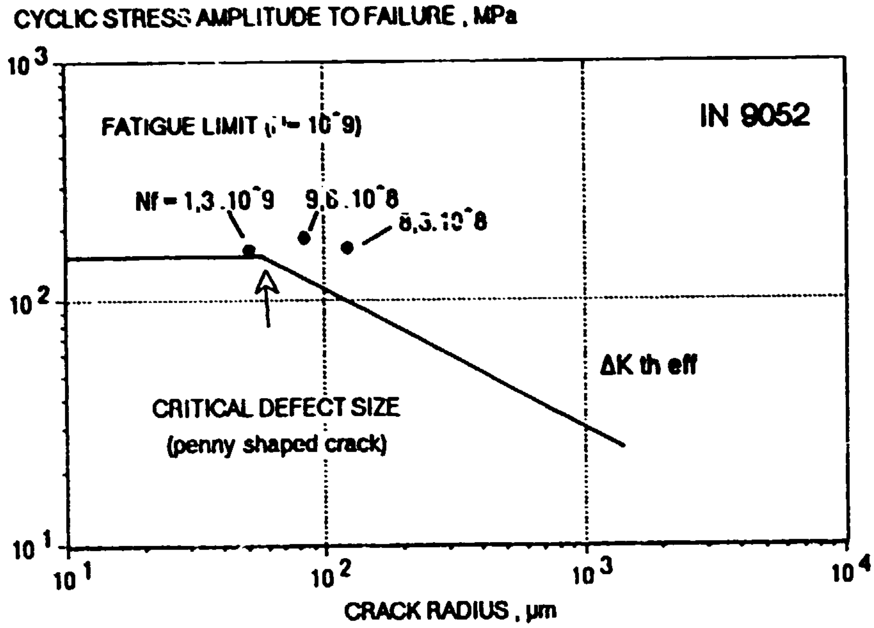

Fig.4a: Kitagawa-diagram for the pm-Al alloy IN-9052, points indicate the size of inclusions initiating a fatigue crack, the corresponding fatigue life is listed

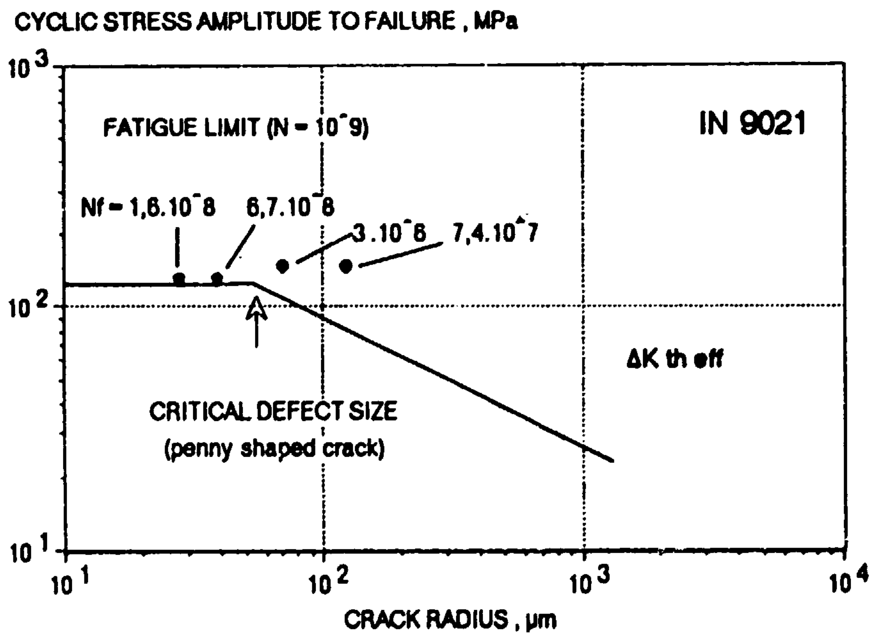

Fig.4b: Kitagawa-diagram for the pm-Al alloy IN-9021, points indicate the size of inclusions initiating a fatigue crack, the corresponding fatigue life is listed

Effect of inclusion size:

Results of two pm-Al alloys with similar values of $\Delta K_{th,eff}$ (26) are shown in Figs.4a (IN-9021) and 4b (IN-9052). The diagrams are drawn for the fatigue limits at $N = 1.10^9$ and the $\Delta K_{th,eff}$ values for a penny-shaped internal crack. From these diagrams critical defect sizes of about 50 µm can be predicted for both alloys. This is in reasonable agreement with fractographic observations. The defect with a size of about 60 µm radius in IN-9021, shown in Fig.5, resulted in a fatigue failure after 3.10^6 loading cycles.

