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Vickers indentation anisometry on thin cylindrical materials

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The required anisometric correction to be made when using Vickers indentations on filaments with radial dimensions of the order of those of the microindenter, is evaluated. For an isotropic plastic material the anisometry of indentation, $\Delta l = l_{\parallel} - l_{\perp}$ increases with decreasing cross-section of the filament. If the calculated correction, Δl^c, is in agreement with experiment the interest lies in the fact that the material preserves the indentation shape after removal of the indenter. Values of $\Delta l \neq \Delta l^c$ reveal the presence of a mechanical anisotropy on metallic filaments due to processing. Furthermore, in the case of thin highly oriented polymeric fibres the inherent microindentation anisotropy is masked by the anisometry effect. Consequently, the proposed correction is very important and particularly valuable when anisotropic cylindrical material surfaces are being studied.

1. Introduction

Recent investigations [1–3] have convincingly shown that the microhardness technique can be a convenient method which yields valuable information on the surface anisotropy developed within oriented polymers. In these materials the stresses are larger along the fibre axis because the material is stiffest and strongest in this direction. The anisotropy of the plastic impression instantly arising on unloading, is hence due to an elastic recovery of the highly oriented material in the fibre direction. As a result the impression shows an asymmetrical diamond shape with the indentation diagonal parallel to the fibre axis, $l_{\parallel}$, smaller than the diagonal perpendicular to it, $l_{\perp}$. In the case of polyethylene ultra-oriented fibres a correlation between indentation anisotropy and draw ratio has been found [3]. Here, $l_{\parallel}$ represents the main contribution to the anisotropy of the material and is somehow related to the elastic modulus which, in turn, depends on the number of interlamellar and interfibrillar tie molecules. In the course of a recent study [4] on very thin PE oriented (50 to 100 μm) filaments with a shish-kebab structure [5] obtained from a stirred solution, $l_{\parallel}$ showed unexpectedly larger values than $l_{\perp}$ thus masking the inherent indentation anisotropy of the oriented fibres. The object of the present contribution is to derive the required corrections which allow a distinction to be made between the anisometry due to the cylindrical shape of the thin filaments and the inherent mechanical anisotropy which is mainly a consequence of the parallelism of polymer chains along the fibre axis.

2. Experimental procedure

The indentations were carried out at room temperature with a microhardness tester using a Vickers square pyramidal diamond. The diagonals of the indentation were measured with a micrometer eyepiece of the microscope using magnifications of $\times 400$ and $\times 100$, respectively. The square pyramidal indenter of 100 μm height was aligned with the XY reference system of the reflection microscope in order to obtain one of the indentation diagonals parallel to the cylindrical axis. Loads of 25, 50, 100, 200, 300 and 385 g were applied for a loading cycle of 30 sec. Further details have been published elsewhere [3].

Table I lists the different materials investigated in this study. These materials were carefully selected for (1) their expected isotropic character, and (2) their perfect cylindrical shape. Each particular indentation was repeated at least five times. The estimated accuracy in the determination of indentation lengths was better than 2%.

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TABLE I Radius of various materials investigated with cylindrical shape

Sample	No.	Radius, r (μm)
Commercial copper wire	1	100
	2*	100
	3*	590
Tin (50% pure)	1	400
	2	960
	3*	960
Polyethylene Lupolen 6011 L (annealed at 125° C for 17 h)	1†	1850
Polyethylene Epolene N-12	2†	2000

*Heated in the flame of a Bunsen burner and then cleaned in a dilute aqueous solution of chlorhydric acid.
†Molten at 165° C and crystallized for 15 min at 40° C.

3. Anisometry correction

Fig. 1 schematically depicts the indentation geometry for a Vickers indenter penetrating a cylindrical surface with a radius r. In the case of an ideal plastic deformation (i.e. on absence of elastic stresses) after load removal, the square pyramidal originates a rhombic indentation with one of its diagonals parallel to the filament axis. Let $2BC$ be the measured indentation length, $l_\perp$, normal to the filament axis and $2DE$ the indentation length, $l_\perp^c$ which would arise on a flat surface for the same penetration depth. For an isotropic material, $l_\perp^c = l_\parallel$. However, as a result of the existing curvature, $l_\parallel > l_\perp$ (anisometric indentation). From Fig. 1, $\overline{DE} = \overline{BC} + \overline{BD} \tan \alpha/2$ and since $\tan \alpha/2 \simeq 7/2$ one has $l_\perp^c = l_\perp + 7\overline{BD}$. Now, by substituting $\overline{BD} = r - \overline{BO} = r - (r^2 - \overline{BC}^2)^{\frac{1}{2}}$ in the latter equation one obtains:

$$l_\perp^c = l_\perp + 7\left\{r - \left[r^2 - \left(\frac{l_\perp}{2}\right)^2\right]^{\frac{1}{2}}\right\}, \qquad (1)$$

an expression which relates the observed diagonal $l_\perp$ to the ideal diagonal length $l_\perp^c$. The geometrical

