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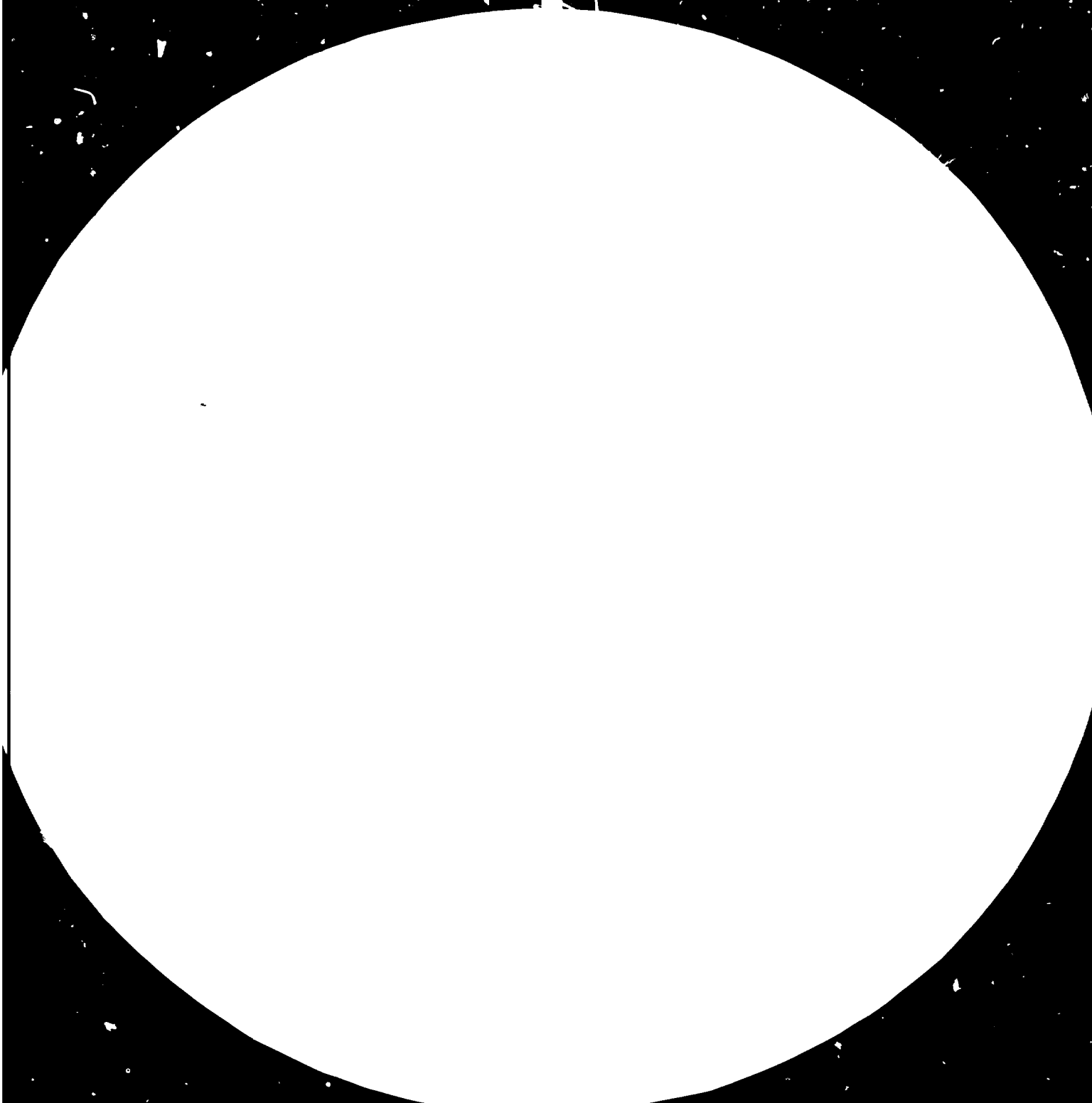
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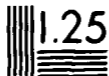
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Survey on Polyester Fibres, their Chemistry and Technology

Dr. Günther Peters

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August, 1979

Summary
of a lecture on
"Polyester Fibres, their Chemistry and Technology"

Polyester is the generic term for any fibre in which the fibre forming substance is any long chain synthetic polymer composed of at least 85 % by weight of an ester of dihydric alcohol and terephthalic acid (TPA).

The specific polyester "polyethylene terephthalate" (PET) obtained from TPA and ethylene glycol which found widespread industrial application was discovered by Whinfield and Dickson in the U.K. in 1941.

The feedstock for TPA and its dimethyl ester "DMT" is primarily p-xylene. This is separated from industrial mixtures of xylenes which are obtained by catalytic re-arrangement (platrefoming) of naphthenic compounds contained in petroleum.

For the conversion into TPA or DMT p-xylene is oxidized by air and in the latter case methylation takes place simultaneously with oxydation at high pressure.

To synthesize polyethylene terephthalate suitable for the manufacture of polyester fibres highly purified raw materials are required, they must be "fibre grade".

As DMT can easily be purified it was for many years preferred as feedstock for the polymer. In the meantime, however, also TPA can be highly purified so that both compounds are available for the production of fibre grade polymer.

The other compound, ethylene glycol, is obtained by means of direct oxidation of ethylene by air on a silver catalyst to ethylene oxide and its subsequent hydration.

TPA or DMT and ethylene glycol are converted by esterification or ester-interchange respectively into bis-hydroxyethyl terephthalate (BETH).

During polycondensation of this diglycol ester of TPA ethylene glycol separates and polyethylene terephthalate (PET) is formed. Usually this is achieved by means of continuous vigorous mixing of the mass and creation of high vacuum at elevated temperatures and in the presence of catalysts.

It has been established that the molecular weight of fibre grade PET is of the order of $\pm 20\ 000$.

In the formation of polyester fibre a grid type or extruder type melting device is used. The molten polymer is forced through spinnerets (i.e. metal discs with numerous apertures) by means of spinning pumps which also serve for metering. The filaments emerging from the spinnerets travel through spinning shafts where they solidify, subsequently they are coated with spin finish and passed to the take-off device.

For the production of filament yarns a wind-up machine is employed whilst in the case of staple fibre production the spin tow is loaded into cans.

In the melt spun fibre the macromolecules are only pre-orientated and still unstable. The fibre needs to be drawn to receive a significant crystalline structure and consequently its valuable physical properties, namely high tenacity and less elongation under load.

Drawing is carried out between two sets of godets which revolve with different speed. After drawing the fibre is crimped in a machine of the Stufferbox type and subsequently heat-set. The special conditions of drawing - with due regard to melt-spinning conditions - in conjunction with heat setting offer a wide variety of possibilities to manufacture fibres with quite different properties. Heat-setting e.g. is necessary in order to decrease shrinkage and to increase the stability of the macromolecules as well as of the fibre crimp for further processing i.e. textile spinning.

The final operations are the cutting and packing of the fibre into bales or alternatively to load "converter tow" into cartons. Staple fibre is supplied to cotton mills and converter tow to worsted mills.

Filament yarns are taken-off from the spinning machine and wound on bobbins. Subsequently drawing is required for the same reason as explained in the case of staple fibre.

In the past draw twisting machines have been employed and are still in operation. In recent years new technique has been developed allowing to spin, draw, twist and even to texturize filaments in consecutive steps in one machine only.

D. van

August 1979

Survey on Polyester Fibres, their Chemistry and Technology

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I

Introduction (World Man-Made Fiber Survey)

In 1970 the production of natural and man-made fibers totalled 21,561 mill. metric tons with a share of 62 % of natural- and 38 % of man-made fibers. 8 years later after the first oil crisis and recession in industrialized countries which began in the second half of 1974 the total production of all fibers amounted to 27,846 mill. tons (+ 29,2 %) of which natural fibers had a share of 52 % (-10 %) whilst the balance of 48 % was attributable to man-made fibers.

During this period man-made cellulosic fibers (rayon and acetate) suffered a loss of 3,5%. Various factories were closed down or production curtailed due to price ruin and/or environment problems like air pollution and contamination of effluent.

On the other hand production of 100 % synthetic fibers grew by 111,6 %. At the beginning of this decade polyamides still had the lion's share. They were overtaken, however, first time in 1972 by polyesters which exceeded production of polyamides by 50 % in 1978.

From 1970 to 1978 polyesters had world-wide a growth rate of 185 % (polyamides + 64 %, acrylics + 101 %).

With the assistance of Western European countries and the USA and by applying their technologies big new plants for the manufacture of synthetic fibers were build up in South America and particularly in the far East. A huge man-made fiber center was e.g. set up in Taiwan. The aforementioned countries showed an overproportional share of the growth rate of synthetics production during the time under consideration (re Table V).

Whilst in West Europe, USA and Japan 1970 still 82 % of all synthetic fibers were manufactured a loss of 16 % of total world production was incurred in these regions until 1978 when their share was down to 66 %. These figures indicate that there was a significant shift of synthetic fiber capacities in favour of developing countries particularly in the far East.

II

Such a development had of course serious repercussions on the man-made fiber and textile industry in highly industrialized countries like West Europe.

