

An X-Ray Crystallinity Index Method with Application to Poly(Ethylene Terephthalate)

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I. INTRODUCTION

The physical and chemical behavior of a polymer is often dependent upon the proportion of chain segments that is in the ordered state; i.e., the polymer's crystallinity. Therefore, one of the most sought-after parameters in characterizing a polymer is some measure of the amount of this crystallinity. Various techniques have been used,¹ such as density, infrared absorption, and x-ray diffraction. These techniques have received extensive study recently²⁻⁶ and have been effectively compared for poly(ethylene terephthalate).^{7,8} The justification for the present study, which adds still another x-ray technique, is that it eliminates many of the difficulties with, and assumptions required of, an absolute measurement. It provides a relative comparison in a straightforward and simple manner which will suffice for the needs of many laboratories.

A few of the difficulties in obtaining an *absolute* crystallinity measurement can be listed. The use of densities requires a knowledge of the theoretical density for a single crystal of the polymer. This ideally requires a knowledge of the exact atomic configuration in crystals of the polymer, which in turn requires patterns of well-oriented samples but which are not always available. Also the density of the amorphous phase must remain constant. This latter point is not always recognized, and the amorphous density has sometimes been found to be variable.^{7,8} The use of the infrared technique requires that there be significant and independent differences in the spectra in the amorphous and crystalline phases which can be accurately measured. Such distinct differences are not always present. The use of the x-ray technique first requires a definition of crystallinity and then a separation of the amorphous halo from the crystalline diffraction; this latter condition is not common in polymer diffraction patterns. For these and other reasons, an absolute crystallinity is seldom obtained. If an absolute value is determined, it is frequently open to debate whether certain assumptions that were used are actually valid.

A *relative* crystallinity, however, does not have as many serious difficulties as an absolute measurement. All ratings can be compared to the

most crystalline sample currently obtainable. No absolute value need be known for this most crystalline extreme; all ratings are relative to it. Such an approach is used for the method described in this paper. It provides a crystallinity index number for a polymer sample having an unknown crystallinity. This index is a comparison of the unknown to the extreme amorphous and extreme crystalline standard samples which can be produced for that polymer. It is a measure of how far the unknown is between these extremes.

The method reported here has general application although it was previously developed and applied only for cellulosic materials.² It has been used in our laboratories for several experimental polymers. Studies of poly(ethylene terephthalate) (PET) fibers, films, and bulk polymer provide examples of its application.

II. EXPERIMENTAL

Important samples for crystallinity studies are apt to be oriented fibers. Since the different segments which give rise to the amorphous halo and crystalline diffraction probably do not have equal orientation, any radial photometer trace of an oriented pattern on x-ray film will give improper intensity data. Therefore, some provision must be made to remove the orientation in the sample. Similarly, a correct diffractometer trace, using counter techniques, requires removal of orientation in the sample. Two ways to accomplish this are:

(1) Dividing the oriented fiber into small enough segments to allow their random packing. Diffraction arcs are thus transformed into circles. This is the classical technique of Hermans et al. in their extensive crystallinity studies in cellulose and rayon preparations.⁹ Its main drawback is the time and difficulty involved in sample division into sufficiently small segments.

(2) Rotating the oriented fiber during the x-ray exposure to spread the arcs into circles. This technique has been frequently used in this laboratory; it has the advantage that the same sample preparation can be used to obtain several types of diffraction patterns. (As an alternative procedure, the film could be rotated instead of the sample. However, there are experimental difficulties in having the center of rotation of the x-ray film occur at the exact position of the main x-ray beam.) It is relatively easy to achieve a perfect rotation of the sample when it is part of the collimating system since the pinhole assembly can be expertly made to be concentric. A simple motor and gear device was designed to rotate slowly the pinhole and sample assembly. This is placed in a vacuum box camera to eliminate all air scattering during the exposure. The details of the above designs may be obtained from the author. Results for an unoriented film show (in Table II) that the rotation, *per se*, does not change the result. If, however, an oriented fiber has major diffractions near the meridian, their large Lorentz-polarization factor will give im-