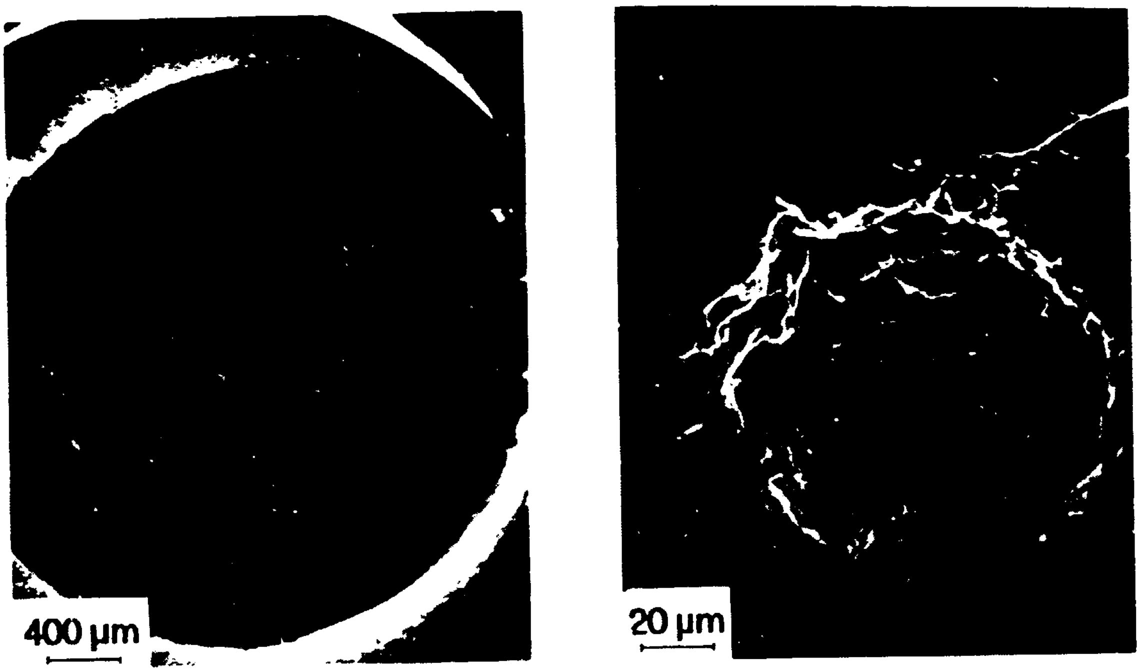

Fig.5: Scanning electron micrograph of the fracture surface of a specimen of IN-9021, note the internal crack nucleation at an inclusion

The fatigue limits shown in the diagrams of Figs.4a and 4b were determined from a large number of specimens. Fractography of specimens failed ·ly or after large numbers of loading cycles just sligh above the fatigue limit revealed in many cases the nce of inclusions giving rise to nucleation and growth c internal fatigue cracks. The sizes of these inclusions and the cyclic amplitudes of loading to failure are indicated for the individual specimens in the diagrams. It can be seen that the particle size in some of the specimens was smaller than the critical size, thus it may be surmised that these specimens would not have failed if tested at the average fatigue limit. However, in several specimens the particle size was larger than the critical size which would have resulted in fatigue failure even if the specimens were tested at stress amplitudes considerably lower than the fatigue limit.

Corroborating results were obtained by evaluating published fatigue life data for pm-TiAl6V4 materials manufactured from prealloyed powder mixed with Al_2O_3 particles of various particle sizes to simulate foreign inclusions (23). The fatigue data showed a decrease of the endurance limit (for $N = 10^6$) with increasing particle size. The loss of fatigue life related to the increasing particle sizes appears larger in the high-cycle than in the low cycle regime. The diagram in Fig.6 is calculated for the case of an internal penny-

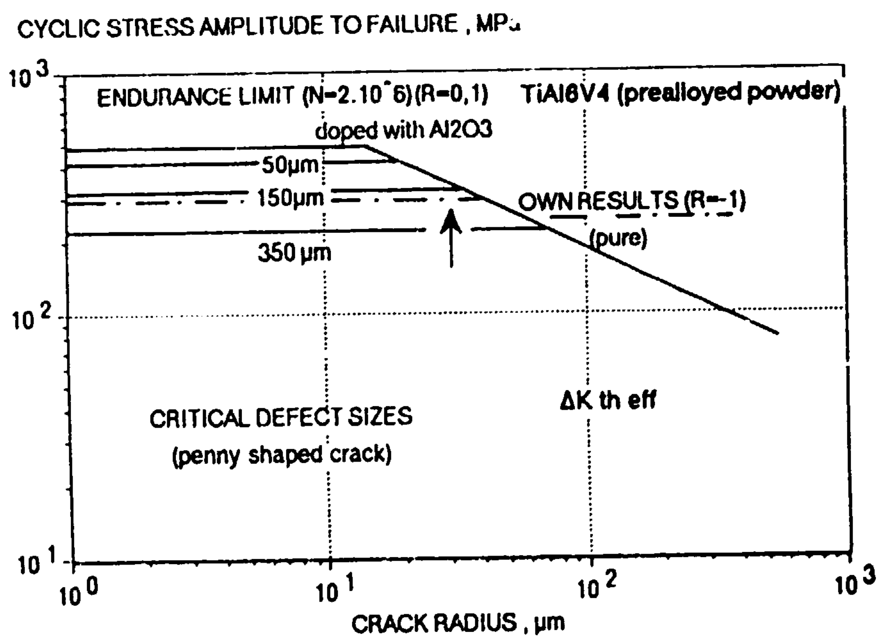

Fig.6: Kitagawa-diagram for doped (23) and pure pm-TiAl6V4 (24)

shaped crack. The value of $K_{th,eff}$ was determined for a similar technical pm-TiAl6V4 alloy (24) and may deviate slightly from that of the contaminated material. It should be pointed out that literature data indicate that the presence of fine particles should not affect measurably the fatigue crack propagation behavior. The fatigue limit (determined at $R = -1$ for $N = 2.10^8$) of the technical material is marked by an arrow in Fig.6. The intersection of the $\Delta K_{th,eff}$-line would predict an internal defect size of less than 40 µm radius. A comparison of the defect sizes deduced from the diagram in Fig.6 with the fractographically observed defect in a failed specimen of the pure pm-TiAl6V4 (Fig.7) shows reasonable agreement considering the possible variations in endurance limits, $\Delta K_{th,eff}$ and the varying locations of defects.