Not only big market shares were lost in export business but also on the domestic market. Synthetics emanating from such new fiber plants are processed on the spot by textile mills and the local clothing industry where rather cheap labour is still available for labour intensive operations. As a result huge quantities of ready made garments are being imported mainly from the far East into West Europe at prices with which many European manufacturers cannot compete anymore. At present for example from every 100 of the following garments sold in West Germany are imported : shirts 85, jackets or coats 67, trousers 60 and suits 50. It cannot surprise that under such circumstances a considerable part of the West European textile and clothing industry is facing big losses, quite a few companies went already bankrupt and most probably there are more to follow. According to an EC-Commission study up to 1,5 million jobs are endangered over the next 10 years in this industry. This is roughly half of the current work force of the branch.

No wonder that also the synthetic fiber industry is seriously affected by the extreme difficulties of its direct customers. For at least the past five years it has been running in the red. Heavy losses were incurred due to unused capacities and ruinous prices.

Under these circumstances mounting pressure is exerted on the authorities to impose protective measures.

Although a member of a suffering club I am still a supporter of free unrestricted trade. In my opinion we must and do cure our problems ourselves by closing down outdated and streamlining the most modern plants, by developing new fiber types with new desirable properties (which cannot easily be copied) for the more sophisticated European textile and clothing industry which will no doubt survive. In fact, highly sophisticated textiles and garments scored sizable gains on export markets even in such difficult times. Although the immediate future does not look to bright or to use a four letter word still looks rather grim I feel that in the longer run the West European synthetic fiber-, textile- and clothing industry stands a fair chance to recover and to return to reasonable profits.

World-Production of Natural- and Man-made Fibres
(Source: Textile Organon, Vol-XLIX. No. 6, June 1979)

Dr. Pe/Ur

Tabel I

Natural/ Man-made Fibres	Type of Fiber	1970		1974		1975		1976		1977		1978		1978	1970
		1000to	%	1000to	%	1000to	%	1000to	%	1000to	%	1000to	%	%	%
Natural Fibres	Raw Cotton	11 782	54,7	14 020	52,7	11 746	49,7	12 432	48,3	13 804	49,8	13 065	46,9	89,6	87,8
	Raw Wool	1 602	7,4	1 510	5,7	1 508	6,4	1 446	5,6	1 445	5,2	1 470	5,3	10,1	11,9
	Raw Silk	41	0,2	45	0,2	47	0,2	48	0,2	49	0,2	50	0,2	0,3	0,3
	Sub-total	13 425	62,3	15 575	58,6	13 301	56,3	13 926	54,1	15 298	55,2	14 585	52,4	100,0	100,0
Man-made Cellulosic	Rayon & Acetate	3 436	15,9	3 532	13,3	2 959	12,5	3 208	12,5	3 277	11,8	3 315	11,9	100,0	100,0
Synthetic tics	Acrylic	1 003	4,7	1 449	5,4	1 391	5,9	1 742	6,8	1 787	6,5	2 017	7,2	20,3	21,3
	Polyamides	1 903	8,8	2 624	9,9	2 488	10,5	2 851	11,1	2 938	10,6	3 128	11,2	31,4	40,5
	Polyester	1 645	7,6	3 267	12,3	3 367	14,3	3 887	15,1	4 293	15,5	4 686	16,9	47,1	35,0
	Other Synth. exc.Olefins	149	0,7	147	0,5	107	0,5	114	0,4	123	0,4	115	0,4	1,2	3,2
	Sub-total	4 700	21,8	7 487	28,1	7 353	31,2	8 594	33,4	9 141	33,0	9 946	35,7	100,0	100,0
World All Fibres	Total	21 561	100,0	26 594	100,0	23 613	100,0	25 728	100,0	27 716	100,0	27 846	100,0		

Tabel II

Synthetics	Actual World- Prod. 1000 to		Increase %
	1970	1978	
Acrylic	1 003	2 017	101,1
Polyamide	1 903	3 128	64,4
Polyester	1 645	4 686	184,9
Other Synth. exc.Olefins	149	115	22,8
Total	4 700	9 946	111,6

Tabel III

Year	1978	1979	1979 vs. 1978	1980	1980 vs. 1979
Type of Fiber	Actual World- Product. 1000 to	World- Producing Capacity 1000 to	Δ %	World- Producing Capacity 1000 to	Prod.Capacity % Increase
Acrylics	2 017	2 512	24,5	2 656	5,7
Polyamides	3 128	3 987	27,5	4 259	6,8
Polyesters	4 686	5 994	26,4	6 453	8,9
Oth.Synth. exc.Olef.	115	215	87,0	220	2,3
Total	9 946	12 688	27,1	13 587	7,5

July 1979
Dr. Pe/Ur.

Austria-Faserwerke Ges.m.b.H.

World Production of Synthetics by Fiber and Area (1000 Metric Tons)

Tabel IV

Area	1 9 7 0						1 9 7 8					
	Acrylics	%	Polyamides	%	Polyesters	%	Acrylics	%	Polyamides	%	Polyesters	%
West Europe	399	39,8	599	31,5	459	27,9	794	39,4	710	22,7	822	17,5
East Europe	74	7,4	181	9,5	91	5,5	199	9,9	436	13,9	403	8,6
USA	223	22,2	614	32,3	666	40,5	329	16,3	1 157	37,0	1 724	36,8
Other America	19	1,9	117	6,1	75	4,6	96	4,7	207	6,6	332	7,1
Japan	263	26,2	303	15,9	309	18,8	373	18,5	309	9,9	637	13,6
All other	25	2,5	89	4,7	45	2,7	226	11,2	309	9,9	768	16,4
Total	1 003	100,0	1 903	100,0	1 645	100,0	2 017	100,0	3 128	100,0	4 686	100,0

Increase of World Production of Synthetics by Fiber and Area (1000 Metric Tons)

Tabel V

Area	Acrylics				Polyamides				Polyester			
	1970	1978	increase	Δ %	1970	1978	increase	Δ %	1970	1978	increase	Δ %
West Europe	399	794	395	99,0	599	710	111	18,5	459	822	363	79,1
East Europe	74	199	125	168,9	181	436	255	140,9	91	403	312	342,9
USA	223	329	106	47,5	614	1 157	543	88,4	666	1 724	1 058	158,9
Other America	19	96	77	405,3	117	207	90	76,9	75	332	257	342,7
Japan	263	373	110	41,8	303	309	6	2,0	309	637	328	106,2
All other	25	226	201	804,0	89	309	220	247,2	45	768	723	1 606,7
Total	1 003	2 017	1 014	101,1	1 903	3 128	1 225	64,4	1 645	4 686	3 041	184,9

1) Definition

According to the United States Federal Trade Commission Rules . . .
Polyester is the generic term for any fibre in which the fibre-forming
substance is any long chain synthetic polymer composed of at least 85 %
by weight of an ester of dihydric alcohol and terephthalic acid
(p - HOOC - C₆H₄ - COOH).

2) History

It is a remarkable and fascinating fact that new discoveries . . .
have . . . very often far reaching repercussions.

More than 60 years ago Dr. Fritz Klatte of a chemical factory in Frankfurt
which to day belongs to Hoechst AG. exposed vinylacetate to sunlight and
after some time he observed the formation of a resin. This was the very first
artificial conversion of a "monomer" into a "polymer". In 1913 a patent was
granted for this invention.

At that time Dr. Klatte was, however, not aware of the nature of the chemical
reaction which took place.

In 1925 Hermann Staudinger, Professor of Organic Chemistry at Freiburg University,
succeeded in elucidating this reaction. He found that the resin had an exact
multiple of the molecular weight of vinylacetate and he could prove that
numerous molecules of vinylacetate were linked together via C-C bonds to form
a macromolecule, a high polymer chain.

These studies gave rise to new investigations into the structure of genuine
products like rubber, cellulose and proteins. All of them proved to be "high
polymers".

H. Staudinger was belatedly awarded with the Nobel Prize in 1953 for his
fundamental researches.

It is obvious that such new knowledge of the existence of macromolecular
structures induced chemists at universities and in industry to look for ways
how to manufacture new products of this kind.

The area of plastics and synthetic fibres came into sight and took shape.

It turned out that basically three different chemical reactions were suitable to build up macromolecules viz.

1. polymerisation
2. polyaddition and
3. polycondensation

re 1. Polymers in the true sense of the word can only be derived from unsaturated monomers i.e. from compounds with vinyl groups $\text{CH}_2 = \text{CH} - \text{R}$ like vinyl-acetate, vinylchloride, the olefins ethylene, propylene and styrene and last but not least acrylonitrile etc., all of them can be polymerized under specific conditions. But today any macromolecule with repeating units and building blocks respectively linked to a chain, is called a polymer.

About 1930 chemists had learned to control and exploit polymerizations on commercial scale.

re 2. The synthesis of polyurethane is an example for a polyaddition. involving the following essential stages

- a) Preparation of a low linear polymer with terminal hydroxyl groups
- b) The low polymer is reacted with excess of a diisocyanate to give a polyurethane which has terminal isocyanate groups.
- c) Water, in deficiency is added to convert some of the terminal isocyanate groups to amine groups.
- d) The linear polymer is cured by heating. Amine and isocyanate react to give urea cross-linkages. The abundances of cross-linkages result in elastomeric property.

re 3. A condensation is concerned when two chemicals react by separating mainly either water or ammonia, or any other compound.

