STRUCTURE AND PROPERTIES

Influence of Rolling Speed on the Mechanism of Adiabatic Shear Band Formation and Plastic Flow in Polyethylene Terephthalate Films

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Abstract—Amorphous polyethylene terephthalate was oriented by rolling at room temperature on laboratory rollers. During rolling, a system of shear bands appeared, which was observed using crossed polarizers. The effect of rolling speed and degree on the shear band system was studied. At low rolling speeds (6.5 mm/min), plastic flow occurs through numerous fine shear bands. At higher speeds (above 1300 mm/min), a single serrated deformation zone is observed. This is explained by thermal softening of the polymer and a decrease in yield strength, which suppresses the formation of competing shear bands. At intermediate rolling speeds, two, three, four, or five serrated zones appear, spaced approximately equally. Increasing the degree of rolling leads to an increase in the number of serrated shear zones and homogenization of the flow process. The temperature in the shear bands was estimated, and it was shown that very slight heating of the bands is sufficient to prevent the formation of multiple fine shear bands. A significant difference was noted between the adiabatic deformation of the sample and the adiabatic shear bands.

DOI: 10.1134/S0965545X24600832

INTRODUCTION

One of the methods for polymer orientation is cold rolling [1–18], in which a polymer film is continuously stretched between two rollers rotating in opposite directions at room temperature. Rolling leads to increased fracture viscosity of polyethylene [19] and suppresses brittle fracture in polystyrene [17, 18], polyethylene terephthalate, and other polymers. Double rolling reduces the brittle temperature of polypropylene, which retains its plasticity at low temperatures down to -30° C [20].

During rolling, two opposing effects are observed: orientation hardening [21] and deformation softening of the polymer, which involves a reduction in yield stress after plastic deformation. Softening occurs at low degrees of rolling, when it does not exceed 1.5. This is related to the formation of shear bands and an increase in free volume within them, with the volume increasing both during stretching and compression of the polymer [22]. There is also a second mechanism of deformation softening, associated with heat generation in the shear bands [23]. During plastic deformation, work is done that almost entirely converts into heat [24, 25]. The fraction of work converted into heat depends on the polymer [25], but it typically amounts to 80-90%. During rapid stretching, the temperature of the polymer can increase by 60–100°C [26]. The heating is especially significant in PET and polyarylate.

In the rolling of PET, a serrated flow zone appeared, consisting of interconnected shear bands with an anomalously large thickness, reaching 100 μ m [27]. The aim of the present work is to study the effect of rolling speed on the formation of shear bands in amorphous PET. Specifically, the conditions for the formation of serrated deformation zones were investigated.

OBJECTS AND METHODS OF RESEARCH

This study investigated industrial PET films with a thickness of 910 μ m. The molecular weight of PET was 20 × 10³, and its degree of crystallinity was 7–10% [21]. Data on the molecular weight were provided by the manufacturer.

Rectangular strips measuring 100×20 mm were rolled at room temperature on laboratory rollers between two rollers rotating in opposite directions. The degree of rolling Λ was defined as the ratio of the length of the rolled sample (*L*) to its initial length (*L*₀): $\Lambda = L/L_0$. The degree of rolling was varied from 1.06 to 1.4.

From the rolled PET film, longitudinal cross-sections with a thickness of approximately $10 \ \mu m$ were



Fig. 1. Flow zones in a polarized transmission microscope on thin side sections of PET films at rolling speeds v equal to (a) 6.5, (b) 26, (c) 260, (d) 310, (e) 416, and (f) 1300 mm/min. Rolling degree $\Lambda = 1.06$.

obtained using a microtome and examined using a VOMO MBS-9 light polarization microscope and a Jeol JSM-5300LV scanning electron microscope.

RESULTS AND DISCUSSION

Figure 1 demonstrates the effect of rolling speed on the appearance of shear bands in PET films. It shows optical photographs of thin lateral cross-sections of the films in polarized light. A rolling degree $\Lambda = 1.06$ corresponds to the initial stage of rolling. After slow rolling at a speed of v = 6.5 mm/min (Fig. 1a), numerous micro-shear bands are observed. These microbands are slightly bent and not strictly perpendicular to each other, as theorized in plasticity theory. This is because the bands appear at different times, and the gradual bending of the film leads to the distortion of the bands. Short surface micro-shear bands are also observed.

