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REFRACTORIES IN USE [1]

by

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INTRODUCTION

The world production of refractories is about 20 millions tons; about 60% of this amount is consumed in the iron and steel industry, the rest being distributed among glass, cement and other industries. The following proportions in the production of refractories were reported in the late sixties: 70% fireclay and high alumina, 17% basic refractories, 8% silica, 5% miscellaneous.

Technological developments in many fields, especially in steel production, evoked considerable changes in proportions of refractory materials demanded by consumers and this factor has had a profound effect on the refractories industry. During the period 1960-1970 the annual production of fireclay bricks has been slightly reduced, the demand for silica brick has decreased, but the demand for high--alumina and basic refractories has considerably increased. Further increase can be expected also in plastics, castables and fused-cast refractories. The general trend in the use of refractories is to lower their specific consumption, i.e. kilograms of refractory per one ton of the product, which mostly means to use refractory materials of high quality.

The selection of refractory materials for a given purpose can often be made from general experience published in literature and commercial catalogues. On the other hand there exist certain rules that can be used for critical analysis of demands on the properties of the required material in order to select the best refractory or to improve its service. The next chapter will show the main criteria and properties that must be taken into consideration. The following chapters will summarize practical experience with use and service of refractories in various industries.

I. SELECTION OF REFRACTORY MATERIALS

The most important types of refractories are summarized in Table 1. They can be modified using slightly different basic compositions, raw materials and manufacturing processes. For example, fireclay bricks are known in various grades according to the composition and properties of starting clays and to the proportion and granulometry of grog. Super-duty and high-duty grades give the best performance (pyrometric cone equivalent higher than 31); medium duty brick has PCE $>$ 29. A fireclay refractory with higher silica content than 72% is called semi-silica. The quality of fireclay refractories can be increased by addition of bauxite or alumina (high-alumina refractories). Sillimanite refractories are manufactured from natural minerals of sillimanite group, mullite refractories can be prepared from synthetic mullite. Only few basic bricks are made of chromite ore; usually they are a combination of magnesite and chromite in various proportions (magnesia-chromite and chromite-magnesite). Corundum materials are produced as ceramic-bonded (with clay or fine-grain alumina) or fused-cast ones with various proportions of β-alumina. Fused-cast refractories of corundum-baddeleyite type have been increasingly used in the most recent years.

When selecting a refractory material for a given purpose the following aspects are to be considered as the main technical criteria:

A/ The resistance to high temperatures (refractoriness), i.e. the capability of maintaining the chemical and physical identity at high temperatures

B/ Thermo-mechanical behaviour (mechanical strength, deformation under load, thermal shock resistance, abrasion)

C/ Chemical interactions: corrosion by high temperature melts and gases, interaction between various types of refractories.

Refractoriness is the primary property. All solid phases present in the material in considerable proportions must exhibit higher melting points than the presumed temperature of use. A melt may be formed only to such an extent as not to cause deformation. This depends partly also on the viscosity of the melt, as well as on the structure: needle-like structures resist deformation better than structures with isometric grains separated by continuous layers of melt.

Melting points can be useful in the case of pure or nearly pure refractory compounds. Deformation can be observed from temperatures 0,8-0,9 T_m (melting point in ^oK). The deformation temperature depends also on grain size; fine-grained structures deform easier than coarse-grain bodies.

Melting points of the most important refractory compounds are shown in table 2. However, the estimate of the maximum temperature of use from melting points and phase diagrams can be inaccurate if impurities forming eutectic melts are present. Therefore, for practical purposes, determination of softening points by comparison with pyrometric cones is usually carried out. The method compares the behaviour of a cone formed from the sample with a series of standard cones, all run in the same furnace with increasing temperature. The softening temperature, indicated by the bending of the cone, is reported by the number of the standard cone showing identical bending (PCE - pyrometric cone equivalent). There is a slight difference between the cone scale used in Europe and that employed in the USA:

Cone No.	Temperature equivalent in °C	
	Seger cones (Europe)	Standard cones(USA)
14	1410	1398
20	1530	1564
26	1580	1621
32	1710	1717
38	1850	1835

Under suitable conditions the accuracy of the method is ± 15°C. The test must be carried out at a prescribed heating rate in an oxidizing or neutral atmosphere.

The maximum temperature of practical use shall be always lower than the PCE because not only softening but also deformation under load must usually be avoided. This property must be examined by another test the result of which is not directly related to the PCE value. For instance, magnesite brick with PCE above 2000°C may deform under load at 1500°C whereas mullite refractory with lower PCE may resist the load at 1650°C. The conclusion is that the estimation of the PCE can exclude an unsuitable refractory material but does not directly indicate the suitability of the material tested.

Cone bending temperatures of some refractories are as follows:

Corundum :	2000°C
Mullite :	1800°C
Silica :	1710-1750°C
Fireclay :	1630-1750°C
Magnesite :	> 2000°C

The resistance to deformation under load is determined by heating the sample under compressive load and measuring the dimmension of the sample simultaneously or after cooling. Torsion, bending and other methods are sometimes used, especially in more detailed studies of rheological properties. The compressive load in standardized methods is 1,758 kp/cm^2 (USA) or 2 kp/cm^2 (Europe). The result is reported as percent deformation or as temperature of a certain degree of deformation.

Approximate temperatures of initial deformation under load, as found by short-term tests, are as follows:

Silica	1600-1700°C
Fireclay	1250-1450°C
High-alumina fireclay	1500-1600°C
Mullite	1600-1700°C
Corundum	> 1700°C
Magnesite	1500-1750°C
Fused-cast (AZS type)	1730°C

Even though the method simulates the conditions of refractories in service, its results often remain far from real practical behaviour and conditions. It must be noted that short-term tests can hardly describe the long-term behaviour of a refractory because the creep characteristics can be time-dependent; furthermore the dependence of flow rate on stress is rather complex (non-Newtonian flow) and cannot be determined by a simple test.

Information on high-temperature deformation under load as found by commonly used methods allows to compare various materials but cannot be taken directly as the temperature of practical use. The temperature of safe long-term performance is usually lower but depends on the load and temperature distribution.

Compressive strength at room temperature is usually measured and indicated in manufacturer's catalogues. Certain strength is required for transport and handling; the strength can also indicate the previous firing temperature and the degree of vitrification, but it cannot be used to predict the mechanical behaviour at high temperatures. Direct measurements at high temperatures show that compression strength mostly decreases with increasing temperature, but an anomaly is found with fireclay refractories exhibiting a considerable increase in strength at about 1000°C.

High-temperature strength in tension or shear is one of the limiting factors of resistance to thermal shock. Stresses arise in refractories as a result of temperature gradients; the part of a refractory brick having a higher temperature expands more than the cooler one and this is the cause of stresses and fracture if the relevant strength is exceeded. The relation to thermal expansion is evident but also other properties are involved in thermal shock resistance: thermal conductivity affects thermal gradients, modulus of elasticity shows the capability of the material to compensate stresses by elastic deformation etc. High strength and thermal conductivity with low thermal expansion coefficient are the main properties of materials with a high resistance to thermal shock.

Thermal shock resistance is usually tested as a complex property by spalling tests performed with single bricks or panels, which are alternately heated and cooled. The result is specified as the number of cycles causing destruction by cracking and rupture. Silica bricks exhibit low thermal shock resistance at lower temperatures (20–700°C) because of structural inversions, but become very resistant at higher temperatures owing to a flat expansion curve (see Fig. I). High thermal expansion of magnesite is the reason of relatively low resistance but considerable improvement has been attained with more flexible structure which can be obtained generally with larger size of grog, higher porosity and low degree of vitrification. The resistance of fireclay varies with the amount and granulometry of grog, burning temperature etc. The resistance of SiC, carbon and graphite is excellent because of high thermal conductivity (see Fig. II).