Hence a polycondensation takes place if a specific condensation can repeat to form a polymer chain.

The very first synthetic fibre was manufactured from polyvinylchloride in Germany about 1931. This fibre had already a high tenacity but only a low melting point of 70°C and otherwise only poor textile properties.

In the 30's, however, the lead in the field of synthetic fibres was taken over by Du Pont. In this company Wallace Hume Carothers investigated numerous "polycondensations" in an endeavour to find new polymers with interesting properties.

In 1935 he eventually enjoyed a big success when he reacted dicarbonic acids with diamines and tried the combination of adipic acid and hexamethylene-diamine resulting in what became known as "polyamide 66"

His observation that this new polymer had fibre forming properties, that the fibre could be considerably stretched where the stretching imparted high tenacity and other related properties, turned out to be of principal importance. We shall see that this principle can be applied to other fiber forming polymers with similar results even if the polymer is entirely different from polyamides from a chemical point of view.

It is worth noting that silk and wool are protein fibres and as such also polyamides. So to speak, Carothers had copied the pattern of nature, as time and again many other chemists had successfully done prior to him.

The new fibre was announced in 1938 by the name of "Nylon" as the first real synthetic fibre having magnificent features in strength, abrasion resistance, elasticity and so on.

But still before commercial scale production was commenced in 1939 which soon amounted to 4 000 tons p.a., Paul Schlack made the next move in Berlin. He had already been concerned with polyamides for some time, when he read the first patent publications by Carothers in summer 1937. He then decided to proceed with his previous studies. Already in January 1938 he succeeded in polymerizing ϵ -caprolactam and to spin the polymer. The second polyamide fibre came into being. It became known as Nylon 6 and Perlon respectively.

I know from witnesses how surprised a group of directors of Du Pont were, when they visited Germany in 1938 and were shown Perlon filament yarns in high quality.

The origin of polyester dates back to early in the 1930's, when W.H. Carothers invented a polyester fibre on an experimental basis. He failed, however, to find a proper combination of a suitable dicarbonic acid and a suitable diol i.e. an alcohol with two hydroxyl groups in one molecule. After his big success with Nylon 66, he concentrated on similar aliphatic dicarbonic acids with which he had made so good experiences in the case of polyamides.

The development of polyester fibres only progressed, however, when aromatic dicarbonic acids and in particular terephthalic acid (TPA) was used to build up polyesters. In the early 40's German and British chemists investigated the performance of TPA with various diols under the conditions of polycondensation. It was the merit of John R. Whinfield and James T. Dickson of the Callico Printers Association in Manchester U.K. to find the most suitable combination amongst innumerable possibilities when they eventually applied the system TPA and ethyleneglycol after a series of painstaking experiments. This special polyester became a similar success as Nylon 66 and Perlon. The original U.K. patent was issued in 1941. World production of polyester fibre has already surpassed that of polyamides since 1972.

As Callico Printers Association was too small a company to develop and exploit "polyethyleneterephthalate fibre" on commercial scale, I.C.I. (U.K.) acquired the license for the production method and the rights of the patented invention in 1947 throughout the world, except the United States, and began to manufacture the fibres under the trademark of Terylene.

In 1953, Du Pont introduced the fibre into the American market, giving it the name of Dacron.

The expiry of the original patents in USA in 1961 and in most European countries 1966/67 has opened the door to numerous companies which previously had not made polyesters.

It was the policy of I.C.I. to sub-license its patent rights and know how - as far as it was developed at that time - to make the greatest possible success of it.

In Germany Hoechst AG. shared a license with Enka Glanzstoff. In 1955 a pilot plant came on stream, whilst commercial production with an initial capacity of 5 000 tons p.a. started only 2 years later. The fibre is marketed under the trade mark of TREVIRA[®]. Today Hoechst ranks amongst the biggest polyester fibre manufacturers in the world and avails of operations not only in Germany but also in the United States, Austria, South Africa, Brazil and Northern Ireland.

3) Raw Materials Intermediates

The major raw materials for polyester fibres are p-xylene, TPA and dimethylterephthalate (DMT) respectively as well as ethyleneglycol.

3.1. p-xylene

This aromatic hydrocarbon has two methyl groups attached to benzene in so called para positions. It is a normal component of coal tar and a by-product of refineries. Particularly for the manufacture of TPA and DMT, today p-xylene is separated from the aromatic fraction accruing after "platreforming" i.e. the formation of aromatic hydrocarbons catalysed by

platinum.

The reformat consists of 25 % by weight of C₈ aromatic hydrocarbons in the following composition.

	<u>%</u>	<u>Bp °C</u>	<u>Fp °C</u>
ethyl-benzene	17 - 20	136,2	- 95,0
para-xylene	16 - 20	138,4	+ 13,3
meta-xylene	35 - 40	139,1	- 47,9
ortho-xylene	19 - 26	144,4	- 25,2

From the aromatic mix benzene (bp 80°C) and toluene (bp 111°C) can easily be separated by distillation.

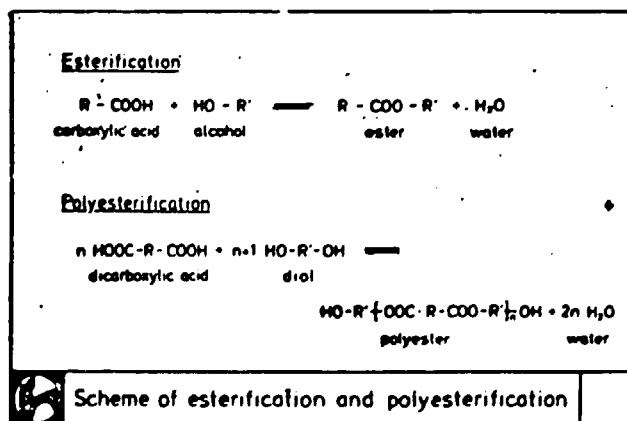
Whilst the boiling points of the a/m C₈ hydrocarbons are very close together, fortunately enough the melting points are quite different. Therefore meta- and para-xylene can best be separated by fractionated crystallisation.

By isomerisation of m-xylene (representing the main portion of the C₈ fraction) the above equilibrium between the four C₈ hydrocarbons is restored. Hence it follows that again 16 - 20 % p-xylene can be obtained from m-xylene (Parex process).

In recent years there was a temporary shortage of p-xylene due to insufficient facilities to separate it from petrol. Left in petrol it improves the Octane count. In the meantime, however, new plants came on stream so that p-xylene has become readily available again.

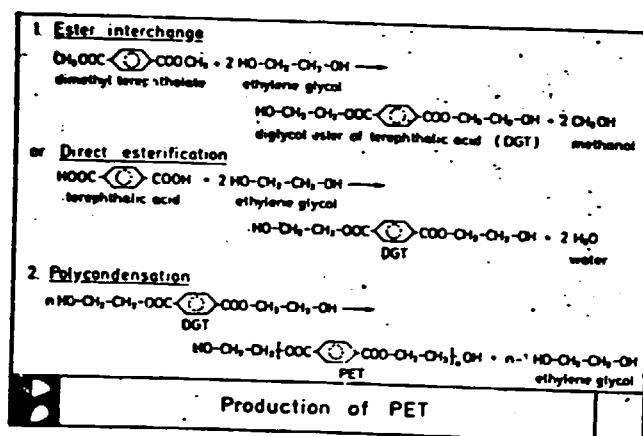
3.2. DMT and TPA

An esterification is defined as a reaction of a carboxylic acid with an alcohol resulting - by separation of water - in an ester (Slide 1)



If a dicarboxylic acid having two carboxylic groups in the same molecule is reacted with an alcohol which also has two hydroxyl groups in the same molecule, it is obvious that the reaction can proceed to form a polyester. In the formula "n" indicates the number of repeating ester units.

As I mentioned already the special polyester Messrs. Whinfield and Dickson found most suitable for the manufacture of fibres, is made from TPA and ethylene-glycol (Slide 2).



The feedstock for TPA is primarily p-xylene $\text{CH}_3 - \text{C}_6\text{H}_4 - \text{CH}_3$ and to a far lesser extent o-xylene and naphthalene (Henkel I process) as well as toluene (Henkel II process). I shall elaborate, however, only on p-xylene which by oxidizing its two methyl groups can be converted to TPA. TPA which is suitable for the manufacture of polyethylene-terephthalate has to meet a very high specification as to purity. It must be "fibre grade". The inventors were faced with the problem, how to purify TPA, how to remove the contaminations, the by-products of the oxidation process. From a chemical point of view TPA is an unpleasant compound because of its properties. It does not boil or melt when heated but decomposes and under normal conditions it is insoluble in common, readily available solvents. In other words the most convenient methods for purification viz. distillation under normal or reduced pressure and re-crystallisation from a solvent were not applicable.

3.2.1. DMT

If an object cannot be achieved on a direct route a by-pass is tried. It is common knowledge of chemistry that many aromatic acids can easily be purified in form of their esters. This method has successfully been applied in the case of TPA. Esterification with methanol gives dimethyl-terephthalate which can easily be distilled under vacuum in the range of 30 to 80 torr and at temperatures from 175 to 202°C. respectively.