When the rolling speed is increased to 26 mm/min (Fig. 1b), the number of bands decreases; they extend from one surface to the other, and their width increases. At speeds of 260 mm/min (Fig. 1c) and 310 mm/min (Fig. 1d), the deformation zones consist

of four and three, respectively, sawtooth-like structures running from one surface to the other. With a further increase in rolling speed to 416 mm/min (Fig. 1e), only two sawtooth-like flow zones are observed on the side cross-section. It is noted that the sawtooth zones in Figs. 1c-1e are spaced approximately equally apart. Finally, at the highest rolling speed of v = 1300 mm/min, a single sawtooth-like zone is visible on the side surface (Fig. 1f). This zone consists of shear bands oriented at an angle $\theta = 50^{\circ} \pm$ 1° to the sample axis. The characteristic of the sawtooth zone is its unusually large shear band width. In Fig. 1f, the width is 45 μ m, but in some cases, it reaches 100 um. Such wide shear zones were first discovered in work [27]. To describe the structural features, with sizes at the limit of the naked eye ($100 \,\mu m$), the concept of mesostructure is introduced, and in terms of their width, the shear bands present in Figs. 1e and 1f are now considered not as micro-shear, but as meso-shear bands.

Thus, as the rolling speed increases, there is a transition from flow along numerous micro-shear bands (Fig. 1a) to flow along several relatively thick sawtooth-like zones (Figs. 1c-1e), and finally to the for-



Fig. 2. (a) SEM image of the top and side surfaces of a PET film with a thickness of 0.9 mm rolled at a speed of 1300 mm/min, rolling degree $\Lambda = 1.06$; (b) schematic representation of a rolled film: (1) shear bands on the side surface of the film, (2) and (3) undeformed areas separated by a shear band, (4) intersection of two perpendicular shear bands on the upper surface of the film.

mation of a single zone (Fig. 1f). The distance between the sawtooth-like zones in Figs. 1c-1e is almost the same. Therefore, increasing the rolling speed leads to a transition from disorder to order in the arrangement of shear zones. Let's examine the zigzag deformation zones in more detail.

Figure 2 shows the images of the side (bottom half of the photo) and top surfaces of PET film rolled at a speed of 1300 mm/min, taken with a scanning electron microscope. The sample was positioned at an angle to the electron beam, so the upper part of the image depicts the film surface, while the lower part shows the side cross-section along the rolling direction, as schematically illustrated in Fig. 2b. On the film surface, the shear bands form a set of parallel stripes extending across the entire width of the film, while on the side, they form a zigzag pattern. The thickness of the mesoshear bands is $40-55 \ \mu m$ on the side surface and $65-75 \,\mu\text{m}$ on the top surface. The sample consists of undeformed triangular prisms 2 and 3, separated by shear bands 1. The bases of the undeformed prisms lie on both surfaces of the film.

Figure 3 shows a scanning electron microscope image of the side surface of PET film, which was coated with a 10 nm thick platinum layer after rolling and then heated to 90°C, slightly above the glass transition temperature (75°C). The heating caused the polymer to shrink, leading to cracking of the coating and the formation of folds in the direction perpendicular to the cracks, allowing the observation of mesoshear bands. Notable is the widening of the shear bands at the film surface, as well as the slight downward shift of the intersection region of the shear bands near the top surface of the film.

Figure 4 presents a photograph of the side surface of the film with $\Lambda = 1.06$ rolled to the point indicated by the arrow. The sample was rolled from right to left.

POLYMER SCIENCE, SERIES A 2024

Rolling in the area of sample thickening (indicated by the arrow) was stopped, after which the sample was removed from the rollers. A small bulge is visible in front of the roller. The lower surface of the film remained flat, and the thickness only decreased on the upper side, as schematically shown in Fig. 4b. The film became thinner only on the upper surface of the sample, while the area near the lower surface remained undeformed. The roller rolled along the lower surface of the film. This behavior during rolling of metals and polymers had not been observed before.

Figure 5 shows a similar photograph of the side surface of the film at a higher rolling degree of $\Lambda = 1.10$. Two zigzag deformation bands are present in the film, and the thickness change occurs on both the upper and lower surfaces. The photo allows the determination of the growth turning points of the sawtooth



Fig. 3. Raster electronic image of the side surface of a PET film, onto which, after rolling, a 10 nm thick platinum coating was applied and then heated to a temperature of 90° C.



