9 Testing and Influencing the Properties of Man-Made Fibers

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9.1 Introduction to Testing

9.1.1 Aims and Tasks

The testing of chemical fibers is a necessary and integral part of the production process. It is multifaceted, can be carried out at various stages in the production process and can be viewed from different aspects with the following emphasis [1].

- Quality control and quality assurance
- Process optimization during fiber production and further processing
- Product development of fibers and yarns
- Input and output controls for monitoring specifications
- Failure analysis in each stage of production
- Investigation into how textile articles behave in use.

Figure 9.1 illustrates the fundamental aspects of measurement and testing technology required for product quality assurance. In addition to the time-proven investigative methods, computer-driven measuring processes are increasingly being used. These are sometimes fully-integrated systems (or lines) which automatically carry out testing and evaluation.

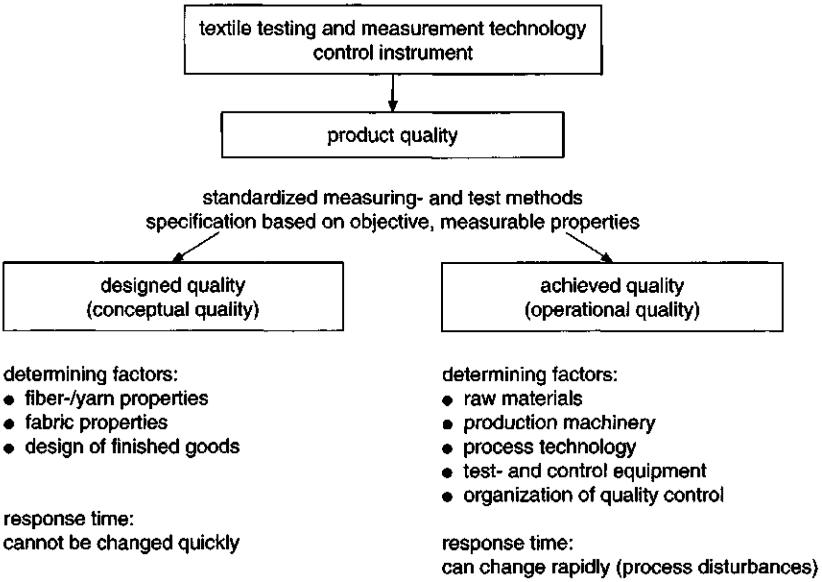


Figure 9.1 Textile testing- and measurement technology as control instrument

As apparel, textiles, carpets and other articles are also judged by their outward appearance, visual methods and sensor-based measurements (such as dye uptake uniformity, pilling, etc.) are still used in addition to textile testing of fibers and yarns. Statistically-evaluated wearer or application trials are still required for performance-in-use evaluation, as, e.g., for fiber development. Similar testing is necessary in the case of new fibers or new types of fibers and for their conformity with design quality.

There is an increasing tendency to move away from the chemical and physical testing of fibers in monitoring producer quality, and to move towards control of process variables on the machine itself:

Process control	replaces	Product control
Process parameters		Fiber/yarn properties
e.g., temperature, pressure, speed, etc.		e.g., tenacity, elongation

This requires a re-orientation in thinking, from product control to the individual process parameters: the more reproducible a production process is, the less need there is for subsequent random samples for chemical or physical testing in the laboratory. Taken to extremes, an absolutely reliable reproduction of process parameters eliminates the need for subsequent product testing in the laboratory, except for a few random samples. Figure 9.2 compares the methodology of process control (left) and product control (right).

- An example of the above is given in Fig. 9.3: The external yarn regularity (Uster CV%) and the elongation at break depend on the quench air flow rate as parameter [6]. For the radial quench chamber, both the lowest Uster value and the lowest elongation occur at 40 Nm^3/h. This process parameter should therefore be held constant within a technically-achievable range. In order to be able to predict the properties correctly, regular confirmatory tests must confirm the relationship established between the parameter and the properties.
- the predictive quality assurance connects:
  cause ⟶ effect
  process ⟶ fiber properties

In this way, the yarn properties prescribe the allowable deviations in the machine set points.

In the case of backward-looking quality control (= product control), the relationship is reversed. During further-processing (such as weaving, knitting, washing, dyeing, thermofixing, etc.), the process variables: tension, temperature and swelling have the greatest effect on the fiber and its further products, which is why a knowledge of the appropriate product properties is important.

When signing purchase contracts for staple fiber and filament yarn, it is usual to specify both the fiber properties and their regularity. Despite the use of predictive quality assurance, it is still necessary to test the sales product for conformity to type. This should not, according to the above described process control system, be necessary, but the further-user (buyer) has no means of evaluating the product other than measuring its properties and their ranges.

9.1.2 Fundamental Principles of Textile Testing

For the fiber producer, textile testing is many-faceted and complicated, since the test must encompass the raw yarn, the fabric and the finished goods. Industrial textiles require many additional, special tests, sometimes requiring other test equipment.

- Laboratory equipment: If the emphasis is on production quality control, not much equipment is needed, but the degree of automation is high and computerized evaluation is required. Quality assurance for customers, for special-application products or for general research, conversely, requires a variety of test equipment specific to cover these diverse needs. Here the test methods could be simple and practical, or they could be complicated research-related analytical methods.

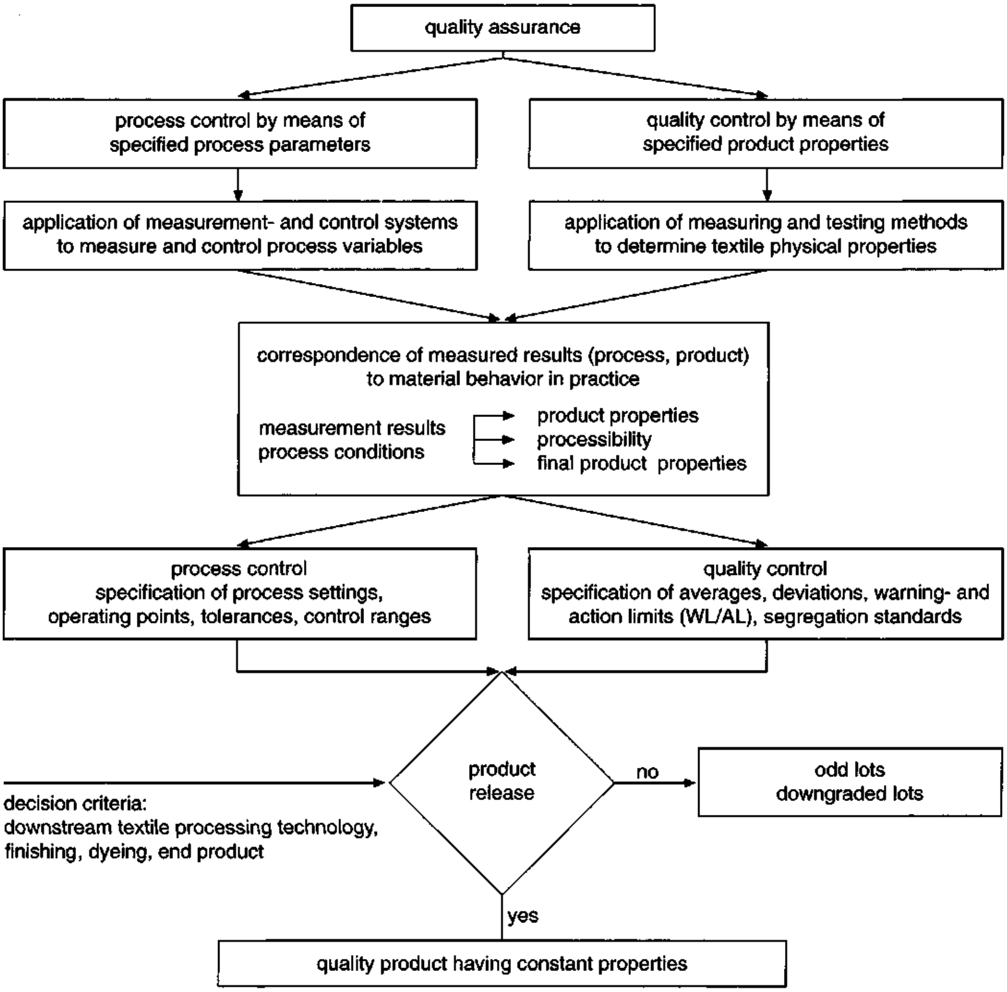


Figure 9.2 Quality assurance by means of process control and quality control

- In order to obtain comparable and reproducible results, it is necessary to use normed test methods having specified conditions and evaluation procedures. Users in different locations at different times must be able to measure given properties of a fiber or a yarn within agreed accuracy at reasonable cost. As an example, Table 9.1 gives test conditions for assessing the performance-in-use of textiles, and points out the consequences of not meeting these requirements.

  Normed methods for testing the properties of man-made fibers and their performance-in-use are provided by various organizations:

  - international norms and rules:

    ISO – International Standardisation Organisation
    CEN – Commission Européenne de Normalisation
    BISFA – Bureau International pour Standardisation des Fibres Artificielles
    IWTO – International Wool Textile Organisation

  - national standards organizations

    ASTM – American Society for Testing and Materials
    AATCC – with emphasis on performance-in-use: American Association of Textile Chemists & Colorists
    DIN – Deutsches Institut für Normung e.V.
    BSI – British Standard Institution (in United Kingdom)
    AFNOR – Association Française de Normalisation (in France)
    SVTM – Schweizerischer Verband für Technik und Materialprüfung

    When standardized test norms are not available, it is advisable to use methods provided by fiber producers, test equipment manufacturers or research institutes [25].

- Testing climate: To eliminate the (sometimes strong) influence of testing climate on laboratory results, climatic conditions are standardized:
  - standard climate (20 ± 2) °C, (65 ± 2)% relative humidity (RH%) (Fig. 9.3, Table 9.1)
  - special conditions for the tropics: (27 ± 2) °C, (65 ± 2)% RH
  - special conditions for testing electrical equipment: (23 ± 2) °C, (50 ± 2)% RH

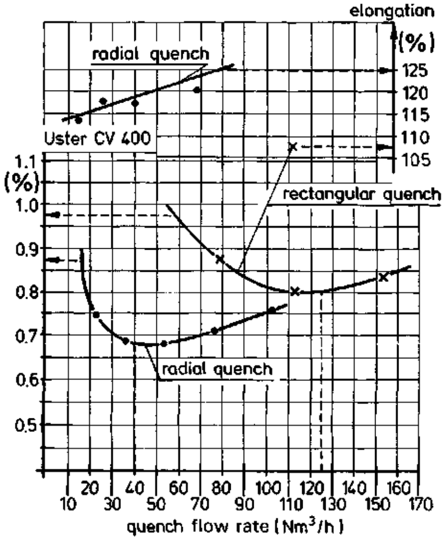


Figure 9.3
Example: Uster C.V. value and elongation as a function of quench chamber conditions (polyester POY, 76 dtex f36, 3200 m/min)

Table 9.1 Assesment of Performance-In-Use

Requirements	Consequences of not meeting requirements
1. Strong correlation between measurements and actual performance-in-use	Incorrect assessment of performance-in-use
2. Good reproducibility and repeatability	No inter-laboratory comparison is possible Test method is not valid for terms of delivery
3. Minimum test effort 3.1 Short testing time 3.2 Minimum of test equipment	Not usable for routine testing because of long testing time needed Ruled out for routine testing on grounds of material usage, costs and time expenditure

The atmospheric pressure should be between 800 and 1060 mbar [6].

- In order to rule out swelling hysteresis, samples should reach equilibrium moisture content by absorption during conditioning. Should the fiber be too moist, it should be somewhat more strongly predried so that it reaches the test condition by absorption in the standard atmosphere [5].
- Sampling and evaluation of results: As textiles are non-homogeneous, the sample taken must be made to be representative of the properties of the lot. To this end, statistical methods must be used to ensure that the property to be measured is accurately estimated (Section 10.10).

  The same reasoning applies to the evaluation of results. Certain characteristics of textiles can only be assessed by using ordinal scales (e.g., gray scale for dye stripiness)
  Appropriate evaluation methods are given by ISO and ASTM [7].
- Measurement units: In 1970, the "Système International d'Unités" (SI unit system) was adopted for use in textile testing on the recommendations of "ISO 1000" and "DIN 1301". Use of the older textile units (such as, e.g., "denier" or "metric number", etc.) has not to date been completely eliminated, and they are still used as standards in certain countries. Conversion factors are therefore given in Section 10.2.

9.1.3 Quality Systems

The fundamental objective of quality systems is to provide quality assurance which ensures failure-free production without interruption to the production flow of goods. The man-made fibers- and textile industry therefore adopted quality systems of high process standard having well-developed instrumentation from other branches of industry, and put them to use in quality control to ensure high product quality. The present-day demands of product liability reinforce this trend, right up to the end-user. This area is covered by DIN/ISO 9000–9004 norms.

ISO 9004 describes which parts of the manufacturing firm are to be incorporated into the quality loop, and lays down the responsibility, from management to product assurance and liability (Fig. 9.4).

For the above reasons, the application of the optimal technology in combination with an appropriate process control system in production is a requirement for mastery of the production process and thus constant product quality. To this end application of textile testing and measurement technology enables proof and assurance of acceptable product quality.

9.2 Terminology and Morphology

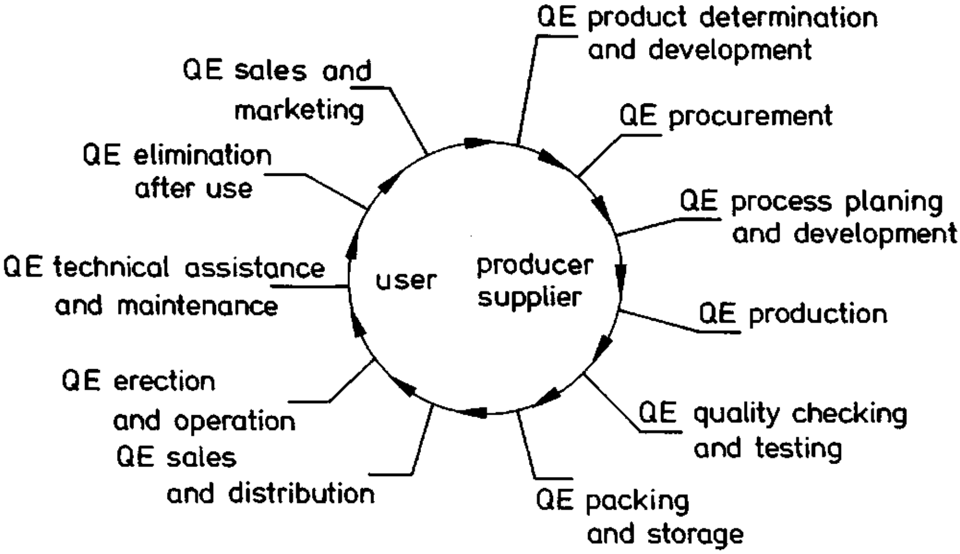
9.2.1 Man-Made Fiber Terminology

Alongside classification of the man-made fibers into various species, the exact specification of concepts and practical definitions is a necessary requirement for common communication. In addition to the abbreviations for chemical fiber classes (Section 10.5), there are also classes according to the spinning speed ranges (Table 10.4), the titer ranges (Fig. 10.3), as well as for tenacity, modulus, staple cut lengths, etc. These are partly laid down in norms, but also include frequently-used terms which have crept into usage through repetition. As most polymers are also used in the plastics industry, a comparison of DIN and BISFA abbreviations is given in Table 10.5.

Figure 9.5 shows man-made fiber classifications according to DIN 60001, part 3, ISO 2076 and BISFA recommendations. These classifications are, however, continuously supplemented and modified in the light of new developments (Fig. 9.4).

The fundamental classification is by polymer type [5]:

- If a polymer or copolymer contains more than 85% w/w of a component, that component determines the polymer type.
- Should each and every component constitute less than 85% w/w, a new category can be formulated or the fiber can be described as a mixture: e.g., *x*% polymer *A*/ *y*% polymer B.



QS (Quality Assurance) Elements

Management duties	
Staff (training)	
Quality assurance system	
Statistical evaluation	
Documentation	
Identification and traceability	
Quality testing	
Test equipment monitoring	
Testing conditions	
Handling faulty units	
Dealing with products (product knowledge)	
Quality recording	DIN/ISO 9003
Sales and marketing (Contract audit)	
Acquisition/auxiliary products	
Production	
Corrective actions	
Internal quality audit	DIN/ISO 9002
Customer service	
Research and development	DIN/ISO 9001
Economic considerations	
Product safety and product liability	DIN/ISO 9004

Figure 9.4
Quality circle according to ISO 9004.
QE means, according to DIN 55350 Part 11, "Quality element(s) on the basis of... " (e.g., Production and responsibility for the Quality Assurance Elements (QS))

BISFA recommends the following terminology for fibers made from mixtures of chemically-different, non-reacting polymers or copolymers:

- Multi-constituent fibers, whose cross-section consists of at least two different polymeric components, practically continuous along the fiber length. The average composition of the components is constant, but composition can vary in any ratio along the fiber length. When only 2 components are present, the fiber is termed biconstituent.
- Multicomponent fibers, whose cross-section consists of at least two polymeric components, each component being relatively constant in cross-section and having a continuous length distribution. In the case of only two components, the fiber is termed bicomponent (Section 5.2).

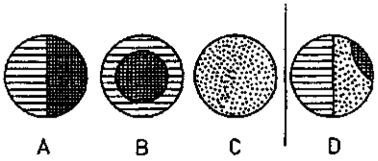


Figure 9.5 Example of a combination of two or more components in a man-made fiber

A: Two semicircular components; the proportions can be widely varied; the boundary between the two components can be straight or sickle-shaped. (See Fig. 5.22)

B: Two components in core/sheath configuration; ratio and centricity are variable.

C: Many single fibrils in an enclosing fiber, the so-called "island in sea" matrix fibers; the mixing ratio is highly variable, as is the number of the island fibrils.

D: A modification composed of examples *A* to *C*, with three components

Figure 9.6 gives definitions and examples of usage. The types S/S (= side by side), S/C (= sheath/core, skin/core), hollow fiber, M/F (= matrix/fibril) and other types are explained in Section 5.2. Many variations are possible by combining the basic types. The mass ratios of the components are usually adjusted according to the end-use. For example, a 50 : 50% ratio is used for texturing, a 10% sheath is used for thermobonding (S/C types), etc.

The fibers can have a round or profiled cross-section. Examples in application are given in Ref. [4]. The multiplicity of cross-sections possible is illustrated in Figure 4.136.

Figure 9.6 shows electrically-conductive fibers produced using a carbon filling.

9.2.2 Morphology of Man-Made Fibers

Table 9.2 and Fig. 9.7, taken from DIN, summarize the most important differences in filament, from staple fiber to bristle. This classification also serves as the definition of special yarns.

Additionally, in the case of meltspun fibers, differentiation is made between the spinning technology (and especially the take-up speed (Table 10.4)) and the single filament titer (Figure 10.3). The two last-mentioned are not defined by norms: every fiber producer has its own definitions. This leads to the usage of macromolecular orientation to describe the structure of LOY yarns and of crystallinity and orientation to describe POY yarn structure. An objective description of the degree of orientation is given by birefringence measurement (Section 9.3.1).

Flat man-made yarns are texturized to improve their bulk (voluminosity) and/or elasticity. The texturizing process is specified in DIN 60900, part 5, and is described in detail in Sections 3.6 and 4.12. Figure 9.8 shows the characteristic visual appearance of various textured yarns, which differ in both crimp structure and crimp intensity.

Hetero-yarns or mixed-filament yarns can be produced either at spinning or during further processing by plying and/or intermingling. Included here are mixtures of dpf (titer per filament) and filament cross-section. Staple fiber blends belong to this group. Here the advantages of the component staples must be synergistic: in blends of PET (or PA 6) staple and viscose (or cotton) staple, the cellulosic component improves the physiological properties, while the polyester enhances abrasion resistance (Figure 9.104). Single filament titer (dpf) differences affect handle and appearance, cross-section blends affect tactile properties and appearance, etc. (Figures 9.6–9.8, Table 9.2).

9.2.3 Application and Fiber Properties

Table 9.3 gives a summarized overview of the field of application of fibers.

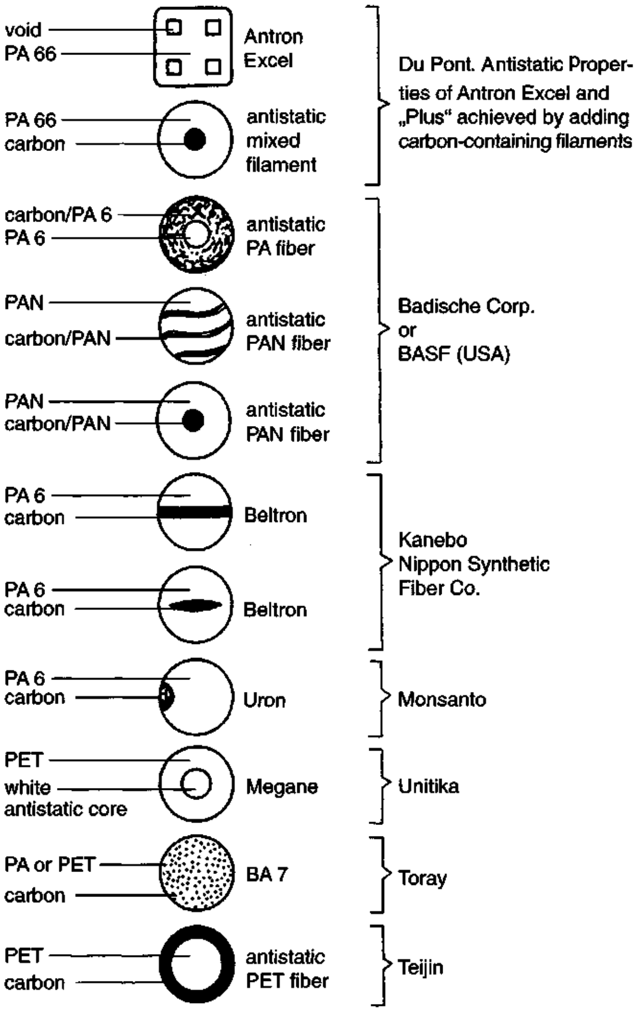


Figure 9.6
Examples of possible mixing combinations for two components (additional text in figure)

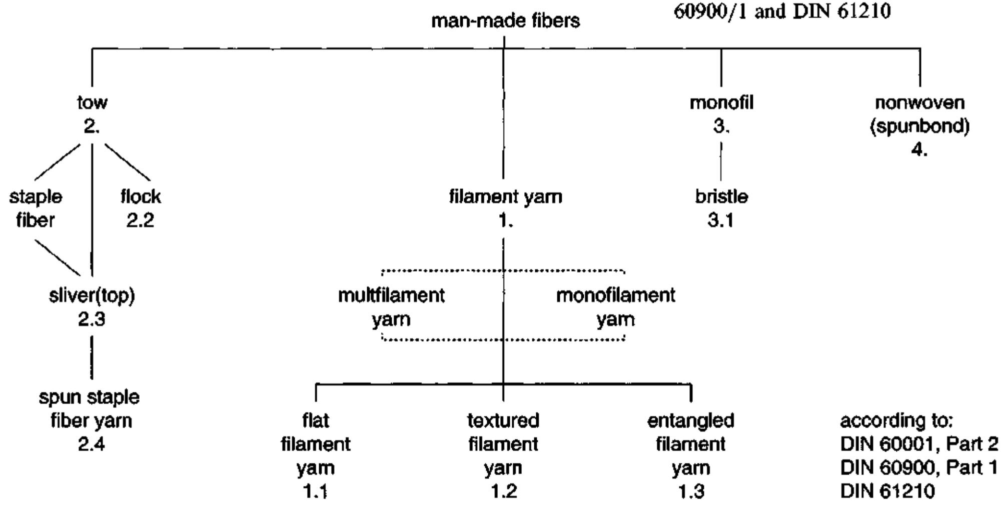


Figure 9.7
Classification of yarns and fibers according to DIN 60001/2, DIN 60900/1 and DIN 61210

Table 9.2 Explanation of the Terms Used in Fig. 9.7

Term	Explanation
Filament	Textile fiber of very great (practically endless) length (acc. to DIN 60000)
Monofil	A filament having a diameter of ca. 0.1 mm or more (acc. to DIN 60900, Part 1)
Tow	A multiplicity of filaments having no significant twist, e.g., a pre-cursor for staple fiber
Filament yarn	A yarn composed of one or more filaments, with or without twist. The single filaments can have a diameter of up to 0.1 mm (acc. to DIN 60900, Part 1/01, 88)
Flat filament yarn	Non-textured filament yarn (acc. to DIN 60900, Part 1)
Textured filament yarn	Filament yarn which has been textured (crimpled, bulked) (acc. to DIN 60900, Part 5)
Intermingled filament yarn	Definition according to DIN 60900, Part 1
Flock	Short fibers deliberately produced for further processing other than by staple-spinning (DIN 60000)
Staple fiber	Textile filament of limited length (from DIN 60000/01.69)
Top (sliver)	A fiber band or sheet produced from a tow, the filaments of which are maintained parallel, and are cut or broken to the same or different lengths
Bristle	Monofilament of limited length

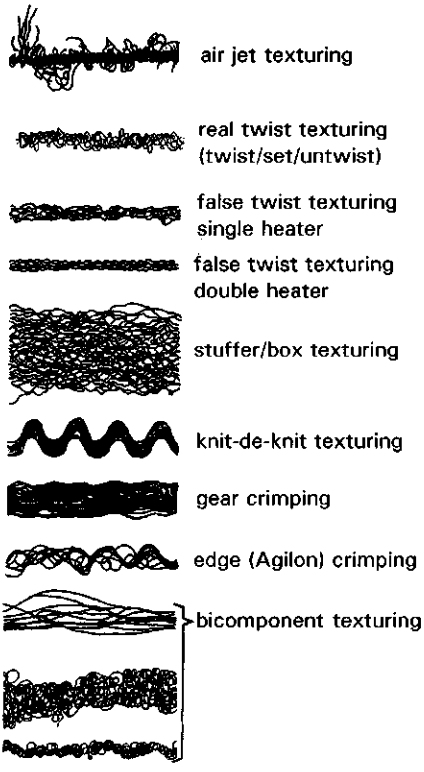


Figure 9.8
Optical representation of yarns textured by various processes. (For the methods, see Figure 4.25, etc.)

Filament yarns are further categorized into industrial and textile applications. Industrial applications require special properties; sometimes other properties become unimportant. Fishing yarns, e.g., must have very high wet tenacity, wet knot-strength, and chemical resistance to seawater, but must additionally be practically invisible underwater. Tirecords must combine high elastic modulus, high tenacity and excellent fatigue resistance. Hollow filaments used for reverse osmosis need only be moderately strong, but must have high porosity (Table 9.3).

Table 9.3 Typical Process/Product Scheme

	Products	Staple fiber and tow	Technical yarns	Textile yarns, flat and textured		
Processes	Spinning	conventional spinning speed (LOY)	conventional spinning speed (LOY)	increased spinning speed (POY)	high spinning speed (HOY)	very high spinning speed (FOY)
	Drawing texturizing	large drawframes and crimpers	Drawtwist or spin-draw-wind	for flat yarn: as per LOY, or drawtexturized	spin-draw-wind or drawtexturized	direct use or texturized
End use, Application		– 100%, and mixed yarns – Apparel fabrics: – Outerwear – Linings – Underwear – Carpets – Upholstery – Industrial yarn – Nonwovens – Sleeping bags – Mattresses – Curtains, covers – Laminates – Filters – Hand towels, etc – Sewing threads – Bedding	High total titer (≥ 500 dtex) – Tires – Drive belts – Seat belts – Hoses – Ropes – Cord – Coated fabrics – Conveyor belts – Fan belts, etc. Low total titer (≤ 500 dtex) – Sewing threads – Sailcloth – Coated fabrics	– Apparel fabrics– outerwear – linings – All-weather clothing – Sports clothing – Automotive textiles – Upholstery – Curtains and covers – Laminates – Sewing threads – Underwear – Bedding – Protective clothing		

9.3 Physical and Textile Properties

9.3.1 Fiber Structure and Fiber Properties

The fiber manufacturing process, in combination with further processing and finishing, results in a definite fiber structure regarding molecular structure and order, from which arises the fiber properties and finally the fiber capability in use. During fiber- and textile further processing, process conditions bring about further structural changes on the next "cause and effect" level. In this way, the final properties of the finished product are determined (in terms of fiber properties). The textile construction and finishing also have a decisive influence on the final properties of the finished product [12]. At the end of the cause/effect chain, the fiber processor, the textile designer and the user are confronted with

- fiber textile physical properties,
- dyeing properties,
- properties-in-use of the end product.

Figure 9.9 shows these relationships, together with some process parameters. Here the

- chemical properties of the polymer,
- process stages of fiber production and further processing,
- process parameters

determine the fiber structure at each process stage during manufacture, further processing and usage:

Temperature
Tension } = the 3 most important "T's" during production and processing
Time

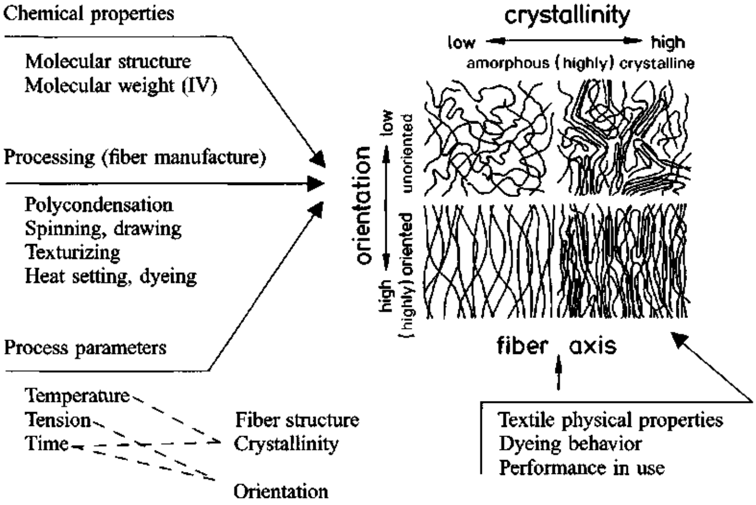


Figure 9.9 Effect of production process, further processing and finishing on fiber structure and properties

In view of the importance of these process parameters in fiber manufacture and in the determination of properties in multi-stage processing to end-products, the effects of these "3T's" on properties are often evaluated after each process stage. The properties measured after each processing stage enable a backwards-evaluation of the process conditions used and their effects, as well as prediction as to the effects to be expected in further processing, in particular:

- Temperature: The process temperature influences the Uster value, shrinkage, birefringence, elongation and tenacity at break of, e.g., PET POY. Higher temperature results in a higher crystallinity, a more dense molecular packing in the crystal, a coarser structure having more disorientation in the amorphous phase.
- Tension: Increasing tension leads to increased orientation, an increase in the crystal length and a reduction in chain back-folding
- Tension/time interaction: The temperature effects described become, in general, greater at low tension (and conversely).

- Time: The above effects increase asymptotically with increasing residence time: crystallinity is particularly affected.

The effect of spinneret temperature on various properties of PET POY can be directly seen in Fig. 9.10 [14], which serves as a model for the application of practical quality control testing. In this case, the fiber structure and its changes are not explicitly examined. Here particular process variables—preferably only one (in this case, spinneret temperature)—are changed, and the results are plotted as a function of the variable changed (Fig. 9.10). The resulting relationships are then used for process control.

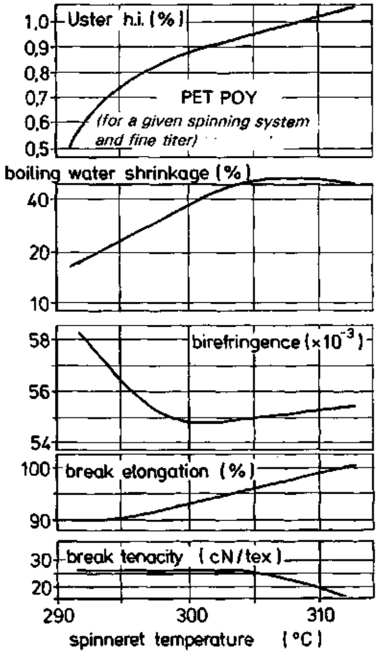


Figure 9.10
Effect of spinneret temperature on various physical properties of PET POY (h.i. = half inert)

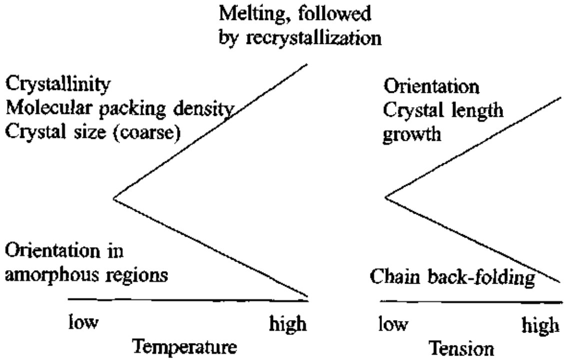


Figure 9.11
Schematic effect of process conditions on fiber properties

Research has shown that higher temperature results in higher crystallinity, denser packing of the molecules in the crystal lattice, larger crystals (= coarser structure) and more disorientation in the amorphous regions. One then establishes (as in Fig. 9.11) which fundamental changes result from an increase in temperature and tension, which effect these have on fiber properties, and how these can be influenced by previously-mentioned process parameters. These analyses are usually costly and complicated, take a long time and must be done in the laboratory. Table 9.4 gives a condensed version of appropriate and typical analytical methods.

The most important aspect of the analytical methods is the investigation of fiber structure and its parameters:

- Crystallinity as degree of order in crystalline, paracrystalline and amorphous regions. Table 9.5 shows X-ray structural analytical methods and Table 9.6 methods for determining density. Figure 9.12 shows characteristic data for a drawn PET yarn, obtained by small-angle X-ray scattering (SAXS), including the long period and fibril width. The long period is ca. 200 . . . 300 A° (1 A° $= 10^{-10}$ m), the crystal height is ca. 50 . . . 100 A° (1.5 × crystal width) and the crystal width is ca. 30 . . . 80 A°. In the case of high speed spun yarns, crystal size depends on the take-up speed, while for drawn yarns, crystal size depends on both temperature and tension.

Table 9.4 Methods for Investigating Fiber Structure

Structural element	Method	Dimensions
Fiber	Optical microscopy	> 0.2 µm
	Electron microscopy	> 100 Å
Fibrils/lamellae	(Irradiation, scanning electron microscope)	
Voids	Small-angle X-ray scattering	1000...10 Å
Crystalline regions	Wide-angle X-ray scattering	10...1 Å
Non-crystalline regions	Small-angle X-ray scattering	100...10 Å
	Spectroscopy (IR, NMR, dielectric, dynamic-mech., etc.)	Molecular groups
Orientation	X-ray scattering (wide- and small angle)	Lamellae, crystallites
	Infrared, birefringence, sonic velocity	Molecular segments or groups

Table 9.5 X-ray Scattering

Wide-angle X-ray scattering

- molecular distances (e.g., unit cell dimensions)
- crystallite size (length, breadth, height)
- crystallite (crystalline) orientation

Small-angle X-ray scattering

- long period = crystallite length + length of amorphous region
- orientation of crystalline and amorphous regions

Note: X-ray structural analysis is deemed to be a "direct" method, as the measured properties are absolute values, given, e.g., as length units (e.g., 10^{-10} m) or as angles

Table 9.6 Determination of Density

Determination of density ($\rho = m/V$) by the flotation method, as a measure of molecular packing density

- Crystalline density ρ_c—the maximum density of a polymer, obtained from the molecular weight and volume of the crystal unit cell
- Amorphous density ρ_a—obtained by measuring the density of a fully-amorphous sample
- Degree of crystallinity (v/v) α:

$$\alpha = \frac{\rho - \rho_a}{\rho_c - \rho_a} \cdot 100\%$$

ρ = measured density (g/cm^3)
ρ_a = amorphous density; PET = 1.3310 g/cm^3
ρ_c = crystalline density; PET = 1.4550 g/cm^3

The result is calculated from the measured density and assumed values for the crystalline and amorphous densities. The degree of crystallinity gives the crystalline volume fraction. The amorphous volume fraction is given by $(1 - \alpha)$

- Density can be easily determined by means of a density gradient column (see Table 9.6). Density is very sensitive to spinning speed and temperature [15]. A distinction is made between the fiber bulk density and the density of the amorphous region. Above 3500 m/min, PET yarns show a distinct increase in the bulk (= filament) density, i.e., the starting point for speed-induced crystallization

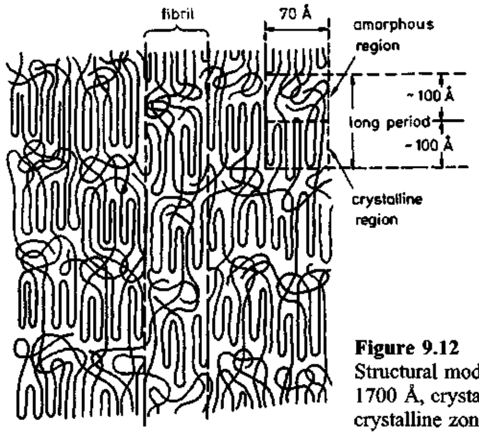


Figure 9.12
Structural model of drawn PET yarn (Given data: average chain length: 1700 Å, crystallinity of the drawn yarn = 10–30%, orientation factor of the crystalline zone = 0.98 and of the amorphous zone = 0.7 . . . 0.85)

(Figs. 9.12, 9.13). Further-processing temperatures have an additive effect on the filament density, as can be seen for texturized PET yarn in Figure 9.14, where the density is given as a function of the primary heater yarn effective temperature. In contrast, the method of texturizing (spindle vs. friction) has no significant effect on filament density, within the limits of experimental error. From the linear increase in density with increasing yarn temperature, one can assume an increase in crystallinity and thus an increase in the density of molecular packing (Figs. 9.13, 9.14).