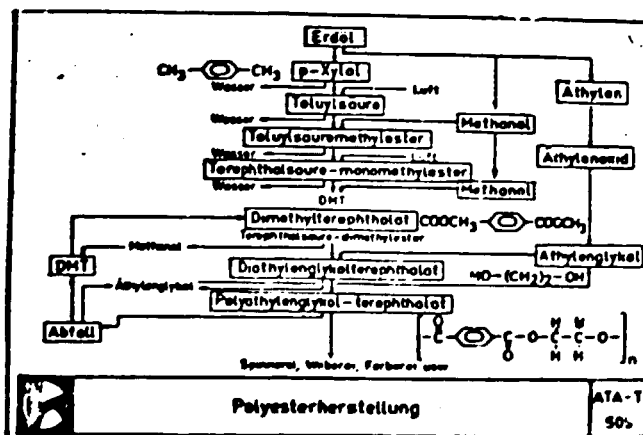
For final purification DMT is recrystallized from methanol.

In the first instance nitric acid under high pressure was used for oxidizing p-xylene by I.C.I. and Du Pont. Also the first commercial plant of Hoechst had been designed for this process. The nitric acid oxidizing method has, however, some shortcomings such as the corrosion of equipment by nitric acid at high temperatures and high pressures, the consumption of a comparatively expensive oxidizing agent and the inclusion of nitrocompounds in the manufactured TPA which is purified via DMT as described above.

Therefore it is not surprising that there was enough incentive to develop a more economic process.

The cheapest conceivable oxidizing agent, of course, is atmospheric oxygen. It was first employed by E. Katschmann of Imhausen at Witten/West Germany in 1951. This method became known as "Witten process" and was first used by Hercules in USA, Imhausen and Hoechst.

It is a continuous production method involving four stages (Slide 3).



The first methyl group of p-xylene can be easily oxidized, but it is difficult to oxidize the second one. The method therefore is intended for changing oxidized carboxylic acid into ester so that the second methyl group may be easily oxidized as well.

The first stage oxidation is performed at a reacting temperature of 130 - 140°C and under a 1 - 5 atmospheric pressure in the presence of a catalyst such as an oil-soluble transition metallic salt like cobalt naphthenate. The conversion rate and yield at the first stage are 40 - 50 % and 85 % respectively. The second stage oxidation is carried out at a temperature of 180 - 210° and under a 1 - 2 atmospheric pressure using the same catalyst. The yield at the second stage is several percent higher than that at the first stage.

A variety of improvements has so far stemmed from this method in an attempt to simplify the four stage processes.

However, the feature of the Witten process is that it only requires p-xylene, methanol for esterification and as solvent for recrystallisation and air i.e. atmospheric oxygen. Methanol, of course, is always recycled. In the past the Witten process was and still is favoured by mayor producers of polyester fibres.

3.2.2. TPA

Nevertheless since many years another process proved to be competitive with Witten DMT. It is the manufacture of highly purified TPA.

As I previously said, TPA is a difficult compound which under "normal conditions" does not melt properly and does not dissolve in common solvents. Not under normal but under extreme conditions e.g. at say $\pm 200^{\circ}\text{C}$ TPA does in fact dissolve in water under approximately 20 atmospheric pressure. On this basis a TPA process was first developed by Scientific Design (S.D.) in 1955. It is intended for obtaining TPA at one-stage, by carrying out a liquid-phase oxidation of p-xylene by atmospheric oxygen in diluted acetic acid as solvent with the assistance of such catalysts as a heavy metal and a bromic compound. This process is now practiced by Amoco and its licensees I.C.I., Aku and Japanese Mitsui Petrochemical Corp.

The method is characterized by oxidizing the difficult to oxidize second methyl group of p-xylene through a proper combination of the action of a heavy metal with the hydrogen drawing action of bromine. P-xylene reacts in diluted acetic acid (2 - 3 times the weight of p-xylene itself) in the presence of cobalt or manganese acetate and ammonium bromide at appr. 200°C and under appr. 20 atmospheric pressure for 30 - 60 minutes with supply of air. Then TPA is produced at a yield of ± 95 %.

The equipment cost of the method is higher than that in the case of the Witten process due to the use of titanium, "Hastelloy" or some other expensive material for the reactor, which must withstand severe reacting conditions.

Final purification is carried out by 3 times recrystallisation from water under pressure at temperatures in the range 130 - 150°C.

3.3. Ethylene glycol

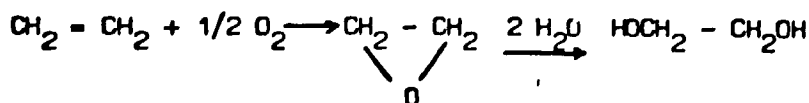
Ethylene glycol which is one of the major raw materials for polyester fibres, is now manufactured on a considerable large scale.

It has wide uses not only for such fibres but also for non-freezing agents, resins and so on. Ethylene glycol is usually manufactured by hydration of ethylene oxide which is in turn, produced by direct oxidizing of ethylene. To a certain extent also the chlorohydrin method may still be in operation, but has widely been replaced by direct oxidation.

In this method the mixture of ethylene and air at a ratio of about 1 : 10 reacts on silver oxide catalyst placed on corundum under normal or reduced pressure. The reacting temperature becomes very high due to the generation of CO₂ and must be carefully controlled by circulating Diphyl or Dowtherm. 60 - 70 % of ethylene can be changed into ethylene oxide at a reacting temperature of 270 - 280°C within one second.

The reaction product is absorbed in water with a pressurized scrubber, separated with a stripper and purified by distillation.

With small amount of acid as catalyst, ethylene oxide can be hydrolyzed give ethylene glycol (EG) at 50 - 70°C.



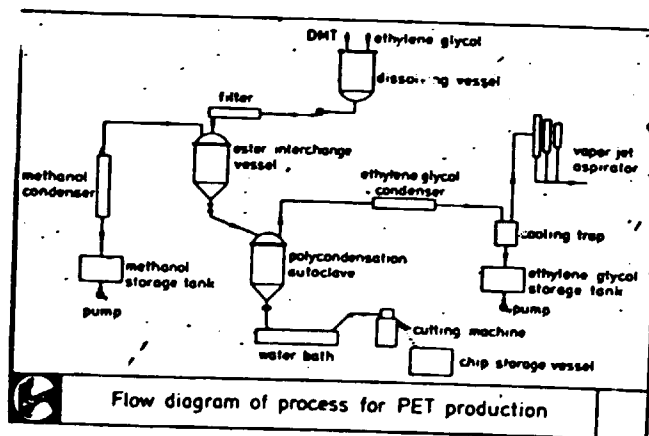
Glycol for polyesters is classified as "fibre grade" which is required to have the highest degree of purity. It is characterized by a negligible content of iron, water and polyglycol as well as by non-coloration when heated.

4) Polymerisation

There are two practical methods for the manufacture of polyethylene-terephthalate (PET), one starts from DMT and EG and the other one from TPA and EG (Slide i).

In both cases diglycol-terephthalate (DGT) which is otherwise known as bis-hydroxyethyl terephthalate (BEHT) in the first instance is formed with the difference that in the case of DMT an ester interchange reaction takes place with separation of methanol and in the case of TPA a direct esterification occurs with separation of water.

4.1. Ester Interchange Reaction (Slide 4)



This reaction between DMT and EG has thus far been used by many manufacturers for synthesizing PET. The reaction between DMT and EG develops uniformly and rapidly in the presence of an adequate catalyst.

Molten DMT and a small excess of EG are agitated and a catalyst is added. The two compounds are then gradually heated from 160 - 220°C until the theoretically expected quantity of methanol is separated and recovered by distillation. The transesterification is completed in 2 - 6 hours, yielding a colorless fluid product. If the reaction is insufficient the two substances won't be polymerized satisfactorily in the second

stage of poly-condensation or DMT will be distilled out of the system together with EG.

Much research has gone into catalyst selection. All types used today give high rates of reactions without sacrificing product quality, notably colour. In transesterification calcium, zinc and manganese acetates are most widely used.

4.2. Direct Esterification Method

Taking the molecular weight of DMT (194) as 100 %, then the molecular weight of TPA (166) is 86 % or 14 % less. These figures reflect the incentive for using TPA, because appr. 14 % less by weight is required for the manufacture of DGT (BEHT) and PET respectively.

The absence of any economical purifying process for infusible and insoluble TPA for many years was the main obstacle to use TPA on large scale. "Fibre grade" TPA i.e. highly purified TPA became a reality only in 1965 once the process of purification by recrystallisation from water under pressure at elevated temperatures had reached maturity.

Direct esterification of TPA with EG is carried out at 200 - 275°C and pressures corresponding to the vapour pressure of EG (2,7 - 5,5 atm). An EG/TPA mole ratio of 2 or a little less is used.

The reaction is usually non-catalytic although the Mobil process is said to use a catalyst.

The main factors which at present restrain polyester producers from switching wholesale to TPA-based polymer are of different nature.

As I said, fibre grade TPA became available only since 1965. This has meant that the lion's share of polymer manufacturing capacity until - say 1970 was designed to use DMT as feedstock. However, there can be little doubt that TPA based processes will gain an increasing share for the manufacture of polyester. To-day approximately 2/3 are still made from DMT and 1/3 from TPA.