Abrasion resistance at increased temperatures is required when solid particles come into contact with, and move along the refractory lining of a furnace. To obtain good abrasion resistance refractories with strong, dense and fine-grained structure have to be chosen. It has been found that abrasion resistance is usually parallel with the resistance to load. Abrasion tests at room temperature are of little value because they do not indicate the behaviour at high temperatures.

The other important criterion for the use of a refractory material is its resistance to chemical attack by molten metals, slags and glasses or by vapours and fine particles captivated by streaming gases. This is often the limiting factor of the life of a furnace. Another type of chemical interaction can occur at the contact of refractories differing in chemical composition.

Rough estimation of the suitability of a refractory for a given medium can be made by comparing chemical compositions

Low interaction can be expected with chemically related media. This is the reason why refractories and heated media are divided into acid, neutral and basic. Materials of the first group contain large amounts of silica whereas the major constituents of basic materials are lime and magnesia. With this criterion refractories can be divided as follows:

Acid: silica, semisilica
Neutral: fireclay, mullite, sillimanite, chromite, corundum
Basic: magnesite, magnesite-chromite, dolomite.

Industrial slags can also be classified as basic or acid according to their SiO_2 and CaO content. Industrial glasses are acid, portland cement clinker is basic. According to this scheme refractories with a high silica content cannot be expected to resist slags with high CaO and MgO etc.

The interaction between various combinations of refractories, as found by experiment, is shown in table 3.

The above described empirical rule is only approximative and not fully reliable in all cases because it does not imply the differences in the rates of corrosion reactions. The theory of corrosion in oxide systems has been considerably improved in recent years; the following parameters have to be taken into consideration in the case of corrosion by high-temperature liquids:

1/ Wetting of the refractory by the corroding liquid; it enables the contact or penetration of the liquid into capillary pores.

2/ Solubility of the solid in the liquid and diffusivity of dissolved constituents in the adjacent layer of liquid; the higher the solubility and diffusivity, the higher the corrosion rate.

3/ The flow velocity of the liquid along the interface; convection accelerates the corrosion process considerably.

Wetting, solubility and diffusivity are invariable parameters for a given composition. Corrosion resistance can be increased only by re-examining and minimizing temperatures, convection and porosity of the material;if this is not possible another refractory material must be chosen.

In the case of interaction with vapours or with another solid the products are not removed from the place of their formation. This process proceeds usually at a lower rate because the product of the reaction makes it increasingly difficult for the reaction constituents to come into contact.

The determination of the corrosion resistance by accelerated tests is difficult. Although many methods have been developped and used they cannot simulate the real conditions of service in all aspects. Direct test in industrial conditions remains as the final proof.

All the above mentioned thermal, mechanical and chemical properties are a function of the chemical composition, phase composition and structure. The relation between intrinsic properties and the behaviour of the material under various conditions is one of the main objects of research in ceramics which has brought many practical improvements for the producers as well as for the users.

II. REFRACTORIES IN THE IRON AND STEEL INDUSTRY

From the total production of refractories for the iron and steel industry approximately 3% are consumed in coke ovens, 9% in blast furnaces and auxiliaries, 59% in all steelmaking processes, 14% in pouring pits and 15% in all rolling and forming processes (5). Thus steelmaking processes consume the greatest portion of the refractories.

There are many new developments in steelmaking. Open-hearth furnaces with a high refractories consumption retreat in favour of the LD process; in 1960 about 72% of the world steel production was produced in open-hearth furnaces, in 1969 only 44%, and further decrease is expected.

This development together with increased quality of refractory materials have the result that the total consumption of refractories dropped from 33 kg per 1 ton of steel (1951) to only 14 kg in 1971 (6). Another consequence is that the production of refractories increases relatively slowly in comparison with the rapid growth of steel production.

In the following chapters the fundamental principles of the most important processes will be described and the best suited types of refractories shown.

A. Blast furnace.

Iron ore is reduced to metal in blast furnaces producing 700-3000 t of iron per day from coke, iron ore and limestone. The blast furnace has the form of a vertical cylinder 15-30m high, with a hearth diameter of 6-8m. Thick refractory walls are surrounded by a steel shell. The mixture of raw materials is fed into the top of the furnace and passes down as the reactions take place forming iron and slag which collect at the bottom and are periodically tapped off through separate notches. Hotter parts of the furnace are water-cooled by hollow metal castings. This contributes to the long life of the furnace which is 2-5 years or even more. The gas escaping from the top (25-30% CO, 1-4% H_2, 50-60% N_2 etc.) is cleaned and part of it burned in 2-3 stoves working on the principle of heat regenerators. Checkers are periodically heated up by combustion gases and cooled, in the following period, by the air to be fed to the tuyères; the air is preheated in this way usually to 700-1000°C, but higher temperatures are used in modern furnaces (up to 1350°C) in order to reduce the consumption of coke.

Temperatures up to 1600°C are reached in that zone of the furnace where preheated air is blasted in, whereas only 250-450°C is the temperature in the upper part of the shaft and about 1400°C at the bottom.

The amount of refractories required for blast furnaces and stoves is large; mostly fireclay bricks of various grades are used. There are severe demands with respect to accurate size; in upper parts of the blast furnace abrasion resistance, thermal shock resistance and also resistance to the disintegrating effect of CO, zinc and alkali vapors are required In the high-temperature zone of the shaft the load-bearing capacity and slag resistance are the most important properties. High-fired fireclay bricks with a dense structure (17-24% porosity) and low reheat shrinkage are used for the hot zone. After long service the largest wear occurs near the top and in the middle portion of the shaft (above the cooling plates), and further in the bottom blocks. In Europe carbon blocks are often used in the hearth of blast furnaces.

Carbon hearths exhibit very long life: 12-18 years. The selection and use of proper refractories is extremely important because no repairs can be done without interrupting the production. With carbon hearths, repeated repairs of shaft linings are possible within a relatively short time by gunning of castables.

A blast furnace of medium size contains about 500t of fireclay bricks and about the same amount of carbon blocks. For 2-3 stoves further 1500 tons of firebricks are necessary. The blast furnace stoves are usually 6-8m wide and about 30m high. Checkers are made of dense high-duty bricks with 40-44% alumina content. Identical material is used for the structure. Firebricks with lower Al_2O_3 content (30-35%) can be sufficient for the middle and lower part of checkers. Also silica is reported to give good service for the dome and upper checkers. Stoves delivering very high hot blast will require high-alumina and possibly, in the future, basic refractories to provide the necessary combination of refractoriness, heat capacity and diffusivity characteristics.

The iron from the blast furnace is transferred in large laddles or transfer cars having a capacity of up to 500 tons of metal. The lining is made of super-duty fireclay. Magnesite bricks are used for miwers where molten iron can be stor ed and homogenized.

B. Cupola furnace.

The cupola is a vertical melting furnace differing from the blast furnace in raw material and by discontinuous operation. The charge consisting of pig iron, scrap, coke and limestone enters the shaft at the top, air for combustion the lower part through tuyères. The shaft is lined with high-

-duty fireclay blocks with dense structure to withstand abrasion and slagging. Because of the periodical character of the process the blocks must be also resistant to thermal shock. Repairs can be made whenever necessary using a plastic material (e.g. plastic fireclay with crushed quartzite); castables consisting of grog, quartzite and high alumina cement can also be used for complete lining.