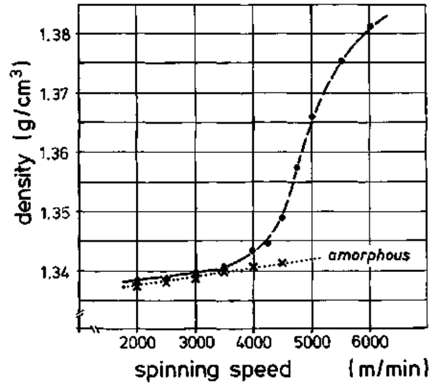


Figure 9.13 Density (g/cm^3) of amorphous and high-speed PET spun yarn as a function of the spinning speed

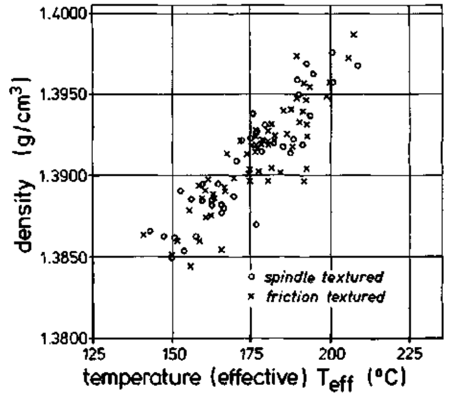


Figure 9.14 Density (g/cm^3) of PET yarn as a function of the effective primary heater yarn temperature

- Orientation can be determined as per the scheme in Table 9.7. Orientation is a measure of the alignment of the crystallites in the crystalline and amorphous regions parallel to the filament axis. Optical birefringence measures the total birefringence of the fiber. X-ray analysis can be used to measure the crystal orientation, thus enabling the amorphous orientation to be calculated. The dependence of crystalline (f_c) and amorphous (f_a) orientation on spinning speed is given in Fig. 9.15, which can be compared with Fig. 9.13. As is to be expected, crystalline orientation occurs only after the onset of sufficient crystallization. Only above ca. 7000 m/min can one talk in terms of total orientation having saturated (Table 9.7, Fig. 9.15). Interference microscopy (= measurement of fringe displacement) enables radial non-uniformity of birefringence to be measured. Such non-uniformity can arise from core/sheath effects or from asymmetrical cooling. Figure 9.16 shows such

Table 9.7 Determination of Orientation

Birefringence*
Optical method for determination of total orientation

- Crystalline orientation Δ_c
- Determined from wide-angle X-ray scattering
- Amorphous orientation Δ_a

Calculated from degree of crystallinity and crystalline orientation

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*only applicable to fibers of round cross-section

Sonic modulus
Measurement of velocity of sound (pulse propagation method) as determinant of **total orientation**
(A higher orientation results in a higher velocity of sound)

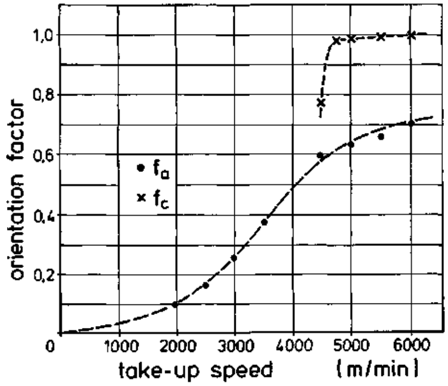


Figure 9.15
Orientation factors of PET yarn as a function of spinning speed: f_c in crystalline region and f_a in amorphous region (vol/vol) [15]

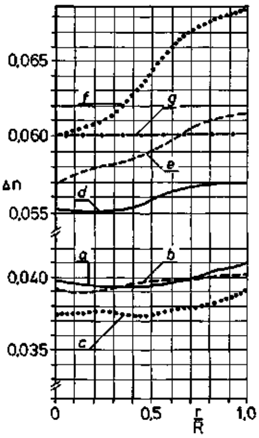
effects for a PA6 POY yarn spun at different extrusion temperatures, and also for the same yarn after drawing. The increase in birefringence from core to sheath of the POY is evident, as is its magnification after drawing [17]. Relationships between orientation and shrinkage and shrinkage force are covered in Section 9.3.4. Figure 9.17 shows the birefringence of PET yarn as a function of shrinkage force (a) and of shrinkage (b), both of which increase with degree of orientation (i.e.

Figure 9.16
Birefringence $\Delta n(r) = n\|(r) - n|(r)$ of a PA 6 fiber as a function of radius r, as well as averaged over the cross-section A: $\overline{\Delta n} = A^{-1} \int_A \Delta n(r) \cdot dA(r)$

Radius R of filament = 12 µ

Curve	Spinneret temperature °C	Draw ratio
a, POY	260	
b, POY	265	
c, POY	270	
d	260	1 : 1,6
e	265	1 : 1,6
f	270	1 : 1,6
g	270	1 : 1,6

g additionally heat-set at constant length for 1 min at 190°C



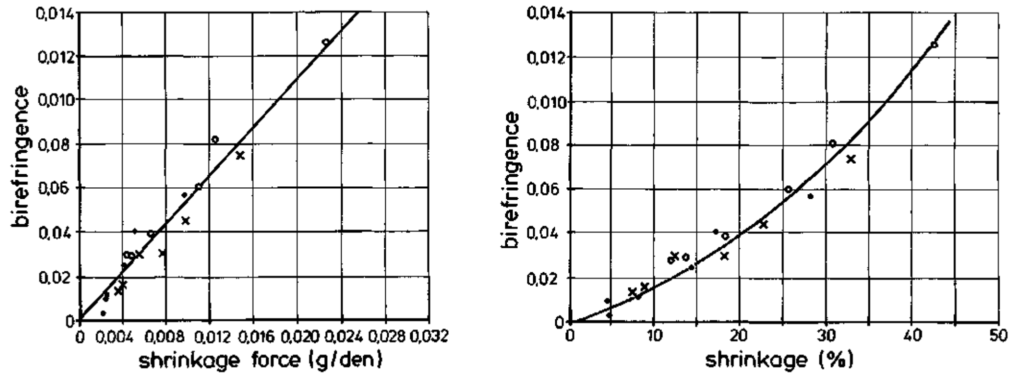


Figure 9.17 Birefringence of PET yarn: ○ Mw = 35 000, × Mw = 27 000, ● Mw = 20 000 *a*) as a function of shrinkage force, *b*) as a function of shrinkage

birefringence) [18]. Combining density and birefringence (measured on the same sample) on a single diagram provides comprehensive information regarding the structural character of a man-made fiber. Fig. 9.18 shows such a plot for PET yarns spun at various speeds. This precise evaluation of structural state enables changes in further processing to be localized in terms of their effect. In particular, it relates which structural parameters are most strongly affected by the change. Tables in Chapters 10 and 11 give the densities of various fiber types, including a few birefringence values from the literature (Figure 9.18).

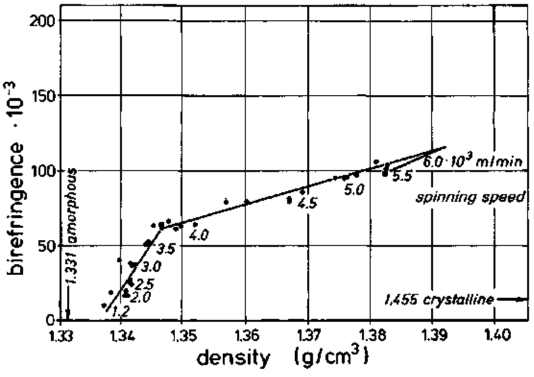


Figure 9.18
Birefringence of PET yarn as a function of density, with spinning speed (m/min) as parameter

- Thermal reactions and melt behavior and the relationships among melt behavior, partial melting and thermal resistance for the different stages of yarn production and further processing and the consequences thereof, are summarized in Fig. 9.19. Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC) (Fig. 9.20) are frequently-used methods of investigating thermal properties, from room temperature through glass transition temperature to complete melting. Here heat flow to and from the sample (enthalpy) is measured as a function of a specified rate of heating. If structural changes occur in the sample during heating, heat can either be given out (e.g. during crystallization) (exothermic reaction) or heat can be absorbed (e.g. during partial melting)

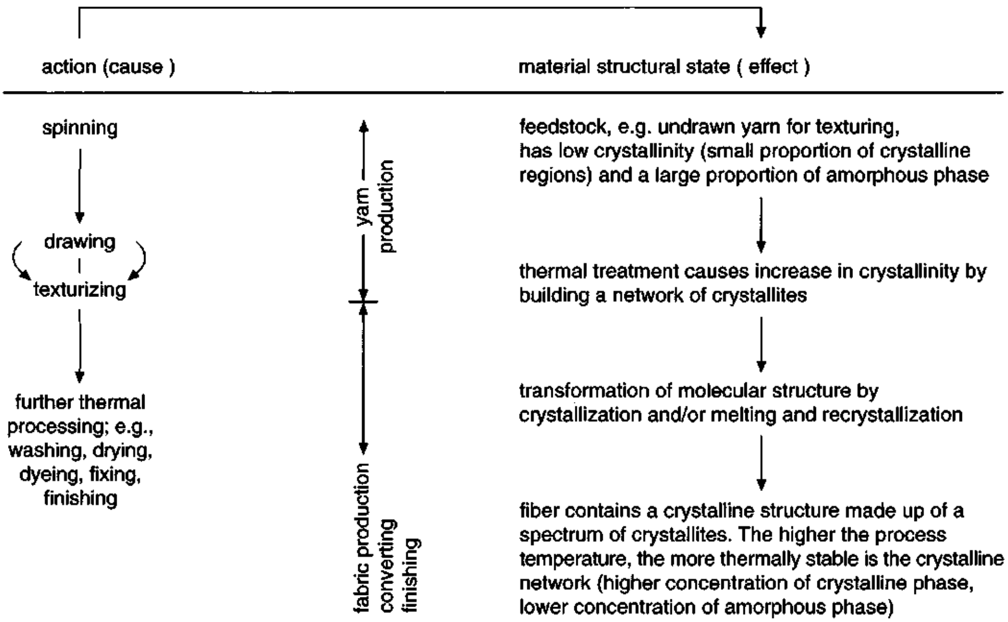


Figure 9.19 Relationship between process stages of yarn production and finishing and their effects on the material structural state

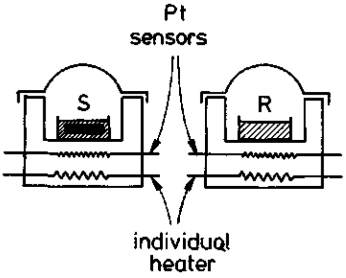


Figure 9.20
One sample holder carries the fiber sample, the second holder contains a reference sample. Both holders are heated uniformly at a linear rate of temperature increase up to the sample melting point. The difference in the required heat input is recorded vertically, the sample temperature horizontally. S = holder + sample, R = holder + reference.
$Q = (Q_S + Q_F) - Q_R$, where Q = heat quantity, and the indices S = sample holder, F = fiber and R = reference sample

(endothermic reaction). These extremely small amounts of heat are recorded on a thermogram as a function of the sample instantaneous temperature, thus enabling the nature of the reaction to be determined (see Fig. 9.21 for undrawn PET). With increasing spinning speed (from LOY to POY at 3500 m/min), the reaction remains exothermic, and the crystallization temperature decreases as the speed increases [20], i.e., the crystallization peak shifts to lower temperatures while the heat of crystallization (peak area) simultaneously decreases (Fig. 9.20).

Typical applications in which the thermal history can be traced are found in:

yarn production: spinning, drawing, heat setting, steaming
texturing: primary- and secondary heater temperature effects
finishing: dyeing, heat setting
general: polymer granulate, ageing

During production and post-processing, man-made fibers are subjected to a wide range of temperature effects, sometimes involving partial remelting and further crystallization. Here the DSC

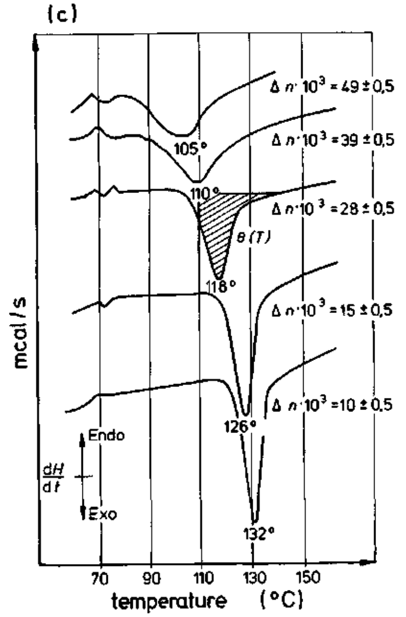


Figure 9.21
Thermograms of undrawn PET filaments) from LOY to POY. (Birefringence is given as parameter)

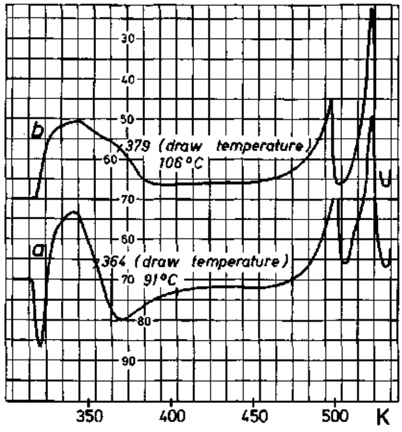


Figure 9.22 Thermograms of two polyester yarns:
a) conventionally drawn on a drawtwister: drawing temperature = 91°C
b) spin-drawn: drawing temperature = 106°C

diagram is a valid indicator of the thermal stability of the crystallites so formed. As an example, Figure 9.22 compares the DSC diagrams of drawtwisted and spun-drawn yarns. From the differences in thermal treatment and effective yarn temperature, one would expect to see differences in shrinkage, shrinkage force and dyeability. This is substantiated in Fig. 9.23, which shows the relationships among pretreatment temperature, residence time and heating medium. At short residence times (0.2 s during texturizing), the effective yarn temperature is lower than the machine temperature [21], while for sufficiently long residence times (e.g., 20 s during thermosetting) the two temperatures approach one another. Hydrothermal treatment (e.g., during high temperature dyeing) results in a significantly higher temperature (by ca. 40 °C) than a dry heat treatment [61,62], which means that crystallites of higher thermal stability are produced. Structure, large crystalline regions and stress on the tie molecules can affect the melting point. If no measurements are available, the glass temperature Tg can be estimated from the melting point temperature Tm using Tm = ca. 3 Tg/2 [13]. In the case of PA6, crystallite modification and -length has an effect on the melting point: an increase in crystallite length increases the melting point. High speed spun PET filaments behave similarly (Fig. 9.24), and the melting point can increase to 270 °C [10].

- Dye uptake depends predominantly on the fiber morphology, i.e. on crystallinity and orientation. The dyestuff molecules diffuse through the structure and lodge themselves in the microscopic- and submicroscopic voids in the amorphous region. Figure 9.25 illustrates how dye uptake is affected by fixation (temperature) in the case of PET yarn: a minimum occurs at 180 ... 190 °C [27]. This results from two opposing processes: on heating to higher temperature, crystallinity increases at the expense of the amorphous region; the diminished amorphous region can hold less dyestuff (lighter dye depth). On heating to still higher temperature, the crystallinity increases further, but at the same time larger crystallites are formed, thus strongly increasing the free volume in the amorphous region and enabling more dyestuff molecules to lodge there (darker dye depth). The dependency of dye uptake on orientation can be indirectly determined from Fig. 9.26: with decreasing elastic modulus (i.e., lower draw ratio = lower orientation), dye uptake increases. Samples which were allowed to shrink

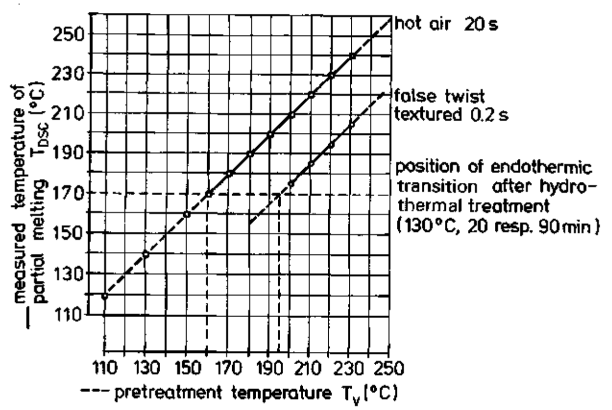


Figure 9.23
Relationship between pretreatment temperature, residence time (or pretreatment medium) and the observed position of the transition in the thermogram (*a*: hot air, 20 s; *b*: false twist texturizing, 0.2 s; *c*: position of the endotherm transition after a hydrothermal treatment at 130°C for 20 resp. 90 min)

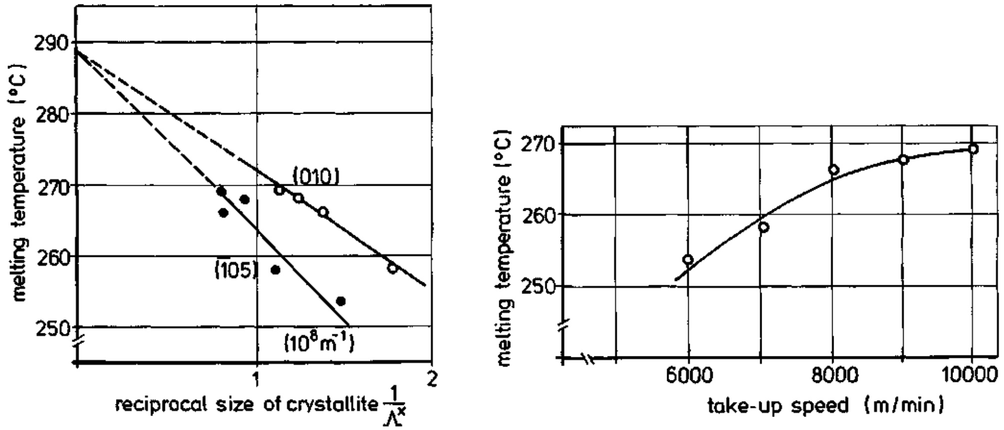


Figure 9.24 DSC-determined melting point of PET fiber as a function of the spinning speed and the reciprocal crystal size

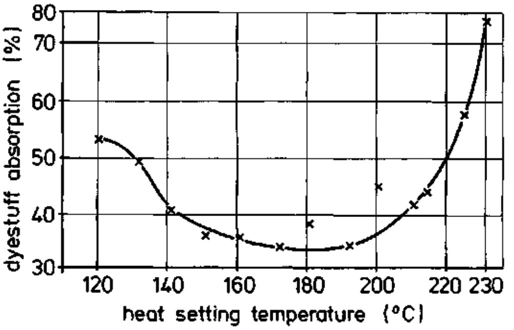


Figure 9.25
Effect of setting temperature on the dye uptake of Terylene (= PET, dyed using 2% Dispersol Scarlet B 150 for 90 min at 100°C [28])

have a tendency to lower dye uptake than samples which were not, as disorientation takes place in the amorphous regions during shrinkage [19]. In finishing, the setting temperature (which determines the crystallization) and the tension during heat setting (which determines the orientation) are particularly important influences. Figure 9.27 shows these relationship contours in three dimensions (a) and projected onto two dimensions (b); these confirm Figure 9.25 in principle [21].

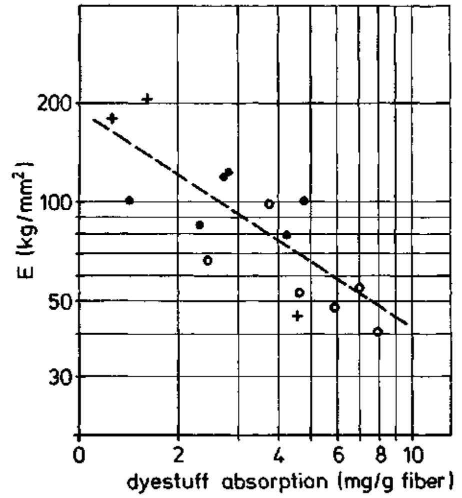


Figure 9.26
Correlation between static elastic modulus at 2% elongation and dye absorption in mg dyestuff/g fiber (1% w/w) Sirius Light Blue BRR (Durazon Blue B) in aqueous dyebath at pH 5 and 95°C for less than 1 hour) for PA 6 yarns of differing pretreatment:
+ drawn, not heat set
● drawn and heat set at constant length
○ drawn and heat set under free shrinkage
- - - heat set in saturated steam at 110...130°C

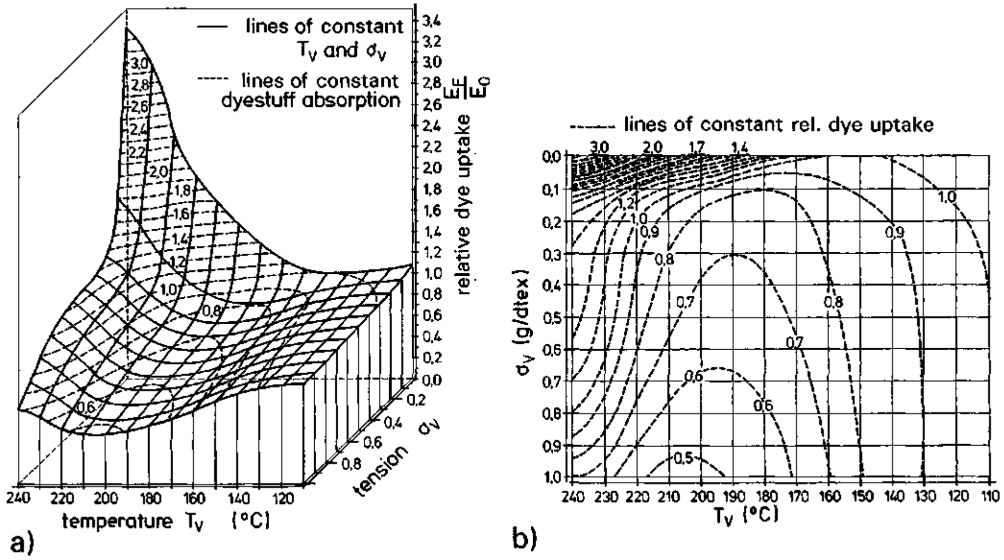
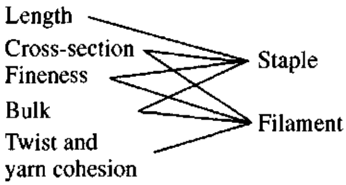


Figure 9.27 *a*) Relative dye uptake ($E_{\text{fix}}/E_{\text{orig}}$) of PET yarns as a function of pretreatment temperature and -tension (20 s fixation time).
Dyeing conditions: 90 min, 130°C, free shrinkage, 3% Resolin Marine Blue GLS, liquor ratio 1 : 500
b) Projection of lines of constant dye uptake onto the base area

In spite of strict adherence to process parameters and their allowed tolerances, differences in dye uptake affinity still occur. These differences can be levelled out either by use of high dyeing temperature or by application of various additives (levelling agents, carrier, etc.), both of which can additionally improve dye fastness.

9.3.2 External Form and Constitution of Fibers

Certain desired properties can be built into man-made fibers during production. Typical characteristics are



The above characteristics confer on the fiber certain properties which are advantageous in further processing and especially beneficial to the properties of the end product ("fitness in use").

The following modifications are also employed in order to achieve the various properties of the final product:

- fiber cross-section and surface
- filament size reduction (microfiber, superdrawing, alkalization)
- filament mixture (fineness, cross-section)
- bicomponent fibers
- bulk/texture (texturing, bi-shrinkage and high bulk yarns)
- after-treatment (crimping through texturizing)
- twisting, intermingling
- mixed yarns (hetero yarns, mixtures)

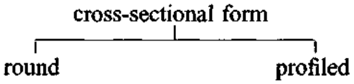
9.3.2.1 Cross-Section and Surface

Cross-section and filament surface form strongly influence the properties of the final product. In the case of melt-spun staple and yarn, the producer can determine the cross-section and surface form, as well as decide on whether to produce solid- or hollow filaments, whereas these are partly determined by the strong influence of spinning conditions in the case of solution-spun yarns. Once chosen, the filament cross-sectional shape and form determine many yarn properties, which, in turn, determine the optical, tactile, physiological and technological properties of the final article. There are further opportunities to modify the cross-section and surface shape of fiber or fabric during further processing (e.g. texturizing, alkalization) in order to change the properties of the final product, but these are less effective than a modification introduced in the production stage.

- Investigation into filament cross-section and surface shape
  - Objectives
    Identification of polymer type (cf Section 9.5)
    Determination of cross-sectional deformation (e.g., as a result of texturizing)
    Failure analysis (process deviations, damage)
    Distribution of delusterant, optical whitener, dyestuff and sheath/core effects
    Determination of fiber fineness (see Section 9.3.2.2)
    Mix ratios of fiber, titer and cross-section mixtures and of filament surface, i.e., with longitudinal pictures

Surface structure, to determine roughness, pores, vacuoles, damage
Surface deposits (oligomers, spin finish residue, etc.)
Surface modification resulting from further processing (e.g., alkalizing of PET) or for process identification (e.g., spindle or friction texturizing)

- Definition of cross-section and surface form
  Their multiplicity necessitates a series of definitions:
  - Cross-sections are firstly subdivided according to



Profiled encompasses 2-lobed, 3-lobed (trilobal), rectangular (rounded, 4-lobed) and star-shaped (n-lobed) (Fig. 9.28)

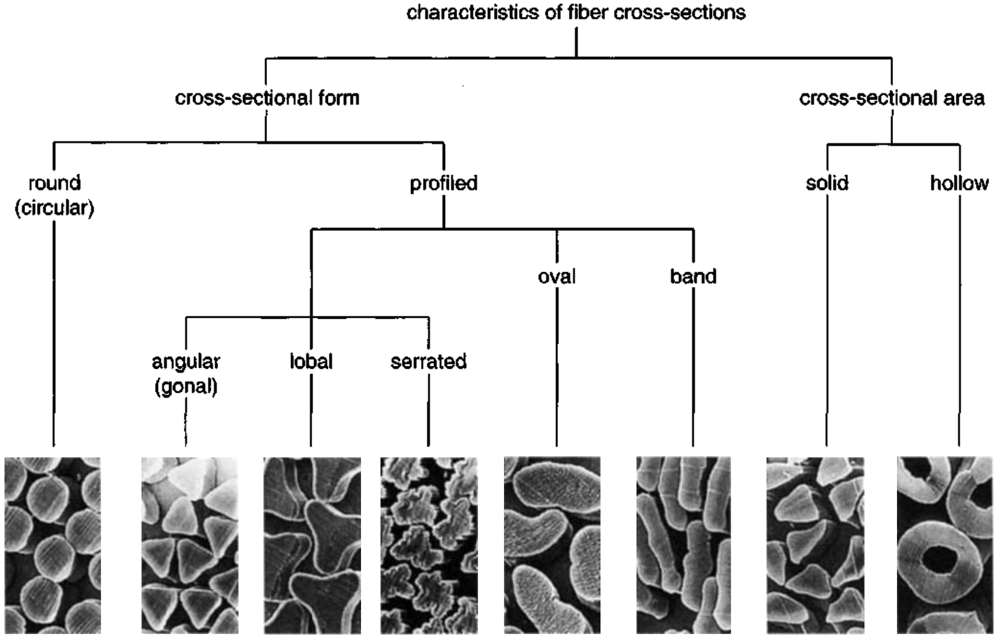
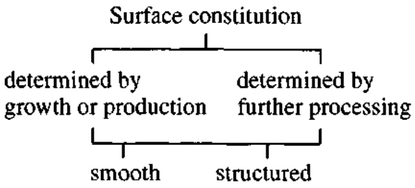


Figure 9.28 Characteristics of fiber cross-sections

- Surfaces can be categorized according to the matrix:



Fibers with structured surfaces can be further subdivided according to morphological fine structure (Fig. 2.29). Cross-sectional deformation and/or surface modification can change the yarn properties, and thereby also the properties of the final product (optical and/or handle).

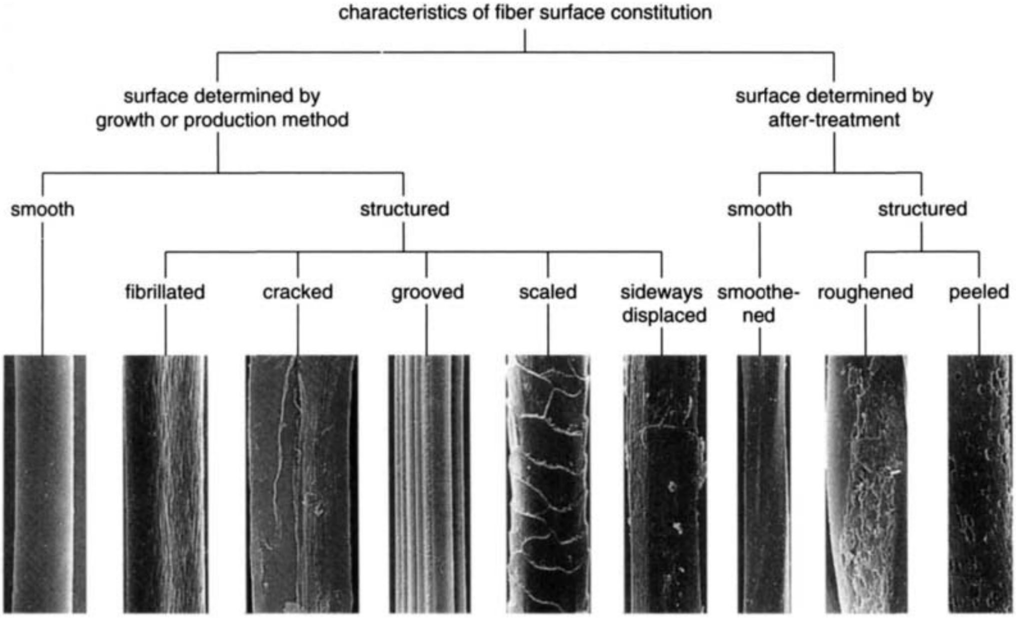


Figure 9.29 Characteristics of fiber surface constitution

- Investigation and evaluation of cross-sections and surfaces can be carried out according to the following scheme:
  - Preparation of a cross-sectional (or microtome) cut of 5 . . . 10 μm thick.
  - Analysis by optical microscopy (using various light sources and illumination techniques) or by electron microscopy (SEM), and
  - Documentation by means of photographs, drawings or computer-assisted picture analysis.

Fiber surfaces are investigated in the same manner, the documentation being in the form of a longitudinal picture (Fig. 9.29).

Geometrically-defined form relationships can give useful information about the cross-sectional shape. A planimeter can be used to measure the cross-sectional area. Frequently the modification ratio $M = R/r$ is used, as shown in Fig. 9.30, the modification ratio being the ratio of the radii of the circumscribed- and inscribed circles. $M = 1$ means that the filament is round. As M increases further, so the deviation from roundness increases. Figure 9.31 shows that, for a triangular PET yarn (upper left, $M = 1.61$), the force/elongation curve is shifted to lower force and higher elongation when the modification ratio (lower right, $M = 1.44$) is deliberately reduced and made less regular. Garments made from the above two yarns are optically different (Fig. 9.30).

In friction texturizing, it is not sufficient to look only at the least expensive friction disk material in terms of lifetime and twist generation, but it is also necessary to observe its effect on the filament surface, as this affects the optical and tactile properties of the finished goods (Fig. 9.32). Additionally, there is a strong correlation between the degree of change of the filament surface and the yarn tenacity, which decreases with increasing roughness (Fig. 9.33). The change to the filament surface partially weakens the tenacity of the sheath structure [23].

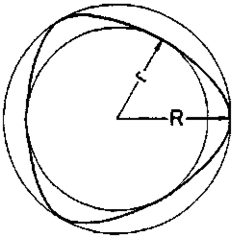


Figure 9.30
Diagram used in determination of modification ratio: modification ratio $M = R/r$. (R = radius of the circumscribed circle and r = radius of the inscribed circle)

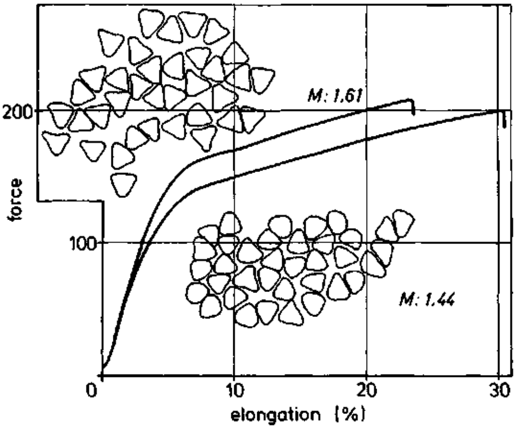
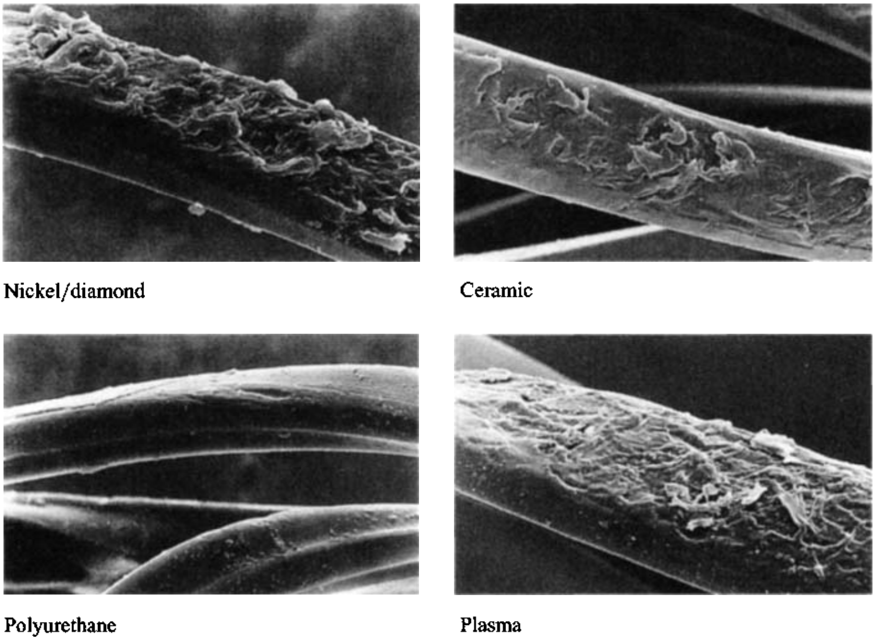


Figure 9.31
Effect of the modification ratio on the tenacity/elongation curve of a triangular (delta) polyester yarn (above, left) and an irregular version of the same (below, right)



Nickel/diamond

Ceramic

Polyurethane

Plasma

Figure 9.32 SEM pictures of filament yarns having been texturized using various disk materials

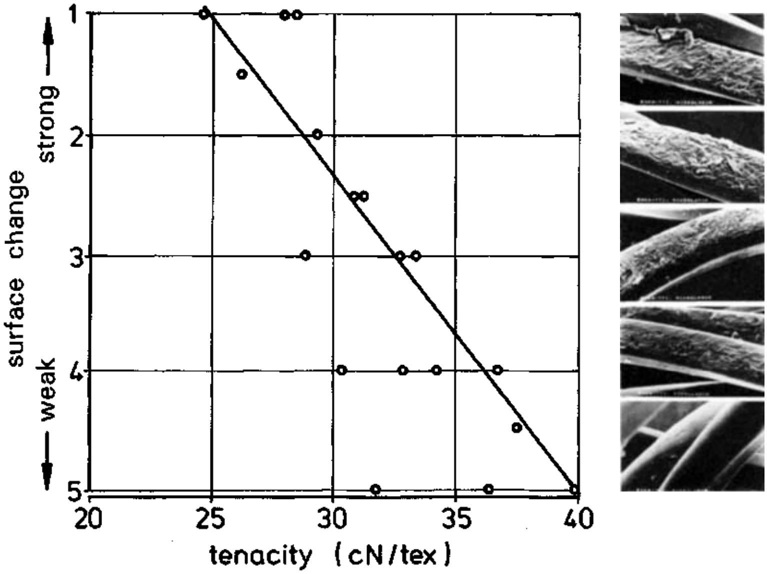


Figure 9.33 Relationship between the degree of surface change and tenacity for a 167 dtex f30 polyester multifilament yarn

9.3.2.2 Fineness of Staples and Yarns

- Definition and concepts

Linear density is the basis for specification of fiber fineness (or titer). In the SI system, the basis is kg/m (see Section 10.2), while DIN 60905, page 1, specifies the Tex system (1 Tex = 1 g/1000 m) for textiles. In addition, certain geographically- or historically-based systems persist. Two, in particular, remain firmly entrenched: 1 den (denier) = 1 g/9000 m (an old Florentine silk measure) and 1 Nm (metric count) = 1 km yarn/1 kg. The relationship between the Tex system and the equivalent diameter of a round filament is given by

$$d\,\mu\text{m} = 20\sqrt{T_t[\text{dtex}]/\varrho(\text{g/cm}^2)\pi}$$

and is shown in Figure 9.34 for various materials. Figure 10.3 gives a classification according to fineness. In the case of mono- and multifilament yarns, the number of filaments per yarn is specified

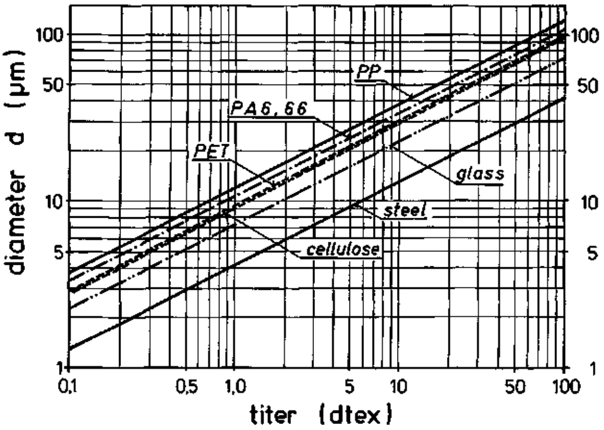


Figure 9.34
Relationship between fiber fineness (titer) and equivalent filament diameter

Table 9.8 Methods for Determining Staple Fiber Fineness

Method	Calculation of fineness T_t	Measuring principle
Gravimetric method	$T_t = \frac{G \cdot 10^4}{Z \cdot l}$ [dtex]	A definite length of parallelized staple fiber is cut and weighed G = fiber mass (mg) l = cut length Z = no. of single fibers in bundle
Counting the no. of fibers in the tow cross section	$T_F = T_g : Z$	Fibers or filaments are counted, and the single filament titer is calculated from the total titer T_F = single filament titer T_g = total titer Z = no. of single filaments
Optical methods Measurement of filament diameter	$T_t = d^2 \cdot \frac{\pi \cdot \rho}{4 \cdot 10^2}$ [dtex] with: $\frac{\pi \cdot \rho}{4 \cdot 10^2} = C$ $T_t = d^2 \cdot C$	Measurement of fiber diameter using a microscope or special measuring equipment d = fiber diameter (µm) ρ = filament density (g/cm^3) C = constant: wool = 0.01021 polyamide 6, 66 = 0.00903 polyester = 0.01084 polyacrilonitrile = 0.00918
Measurement of filament surface area	$T_t = 0.01 \cdot A \cdot \rho$ [dtex]	Planimetry of microtome-cut section under microscope magnification A = cross-sectional area (µm^2) ρ = filament density (g/cm^3)
Vibration methods	$T_t = \frac{C}{f^2}$ [dtex]	Determination of the resonance frequency of a fiber of a defined length oscillating under a defined load C = constant depending on length and pretension of the fiber f = fiber resonance frequency
Air permeability	Air flow is proportional to fineness	Measurement of air flow rate or pressure drop through a fiber sample under defined conditions

in addition to the total titer of the yarn. Thus, e.g., 167 dtex f 32 denotes a multifilament yarn of total decitex 167 having 32 individual filaments of 167/32 = 5.22 dtex per filament. DIN and BISFA make the following distinctions:

- Nominal fineness: Staple or yarn is specified by its nominal titer
- Actual fineness; Titer measured under actual, specified conditions. Here distinction is also made between spun (undrawn) and drawn fineness.
- Target fineness: Titer to be achieved in the production process.
- Commercial fineness: Titer appearing in the sales document, including moisture- and oil content.