For the production of polyester staple fibres nowadays are required :

	Naphtha to/to fibre	Crude oil equivalent to/to fibre
DMT route	1,31	1,74
TPA route	0,96	2,55

4.3. Polycondensation (Homopolymers)

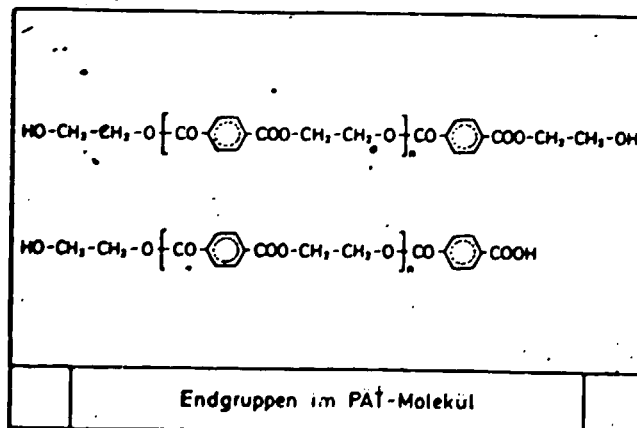
After DGT (BHET) has been manufactured by one way or the other it is passed into the polymerization vessel. Simultaneously with the transfer of the melt, a suspension of TiO_2 is added. TiO_2 is necessary to obtain a delustered fibre. According to the amount introduced, a dull or semi-dull fibre can be produced.

Ultraviolet radiation or "black light", comprising only 5 % of the radiant energy reaching the earth from sun, is responsible for the photo-chemical degradation of many chemicals. This damaging radiation has wavelengths between 290 and 400 millimicrons. When absorbed, this energy is capable of changing the colour, physical form, and reactivity of many synthetic and natural structures. By adding ultraviolet absorbers to a polymer, the stability of the fibre produced from the polymer can be improved.

The inhibitor in the polymer absorbs the ultraviolet rays and, without detriment to the polymer, dissipates the energy.

DGT (BHET) is condensed under heat and vacuum to drive off one mole of EG per mole of DGT. When agitated and heated to $270 - 280^\circ C$ EG initially generates under normal pressure and is removed.

Under gradually increasing vacuum to $100 - 10$ torr and in the presence of a catalyst, the polycondensation develops further and the viscosity of the polymer increases. When finally the pressure is reduced to $10 - 0,1$ torr the required polymerisation degree is reached. The desirable molecular weight of the final polymer for fibre use is in the range $14\ 000 - 20\ 000$ ("n" is $70 - 100$ in the aforementioned equation). When a polymer chain stops growing there are several possibilities for the formation of terminal groups as shown in Slide 5.



For special purposes it is desirable to have a low content of terminal carboxylic groups.

The viscosity of the melt depending on the polymerisation degree can easily be controlled by measuring the power consumption during agitation. The melt viscosity of polyester required for the manufacture of fibres and the molding of films is usually 2 000 - 4 000 poises.

Antimony trioxide Sb_2O_3 has proven to be of particular value as catalyst in the polycondensation reaction. In practice a mixture of an acetate of either Ca, Zn or Mn and Sb_2O_3 is normally added before esterification. It is very important to consider the effect of metal compounds on thermal stability since the catalyst remains in the polymer. Phosphorous compounds are successfully used to improve thermal stability and anti-oxidizing property.

After the molecular weight is attained in accordance with manufacturing formulation pure nitrogen is passed into the vessel to create sufficient pressure to force the polymer through an extrusion nozzle for further cooling and pelletizing.

The melt coming off the nozzle pours over the surface of a band caster in the form of a ribbon which solidifies when conveyed under water. Finally the polymer ribbon enters a dicing machine and is cut into pellets also called chips.

In batch processes as described above polymer chip is first produced. This offers flexibility since it can easily be transported but it needs to be stored and has to be melted before spinning.

Since several years methods for continuous processing of DMT or TPA to polymer and fibre have been developed by a number of companies in several countries. The major producers of polyester fibre today avail of both batch and continuous installations.

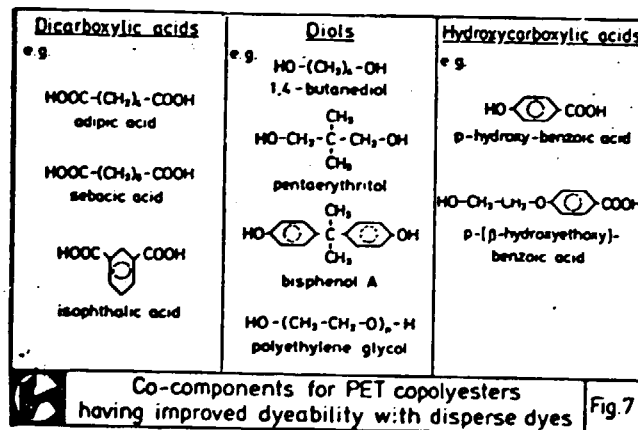
In a complete continuous process molten polymer is passed straight to the spinneret thus cutting out the expensive chip handling facilities. Normally some chip is produced even in continuous processes so as to provide flexibility in product quality.

Choosing between batch and continuous processes depends not only on economics but also on the demands of the market. If filament yarns of various deniers and delusterant levels are required batch production is usually best since chip has to be made anyway. To change deniers the spinning plant must be stopped and the spinnerets changed; to give different delusterant levels additives must be mixed in with the chip. On the other hand, when large quantities of uniform staple fibre, tyre cord or carpet yarn are required the real benefit of a continuous operation with direct spinning can be achieved. In addition, continuous processes offer more uniform product quality and higher molecular weights which in certain cases are of particular importance.

4.4. Co-polyesters

Besides homopolymers, a lot of work has been done on co-polyesters. Such products were originally developed e.g. in an attempt to avoid the basic Whinfield and Dickson patents.

In this context I would like to quote Swiss Inventa's co-polyether ester, made by substituting p-hydroxyl-benzoic acid for about 10 % of TPA (Slide 6).

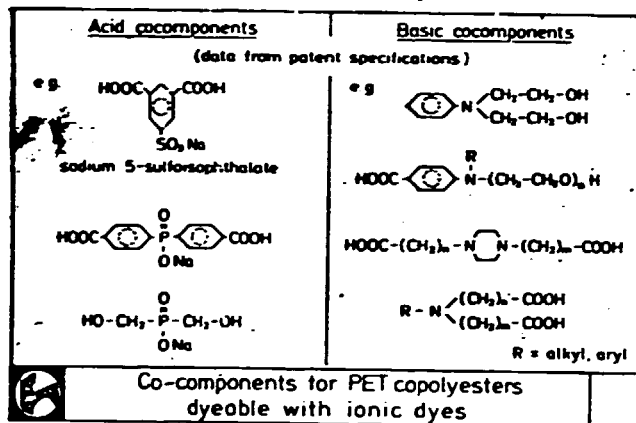


This variety is still produced on a larger scale.

Other incentives for the manufacture of co-polyesters were the general desire to achieve special - or let me put tailor made - properties like improved dyeability, low pilling performance or retarded inflammability.

Slide 6 illustrates co-monomers for the manufacture of co-polyesters characterized by improved dyeability with disperse dyes. The effect of building in such compounds is a certain disintegration of the orientated macromolecular structure thus facilitating the penetration of disperse dyes.

The next slide 7



shows co-monomers yielding co-polyesters dyeable either with cationic (basic) dyes provided excessive acid groups have been incorporated or with anionic (acid) dyes provided basic groups are easily accessible in the co-polyester.

5) Spinning Process

The fibre spinning process is the same whatever the upstream technology is. Except in the case of direct spinning where the molten polyester made in a continuous process is passed straight to the spinnerets, it is essential to dry the polymer before re-melting it.

5.1. Drying of Chips

In the state of equilibrium the water content of PET chips amounts to 0,4 % but is normally less. Anyway, chips containing water suffer an undesirable decrease of their polymerisation degree due to hydrolysis (i.e. the reverse reaction to condensation) at fusing temperature. Therefore chips need to be dried in the spinning area

before going to the fibre forming equipment.

Several drying systems are in use e.g. vacuum drying as well as rotary dryer using counterflow heated air. The chips must be dried to a moisture content of say 0,02 % or less.

When heated the chips pass - the so called glass transition temperature - at a temperature of about 76°C. At this temperature PET somewhat plasticizes because certain segments of the polymer chain get mobile and crystallize. This transition point should be passed rather quickly because otherwise the chips can agglomerate and pack due to their sticky surface.

In vacuum drying the dryer rotates continuously and a liquid heating medium is circulated through an insulated jacket. The chips are heated and the entire batch is uniformly dried. A stationary vacuum line installed through the center of the main shaft extends up into the free air space above the tumbling batch which permits exhausting of the vapor laden air. An air strainer prevents dust from leaving the dryer. As the dryer rotates constant change in location of the chips is assured. Every chip makes positive contact with the heat transfer surface of the dryer. The chips do not pack but take random orientation leaving voids for the escape of vapor. After drying the chips are discharged to the transfer hoppers or to the hoppers of the spinning machine. Vacuum drying, of course, warrants exceptional mild conditions.

Insufficient drying is surely indicated if the degradation of the molecular weight is found too high after spinning.