TABLE I
Time Required for Various Steps in the Determination
of a Typical Crystallinity Index

Operation	Machine time	Operator time
Sample preparation (yarn windup on pinhole)	—	5 min.
X-ray exposure and processing	2 hr.	10 min.
Microphotometering x-ray pattern	25 min.	5 min.
Obtain data from photometer trace	—	15 min.
Prepare data tape for computer	10 min.	10 min.
Computation of index	12 min.	—
Handling data tapes	—	5 min.
Setting up computer (for first run only)	4 min.	4 min.
	2 hr., 51 min.	54 min.

proper intensities. PET fibers have their major diffraction spots close to the equator.

A bundle of fibers 0.020 in. thick is given an exposure to CuK_α (Ni filter) x-rays for usually two hours at 5.0 cm. sample-film distance. Collimation is by 0.025 in. diameter pinholes 3 in. apart. Fine-grained Eastman Kodak Type AA film is used, and photometer traces are obtained using the Leeds and Northrup Knorr-Albers microphotometer.

The development time of the x-ray film was found to be critical for accurate results. If 3-min. development is used, inconsistent background values are obtained. Agitation of the film at least once a minute during a 5-min. development at 68°F. is advisable.

The intensity data obtained from microphotometer traces are handled rapidly in our laboratory by a Bendix G-15 small-scale computer in the determination of the crystallinity index, as described in the next section. The use of a computer greatly facilitates calculations, but it is not vital; a desk calculator could be used.

Undrawn, melt-cast film of PET provides the amorphous standard. An annealing treatment of this film at 245°C. for 65 hr. in air in a small muffle furnace provides the extreme crystalline standard.

Table I shows the times required for the various steps in the determination of a crystallinity index in our laboratory. Actual operator time is short, and it would be further reduced if the data were taken directly from an x-ray pattern to the memory of the computer by digital read-out of the intensity data.

III. PRINCIPLES OF THE CRYSTALLINITY INDEX

A. Normalization of Intensity Data

Since it is difficult to obtain identical x-ray exposures of different fiber samples, some normalization of the intensity data is usually necessary.

This can be accomplished rapidly and accurately by the computer. The normalization requires one basic assumption: that the total blackening on the x-ray film will be the same for a given mass of polymer and a given exposure no matter whether the sample is crystalline or amorphous. This assumption is valid since it is based on the fact that the x-ray film registers only the result of interaction of x-rays with atoms; the state of aggregation of the atoms will determine the location and phase of the scattering but will not affect the total intensity. Thus, amorphous scattering is traded for crystalline diffraction when a polymer is crystallized. This assumption permits us to normalize x-ray diagrams for two samples having different masses or exposures by using the following straightforward steps. (1) A vacuum camera is always used to eliminate air scattering. (2) The relative amount of white radiation is not changed in different exposures. (3) The area under the curve of intensity vs. diffraction angle for one of the standards is obtained by conventional numerical integration. The data for the unknown are handled similarly to get the area under its curve. (4) The ratio of these areas is obtained and then used as a multiplier for each of the data for the unknown. These new, normalized data are then used to determine the crystallinity index. One of the greatest advantages in the use of a computer is that this normalization can be done so easily and accurately.

B. Treatment of Data

The method was suggested by Wakelin¹⁰ and was recently used for cotton fibers.² Since the latter paper gives much of the necessary detail of the development of the technique, the following will only outline the adaptation and modifications used in the present study. The method provides a numerical comparison of an unknown sample to the most amorphous and the most crystalline extremes that can be obtained for the polymer species. The unique way in which the data are handled provides this numerical comparison.