C. Open-hearth furnace.

Siemens brothers were the inventors of the glass melting tank furnace with regenerators; Martin applied this invention for steelmaking. Therefore the open-hearth furnace is also called Siemens-Martin furnace. The fuel(producer gas or more calorific gas, or liquid fuel) burns in the free space above molten metal and the outgoing combustion gases heat up the checkers. After a period of time (10-30 min) the flow of gases is reversed and the incoming air and gas are preheated in hot checker chambers. Natural gas and coke-oven gas cannot be preheated because of the tendency to cracking; in this case only air is preheated.

The open-hearth furnace operates periodically; the processing of one charge lasts 6-10 hours and after that small repairs can be made if necessary. The charge consists of scrap and cold or hot pig iron. These materials are melted to achieve oxidation of the carbon, elimination of phosphorus in the basic process, and a decrease in the content of silicon, manganese and sulphur by transfering them into the slag. In the acid process (i.e. with acid slag and acid furnace lining) the phosphorus cannot be eliminated. The acid process is used only to a small extent in comparison with basic process, with low-phosphorus raw materials and for special purposes. The basic process operates with basic slag and basic refractories.

The size of the furnace corresponds to a capacity of 50-300 tons of metal and more; the dimmensions of the hearth are 4-6x8-15m. The temperatures are 1650-1750°C at the roof and 1550-1660°C in molten metal. The consumption of refractories for a 100 t furnace is about 500 t. Specific consumption decreases with increasing size of the furnace: 6-8 kg of refractories per 1 ton of steel can be taken as a good result in the basic process. The amount of refractories in checkers is 6-13 t per m^2 of the hearth area. The temperature in checker chambers is 1200-1450°C in the upper section and about 900°C in lower one.

Among the requirements on refractory materials for open-hearth furnaces the most important are the following: good load-bearing capacity, low reheat shrinkage, thermal shock and abrasion resistance in parts where the charge is fed in, and resistance to corrosion by molten slag.

The hearth of the basic furnace is made of magnesite bricks with a working layer of rammed sintered magnesite; to a less extent, chrome-magnesite and dolomite materials are used. If some parts of the furnace are made of silica(e.g. roof and upper courses of front and back walls), a layer of chrome-bricks is introduced between silica and basic material to prevent damage due to chemical interaction. At pre sent chemically-bonded and metal-cased magnesite-chrome bricks are used for the walls. The insulation of the hearth is made of firebricks.

Some years ago the roof used to be constructed of silica because its expansion coefficient is very low and thermal shock resistance is excellent if temperature does not drop below 600°C. However, silica cannot withstand temperatures higher than 1650°C in contact with iron oxide. Higher temperatures were demanded to achieve better heat transfer and better reduction of Mn and Cr from the slag; this can be managed only with basic refractories. Chrome-magnesite bricks together with an advanced roof construction (suspended roof) give very good service and are used especially in furnaces heated by high-calorific gaseous fuels.

The downtakes to the checkers, as well as the upper part of checker chambers, were usually lined with silica brick, but also here basic refractories are in progress. Checkers consist of hard-burned fireclay blocks and more recently also of high-alumina or basic bricks.

As a result of the developments in basic refractories, open-hearth furnaces can be almost completely lined with basic refractory materials except for the bottom insulation and lower courses of checkers, which are made of fireclay bricks. The basic materials used in such a furnace are as follows:

Magnesite (bottoms),
magnesite-chrome (downtakes, walls),
chrome-magnesite (crowns).

Molten steel from the open-hearth furnace is poured into ladles where addition of deoxidizers or alloying elements can be performed. Steel is then teemed into ingot molds through a nozzle in the bottom of the ladle. Ladle linings represent up to 1/3 of the consumption of refractories in the steel production by the open-hearth process. Following properties are required:

a/ thermal shock resistance (abrupt heating up by molten steel)
b/ chemical resistance to the slag
c/ resistance to abrasion by molten steel; particles of the refractory material must not contaminate the steel by oxide inclusions
d/ low thermal conductivity

Several materials were found to give satisfactory service: high-siliceous clay bricks or a rammed mixture of quartzite and clay, high-duty firebricks, or basic materials in special cases.

In order to get a uniform rate of flow from the ladle a material is required that will slowly erode and increase the diameter of the nozzle during the pouring operation. Fireclay or magnezite nozzles are used. The stopper must guarantee a tight fit between the nozzle and stopper without sticking. Both parts therefore can be made of fireclay one of them being less refractory so to be slightly plastic at the pouring temperature. The stopper can be also made from a graphite-clay mixture. The sleeves protecting the steel rod are made of fireclay resistant to thermal shock. Oxide inclusions must not be formed due to the contact of the described parts with molten steel.

D. Converters.

In converters air or oxygen is blown into molten iron in order to oxidize carbon, silicon, manganese and phosphorus Molten iron is supplied from the blast furnace or from the mixer. Bessemer converter is lined with an acid refractory (silica bricks or sandstone). The bottom consists of rammed mixes with holes through which air can be blown into the metal. For iron with higher phosphorus content basic lining must be used (Thomas converter). Tar-bonded dolomite or magnesite lining (bricks or unshaped mixes) are commonly used.

The most important recent development in converter techniques is the use of oxygen (LD process). This process can be applied for low-phosphorus iron using silica converter lining, but the basic process is most frequently employed. The LD process is responsible for considerable decrease of steel production by open-hearth technique which is going to be used mainly for the melting of scrap, in competition with electric arc furnaces. The main advantage of the LD process is a high output from a small vessel and low consumption of refractories.

LD convertor is a pearshaped vessel which can be tilted to the horizontal position. Molten iron and scrap is fed in through the top opening which serves also for pouring. The process is performed in vertical position by inserting a water cooled oxygen lance.

The life of the converter lining was increased considerably when combinations of dolomite, magnesite bricks were applied. High density of the refractory material is required. Converters of 100-300 tons capacity are available and the life is about 600 cycles (compared with 200 cycles ten years ago). The specific refractory consumption is only 2,5-3 kg per 1 ton of steel. To increase the life of the lining, more resistant-even though more costly-refractories are being searched for. For instance, fusion-cast basic blocks have been used around the mouth opening of the converter where the wear is critical. Periclase refractories are expected to be used in larger amounts in the future.

E. Electric furnaces.

Electric furnaces are used especially in the production of high-grade steels and alloys. Electric-arc furnaces are heated by an arc formed between the slag layer and electrodes. The bottom of these furnaces is built from successive layers of firebricks, magnesite bricks and dolomite. The side walls are usually metal-cased magnesite bricks, with fused-cast blocks at the slag line; also large blocks of tar-bonded dolomite are used. The roof is of silica, high-alumina or chrome-magnesite bricks.

In induction furnaces the heat is generated directly in the metal. This type of furnaces can be constructed in a wide variety of sizes, with capacity from experimental melts up to 5 and more tons furnaces. The induction coil is protected by a sillimanite layer. The melting crucible is made of a thin layer of rammed refractory mix (e.g. quartzite with clay, boric acid or sodium-silicate for acid furnaces, magnesite or magnesia-alumina masses for basic process). Fused alumina and zircon refractories have been also used in special cases. Linings are mostly fritted in position; prefired crucibles of magnesite can be used for small furnaces.