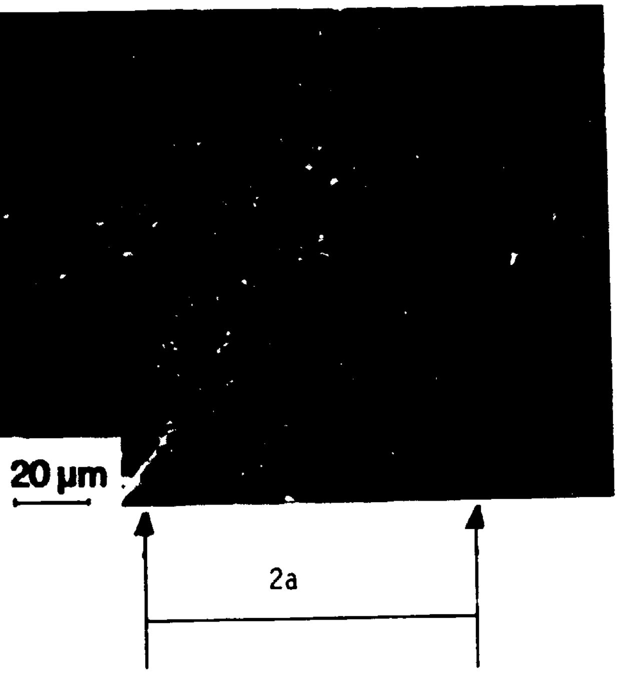

Fig.7: Scanning electron micrograph of a specimen of "pure" pm-TiAl6V4, note the crack initiating inclusion surrounded by a circular short crack of diameter 2a

Effect of porosity:

The effect of porosity is demonstrated in Fig.8 for pm-Fe (25) and the pm-steel Distaloy-SA in Fig.9 (24). For both

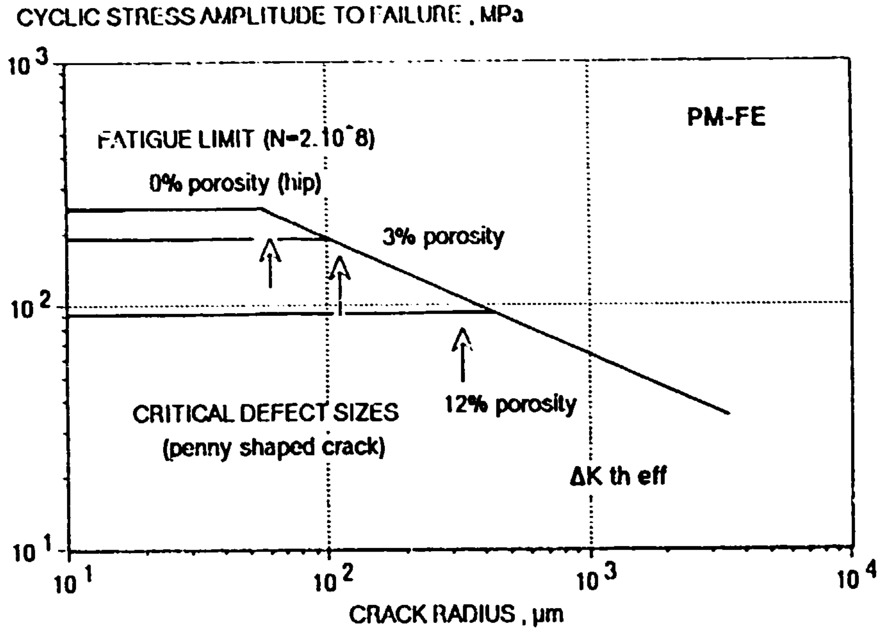

Fig.8: Kitagawa-diagram for pure pm-Fe containing various amounts of porosity

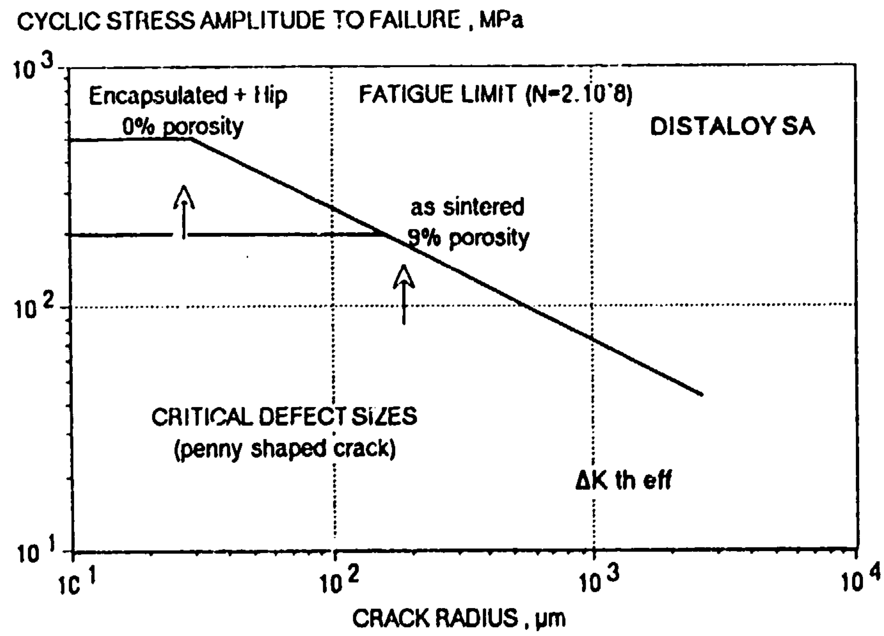

Fig.9: Kitagawa-diagram for pm-DISTALOY-SA in sintered and hipped conditions

materials the fatigue limit (for $N = 2.10^8$) decreased with increasing porosity. The results with porous specimen materials clearly indicate that pore distribution and geometry have a strong effect on the fatigue behavior. As long as pores act as isolated defects crack nucleation occurs preferentially in the interior of the specimen. For the pm-Fe material this condition is prevalent up to a total porosity of approx. 4% . The critical defect size deduced from the corresponding Kitagawa diagram coincides with the experimentally observed size of the largest pores. With the porosity increasing further a pronounced effect of the open (interconnected) porosity was found. The critical defect sizes predicted from the Kitagawa diagram are much larger than the observed pore sizes. A better correlation with the predicted sizes is found if the size of pore aggregates (clusters) is taken into account (25).