5.2. Melting, Metering and Filtering PET

The re-melting spinning process of dried chips is most popular due to its simple operation and high productivity. Typical equipment used for the process are :

- grid type
- pressurized melter and
- extruder type

In the first two instances steam or Dowtherm are used for heating the melter and N_2 gas in place of air for transporting the melt to prevent oxidation. The extruder type equipment requires power as direct source for melt heat supply.

The molten polymer delivered from the melting device is measured with a metering pump and transmitted to each spinneret in an equal quantity. A gear pump is preferred for this purpose.

When a melting device of the grid type is used a booster pump is sometimes installed in front of the metering pump. This not only improves the measuring accuracy but also increases the polymer transmitting capacity with the pressure of N_2 gas. The metering pump requires strict testing with highly viscous oil under pressure before installation.

The extruder possesses an obvious but important characteristic in that it melts polymer only as fast as required; that is, only as much polymer is melted as is needed to supply the metering pumps. The pressure and temperature are controlled precisely. Extruders (as well as other types of fibre forming equipment) are fabricated to high precision standards of an alloy of high hardness and wearing qualities and they retain these qualities at high temperatures. The feed zone of a spinning extruder is water cooled. The other cylinder zones are electrically heated. For the best homogenizing of the melt and better yarn quality the front section of the extruder screw is designed as a mixing torpedo.

Molten polymer is fed to the metering/spinning pumps through ducts extending from the extruder. Before transmitted to the spinnerets the melt needs to be filtered for removal of solid impurities in order to protect the tiny capillary exit holes of the spinneret. Both the filter and the spinneret are incorporated into a single cylindrical body called the spinpack which is replaceable whenever the spinning process is found unsatisfactory or at regular intervals.

The coordination for the operation of the spinning extruder and spinning head/spinpack requires special instrumentation for temperature and pressure control. A pressure transducer especially designed for hot thermoplastics senses a controller which regulates the revolutions of the screw thus fulfilling any requirement of the pressure control

and allows constant flow of the melt to the spinning pump. At every spinning position the supply of melt may be cut-off by stopping the respective metering pump. This allows the changing of a spinpack without the interruption of other positions. When a spinning pump is stopped the revolution of the extruder screw is automatically regulated by the pressure control that reduces the speed. The extruder will pick up speed as soon as the pump is put in operation again.

The changing of a spinpack takes only a few minutes.

As I said the filtering of the melt is of great importance. A spinpack consists of screens (fine-mesh wire nets), layers of well-selected sand, a distribution plate and a spinneret. Several packs such as assembled ready for use, are held in place in Dowtherm boxes and pre-heated to facilitate rapid pack changes. Besides filtering any foreign particles from the polymer, the pack is also designed to shear the polymer in order to reduce agglomerates such as gel particles to a smaller size. A distribution plate placed above the spinneret serves to spread the melt evenly over the spinneret face and improves interfilament denier uniformity.

5.3. Forming Filament and Quenching

Spinnerets are metal discs with mostly numerous small holes. They vary from less than 100 as used for the manufacture of continuous filament yarns to several hundreds designed for staple fibre production.

Although most spinnerets have holes with a circular cross-section also other patterns as trilobal and pentalobal cross-sections have been developed to serve special purposes. It is evident that a different surface of a yarn affects properties like handle and bulk.

The filament diameter is not normally the same as that in the hole. Immediately upon being extruded and in the moment the polymer emerges from the spinneret capillary hole the thread just formed bulges to a diameter considerably larger than that one of the hole. Thereafter, when still in molten state the diameter of the filament decreases rapidly. This denier reduction is brought about by the pulling action

of the feed rolls of the take-up/winding device by means of which the still thermoplastic filaments are drawn and elongated. The filaments solidify by the time they travel approximately halfway down a chimney called spinning shaft.

Cooling and solidifying of the molten filaments is accomplished by cross-current flow of air. Air is supplied from a compressor via an air quenching system involving a screen assembly before passing across the filaments. The assembly is required to ensure that air is evenly distributed over the cooling area.

I wish to emphasize that the state of the filaments before solidifying is a very critical one. It endangers that one or two or even more of the numerous filaments emerging from the spinneret with very little distance to each other amalgamate and/or break. To avoid such occurrences which give a lot of trouble the proper functioning of the quenching system must always be given special attention.

- 5.4. Application of Finish and Take-off. After travelling through the spinning shaft the next operation is the application of a finish. Finish solution is usually an emulsion of oils and other components. The oil is intended for lubrication. Another component serves to reduce the surface tension which affects the finish pick-up of the filaments as they move over the finish rolls. These rolls in turn rotate in finish supply pans. One of the most important functions of the finish application is to reduce the electrostatic charge which builds up on the filaments as they pass over guide surfaces.

So far the technology applied for the manufacture of filament yarns and staple fibre is basically the same. Of course, there are important differences in the selection of spinnerets, in the size of metering gear pumps and other equipment. The ramification as far as principal technology is concerned, however, begins with different methods of further processing filaments designed for staple fibre and those for filament yarns. The first difference being that in the case of filament yarns the number of filaments emerging from one single spinneret are wound upon a bobbin and from then onwards are treated individually whilst in the case of staple fibre the total number of

filaments extruded from all spinning positions of a spinning machine are taken-off together.

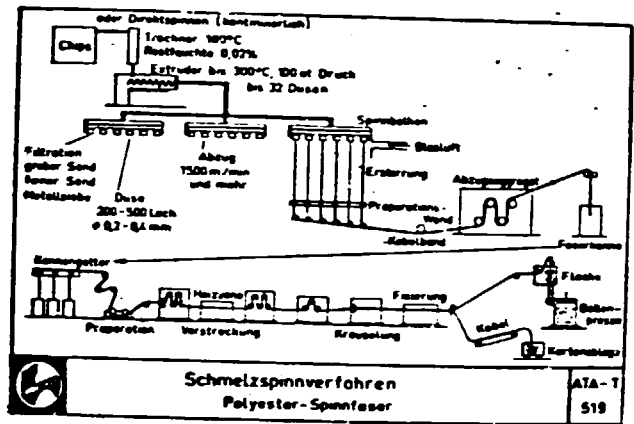
5.4.1. Take-off in case of Staple Fibre

This method shall be dealt with first. As I said all filaments spun from one machine comprising 10, 20, 30 or even more spinnerets of e.g. 500 holes each are allowed to converge over guides thus forming a spin tow. A set of godet rolls with high revolving speed delivers the spin tow to a can loader from which the tow is discharged into spincans rotating to ensure even loading.

At this point I feel I must make a supplementary remark as to the final denier which is desired. It results from the feeding rate of the metering pump and the take-off speed of the set of godets taking into account a certain slip on the godets and the draft of the molten filaments before solidifying. To give you a rough figure the spinning speed ranges from 500 to 2 000 m/min. Fine deniers are obtained with low feeding rates and high take-off speeds. The finest denier of staple fibre we supply amounts to 1,2 den i.e. that 9 000 m of a single filament weighs 1,2 g. In recent years the gauge has been changed to the decimal system. Dtex is defined as the weight in g of 10 000 m filament. 1,2 den therefore equals 1,3 dtex.

It is evident that a complex spinning unit as described above cannot be operated manually. It requires full integration of all machinery and equipment involved. The coordination of the various steps of the process can only be attained by highly sensitive instrumentation which is installed in a panel and designed for widely automatic control. This panel is the heart of the whole set-up. All important data are continuously recorded. An alarming system ensures that any significant deviation from the manufacturing formulation is immediately indicated so that the responsible person can step in without delay.

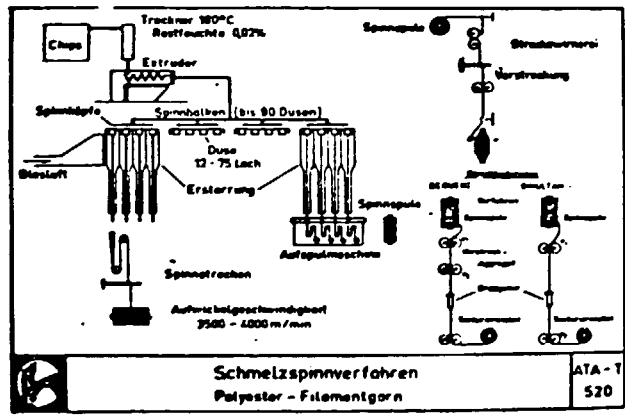
Slide 8

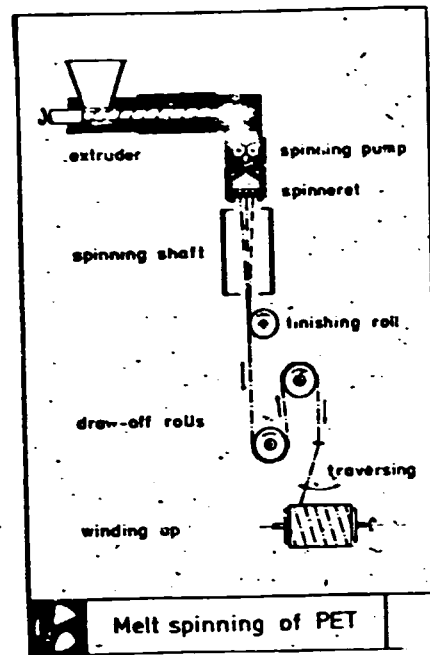


shows a flow sheet of a spinning plant. It summarizes in a condensed manner my preceding explanation on spinning PET staple fibre.