- Methods for determining fineness are listed in Table 9.8. The gravimetric method is predominantly used for yarns and twisted products. There are two necessary steps in the gravimetric method:

Table 9.9 Formulas for Calculating Change of Fineness

Formula	Comment
$T_{\text{total}} = T_1 + T_2 + \cdots + T_n$ $T_{\text{Total}} = T_1 \cdot Z$	Total titer (no twist) T_{total} = total titer T_1, T_2, T_n = individual titers Z = number of plies
$T_b = T_o \dfrac{100}{100 - S}$ $T_b = T_o \dfrac{100}{100 - e}$	Change of titer caused by length changes T_b = titer after treatment T_o = titer before treatment S = shrinkage e = twist contraction
$T_b = T_o \dfrac{100 - p}{100}$ $T_b = T_o \dfrac{100 + q}{100}$ $T_b = T_o \dfrac{100 + \psi t}{100 + \psi t_1}$	Change of titer caused by change of mass T_b = titer after treatment T_o = titer before treatment p = mass loss, due to washing, loss of spin finish, etc. q = increase in mass due to process additives ψt, ψt_1 various moisture contents at various RH levels

- generation of a specified length by laying or wrapping
- weighing using a balance of sufficient accuracy
- calculation of fineness in dtex (with recourse to statistical methods, as per Section 10.6.9).

Manual or motorized wrap-reels are normally used, although fully-automated devices are also employed, particularly as part of an automatic tensile tester. Because of the limited length tested, deviations are experienced, typically 1–5% coefficient of variation on 0.1 m lengths, 0.9% on 1 m lengths, 0.5% on 10 m lengths and 0.3% on 70 m lengths. This leads to continuous titer measurement (see Sections 9.3.5 and 7.8).

- Significance of fiber fineness
  Process parameters which determine fineness (spin pump speed, take-up speed, draw ratio, relaxation, temperatures, etc.) are set in the production process in such a manner that the nominal fineness is achieved. Table 9.9 shows how corrections can be made during each stage of production. Changes in mass occurring in further processing can be similarly taken into account. Fineness affects, above all, the handle and aesthetics of the finished goods. In many instances, one strives to reproduce the titer of natural fibers. Originally this was done with a view to substitution by the new specific staples and yarns, but today it represents a valuable supplement.

9.3.2.3 Spun Fiber (Staple) Length

- Significance of staple length
  Staple length is a very important characteristic for further processing and yarn- and final product properties. It affects, e.g., carding performance, the nip point distances in drafting, and determines the spun yarn fineness, tenacity and elongation and their uniformity. (For the effect on downgraded goods, see Section 9.4).
- Measuring methods for determination of fiber length are summarized in Table 9.10. The "fiber beard" determination incorporates computer support. Above all, the following characteristic values must be given:
  - average fiber length (mm)
  - coefficient of variation of fiber length (%)

Table 9.10 Summary of Various Methods for Measuring Fiber Length

Method	Comments on procedure
Measurement of single fibers (acc. to BISFA and DIN)	Measurement of the length of single, uncrimped fibers Staple fiber length distribution diagram
Combing process (Johannsen-Zweigle-Schlumberger)	Using appropriate equipment, the fibers are sorted according to length
Fiber beard methods (Fibrograph, WIRA, Almeter)	A straightened-out staple fiber sample positioned on a baseline is scanned using various measuring methods: a) Measurement of the dependence of light transmittance on the thickness of a fiber bundle using photocells b) Measurement of the light reflection of a staple fiber layer on a black background c) Measurement of a straightened-out staple fiber using a capacitor, and display of the length distribution diagram by means of a recorder

- most frequently-occurring fiber length (%)
- proportion of most frequently-occurring fiber length (%)

The measurement basis could be either weight or fiber frequency, in which case the characteristic values can show considerable differences. The man-made fiber lengths are determined by the process conditions (Section 4.13.12 and 5.8), whereby the staple cutter delivers a seemingly constant distribution of fiber length (e.g., Fig. 4.31), while the converter generates a more natural fiber length distribution (e.g., Fig. 5.69).

Mechanical demands during the various further processing stages (e.g., carding) can give rise to staple length reduction, which has a negative effect on certain yarn properties (tenacity, yarn cleanliness). Figure 9.35 shows an example (measurement principle: measurement of a staple using a condenser). A sample taken after carding shows deviations to shorter staple length in the range of 5 ... 15 mm (length reduction). This points to fiber damage during the carding process (Table 9.10, Fig. 9.35).

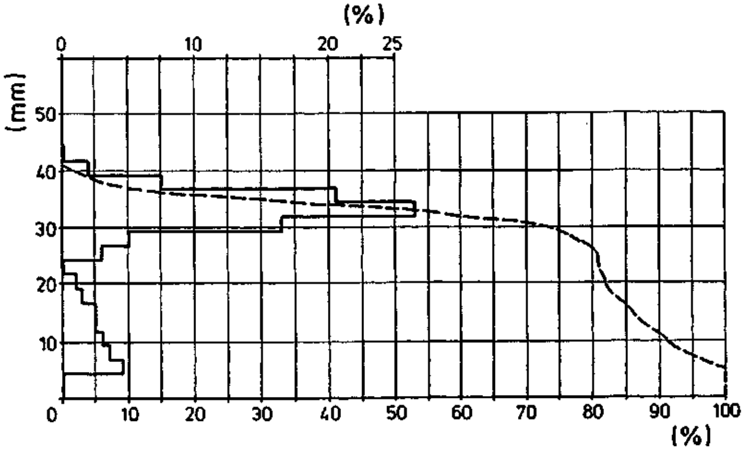


Figure 9.35
Polyester staple fiber diagram, showing a most-frequently occurring fiber length of ca. 34 mm. The shorter fibers arise from damage during the carding process

9.3.2.4 Crimping (Bulking) Properties

There are two basic ways of measuring crimp in staple fibers or textured yarns:

a) geometrically-based crimp properties: number of crimps per unit length—their form—their amplitude—degree of crimping—crimp development
b) crimp properties developed in the fiber: decrimping force and work—crimp stability—crimp modulus.

The concepts for evaluating crimp properties given in Table 9.11 are valid for both staple fiber and textured yarn.

- The staple fiber crimp and its subsequent interaction with the spin finish is important in further processing: smooth fibers require higher crimp, and vice versa. Because of the relatively low crimp stability of staple, crimp properties hardly manifest themselves in the finished product.
  All chemical fibers, however, are more or less strongly crimped and there can be large differences in the form and size of the crimps. Figure 9.36 (left) shows the principle of measuring crimp and the number of crimps per unit length. A single fiber is pretensioned to a defined tension (0.0025 cN/tex) in an apparatus (e.g., torsion balance, tensile tester, crimp balance, et al.), the length L1 is measured, the number of crimps in the spanned length is counted and the crimp number per 100 mm is calculated [25]. In a simplified version of the measurement, a defined length of fiber L1 is cut, laid in a slide frame, the number of crimps is counted and the crimps per 100 mm is calculated. Here the form and size of the crimps can be determined optically or the image can be electronically measured.

Table 9.11 Concepts for the Evaluation of Crimp (Bulk) Properties

Material property	Calculation	Comment
Crimp before decrimping	$E_1 = \frac{L_1 - L_2}{L_1} \cdot 100\ [\%]$	L_1 = length at force F_1 L_2 = length at force F_2, $F_1 > F_2$ L_3 = length at force F_2 after loading
Crimp after decrimping	$E_2 = \frac{L_1 - L_3}{L_1} \cdot 100\ [\%]$	
Degree of crimp	$k = \frac{L_1}{L_2}$	
Crimp stability	$KB = \frac{E_2}{E_1} \cdot 100 = \frac{L_1 - L_3}{L_1 - L_2} \cdot 100\ [\%]$	Point of inflection in the decrimping diagram
Decrimping force	F	
Bending resistance (acc. to Böhringer)	$B = \sqrt{\frac{W_1}{W_0}} \cdot 100\ [\%]$	W_0 = work of decrimping W_1 = work of decrimping after one load/relax cycle
Bending resistance (acc. to Onderstal)	$B = \frac{W_1}{W_0} \cdot 100\ [\%]$	
Bending stiffness (acc. to Böhringer, Herzog)	$S_B = \frac{st_i \cdot T_t^2 \cdot p}{\rho} 8 \cdot 10^{-7}\ [p \cdot cm^2]$	st_i = initial tensile stiffness T_t = fiber fineness (titer) ρ = filament density p = cross-sectional form factor
Average curvature	$\frac{1}{R} = \sqrt{\frac{2 \cdot W_0}{S_B \cdot L_1}}\ [cm^{-1}]$	

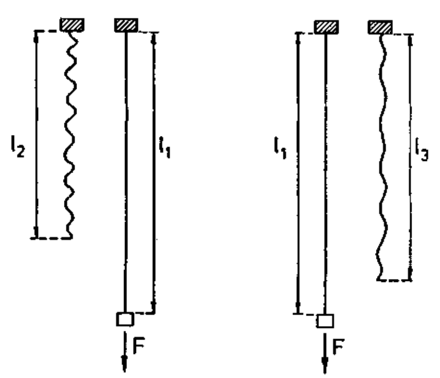


Figure 9.36
Diagram illustrating the measurement of crimps per 100 mm (see Table 9.11)

Typical crimp values are 40...200/100 mm, depending on fiber type, end use, etc. For stuffer box crimping, 80...140 crimps/100 mm is the preferred level. Two additional characteristics, crimp level and crimp stability, can be evaluated using Fig. 9.36.

- The crimp level is defined as the difference between the drawn-out (de-crimped) length L1 and the crimped length L2, divided by the de-crimped length L2, expressed as a percentage
- The crimp stability is based on length measurements before and after one or more mechanical and/or thermal loading(s), and is defined as:

$$\text{crimp stability} = \frac{\text{crimp after loading}}{\text{crimp before loading}} \times 100\%$$

Crimp stability can lie between 100% (full recovery) and 0% (no recovery).

- Textured yarns have greater elongation, elasticity and voluminosity than flat yarns. Here the following textured yarn properties are relevant:

– Crimp elasticity.

The measurements are done predominantly on skeins according to the sequence in Table 9.12. Skeins can be measured either manually on measuring stands or automatically using suitable test equipment, as, e.g., the Texturmat supplied by Textechno [39]. With the equipment described in Section 9.3.5.2, it is possible to measure crimp elasticity continuously. The conditions for developing latent crimp are matched to the further-processing conditions: dyeing requires generation of crimp in water, hot air texturizing requires the same in hot air, etc. Crimp contraction values of texturized yarns in water (Fig. 9.37a) and in hot air (b) [24] are very dependent on the polymer type, and are important for further processing (washing, de-sizing, dyeing, thermofixing). For PET textured yarns, water temperatures below 70 °C (= glass temperature) have very little effect on loss of crimp. In texturizing or friction-drawtexturizing (Fig. 9.38), choice of process parameters enables the crimp contraction to be selected according to the requirements of the end product [31]. False-twist textured yarns can be categorized in terms of further processing requirements as follows:

Crimp $E = 35 \ldots 50\%$–highly elastic (HE) yarns for weaving
$E = 25 \ldots 35\%$–medium elastic (ME) yarns for weaving
$E = 25\%$ –lowly elastic (LE, usually SET) yarns for knitting, curtaining, etc.

— Crimp stability

For textured yarns, the same definition is used as for staple fibers (Fig. 9.36). Further processing can be simulated by mechanical loading of the yarn. According to Fig. 9.39, two textured yarns can have the same crimp stability at low stress but one yarn (b) can have much lower crimp stability than the other (a) when more highly mechanically stressed. A high crimp stability is important in reducing waste during the production of elastic and bulky woven- and mesh fabrics. In practice, crimp stability values of over 50% are deemed necessary.

Table 9.12 Determination of Crimp Elasticity

Measuring method	*Heberlein*	DIN 53840
Hank length Hank titer Pretensioning during hank wrapping	10 m variable 1 cN/tex	variable 2500 dtex 1 cN/tex
Crimp development	Polyamide: 10 min in distilled water at 60...70 °C, followed by centrifuging and drying Polyester: 10 min in distilled water at 60 °C, followed by boiling for 15 min, then centrifuging and drying	Polyamide and Polyester: 10 min in hot air at 120 °C Polyacrilonitrile: 80 °C
First measurement	30 s in water at 60 °C (2 g/l wetting agent) Apply force of 1.8 cN/tex for 1 min Measure L_0	Apply force of 0.01 cN/tex and 0.99 cN/tex Measure L_0 after 10 s
Second measurement	60 min at 60 °C; dry Cool for 60 min Apply force of 0.018 cN/tex Measure L_1 after 1 min	Allow to relax under a load of 0.01 cN/tex Measure L_1 after 10 min
Calculation	$KK = \frac{L_0 - L_1}{L_0} \cdot 100\%$	$E = \frac{L_0 - L_1}{L_0}$

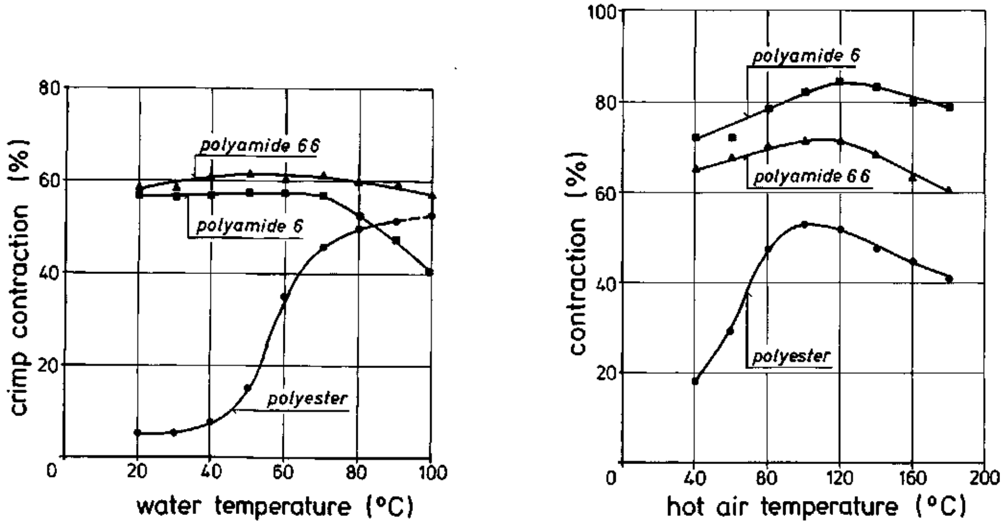


Figure 9.37 Results of crimp contraction measurements

a) as a function of water temperature (Treatment time: polyamide 10 min, polyester 10 min and 15 min at the boil)

b) as a function of hot air temperature (Treatment time: 10 min)

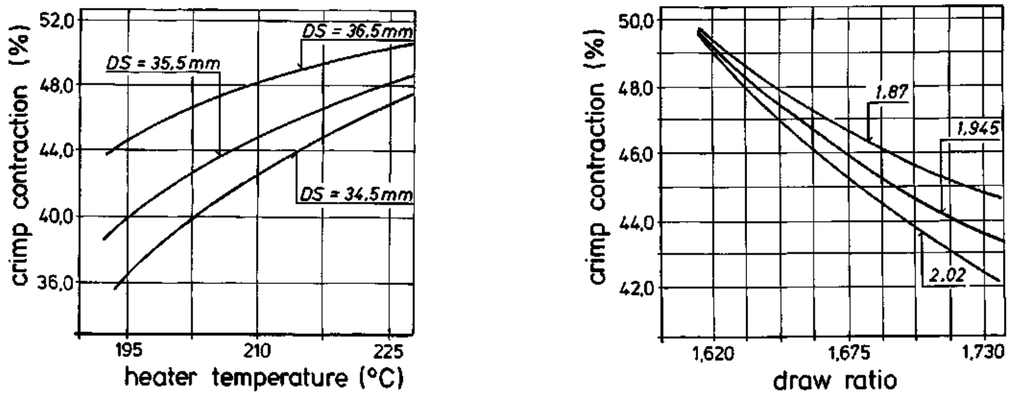


Figure 9.38 Crimp contraction values of texturized yarn
a) as a function of primary heater temperature, with disk spacing (DS) as parameter
b) as a function of draw ratio for POY yarns, with D/Y ratio as parameter

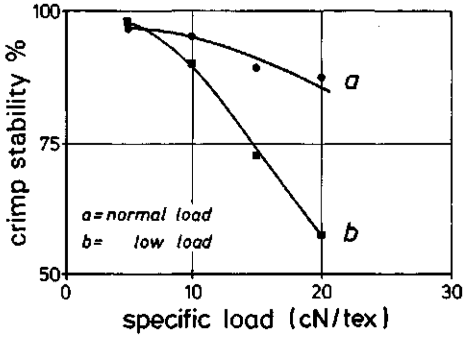


Figure 9.39
Crimp stability of two yarns as a function of load

9.3.2.5 Twist and Intermingling (Tangling)

- Twisting of yarns and cables

  For staple fiber yarns, the insertion of twist is done primarily for tenacity. For continuous filament yarns, twist is inserted in order to obtain certain yarn properties, as well as to improve further processing and reduce waste. Twist insertion influences important technological yarn properties, e.g., tenacity, elongation, modulus, elasticity, fatigue resistance (in tirecords) and filament cohesion; it also affects the optical and tactile properties of fabrics, such as surface constitution, glitter (Fig. 9.92), volume and handle. All these characteristics depend on the level of twist inserted.

  – Determination of twist.

    Twist can be determined by mechanical (e.g., twist counter) or optical methods. In the latter case, the angle of inclination of the single filaments is measured. Table 9.13 lists possible methods for measuring twist in yarns and cables. The de-twisting method is, however, of limited use in the case of highly twisted- or entangled yarns, as it is not always possible to parallelize the single filaments. Entangled and twisted yarns are produced by intermingling prior to the spinning or drawing machine take-up, and are subsequently twisted.

  – Characteristic properties of twisting are the:

    twist number (level) = number of turns/m (of twisted yarn)
    twist direction = spiral inclination of the twisted filaments
    from above, left to below, right = S-twist
    from above, right to below, left = Z-twist (see Fig. 3.32).

Table 9.13 Methods for Determining Twist in Yarns and Cables

Measuring method	Measurement principle	Calculation of twist
Direct untwisting	A defined length of twisted yarn or cable is untwisted under defined tension until the filaments are parallel Clamped length L_o acc. to DIN 53832 Staple yarns: 10, 25, 50 mm Filament yarns: 250, 500 mm	$n = \frac{T \cdot 1000}{L_o}$ (1/m) L_0 = clamped length (mm) (twisted length)
Torsion-break method acc. to Marschik	Twisting of a sample at constant length against the twist direction until it breaks (T_r), and twisting of a second sample in the direction of twist until it breaks (T_v)	$n = \frac{1}{2}(T_r - T_v)$ (1/m) T_r = no. of reverse twists to break T_v = no. of forward twists to break
Tension-sensor method	Untwisting the yarn until $\frac{1}{4}$ of the sliding length of the sample is reached, then twisting back to the original length The clamped Length L_0 is the same for all yarns	None, since the rev. counter gives n (1/m)
Microscopic measurement on staple yarns	Using a microscope, the helix angle φ (relative to the fiber axis) and the yarn diameter d are measured	$n = \frac{\tan \varphi \cdot 1000}{d \cdot \pi}$ (1/m) d = yarn diameter (mm)
Microscopic measurement on monofilament yarns	Using a polarizing microscope, the helix angle φ is measured. Using the measured diameter d, the twist is calculated	$n = \frac{\tan \varphi \cdot 10^6}{d \cdot \pi}$ (1/m) d = yarn diameter (μm)

- Twist coefficient (factor)

  Table 9.14 gives various relationships. The twist coefficient α relates the yarn diameter (fineness in Table) and the twist level (indirectly as angle of inclination of the filaments relative to the yarn axis in Table), and is, in practice, based on the twist level for 1000 tex. This leads to the conclusion that yarns of different fineness having the same twist angle of their filaments will display the same yarn properties. In the lower part of Table 9.14, twist factors are given for cabled yarns and for spindle false twist textured yarns, the latter designed to give a similarity relation between twisted and textured yarns.

- Intermingling (entanglement, tangling, interlacing) of multifilament yarns

  Intermingling is carried out mainly to give the non-twisted multifilament yarn sufficient filament cohesion for further processing, to enable interruption-free take-off from yarn packages at high speed, and—inter alia—to simulate the protective twist obtained in drawtwisting. As this is the case, most synthetic multifilament yarns are nowadays intermingled:
  - flat multifilament (undrawn or drawn) for textile and technical application,
  - texturized yarns for textile products and carpets (tufting)

  Section 7.4.2 describes intermingling using compressed air. The intermingling process results in a loose association of filaments having intermittent plaited sections (cohesion points) which are responsible for the filament cohesion. The number of entanglement nodes per unit length, their mechanical stability and their distribution is determined by:
  - construction details of the tangling jets and their positioning in the yarn path,
  - process conditions (air pressure, yarn speed and tension, etc.),
  - yarn constitution (flat, textured, filament count, spin finish, wet or dry)

  The intensity and structure of intermingling determines the yarn behavior during further processing and the waste levels. Further processing of textured yarns requires tight, mechanically-stable nodes in

Table 9.14 Formulas for Calculating the Twist of Yarns (According to Various Authors)

Formula	Comments	Used for
$n = \alpha\sqrt{\dfrac{1000}{T_t}} = \alpha\left(\dfrac{1000}{T_t}\right)^{0.5}$ $n = \alpha\left(\dfrac{1000}{T_t}\right)^{x}$ $n = q - \alpha\left(\dfrac{1000}{T_t}\right)^{x}$ $n = \dfrac{\tan\alpha}{\pi \cdot d \cdot K}$	acc. to *Köchlin* α = twist coefficient T_t = yarn titer acc. to *Laetsch* x = minimum 0.62 x = maximum 0.75 acc. to *Matthes* q = 0.8 ... 1.2, depending on fiber type x = 0.60 ... 0.64, depending on titer acc. to *Schwarz* α = angle of inclination d = yarn diameter K = constant	Staple, yarn, filament yarns
$n_z = \alpha_z \cdot \sqrt{\dfrac{1000}{T_{tz}}}$ $n_z = c\left(\sqrt{\dfrac{1000}{T_{tz}}} - v\right)$	acc. to *Köchlin* d_z = twist coefficient T_{tz} = effective twisted titer in dtex acc. to *Holtzhausen* c = twist constant v = reduction factor, dependent on the packing	Twists
$n = 800 + \dfrac{305800}{67 + T_t}$ $n \geq \dfrac{30000}{\left(1 + \dfrac{16.6}{T_t}\right) \cdot \sqrt{T_t}}$ $n = 3840 - 10.8 \cdot T_t$ $n = \dfrac{285000}{\sqrt{T_t}}$	acc. to *Heberlein* T_t = yarn fineness in dtex acc. to *Fourné* T_t = yarn fineness in dtex acc. to *Press* T_t = yarn fineness in dtex acc. to *Köchlin* T_t = yarn fineness in dtex	Texturized yarns

order to avoid later snagging of filaments. Sheen, handle, volume and aesthetic are important properties of finished goods. Intermingling nodes can cause, e.g., stripiness and Moiré patterns. To minimize the amount of fabric downgrading, the number of tangling nodes remaining should be small—which is contrary to processing requirements.

- Definition
  According to DIN 60900, Part 1, intermingled filament yarns are single- or multiple, flat or textured, filament yarns which have common cohesion points. Regarding intensity and structure, differences are possible (Fig. 9.40)
  - periodic interlacing
  - statistically random intermingling nodes (a)
  - continuous interlacing
  - partly intermingled, without prominent nodes (b)
  - periodically continuous intermingling
  - combinations of (a) and (b), in accordance with (c)
  - knots formed from some or all entangled filaments of a yarn.
- measurement of intermingling characteristics
  Figure 9.41 gives an overview for determination of the following characteristics:
  - open length (nodal distance) = distance (cm) between consecutive nodes
  - density of nodes = number of nodes/m
  - distribution function of the characteristic values.

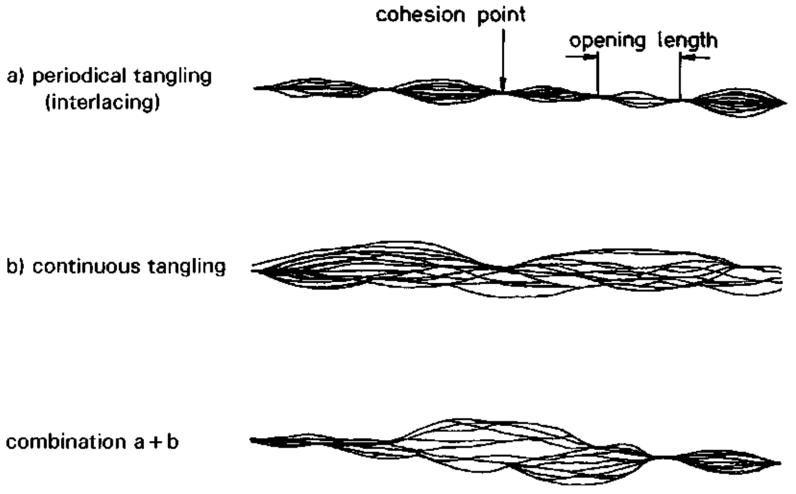
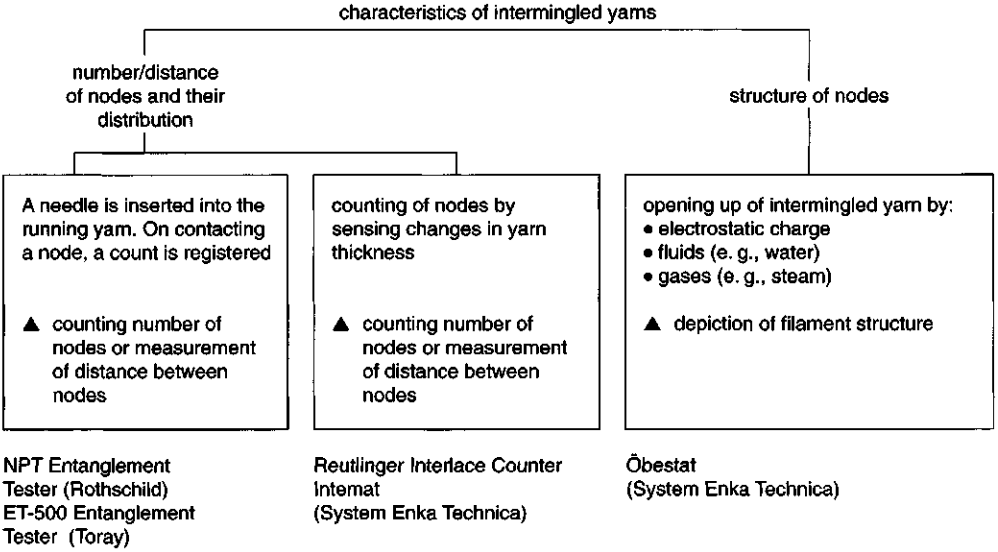


Figure 9.40 Definition of tangled (intermingled) yarn *a*) periodic entanglement *b*) continuous entanglement *c*) combination of *a*) and *b*)



A needle is inserted into the running yarn. On contacting a node, a count is registered

▲ counting number of nodes or measurement of distance between nodes

counting of nodes by sensing changes in yarn thickness

▲ counting number of nodes or measurement of distance between nodes

opening up of intermingled yarn by:

- electrostatic charge
- fluids (e. g., water)
- gases (e. g., steam)

▲ depiction of filament structure

Figure 9.41 Characteristics of intermingled yarn

Figure 9.42 [22] shows the frequency distribution of internodal distance of three textured yarns. A yarn having a narrow distribution of internodal length has advantages for further processing: sizing and/or twisting is not required for a weaving weft yarn, but—on the other hand—fabric downgrading increases, and special equipment may be required. Figure 9.43a shows additionally that the maximum number of nodes is reached with low titer/low air pressure (55 dtex–2.5 bar), and that the number of nodes decreases with increasing titer, in spite of increasing the air pressure.

- Stability of the nodes

  In further processing, the tangled yarn experiences considerable applied tension, sometimes of a periodic nature, and unstable nodes can be pulled out, leaving the yarn more open. The node stability is defined as:

$$\text{node stability} = \frac{\text{number of nodes after processing}}{\text{number of nodes before processing}} \times 100\%$$

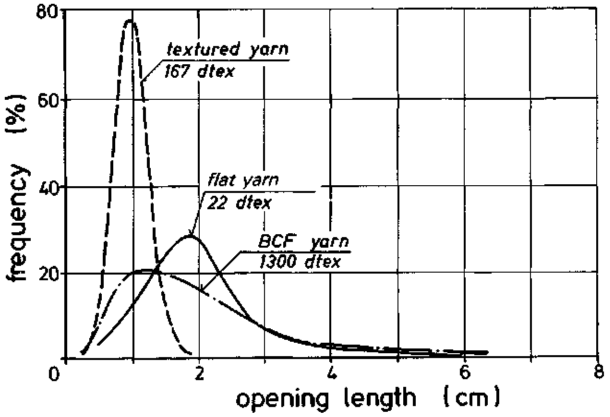


Figure 9.42
Opening length frequency distribution for various intermingled yarn types

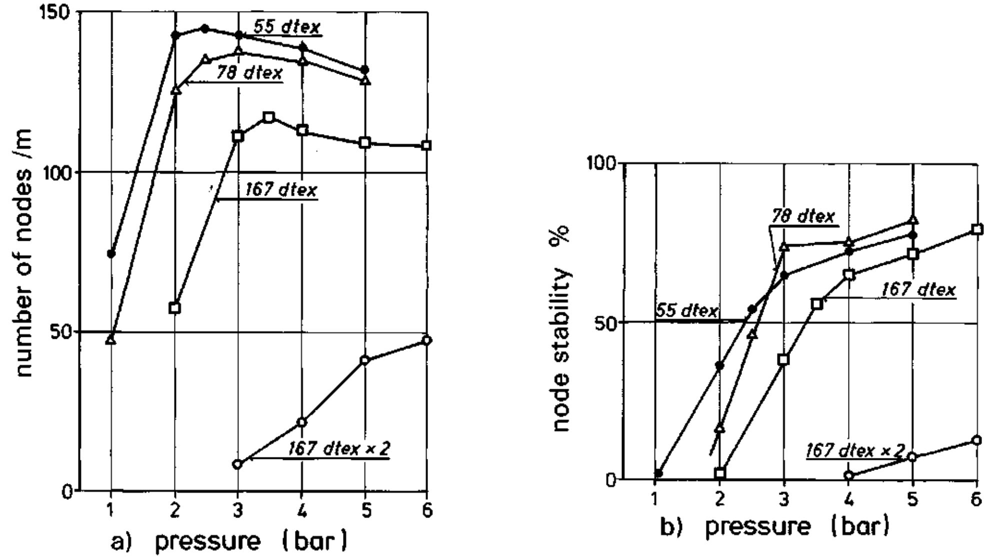


Figure 9.43 Number of cohesion points (nodes) (*a*) and cohesion point stability (*b*) of intermingled yarns as a function of air pressure and multifilament titer

The determination of node stability requires a second measurement, which will simulate the processing conditions (yarn path, mechanical loading). Figure 9.43b shows an example. From practical experience, knot stability should be greater than 50% (0% = nodes totally removed; 100% = nodes totally retained).

9.3.3 Mechanical Properties

By mechanical properties one understands characteristic material properties derived from dynamometric drawing or from elastic recovery testing.

Modern equipment for tensile testing works with constant rate of extension in order to ensure that samples having different force/elongation characteristics are treated rheologically equivalently, thus ensuring comparability of sample characteristics.

Fig. 9.44 shows a schematic of tensile testing.

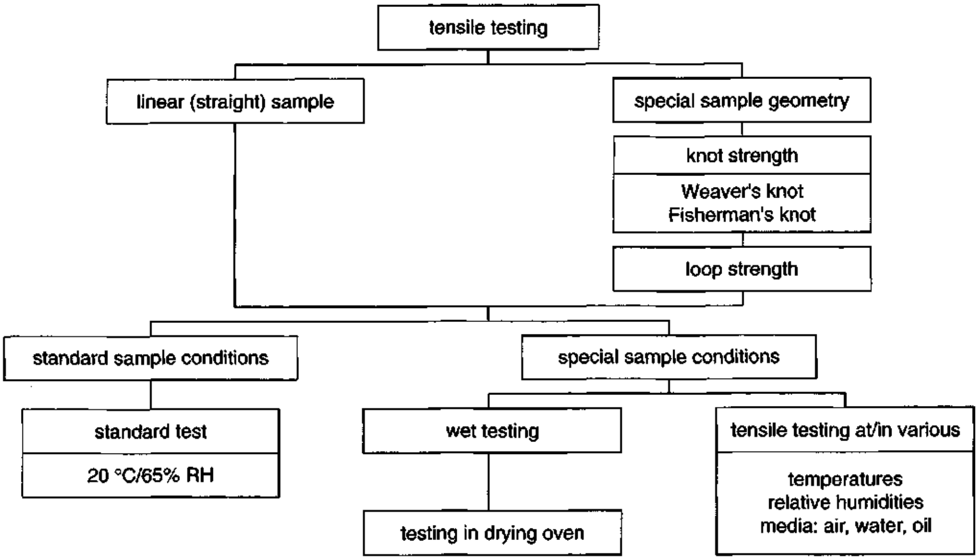


Figure 9.44 Schematic of tensile testing

- Principle of tensile testing:
  The sample, pre-tensioned between two jaws, is drawn to break in the tensile testing mode. For elasticity measurements, the pre-tensioned sample is subjected to a defined loading program, e.g., cyclic tensioning and relaxation.