Experimental data for as-sintered porous DISTALOY-SA specimens (up to 8% porosity) showed that increases in porosity lead to a reduction of the fatigue limit. The evaluation of the data for specimens of hipped and practically p -free material indicated a critical defect size of 20 µm. This is in agreement with the observed size of inclusions initiating cracks during extended fatigue loading.

Summary and conclusions

In this paper an attempt was made to emphasize the advantages of exploiting the information included in a modified Kitagwa-diagram for the prediction of the fatigue limit of pm-materials containing defects (sintering voids, foreign particles, precipitates):

(i) During fatigue loading near the (high-cycle) fatigue limit the microstructural defects give rise to the formation of surrounding microcracks. The observations support the statement of Murakami (11) that the fatigue problem associated with materials containing pores or inclusions is in fact a problem of defining the fatigue limit of materials containing short cracks of various location and sizes. So far our tests failed to reveal the presence of non-propagating circular microcracks around inclusions or voids in run-out specimens, obviously related to experimental difficulties. However, the existence of such non-propagating cracks was clearly demonstrated in run-out specimens of technically pure Cu containing artificial small holes at the specimen surface. It should be pointed out that the influence of microscopic defects on the fatigue properties is revealed with high sensitivity only by cyclically loading of a specimen near the fatigue limit. Due to the random distribution of such microstructural defects an experimental verification of the predictions would require testing of a statistically significant number of specimens.

(ii) The critical defect size can be deduced from the proposed modified Kitagawa-diagram with $\Delta K_{th,eff}$ as parameter. Since the critical defect is composed of the particle and the surrounding near-circular microcrack, the critical defect size is only to a minor degree dependent on the shape of the particle assumed to be globular. The shape of the particle, however, certainly influences the initiation life. The experimentally determined Kitagawa diagrams reveal that there exist critical defect sizes below which the fatigue limit of defect-containing materials is not affected. The knowledge of such non-critical defects should be of significance for the design of improved pm-materials. On the other hand, the diagrams permit the prediction of endurance limits of materials containing larger defects. An important advantage of the presentation in form of Kitagawa-diagrams is the fact that the same diagram applies for a wide range of mean stresses as long as for the fatigue limit the value of the maximum stress is plotted. The $\Delta K_{th,eff}$ values are practically not affected by mean stress.

(iii) The position of the defect has a strong influence on the fatigue limit as can be deduced from the calculations of the stress intensity factors. Most detrimental are inclusions or pores at tangential and near sub-surface positions. Internal defects of comparable size are least detrimental and show the longest propagation life, in agreement with the findings of Betz&Track (3).

(iv) The proposed methodology requires for each material to establish a odified Kitagawa-diagram based on its effective threshold value, supported by results of a quantitative metallographic analysis (extreme value statistics for estimating the maximum expected inclusion or pore sizes). From the fatigue limit determined for specimens of a particular material the maximum size of the defects can be deduced. The upper bound of the fatigue limit would apply to a defect-free material, in which the magnitude of microstructural paramaters, such as grain size, dislocation or slip-line arrangements, are the dominating features limiting the fatigue strength.

(v) The determination of these material parameters involves considerably less efforts than the determination of high-cycle S-N curves and fatigue limits for each material in each microstructural condition. The proposed method appears of advantage in characterizing the effects of changes in production schedules, compositional variations, optimization treatments, etc. on the high cycle fatigue limit.

Acknowledgements

The authors thank Mrs. H.Sychra for performing most of the fatigue tests and SEM-fractography, Dr.H.Kemper for the threshold and closure measurements, and Dr.W.Hessler for the determination of the dynamic Young´s moduli. The work was supported in part by grants of the Fonds zur Förderung der Wissenschaftlichen Forschung, Vienna, by the Metallwerk Plansee GmbH, Reutte, and the Bundesministerium for Wissenschaft und Forschung, Vienna (support for participation in the European Concerted Action COST-503).

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13. PUBLICATIONS

Major PM publications in 1990

Following PM'90 (London, 2-6 July) The Institute of Metals will be launching an integrated series of texts to meet the needs of undergraduates and practising engineers.

An Introduction to Powder Metallurgy

This volume, designed for students, covers the basic scientific and technological concepts with a number of practical examples for illustration. Sections on fabrication, powder characterization, consolidation, property development, and specific materials (e.g., ceramics, ferrous and non-ferrous components) are included. An emphasis is placed on the advantages and limitations of applying these principles in practice. (Approximately 150 pages.)

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Selected Case Studies in Powder Metallurgy

Intended for students and materials engineers working in industry and elsewhere. Volume 2 contains a series of short case studies of component design and manufacture covering many traditional and non-traditional applications of powder metallurgy. These range from automotive and aerospace parts to magnetic and electronic uses. Each case study is written to a specific set of objectives covering: a description of the part/specifications, reasons for choosing PM, where basic principles are relevant to finding a technological solution, design criteria. These are followed by a brief description of the actual processing and finishing route. (Approximately 150 pages.)

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Powder Metallurgy - an Overview

A series of review articles summarizing the current status of powder metallurgy technology for practising PM engineers. This volume includes powder production, characterizations, sintering theory and practice, rapid solidification technology, injection moulding, alloy development, quality assurance, and selection procedures. (Approximately 400 pages.)

The series was initiated through Dr. Ivor Jenkins, the late Professor Malcolm Waldron, and Professor John Wood. Contributions have been commissioned after consultation with the international PM community and with the aim of responding to the widely-expressed need for an integrated package grounded in technological reality.

The Institute of Metals is offering a competitive pre-publication price for the series. For details please contact: Sales and Marketing Department (004), The Institute of Metals, 1 Carlton House Terrace, London SW1Y 5DB. Tel.: 01-839 4071. Telex: 8814813. Fax: 01-839 2289.

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Powder Metallurgy and Related High Temperature Materials

Editor: P. Ramakrishnan, Dept. of Metallurgical Eng., Indian Institute of Technology, Bombay, 1985, 520 pp. Oxford & IBH Publishing Co.