The intensity of the x-ray diffraction is measured at equal increments along a radial photometer trace of an unoriented pattern. These increments have their boundaries at the same 2θ values in the traces for all samples of a polymer species. The choice of increment size and 2θ reference positions are dependent on the needs of the individual experiment. The more increments used, of course, the more exact will be the determination. The positions should be chosen to coincide with as many peaks in the pattern as possible. These reference positions are used to obtain the intensity data for the amorphous and crystalline extremes and also for the unknown, as shown in Figure 1 for a few of the increments for PET. A practical compromise for this polymer was to use increments of $2\theta = 21'$ from $2\theta = 12.5^\circ$ out to values of $2\theta = 38^\circ 48'$.

At each of these incremental positions, i , along the crystalline standard trace, a difference is obtained for its intensity compared to the amorphous standard curve, $(I_c - I_a)_i$. This provides a collection of differences.

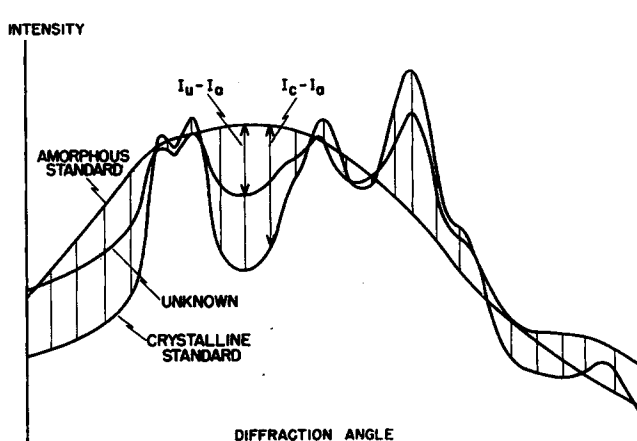


Fig. 1. Radial intensity distribution of poly(ethylene terephthalate) showing some of the incremental intensity differences used to calculate the crystallinity index.

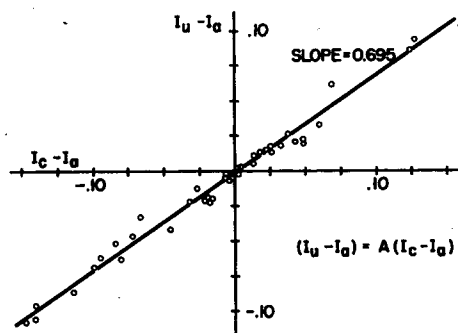


Fig. 2. Plot of incremental intensity differences used to determine a typical crystallinity index by the correlation method.

Similarly the differences are obtained for the unknown curve, $(I_u - I_a)_i$, to provide another collection.

These data can be handled in two ways:

1. The *absolute* values of the differences $(I_u - I_a)_i$ can be used in an integration to get the area between the amorphous curve and the unknown curve. Similarly, the differences $(I_c - I_a)_i$ can be used to get the area between the amorphous curve and the crystalline standard curve. A ratio of these areas is then a relative measure of the crystallinity of the unknown sample. This is similar in principle to the standard method so often used for absolute crystallinity measurement, but it does not require a decision about the proper background nor does it require a resolution of the curve into crystalline peaks and an amorphous halo. The method is very useful if there is a shifting of a peak's 2θ position, as is sometimes found. However, the index obtained in this way is quite sensitive to errors in experimental technique; a much lower precision was obtained for PET

samples for the area or integral index than for the slope or correlation index as determined in the following paragraphs. It was also shown for cotton fibers² that the integral index may have a different value from the correlation index.

2. The *numerical* value of the differences $(I_c - I_a)_i$ and $(I_u - I_a)_i$ can be plotted against each other for each increment, i . This results in the straight-line relation and is the essence of this new method of obtaining a crystallinity value. The treatment of the data can be shown as an X-Y plot in Figure 2. The X position of each dot represents the value of $(I_c - I_a)$ at one incremental position along the curve; the Y position of each dot is the corresponding $(I_u - I_a)$ value. The dots will be in both the first and third quadrants because the crystalline curve goes both above and below the amorphous standard curve; this creates both positive and negative differences. Now, if a regression line is drawn through these difference points, its slope will be the direct measure of where the unknown is between the crystalline and amorphous extremes, as follows. The equation for this straight line is

$$(I_u - I_a)_i = A(I_c - I_a)_i + B$$

The intercept, B , should be zero if the normalization, discussed in the previous section, is properly performed. The computer program type-out includes the value of B ; if it is not a very small number, an error has been made. The slope, A , will be given by

$$A = \frac{(I_u - I_a)_i}{(I_c - I_a)_i}$$

Thus, as the values of I_u approach I_c , the value of A will approach 1. On the other hand, if the values of I_u approach I_a , A becomes 0.