A tensile tester comprises: clamping jaws to hold the sample and a drive to pull the jaws—force-measuring cell with amplifier—recorder for force/elongation diagram (recorder or computer) and possibly automation of the test cycle. Some force/extension diagrams of man-made fibers are shown in Fig. 9.45 [28]: further examples are found in the Fiber Table (Chapter 11) and in Chapter 2.

Knowledge of these diagrams is important for fiber production, processing and further processing, as Fig. 9.46, for example, shows for the effect of further processing speed. For all man-made fibers, the effect of temperature during tensile testing is important (hot drawing, texturizing, sizing, etc.). This also

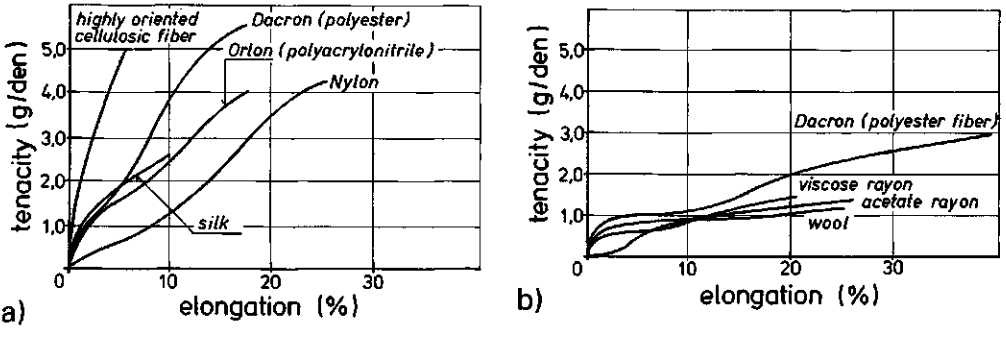


Figure 9.45 Tenacity/elongation curves for (*a*) "silk-like" and (*b*) "wool-like" man-made fibers
(For details of modifying PET fibers to resemble natural- or chemical fibers, see Fig. 2.87)

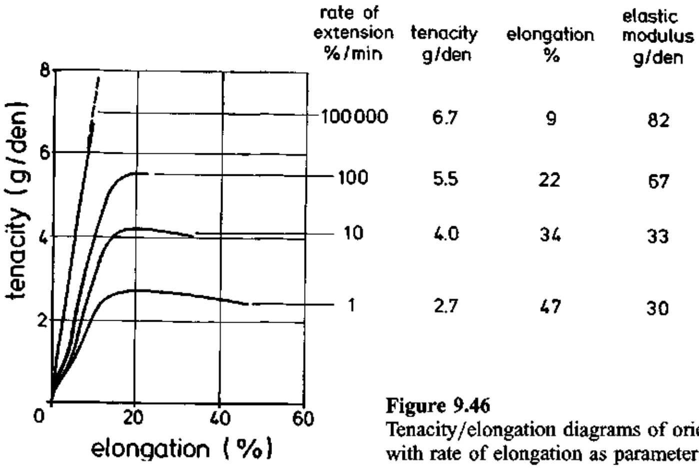


Figure 9.46
Tenacity/elongation diagrams of oriented linear polyethylene (HDPE), with rate of elongation as parameter [26]

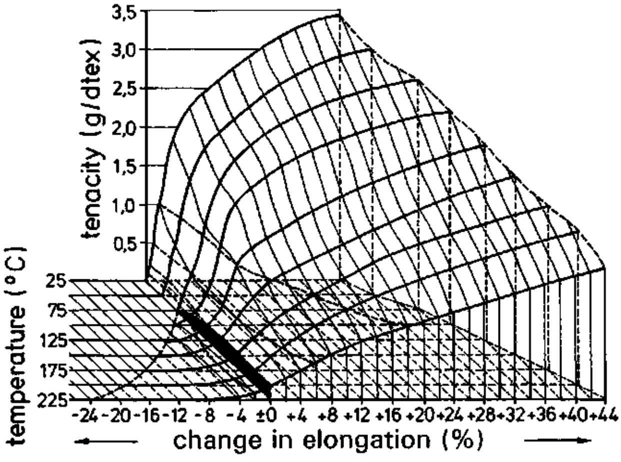


Figure 9.47
Tenacity/elongation curves of a PET multifilament yarn measured at various temperatures

applies during application (Fig. 9.47). In order to maintain the constancy of results, it is necessary to observe the conditions listed in Table 9.15 [24]. Table 10.2 gives calculation factors for converting between the SI System and other units still in use.

9.3.3.1 Tensile Testing and Properties Derived Therefrom

The tenacity/elongation curve gives significant information for process control, further processing and the end product properties. The elastic modulus (E-modulus) belongs to the tensile properties. There are three formulations for the E-modulus: the initial tangent to the curve passing through zero (initial modulus), the straight line passing through zero and a point on the curve at a specified elongation (secant modulus) and the tangent at any desired point on the curve (tangent modulus). For textile applications, the initial modulus is usually the most important; the same applies for textured yarns, where it is termed the crimp modulus (Fig. 9.48).

The secant modulus is mainly used for technical yarns (e.g., as M_{10} in Fig. 2.61 for the modulus at 10%); it gives the average slope of the curve over a given region. The tangent modulus corresponds to the first derivative ($d\sigma/d\varepsilon$) of the tenacity (σ)/elongation (ε) curve, as, for example, in Fig. 9.49 [27]. Here the difference between a drawn (only) and a drawn and thermoset PA6 fiber can clearly be seen. The curve can be divided into three sections [12,13,27]:

Table 9.15 Overview of Various Influencing Factors in Tensile Testing (left) and the Derived Characteristic Values (right) (ρ = Density)

Influence	Source of the effect
Type of tester Accuracy Sensitivity and time constant of the display Type of jaw Jaw geometry Slip in the jaws	Test equipment
Sample conditioning time Tension during conditioning Movement towards moisture equilibrium Relaxation during conditioning Test length Rate of extension or rate of force increment of the sample Pretension Type of test (knot-, loop strength)	Test conditions
Testing medium: air, gas, water, steam Degradation during testing Temperature of the test medium Relative humidity	Environmental conditions

Concept	Symbol	Formula
Pretension, based on linear density	R_V	$R_V = \frac{F_V}{T_{t_V}}$
Tenacity, based on linear density	R	$R = \frac{F}{T_{t_V}}$
Max. tenacity, based on linear density	R_H	$R_H = \frac{F_H}{T_{t_V}}$
Tenacity at break, based on linear density	R_B	$R_B = \frac{F_B}{T_{t_V}}$
Pretensioning stress	σ_V	$\sigma_V = \frac{F_V \cdot \rho}{T_{t_V}}$
Tensile stress	σ	$\sigma = \frac{F \cdot \rho}{T_{t_V}}$
Max. tensile stress	σ_H	$\sigma_H = \frac{F_H \cdot \rho}{T_{t_V}}$
Tensile stress at break	σ_B	$\sigma_B = \frac{F_B \cdot \rho}{T_{t_V}}$
Elongation	ε	$\varepsilon = \frac{\Delta L}{L_V} \cdot 100$ [%]
Elongation at max. force	ε_H	$\varepsilon_H = \frac{\Delta L_H}{L_V} \cdot 100$ [%]
Elongation at break	ε_B	$\varepsilon_B = \frac{\Delta L_B}{L_V} \cdot 100$ [%]

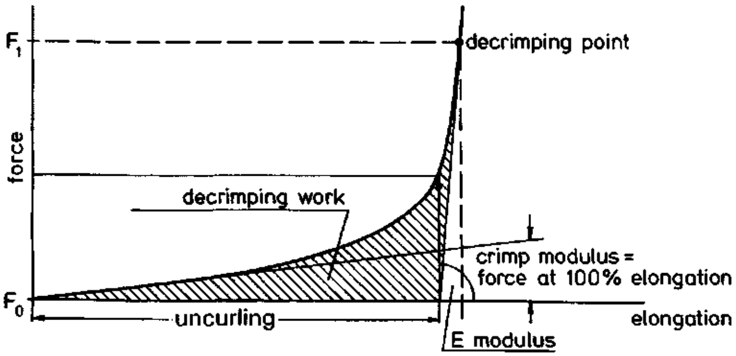


Figure 9.48 Schematic diagram for defining crimp modulus and other relevant properties

stretch region 1: – quasi-elastic extension region.

stretch region 2: – predominantly loading of the tie molecules linking the crystalline and amorphous regions. With increasing modulus, further orientation of the still intact tie molecules and the crystalline regions occur.

stretch region 3: – region of irreversible plastic flow (sliding); slipping of the tie molecules in the amorphous and crystalline phase, terminating in fiber breakage.

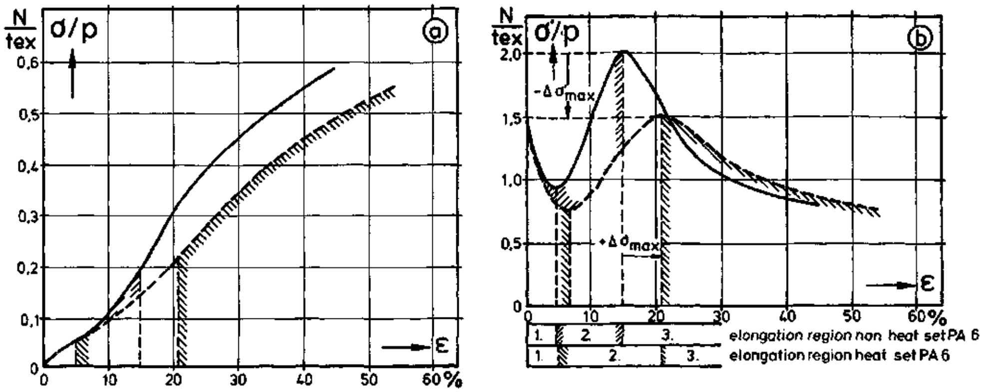


Figure 9.49 Tenacity/elongation characteristics of oriented PA 6

a) $\sigma(\varepsilon)/\varrho$; drawn, non heat setted

b) $\sigma'(\varepsilon)/\varrho$: drawn and thermally treated yarn (heat set in saturated steam without tension at 130°C). The shaded region shows where the gradient changes sign

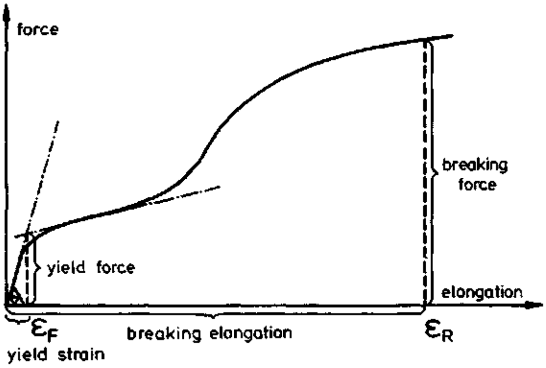


Figure 9.50 Characteristic properties derived from *tensile* testing

Initial modulus	$M_A = \tan\alpha$	
Yield elongation (strain)	ε_F	Predominantly orientation-dependent
Yield force	P_F	
Yield tenacity	$\sigma_F = \dfrac{P_F}{\text{Initial titer}}$	
"True" yield tenacity	$\sigma_F(1+\varepsilon_F) = \dfrac{P_F}{\text{Yield titer}}$	
Elongation at break	ε_R	Orientation- and crystallinity-dependent
Breaking load	P_R	
Tenacity at break	$\sigma_R = \dfrac{P_R}{\text{Initial titer}}$	
"True" tenacity at break	$\sigma_R(1+\varepsilon_R) = \dfrac{P_R}{\text{Titer at break}}$	
"Quality factor"	$T\sqrt{\varepsilon}$	
Work to break	$\int_0^{\varepsilon_R} \sigma \, d\varepsilon$	

These relationships can be used to show which structural parameters affect the measured fiber characteristics obtained during tensile testing. Figure 9.50 defines these: the orientation predominantly affects the characteristics of the initial phase of stretching, while both orientation and crystallinity affect the latter part of the force/elongation diagram [12,13]. Figure 9.51 gives an overview of the initial modulus of textile fibers.

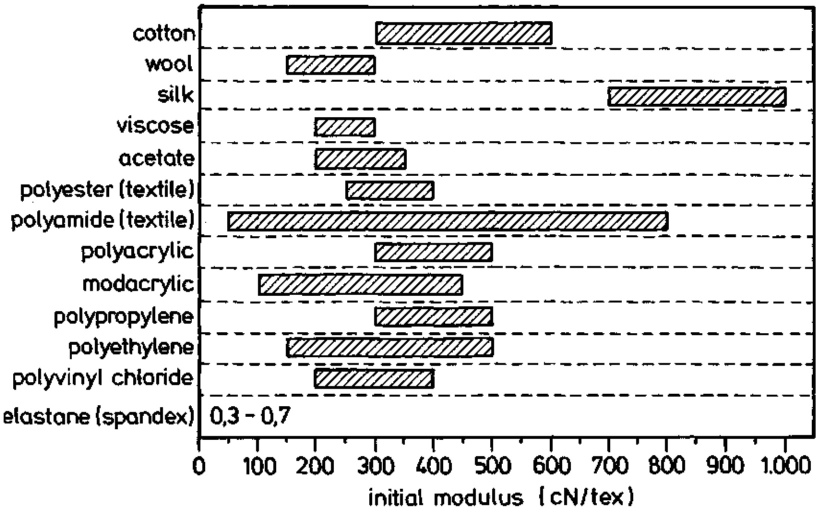


Figure 9.51
Initial modulus ranges of various fiber types

9.3.3.2 Tenacity in the Non-Axial Direction

In addition to the (usual) tenacity in the fiber axial direction, tenacity in the non-axial direction is of great importance for some processes and end uses.

Sewing thread yarns are usually tested in the form of a loop (Fig. 9.52), and fishing net yarns in the form of a knot. The respective tenacities are usually determined in this way [2].

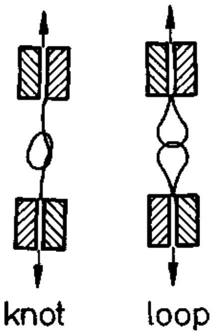


Figure 9.52
Yarn geometry in instrument clamps for loop- and knot strength testing

- Knot tenacity (knot strength):
  For knot tenacity, the following knots are employed in sequence, corresponding to the stage in the fishnet production process: overhand knot (S or Z direction)—weaver's knot—fisherman's knot. Knot tenacity is quoted as a percentage of the normal tenacity.
- Loop tenacity (loop strength).
  Two pieces of the same yarn are interlooped, pretensioned and drawn to breaking point on the tensile tester. The breaking load is divided by a factor of two, since the load is borne by two yarns. Loop

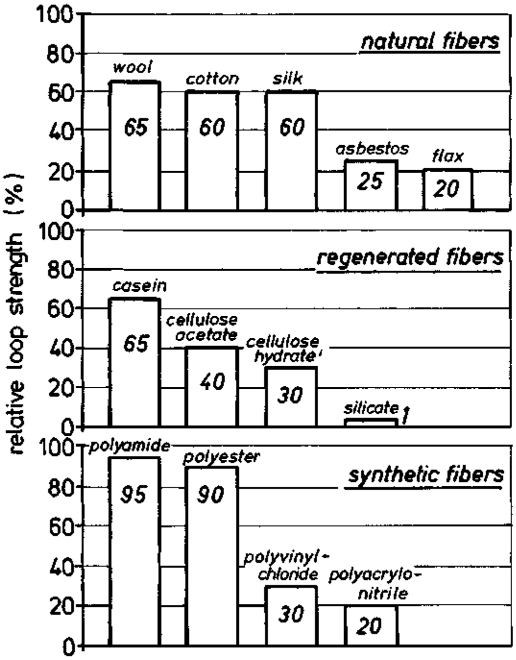


Figure 9.53
Loop strength of various fiber types

tenacity is similarly quoted as a percentage of the normal tenacity. Figure 9.53 shows some comparative values.

9.3.3.3 Elastic Properties

During production and further processing, external forces act on the fibers and yarns, tending to deform them. Intermolecular binding forces, however, oppose these actions, and seek to restore the fiber to its original condition. By the term elasticity one understands the more or less complete recovery of the fiber from its deformation once the deforming force has been removed. One also differentiates according to the nature of the deformation:

tension or compression – tensile or compressive elasticity
bending – bending elasticity
torsion – torsional elasticity

Knowledge of elastic properties is important for further processing and particularly so for selection of the polymer with regard to the end-use application.

- Elastic tensile properties
  The above-mentioned concepts, definitions and knowledge can be partly or wholly used for measurement of elastic properties under different loading conditions. Figure 9.54 gives a systematic overview of elasticity tests described in DIN 5385. The multiplicity of test variations is explained by the multiplicity and complexity of the demands made in further processing and application, e.g., hundreds of load cycles in weaving, repeated stretching of clothing across the knee or the repeated flexing of automobile tires, etc. As an example, Figure 9.55 shows a single-loading elasticity test having fixed extension limits and immediate jaw return. Using diagram (b), frequently-used elasticity concepts can be defined:

  total elongation $\varepsilon_{\text{total}}$ — the total elongation experienced, either as the limit set in an elongation-specified test, or the elongation achieved in a test where a force limit is set.

number of cycles	clamp (jaw) movement	conditions imposed
single cycle	immediate return	constant elongation limits constant force limits
	with pauses at clamp return	constant elongation limits constant force limits
multiple cycle	immediate return	constant elongation limits constant force limits
	immediate return; large number of cycles until constant hysteresis is reached	constant elongation limits constant force limits

Figure 9.54 Schematic of elasticity tests according to German Standard DIN 53835

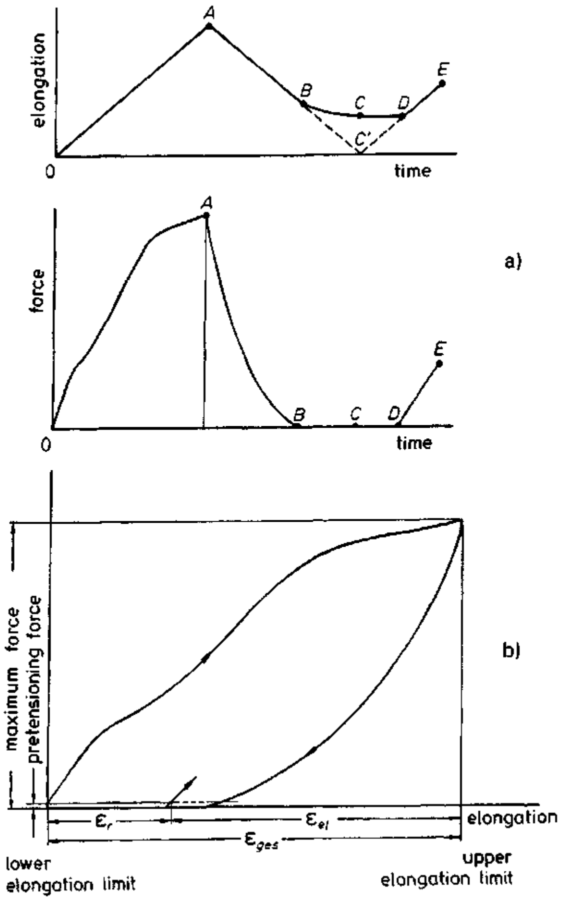


Figure 9.55
Composition and combination of the phases of an elasticity test (a)
Point *0*: Start of the test
Curve *0A*: Elongation of the sample at constant rate of extension (above) and resulting force (below)
Point *A*: Upper (selected) elongation limit and corresponding force are achieved. Reversal of the clamp begins.
Curve *AB*: Sample can relax at the same rate of extension as in the loading cycle. Force reduces.
Point *B*: Sample is completely relaxed. Recovery cycle shows change of speed.
Curve *BC*: Further time-retarded recovery of the deformation; sample shows residual elongation.
Point *C*: A fully-elastic material would show no residual elongation, i.e., Point *C* would coincide with Point *C'*.
Curve *CD*: Further possibility of deformation recovery
Point *D*: End of the recovery. Extension and loading start again.
Curve *DE*: Elongation of the sample as per previous cycle.

By combining the above force and elongation curves in a co-ordinate system, one obtains a characteristic diagram (*b*) for the elasticity test. Using this diagram, the concepts and definitions of tensile elastic behavior can be established

residual elongation ε_r — the elongation remaining when the load is removed and the specified pretension is attained.
elastic elongation ε_{el} — the difference between the total elongation ε_{total} and the residual elongation ε_r.
elastic elongation ratio D = degree of elasticity — the ratio of elastic elongation to total elongation.

The work of deformation can also be used to specify the elastic properties. According to Fig. 9.55, the total work comprises the reversible, elastic work and the irreversible, non-recoverable work. The ratio of non-recoverable work to total work is the hysteresis loss. This concept is applied particularly to elastane (spandex), wrapped yarn, textured yarn, etc. Figure 9.56 shows the degree of elasticity for some important fibers, quoted for 2% and 10% extension.

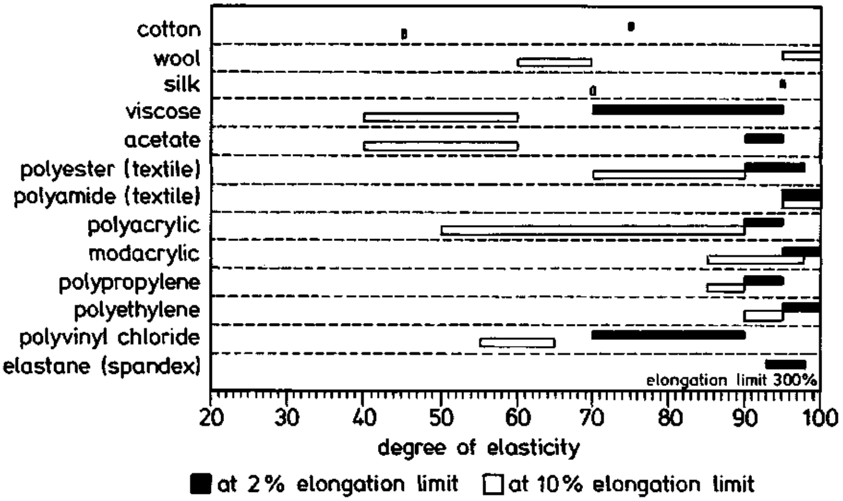


Figure 9.56 Degree of elasticity ranges of various fiber types

9.3.4 Shrinkage and Shrinkage Force

Thermal or hydrothermal treatment of man-made fibers can result in changes in length (usually reduction in length). In the case of fibers and yarns, this is termed "shrinkage", while for fabrics the term "dimensional change" is used. To avoid dimensional changes, the article can, e.g., be held to length and thermoset, in which case internal restoration forces arise, the shrinkage force. Both shrinkage and shrinkage force are characteristics which need to be added to the description of the temperature and tension-related behavior of man-made fibers. Factors affecting shrinkage are given in Table 9.16; these again depend on fiber structure.

9.3.4.1 Shrinkage of Fibers and Yarns

- The shrinkage mechanism can be explained in terms of structural models involving molecular chains. In the case of PET, the two phase fibril model (Fig. 9.12) can be invoked. A drawn man-made fiber consists of fibrils comprising alternating ordered (crystalline) and non (or weakly)-ordered (amorphous) regions. A molecular chain can wander through both regions, in which case it is termed a tie molecule. It is possible for a chain to exit and re-enter the same crystalline region: a back-folding of the chain [30]. Figure 9.57 can be used to explain shrinkage in the molecular structure [29]: on heating to above a fiber-specific temperature (glass temperature, preferably termed glass transition temperature or second order transition temperature), the molecules in the amorphous region become more mobile, thus freeing restoration forces which had previously been blocked at lower temperature. The molecules then strive to attain the lowest energy state and coil up, i.e., they shrink (state C). On

Table 9.16 Factors Influencing Shrinkage

	Source
Polymer Chemical constitution Crystallinity Orientation	Material
Technology (single stage) Temperature Drawing Time Medium Heating rate	Process
Temperature Medium dry (air) moist (steam) wet (water) Time, Conditioning in medium Measuring rate	Treatment testing

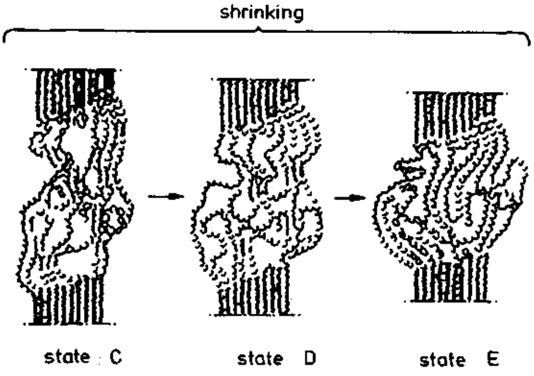


Figure 9.57
Intermediate crystalline stages during shrinkage
C State when allowed to shrink above T_g but below T_{eff}
D State when allowed to shrink at T_{eff}
E State when allowed to shrink above T_{eff} but below T_S

further temperature increase, the less thermally stable crystallites melt, followed by the more thermally stable network points, whereby the amorphous regions become fully disoriented and kink and shrink (state E). Further temperature increase causes melting of the remaining crystalline regions (except for liquid crystals).

The various highly-oriented molecules in the amorphous region of a fiber largely determine the shrinkage behavior [30]. Designating f_a = amorphous orientation factor, V_c = crystalline volume fraction and $(1 - V_c)$ = amorphous volume fraction, the shrinkage can be plotted as a function of $(1 - V_c)f_a$ as, e.g., in Fig. 9.58 for the hot air shrinkage of PET at 160 °C. The shrinkage is therefore a function of both the amorphous orientation and the degree of crystallinity. In general, shrinkage and orientation increase with increasing processing tension. Contrarily, increasing processing temperature results in a reduction of shrinkage and an increase in crystallinity [31]. Accordingly, with increasing spinning speed, the speed-induced crystallinity increases, thereby initially stabiliz-

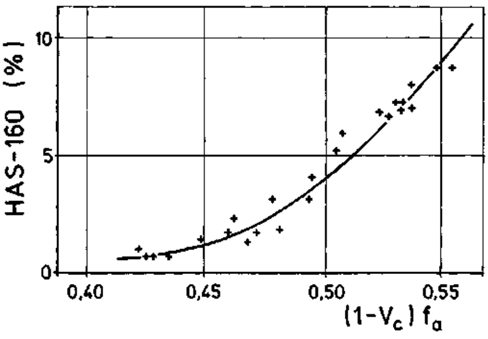


Figure 9.58
Hot air shrinkage (HAS) of a fiber at 160°C as a function of $f_a \cdot (1 - V_C)$

ing the shrinkage. Increasing the spinning speed yet further, the increasing crystallinity reduces the shrinkage more strongly. In the example (Fig. 9.59), shrinkage reaches a maximum at a spinning speed of 2100...2200 m/min.

Methods of measuring shrinkage

The preceding considerations (but not the actual values) are, in general, valid. Using different test methods, however (shrinkage with or without tension, thermomechanical analysis or continuous shrinkage force measurement), different results are obtained, results which can, however, be generally given a common interpretation. The test conditions should be as closely matched to the processing conditions to be used as possible, thus:

boiling water (boil-off) shrinkage for washing and dyeing treatments
hot air shrinkage for heat setting and drying processes
shrinkage in saturated steam for steam processing.

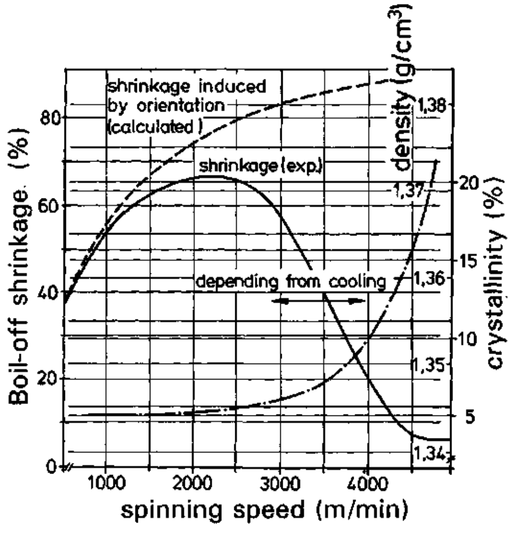


Figure 9.59 Boil-off shrinkage of spun PET fiber as a function of spinning speed. (Spun shrinkage = orientation-induced shrinkage minus crystallization-blocking effect)

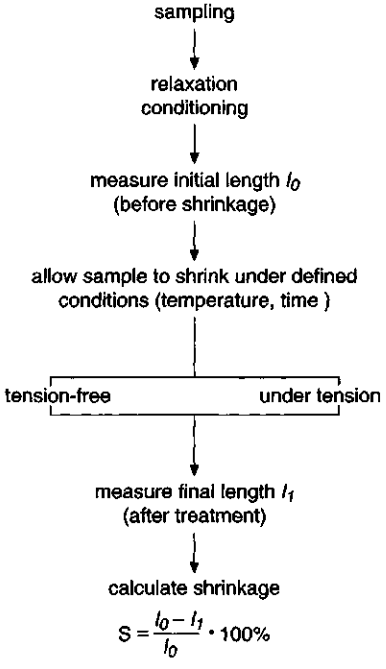


Figure 9.60 Shrinkage testing sequence

Table 9.17 Methods for Determining Boiling Water Shrinkage, Hot Air Shrinkage and Shrinkage in Saturated Steam

Type of Shrinkage	In boiling water	In hot air	In saturated steam
Hank length	10 m for yarns ≤250 dtex; 1 m for yarns >250 dtex		
Pretensioning during wrapping	Flat yarns: 0.5 cN/tex Textured yarns: 2.0 cN/tex		
Relaxation time before treatment	24 h in normal climate		
1st length measurement at a pretension1) of	Flat yarns 0.5 cN/tex; Textured yarns 2.0 cN/tex Measure length l_1 on measuring stand after 10 s		
Thermal treatment	Boil in cotton bag	15 min, 190 °C hanging freely	PA 6: 3 min, 125 °C PA 66: } 3 min, 130°C PET: }
Relaxation time after treatment	24 h in normal climate		
2nd length measurement at a pretension1) of	Flat yarns 0.5 cN/tex; Textured yarns 2.0 cN/tex Measure length l_2 on measuring stand after 10 s		
Calculation of shrinkage	Shrinkage $= \frac{l_1 - l_2}{l_1} \cdot 100\%$		

Table 9.17 gives an overview of commonly used shrinkage methods; Figure 9.60 describes the test method sequence. Shrinkage can be measured tensionlessly or under tension (the latter for tirecord or HMHT PET staple fiber), continuously or before and after thermal treatment (tirecord).

- Shrinkage measurement results are given in Figs. 9.61–9.66.

According to Fig. 9.61, the three main shrinkage methods (boiling water, hot air, saturated steam) give different results with PA 6, PA 66 and PET. Hot air shrinkage is most dramatic for PET, much weaker for PA 6, but PA 6 shrinks the most in saturated steam (Fig. 9.61).

Up to a temperature of ca. 100 °C, the shrinkage of PET (without swelling) is independent of the heating medium, but at temperatures above this, hot air shrinkage is lower than shrinkage in oil (Fig. 9.62) [33]. According to Fig. 9.63, an increase in tension during shrinkage in hot air reduces the shrinkage, i.e., disorientation becomes smaller. When the externally-applied load exactly equals the internal restoration forces, the shrinkage becomes zero (Fig. 9.63). Treatment time and rate of heating have, according to Fig. 9.64, an effect on the development of shrinkage [24]. A shock-temperature treatment results in shock-shrinkage. Here, in general, the crystallization proceeds more slowly than disorientation, particularly if the heating rate is lower. Orientation and crystallization developed during thermal treatment can be measured using birefringence (Fig. 9.65). Density measurement enables crystallinity to be calculated [33] (Fig. 9.66). Additionally, the birefringence reduction (= disorientation relative to the starting material) can be established. The disorientation arising with increasing temperature depends on the heating medium, as can be seen in Fig. 9.62. During shrinkage at constant length, the orientation hardly changes. This is of importance in fiber production or later thermal treatment. When measured after shrinkage, filament density increases almost linearly with treatment temperature, i.e., the crystallinity increases. This enables shrinkage to be stabilized after a correctly-determined thermal treatment. Heat setting at constant length = dimensional stability during further processing.

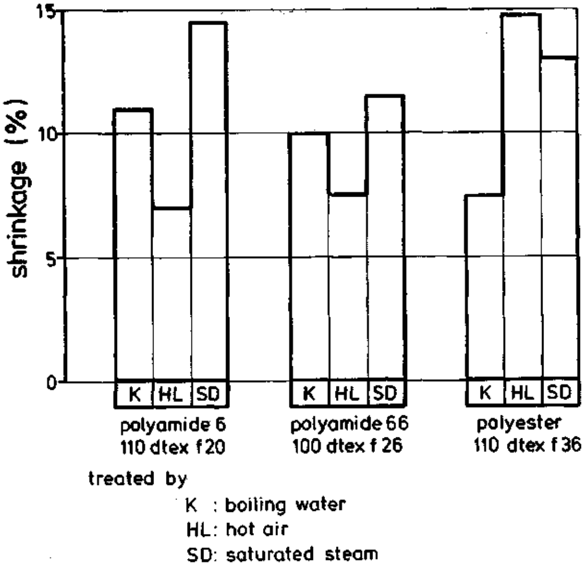


Figure 9.61 Results of shrinkage measurements on PA 6, PA 66, PET; K = Boiling water (boil-off) shrinkage, SD = Saturated steam shrinkage, HL = Hot air shrinkage

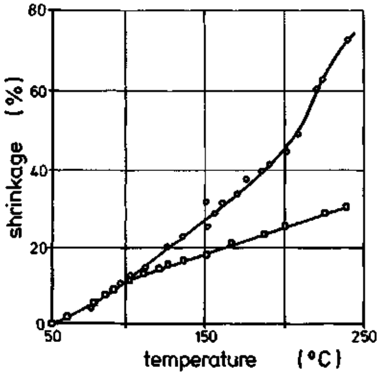


Figure 9.62 Shrinkage of a polyester fiber as a function of shrinkage temperature
☐ Shrinkage after 10 min in a hot air oven
○ Shrinkage after 1 min in oil

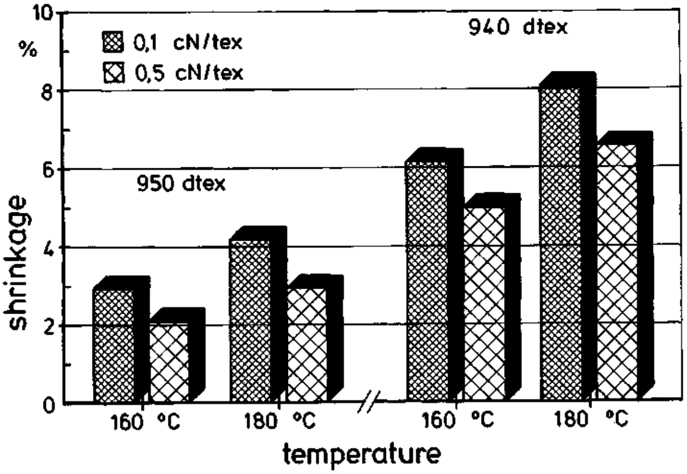


Figure 9.63
Effect of temperature and tension on shrinkage (left: low shrinkage yarn; right: high shrinkage yarn)

9.3.4.2 Shrinkage Force

- Mechanism of shrinkage force
  The shrinkage force determines the magnitude of the shrinkage achievable, as the shrinkage force developed acts in opposition to the externally-applied forces on the fiber. A fiber cannot therefore shrink freely, as the external forces counteract such shrinkage (Fig. 3.66). In general, shrinkage is possible when the internal shrinkage forces are greater than the externally-applied forces on the fiber; the converse is also true. The same molecular relationships apply for shrinkage force as for shrinkage.