International Symposium on Advanced Structural Materials (1988: Montreal, Quebec)

Proceedings

Edited by D.S. Wilkinson. NY: Pergamon, 1989. 318p, $56.25 (Proceedings of the Metallurgical Society of the Canadian Institute of Mining and Metallurgy; Vol. 9) 620. 1'1 TA401.3 88-38801 ISBN 0-08-036090-4.

Contents: Metal matrix composites. Structural ceramics. Interfaces. Polymeric composite material - advanced processing methods and applications. Powder metallurgical materials.

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Powder Metallurgy Design Manual covers design, shapes and forms, compositions, properties, manufacturing (pressing, sintering, heat treating, surface finishing, machining and joining) of powder-metal parts, 110 pp, $75, Metal Powder Industries Federation (Princeton, NJ, USA).

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"P/M Design Guidebook" devotes 40 pages to topics such as the benefits of powder metallurgy, process steps, secondary operations, and material selection. Other chapters cover engineering properties, designing powder-metal parts, applications, terminology, and how to specify. Many useful drawings and data tables accompany the discussion. Photographs show a variety of powder-metal parts formed in various processes. Metal Powder Industries Federation, 105 College Rd. E, Princeton, NJ 08540, USA.

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PM sintering

Atmosphere requirements for sintering of powder metallurgy (PM) components are outlined in literature from Liquid Air Corp., Walnut Creek, Calif. Also discussed are atmosphere setup and selection, control instrumentation, and selection of alloys for furnace belts.

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PM parts

Eight-page brochure details powder metallurgy (PM) manufacturing capabilities of Ferralloy, Troy, Mich., USA. The company manufactures PM components for automotive, appliance, power transmission, farm equipment and hydraulic applications. In-house computer-aided design facilities and statistical quality control (SQC) techniques also are described.

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Advanced Technical Ceramics, edited by Shigeyuki Somiya, is an updated English translation of the 1984 Japanese edition. The term technical ceramics as defined by Dr. Somiya refers to "ceramics that exhibit a high degree of industrial efficiency through their carefully designed microstruture and superb dimensional precision ... Rigorously controlled conditions of shaping and firing." The 23 contributors are largely from Japanese industrial laboratories, with several, including the editor, from technical institutes.

The book is divided into three major sections. The introduction includes chapters on definitions and types of ceramics: synthetic raw materials; production processes; and methods of evaluating mechanical and thermal properties. The section on properties and applications covers electrical, electronic, magnetic, thermal, chemical, optical and mechanical properties in separate chapters, plus a chapter on biological applications. The final section covers precision machining methods. An appendix that presents a chronology of the development of advanced electronic and engineering ceramics from 1893 to 1982 is a novel and informative addition. (Academic Press Inc., 465 South Lincoln Drive, Troy, MO 63379. Phone: (800) 321-5068)

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Inherently Conducting Polymers: Processing, Fabrication, Applications, Limitations, by M. Aldissi of Los Alamos National Laboratory, was originally written in 1987 as a report for the Department of Energy. As the author points out, the many potential applications for electrically conducting polymers have made them the subject of much research. Unfortunately, the commercial applicability of these potentially useful materials has been hampered by their instability and intractability due to their conjugated backbone, which is responsible for their conduction. The book describes the progress that has been made in solving these problems. It discusses several methods of synthesizing conjugated polymers and ways of doping the backbone to render them more highly conductive. Various stabilization techniques are covered including chemical doping, ion implantation, plastification, copolymerization, anti-oxidative treatments, surface protection, and the use of crown ethers. The author notes that while considerable progress has been made, conducting polymers still need further investigation for improving their processability, high performance in various applications, and long-term stability. Possible uses of conducting polymers are described along with the conditions for successful applications. The relative advantages and disadvantages of currently available polymers are also discussed. (Noyes Publications, Mill Road at Grand Ave., Park Ridge, NJ 07656. Phone: (201) 391-8484. Fax (201) 391-6833)

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Composite Applications: The Future is Now, edited by Thomas J. Drozda, is a publication of the Society of Manufacturing Engineers (SME) Manufacturing Update Series. It is a compilation of 31 papers presented at various SME conferences between 1985 and 1988. The purpose of this series is to provide up-to-date information on various topics relating to manufacturing for engineers working in the field and as a reference source. The papers are divided into six chapters: ceramic matrix composites; metal matrix composites; resin matrix composites; tooling; testing and inspection; and applications. Among the presentations included are "Structure and Properties of Hybrid SiC/LAS III Glass Ceramic Composites"; "Metal Matrix Composite Materials for Manufacturing"; "Advanced Thermoplastic Preforms"; "New Materials for Composite Tooling"; "Pultrusion - Flexibility for Current and Future Automotive Applications"; "Damage-Assessment Nondestructive Inspection Methods"; "Ultrasonic NDE Potential in Composite Manufacturing"; "Design of Composite Automotive Parts: A General Discussion"; "State-of-the-Art Materials for Orthopedic Prosthetic Devices". (Publication Sales, the Society of Manufacturing Engineers, One SME Drive, P.O. Box 930, Dearborn, MI 48121-0930. Phone: (313) 271-1500, Ext. 418 or 419. Fax (313) 271-2861)

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Basic Research in Superconductor, Ceramic and Semiconductor Sciences at Selected Japanese Laboratories, by Robert J. Gottschall, is a report based on visits in 1988 to 14 major industrial, governmental and university R&D organizations and laboratories. The subjects covered in this most interesting and informative 194-page report are: superconductivity; diamonds; cubic boron nitrides; synchrotron radiation, accelerators, and applications; beam technology and lithography; semiconductor sciences; ceramic process science; high pressure and bonding ceramic process science; mechanical behaviour and characterization of ceramics; ceramic design and engine applications; ceramic surface modification and behaviour; ceramic superplasticity; ceramic matrix composites; carbon; ordered alloys; metal matrix composites; and advanced instruments and facilities. This is highly recommended for obtaining an overall picture of the thrust and scope of materials R&D in Japan. While the supplies last the report is available gratis. (Dr. Robert J. Gottschall, Division of Materials Sciences, Office of Basic Energy Sciences, Mail Stop G-256 GTN, Washington, DC 20545. Phone: (301) 353-3428. Also available for purchase: The National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161 as PB89-174264/WFT. Phone: (800) 336-4700)