F. Vacuum degassing, continuous casting, heat treatment

A small amount of refractory materials of high quality is required in vacuum degassing of steel. Magnesite-chrome and high-alumina bricks give the best service in this process

Continuous casting was developped in 1955 and in 1971 as much as 12% of the world steel production was processed by this method (70-80 mil.tons). A further increase is expected. Continuous casting makes possible to leave out several traditional operations (ingot casting, reheating in soaking pit etc.) which are very expensive. The amount of refractories is small but high quality is required because of important function. The materials used are the following:

Holding ladle: superduty fireclay brick with a high-temperature insulating layer. New development is directed to high-alumina materials based on corundum and to monolithic high-alumina linings

Tundish: superduty fireclay, high-alumina bricks or monolithic lining

Nozzle: dense fireclay, high alumina, magnesite, zircon, zirconia

Tubing: fireclay-graphite, high alumina-graphite, vitreous silica

Stopper rod: fireclay, zircon, zirconia.

The present technique using the stopper rod is going to be replaced by an external sliding-gate system requiring zirconia and high-alumina parts resisting erosion and abrasion.

Only moderate temperatures are attained in the furnaces for heat treatment. Depending on the temperature, insulating firebricks can be used or high-alumina castables and plastics for monolithic linings. Fused-cast corundum-mullite-baddeleyite refractories have been used with good result for the bottoms of soaking pits and for skid rails where resistance to abrasion and to sticking of iron scale is required.

III. REFRACTORIES IN NONFERROUS METALS PRODUCTION

A. Aluminium.

Aluminium is made from aluminium oxide which has been prepared from bauxite. Alumina is calcined to 1200-1300°C in rotary kilns lined with high-alumina bricks in the hot zone and with high-duty fireclay bricks in cooler areas.

Aluminium is produced by electrolytical reduction of calcined alumina in reduction cells with fused cryolite electrolyte at about 950°C. Alumina is dissolved in cryolite and reduced to metal whereas evolving oxygen reacts with carbon anode producing CO and CO_2. The inner lining of electrolytic cells is made of carbon(blocks or rammed paste) with an insulation layer.

Furnaces for the remelting of aluminium are constructed as tank furnaces heated with gas or electricity. They are lined with magnesite and an insulating firebrick layer. Silica-containing materials cannot be used in direct contact with aluminium because SiO_2 would be reduced.

B. Copper and nickel

Copper ores contain usually 1-5% of Cu as $CuFeS_2$, Cu_2S and Cu_3FeS_2 and must be treated by ore-dressing methods. Concentrated ore is roasted in furnaces with hearths of abrasion-resistant firebrick. The roasted ore is smelted in a reverberatory or shaft furnace; after that it is treated in a convertor by blowing air through the melt. The crude copper is then melted in a refining furnace, desoxidized by coke and cast into anodes which are treated electrolytically and remelted in a fining furnace. The complex procedure requires the following refractories in individual steps:

Smelters: similar to a long open-hearth furnace in construction with bottom made of dense rammed silica fritted in place. The side walls are lined with silica bricks (also with magnesite in lower parts) and high-alumina bricks in the end walls. The roof is made mostly of silica (about 100 days life); suspended roofs of chemically bonded chrome-magnesite give longer life. Temperatures of about 1500°C are reached.
Converters: lined with magnesite or chrome-magnesite bricks and with firebrick insulation. The life of the lining is 5-10 years; however, near to the tuyères the wear is rather extensive and repairs must be made after about 1000 hours of operation.
Anode refining furnace: reverberatory type of furnace (oil or gas heated) with acid bottom. In the side walls magnesite and silica are used; the roof is made of silica, magnesite or forsterite bricks. Electric arc furnaces with basic lining are also used for this operation.

Nickel ores contain /Fe,Ni/S and $/Ni,Mg/SiO_3$. The process depends on the type of ore; it is in many respects similar to that of copper production in the case of sulphur containing ores. The refractories used are fireclay, high alumina and magnesite.

C. Lead.

The most important ores contain lead in the form of PbS. The ore must be concentrated by flotation and oxidized by roasting and sintering. The reduction to metal is performed in blast furnaces with water cooling. In the upper part, above the water jacket, high duty fireclay bricks of low porosity are used. The temperatures are relatively low but good abrasion resistance is required. Dense firebricks are used also in the crucible of the furnace. Magnesite bricks can be used in parts of heavy operating conditions.

Refining furnaces of reverberatory type must withstand the effects of PbO-containing slag. Air is blown into the bath to oxidize As, Sb and Sn. The portions of the furnace coming into contact with the slag (hearth and walls) are constructed of magnesite, the upper parts of medium duty firebrick (maximum temperatures about 1100°C) or high-alumina firebricks. Identical materials are used in other types of furnaces for lead refining.

D. Zinc.

Zinc ores usually contain ZnS or $ZnCO_3$. Sulphur containing ores must be desulphurized by roasting or sintering, carbonate ores are calcined in rotary or shaft kilns. The product is reduced by carbon at about 1100°C, i.e. above the boiling point of Zn (906°C) so that evaporation of Zn takes place and the process must be performed in closed vessels (retorts) about 1-3 m long, with condensers at the open end retaining the liquid zinc. Retorts and condensers are pressed of fireclay mixtures. Low porosity is required to prevent loss of zinc vapours. Retorts are placed in long gas fired furnaces of regenerative type operating at about 1400°C. The centre wall of the furnace holds the retorts and is heated from both sides; therefore it must be made of high-quality refractory, e.g. high-alumina bricks. The side walls are built of fireclay bricks. The life of the retorts is 25-50 days, that of the furnace is several years. Continuous zinc distillation can be also performed with vertical retorts of silicium carbide.

IV. REFRACTORIES IN THE GLASS AND CERAMIC INDUSTRY

A. Glass melting furnaces.

Recent developments in the glass melting process and furnace design are characterized by increased temperatures of glass melting that are between 1450-1550°C with common glasses and even higher with hard borosilicate and special glasses. The reason is to achieve higher outputs and better properties, especially chemical durability. Another trend in melting techniques is the use of direct electric heating achieved by molybdenum electrodes immersed into molten glass which is a good ionic conductor at high temperatures. A combination of gas and electric heating is mostly used (electric boosting) in continuous glass tank furnaces.

The increase in melting temperatures and especially electric heating brings about more intensive convection in the melt which is responsible for higher corrosion activity; therefore high-quality special refractories are used at least for the most exposed parts of glass melting furnaces. The batch consisting of sand, limestone, soda etc. is charged at the feeding end of the tank furnace; the batch is taken by pull current to the hot spot (maximum temperature point) where extensive mixing proceeds by convection currents arising due to temperature and density gradients. Molten, homogenized and refined glass has to pass into the working end through a throat or neck. The combustion space is heated by gas or liquid fuel and it is divided into two separate chambers by a shadow wall making possible conditioning of molten glass for forming operations. Continuous flat glass tanks are up to 50 m long and have a fireclay floater on the surface of molten glass between the melting and working space of the furnace. The output of a large melting furnace in flat glass production is up to 300 tons per day; container glass furnaces produce 50-150 tons of glass per day. Specific output amounts 800-4000 kg per day and m^2 of melting area (the higher value is attained with electric boosting); thermal efficiency of gas fired tanks with heat regeneration is 20-35%.

The mean consumption of refractories in melting tanks for sodalime glass is about 15 kg per ton of glass but only 0,5 kg of that amount is dissolved in glass during the life of the furnace which is about 2 years. The average relative consumption of various refractories in glass tanks is as follows (weight units): 48% fireclay products, 30% silica, 15% fused-cast refractories and 7% basic materials. Fused-cast refractories are the most expensive ones; in spite of only 15% share in weight the cost can be more than 50%.

The consumption of various refractories per 1 ton of produced glass in an average tank furnace is about 7 kg fireclay, 4,5 kg silica, 2,5 kg fused-cast and 1 kg basic. New developments in glass tank construction bring about lower total consumption with increased proportion of fused-cast and basic refractories.