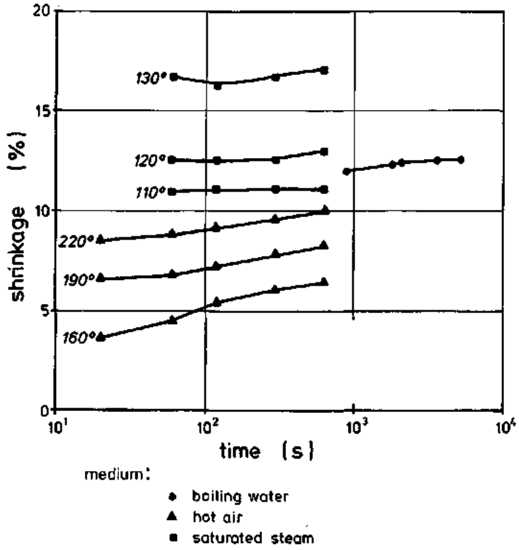


Figure 9.64 Effect of shrinkage time and -temperature on the shrinkage of a polyester yarn

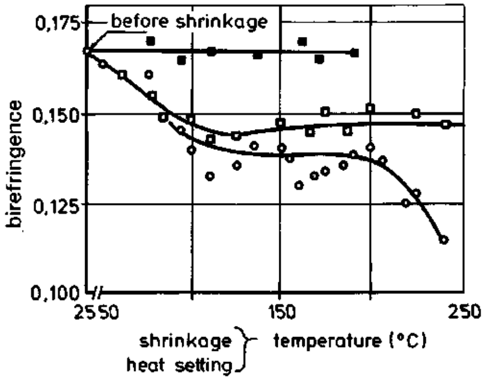


Figure 9.65 Birefringence of a polyester yarn after shrinkage and heat setting at constant length as a function of shrinkage- and heat setting temperature
■ in oil, at constant length
□ with shrinkage in air
○ with shrinkage in oil

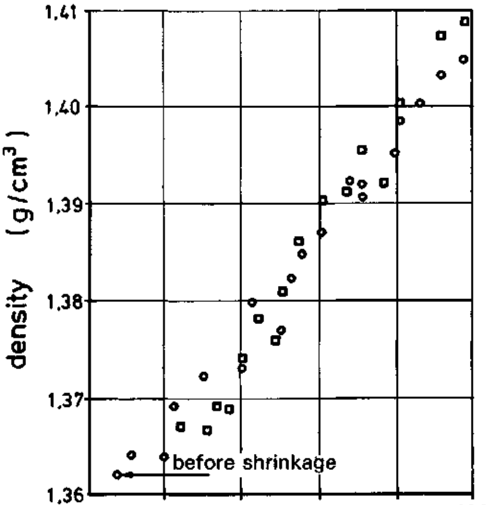


Figure 9.66
Density of a polyester yarn as a function of the shrinkage temperature

Above the glass transition temperature, the chain segments become mobile, changing the amorphous regions from ordered (oriented) to less ordered (unoriented), thereby releasing the "frozen-in" tensions (restoration forces). If the sample length is held constant (= shrinkage blockage), the released restoration forces (= shrinkage forces) can be recorded (Fig. 9.67 for PET). Shrinkage and shrinkage force are minimal below the glass transition temperature. With increasing temperature, shrinkage force rises to a maximum (F_{SH}, Fig. 9.67), corresponding to the maximum disorientation of the amorphous region (Fig. 9.57, state E). Further temperature increases then continuously release more blocked forces (thermal relaxation): the continuous melting of the crystalline regions

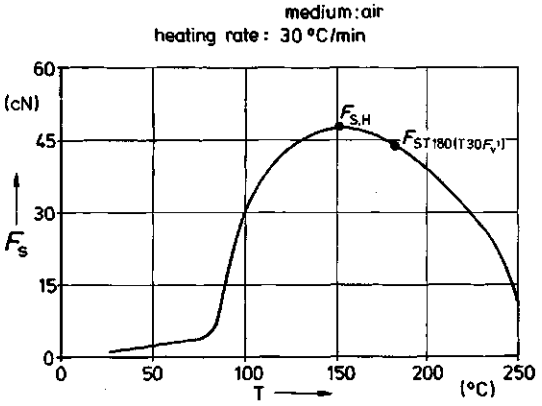


Figure 9.67
Shrinkage force (F_S) as a function of shrinkage temperature (T) for a 15 tex f34 PET yarn (medium: air; heating rate: 30°C/min)

begins. By this action, the binding points of the tie molecules are successively weakened: they lose their position in the crystallites by sliding action. In the melt region there is complete flow; the locked-in tensions tend to zero.

The shrinkage force is a necessary piece of information for a comprehensive description of the shrinkage behavior of fibers. Figure 9.68 therefore shows the shrinkage force as a function of hot air shrinkage at constant temperature measured in a tension-free state. There is no clear relationship between high shrinkage and high shrinkage force. The tangent to the shrinkage curve (shrinkage modulus) does, however, enable predictions to be made concerning expected shrinkage behavior as a function of the temperature and applied tension during further processing.

- Methods of measuring shrinkage force.

The apparatus used needs to be able to measure the force/elongation curves, and needs to be fitted with a temperature-controlled heating zone for the sample. Shrinkage force measurement and thermomechanical analysis require the following steps:

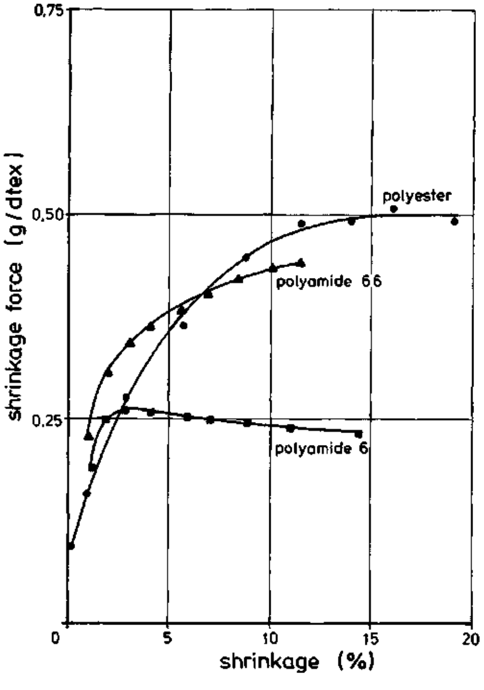


Figure 9.68
Shrinkage force as a function of shrinkage for PA 6, PA 66 and PET

- Preheating and regulation of the measuring zone at the required measuring temperature.
- Clamping the sample and pre-tensioning.
- Insertion of the sample into the temperature zone by positioning of the oven.
- Recording of the shrinkage force/time curve, and evaluation of results.

On insertion of the sample into the pre-set temperature zone, very high and undefined heating rates can occur. These must be taken into account when interpreting the results. Figure 9.69 shows shrinkage force as a function of time for a given PET yarn, with shrinkage temperature as parameter. As is to be expected (because of the higher heat transfer rate), samples at higher temperature reach their maximum shrinkage force (F_S) earlier; the times to reach maximum shrinkage force can differ by many decades. At higher temperatures, relaxation occurs after attainment of maximum shrinkage force, i.e., the melting of the crystalline regions begins with the loss of the binding points of the tie molecules. These material-dependent shrinkage/time curves are relevant for residence time selection in thermal treatment processes.

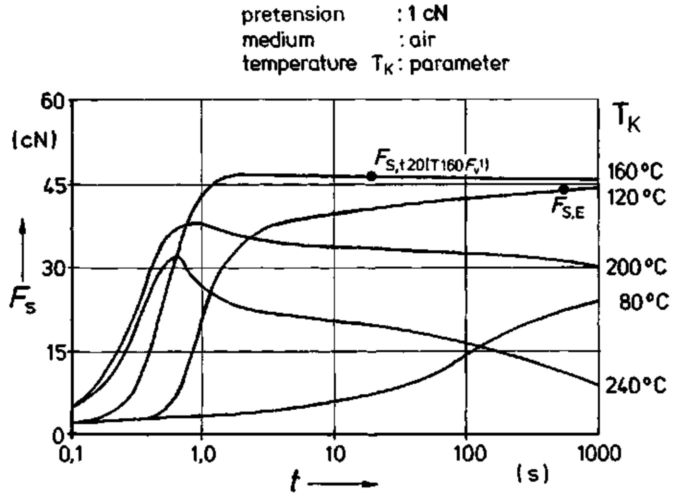


Figure 9.69
Shrinkage force (F_S) as a function of shrinkage time (t) for a 15 tex f34 PET filament yarn. Pretension = 1 cN; shrinkage medium: air; shrinkage temperature = T_K (parameter)

- Shrinkage force measurement results
  Shrinkage force/time diagrams of three materials are shown in Fig. 9.70 at 180 °C under different tensions. At low tension (= external force), the shrinkage force tends to a constant value after only 4 ... 7 s, after which the force decreases asymptotically to its final value after about 10 ... 20 s. From this it follows that tension equilibrium is only attained when the external forces counterbalance the internal, "frozen-in" forces [34]. Figure 9.71 shows the dependency of shrinkage force on externally-applied pretensioning. These cut the 1 : 1 equivalence line at a definite pretension: the equilibrium shrinkage force corresponding to the "effective tension" at the given temperature, i.e., the prevailing blocking tension.

9.3.5 Uniformity of Yarns and Fibers

Non-uniformity (= fluctuation of characteristic values, DIN 53829) can be of an internal or external nature (Fig. 9.72). External uniformity is often judged visually (sometimes using auxiliary agents) or can be recognized in textile fabrics [35]. Internal uniformity is principally characterized by the properties of the fiber structure, and is not usually directly visually recognizable. It can, however, affect color,

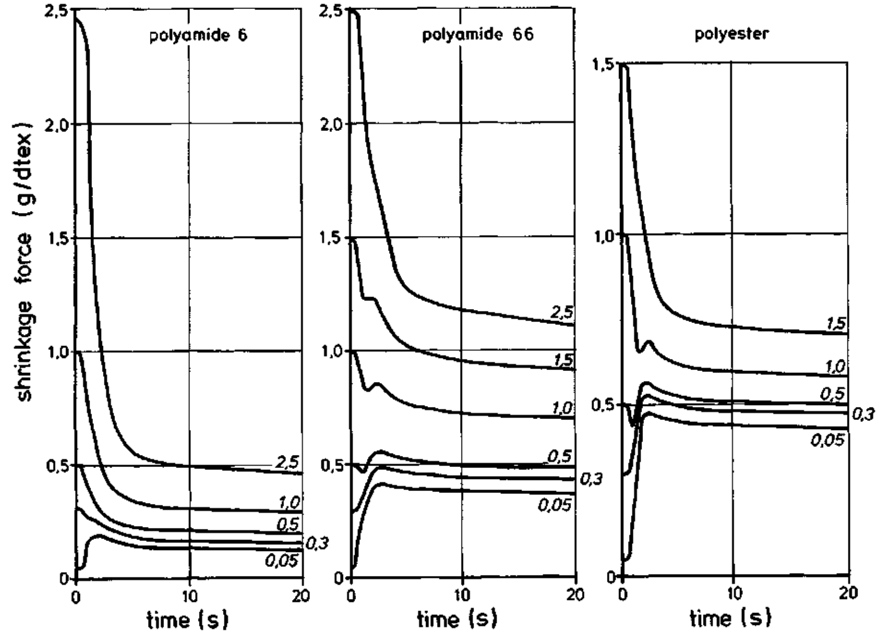


Figure 9.70 Shrinkage force as a function of time, with pretension as parameter, for PA 6, PA 66 and PET

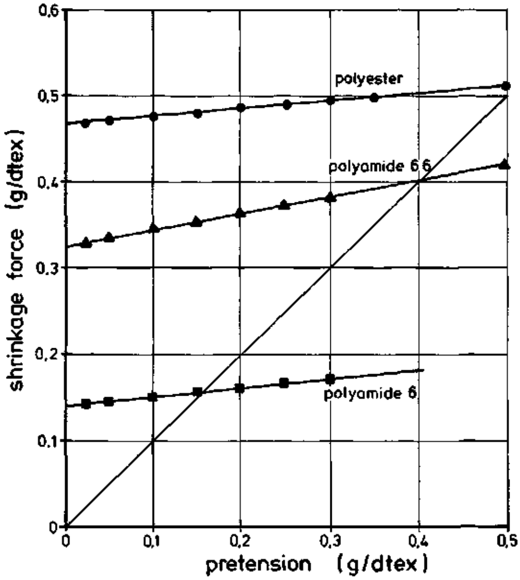


Figure 9.71
Shrinkage force as a function of pretension for PA 6, PA 66 and PET

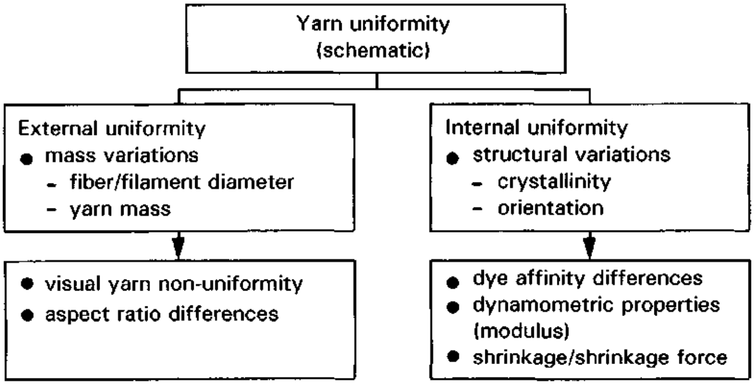


Figure 9.72 Uniformity of yarns (schematic)

dyeability, modulus, tenacity, elongation, etc. Both types of non-uniformity can occur at the same time. A change in linear mass can, e.g., manifest itself visually as a change in thickness, be less drawn, take up more dyestuff and therefore appear darker.

Figure 9.73 shows, as an example, the production stages in which testing can be done, and Fig. 9.74 how the upstream properties can affect the properties after further processing: for example, texturizing cannot improve the Uster CV value. The CV value of dry-spun PAN tow of ca. 7% can increase to ca. 11–12% as a result of washing and drawing. Figure 9.74 confirms this strong correlation between titer uniformity of a POY yarn and the resulting textured yarn [36].

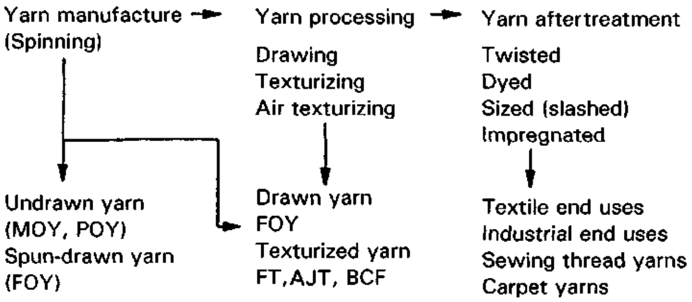


Figure 9.73 Application areas for yarn uniformity testing

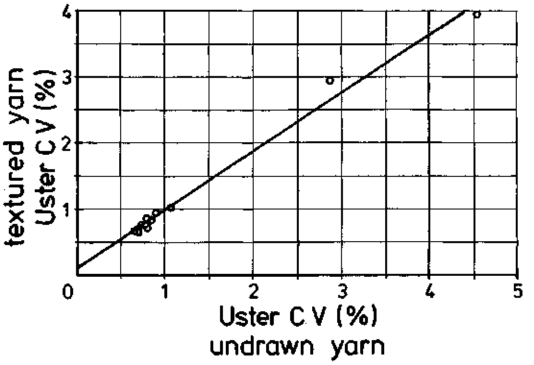
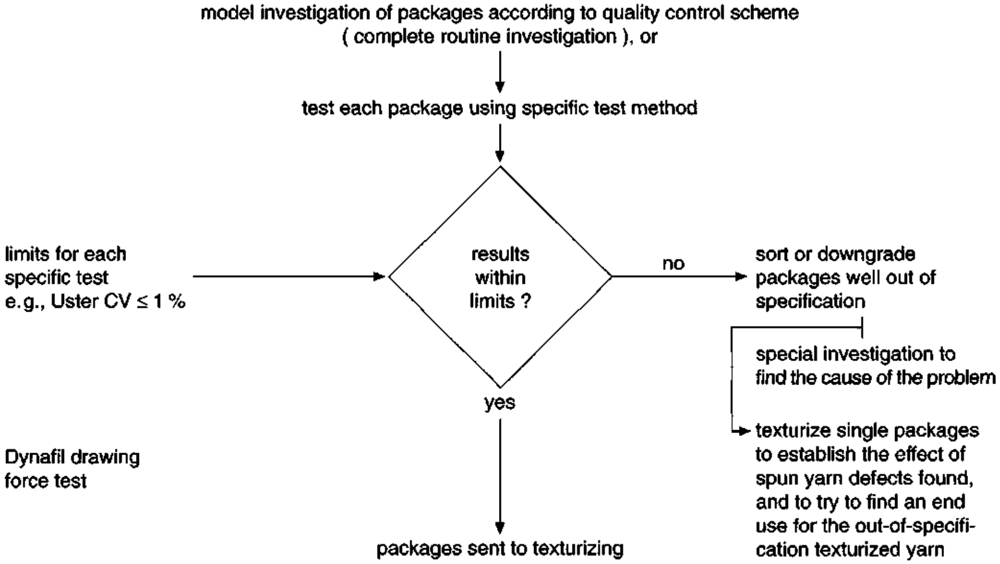


Figure 9.74
Relationship between Uster CV values of undrawn- and drawtexturized filament yarn

To this end, a generally-valid testing scheme is given in Fig. 9.75. With appropriate sorting norms, each production stage can be controlled and the causes of out of specification production traced and found.



This test scheme can also be used for other undrawn yarns, e.g., for LOY, MOY.

Figure 9.75 Model investigation of packages according to a quality control scheme

9.3.5.1 Test Methods for External Uniformity

Discontinuous Testing

The samples are of limited length: e.g., 20 cm for tensile testing, 8 m hanks for crimp contraction testing, 32 ... 160 mm for single fiber testing. The following tests are typical:

- Yarn samples are tested visually (for example, for thick places, filamentation, etc.) by wrapping on an inspection table (mostly black, Seriplane).
- Fabrics are made from yarns to be tested (raw white or dyed), e.g., knitted socks or narrow woven bands (without changing the weft yarn) for periodic or start marks.
- Tenacity/elongation testing.
- Crimp testing: Fig. 9.76 (left) shows results from a Dynafil test [39,40] on a PET 167 dtex set (= double heater) yarn: a periodicity of ca. 200 m is evident. The diagram on the right shows results of repeated testing using 8 m hanks: a periodicity of 25 to 28 hanks can be seen.

Continuous Testing

The samples are very long and are taken off from a bobbin or as spun tow. The most common method of measuring titer variations uses a capacitor. The most widely-used test apparatus is the "Uster" tester, described in detail in Section 7.8 for diagnosis of causes of failure in production machines which manifest themselves in yarn deviations, and where many results are presented. The measurement principle requires the yarn to be tested to be run between the two capacitative plates under uniform tension and torsion. In order to eliminate moisture and spin finish influences, the sample must be sufficiently conditioned.

The effect of poor quench flow uniformity is shown in Table 9.18b. The take-up tension varies in parallel with the quench air variation. The individual components can be obtained by performing a Fourier analysis on the Uster diagram.

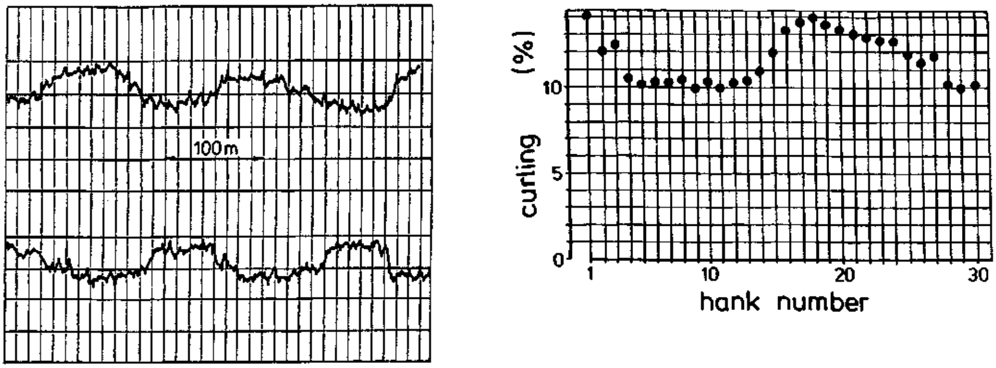


Figure 9.76 Textured yarn test according to German Standard DIN 53840 on 167 dtex f30 PET drawtexturized set (double heater) yarn:
Left: continuous crimp contraction force test with 5% underfeed, 20 m/min test speed, 120°C heater temperature and 25 g pretension.
Right: comparable crimp test according to German Standard 53840 DIN on consecutive yarn hanks

Table 9.18a Titer Variations, their Most Frequent Causes and Magnitudes (These Variations are only sometimes Additive)

Take-up speed (m/min)	800–1700	3200–3800	=5200
– Quench air variations	30–40 m/1.2	40–60 m/0.6	50–80 m/0.4
– Pulsation of air ducting	1–2 s/0.4	1–2 s/0.3	1–2 s/0.2
– Spinneret temp. variations (T)	acc. to T/0.1/°C	acc. to T/0.1/°C	
– Turbulence in quench	10–40 m/1.0 (broad)	20–60 m/0.5 (broad)	25–80 m/0.3 (broad)
– Traverse:			
with godets	0.7–2 m/0.8	1–1.3 m/0.4	1.3–2 m/0.25
with integrated overfeed roll (e.g., grooved roll)	–	3 m/0.3	3.5 m/0.2
– Package running out-of-round*	1–3 m/0.5	1–1.3 m/0.3	1–1.3 m/0.2
– Out of round godets	0.5 m/0.1	0.5 m/0.1	0.5 m/0.1

*and wound yarn length per double stroke

Table 9.18b Investigation into Variables Affecting the Uster Value (Compare Table 7.11 and Fig. 7.32F)

No.	Variable	Parameter	Period	Example of investigation Approx. fractional contribution	 CV contribution %
1	Spinning extruder	Temperature	1.8 min	0.03	0.4
		Pressure	0.83 s	0.01	0.28
2	Spinning beam	Temperature	2.6 min	0.09	0.8
3	Melt temperature (immediately below spinneret)	Viscosity	2.6 min	0.06	0.10
4	Melt delivery pressure	Melt	Pump speed	0.04	0.44
5	Quench	Cooling	ca. 2 s to irreg.	0.25	1.60
6	Yarn position	Cooling	ca. n × 2 s	0.05	0.32
7	Yarn tension		ca. 2 s	0.20	1.35
8	Traverse		Stroke frequency	0.16	0.56
9	Miscellaneous			0.11	0.65
				1.00	6.50

The Uster test gives: (see also Section 7.8)

- the average (mass) titer (= AF value, average factor)
- a diagram of titer variation
- the CV value (% coefficient of variation): the most important characteristic value for the external uniformity
- the spectrogram, for periodicity analysis and for back-relating to the machine settings (Section 7.8).

Figure 9.77 gives a further example, including the necessary explanations of causes. The Uster Tester II (old model) can register periods of 2 cm ... 40 m, the Model III up to 500 m — which makes it the preferred choice of yarn producers. This model was used to determine the values in Table 7.10, which serve as an example of complete evaluation [43]. The main influences on the Uster value in this example were deviations in melt temperature, quench and the traverse. Figure 9.78 shows that it is possible to convert the linear mass variations along the fiber length into amplitude frequency, thus helping to find the cause of the disturbance (see also section 7.8).

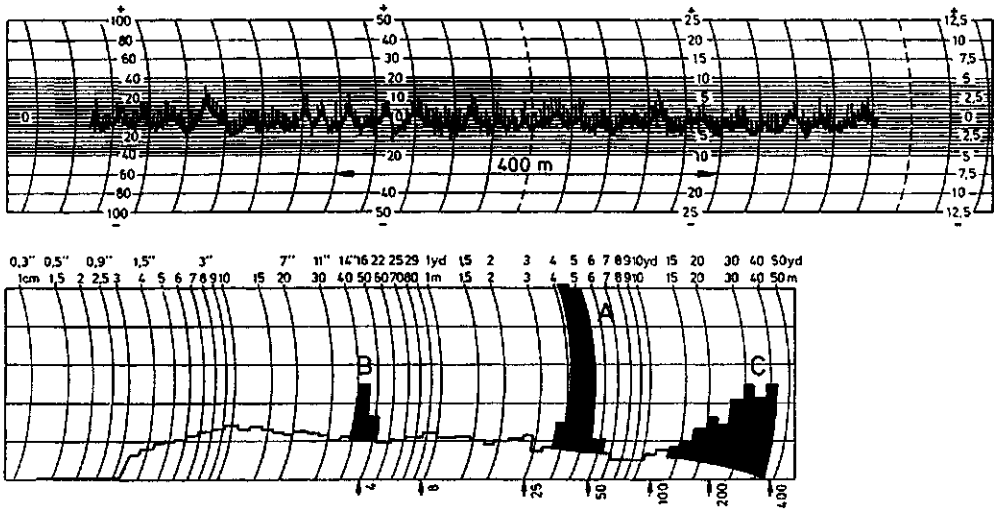


Figure 9.77 Uster diagram and wavelength spectrogram of a PA 66 22 dtex f7 multifilament yarn showing three faults:

A) periodic cross-sectional changes having a wavelength of 4.5 m, caused by an eccentrically-running spun yarn bobbin,

B) periodic fault showing a ca. 35 cm wavelength caused by an eccentric drawroll on the drawtwister,

C) variation of linear density (titer), caused by pulsating cross-flow quench and an upwardly-pulsating air flow in the interfloor tube

Table 9.19 Acceptable Uster CV Values (*1990*)

		Uster CV value %	Quality classification
Usable	yes	≤ 0.6	Very uniform (normally not visible after dyeing)
	possibly	0.6 ... 1.0	Uniform
	no	1.0 ... 2.0 2.0 ... 3.0 3.0	Less uniform Non-uniform Very non-uniform

Finally, Table 9.19 gives typical 1990/91 Uster CV criteria for yarn acceptability. The correlation between these Uster values and end product acceptability determines which values can be accepted in practice. Uster values above 0.6% become evident in the final product, e.g., as stripiness in solid shades.

In the case of staple fibers, CV values of 7% are considered very good, values of 11 ... 12% are still acceptable, while—in the case of mixed-spun yarns having a high content of another component—CV values of up to 25% can be used.

9.3.5.2 Test Methods for Internal Uniformity

The internal uniformity is determined by the morphological and chemical structure of the molecular constitution and their deviations, which in turn affect the dyeing, the dynamometric properties (modulus), shrinkage, shrinkage force, crimp, etc. The following tests are mainly used for the investigation of internal uniformity:

- Dye uptake test
  A circular-knitted sock (or a piece of woven fabric for the weft) is dyed in a large vessel at the appropriate temperature using a test dyestuff, is washed, dried tensionlessly and is then visually assessed:
- as rings and bands in the knitted sock [74]
- or the sock is unravelled and the yarn dye depth is measured optically
- or the stripes in the sock are marked and the affected yarn lengths are measured (Fig. 9.78, Table 9.19).
- The six stage Toray FYL-500 test system [44] lends itself to continuous evaluation (Fig. 9.79). From the bobbin magazine (left), a 10 m length of yarn (mainly textured) is taken from each sample and knotted to the next sample, up to 360 samples per hour being possible. The yarn is dyed with a special dyestuff (Toray Blue), followed by rinsing. The dye uptake and its variation is then measured in an optical analyzer, speeds up to 60 m/min being possible. The computer evaluates the so-called FYL value and its deviations from these inputs. Figure 9.80 shows the dependence of the FYL value on false twist level and heater temperature [44]. The system lends itself to setting temperature controls on textile machines, to temperature optimization at texturizing and to establishing the relationship between texturizing conditions and downgrading. It can be used for all yarns (staple and filament) in all material states (undrawn, drawn, texturized, dyed, etc.), as well as with supplementary equipment in gaseous or fluid mediums at various temperatures.
- Force measurement at defined change of length.
  The "Dynafil" tester [39] operates according to this principle (Fig. 9.81, left). In the case of undrawn yarn or POY, a defined draw ratio is used, whereas a specified overfeed is used for textured yarn. The yarn runs through a heated tube at a defined pre-tension (g/den), over a roller to which is fixed a force measuring cell, and is then taken off by means of a take-off roll. The drawing force (or crimp contraction force) and its deviation are recorded.
- Changes in length at defined force [47].
  The Lawson–Hemphill Textured Yarn Tester (TYT, Fig. 9.81, left) measures changes of length and their deviations. The yarn runs over godets or duos into a heated tube at defined pretension (g/den), after which the first sensor measures the crimp contraction (total recovery). The speed ratio of input : output roll gives the percentage change in length. In addition to crimp contraction, the apparatus measures yarn shrinkage and actual bulk at a specified temperature. It can also be used for undrawn MOY and POY yarns.

The test results from these latter two methods can be compared, although the reactions of the different characteristic values to process changes differ (Fig. 9.82). An increase in the first heater temperature, e.g., results in an increase in crimp intensity (total recovery), whereas the Dynafil shows a reduction in the crimp contraction force. The relationships given in Fig. 9.83 show the influence of the process parameters on the crimp contraction and permit interpretation of the source of the causes of error (Fig. 9.83).

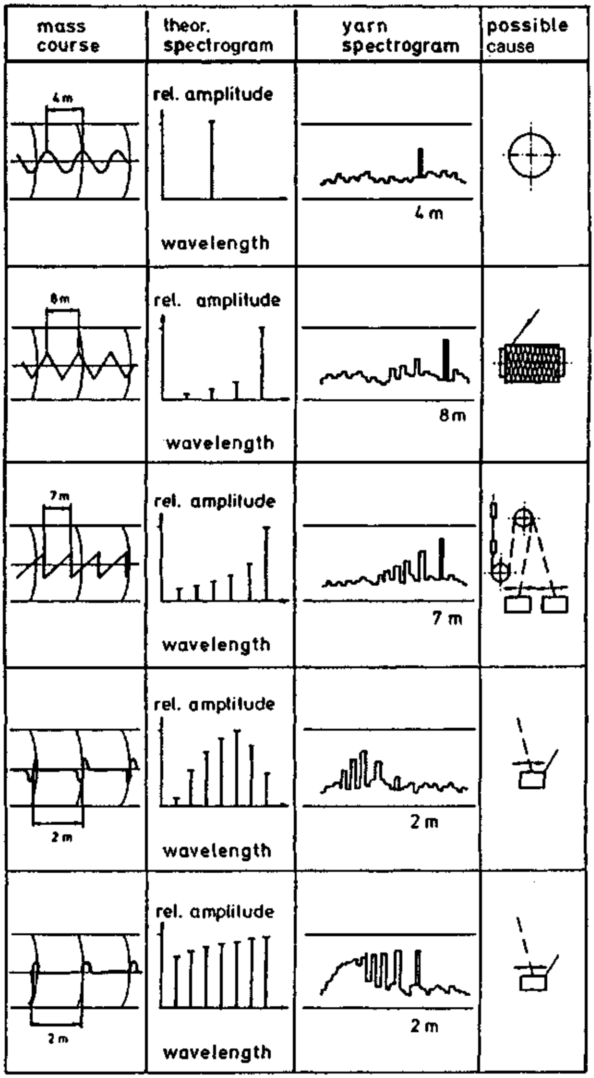


Figure 9.78
Evaluation scheme for Uster spectrograms [42]

In addition to the diagrams giving deviations in the characteristic values of internal uniformity, the following computer/statistical criteria must be added:

- average values of one or more bobbins, e.g., of a machine doff
- standard deviation or coefficient of variation within and/or between the tested values.

Tolerance limits can be given and strongly-deviating packages can be marked or eliminated. Here, too, mathematical analysis (similar to the Uster spectrogram) can be used to analyze frequencies and wavelengths with a view to finding their causes. Figure 9.84 shows an example of how texturizing errors can affect crimp intensity, and how causes of failure can be systematically assigned.

- Dynamic force/elongation diagrams
  Dynamic tensile testing, with or without the influence of temperature, is another method for analysis of internal uniformity [38]. Figure 9.85 compares a static force/elongation curve with that of a dynamic one derived from the Dynafil (175 °C heater, 100 m/min) for a 335 dtex f48 PET MOY yarn.

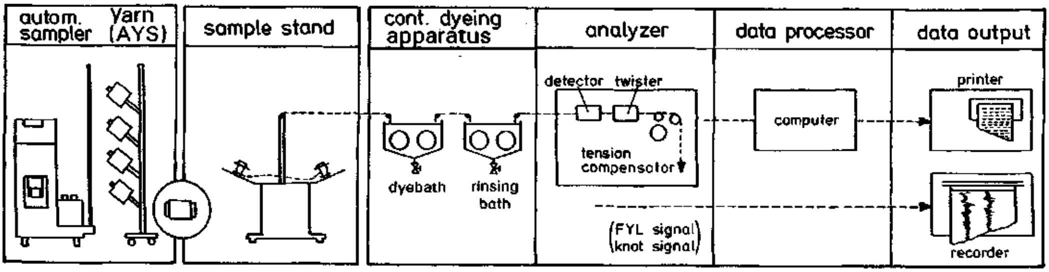


Figure 9.79 Continuous yarn testing using the six-stage Toray FYL-500 S dye uptake tester

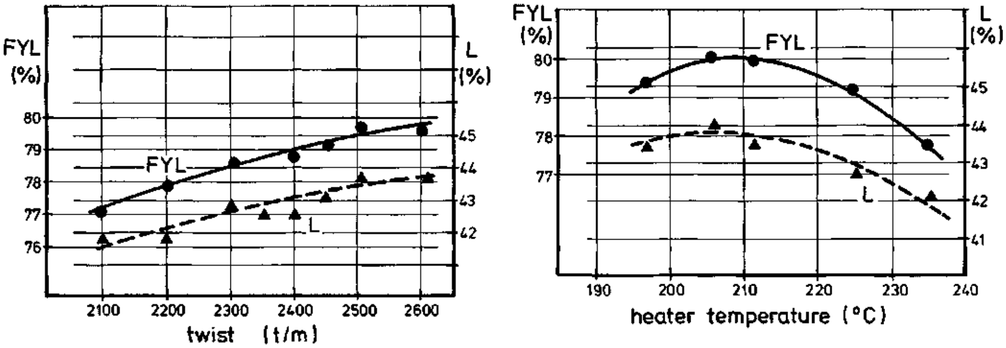


Figure 9.80 Results of an evaluation on a PET false twist yarn using the equipment in Fig. 9.79
a) dependence of the *FYL* (or *L*) value on twist level
b) dependence of the *FYL* (or *L*) value on first heater temperature

Next, the drawing force is measured at different draw ratios at otherwise constant settings, and the results plotted on a co-ordinate system—which gives the dynamic force/elongation diagram. Because of the effect of temperature, this curve is flatter than the static curve, and the typical plastic flow region of the under-drawn yarn is absent. The dynamic force/elongation curve is more representative of the real (thermal) process, as here the rheological relationships at higher temperature (e.g., hot drawing or drawtexturizing) can be analyzed.

9.3.5.3 Results from Investigations into Uniformity, and Causes of Non-Uniformity

If one arranges the measurable characteristics of internal and external uniformity with regard to their correlation with the most noticeable faults in fabric in a matrix, as in Fig. 9.86, one notices that average titer and its coefficient of variation (CV) affect stripiness strongly, while average drawing force and its CV affect dyeability in particular. In the case of textured yarn, changes in length or crimp contraction force characteristics are mainly an indicator of changes in voluminosity (volume stripiness), but also indicate changes in dyeability.

By means of analysis and interpretation of Uster diagrams and spectrograms, the sources of titer non-uniformity can be assigned, as per Fig. 9.87.

- Polymer condition
  Viscosity (molecular weight and temperature uniformity)
- Spinning machine
  Extruder faults, e.g., loss of pressure—melt temperature and deviations—spinneret temperature—insulation or bedding in of the spinnerets—quench chamber faults and flow pattern—quench air flow

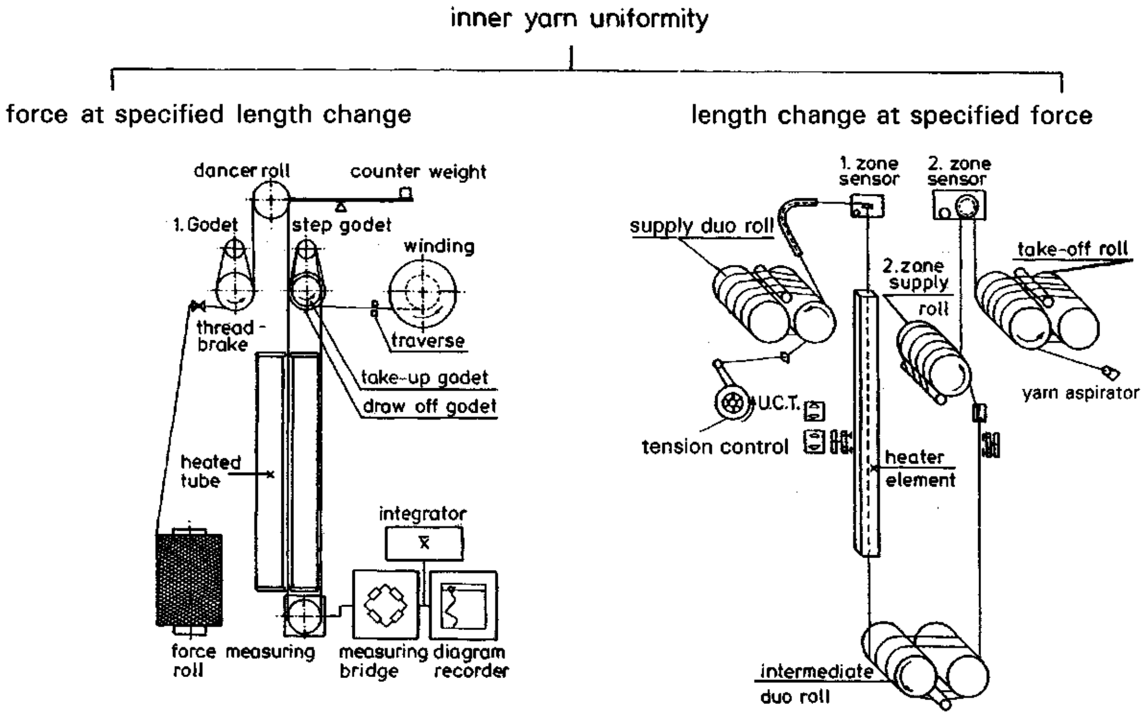


Figure 9.81 Apparatus for determining the internal uniformity of yarns.
Left: Dynafil apparatus (Textechno) — force measurement at specified length change.
Right: Textured Yarn Tester (Lawson-Hemphill) — measurement of length changes at specified force.