The slope can be converted to a "percentage relative crystallinity" or to some arbitrary "crystalline index." It is preferable not to use the term "per cent crystallinity" as it is too easily confused with the absolute crystallinities that are sometimes obtained. Since it is a relative number only, the word "index" is used. An index limit of 100 was chosen for the crystalline standard, and 0 was chosen as the opposite standard for the amorphous material. The decision of which patterns are likely standards can usually be made visually, and then successive testing of them as "unknowns" will determine the combination which gives the greatest range of values.

It is conceivable that an unknown will in the future be more crystalline than a standard now being used. The method easily accommodates this situation as it produces a slope greater than 1 and, therefore, an index greater than the defined limit of 100. This new maximum in crystallinity could then be used as the new standard, although this change in standard could be confusing in a continuing research program. In the latter case, the index could be unlimited and be allowed to assume a required value

above 100. An analogy is found in gasoline octane numbers where numbers greater than 100 are used.

C. Precision and Accuracy

Values obtained for repetitive measurements of a PET fiber are shown in Table II. The precision is satisfactory for the slope index since the variability is within the probable error which is always computed for this index as a guide to the reliability and scatter of the data. Table III

TABLE II
Repetitive Measurements of Crystallinity Index

Sample description	Slope index	Probable error
PET Fiber		
Exposure 1 of sample windup no. 1	41.3	1.6
Exposure 2 of above	41.4	1.6
Exposure 3 of above	41.6	2.0
Exposure 4 of above	40.7	1.4
Exposure 5 of above	40.8	1.4
Exposure 6 of above	42.2	1.5
Exposure 7 of above	41.4	1.3
Exposure 1 of new sample windup no. 2	40.7	1.7
Exposure 1 of new sample windup no. 3	40.0	1.7
PET Film		
Unoriented, annealed; sample not rotated	100.0	(Standard)
Annealed; sample rotated	100.6	0.6

TABLE III
Crystallinity Index Accuracy

Sample Composition*		Slope index	Probable error
$\frac{2}{3}$ crystalline, $\frac{1}{3}$ amorphous	Sample no. 1	63.4	1.1
	Sample no. 1	63.7	1.0
	Sample no. 2	59.7	0.5
	Sample no. 2	58.4	0.8
	Sample no. 3	62.5	0.8
$\frac{1}{2}$ crystalline, $\frac{2}{2}$ amorphous	Sample no. 1	30.3	0.6
	Sample no. 2	25.0	0.9
	Sample no. 2	24.8	0.8
	Sample no. 3	32.2	0.9
	Sample no. 3	32.3	0.7
$\frac{1}{4}$ crystalline, $\frac{3}{4}$ amorphous	Sample no. 4	29.9	0.8
	Sample no. 5	34.6	1.9
		23.3	0.7

* A composite was used of several layers of films of the amorphous and crystalline standard. Although micrometered, the *exact* thickness in the x-ray beam cannot be determined due to the uneven thickness of the crystalline standard.

shows the values obtained on "standard samples" which were prepared by sandwiching together known thicknesses of the extremes in varying ratios. Since the index is always a relative number, precision is more important than accuracy. In the study of cotton fibers,² better preparation of "standard" samples showed that excellent accuracy can be achieved.