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Intelligent Processing of Materials is the Report of an Industrial Workshop Conducted by the National Institute of Standards and Technology (NIST) in 1988. The 50-page report includes a description of the concept of intelligent processing and reviews its advantages; the reports of three working groups - polymer processing, thermomechanical processing and ceramics processing; and a summary of two workshops on the hot isostatic pressing of metal alloys. Benefits from the intelligent processing of materials noted in the report are a marked improvement in the overall quality of the product and a reduction in rejected products; a lowering of the cost of post-manufacturing inspection and rejection; the flexibility to change manufacturing processes or material types quickly and economically; and a shortening of the long lead time needed to bring new materials from the development stage to mass production. The report is available gratis. (Dr. H. Thomas Yolken, Chief, Office of Nondestructive Evaluation, NIST, Gaithersburg, MD 20899. Phone: (301) 975-5727)

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Surface Modification Engineering, Volume I, Fundamental Aspects; Volume II, Technological Aspects, editor Ram Kossowsky. The intended audience for these volumes is the general practitioner of materials and engineering sciences. The first volume begins with a discussion of the properties of surfaces and how surfaces are characterized. This is followed by three chapters on environmental effects on surfaces including aqueous corrosion, the biological interfacial phenomenon, and friction and wear. The last four chapters discuss the fundamentals of methods developed to protect surfaces from their environments: chemical vapour deposition, physical vapour deposition, ion implantation and ion beam mixing, and modification of polymer surfaces. The second volume covers broad applications of surface modification engineering: surface engineering of materials for biological and medical applications, protective coatings for high temperature technology, applications of surface modification techniques to electron device technology, plasma (ion) processes for case hardening of metals, technological applications of surfaces modified by ion beams, surface modification with laser beams, laser cladding, and alloying for surface modification. (CRC Press, Inc., 2000 Corporate Blvd., Boca Raton, FL 33431. Phone: 1-800-272-7737 or (407) 994-0563)

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Practical Handbook of Materials Science, edited by Charles T. Lynch, includes an enormous amount of useful information in a little over 600 pages. Much of the information is similar to that in the original four-volume CRC Handbook of Materials Science, from which it is drawn. However, wherever necessary, particularly in the newer area of composites, the information has been updated. The conveniently sized book is divided into 13 sections: the elements; elemental properties; physical properties of compounds; conversion tables; properties of miscellaneous materials, e.g., building materials, concrete, foundations, rocks, soil, wood, cryogenic, flame retardant, paints, coatings, textiles, adsorbents and acoustic materials; ceramics and glasses; composites (ceramic, metal and polymer matrix) and reinforcements; electronic materials; graphitic materials; metallic materials; nuclear materials; polymeric materials; and materials information. This last section covers major compilations of information on materials plus comprehensive lists of federal materials information centres and federal materials research centres, with brief descriptions of each. The exhaustive table of conversion factors alone should prove invaluable to students and others working in materials science and engineering. (CRC Press, Inc., 2000 Corporate Blvd., Boca Raton, FL 33431. Phone: 1-800-272-7737 or (407) 994-0563)

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Energy Pulse and Particle Beam Modification of Materials, edited by Klaus Hennig, is the published proceedings of an international conference held in September 1987 in Dresden, DDR. The 157 conference papers (in English) were divided among the following topics: implantation into silicon; implantation and annealing of compound semiconductors; implantation into metals; transient heat treatment of semiconductors; formation of silicides; ion beam-assisted deposition and ion beam mixing; deposition, modification and structurization; silicon on insulator (SOI); diagnostics and focused ion beams. While about 15 countries were represented at the conference, the majority of the papers were from East and West Germany and the USSR with several from Austria, Hungary, Poland, Czechoslovakia and even Romania. Among the papers categorized as miscellaneous is one of particular interest by H. Gleiter and co-workers on the structure and properties of nanometer-sized solids. (Akademie-Verlag Berlin, Leipziger Strasse 3-4, DDR-1086 Berlin, DDR)

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Shock Waves for Industrial Applications, edited by Lawrence E. Murr. As its name implies, this book is aimed at the industrialist, the managers of technology, the manufacturers and anyone involved in manufacturing processes where shock wave fabrication could offer a new and novel approach. It is also intended for use by students and others as a reference in this field. The topics covered are: shock wave fundamentals; effects on the structure and behaviour of engineering materials; shock hardening and strengthening; explosive forming and material working applications; process parameters of explosive forming and applications in the automotive industry; explosion welding; parameters, structures, properties and applications of bimetals; explosive welding and bonding of multilaminates; principles and applications of shock wave compaction and consolidation of powdered materials; explosive shock wave consolidation of metal and ceramic powders; and the fabrication of novel, bulk superconductor composites by simultaneous explosive consolidation and bonding. The authors of this last section, who include its editor, have done pioneering work in the explosive compaction of the new ceramic superconductors. An attractive feature of this book is its many excellent illustrations, both diagrams and microstructures. (Noyes Publications, Mill Road at Grand Ave., Park Ridge, NJ 07656. Phone: (201) 391-8484. Fax (201) 391-6833)