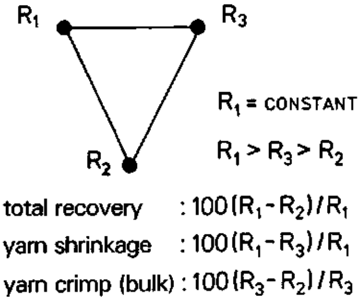


Figure 9.82
Measurement results of the TYT tester (Fig. 9.81)

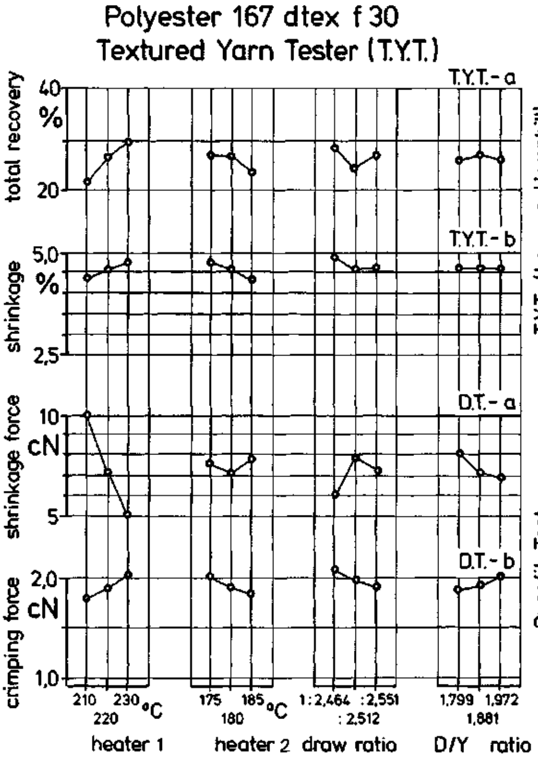


Figure 9.83 Comparison of results on 167 dtex f30 PET using methods in Figs. 9.81 and 9.82 with Dynafil and with TYT tester

rate, quench air velocity, including its distribution and turbulence—godet and winder eccentricity—and bearing problems, roll contamination and damage—traverse mechanism and its faults—spin finish faults (defective or false, too high or irregular)—variation in intermingling intensity and poor filament cohesion—take-up speed errors

- Drawtwist machines.

Godet eccentricity, bearing problems, roll contamination and damage—defective separator rolls—differences in drawing, drawing faults, yarn slipping over the rolls—defective yarn guides or ring

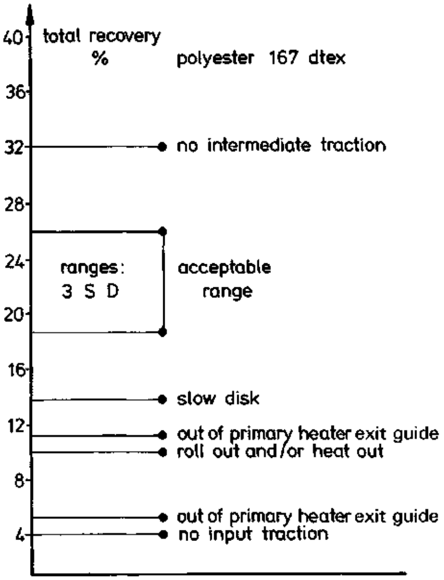


Figure 9.84 Interpretation of Textured Yarn Tester TYT results on PET 167 dtex, and assignment of faults found in terms of total recovery

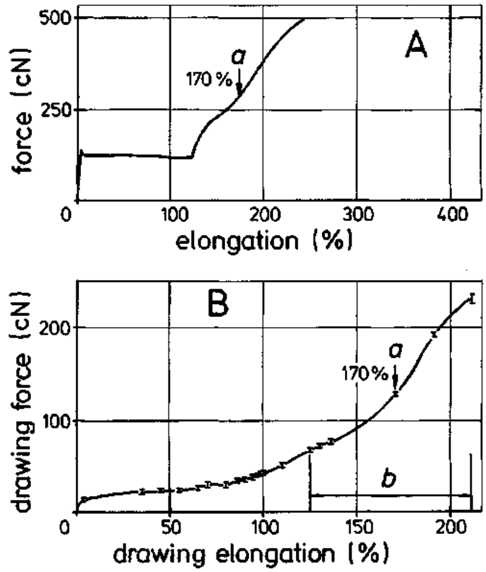


Figure 9.85 Tenacity/elongation diagrams
A) from static measurements (tensile test)
B) from dynamic measurements (Dynafil apparatus)
a) reference point (170%)
b) usable range for drawing force measurements

degree of uniformity

		titer (fineness)		drawing force	
		average	variation coefficient C V	average	variation coefficient C V
down grades	dye uptake	O	O	XX	X
	stripiness	X (in one bobbin) XX (between several bobbins)	XX	O	O

XX strong correlation X weak correlation O no correlation

Figure 9.86
Relationship between yarn uniformity and downgrading, as a correlation matrix

travelers—eccentric and vibrating spindles—yarn tension variations (e.g., from ring rail stroke: which results in titer deviations in the drawn cop)

- Warp-draw machines
  deviations in the draw ratio—defective reeds—problematic intermingling—start and stop lines after a yarn break—warp yarn end change resulting from a yarn break.

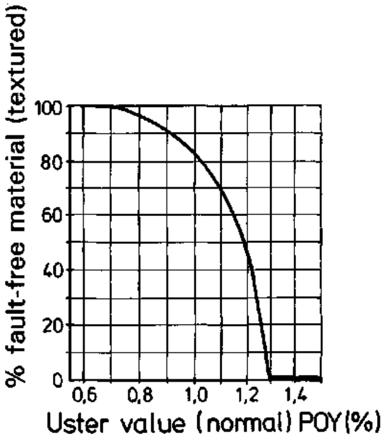


Figure 9.87
Relationship between the Uster CV value of PA 66 POY yarn and the % of stripe-free, dyed textured yarn

- Twisting machines
  changes in the number of twists (e.g., through deviations in spindle speed or twist build-up in front of yarn guides)—yarn tension variations due to twist generation—winding problems (with multi-tier or doubling machines)
- Texturizing machines
  bad package formation—drawing problems during drawtexturizing—heater temperature uniformity (venting, coking)—too high a yarn speed for a given heater and cooling zone—input and delivery roll slippage—twist slippage at the twist aggregate—rotational speed of the twist aggregates and their uniformity—disk surfaces—deposits
- Further processing
  Assessment of the suitability of the yarn already produced for the planned further processing—prediction concerning expected quality fall-out, in particular concerning visual uniformity (Figure 9.87). There are sufficient results, both from investigations and from experience, to assess the characteristic values of internal uniformity, and to use these to establish adequately the causes of deviations in the process conditions and to demonstrate errors in
- Polymer condition/state
  viscosity, factors affecting viscosity and its uniformity—differences in chip moisture
- Melt spinning and feeder yarns
  spinning pumps and their delivery accuracy—spinning temperature, and its effect on viscosity and degradation—ageing of MOY and POY—spin finish pick-up and its influence on orientation—moisture in feeder yarn
- Drawtwisting
  Temperature deviations of individual components and their effect on the stability of drawing and the draw point—variations in draw ratio—changes in yarn modulus caused by the ring rail stroke—yarn tension variations caused by ring rail and spindles (bearings, ring traveler, etc.)
- Warp-drawing
  Variations in draw ratio—yarn temperature effects at starting and stopping
- Twisting
  tension changes during twisting (tension accumulation)
- Texturizing
  temperature effects of primary and secondary heaters—variations in draw ratio—variations in disk/yarn speed (D/Y ratio)—slippage of belts—yarn slippage on disks—incorrect yarn path—package builds (volume changes on package sides)
- Yarn package
  modulus changes through package (outside/middle/inside effect) caused by relaxation—winding effects and package side effects in spun and (particularly) texturized packages
- Further processing
  temperature and yarn tension effects (e.g., during warp preparation, sizing, dyeing, steaming).

From these individual, listed causes of uniformity variation and from experience, it is possible to establish

- Tolerances for process parameters, particularly for a regulated process control (see Section 9.1.1). These then form the basis for optimal manufacturing conditions for producing optimal products, as well as for derived properties and uniform end product quality. The process parameter tolerances established are "backward-looking", i.e., from the quality of finished fabrics backwards to the properties of the feeder yarns in each stage of production, right up to the polymer. As an example, Fig. 9.88 shows a cause/effect model relating the finished goods quality (stripiness) to the yarn properties, the textile further processing and yarn manufacture. Both objective and subjective criteria are included in the establishment of the tolerances, e.g., yarn type, end use, dyestuff, etc., constitute the objective criteria, while, e.g., fashion and customer wishes are taken into account as subjective factors.

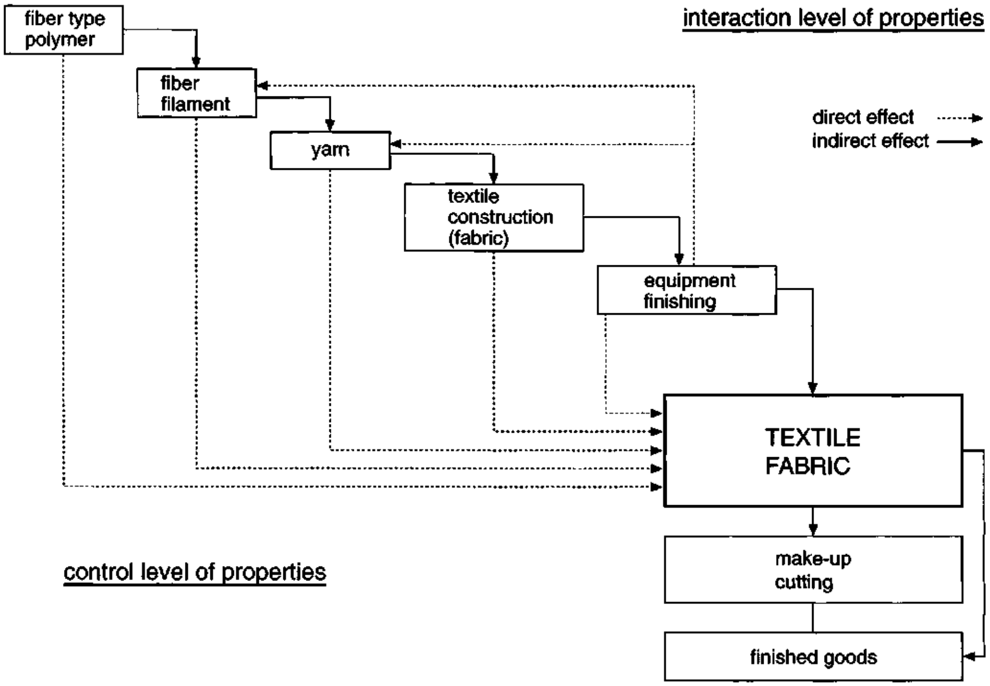


Figure 9.88 Cause/effect model for investigation of causes of faults in fabrics, relating back to polymer

Figure 9.87 clearly shows that an Uster value of $U=0.6\%$ is required in order to achieve 100% stripe-free goods in the case of PET (a similar relation holds for PA 6 and PA 66), while an Uster value of $U=1.0\%$ will result in only 85% of stripe-free product. Yarn having an Uster value of 0.7% is therefore not suitable for general textile application, but can only be used for special applications in which dye-levelling is used.

For staple fiber production, similar relations can be established, and—from these—the production parameters and their allowable deviations. From polymer to spun tow, the relations are similar to those mentioned above, but—after tow stretching—the relationships become much more complex and many-sided.

9.4 Fiber and End-Use Properties

As a consequence of the cause and effect relationships on many levels, the relationship between fiber- and finished product properties is extremely complicated and multi-faceted. All stages in the production chain can be treated as a series of input–output relations. The following four stages determine the properties of the end product:

- Fiber properties
  Polymer and additives—fiber mixtures—fiber form (cross-section)—filament properties (smooth, textured, chemically-treated)—elongation
- Type of fabric (woven, knitting, non-woven)—construction (binding, number of yarns, surface)
- Finishing/after-treatment
  stentering, type of dyeing and dyestuff selection—chemical/mechanical/thermal post-treatment (resin finishing, alkalization, roughening, singeing, surface treatment)
- Make-up
  type of cutting—form and assembly of individual components in the case of technical textiles.

"Fiber engineering" therefore requires that the appropriate polymeric fiber meeting the criteria above is used, so that the article produced has a maximum value in use, the so-called "fitness for use".

9.4.1 Cause and Effect Chain Between Fiber and Endproduct

Figure 9.88 is an interaction diagram showing how the polymer and fiber properties persist through into both the fabric structure properties and the final article properties. While the original polymer/fiber properties persist into the final article, they can, however, be indirectly changed to a greater or lesser extent during each process stage. The physical or chemical properties of the fiber can be so strongly modified by other factors (e.g., crimping, twist, weaving construction and finishing) that the latter predominantly determine the properties of the final article.

There is a direct relationship between fiber cross-sectional form and, e.g., fabric handle: non-round or hollow cross-sections have a greater bending moment than round fibers and therefore confer higher bending stiffness, which results in a harsher handle in the end product. Here the indirect effects of fabric construction and equipment interact strongly and can gradually weaken the former effects. For orientation, Fig. 9.89 gives an overview of the final product properties and the factors which affect them; fiber type, fabric construction and finishing. These illustrations, to be understood as sets, are to be interpreted as follows: certain properties (e.g., moisture relations, antistatic, antiflam) can be influenced by the fiber properties or the finishing. Here it is to be noted that, in regard to the permanence of properties, the original property represents the more durable form.

9.4.1.1 Fiber Shape: Tactile and Optical Properties

The surface constitution of a fabric construction can be deliberately influenced (Fig. 9.90) by the choice of fiber cross-section. In melt spinning, this is done by selecting the appropriate spinneret hole geometry (e.g., Fig. 4.136). This, in turn, determines the optical, tactile, physiological and technological properties of the final product. Figure 9.90 summarizes the dependence of various properties on cross-sectional shape, area and fiber surface.

To influence luster, titanium dioxide is added to the polymer (Section 6.8.1). The relationship between luster, cross-sectional shape and degree of delustering is shown in Fig. 9.91 for a carpet yarn [55]. Even semi-dull (= 0.35% TiO_2) already shows a noticeable reduction. The addition of TiO_2 results in duller colors after dyeing. In addition, triangular or Y-shaped profiles produce strong luster, possibly even glitter effects.

The variation of fiber/filament fineness (which can be reduced down to 0.1 dtex) is important in "engineering with fibers". Reducing fineness achieves more attractive handle, aesthetic, drape, etc., while coarser filaments lend themselves to easier fiber production and further processing, have some better properties in use (abrasion, pilling) and are more suitable for carpet fibers (recovery). Dependence tendencies are given in Table 9.20.

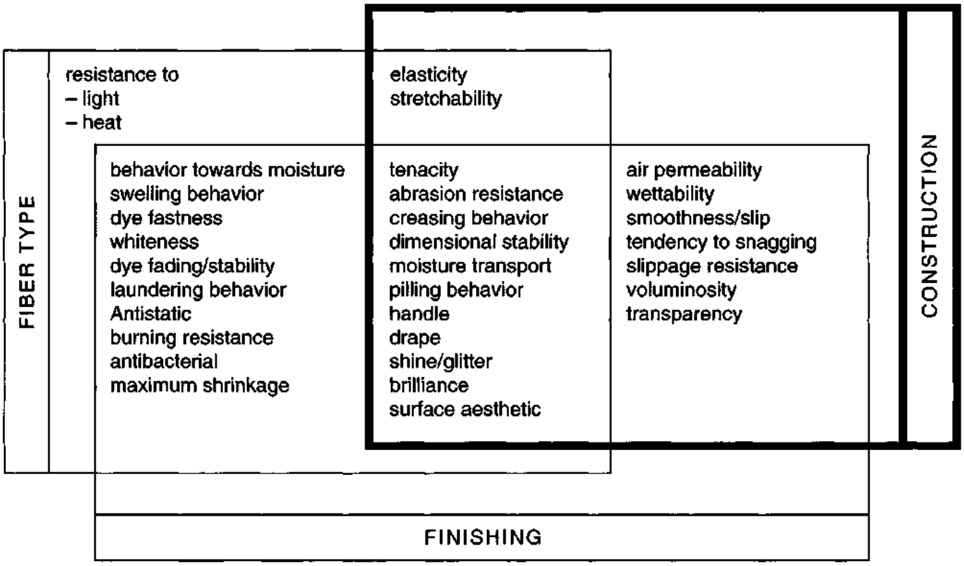


Figure 9.89 Relationship between finished goods properties, fiber type, fabric construction and finishing

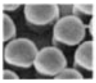
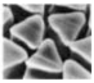
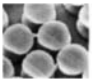
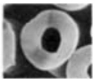
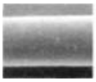
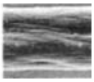
	cross-sectional shape		cross-sectional area		fiber surface	
	round	profiled	solid	hollow	smooth	structured
						
optical properties						
brightness (luster)	weaker	stronger	stronger	weaker	stronger	weaker
transparency	higher	lower	higher	lower	higher	lower
covering power	lower	higher	lower	higher	lower	higher
color impression (dye depth)	darker	lighter	lighter	darker	lighter	darker
dyestuff consumption	lower	higher	lower	higher	lower	higher
dirt visibility	higher	lower	higher	lower	higher	lower
particle adhesion	weaker	stronger	-	-	weaker	stronger
tactile properties						
handle	softer	harder	softer	harder	softer	harder
friction	smoother	rougher	-	-	smoother	rougher
bending resistance	lower	higher	lower	higher	-	-
voluminosity	lower	higher	lower	higher	-	-
physiological properties						
fiber surface area	smaller	larger	smaller	larger	smaller	larger
moisture transport	smaller	larger	smaller	larger	smaller	larger
thermal insulation	lower	higher	lower	higher	-	-

Figure 9.90 Overview: Influence of fiber cross-sectional shape and surface area on optical and tactile properties

One can also achieve certain effects by means of yarn twisting (Table 9.20a), while—at the same time—influencing further processing (yarn take-off, beaming, air consumption in air-jet weaving). According to Fig. 9.92, luster also decreases with increasing twist: this is explained by the helical form of the filaments after twisting.

Further processing, such as texturizing or alkalization, modifies the cross-section and/or surface, and thereby the appearance or handle of the final goods. These modification possibilities are, however, of less importance than the original fiber cross-section.

Table 9.20 Relationship between Fineness (Single Filament Titer) and the Properties of the Endproduct

Property	Single filament titer	
	fine ⟷	coarse
Elasticity	lower	higher
Crease resistance	lower	higher
Snagging	lower	higher
Air permeability	lower	higher
Hand (handle)	softer	harder
Bending stiffness (drape)	lower	higher
Voluminosity (bulk)	higher	lower
Covering power	higher	lower
Optical appearance	more matte	more shiny

Source: Lenzinger Berichte, Folge 38 and Enka results

Table 9.20a Effect of Twist

Property	Twist	
	low ⟷ (non-twisted)	high
Creasing resistance	lower	higher
Air permeability	lower	higher
Hand (handle)	softer	harder
Voluminosity (bulk)	higher	lower
Covering power	higher	lower
Fabric appearance	more diffuse	sharper
Snagging	lower	higher

Source: Lenzinger Berichte, Folge 38

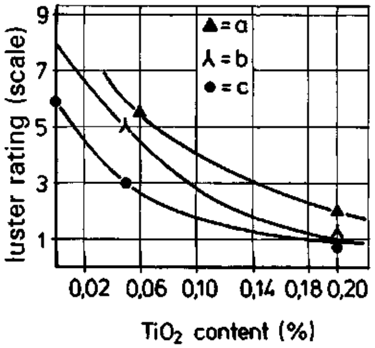


Figure 9.91 Relationship between luster, cross-sectional shape and titanium dioxide content

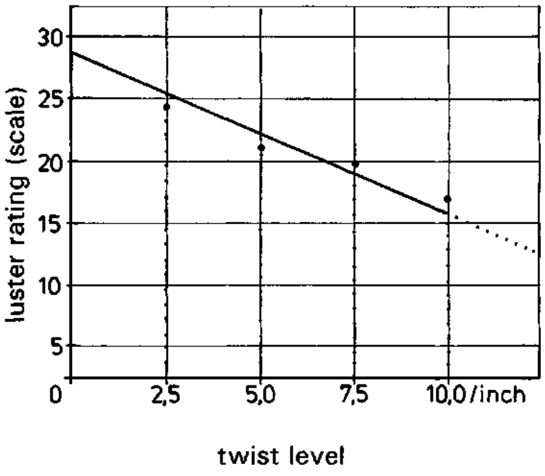


Figure 9.92 Effect of yarn twist level on luster

9.4.1.2 Influence of the Fiber Properties on Various Endproduct Properties

Table 9.21 shows how weaving density affects the properties of the end product in use. A similar relationship holds for the effect of the length of the float stitch (Table 9.21a). These properties are also affected by the finishing, particularly regarding the shrinkage developed, which increases the weaving density.

Table 9.21 Relationship between Yarn Stitch Density and Finished Goods Properties

Property	Yarn density low ⟷ high	
Tenacity	lower	higher
Areal density	lower	higher
Creasing resistance	higher	lower
Seam strength	lower	higher
Air permeability	higher	lower
Water vapor permeability	higher	lower
Fabric quality	poorer	better

Source: Lenzinger Berichte, Folge 38

Table 9.21a Effects of Woven Fabric Float Stitch

Property	Length of float stitch short ⟷ long	
Elasticity	lower	higher
Snagging	more	less
Seam strength	higher	lower
Voluminosity (bulk)	lower	higher
Fabric appearance	more matte	more shiny

Source: Lenzinger Berichte, Folge 38

- Pilling tendency:
  Pills are ball-shaped fiber entanglements which are pulled out of the fabric by abrasion during wear. They are considered to be visually so objectionable that their presence often results in the garment being discarded. There is a wealth of information covering investigations into the pilling phenomenon, from which methods of reducing or completely eliminating pilling have been derived. Figure 9.93 shows the dependence of pill formation on fiber fineness, fiber length and fiber cross-section as a function of the number of abrasion cycles. The course of pill formation should be noted: more fibers entangle themselves into pills, the pill density reaches a maximum, the pill density then reduces and finally (possibly) totally disappears as the anchor fibers in the fabric surface break off. One solution is to use fiber-forming polymers having low tenacity in the non-axial (= radial) direction: these can be produced, e.g., by including comonomers in the polymer chain. Such polymers promote the breaking off of pill-forming fibers on the fabric surface. Figure 9.94 shows the abrasion resistance (proportional to cycles) of PET as a function of the solution viscosity η_{rel}, with qualitative pilling resistance limits indicated. Short chain molecules also tend to result in earlier breaking off of the filaments. The number of flex cycles to break is dependent on the draw ratio (Fig. 9.95). Thermal treatment (crystallization) also reduces flex fatigue resistance. Figure 9.96 gives a summary of the interactions of fiber, yarn, fabric construction and finishing on pilling behavior. Here the influence of the fiber and its further processing can clearly be seen.

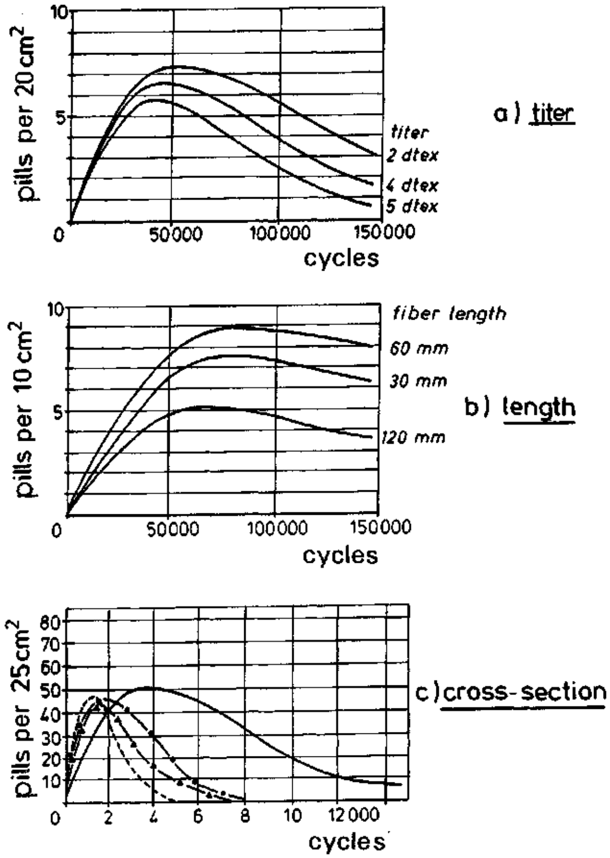


Figure 9.93
Relationship between pilling behavior and fiber properties
a) Titer *b*) Length *c*) Cross-section

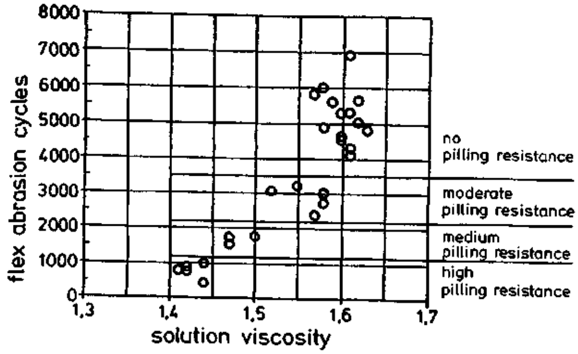


Figure 9.94 Relationship between flex abrasion cycles and solution viscosity of polyester, with pilling behaviour indicated

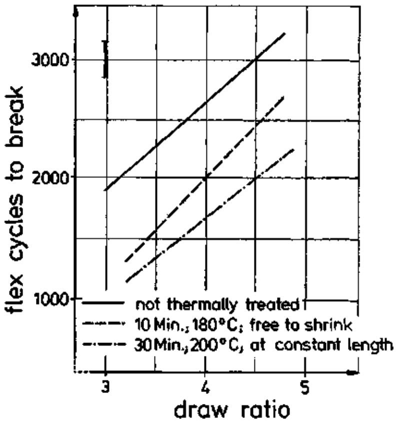


Figure 9.95 Flex cycles to break of a textile PET fiber of average molecular weight as a function of draw ratio and thermal treatment

- Creasing behavior

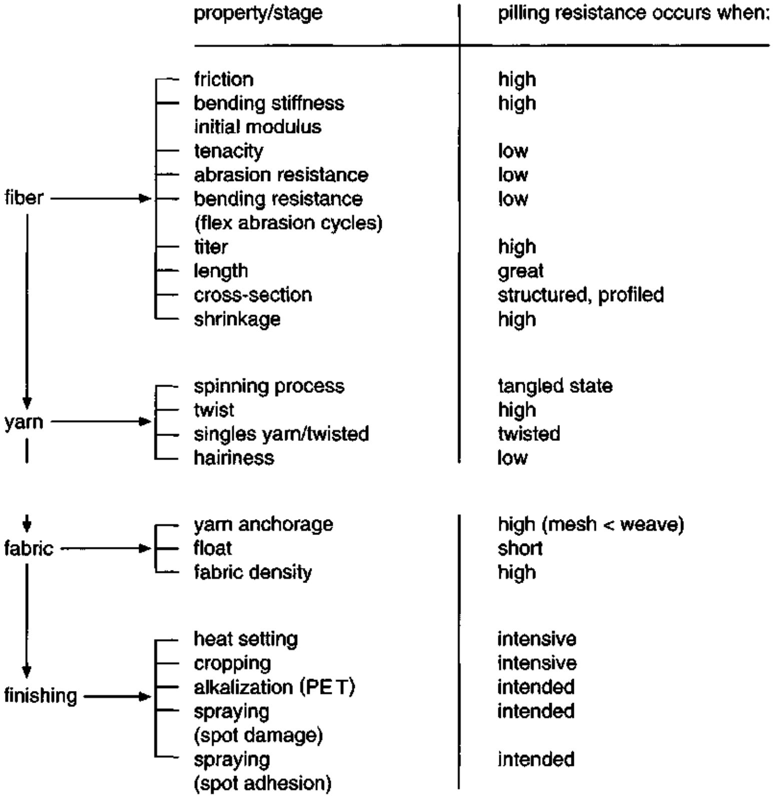
  During the wearing of garments (particularly on sleeves and trouser legs) and through incorrect laundering, folds and creases occur (Fig. 3.62), which subsequently more or less disappear. They may, however, persist, thereby making the garment unacceptable to the wearer.

  Crease formation involves two interlocking processes

  - Crease formation —, the bending of a fabric structure — the magnitude of the effect depending on the effective bending moment, the bending resistance and climatic conditions, particularly humidity and temperature
  - Crease recovery — the time-dependent recovery of the crease deformation —, itself a function of the bending elasticity and the factors which influence it (Fig. 3.68).

According to Fig. 9.88, the factors influencing creasing can be categorized as
internal influences: polymer — fiber, filament — yarn structure — textile construction of the fabric — finishing, after-treatment, make-up
external influences: loading (tension, pressure, etc.) — climate (humidity, temperature) — time

The method of measuring crease recovery is relatively simple: the fabric is bent 180 °, held under a defined force for a defined time, then the angle of recovery is measured as a function of time, or the fabric appearance is assessed visually (Fig. 9.96). Figure 9.97 shows the crease recovery of PET as a function of both orientation and crystallinity, an increase in both of which results in higher crease recovery. The dependence of creasing behavior on yarn fineness can be seen in Fig. 9.98 for a PET yarn (note: 8 ≈ best value): increasing filament titer improves crease resistance, because the resistance



	property/stage	pilling resistance occurs when:
fiber	friction	high
	bending stiffness initial modulus	high
	tenacity	low
	abrasion resistance	low
	bending resistance (flex abrasion cycles)	low
	titer	high
	length	great
	cross-section	structured, profiled
	shrinkage	high
yarn	spinning process	tangled state
	twist	high
	singles yarn/twisted	twisted
	hairiness	low
fabric	yarn anchorage	high (mesh < weave)
	float	short
	fabric density	high
finishing	heat setting	intensive
	cropping	intensive
	alkalization (PET)	intended
	spraying (spot damage)	intended
	spraying (spot adhesion)	intended

Figure 9.96 Interaction model: pilling behavior of and between fiber, yarn, textile fabric and finishing treatment

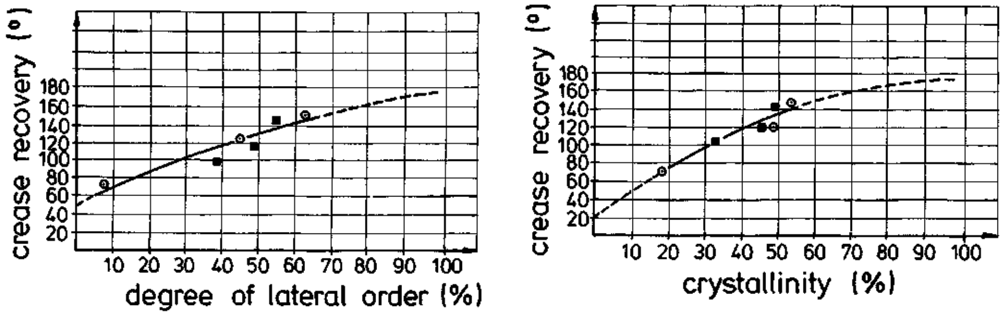


Figure 9.97 Crease recovery of PET as a function of morphological order

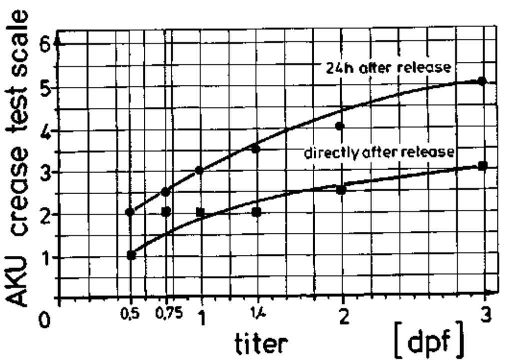


Figure 9.98
Creasing behavior of "Diolen Ultra" as a function of single filament titer (dpf)

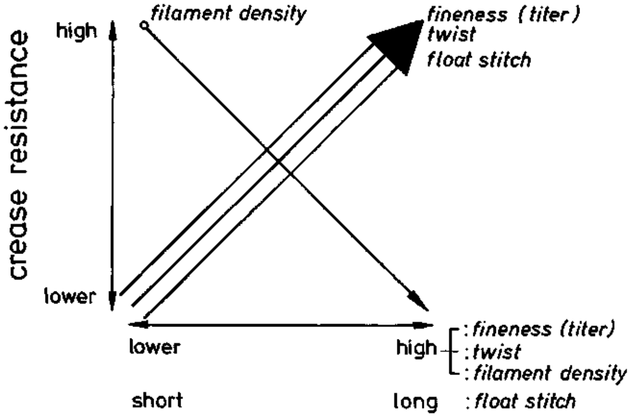


Figure 9.99
Main yarn and woven fabric property determinants of creasing behavior (schematic)

moment of the fiber increases likewise. Figure 9.99 shows schematically the dependence of creasing behavior on woven construction. In addition, finishing used has a decisive effect on creasing behavior, especially the use of substances which hinder crease formation.

- Electrostatic behavior

Table 9.22 gives an overview of the applications where the electrostatic behavior of textiles is relevant [58]. The effects can vary from irritation in use in the case of apparel to risk of explosion in the case of technical yarns. The mechanism of static generation in fibers is that rubbing during use generates an electrical charge, which—because of the poor electrical conductivity of the fiber—cannot be

Table 9.22 Relevance of Static Electricity for Textiles

Discomfort	Difficulties, poor functioning, financial loss	Dangers, risks
– Dirt attraction, soiling of wall hangings, furniture, curtains – Electric shocks Carpets, furniture Automotive textiles – Clinging, sticking of clothing	– Interference in computers, switching circuits (in automobiles, aircraft), "high tech." equipment – Machine- and production stoppages Spinning Textile processing Cutting and make-up	– Ignition, explosion Mining Petrochemical industry Hospitals

discharged quickly enough. In this way, the textile user can, under certain conditions, acquire a certain electrical charge, which is then suddenly discharged to earth, resulting in the phenomena shown in Table 9.22. To achieve an antistatic effect, the specific electrical resistance of the fiber must be so reduced that the charge developed can be quickly conducted away. This requires that the limits

Table 9.23 Determination and Classification of Antistatic Properties

Resistances:	
– Solids (acc. to Ges. der chem. Industrie, Germany), at 23 °C/50% RH	$< 10^{9}$
– Automotive textiles (acc. to General Motors/Opel), at 22 °C/65% RH	$< 10^{10}$
– Clean room clothing, at 22 °C/40% RH	$< 10^{6}$
– Carpets, at 23 °C/25% RH	$< 10^{10}$
Field strengths:	
– Product = log (Field strength × decay half-life), e.g., log (100 V/cm × 1 s) = 2	< 2
Cling time:	
– Linen, linings, light female clothing (PA 6) Development and classification according to experience	

Cling time min	Classification according to antistatic comfort	
3	Exceptional to very good	at 22 °C/25% RH
3...5	Good	
5...8	Acceptable	
8	Bad	

given in Table 9.23 are not exceeded. From the dependence of the electrical resistance of various fibers on the relative humidity of the environment, the following cause and effect dependence can be derived:

relative humidity
└──→ moisture content of the fiber
  └──→ electrical resistance of the fiber
    └──→ conduction of the charge

As Fig. 9.100 shows, the electrical resistance decreases by a few decades between 20% and 80% relative humidity. This leads to a solution for the problem:

- staple—spinning of polymer mixtures containing highly conductive substances (Section 2.12.8), or a combination of carbon-containing polymer from the same source as the main polymer (Fig. 9.6)

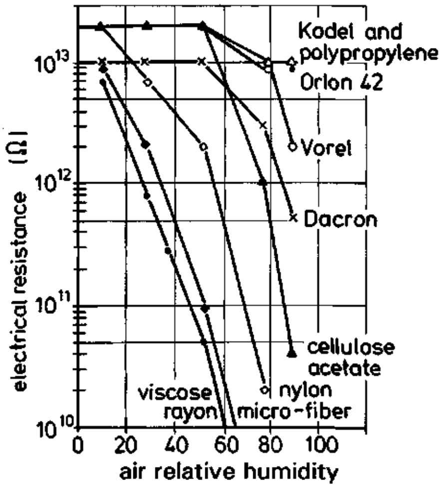


Figure 9.100
Electrical resistance of various fibers as a function of air relative humidity

- Continuous filament—combination containing one or more conductive filaments (containing carbon or metal)
- staple fiber yarns—co-mixing of up to 1% of conductive fiber (containing carbon or metal)
- fabrics—systematic incorporation of conductive yarns or application of antistatic surface coating (the latter is usually washed out and is not durable)

The co-spinning of conductive substances has proved to be optimal in terms of antistatic properties, but carbon-containing polymer causes a change in dye shade. This effect can be reduced by using bicomponent techniques (Fig. 9.6).