IV. STUDY OF POLY(ETHYLENE TEREPHTHALATE)

A. Maximum Crystallization

Samples of PET in a variety of forms were given an extreme thermal treatment of prolonged heating at 245°C. for 65 hr. This high temperature should mean that the polymer chains have nearly reached their ultimate thermal motion short of melting; this long time should be sufficient for them nearly to reach an equilibrium state. Thus, "ultimate crystallization" should be achieved. The results given in Table IV

TABLE IV
Maximum Crystallization—Poly(ethylene terephthalate)

Sample	Index after annealing at 245°C. for 65 hr.
Film—amorphous, unoriented	100
Fiber—amorphous, unoriented	89
Film—amorphous, oriented	69
Fiber—crystalline, oriented	69

show that an unoriented amorphous film will go to a higher level of crystallinity than will a fiber in any of its possible starting states. Furthermore, an unoriented fiber reaches a higher crystallinity than an oriented one. These results lead to two interesting interpretations:

1. The spinning process, even without windup orientation, produces some type of strained or nucleated aggregation which is not free to crystallize to the level that is possible if the polymer is simply cooled down from a melt without being spun into a fiber.

2. The orienting process imposes some type of constraint on the freedom of movement of the chains which prevents the highest amount of crystallization. This is contrary to the established viewpoint that an orienting process should aid in the rate of crystallization, a viewpoint which probably resulted from the work on the crystallization of natural rubber.

B. Crystallizing Treatments

A variety of treatments was given to fiber and film samples, as listed in Table V. The results are in agreement with the known¹² slow rate of crystallization of PET. It appears that many seconds of time are needed to achieve equilibrium at any elevated temperature; for instance, the index after 15 sec. at 240°C. does not equal the index after 30 min. at

220°C. In another case, 10 sec. at 245°C. is not equivalent to 65 hr. at 183°C. Thus, time and temperature may be traded in crystallizing treatments for this polymer. For instance, a long 65-hr. treatment at 183°C. will achieve a higher crystallinity than a 30-min. heating at 220°C.

Another result seen in Table V is the well-known plasticizing effect of water; the boil-off treatment is equivalent to 150°C. dry in crystallizing effect. This same plasticizing effect was seen in another characterization of these fibers¹³ in which their long period x-ray diffractions were found to be equal after these treatments.

TABLE V
Crystallization Treatments—Poly(ethylene terephthalate)

Sample and treatment	Index after treatment
Amorphous, Unoriented Film	
Control	0
Heated at 183°C. for 10 sec. in air	15
Heated at 183°C. for 65 hr. in air	64
Heated at 245°C. for 10 sec. in air	43
Heated at 245°C. for 65 hr. in air	100
Drawn, Slightly Crystallized Fiber	
Control	20
Boiled in H ₂ O for 30 min.	25
Heated taut in air at 150°C. for 30 min.	26
Heated taut in air at 200°C. for 30 min.	39
Heated taut in air at 220°C. for 30 min.	57
Heated slack in oil at 220°C. for 15 sec.	46
Heated taut in oil at 220°C. for 15 sec.	49
Heated slack in oil at 240°C. for 15 sec.	51

C. Comparison of Crystallinity Index with Density

Density has been used as a measure of crystallinity of PET¹⁴⁻¹⁶ and so was obtained for most of the samples in order to compare with the results of the crystallinity index technique. The densities were obtained by a standard gradient tube method¹⁶ and are shown in Figure 3. It appears that density is *not* a reliable measure of *x-ray* crystallinity of PET; this has been recently shown also by Farrow and Ward.^{7,8} Figure 3 shows that samples of the same density can have a great variation in index, or vice versa. Thus, other factors must be important in the density determination. It has been suggested^{7,8,14,17-19} that the amorphous regions can exist with different densities. The data in Figure 3 appear to be ample proof of this concept. That is, the amorphous regions have a variation in packing and entanglements so that different amounts of openness and randomness exist. A preordering may occur in which the polymer segments are densely packed and are nearly crystalline in the density test but do not

have perfect enough packing to cause diffraction of x-rays. Thus, a measurement of crystallinity by a density determination could be in serious error in terms of x-ray crystallinity. On the other hand, density may be more useful than x-rays to characterize a sample in relation to some property which would depend on the amount of close packing and preorder.