Traditional refractory materials are fireclay blocks for the bottom and side walls (30-43% Al_2O_3), silica for the roof and high-duty fireclay for checker chambers and checkers. Heavy corrosion occurs at the level of molten glass and therefore fused-cast blocks have been used at least for this part of the tank. High corrosion resistance of fused-cast refractories is due not only to the chemical and phase composition (mostly materials on corundum-baddeleyite basis with 32-36% or about 40% ZrO_2), but also due to the absence of capillary pores. This type of material can be used also for throats, feeders, electrode and bubbling blocks, doghouse corners, burner ports etc. At present, in hard-driven furnaces, all basin is lined with fusion-cast blocks. For low alkaline glasses fused quartz blocks are widely used in continuous tanks. Low expansion, low porosity and easy crystallization to cristobalite are the main characteristics of this material which is also suitable for special parts with high thermal shock resistance.

Silica is the traditional material for the superstructure. More recently, in areas such as breast and back walls, port arches and port necks, extensive use has been made of fused-cast blocks, sillimanite and mullite blocks. Magnesite and magnesite-chrome have also found application especially in port necks. Silica remains the major material for crowns but increasingly super-duty silica refractories with low alumina content (0,15-0,3%) are being used. In order to withstand alkali vapour attack silica brick must be of low porosity; quartz content must be low to reduce afterexpansion. However, silica crowns are unsuitable for melting temperatures above 1600°C and for glasses containing agressive components. In furnaces for hard borosilicate glasses, fused -cast refractories of corundum-baddeleyite type have been used since 1960; such crowns can be heated up more easily than silica crowns, they are corrosion-resistant, and furnace pull can be increased by raising the melting temperature. Also magnesite has many desirable properties with proper crown design and suspended construction.

Refractories in glass tank regenerators fail mainly through a combination of attack by batch carry-over, alkaline vapour and temperature cycling. The conditions vary considerably in various regions. Usual materials are silica, various grades of firebricks and high-alumina bricks. In recent years magnesite and magnesite-chrome have been used with succes in most regions of regenerators including the crowns and dividing walls. Basic materials exhibit good resistance to alkali and good heat capacity, but do not resist SO_3 and thermal shocks.

Zircon refractories ($ZrO_2 \cdot SiO_2$) are often used in various places of tank furnaces for melting borosilicate and fluoride glasses (basin, feeders, throats).

Special glasses, for example optical, coloured, decorative etc., which are produced in small amounts with wide variety of composition, are still preferably prepared by melting in clay pots. The pots are made of highly plastic clays with grog and formed by slip-casting or isostatic pressing. They are supplied in non-fired state and heated, before use, in special furnaces ("pot arch"). From there they are transferred directly into the melting furnace; sintering of the pot proceeds in position. Pot furnaces exhibit very low thermal efficiency; there are usually no special requirements on refractories because they do not come into contact with molten glass.

B. Cement and lime industry.

Portland cement is the most usual type of cement; it is produced from a mixture of limestone, clay and minor constituents. Sintering temperatures are about 1450°C. The products of high-temperature reactions are calcium silicates and aluminates which are capable of hardening when mixed with water. Approximate chemical composition of the cement clinker is: 66% CaO, 22% SiO_2, 6% Al_2O_3 and 3% Fe_2O_3.

In cement industry mostly continuous rotary kilns are used. The raw materials mixture is fed at the upper end of the inclined cylindrical furnace rotating at a speed of about 1 rev/min. The charge is introduced dry or as a slurry and moves to the lower end, in opposite direction to the flame and hot gases. The product must be rapidly cooled after leaving the furnace to sustain the high temperature phase composition.

The length of rotary cement kilns is 70-200 m, the diameter 3-6 m. Heat consumption depends on the system of preheating; it is possible to attain about 900 kcal per kg of clinker but values up to 1700 kcal/kg are reported in the wet process. The production of rotary kilns is 400-3000 tons per day.

Different refractories are used in various zones of the furnace. In the cooler part good mechanical strength and abrasion resistance are required; dense fireclay bricks perform best here, and dense superduty firebricks in the intermediate zone. In the hot zone, with clinker temperature of 1450°C and with about 25% of liquid phase in the clinker, high-alumina blocks and in recent years mostly basic linings have been used. Good results have been obtained with magnesite or magnesite-chrome, fired or chemically bonded and metal-cased. In the hot zone a coating of clinker mass is formed on the lining which reacts partly or infiltrates into the refractory material. Its function is to reduce the temperature and heat losses, and to extend the life of the lining which is usually about 9 months in this case. The life is considerably shorter if the coating is not formed; basic linings hold the coating better than high-alumina linings. The specific consumption of refractories is 0,5-1,0 kg of magnesite and about 0,7 kg of fireclay per 1 ton of clinker.

Insulating layer between the lining and the shell should lower the heat losses but it also raises the temperature of the lining and disturbs the conditions for the building up of the protective coating. Therefore insulation is used only rarely (fireclay plates).

Shaft kilns are still sometimes used in cement production. Identical refractories are applied as in rotary kilns: fireclay bricks in the upper part, high-alumina or magnesite in the hot zone. The life is 12-24 months.

Shaft kilns of similar construction are also used for lime burning. In lime production a mixture of coke and limestone is charged into the top and air is blown at the bottom. Exterior dimensions are up to 8 m diameter and 30 m height, with an average of 3,5x15 m and output of 200-300t/day. Gas-or oil-fired kilns have been also developed using a center burner. The temperatures in lime burning are lower (1250-1350°C) than in cement production and there does not arise any liquid phase; the conditions of service are therefore not so severe. The kilns are usually lined with high-duty or super-duty firebricks; high-alumina linings have been found useful in high-output kilns and more recently also magnesite in the hot zone.

Rotary kilns are used to a smaller extent for lime burning. The lining consists of super-duty fireclay or high alumina blocks, and of magnesite with steel-sheet inserts in the hot zone.

C. Ceramic kilns.

Periodic kilns are still sometimes used in ceramic industry, mainly for the firing of refractories and heavy clay products. There is no direct contact between refractory lining and fired products so that the corrosion problem need not be taken into consideration except for slagging by the coal ash. Periodic kilns operate between 1000°C and 1540°C. Firebricks are mostly used for the construction, employing a quality corresponding to the temperature required. Because the kilns are periodically heated and cooled, silica bricks are not satisfactory. Periodic kilns have recently been constructed with linings of insulating firebrick. The main advantage is the reduction of heat accumulated in the walls and roof; this brings about a substantial savingin fuel and energy, a quicker turnover and more even temperature distribution over the space of the kiln. This development allows wall thickness and weight to be reduced. Insulating bricks are nowadays available for operation up to 1600°C. The use of outer insulation is likewise increasing, especially in the crown in order to save fuel. On the other hand, insulation requires a better grade of inner refractory because higher temperatures are reached in the lining.

Continuous compartment kilns are sometimes used for firing common bricks, lime and silica. Each chamber is similar to a single periodic kiln. Cold air and combustion gases pass from one chamber to the next; in this way the heat of fired bricks is used to preheat the air for combustion, and the heat from combustion gases is used to preheat the cool products. The fuel consumption is only 1/2 - 1/3 of that of single periodic furnace. The main disadvantage is difficult charging and discharging which cannot be mechanized to such an extent as with tunnel kilns.