9.4.1.3 Fiber Properties and Physiological Behavior

For clothing applications, the term "wearer comfort" is used: this, in turn, can be subdivided into four categories [59]:

- thermophysiological properties
- sensory/tactile comfort
- comfort through movement/dexterity
- psychological comfort

In Table 9.24, the above comfort concepts are causally related to stages in the textile manufacturing process. Also included are environmental factors (climate) and subjective wearer opinion.

- thermophysiological comfort
  When the heat generated by the wearer's body is in equilibrium with the bodily heat transported through the clothing into the environment, the wearer feels himself to be in a state of thermophysiological comfort. The textile construction of the yarn and/or the fabric construction is mainly responsible for the thermal insulation of the garment. To achieve such insulation, the structure should contain as much air as possible, since air has a lower thermal conductivity than the fiber (Table 9.25). The amount of air contained in a garment depends mainly on the fabric construction, this amount being directly proportional to the fabric thickness. Cutting and layering can also influence the thermal regulation of the garment via ventilation. To achieve good wearer comfort, bodily sweat generated during physical activity should be transported away. Moisture removal can be achieved by absorption, transport, textile construction or a sufficiently high permeability. The main contributors to physiological comfort are the moisture absorption of the fiber, its morphological constitution, the textile construction and the garment construction. The moisture absorption of various fibers at 65% relative humidity is given in Table 9.26. From Fig. 9.101, it can be seen that the fiber surface area increases

Table 9.24 Comfort in Clothing

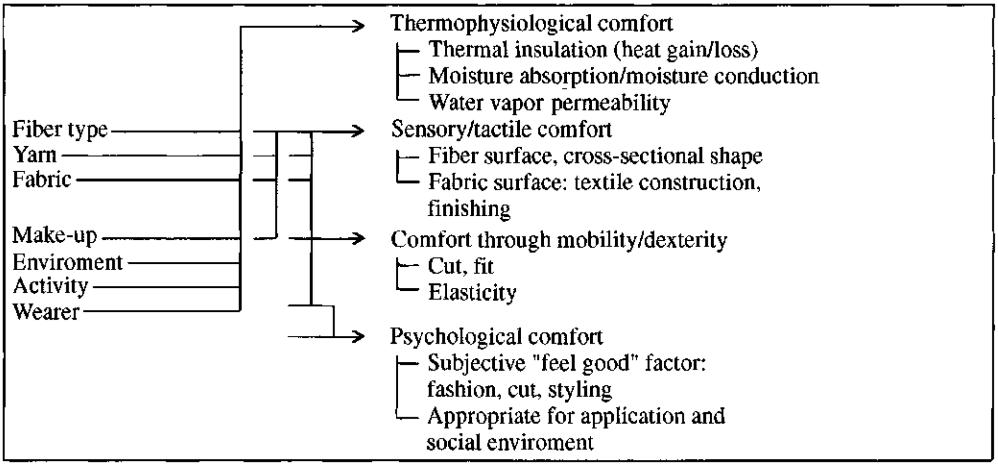


Table 9.25 Thermal Conductivity of Fibers

Fiber	Thermal conductivity W/m × K
Air	0.026
Aramid	0.130
Cellulose acetate	0.226
Cotton	0.461
Polyacrylonitrile	0.200
Polyamide	0.243
Polyester	0.141
Polypropylene	0.117
Polyurethane	0.126
Polyvinyl chloride	0.167
Viscose	0.289
Wool	0.193

strongly with decreasing filament titer. It therefore follows that finer fibers have greater surface-dependent moisture transport potential. Profiled cross-sections also have greater surface area than round sections. Figure 9.102 gives the schematic relationships between the main properties required for physiological comfort and the morphological shape properties of the fiber (Table 9.25).

- sensory/tactile comfort

  Sensory/tactile notions of comfort are subjective, and are derived from the feeling arising from skin contact with fabrics. These sensations include unpleasant ones, like skin irritation, scratching, itching or clinging of the fabric to the skin (caused by perspiration or electrostatic charge). The fiber properties contributing to these effects are given in Fig. 9.90. Woven construction, finishing and make-up also contribute to this aspect of wearer comfort.
- Comfort through movement/dexterity

  This aspect of comfort is related to the functionality of the clothing and is significantly affected by the cut and make-up. Clothing which is cut too small or has too little stretch is considered uncomfortable.

Table 9.26 Moisture Absorption and Water Retention of Fibers

Fiber type	Moisture absorption (at 65% RH) %	Water retention %
Cotton	7...11	45...50
Wool	15...17	40...45
Silk (boiled-off)	9...11	40...45
Viscose	11...14	85...120
Cupro	11...14	85...120
Acetate	6...7	20...28
Polyester	0.2...0.5	3...5
Polyamide	3.5...4.5	10...15
Aramid	4.5...5.0	12...17
Polyacrilonitrile	1.0...2.0	5...12
Modacrylic	0.5...4.0	10...20
Elastane (Spandex)	0.5...1.5	7...11
Polypropylene	0	unknown
Polyethylene	0	unknown
Polyvinyl chloride	0...0.2	25...35
Vinal	3.5...5	4...6
Glass fiber	0	unknown
Carbon fiber	1.0	unknown
Superabsorbent	≤ 10	500...3000

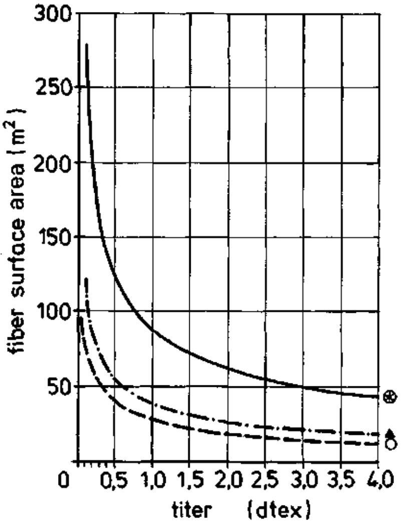


Figure 9.101
Filament surface area for various linear densities (titer) and cross-sections

A model showing the relationship between clothing fit and stretchability of textile structure is given in Fig. 9.103. Elastane (spandex), textured and core-spun yarns, in particular, are used in stretchable and elastic fabrics (corsetry, sports clothing, special medical clothing, etc.). A garment which fits well, is correctly designed for its end use, allows free movement of the body extremities and frees the hands for work, is considered comfortable. Other components of comfort come from fitting the fabric construction to the end use: fabric weight, bending stiffness, stretchability and elasticity (Fig. 9.103). Here the fiber contributes only partly to the nature of the fabric.

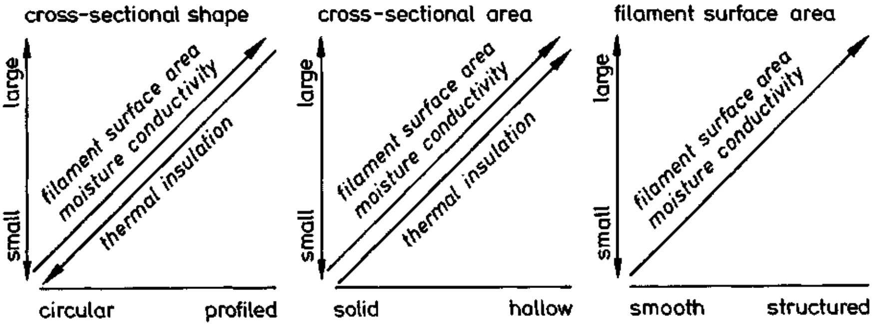


Figure 9.102 Schematic influence of cross-sectional shape, cross-sectional area and filament surface area on thermophysiological comfort

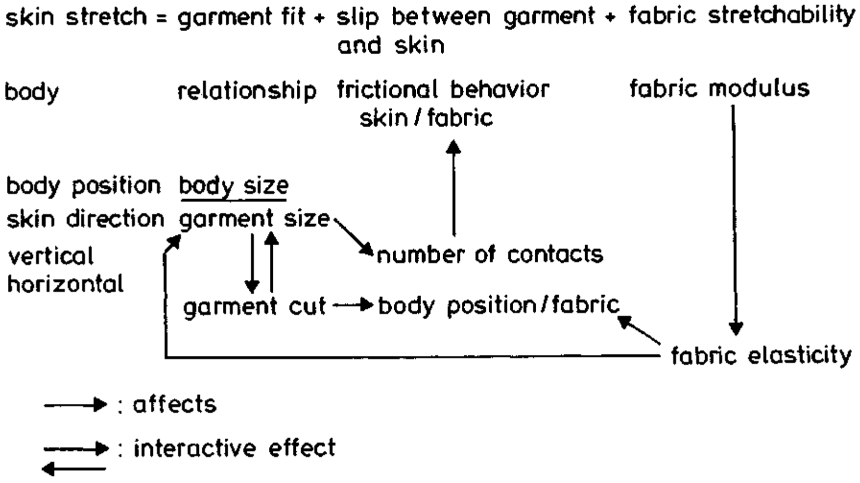


Figure 9.103 Relationship between fit and stretchability of textile fabrics

- Psychological comfort:
  Psychological comfort implies that the wearer feels that the clothing has the right cut, is fashionable and is correctly appointed for the activity in mind (e.g., job, sport, leisure), that the clothing matches his/her financial, social and functional status, and that the clothing confirms these values. The physical properties of the fibers, yarns and fabric have here hardly any contribution; styling, dyestuff selection, etc., are far more important (Fig. 9.103).

9.4.1.4 Fiber Mixtures

Synthetic-, chemical- and natural fibers each have their own specific properties, none of which alone may satisfy the requirement of a specific end use. In such cases, it can be advantageous to combine the properties of two or three fiber types in order to obtain an optimum for the end use in question or to enhance a particular product characteristic. As an example, a mixed yarn made from 84 dtex f 24 viscose (ca. 13% moisture content at 65% RH) and 44 dtex f 10 PA 6 (4.0% moisture content at 65% RH) has a cross-section as shown in Fig. 9.104 (Neva Viscon®) and constitutes a lining material having optimal wear properties in terms of physiology and behavior in usage.

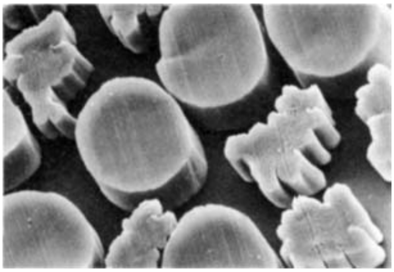


Figure 9.104
Heteroyarn: mixed filament yarn comprising "Neva Viscon" (viscose) and PA 6 (Akzo Nobel)

In principle, the following methods can be used to mix or blend fibers or yarns:

- combining different staple fibers or staple fiber with natural fiber in flock or at carding
- combining filament yarns and/or staple yarns by means of plying, twisting or entangling
- combining yarns during fabric manufacture (at weaving, in warp and/or weft; in netting structures by means of the yarn delivery system).

The following fiber combinations have proved themselves successful in terms of processability and optimization of product properties:

a) chemical- or synthetic fibers with natural fibers
polyamide with wool—polyester with wool, cotton or flax—polyacrylonitrile with wool—polypropylene with wool—viscose with wool and cotton
b) chemical- or synthetic fibers with one another
polyamide 6 with viscose, acetate, polyvinyl chloride, polyamide 66, polyester or electrically-conductive fibers (containing carbon or metal for antistatic protection)—polyester with polyacrylonitrile, elastane (spandex) or viscose—viscose with acetate, polyacrylonitrile or polypropylene
c) natural fibers with one another
wool with ramie, silk or mohair—mohair with silk—cotton with flax

The above list is by no means complete in itself, and furthermore, new fiber mixtures are constantly being devised for reasons of fashion and technological advance. Existing mixtures disappear from the market, sometimes re-appearing a few years later.

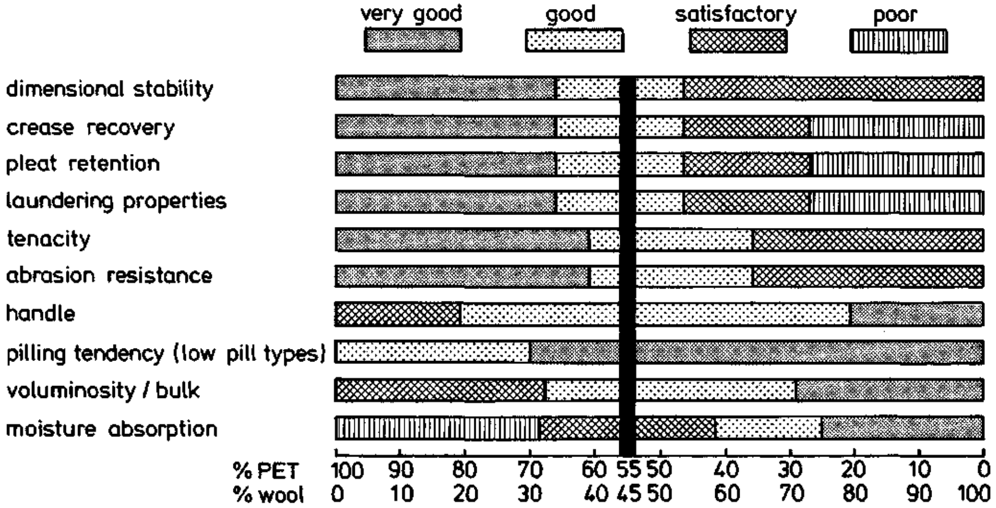


Figure 9.105 Effect of the blending ratio of two fiber components on aesthetic properties

Figure 9.105 shows the effect of continuously varying the proportions of PET and cotton on various aesthetic properties of the widely used cotton/wool blend [53]. From these results, one can conclude that the optimum ratio is 55% PET/45% wool.

Fiber combinations of staple or continuous filament yarns include mixtures of the same or different polymers having different morphological, physical and chemical properties (compare Section 9.23), e.g.:

a) morphological combinations involving fineness (fiber/filament), cross-section and crimp
b) physical combinations, particularly involving shrinkage (high/normal shrinkage), elastic modulus, flat/textured, luster (bright/matte).
c) chemical combinations, with a view to dyeing effects (differential dyeing), solution viscosity.

Table 9.27 gives a summary of the most commonly used blends, typical blend ratios and their end uses.

Table 9.27 Typical Apparel Fiber Blend Ratios

Blend components	Blend ratios %	Application examples
PET/cotton	50/50, 65/35, (67/33),	Underclothing, shirts, blouses, nightwear, clothing, poplin coats, work- and sportswear
PET/cellulose	70/30	
PET/linen	65/35, 80/20	Leisurewear, clothing
PET/silk	70/30, 75/25, 80/20, 85/15	Leisurewear, clothing
PET/wool	55/45, 70/30	Suits, trousers, costumes, dresses, coats, jerseys, pullovers, uniforms
PET/PAC	50/50, 60/40, 65/35, 70/30	Leisurewear, clothing, women's slacks, pullovers, jerseys, tableware
PAC/wool	55/45, 70/30, 60/40	Jerseys, clothing, pullovers, socks
PAC/cellulose	55/45, 70/30	Jerseys, clothing
PAC/linen	55/45, 80/20	Leisurewear, knitted goods
Wool/PA	75/25, 80/20, 85/15	Uniforms, socks, handknitting yarns
Wool/cellulose	50/50, 70/30	Suits, jackets, sports coats
PET/PAC/wool PET/PAC/cellulose PET/*PAC/others*	55/15/30, 30/40/30	Jerseys, clothing, pullovers
Staple combinations of high shrinkage PAC or high shrinkage PET	30...40% high shrinkage component	Jerseys,clothing, pullovers, handknitting yarns Trousers, clothing, jackets

9.5 Methods of Fiber Identification

Because of the existence detailed, specialized literature [46, 60, 23] and the availability of normed test methods [63], we confine ourselves here to identification tests related to application. The most commonly used fiber types are listed in Tables 9.28 and 1.1, as well as in the Fiber Table. Furthermore, many fibers are only known by their trade names, even when they are, in fact, normal fiber types, and show only small differences in identification tests and further processing. Table 9.28 gives the simplest characteristic differences between various fiber types. In Germany, the most commonly used identification methods involve dyeing or dissolution in solvents, while in the USA (as per ASTM) [63], infrared spectroscopy is the preferred method.

Table 9.28 Characteristics for Differentiation of the Most Important Textile Fibers

Investigative methods and fiber differentiation						Identification and separation of synthetic fibers			
Characteristic fiber	Color	Microscopic view		Burning test	Dry distillation	Fluorescence in UV light	Melting point determination	Solubility	Dyeing
		longitudinal	cross-section						
Nylon 66	Bright luster, white	Smooth structureless	Round to weakly oval	No ignition Melts slowly to form a dark brown mass having small bubbles Pyridine smell	Litmus paper: alkaline (blue)	Blue-white	S_p: 250 °C Melting point salt: $KClO_3$ (250 °C)	Easily soluble in chlorohydrate 1.9 g/cm^3 H_2O Insoluble in chloralhydrate 1.25 g/cm^3 H_2O	Dyestuff III: greenish-black Neocarmin W: yellow Neocarmin MS: red-orange RI: bright yellow
Nylon 6	Lustrous, white, transparent	As for Nylon 66	As for Nylon 66	As for Nylon 66	As for Nylon 66	As for Nylon 66	S_p: 215 °C Melting point salt: $KHSO_4$ (210 °C), p-oxybenzoic acid (215 °C), anthracene (213 °C)	Slowly soluble in chloralhydrate 1.9 g/cm^3 H_2O Slowly soluble in chloralhydrate 1.25 g/cm^3 H_2O	Neocarmin W: yellow Neocarmin MS: red-orange
Perlon U		Lengthwise cracks	Round	Melts together without turning brown	Alkaline fumes			Soluble in m-cresol, conc. formic acid Insoluble in warm conc. HCl	No affinity for most acidic dyestuffs; not dyed by Sänger reagent: other polyamides stain light yellow (66% ethyl alcohol + 2% dinitrofluorobenzol + 2% sodium bicarbonate)

Table 9.28 (*Continued*)

Investigative methods and fiber differentiation						Identification and separation of synthetic fibers			
Characteristic fiber	Color	Microscopic view		Burning test	Dry distillation	Fluorescence in UV light	Melting point determination	Solubility	Dyeing
		longitudinal	cross-section						
Polyester (Terylene)	Slightly ivory to white, opaque	Smooth, structureless	Round, limited smoothness	No ignition Melts together No specific smell	Initially neutral, on further heating, acidic		S_p: 257 °C	Insoluble in chlorinated zinc iodide solution: is tinted weakly yellow; Insoluble in cold, liquid phenol; Soluble in hot phenol and in hot nitrobenzene	Dyestuff III: dirty yellow Neocarmin MS: pale violet
Polyacrilonitrile (Orlon)	Weakly amber (cream colored)	Slight lengthwise cracks	Dumb-bell to bisquit-shaped	Browns and melts together, then burns, emitting yellow soot; Flames; No to slightly sweet smell	Initially neutral fumes; On further heating, alkaline	Intense yellow	Becomes sticky above 225 °C	Insoluble in p-nitrophenol (70%, hot), acetone, phenol (80%); Soluble in calcium rhodanide (70%, cold), dimethylformamide (hot), cold, conc. nitric acid	Dyestuff III: brownish pink Neocarmin MS: Orlon, Pan, Dralon, etc. dye green Other polyacrolonitriles dye yellow to yellow-green
Dynel	Golden yellow to clear	Smooth	Dumb-bell shaped	Not flammable; Burns while in the flame, but self-extinguishes on removal	Litmus paper: acidic (red)			Soluble in acetone, cyclohexanone, phenol (80%)	Dyestuff III: red-brown Neocarmin MS: pink

Table 9.28 (*Continued*)

Investigative methods and fiber differentiation						Identification and separation of synthetic fibers			
Characteristic fiber	Color	Microscopic view		Burning test	Dry distillation	Fluorescence in UV light	Melting point determination	Solubility	Dyeing
		longitudinal	cross-section						
PCU	White	Fine grooves	Elongated, with fine grooves on circumference	Incombustible, not flammable; Fuses into a black mass Smell of burning	Litmus paper: acidic (red)			Insoluble in trichloroethylene, xylene, acetone	Neocarmin MS: dark pink
PC	White	Smooth	Kidney-shaped	As for PCU	As for PCU	Matt blue-green		Soluble in trichloroethylene, xylene, acetone	Neocarmin MS: dark pink
Polyvinyl alcohol		Fine lengthwise cracks	Bean-shaped	Difficult to ignite			Shrinkage point 210 °C; browns noticeably		Dissolve in 30% H_2SO_4 and add a few drops of iodine/potassium iodide or carbazol sulfuric acid: blue color
Polyethylene	Dirty gray-white	Smooth	Round	Ignites and burns slowly, like high melting point wax			S_p: 105–110 °C	Soluble in boiling xylene	
Teflon		Smooth	Round	Glows in the flame, but does not burn; Melts at 400 °C with decomposition			Decomposition temperature: 399 °C	Soluble in molten alkali metals; Soluble in fluorine gas and trifluorochloromethane at high temperature and pressure	

Table 9.28 (*Continued*)

Investigative methods and fiber differentiation						Identification and separation of synthetic fibers			
Characteristic fiber	Color	Microscopic view		Burning test	Dry distillation	Fluorescence in UV light	Melting point determination	Solubility	Dyeing
		longitudinal	cross-section						
Polystyrol	Transparent	Smooth	Round					Insoluble in 20–25% H_2SO_4 and glacial acetic acid; Soluble in chloroform and tetrachloromethane	Dissolve 0.1 g of polystyrol in 5 cm^3 conc. HNO_3 to form a clear yellow solution; Addition of 20 cm^3 H_2O results in a lemon-yellow precipitate; Extract twice with ethylether, then twice with dil. NaOH; Neutralize with HCl Add zinc and warm, followed by a few drops of 5% aqueous sodium nitrate solution and alkaline β-naphthol solution: bright red color
Polybutadiene	Gray to black	Smooth	Round, square, etc., according to spinneret hole						Not dyeable except in spinning solution
Wool		Overlapping scales	Round or weakly elliptical	Leaves an irregular, bloated, black mass, in some ways like silk		Bluish-white		Insoluble in cold H_2SO_4 (80%) and conc. HCl up to 40 °C Soluble in boiling 5% NaOH and sodium hypochlorite	Dyestuff III: black; RI: green

Table 9.28 (*Continued*)

Investigative methods and fiber differentiation						Identification and separation of synthetic fibers			
Charact-eristic fiber	Color	Microscopic view		Burning test	Dry distillation	Fluorescence in UV light	Melting point determination	Solubility	Dyeing
		longitudinal	cross-section						
Silk		Smooth	Round to rounded triangle	Burns constantly with weak hissing Smells of burning hair; Extinguishes on removal from flame		Bluish-white	Decomposition temperature ca. 170 °C	Soluble in 10 parts $ZnCl_2$ + 10 parts H_2O + 2 parts ZnO and in 10 g $CaCl_2$ in 100 cm^3 90% formic acid, or in boiling 5% NaOH	Dyestuff III: black; RI: blue
Cotton	Matte white	Tortuous	Irregular, dumb-bell shaped	Burns with a blue flame		Yellow-white to yellow		Soluble in ammoniacal copper oxide	Dyestuff III: mid-gray; RI: bright-to mid-yellow
Cellulose hydrate (viscose)	Slightly off-yellow	Lengthwise stripes to fine surface structure	Irregular, with more or less large notches	Burns with blue flame; Smells of burning paper		Yellowish-white to yellow (Cupra: reddish white)			Dyestuff III: pink (violet-gray) Neocarmin W: red to red-violet Neocarmin MS: blue-violet to violet RI: deep yellow
Cellulose acetate	Lustrous white	Lengthwise stripes	Highly folded			Bluish-violet		Soluble in acetone	Dyestuff III: bright olive Neocarmin W: greenish-yellow Neocarmin MS: orange RI: red

Note:
RI = Réactive Identex (S.A. Francolor, Paris, France). Dye using 2 g/l at 90 . . . 95 °C, liquor ratio 1 : 40, wash and treat twice for 3 min in 5 g/l Primatex CG at 80 °C. Wash cold and dry.
Neocarmin: Supplied by Fesago, Chem. Fabrik Dr. Gossler GmbH, Heidelberg, Germany.
Dye mixture III: Chromorhodine BR + Cellitonechtgelb C + Acilandirektblau: 0.5 g in 200 cm^3 H_2O + 1 cm^3 acetic acid (56%). Boil fiber in mixture for 3 min, then rise at 40 °C (according to Schaeffer).

9.5.1 Diagnostic Dyeing Tests

Four diagnostic dyestuffs [60] are used: Neocarmin W, -MS, -TA and Carmine Azurol [65]. Each dyestuff will dye a given fiber to a particular color which is characteristic for that fiber type (Table 9.29).

Dyeing method: Neocarmin dyeings require 5 minutes. Neocarmin W is dyed cold: Neocarmin MS and TA at the boil; Carmine Azurol requires 10 minutes at 85–90 °C, followed by rinsing until the water is clear. These test dyeings can only be used as a pre-test, as slight differences in polymer constitution make themselves noticeable. Pretreatment and copolymerization can also produce considerable differences. Mixed yarns or differences in the warp and weft can easily be detected. Fibers which have dyed differently can be easily separated mechanically. Heavily dyed samples cannot be dyed with the above diagnostic dyes. Bicomponent fibers can usually be easily identified in microscopic cross-section.

9.5.2 Microphotographs of Fibers

Longitudinal- and cross-sections of unidentified fibers can be compared with sections of known fibers as an aid to identification. For longitudinal viewing, the fiber is laid in distilled water or diluted glycerin. During cutting, the cross-section must not be damaged: this can happen in the case of shear- and cross-sections. A darning needle can be used to pull a fiber bundle through a cork (or Plastiline), after which the cork is sectioned using a sharp razor blade in a pulling mode to give a thin section, which can be placed on a glass slide with a drop of water, covered with a cover glass and examined under the microscope. Examples of fiber microphotographs can be found in this chapter, in the Fiber Table and in the literature [62,63]. Particularly clear pictures are obtained from the scanning electron microscope (SEM).

9.5.3 Solubility

No knowledge of chemistry is necessary for these tests. A requirement, however, is an unambiguous solubility reaction, e.g., solution of PAN in cold glacial acetic acid and acetate is conclusive, but dissolution in boiling DMF, however, is not conclusive proof of PAN. For this reason, it is necessary to complete the whole solubility test scheme [46,63,67], from which Table 9.30 is derived [60]. A simplified scheme, valid for synthetic fibers and acetate [62], is given in Fig. 9.106 and in [63].

These tests can be further refined by modifying the solvent by addition of a miscible component, distilled water (usually) or methanol. Fiber/solution mixtures which are difficult to resolve can be made to reveal their components by this method, or the dissolved component can be precipitated.

These investigations can be done either macroscopically (in a beaker) or microscopically on a glass slide. The type reactions below are based on the above methods.

9.5.4 Type Reactions [60]

These are special reactions, valid for every type of fiber, which—if fully carried out—give an unequivocal identification. They should only be applied when the identity of the fiber has already been guessed, or a preliminary test has suggested a fiber type.

With this as prerequisite, the fibers can be allocated to solubility groups (according to Stratmann: I to IV [60]). Tables 9.29 and 9.30 show how to proceed further.

9.5.5 Embedding the Fiber in Specific Reagents

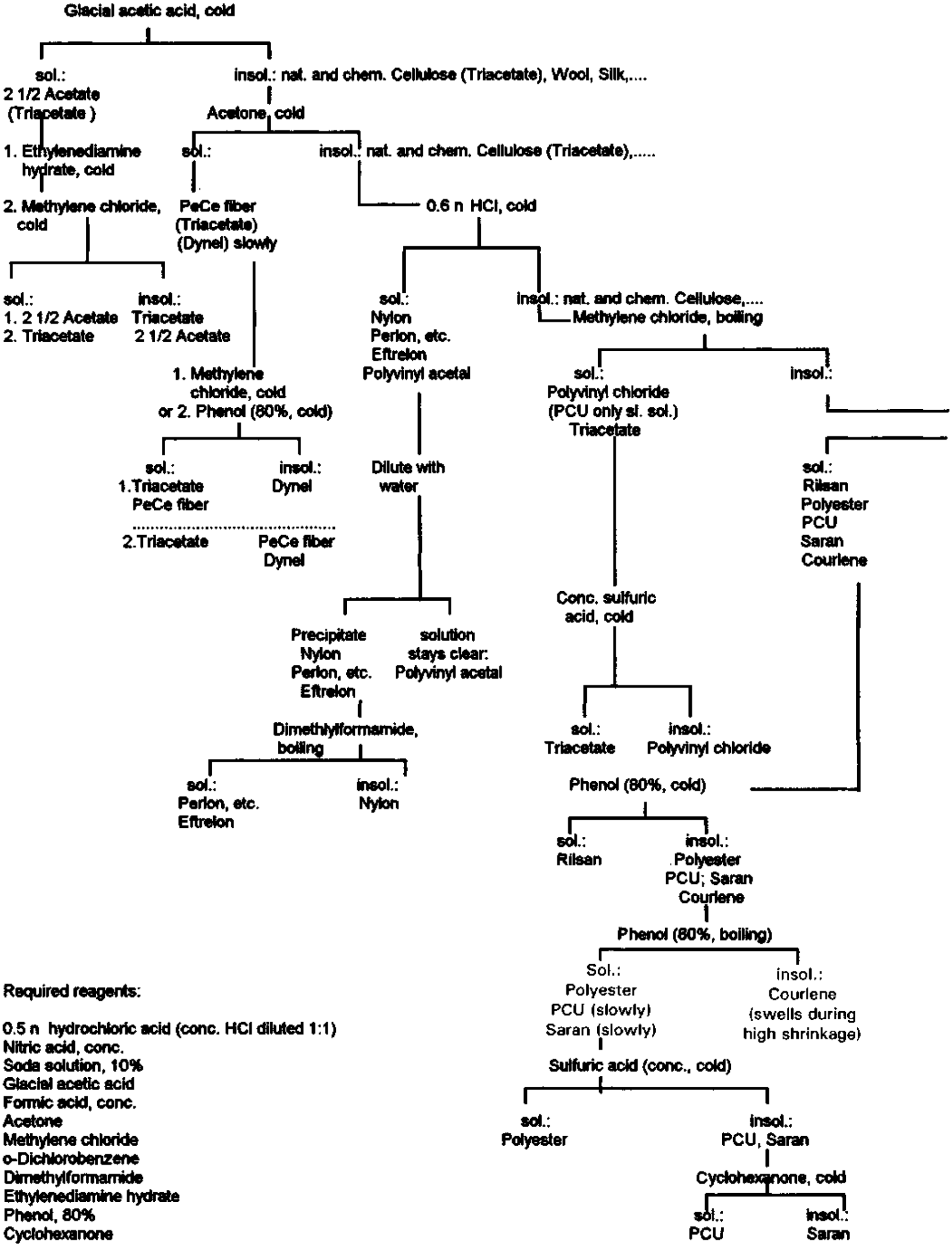
If the microscopic examination is carried out with the fiber immersed not in an inert medium, but in a swelling or dissolving medium, many fibers follow a specific reaction course. According to the speed of penetration through the skin or the cut end, the swelling can appear tulip-, trumpet-, hat- or mushroom-shaped [60].

Table 9.29 Diagnostic Dyeing of Fibers Using Test Dyestuffs [25]

Dye Fiber		Neocarmin W	Neocarmin MS	Neocarmin TA	Carminazurol
Cotton	Co	pink to blue	violet	–	wine red
Kapok	Kp	yellow	pink-violet	–	blue-violet
Flax	Fl	pink to blue	purple	–	wine red
Hemp	He	pink to blue	bright violet	slightly dirty blue-gray	dark wine red
Ramie	Ra	pink to blue	lilac-blue	–	wine red
Jute	Ju	dirty yellow	red	slightly dirty pink	dark blue
Wool	Wo	strongly yellow	red	strongly blue	bright blue
Mulberry silk	Ms	Fibroin: yellow Serizin: blue	Raw silk: strongly red Boiled-off: strongly pink	Fibroin: pale yellow Serizun: blue	red-violet
Tusseh silk	Ts	Fibroin: yellow Serizin: blue	red-blue	strong dirty blue	strong bright blue
Cupro	CC	blue	strongly blue	–	red
Viscose	CV	pink, lilac, blue	strongly violet	–	red
Acetate	CA	yellow	red-orange	pink-yellow	–
Triacetate	CT	pale yellow	yellow	golden yellow	–
Alginate	AL	brown-violet	blue-violet	dirty bright blue-green	red-brown
Regen. protein	KA	strongly yellow	red, purple, brown, violet to black	dark blue-black	strongly violet-blue
Polyamide 3, 6, 66	PA 3, 6, 66	bright yellow PA 3: orangish	strongly orange	dark blue-green	blue
Polyamide 11	PA 11	pale yellow	yellow-orange	dark blue-green	pale gray-blue
Polyamide 12	PA 12	pale yellow	yellow-orange	strongly blue	pale lilac
Qiana	PA (Q)	–	pale beige	bright greenish-blue	pale gray-blue
Nomex	PA (N)	–	pink	–	bright greenish-blue
Polyurea	PUA	bright yellow	orange-yellow	dark greenish-blue	gray-blue
Polyester	PES	–	pale dirty pink	yellow (Dacron 64: pink)	pale blue
normal types					
Vycron	PES (V)				
PES Grilene	PES (G)	–	very pale pink	yellow	pale blue
A-Tell	PES (A)				
PES Kodel	PES (K)	–	–	yellow	pale blue
Polycarbonate	PC	–	– or pale pink	bright yellow	bright blue
Acrylic	PAC	–	yellow-green, orange, pink-blue	blue (basic copolymer) pink (acidic copolymer)	bright blue
Polyvinylchloride	PVC	–	strong pink-blue	dirty yellow-pink	bright blue
PVC post-chlorinated	PVC+	–	strong pink-blue	dirty yellow-pink	bright blue
Polyvinylidene	PVC	–	strong pink-blue	bright yellow	bright blue
Copolymers	PVM	–	pink	dirty pink	pale blue
Vinal	PVA	blue, starts dissolving	dissolves	dissolves	dissolves
Vinylal	PVA+	yellowish, blue	violet	dirty pink; yellow-green	violet-blue
Polyolefine	PE, PP	–	pale to strong pink	yellow	bright blue
Fluoroethylene	PTFE	–	–	–	–
Polyurethane	PUR	pale yellow	salmon	bright blue-green	very pale gray-blue
Rubber	LA	yellow	red-blue	dirty olive	bright blue
Elastomer	PUE	yellow	red	olive, green to blue-green Vyrene: dirty pink	blue

The information in the table is based on results from normal production. Deviations can be caused by polymer modification.