The data in Figure 3 agree with the above concept of the preordered state since most of the points fall above a line drawn from index zero to index 100. Since the index is based on these two extreme samples, all results should fall on the line if there is a linear relationship between density and x-ray crystallinity index. Thus, most samples are more

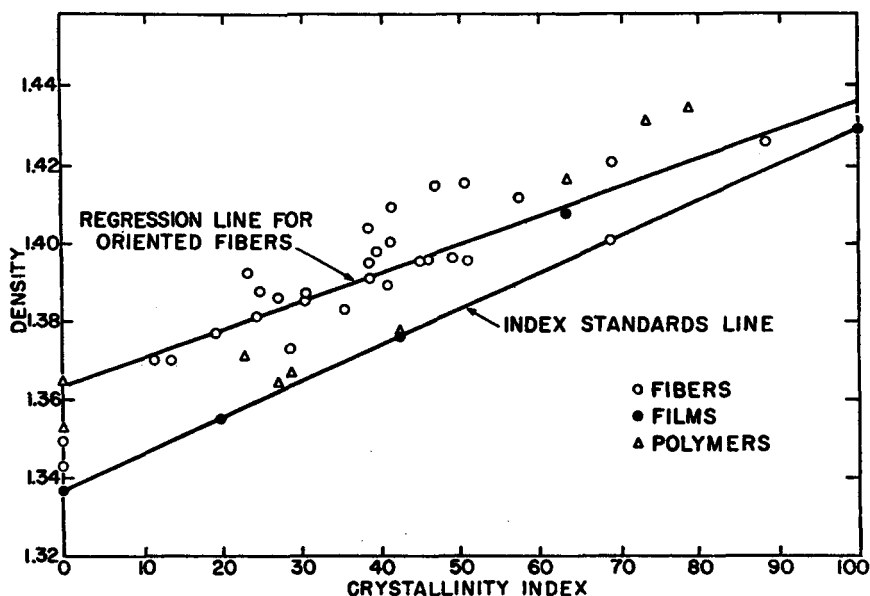


Fig. 3. Density-crystallinity index relationship for poly(ethylene terephthalate).

dense than the x-ray determination would indicate, suggesting that preordering exists. It appears that either the spinning or the orienting operation is the factor causing the preordering, since the samples which occur on the line are films or bulk polymer; drawn fibers occur above the line. A least squares regression line is drawn through all the data points in Figure 3 for oriented fibers. The line has a zero intercept at density = 1.363 g./cm.³, a value close to the value, 1.355 g./cm.³, obtained recently from other considerations by Farrow and Ward⁷ for the density of the amorphous regions in drawn materials.

The data in Figure 3 can also be interpreted in a different way. Since the x-ray crystallinities are lower than the density values would predict, it can be interpreted that the spinning or drawing disrupts the perfect lattice enough to reduce the x-ray diffraction, but not enough to lower the