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Advanced Materials in the Manufacturing Revolution is the proceedings of a conference held in June 1988 at Argonne National Laboratory (ANL). The topic and speakers were chosen, according to Alan Schreisheim, Director of ANL, to help manufacturing industry leaders gain a broader awareness of the advances in materials and processes that are currently under way. Two areas were emphasized in the presentations: management issues and initiatives involved in adapting technological advances in materials and manufacturing methods, and exploiting these advances to gain a competitive advantage; and specific trends in the development and commercial availability of advanced metallics, ceramics, polymerics and their composites. The presentations included: The Process for Processing: How to Commercialize It All; Developing a New Product: Disconnects that Exist Between Design, Materials Selection, Materials Performance and Manufacturing; Exploiting New Materials Technology for Competitive Advantage; An Assessment of New Engineered Metallic Materials; Developing Trends and Characteristics of High Performance Polymers and Composites: Manufacture, Supply and Use; The Materials Effect in the Manufacturing Revolution: Emphasis on Advanced Ceramics; Manufacturing Flexibility for Competitive Advantage: The Strategic Imperative for CIM. (ANL-89/3, CONF-880603; U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161. Phone: (703) 487-4630)

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Materials Research Society's (MRS) new publications: (MRS: 9800 McKnight Road, Pittsburgh, PA 15237, USA)

Materials Processing in the Reduced Gravity Environment of Space

Volume 87 (1986 MRS Fall Meeting, Boston, MA)
Editors: R.H. Doremus, P.C. Nordine

Describes recent work in processing and measuring materials properties in microgravity environment, including flight experiments which provide new and unexpected results in electrophoretic processing, crystal growth and fluid behaviour, as well as ground-based experiments and plans for future research. Topics: space processing viewed by a scientist-astronaut; theoretical studies of gravitational effects in chemical vapour deposition; isothermal dendritic growth; solidification of undercooled Ni-Sn eutectic alloy under microgravity conditions in the space shuttle; thermogravimetric measurements in an electrodynamic balance; microgravity materials processing for commercial applications; floating-zone processing of indium in earth orbit; ultrafine particle and fibre production in microgravity; containerless polymeric microsphere production for biomedical applications; glass formation in microgravity; noncontact true temperature measurement; and effects of an applied magnetic field on directional solidification of off-eutectic Bi-Mn alloys. 1987, hardcover. 38 papers, 366 pages. ISBN: 0-931837-52-9.

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Nondestructive Monitoring of Materials Properties

Volume 142 (1988 MRS Fall Meeting, Boston, MA)
Editors: J. Holbrook, J. Bussiere

This volume has a dual focus: (1) using nondestructive inspection of materials for process control during manufacturing to increase quality, and (2) nondestructive monitoring of the aging and degradation of materials in service to obtain estimates of remaining useful life. Two conclusions emerge from this research. Based on the apparent breadth and intensity of international activity in this area, there is a strong industrial and government need for further development of NDE monitoring technology. On the other hand, because almost all the development is still in the laboratory stages, the community's job of technology development has just begun. Also, continued progress in NDE technology will require a full interaction between materials scientists with the knowledge of how properties depend on microstructure, and NDE physicists who understand how changes in NDE signals provide information on differences in microstructure. 1989, hardcover, 45 papers, 374 pages. ISBN: 1-55899-015-1.

New Materials Approaches to Tribology: Theory and Applications

Volume 140 (1988 MRS Fall Meeting, Boston, MA)
Editors: L.E. Pope, L. Fehrenbacher, W.O. Winer

As this volume shows, atomic and molecular-scale tribobehaviour calculations are being made which explain interfacial interactions between materials undergoing relative motion. These calculations accurately predict the location of shear planes and thereby provide guidance to experimental programmes aimed at modelling friction and wear processes. Also discussed are applications for severe environments which require materials that operate at high temperatures. An emerging area of investigation is the evaluation of ceramics for these environments; and a new class of materials, lubricious oxides, is discussed. Hardbound, 1989. ISBN: 1-55899-013-5.

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Materials Futures: Strategies and Opportunities

(1988 US-Sweden Joint Symposium, Philadelphia, PA) Editors: R.B. Pipes, R. Lagneborg

The technological race which has captured the attention of the world's industrial nations has stimulated considerable discussion about the future. While that was also a major concern of participants at this symposium, conferees addressed this great unknown in part by examining the past - specifically, the materials developments which have shaped commerce over the past four centuries. Using the 350th anniversary of the founding of New Sweden Colony in North America as the occasion for their investigations, academic and industrial leaders from Sweden and the United States gathered in the birthplace of American independence to discuss the importance of materials in developing wealth for the citizens of these two nations. Topics: challenges and opportunities of high temperature superconductivity; materials technology for space applications; catalysis on zeolites; impact of new materials on products and systems in the information technology industry; the role of sensors in intelligent materials processing; the transformation from aluminium to advanced materials; and strategic challenges for materials-oriented firms in the year 2000. 1988, hardcover, 16 papers, 150 pages. ISBN: 1-55899-000-3.

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Computer-Based Microscopic Description of the Structure and Properties of Materials

Volume 63 (1985 MRS Fall Meeting, Boston, MA)
Editors: J. Broughton, W. Krakow, S.T. Pantelides

This volume is of interest to scientists working on electronic structure and the dynamics of atomic motion, as well as those who design and use special-purpose computers and who simulate experimental data. ontents: structural, electronic and magnetic properties of surfaces, interfaces and superlattices; study of surface phonons by electron energy loss spectroscopy - theory of the excitation cross section; computer simulation of electron microscope images from atomic structure models; simulation of equilibrium in alloys using the embedded atom method; dynamics of compressed and stretched liquid SiO_2 and the glass transition; pseudopotential calculations of structural properties; fracture and flow via nonequilibrium molecular dynamics, interatomic forces and structure of grain boundaries; and special-purpose processors for computing materials properties. 1986, hardcover, 36 papers, 289 pages. ISBN: 0-931837-28-6.