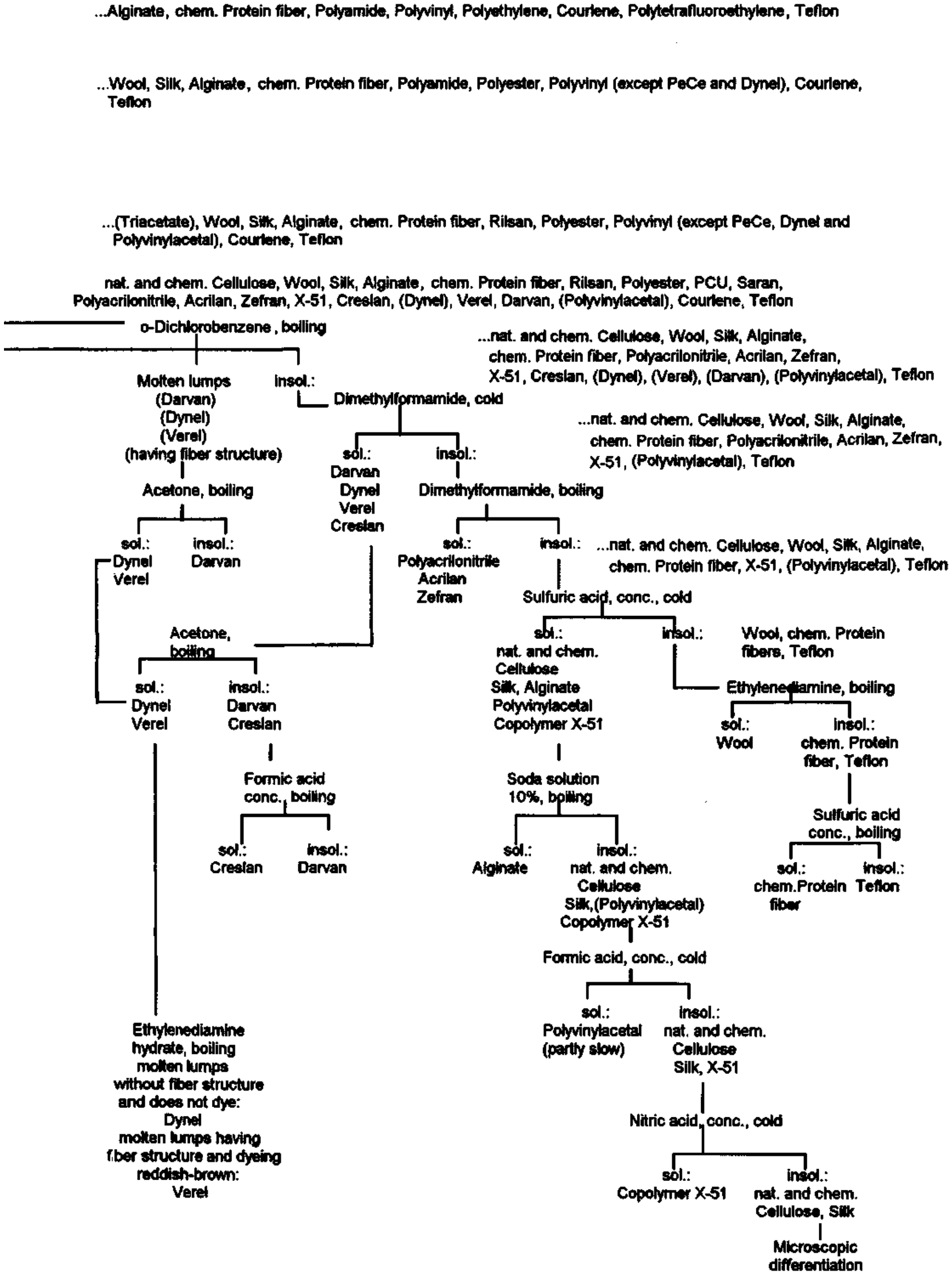
Table 9.30 Scheme for Qualitative Chemical Analysis of Fibers (acc. to M. Stratmann)



Required reagents:

0.5 n hydrochloric acid (conc. HCl diluted 1:1)
Nitric acid, conc.
Soda solution, 10%
Glacial acetic acid
Formic acid, conc.
Acetone
Methylene chloride
o-Dichlorobenzene
Dimethylformamide
Ethylenediamine hydrate
Phenol, 80%
Cyclohexanone

Table 9.30 (*Continued*)



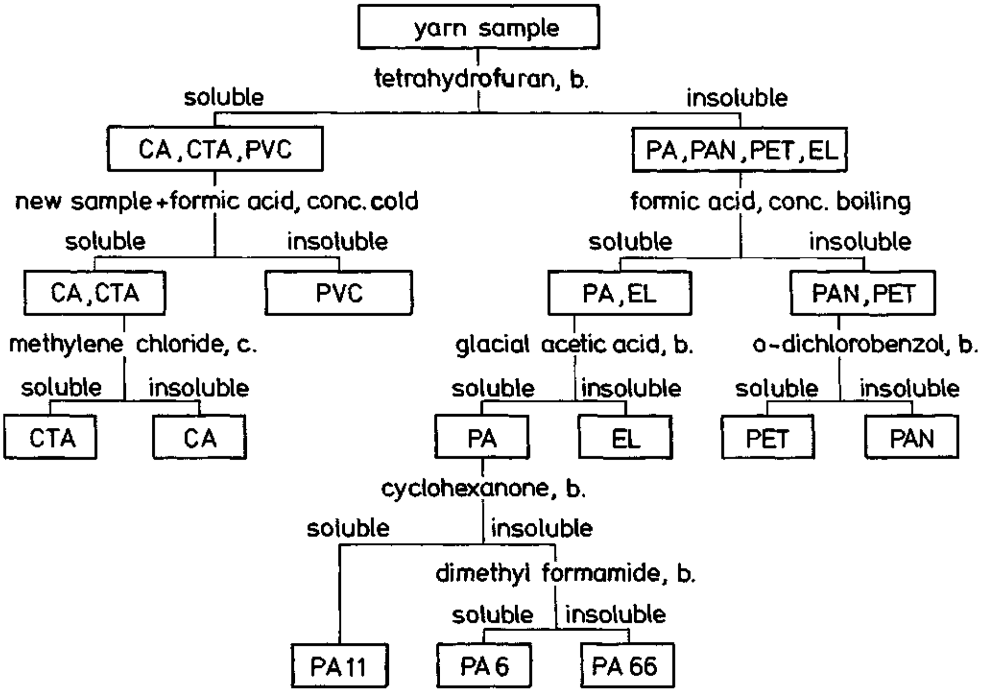


Figure 9.106 Simplified analytical scheme for identification of fibers, based on solubility [29]
c = cold, for 30 min; b = boiling, for 30 min. For fiber type abbreviations, see Table 10.5

Using this method, the degree of heat setting of polyester yarns can be determined by iodine absorption [68]. The iodine absorption (mg I/g fiber) depends on the specific surface area. Figure 9.107 shows the functional relationship.

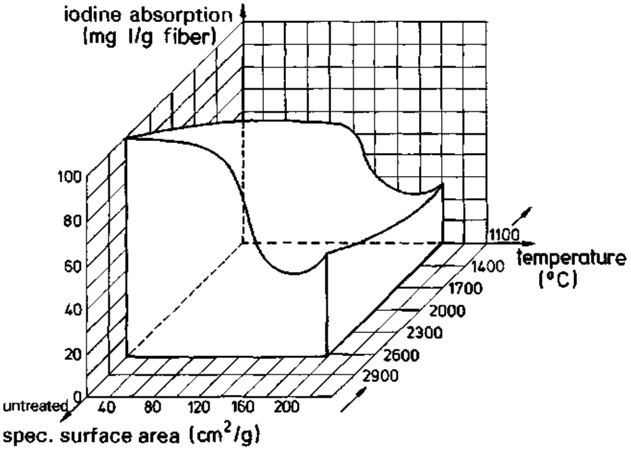


Figure 9.107
Iodine absorption of polyester yarn as a function of specific surface area and heat setting temperature [35]

9.5.6 Thermal Tests

Included here are:

- Melting point determination: This can be done under a microscope fitted with a melting stage or, more indirectly, by thermomechanical analysis (see Section 9.3.1). If the heating rate is sufficiently low, the melting point determination gives exact results. Even small differences can be used to differentiate fiber types. The melting point can also be determined microscopically under polarized light, since birefringence disappears instantaneously on reaching the melting point.
- Dry distillation: The fiber sample, together with a moistened cotton-wool ball, is sealed in a glass tube and heated to red heat. The tube is then cooled down, broken and the pH value of the moist cotton-wool ball is measured and the result compared with the values given in Table 9.31. The pyrolysis residue can also be examined using thin layer chromatography.

Table 9.31 Typical Behavior of Man-Made Fibers During Burning and Dry Distillation

Fiber	F_P °C	Smell	Residue	pH value of vapor evolved during dry distillation
PAC	carbonizes	Sweet, aromatic	Hard, brittle, black irregular pearls (globules)	10...11
PA 6	215...220	Weak smell of burning hair, not pungent	Hard, tough, gray-brown, round pearls	10...11
PA 66	255...260			
PET	250...260	Sweet aromatic, slightly pungent	Hard, tough black-brown round pearls	3...4
PVC	160...200	Pungent, irritant, causes coughing	Hard, black irregular pearls	1
PVA	carbonizes	Similar to burnt sugar	Brownish, brittle	4...5
PP	160...175	From burning paraffin to undefinable	Light brown, round pearls	6...7

- Burning test: A small fiber sample is brought close up to a flame, is slowly introduced into the flame and is then removed from the flame. All changes and odors are noted and compared with those listed in Table 9.31 or with experience.
- DSC or DTA methods: Here it is also possible to identify partly crystalline polymers, relying particularly on melting point, heat of fusion and reaction temperature and -energy. The melting point is characterized as an endothermic peak in the diagram, while solid state polycondensation is an exothermic process. The total energy can be measured by planimetry.

Measurements are usually carried out from 40 °C to 40 °C above the melting point on samples of 0.3 ... 3 mg size at heating rates of 20 °C/min. Figure 9.108 shows an evaluation of the results of such a measurement, giving the enthalpy H and the specific heat cp, as a function of temperature for isotactic polypropylene and two polyethylene types. Table 9.32 gives results for other polymers.

These methods can also be used to elucidate the prehistory of the fiber. Figure 9.109 shows, for PA 66 (Nylon) and PET (Terylene®), the effect of changing quenching conditions at spinning (with quench or without quench, as per program). Interpretation is, however, a matter of experience and still needs to be catalogued.

9.5.7 Infrared Spectral Analysis

ASTM [63] gives detailed rules. Figure 9.110 shows an on-line measuring equipment, Fig. 9.111, a scheme for the analytical procedure. A double-beam infrared spectrometer is used. Measurements require both experience and reliability of equipment. Measurements can be done on solids (fibers or films), as well as on melts, the latter being particularly useful for analysis of components and impurities.

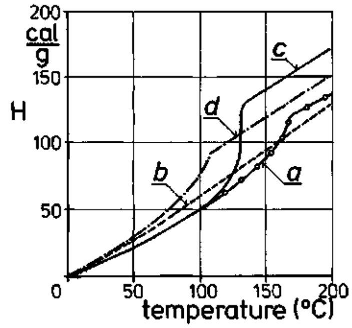
Table 9.32 Results from DSC Measurements on Various Fiber Types [38]

Fiber	Name/trade name	Melt		Filament
		Melting temp. °C	Heat of fusion cal/g	°C
PA	PA 6	217	12	
	PA 66	257	14	
	PA 610	218	14	
	PA 11	185	10	
PET	Terylene, Dacron, Diolen Kodel 411, Kodel 211	249	7.5	140...150
Polyolefin	PE (branched)	102	14	
	PE (linear)	128	35	
	PP (isotactic)	156	15	
Polyvinyl chloride	Saran	162	4.0	
Cellulose acetate	Arnel, Tricel	278...296	2.2...7.8	120...145 206

Table 9.33 Recommended Methods for Producing Films of Acceptable Quality for Infrared Spectroscopy

Fiber type	Film cast from
Acetate	Acetone solution
Modacrylic	Acetone- or butyrolactone solution
PA 6, 66, etc.	Melt
Polyacronitrile	Acetone- or butyrolactone solution
Polyolefin	Melt
Polyester	Melt
Saran	Melt
Cellulose triacetate	Methylene chloride solution
Vinal	Methylene chloride solution
Vinyon	Methylene chloride solution

(The methods listed are known to be practicable. The list is not, however, complete in terms of fiber types and/or alternative solvents)



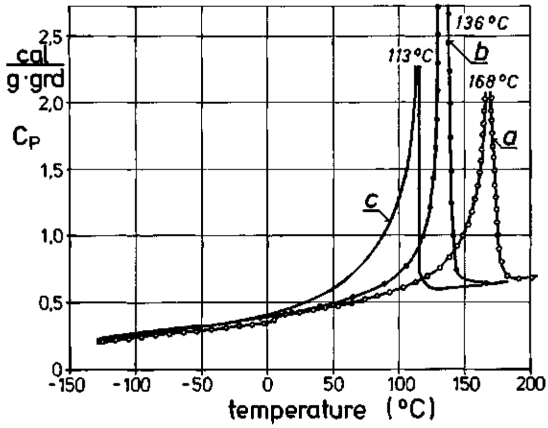


Figure 9.108
Enthalpy H, based on 0°C (top), and specific heat c_p (bottom) as functions of temperature *a*) Isotactic polypropylene *b*) Atactic polypropylene *c*) Linear polyethylene *d*) Branched polyethylene

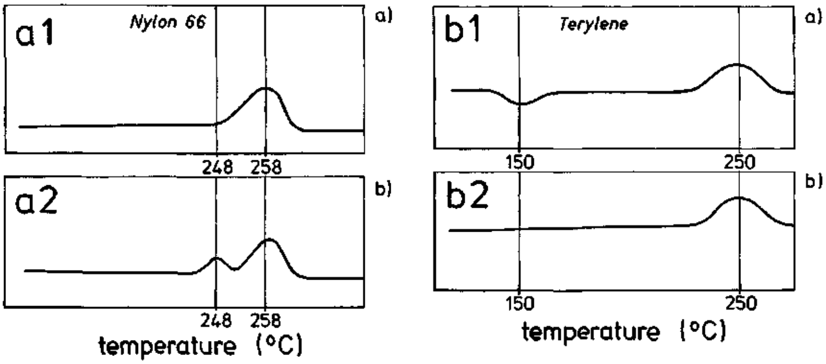


Figure 9.109 DSC heat flow diagrams for two synthetic fibers cooled under different conditions
a) PA 66 (Nylon 66) *1* cooled in quench chamber
b) PET (Terylene) *2* cooled according to program

- Fiber analysis (Table 9.32 [63]).

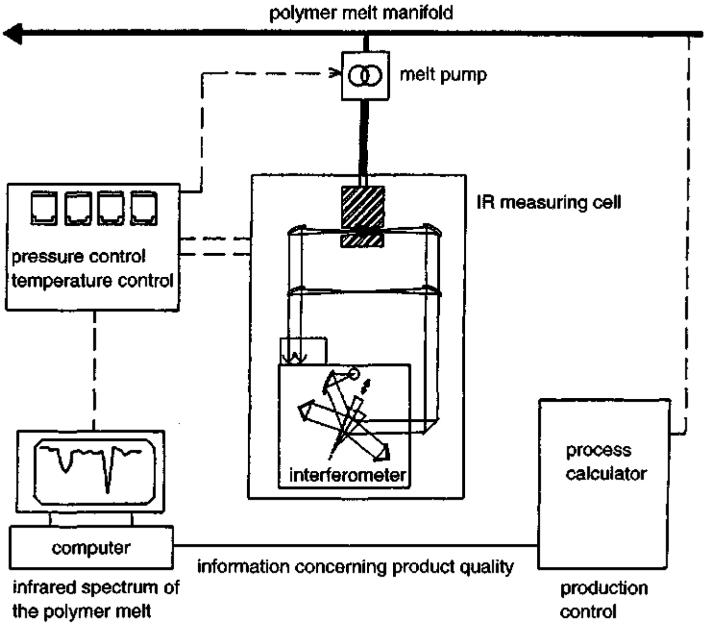


Figure 9.110
Schematic of on-line infrared spectrometer

In the case of a mixed yarn, the fiber components must be separated, optimally by dissolving the fiber to be investigated using a very pure solvent taken from Table 9.33. Alternatively, separation can be done by melting under an inert gas in order to obtain a cast film. These are TFE-fluorocarbon platelets which are pressed in a hydraulic press at a pressure of 14 MPa (ca. 15 bar) for 2 ... 5 min. In the measuring cell they are scanned over a wavelength range of 2.5 ... 15 µm. One obtains infrared absorption diagrams which can then be compared with known, catalogued diagrams. Here the smallest deviations in polymer composition or the presence of impurities can be detected in the spectrogram. ASTM [63] cites 31 examples, of which Fig. 9.112 is an extract of frequently-occurring

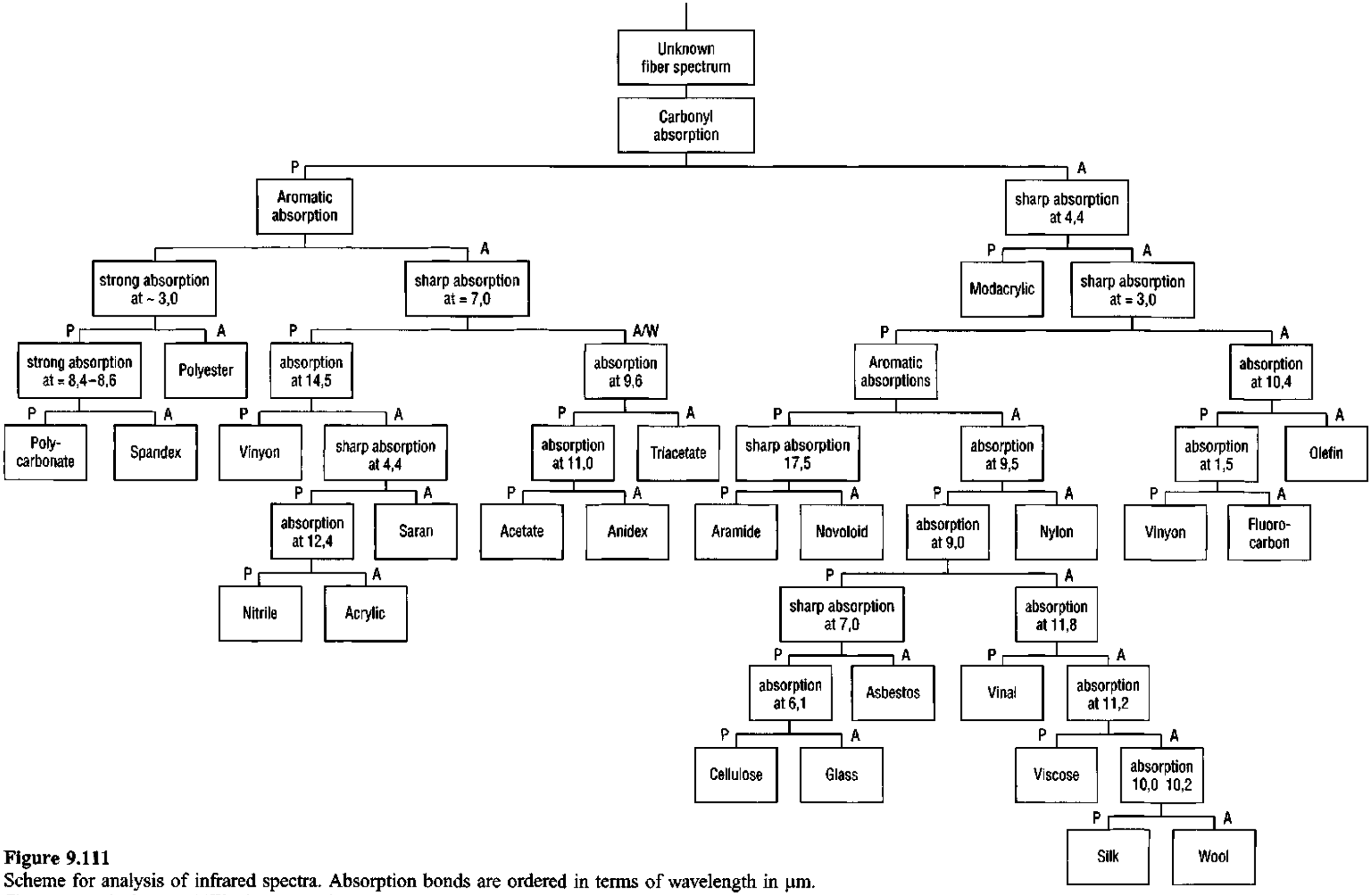
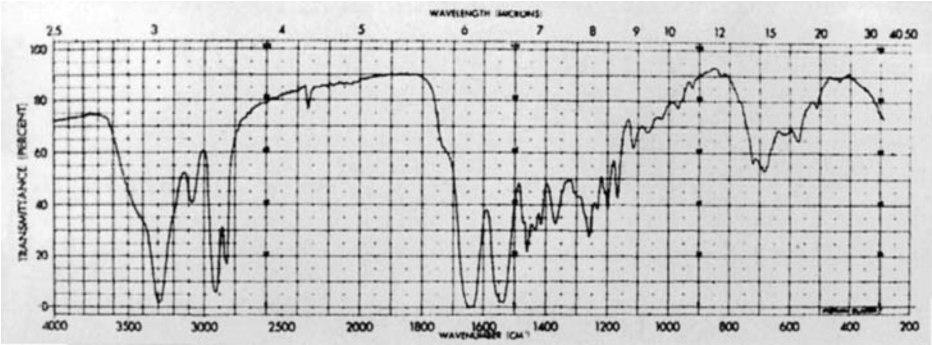
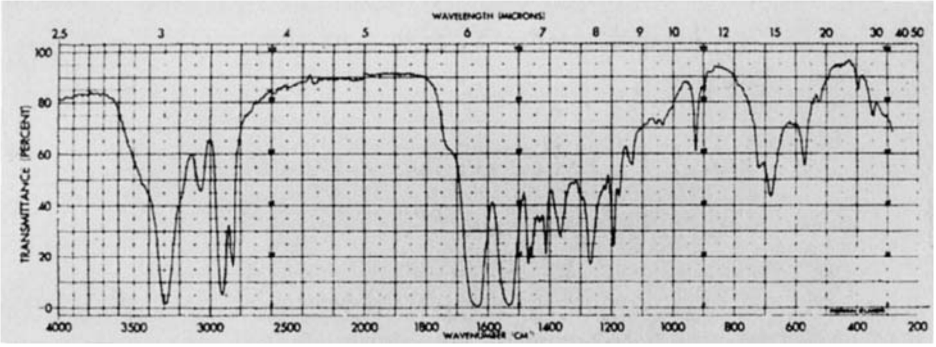


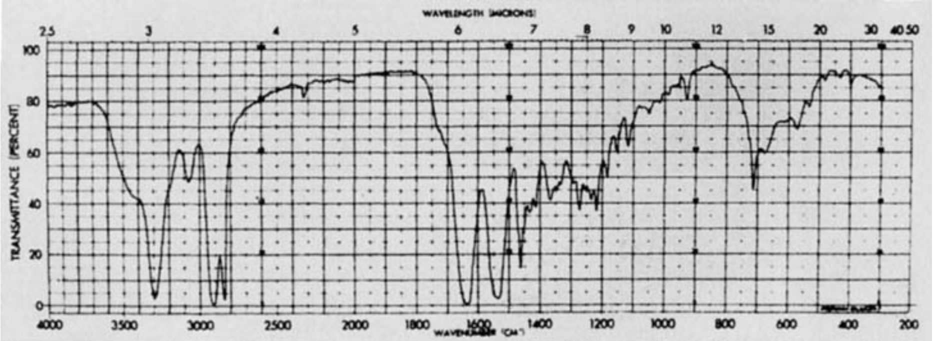
Figure 9.111
Scheme for analysis of infrared spectra. Absorption bonds are ordered in terms of wavelength in µm.
P) present; *A*) absent: *A*/*W*) absent/weak
A) present at 5.75 µm; *B*) present at 6.23, 6.30 and 6.70 µm



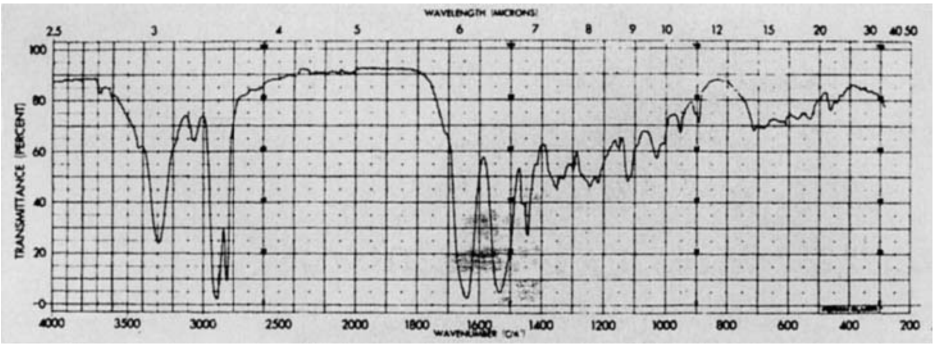
a)
PA 6



b)
PA 66

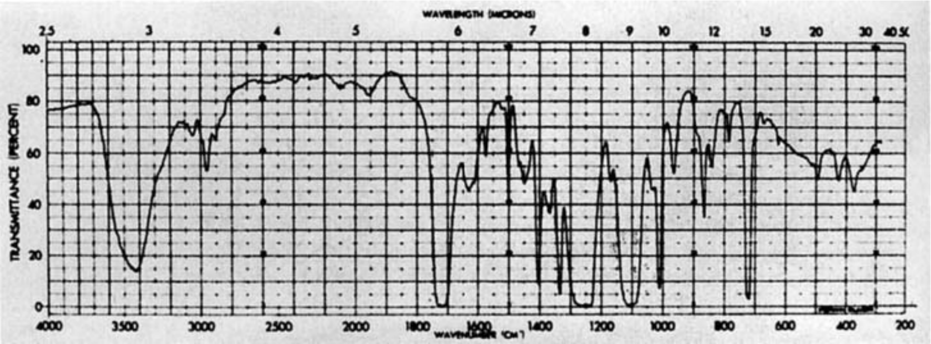


c)
PA 11

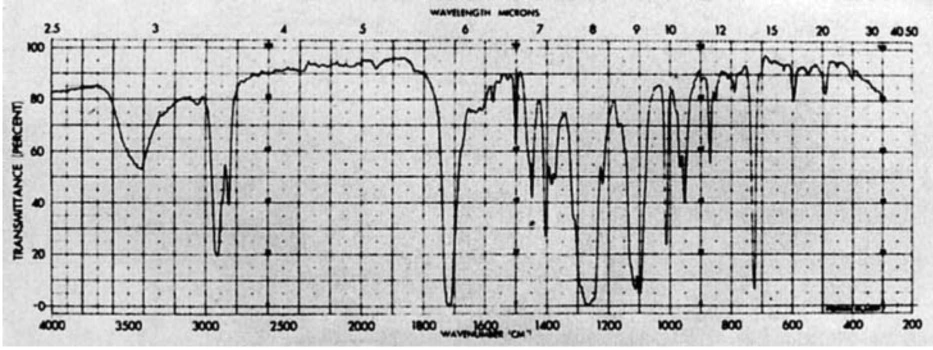


d)
Qiana
(DuPont)

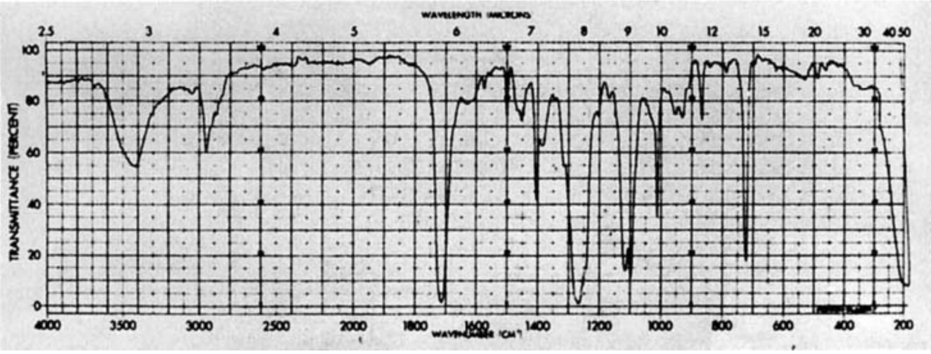
Figure 9.112 Typical fiber (film) infrared spectrograms
Typical polyamide fibers



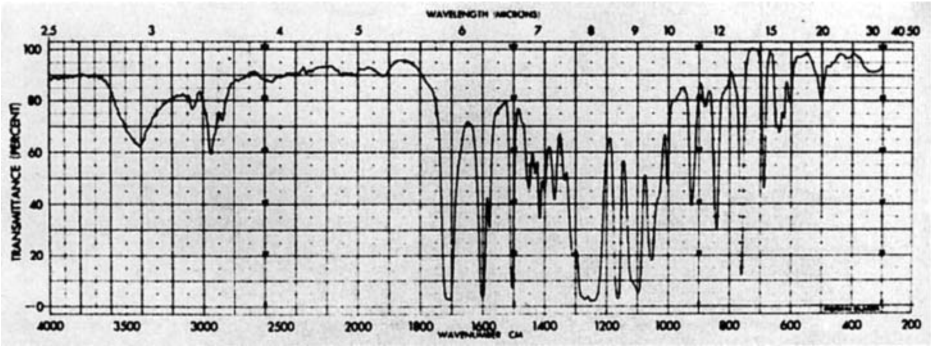
e) PET (e.g. Dacron, Terylene, Diolen, Trevira etc.)



f) (1,4-) Cyclohexane-dimethano (Kodel)

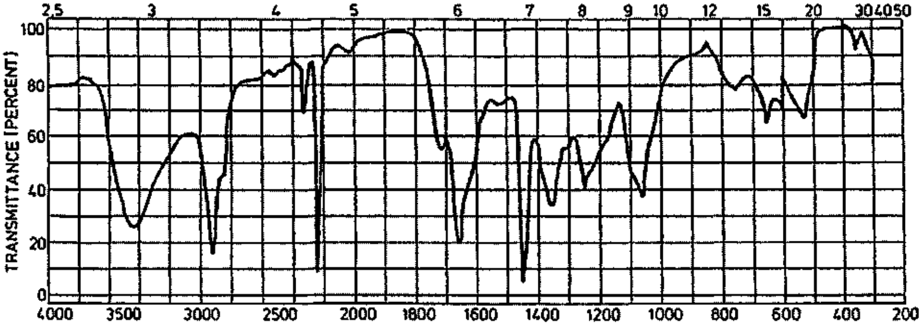


g) (1,4-)Butanediol (Fortrel)

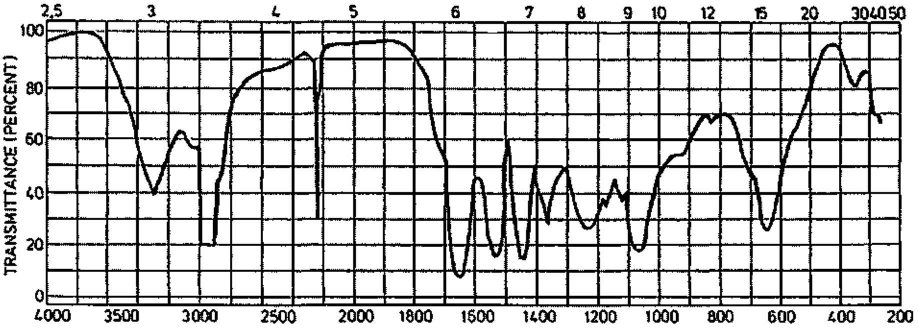


h) PET-oxy-benzoat (A-Tell)

Figure 9.112 (*continued*) Typical fiber (film) infrared spectrograms
Typical polyester fibers

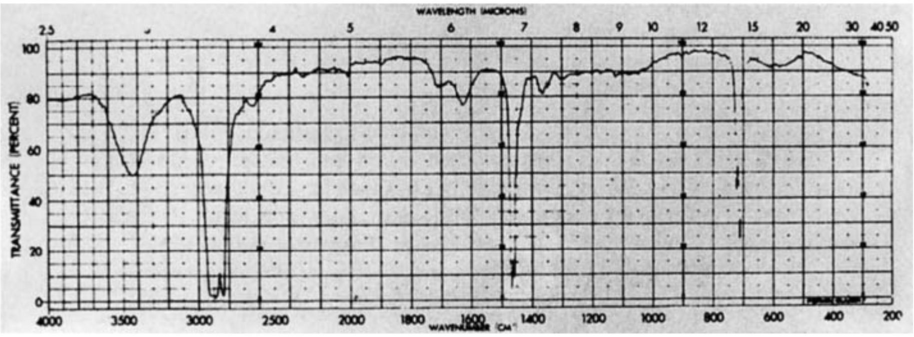


i)
PAN
(Acrilan,
Orlon,
Dralon, etc.)

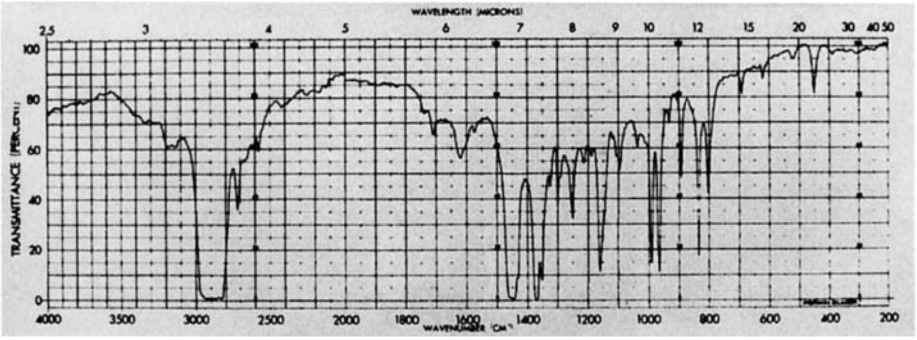


j)
Modacrylic
MAC
(Dynel,
Verel, etc.)

Olefin fibers

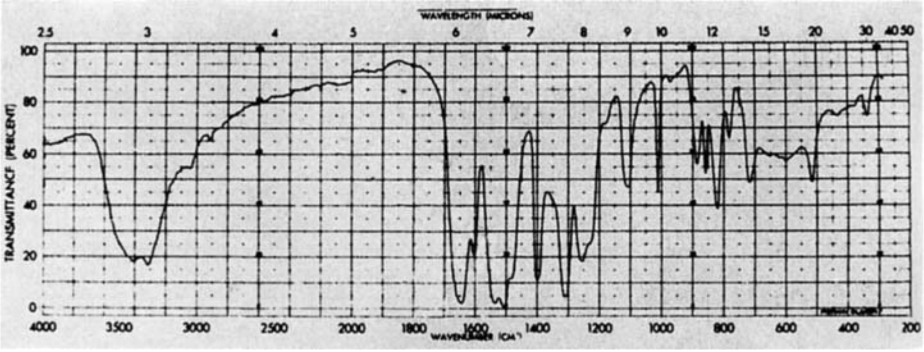


k)
Polyethylene

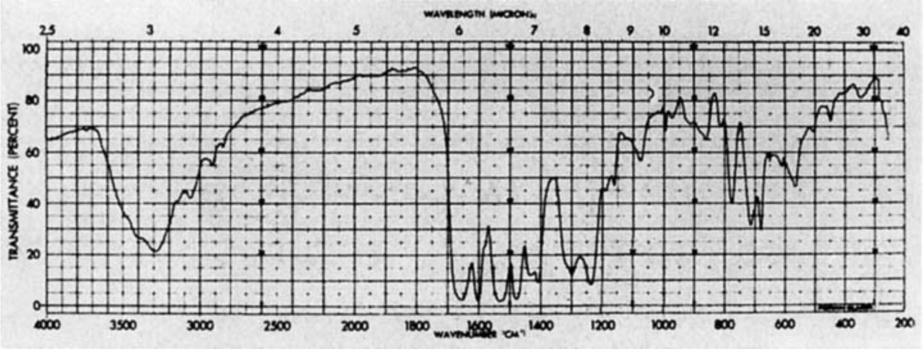


l)
Polypro-
pylene

Figure 9.112 (*continued*) Typical fiber (film) infrared spectrograms
Typical PAN fibers

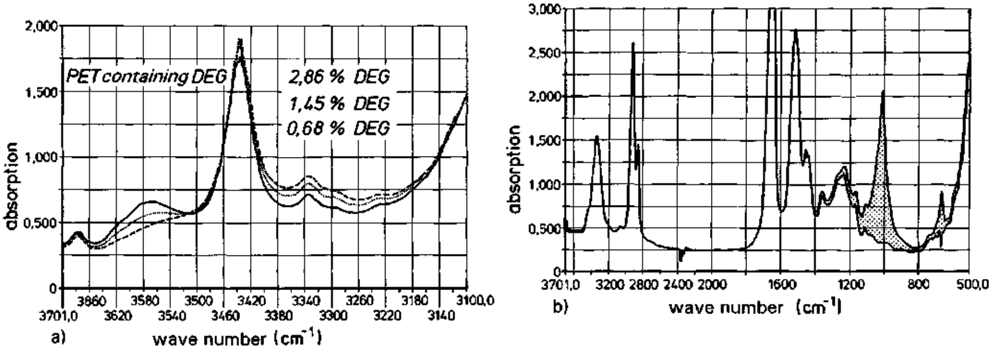


m)
Kevlar



n)
Nomex

Figure 9.112 (*continued*) Typical fiber (film) infrared spectrograms
Typical polyaramid fibers



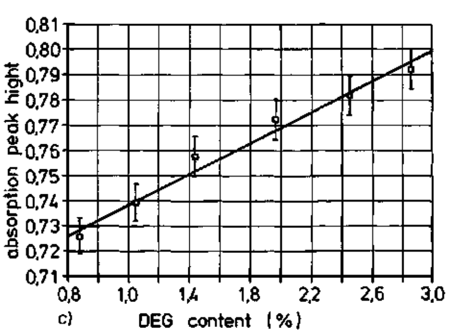


Figure 9.113
(*a*): Section of IR spectrum of molten PET containing various DEG (diethylene glycol) concentrations: $a = 1\%$, $b = 2\%$, $c = 3\%$ DEG
(*b*): IR spectrum of pure PA 6, and PA 6 containing 13% talcum powder as filler (shaded). The main differences are found at a wave number of 1030 cm^{-1}
(*c*): Absorption peak height versus DEG content of PET

synthetic fibers. One should, however, identify one's own measurements by fiber type, chemical analysis, trade- and manufacturer's name, date, etc. and archive it for categorization later.

- Melt investigation
  Instead of using static films derived from solution or melt, one can pump a melt or solution continuously through a specially-constructed cell. Since the main product is known, one can obtain a deviation signal by comparing the actual signal with that of a standard infrared spectrogram, and thereby obtain a continuous product analysis: uniformity, purity, etc. [69]. Figure 9.113 shows two examples of PET and PA 6 having foreign contamination. The apparatus can operate up to 200 bar/400 °C.

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