Tunnel kilns have a thermal efficiency approximately equal to that of compartment kilns because a preheating system is also utilized. Their advantage, in comparison with compartment kilns, is in the possibility of setting the products on cars outside of the kiln. As regards refractories the conditions of service are much better because temperatures are kept constant. Low-duty fireclay bricks are used in low-temperature zones, and medium to superduty firebricks in the hot zone. The tunnel kiln is insulated to prevent high heat losses.

Some tunnel kilns have a muffle protecting the products from direct contact with combustion gases. The material of the muffle should have high thermal conductivity and good hot strength. Muffles are usually made of clay-bonded silicon carbide or clay-bonded fused alumina.

Car bottoms are exposed to hard conditions of service because they are periodically cooled and heated up. They are usually made of large blocks or of light-weight castables. For firing of porcelain and other products saggers and settings are used made of high alumina or bonded silicon carbide refractories.

V. MISCELLANEOUS INDUSTRIES

A. Coke ovens.

The production of coke and gas is carried out in ovens consisting of chambers coupled into batteries and separated by vertical heating walls. The chambers are 0,35 - 0,55 m wide, 3 - 5 m high and 8 - 13 m long. The coal is fed in at the top of the chamber and the coking process takes place during a period of 15 - 30 hours. Then the charge is pushed out laterally and quenched. The gas escaping during the coking process is collected in a large main and partly used for firing the coking chambers (1250-1350°C). The heating is of regenerative type.

Silica bricks are used for the walls of heating chambers. They have very low thermal expansion at working temperatures and therefore very good thermal shock resistance, good mechanical strength and resistance to abrasion by entering and leaving charge. The temperature must not be allowed to drop below the critical temperature of silica involving modification changes, e.g.600-700°C.

The pores of the brick are filled with coke after long operation . The phase composition is changed to tridymite on the side of the coke chamber, whereas cristobalite is the prevailing phase on the hot face with a surface glassy layer formed in contact with the flame.

The amount of refractories in a coke oven battery is large - it attains several thousands tons. The specifications for silica bricks for the chamber walls can be summarized as follows: $SiO_2 > 93\%$, $Al_2O_3 < 2,5\%$, $CaO < 3,5\%$, load resistance > 1620°C, true density $< 2,35$, porosity $< 25\ \%$.

Lower quality is allowed for other portions of the coke oven. Semisilica bricks containing 70 - 80% SiO_2 are still used in small or horizontal types of coke ovens. Semisilica is also used for checker chambers. The heating up of a coke oven battery takes 8 - 10 weeks because certain limits of silica expansion must not be exceeded. The most critical region is between 100-300°C where transformations of tridymite and cristobalite occur accompanied by considerable expansion.

There is not expected any change in the type of refractory material for coke ovens. There is a tendency to decrease the number of special shapes required for a new oven in order to decrease the cost of coke oven refractories.

Improved refractories have been developed for coke-oven doors; materials ranging from aluminate cement-bonded castables to fused silica shapes are now used.

B. Steam boilers.

The use of refractories in steam-power generation has decreased because water-walls are used with little demand on the volume and quality of refractory materials. In the zones with temperatures lower than 450°C common bricks can be applied excepting places where interaction with combustion gases containing SO_2 can occur. Otherwise fireclay refractories (bricks and plastics) are used in combination with insulation firebricks and diatomaceous blocks. Under conditions of severe slag erosion chrome-based plastics are used, especially in the studwalls of boiler furnaces; they can be rammed around the water-wall tubes. This type of plastic can be made of raw or calcined chrome ore with clay or sodium silicate bonding. African and Cuban chrome ore is preferred for this purpose.

C. Enamelling.

Furnaces used in enamelling of cast iron and steel-sheet operate at relatively low temperatures, below 1000°C for ordinary enamels. Periodic as well as continuous tunnel kilns are used and heated by gas or electricity. Gas fired furnaces are provided with muffles of SiC or bonded alumina.

The walls of periodic kilns are made of firebrick with an outside insulation. The doors are often lined with a light-weight castable or with insulating firebrick; this enables rapid operation with the door which is necessary because of very short firing intervals (minutes in the case of small sheet-steel products). In continuous furnaces walls can be made of insulating firebrick.

D. Chemical industry.

Refractories are used in the chemical industry only to a very limited extent but conditions of service can be hard as regards chemical attack. For example SiO_2 containing refractories cannot be used in contact with hydrofluoric acid; in this case carbon or zirconia materials are suitable. On the contrary, carbon must not be used in contact with nitrates and other oxydizing agents. For chlorine containing substances the reaction of Cl_2 with metal oxides must be taken into consideration; fused quartz gives better results than alumina containing refractories. For sodium salts (soda, sulphate) chrome brick can be employed. For sulphur and pyrite burning furnaces high duty fireclay is satisfactory. Petroleum industry needs refractories for the crude oil cracking process. At present, for reaction vessel linings mostly castables are used which can be placed by gunning inside the steel shell and reinforced, if desirable, with steel mesh. Monolithic linings are made of fireclay with aluminate cement or of alumina with phosphate bond.

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Table 1. Composition of refractory materials

Material	Chemical composition	Main refractory compounds
Silica	93-97% SiO_2 CaO,Al_2O_3,Fe_2O_3	SiO_2(tridymite, cristobalite,quartz)
Fireclay	15-46% Al_2O_3 50-80% SiO_2	$3Al_2O_3.SiO_2$(mullite)
High-alumina	50-99% Al_2O_3	$3Al_2O_3.2SiO_2$(mullite) α-Al_2O_3 (corundum)
Sillimanite	theor. 63% Al_2O_3 37% SiO_2	$3Al_2O_3.2SiO_2$(mullite)
Mullite	theor. 72% Al_2O_3 28% SiO_2	$3Al_2O_3.2SiO_2$(mullite)
Magnesite	$>$83% MgO Fe_2O_3,CaO,SiO_2	MgO (periklas) $MgO.Fe_2O_3$(magnesio-ferrite)
Chrome-magnesite	25-55% MgO 20-35% Cr_2O_3 Al_2O_3,Fe_2O_3,SiO_2	MgO (periklas) spinel
Chromite	35-45% Cr_2O_3 Fe_2O_3,MgO,Al_2O_3	$(Fe^{2+},Mg)(Fe^{3+},Al,Cr)_2O_4$ (spinel)
Dolomite	32-60% CaO 32-45% MgO 1-15% SiO_2	MgO,CaO,($3CaO.SiO_2$)
Forsterite	50-58% MgO 30-39% SiO_2,Fe_2O_3	$2MgO.SiO_2$(forsterite)
Zircon	60-66% ZrO_2 30-35% SiO_2	$ZrO_2.SiO_2$(zirconium silicate)

Table 1. (Continued).