5.4.2. Winding up Filament Yarn

To demonstrate the main difference between staple fibre and filament yarn technology I wish to refer to Slides 9 and 10





From the finish roll the final number of filaments (multifilaments) are taken-up by two godet rolls intended as feed rolls. The lower feed roll is the first point at which the multifilament yarn is pulled or a controlled linear velocity applied. The force applied on the yarn at this point serves to pull the thermoplastic polymer from the spinneret. The peripheral speed of the feed wheel is one of the two factors involved in controlling the spun denier. The other factor - c_w in the case of staple fibre - is the throughput of polymer in the fibre forming unit.

The upper feed roll of the take-up unit is the same as the lower feed roll, however, the diameter of this roll is slightly greater than that of the lower wheel so that there is a slight tension on the yarn between the rolls.

Guide pins directly over the traverse position the yarn before it is wound upon the bobbins. The traversing of the yarn provides an even cake and stability to the package.

Diameter and speed of the take-up is precision controlled as the speed affects the spun denier and also affects the orientation of the macromolecules in the yarn. Tandem wind-up is normally used.

In wind-up the tension change on the yarn must be held to the minimum of turn-around points of traverse.

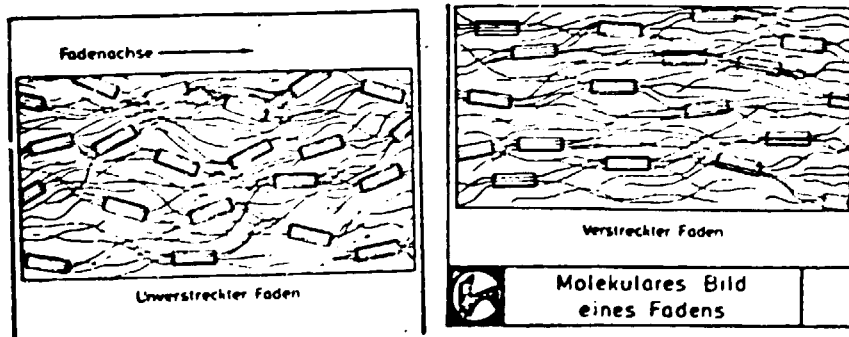
Wind-up chucks for take-off can be parallel or at right angles to the machine. When a surface drive is used the chuck pressure must change with package build-up. The chuck must take and hold securely the wind-up tube.

6) Staple Fibre/Stretch-Line Process

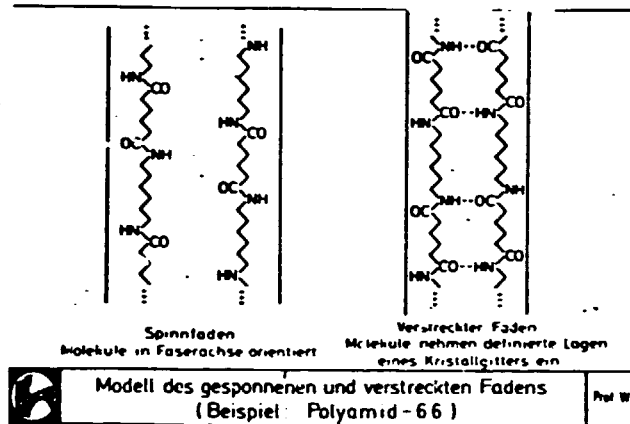
The final product of the spinning process is either spin tow collected in spin cans or spun multifilament yarn on bobbins.

For obvious reasons the so far continuous operation must be interrupted at this stage. It is followed by another process which is normally also a continuous one of not less importance. As I have initially remarked already the fundamental discovery of W.H. Carothers was his observation that fibre forming polymers can be considerably stretched or drawn. Melt-spun undrawn yarns have a 400 - 500 % elongation. As their physical properties are unstable they cannot be practically used. They receive their valuable physical properties, namely high tenacity and elasticity and less elongation when the yarn is under load only from drawing. When the melt-spun yarn is drawn beyond its elastic limits the filaments are reduced in size to a fraction of their spun diameter. In practice they are drawn 3 - 4 times their length which imparts a tenacity in the brackets of 40 - 60 pond/tex and an elongation of roughly 20 - 40 %. There is a change in the cross-sectional area of the filament and also a change in the molecular and crystalline structure. The filament structure takes a highly ordered and orientated arrangement in the direction of the fibre axis.

Slide 11 shows schematically the different structures of an undrawn and drawn filament.



Slide 12 illustrates the effect of drawing from a chemical point of view.



As I could not find a slide related to polyester the model of polyamide must do, but it reflects the feature as well. On the left hand side undrawn macromolecules are positioned at random and only pre-orientated in itself along the fibre axis. On the right hand side two macromolecules are shown when drawn. They have established a more regular and stable structure which can be compared with a crystal grating. The arrangement is characterized by the formation of intermolecular hydrogen bridges between nitrogen and oxygen. Such hydrogen bridges considerably contribute to the stability of the arrangement.

Now I wish to give you a little demonstration on the drawing of a spin tow. This, however, must be done in an economic way.

What I have called stretchline process comprises six essential steps :

1. Finishing
2. Drawing
3. Crimping
4. Heat-setting
5. Cutting) alternatively loading "converter tow" into cartons
6. Baling)

Whilst the spinning unit runs from top to bottom and its machinery is mounted on several floors accordingly the equipment of a stretchline extends in a horizontal direction.

Many spin cans loaded with spin tow are positioned in front of the stretch line. With the aid of a creel equipped with thread guides the spin tows are pulled out of the cans and aligned into a broad tow. The spin tows do not tangle provided the cans have been evenly loaded. Before the broad tow is transmitted to the drawing device it is dipped in a finish bath to pick-up fibre finish for the following reasons :

1. To make the filaments easier to draw as it gives the filaments a plasticizing effect.
2. To eliminate static charges for easy processing from drawing to cutting and last but not least
3. To make them spin properly during textile spinning processes.

I wish to stress the importance of item 3. Satisfactory performance of synthetic fibres in textile mills is to a great extent dependent on the application of an adequate fibre finish. I do not exaggerate when stating that fibre finish must meet highly sophisticated requirements. For that reason tailormade finish formulations had to be developed for any of the various polyester fibre types to ensure troublefree spinnability on their respective textile machinery.

From the finish bath the tow is delivered to the drawing device which consists of two three to seven-roller draw stands of heavy construction. Between these two sets of godets revolving with different speed the tow is drawn accordingly. It is obvious that the draft ratio corresponds with the speed ratio of the two set of godets. It normally ranges from approximately 1 : 3 to 1 : 4. Contrary to polyamides polyester fibres are usually drawn at elevated temperatures. The heat may be supplied by means of power or steam. It is advisable to have the first roll of the first set of godets equipped with a pneumatically operated niproll to squeeze abundant fibre finish.

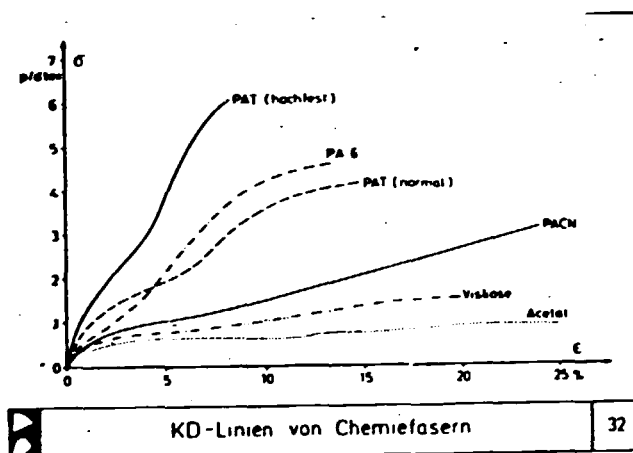
From the second set of godets the tow is guided to a third one. The speed of the third set of godets may be adjusted to allow for shrinkage of the tow or can be so arranged as to give a two-zone draft ratio which may be preferred in certain cases.

The third set of godets also serves to feed the crimping machine.

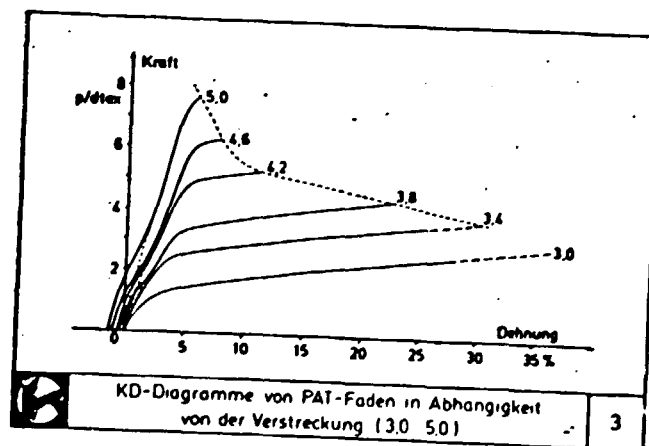
The special conditions of drawing - with due regard to melt-spinning conditions - in conjunction with heat setting which may be performed with or with no tension, offer a wide variety of possibilities to manufacture fibres with quite different properties. It is evident that the physical properties of the undrawn yarn in particular the degree of pre-orientation reflected in spin shrinkage must be given due regard when the conditions for drawing are formulated. E.g. higher degrees of pre-orientation require less drawing.