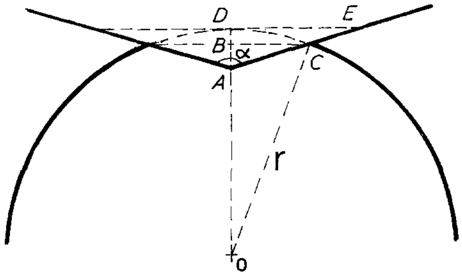

Figure 1 Schematic illustration of a Vickers indenter, edge on, penetrating the surface of a cylindrical material of radius, r.

anisometry, $\Delta l^c = l_\perp^c - l_\perp$, calculated for a given value of r can now be compared with the measured indentation anisometry, $\Delta l = l_\parallel - l_\perp$. For an isotropic material, evidently $\Delta l^c = \Delta l$. Fig. 2 gives a plot of $l_\perp^c$ against $l_\perp$ according to Equation 1 for different values of r. The bisecting straight line, $l_\perp^c = l_\perp$ corresponds to an isotropic fully flat surface ($r = \infty$) with no identation anisometry. This plot immediately indicates that for a given value of r, Δl^c increases with $l_\perp$. However, if $l_\perp$ is maintained constant, Δl^c decreases markedly with increasing r.

4. Results and discussion

Fig. 2 shows the experimental indentation lengths obtained from the three copper filaments investigated. It is noteworthy that the data for the heat-treated filaments Cu(2) and Cu(3) (Table I) are lying on the curves corresponding to the predictions of Equation 1. On the other hand, the data for the untreated filament Cu(1), lie off the $r = 100\,\mu$m expected curve, the observed anisometry Δl being lower than Δl^c. Fig. 3 illustrates the typical bulging of the sides of the rhombic impression giving a "convexity" for Cu(1) (untreated). This convexity has been ascribed to an elastic effect on unloading [6]. After heat treatment and subsequent cleaning (Table I) of the wire, the sides of the impression do not bulge anymore. The relative anisometry increases from 24% up to 43% after heat treatment. The discrepancy found between calculated and experimental values in Fig. 2 for the untreated metallic filament, hence, suggests the occurrence of a

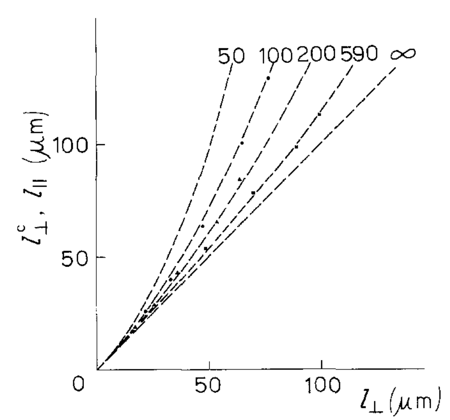

Figure 2 Corrected value of indentation length, $l_\perp^c$ (according to Equation 1) as a function of the observed length $l_\perp$, for different r values. Experimental values ($l_\perp$, $l_\parallel$) for the three copper filaments are also given: Cu(1) (▲) and Cu(2) (•) with $r = 100\,\mu$m and Cu(3) (▪) with $r = 590\,\mu$m.

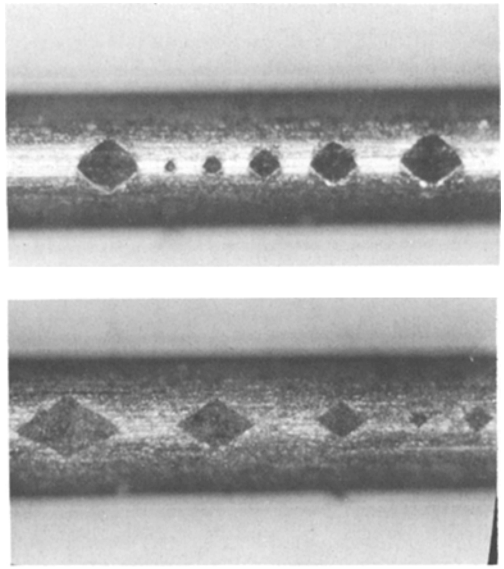

Figure 3 Microindents for different loads (25, 50, 100, 200 and 300 g) on a copper filament with $r = 100\ \mu m$. Top: untreated Cu(1). Bottom: after treatment, Cu(2). See Table I.

release of elastic stresses when unloading which are removed by heat treatment. Corrected indentation lengths for elastic recovery [7] are $\sim 5\%$ smaller thus allowing the data for Cu(1) to fit into the predicted curve for $r = 100\ \mu m$. Other experiments carried out on copper filaments after annealing, without chemical treatment, under vacuum at 860° C also show anisometry values which deviate from the geometrical expectations of Equation 1. The same trend is obtained for soldering tin filaments. While Sn(3) closely satisfies $\Delta l \simeq \Delta l^c$ for Sn(1) and Sn(2) $\Delta l < \Delta l^c$. In all polymer filaments it was found $\Delta l \simeq \Delta l^c$. In addition, the microhardness values are similar to those determined on isotropic flat sheets of the polymer crystallized under similar conditions. This result emphasizes the isotropic character of the melt-crystallized PE filaments examined in this study.