Material	Chemical composition	Main refractory compounds
Zirconia (stabilized)	92-96% ZrO_2 2-6% CaO	ZrO_2-CaO solid solution (cubic)
Fused-cast (Al_2O_3-ZrO_2-SiO_2) type	47-54% Al_2O_3 30-41% ZrO_2 10-14% SiO_2	α-Al_2O_3 (corundum) ZrO_2 (baddeleyite)
Silicium carbide (clay bonded)	50-90% SiC Al_2O_3, SiO_2	SiC (silicium carbide) (mullite)
Carbon	90-98% C	carbon

Table 2. Melting points of refractory compounds

Compound	Melting point in °C
Al_2O_3	2050
CaO	2570
MgO	2830
SiO_2	1710
ZrO_2	2720
SiC	2700
$3Al_2O_3 \cdot 2SiO_2$ (mullite)	1810
$2\ MgO \cdot SiO_2$ (forsterite)	1890
$ZrO_2 \cdot SiO_2$ (zircon)	2500
$FeO \cdot Cr_2O_3$ (chromite)	1770
$MgO \cdot Al_2O_3$ (spinel)	2135

Table 3. Interaction between individual types of refractories

	Fireclay				High-alumina (90%)				Silica				Magnesite				Chrome-magnesite				Chromite			
	1400°	1500°	1600°	1700°	1400°	1500°	1600°	1700°	1400°	1500°	1600°	1700°	1400°	1500°	1600°	1700°	1400°	1500°	1600°	1700°	1400°	1500°	1600°	1700°
Fireclay					0	0	0	0	0	2	3	3	2	3	3	3	0	0	1	3	0	0	2	3
High-alumina (90%)	0	0	0	0					0	0	0	3	0	0	2	3	0	0	0	2	0	0	0	2
Silica	0	2	3	3	0	0	0	3					0	2	3	3	0	0	2	3	0	0	0	2
Magnesite	2	3	3	3	0	0	2	3	0	2	3	3					0	0	0	0	0	0	0	2
Chrome-magnesite	0	0	1	3	0	0	0	2	0	0	2	3	0	0	0	0					0	0	0	0
Chromite	0	0	2	3	0	0	0	2	0	0	0	2	0	0	0	2	0	0	0	0				
Forsterite	1	2	3	3	0	0	0	3	0	0	2	3	0	0	0	2	0	0	1	2	0	0	2	2

0 ... no interaction; 1,2,3 ... poor, medium and strong reaction, respectively

Figure I. Thermal expansion of refractory materials

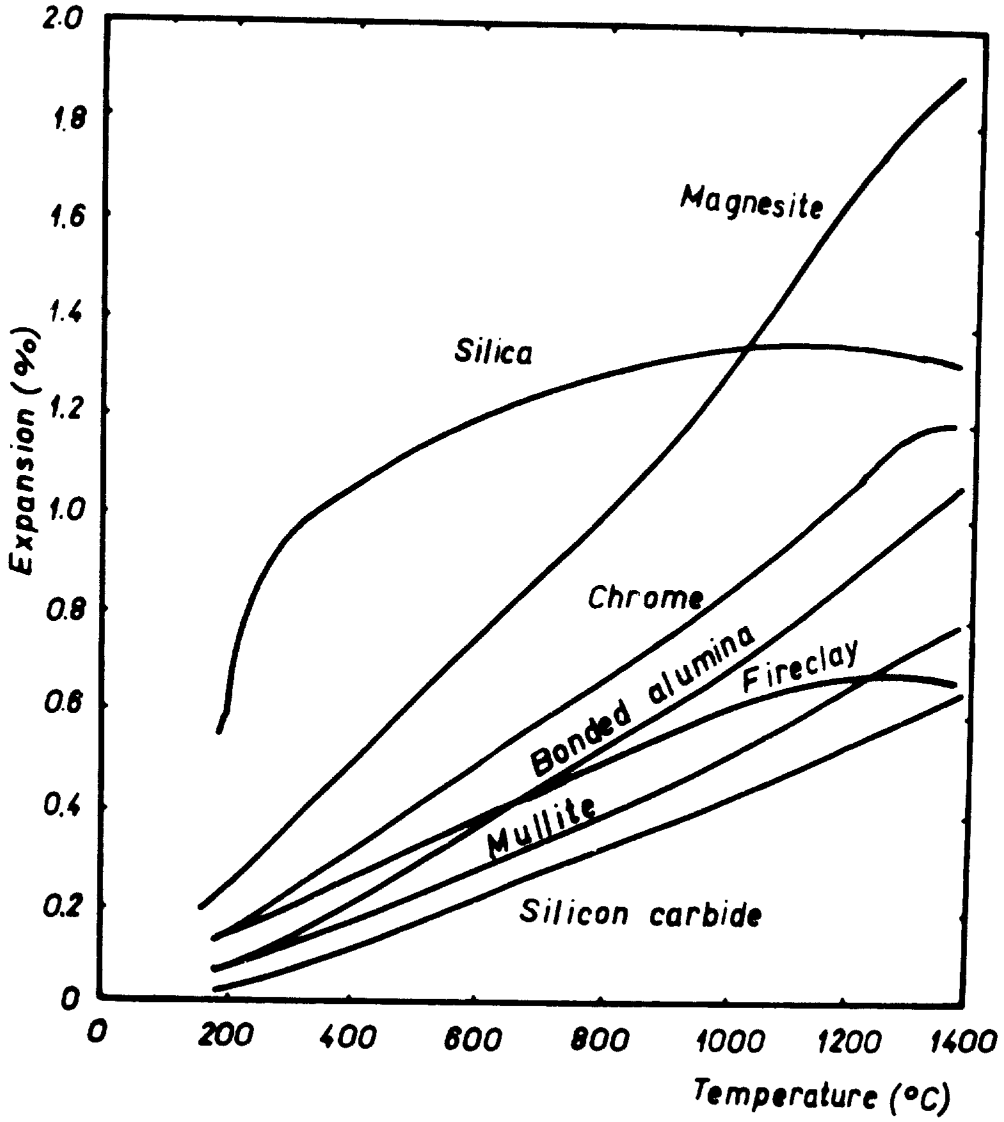
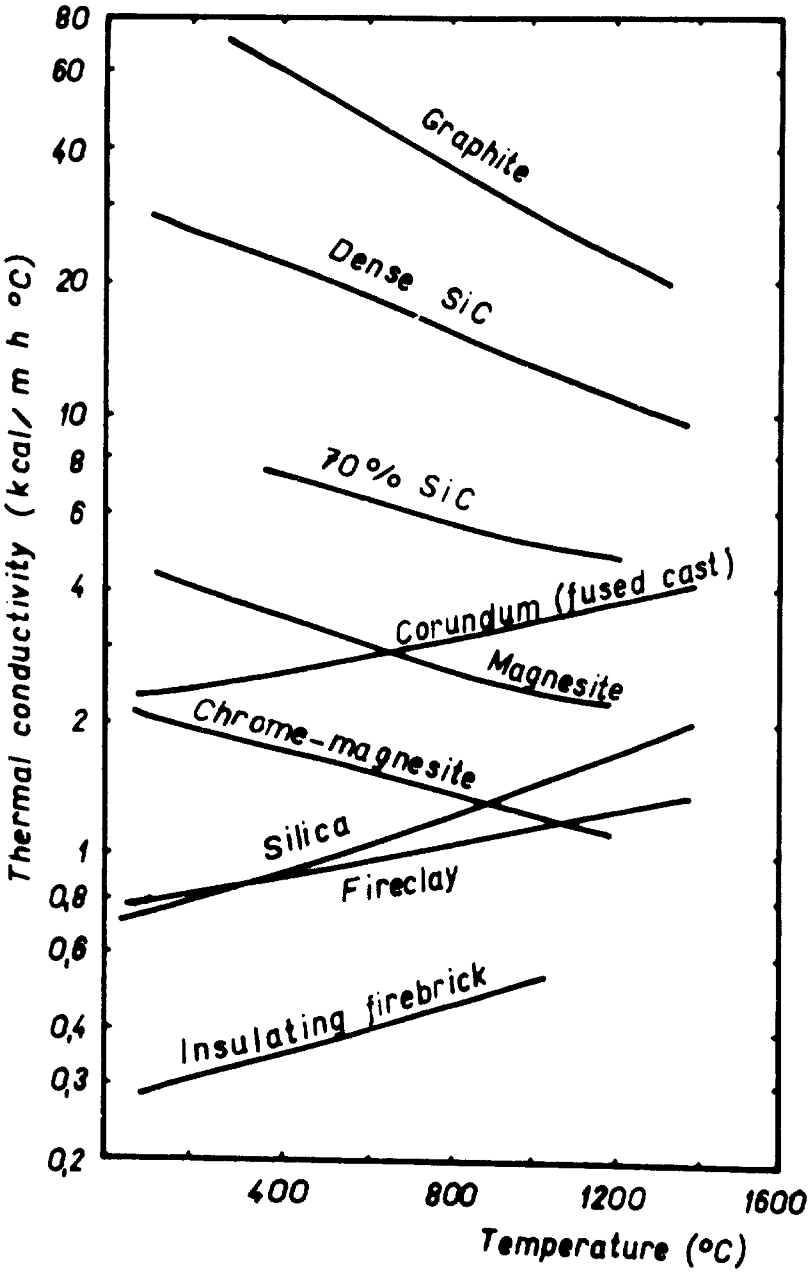