In other words important properties as tenacity, elongation, elasticity, shrink performance and dye affinity are affected and can be varied by applying different drawing and setting formulations.

Slide 13 illustrates stress-strain curves of typical man-made fibres. Tenacity is plotted against elongation.



Slide 14 shall serve to demonstrate the strong effect of different draft ratios on tenacity and elongation detected on polyester filament yarns.



I have mentioned the dimension of tenacity in terms of gram or pound per denier/detex. More precisely the load in grams required to break yarn is referred to as the breaking strength of the yarn. The tenacity of the yarn (or fibre respectively) is calculated by dividing the observed breaking strength by the average denier of the yarn.

Yarn elongation is the percent increase in length before the yarn breaks.

Highly sensitive (and expensive) instruments have been developed to measure and record stress-strain curves.

From such curves experts can conclude interesting details of the manufacturing process.

The next step on the stretch-line is crimping which, in fact, means folding. Before entering the crimping machine the tow needs to be slenderized and thus to be adjusted to the width of the crimper. With the aid of two rollers which rotate one upon the other under pressure and with equal speed the tow is pulled into the stuffer box. The pressure of the rollers is required to push the tow against an obstruction, namely against the fibre which is already in the stuffer box and to avoid any escape in the opposite direction. On the movable bottom of the box a controlled pneumatic pressure is exerted and transferred to the tow. In a series of consecutive cycles and by the joint action of forward pushing and upward pressing the fibre is crimped.

When a small section of tow enters the stuffer box the fibre is stiff and moves the obstruction until the slenderness ratio becomes critical. Then the fibre starts to buckle and the thrust it exerts against the obstruction is reduced therefore the obstruction instantly stops moving. In the next moment the fibre has buckled completely in the sense that it will sustain an end load great enough to move the obstruction. At this point half a crimp has been formed. Now the moving fibre again becomes a stiff column and pushes the obstruction back until the slenderness ratio becomes critical. The fibre begins to buckle but in the opposite direction due to the binding stress at the junction of the first half of the crimp. The pressure on the obstruction is again released for a short moment until the buckling is completed to form the second half of the crimp or one full crimp. At this point the cycle begins to repeat.

Crimping is necessary to impart bulk and handle and to make the fibre spinnable. Remember that all natural fibres are crimped too.

After crimping, the tow may be or may not be heat set. In the first case the fibre is allowed to freely shrink as it is under no tension. The percentage of shrink, of course, depends on the temperature to which the fibre is exposed.

At this point there is a ramification as the fibre can either be cut to make staple fibre of a definite staple length or it can be discharged into cartons in form of "converter tow".

Staple fibre is supplied to cotton mills and converter tow to worsted mills.

In the case of staple fibre the tow is transmitted to a cutting machine. The mode of action of such machine normally used can briefly be described in the following way :

There are two slot-wheels with vertical revolutions which touch under pressure at a clamp point. Between the slot wheels a set of extreme sharp knives mostly three or six ones rotate in a horizontal movement. The fibre tow charged to the cutting machine is clamped between the two revolving slot wheels and in the next moment is cut by the revolving knives.

Of course, the speed of the revolving knives must be carefully adjusted to the speed of the revolving slot wheels.

The staple length depends on the distance of the slots and the number of knives employed. Using a specific set of slot wheels designed to yield 1 1/2" staple length, it is possible to make 3" fibre by employing only half the number of knives. In this event the knives omit one slot and hit the tow in each second slot only.

After cutting the staple fibre is blown into the bale press for final baling.

In the case of manufacturing converter tow the tow is packed into cartons by means of an automatic traverse travel-and doff system.

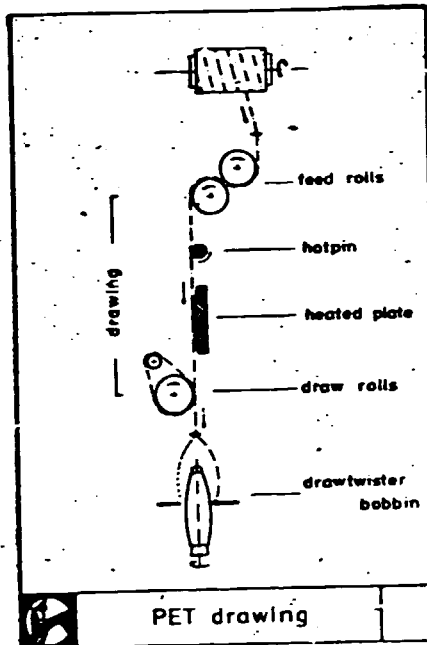
7) Multifilaments/Drawtwisting

In the past spinning and drawing^{of} filament yarns has been usually done in two different steps.

We have seen that the final product of the spinning process is undrawn filament yarn wound upon bobbins. In principle drawing^{of} melt spun filament yarns is performed in a similar way as in the case of staple fibre and for the same

reason. As each bobbin has to be treated separately an economic machine must be able to cope with numerous bobbins placed side by side simultaneously.

Slide 15 shows one position of a draw twisting machine.



Drawing takes place by passing the yarn from the bobbin over feed rolls and drawrolls. The drawing is released on the yield point that is the point where the filaments start to be elongated. This yield point is localized on heating equipment, namely hot pin and hot plate. Remember that heating is required to draw polyester filaments. Otherwise drawtwisting consists of drawing the yarn several times its original length imparting a slight twist to the yarn and winding the yarn into a package for shipping. A small amount of twist is added to the yarn to hold the filaments together. This is imparted due to the difference in drawing speed and wind-up speed of the drawtwister.

The yarn, after being drawn is partially retracted or recovers from the drawing imparted. Some of the retraction takes place between the drawroll and the bobbin. This action decreases the effective draw ratio. Further retraction takes place upon the bobbin or cop after the yarn has been wound. This action imparts a crushing force of great magnitude upon the bobbin case. The bobbin must be substantially constructed, to withstand this force.

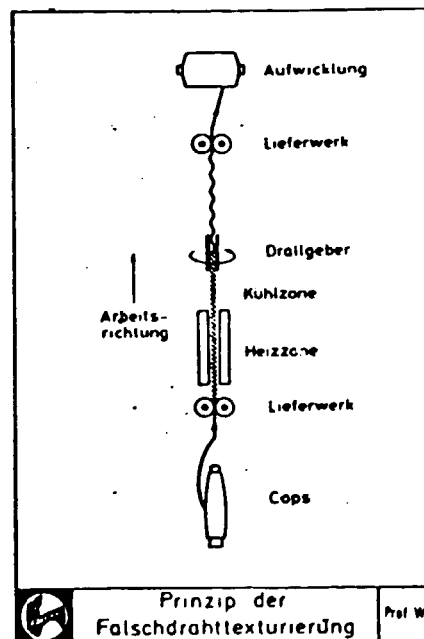
Drawtwisting requires air conditioning as temperature and humidity have an important effect upon the performance of the yarn.

The two step method for separate spinning and drawtwisting as described has been the standard operation for many years. There are still numerous installations in the world employing this method.

However, technology is always progressing. In recent years by close co-operation between textile machinery industry and synthetic fibre manufacturers a more modern machine has been developed providing new technique which permits the two operations to be combined on one machine and in one operation. The process involved is called draw- or stretch spinning. It is evident that direct stretching at already high spinning speeds requires extreme high take-up or wind-up speeds.

8) Texturizing

Polyamide and polyester multifilaments enjoyed a great boost with the introduction of texturizing techniques. For broader application multifilaments had one important shortcoming namely the lack of crimp and related properties as bulk and handle. This shortcoming, however, could be eliminated by texturizing the yarns. This process is schematically illustrated in the last slide 16.



The method is briefly characterized by high twisting a drawtwist yarn at elevated temperatures. Subsequently the yarn passes a cooling zone and is rewound in the opposite (false) direction, hence the name false twist texturizing. This method imparts a considerable bulk and makes the yarn

suitable for many more applications. TREVIRA[®] 2000 and Crimplene are well known trade marks for garments made from such texturized yarns.

The latest development in this field endeavours to incorporate the texturizing process into stretch spinning as well.

9) Product control

I cannot conclude my report without stressing the importance of product control. It cannot surprise that products manufactured by applying highly sophisticated technologies require permanent control to ensure high standard quality at all times.

The responsibility for final release for shipment is vested in the textile laboratory and in the chemical laboratory. A considerable number of specifications has to be met before the fibre is released for sale.

At regular intervals samples are taken from all important steps of the process and passed on to the lab's which undertake a series of tests starting from moisture content of undried and dried polymer, *molecular weight control at various stages of the process and extending to final properties of fibre and yarn comprising control of denier, staple length, tenacity, elongation, crimp, dye affinity and many more. The responsible persons are immediately advised if the limits of any specification are surpassed so that instant action can be taken.

In concluding I hope that I was able to give you a fair and comprehensive report on what I promised in the heading :

"Survey on polyester fibres, their chemistry and their technology."

I have tried to touch any aspect of significance but should I have omitted an important item, please do not hesitate to ask questions.

* Measuring the velocity of flow of dissolved yarn through a viscosimeter