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Better Ceramics Through Chemistry II

Volume 73 (1986 MRS Spring Meeting, Palo Alto, CA) Editors: C.J. Brinker, D.E. Clark, D.R. Ulrich

Covers developments of ceramic materials through synthetic chemical routes, e.g., solution processing and polymer pyrolysis, as alternatives to conventional processing of natural minerals mined from the earth; solution chemistry and synthesis of gels and powders; characterization of chemically derived ceramics; drying and consolidation; structure of random and ordered systems; nonoxides; comparisons of chemically and conventionally derived ceramics; applications of MO/MD calculations; and materials for electronic packaging. Also: 22 poster session papers cover ceramics from hydridopolysilazane, bulk glass and glass film compositions determined by inductively coupled plasma-atomic emission spectrometry, the physico-chemical characterization of alumina sols prepared from aluminium alkoxides, etc. 1986, hardcover, 99 papers, 832 pages. ISBN: 0-931837-39-1.

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Better Ceramics Through Chemistry III
Volume 121 (1988 MRS Spring Meeting, Reno, NV)
Editors: C.J. Brinker, D.E. Clark, D.R. Ulrich

Polymer scientists, geologists, microscopists and many other specialists have joined ceramists and chemists to make this a highly successful symposium proceedings series. Principal topics here include sol gel routes for preparing oxides, powder processing and non-oxides. However, this symposium was unique in that it emphasized thin film formation, high temperature superconductors and insitu methods of characterization. Highlights: sol gel chemistry of silicates; thermodynamic versus kinetic control in silicate polymerization pathways; in situ methods such as small angle scattering, photo-physical probes and cryogenic TEM; and an approach to thick films based on organic modification. 1988, hardcover, 122 papers, 844 pages. ISBN: 0-931837-91-X.

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High-Temperature/High-Performance Composites

Volume 120 (1988 MRS Spring Meeting, Reno, NV)
Editors: F.D. Lemkey, A.G. Evans, S.G. Fishman, J.R. Strife

Covers novel processing methods for metal-based composites; deformation mechanisms in metal matrix composites; ceramic composite microstructural development; ceramic composite mechanical performance; composite interfacial effects; novel composite structures; and potential applications in aerospace structures, propulsion devices and energy conversion systems. 1988, hardcover, 41 papers, 382 pages. ISBN: 0-931837-90-1.

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Advances in Structural Ceramics

Volume 78 (1986 MRS Fall Meeting, Boston, MA)
Editors: P.F. Becher, M.V. Swain, S. Somiya

Covers recent research in the field of toughened ceramics, including studies on transformation toughening and fibre- and whisker-reinforced ceramics. Headings: transformation analysis; transformation plasticity and toughness; mechanical properties and microstructures of zirconia-toughened ceramics; mechanical behaviour of reinforced ceramic composites; and fracture and deformation behaviour in ceramic composites. Specific topics include: texture on ground, fractured and aged Y-TZP surfaces; crack propagation in Mg-PSZ; effect of changes in grain boundary toughness on the strength of alumina; and analysis by SIMS and EELS-EDX in a stem of SiC. 1987, hardcover, 30 papers, 306 pages. ISBN: 0-931837-43-X.

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Interfacial Structure, Properties and Design

Volume 122 (1988 MRS Spring Meeting, Reno, NV)
Editors: M.H. Yoo, W.A.T. Clark, C.L. Briant

Covers interrelationships of structure, properties and chemistry of interfaces in metals, ceramics and semiconductors; recent advances in structural characterization and analysis of internal interface and interphase boundaries; structure and thermodynamics; diffusion and segregation; elasticity and localized deformation; cohesive strength and intergranular fracture; processing and alloy design; structural ceramics and superconductors; heterophase interfaces and thin films; effects of processing on the grain boundary structure and chemistry of high temperature ceramic superconductors and the resulting changes in critical currents; and a significant advance in processing and crystal growth techniques, which include the UHV diffusion bonding and MBE techniques for the controlled production of homo- and heterophase interfaces. 1988, hardcover, 80 papers, 606 pages. ISBN: 0-931837-92-8.

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Adhesion in Solids

Volume 119 (1988 MRS Spring Meeting, Reno, NV)
Editors: D.M. Mattox, J.E.E. Baglin, R.J. Gottschall, C.D. Batich

Covers fracture mechanics; fracture mechanics and tribology; postdeposition treatments; deposited inorganic films; adherence of natural layers and surface treatment of polymers; surface treatment of polymers and analytical techniques; and analytical techniques such as electron microscopy and Auger analysis. 1988, hardcover, 40 papers, 312 pages. ISBN: 0-931837-89-8.

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Processing Science of Advanced Ceramics

Volume 155 (1989 MRS Spring Meeting, San Diego, CA) Editors: I.A. Aksay, G.L. McVay, D.R. Ulrich

Covers the role of advanced ceramics and ceramic matrix composites in complex systems for structural, electronic, magnetic and optical applications. The tailoring of composites that display spatial resolution in the micro- and nanometer range; fabrication techniques ranging from consolidation of submicron-sized powders to vapour phase deposition as well as two fundamental fabrication technologies: liquid/solid and gas/solid processes. Topical headings: powder synthesis and colloidal processing; sol-gel processing and ceramic-polymer composites; sol-gel processing of thin films and electronic ceramics; plasma-assisted processing and novel composites; and fibre and whisker-reinforced composites. 1989, hardcover, 40 papers, 387 pages. ISBN: 1-55899-028-3.

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Composite materials

Brochure details specifications of new thermoplastic powder prepregs and commingled yarns from Thermoplastic Composites Div., BASF Structural Materials Corp., Charlotte, N.C. The document also gives an overview of thermoplastic powder prepreg technology, and gives complete specifications for several commercially available products.

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We should appreciate it if readers could take the time to tell us in this space what they think of the 22nd issue of Advances in Materials Technology: Monitor. Comments on the usefulness of the information and the way it has been organized will help-us in preparing future issues of the Monitor. We thank you for your co-operation and look forward to hearing from you.