Figure II. Thermal conductivity of refractory materials



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Curso práctico de capacitación en el trabajo
sobre fabricación de productos refractarios

Pilsen (Checoslovaquia)

11 - 28 junio 1974

USO DE LOS PRODUCTOS REFRACTARIOS[1]

por

J. Hlaváč*

RESUMEN

En la utilización de materias refractarias se observa una tendencia general a emplear materiales de buena calidad y a reducir el consumo específico. Durante el período 1960-1970, ha bajado ligeramente la producción de ladrillos refractarios, ha disminuido la demanda de materiales refractarios de sílice, y en cambio ha aumentado la de materiales refractarios con elevado contenido de alúmina y básicos. También se puede prever que seguirá aumentando la demanda de materiales refractarios plásticos, moldeables y colados por fusión.

Los artículos de arcilla refractaria representan más del 60% de la producción de materiales refractarios. Los ladrillos de arcilla refractaria se utilizan para revestimientos interiores de diversos hornos, por ejemplo, altos hornos y cubilotes, y en las zonas de los hornos de cemento, las caleras, los hornos para productos cerámicos, etc. en que las temperaturas son más bajas. Se consumen grandes cantidades de productos de arcilla refractaria para recuperar calor en termorregeneradores y como

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[1] Las opiniones que el autor expresa en este documento no reflejan necesariamente las de la Secretaría de la ONUDI. La presente versión española es traducción de un texto no revisado.

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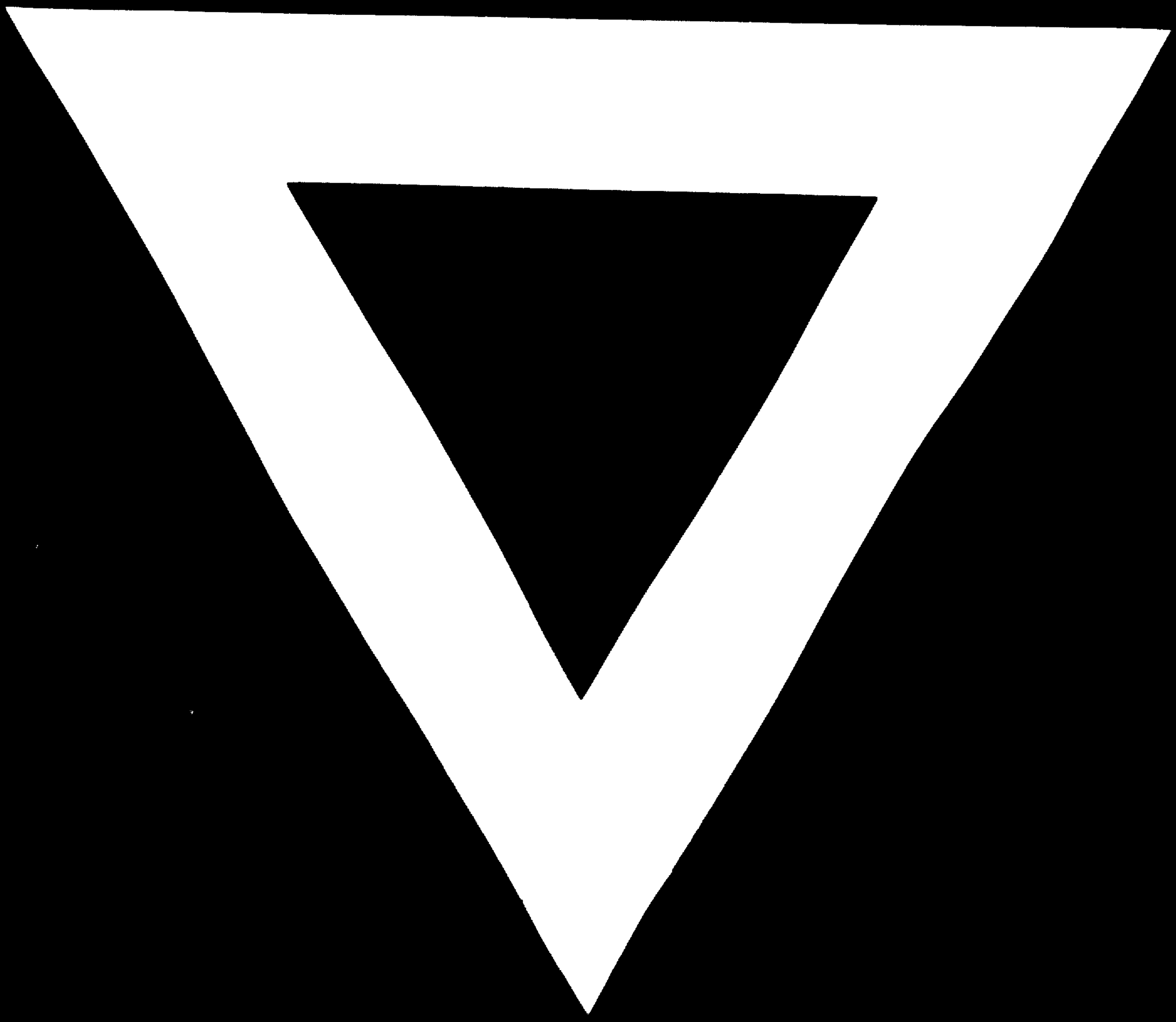
materiales refractarios para termoaislamientos y fosos de colada. Los materiales refractarios con elevado contenido de alúmina cada vez se utilizan más en recuperadores de calor y revestimientos interiores que han de soportar temperaturas elevadas.

Los materiales refractarios básicos se necesitan especialmente en los procesos siderúrgicos. El interior de los hornos de solera abierta se puede revestir casi totalmente con materiales básicos. Sin embargo, está disminuyendo el empleo de los hornos de solera abierta, que van siendo sustituidos por convertidores LD con revestimientos básicos, los cuales consumen menos materiales refractarios a base de dolomita y magnesita. Los revestimientos interiores de magnesita suelen utilizarse para las zonas de los hornos rotatorios de cemento donde reinen temperaturas elevadas.

La sílice es el material tradicional para bóvedas, pero en muchos casos se la ha sustituido por materiales refractarios básicos. Todavía se la utiliza en bóvedas de hornos de fundir vidrio. Se consumen grandes cantidades de materiales refractarios de sílice en los hornos de coque.

Los materiales refractarios colados por fusión se utilizan especialmente en la industria del vidrio. En la actualidad, la cuba de muchos tanques de fundir vidrio se reviste con bloques de Al_2O_3-ZrO_2-SiO_2 colados por fusión. Los materiales de zircón se utilizan especialmente en los hornos de fusión para vidrios a base de borosilicatos y fluoruros.



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