A quantitative check concerning the isotropic character of samples with cylindrical shape is straightforwardly given by plotting the ratio $\Delta l/\Delta l^c$ as a function of $l_\perp$ (Fig. 4). Large deviations of this ratio from unity immediately evidence the presence of some kind of mechanical anisotropy. In accordance with the foregoing, the data corresponding to the isotropic filaments – PE samples, Cu(2), Cu(3) and Sn(3) – fluctuate around the $\Delta l/\Delta l^c = 1$ horizontal line. In contrast, the data for untreated metallic filaments largely deviate from unity.

We wish finally to show that the anisometric correction is also of great value when determining the anisotropy of mechanical oriented polymers. Indeed, to evaluate properly the indentation anisotropy, ΔMH, in cylindrical polymer fibres the corrected $l_\perp^c$ value must be used. We define the "microindentation anisotropy"* in a highly oriented system as:

$$\Delta MH = 1 - (l_\parallel / l_\perp^c)^2 . \qquad (2)$$

The critical influence, if using $l_\perp^c$ instead of $l_\perp$ in Equation 2, now becomes evident because $l_\perp^c > l_\perp$ and ΔMH (uncorrected) is a function of $1/l_\perp^2$ thus leading to a depression of anisotropy. Fig. 5 illustrates the conspicuous enhancement of the ΔMH values on the surface of ultra-oriented PE strands with $r \simeq 680\ \mu m$ (the data are taken from [3]), as a function of draw ratio, when Equation 2, instead of the uncorrected ΔMH value, is used. These values entail an elastic recovery of the highly oriented material perpendicular to the fibre axis. This recovery was not previously

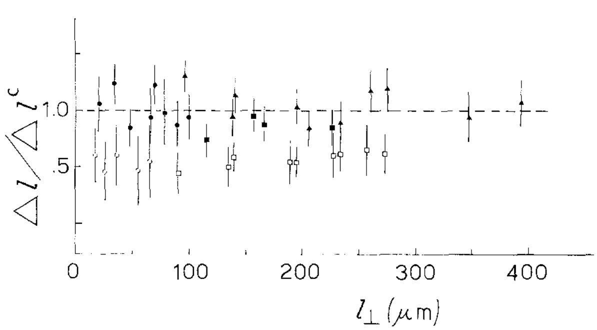

Figure 4 Ratio of observed to calculated indentation anisometry against $l_\perp$ for the filaments investigated: copper (○, ●), 50% pure tin (□, ■) and polyethylene (▲). Open symbols correspond to untreated metallic filaments.

*Since the term "microhardness anisotropy" used in previous publications [1–3] and defined by Equation 2 seems rather ambiguous – $l_\parallel$ involving an elastic recovery of the material along the fibre axis – we propose to call ΔMH from now on "microindentation anisotropy".

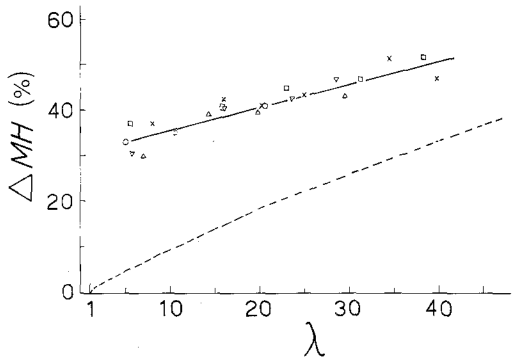

Figure 5 Microindentation anisotropy $\Delta MH = 1 - (l_{\|}/l_{\perp}^{c})^2$ on the surface of ultra-oriented PE as a function of draw ratio. Bottom: uncorrected ΔMH [3]. Top: values derived using the anisometric correction. The data are taken from [3].

recognized [3] due to the lack of an adequate anisometric correction.

5. Conclusions

The study of hardness indentations formed in cylindrical surfaces of thin filaments using a square-based diamond indenter (Vickers), shows that the impression is not symmetrical but gives longer diagonals parallel to the cylindrical axis, $l_{\|}$, than at right angles, $l_{\perp}$. A simple analysis relates these two diagonals (Equation 1) as a function of the cylinder radius for a perfectly isotropic material. Measurements made on non-isotropic filaments show a deviation from the theoretical curves giving a measure of the anisotropic properties. This technique is of particular interest as a convenient means of assessing the anisotropy of polymer filaments.

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