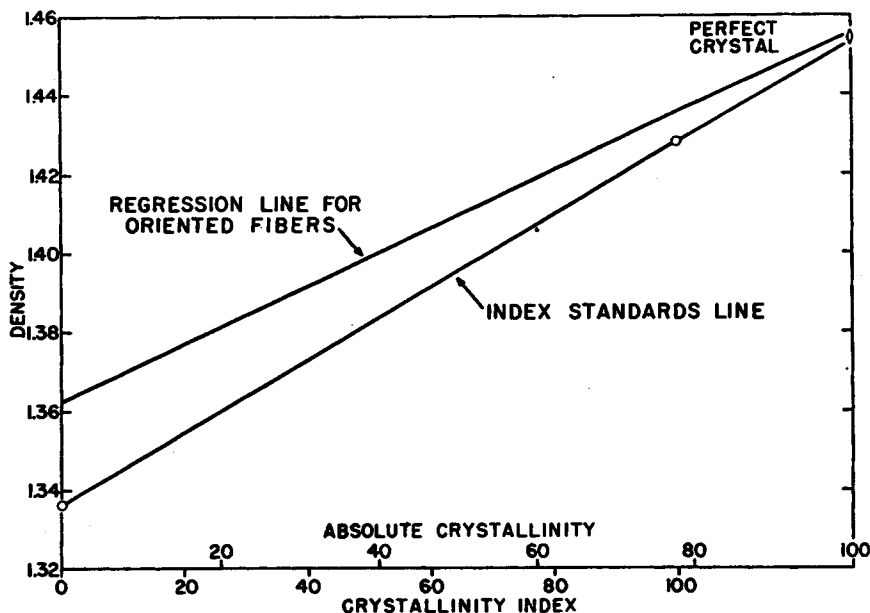


Fig. 4. Density-crystallinity comparison of poly(ethylene terephthalate) in terms of absolute and relative indices.

density. This would be a good argument for the paracrystalline state of Hosemann²⁰ and would be in line with the proposal of Kargin.¹⁷

D. Absolute Crystallinity Scale

Although the crystallinity index is meant to be only a relative determination, there is a natural question of its relation to absolute standards. Three data points from early infrared measurements¹⁴ are close to the index line in Figure 3 and suggest that the crystallinity index is similar to what the infrared technique measures. However, recent infrared measurements⁷ appear to show conclusively that no such relation can be sought.

The density of the perfect crystal²¹ can be used as a primary standard, as shown in Figure 4 in which the 100 index corresponds to 78% absolute crystallinity. It is interesting to note in Figure 4 that the straight line connecting the relative standards extrapolates on to the perfect crystal value. Also, the regression line for drawn fibers taken from Figure 3 extrapolates on to the same value at 100% absolute crystallinity. This suggests that drawn fibers, *on the average*, could give an absolute crystallinity value when their relative index is determined. Unfortunately, this surprising and interesting conclusion is not at all useful in detailed work since there is so much deviation from the regression line for individual results.

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Synopsis

Wakelin's method of correlation analysis of x-ray diffraction patterns was adapted for fibers, films, and polymer samples of poly(ethylene terephthalate). This method gives a number called a crystallinity index, which is a measure of how far an unknown sample is between the extremes of the most amorphous and most crystalline samples obtainable for a polymer species. By being relative, this index eliminates the controversial assumptions of absolute crystallinity determinations. It is especially suited for routine studies since the calculations are readily adapted to the use of a computer and since the total time needed to obtain an index is short. A study of the indices of a variety of poly(ethylene terephthalate) materials produced the following conclusions:

(1) Maximum crystallization is achieved only by starting with an unoriented amorphous film. This is contrary to the view that orientation helps crystallization. Also, this shows that the act of spinning a fiber sets up an apparently strained state which will not crystallize to the same high level as a cast film. (2) Time of heating can be exchanged for temperature of heating, since the rate of crystallization is slow for this polymer. Thus, a longer time at a lower temperature is the equivalent of a shorter time at a higher temperature in producing equivalent crystallinities. (3) The crystallinity measured by density is not the same as the crystallinity measured by x-rays. (4) A preordered state must exist in fibers in which segments of molecules have lost their complete randomness and are densely packed, yet the segments are not perfectly enough placed to produce x-ray diffraction. Or, it can be concluded that spinning or drawing disrupts the fiber's perfect lattice so that less x-ray diffraction is produced than the high density would indicate.

Résumé

On a adapté pour des fibres, des pellicules et des échantillons de polymère de polytéréphtalate d'éthylène la méthode de Wakelin concernant l'analyse corrélationnelle des diagrammes de diffraction des rayons-X. Cette méthode fournit un nombre appelé indice de cristallinité qui mesure à quel degré de cristallinité se trouve un échantillon inconnu par rapport aux extrêmes les plus amorphes et les plus cristallins qu'on peut obtenir pour une sorte de polymère. Par son caractère relatif, cet indice élimine le concept controversé de la détermination de la cristallinité absolue. Il est spécialement adapté aux études routinières puisque les calculs sont facilement adaptés et obtenus rapidement en utilisant une machine à calculer. Une étude des indices de cristallinité d'une série de polytéréphtalate d'éthylène-glycol amène aux conclusions suivantes: (1) On parvient à une cristallisation maximale uniquement au départ d'un film amorphe non orienté. Ceci est contraire à l'opinion d'après laquelle l'orientation aide la cristallisation. Ceci montre également que le fait de filer une fibre provoque un état de tension apparent qui ne cristallisera pas au même degré qu'un film coulé. (2) Le temps de chauffage et la température de chauffage ont un effet interchangeable puisque la vitesse de cristallisation est lente pour ce polymère. Donc, un long temps de réaction à basse température et un court temps de réaction à plus haute température s'équivalent pour produire des cristallinités identiques. (3) La cristallinité mesurée par densité est différente de celle qu'on mesure par rayons-X. (4) Un état préordonné doit exister dans les fibres où des segments de molécule ont perdu leur arrangement purement statistique et sont ordonnés d'une façon dense. Cependant, les segments n'ont pas une ordonnance assez parfaite pour causer une diffraction des rayons-X. Ou bien on peut conclure que le filage ou l'étrépage rompt le réseau parfait de la fibre de telle sorte qu'il se produit une diffraction de rayons-X moindre que ne le ferait prévoir la densité élevée.

Zusammenfassung

Die Methode der Korrelationsanalyse von Röntgenbeugungsdiagrammen von Wakelin wurde für Fasern, Filme und Polymerproben aus Polyäthylenterephthalat angepasst. Diese Methode liefert eine, Kristallinitätsindex genannte, Zahl, die ein Mass für die Lage der Kristallinität einer unbekanntenen Probe zwischen den beiden für eine Polymerart erhältlichen Extremen, nämlich höchstgradig amorphe und höchstgradig kristalline Proben bildet. Dieser Index ist zwar relativ, beseitigt aber die widersprechenden Annahmen bei absoluten Kristallinitätsbestimmungen. Er ist besonders für Routineuntersuchungen geeignet, da die Berechnungen rasch und leicht mit Hilfe eines Computers durchgeführt werden können. Eine Untersuchung der Indices einer Reihe von Polyäthylenterephthalatproben lieferte folgende Ergebnisse: (1) Maximale Kristallisation wird nur bei Verwendung eines nicht-orientierten amorphen Films als Ausgangsmaterial erhalten. Das steht im Gegensatz zu der Ansicht, dass Orientierung die Kristallisation erleichtert. Es zeigt auch, dass der Spinnvorgang einer Faser einen offenbaren Spannungszustand erzeugt, der nicht einer gleich hochgradigen Kristallisation fähig ist wie ein gegossener Film. (2) Erhitzungsdauer kann durch Erhitzungstemperatur ersetzt werden, da die Kristallisationsgeschwindigkeit bei diesem Polymeren gering ist. Eine längere Dauer bei niedrigerer Temperatur ist zur Erzeugung eines gleichen Kristallinitätsgrades einer kürzeren Dauer bei höherer Temperatur äquivalent. (3) Die aus der Dichte bestimmte Kristallinität stimmt nicht mit der Röntgenstrahl-Kristallinität überein. (4) In Fasern muss ein vorgeordneter Zustand vorhanden sein, bei welchem die Molekülsegmente ihre völlige Regellosigkeit verloren haben und dicht gepackt sind, die Lage der Segmente aber noch nicht vollkommen genug ausgebildet ist um Röntgenbeugung zu erzeugen. Oder es ergibt sich der Schluss, dass Spinnen oder Ziehen das vollkommene Gitter der Faser zerstört, so dass eine geringere Röntgenstreuung entsteht als der hohen Dichte entsprechen würde.

Received January 17, 1962