Fig. 4. Lateral surface of the film at $\Lambda = 1.06$ in reflected light. Rolling was stopped in the area marked by the arrow. The rolling diagram is shown on the right.



Fig. 5. Side surface of PET film during rolling process. Rolling degree $\Lambda = 1.10$. In the middle of the sample, rolling was stopped and the sample was removed from the rollers. The schematic drawing on the right illustrates the direction of shift in the stripes.

zones. These appear at the points where the rollers contact the unraveled part of the film and are directed towards the unoriented polymer, as shown in the schematic drawing on the right.

Visual examination of the rolling process revealed that at $\Lambda = 1.06$ and high rolling speeds, the roller alternately rolls over the top (Fig. 6) and then the bottom surface of the sample, etc. The unrolled tip of the sample in the rollers oscillates up and down, as shown in Fig. 6. In the schematic Fig. 6a, the top roller rolls while deformation occurs on the bottom surface. A shear band A first appears at this surface. Subsequently, a new shear band B forms at the tip of band A, and deformation occurs at the top roller. Finally, a new shear band C starts at the bottom roller (right side of the figure). The oscillation of the sample tip is caused by the alternating rolling of the roller over the top and bottom surfaces of the film. Consequently, the sample bends alternately at the top and bottom surfaces. In a separate experiment, the number of oscillations of the sample tip was measured and found to be twenty, which matches the number of parallel bands on the top surface of the sample (4 in Fig. 2b). This confirms that the rollers alternately roll over one surface and then the other of the sample.

ROLLING DEGREE

Figure 7 illustrates the effect of rolling degree Λ on the deformation mechanism of the film. The photo shows the side surfaces of the film in reflected light at different rolling degrees. At $\Lambda = 1.06$ and 1.085, the bottom surface is smooth, but at rolling degrees $\Lambda = 1.125$ and higher, the sample undergoes plastic deformation simultaneously, though unevenly, through multiple sawtooth shear zones on both surfaces. Thus, as the rolling degree increases, the number of sawtooth zones gradually increases and transitions to more homogeneous deformation.

The corresponding microtome-cut side sections of the film are shown in Fig. 8. In the upper left corner of Fig. 8a, a thin micro shear band reflected from the top surface of the film is marked with an arrow. After the shear band emerges onto the surface of the film, a perpendicular band forms at its tip, extending to the



Fig. 6. Schematic drawing illustrating the rolling mechanism.



Fig.7. Lateral surface of the PET film at the degree of rolling Λ equal to (a) 1.06, (b) 1.10, (c) 1.085, and (d) 1.125 in reflected light.

opposite surface. Mesoshear bands develop from thin micro bands by increasing in thickness. The "reflection" of the shear band is explained by the heating of the polymer, which causes the perpendicular band to form in the old band, resulting in a unified sawtooth deformation zone.

Increasing the rolling degree leads to the formation of several overlapping sawtooth deformation zones. The number of such zones increases with rising Λ . An increase in the rolling degree results in more shear zones and a gradual homogenization of the deformation process. As a result, the rollers deform the film almost symmetrically.

It was hypothesized that the formation of the sawtooth deformation zone is due to thermal softening of the polymer in the shear bands, which suppresses the formation of competing micro bands. To test this hypothesis, the PET film was dusted with micro-sized particles of colored benzyl (melting temperature 95°C), approximately 10 μ m in size, and covered with a second PET film. The double-layer sample was sealed at both ends to prevent mutual slippage of the films and then rolled at a speed of 1300 mm/min. After rolling, the sample was separated, and the powder was removed with a soft brush. Figure 9 shows the rolled film under a transmitted light microscope after the powder particles were removed. In the areas marked by arrows, the powder melted and fused to the polymer. In the other areas, the particles did not melt and were removed with a brush. The melting of the particles indicates that the temperature in the shear bands exceeded 95°C. The glass transition temperature of PET is 70–80°C, so the polymer in the shear bands was in a viscoelastic state.

Figure 10 presents an optical image of the side section of the double-layer PET sample, rolled at a speed of 1300 mm/min. Both layers of the polymer deformed together, and the thickness of the bands is $110-150 \mu m$. The vertical lines are artifacts caused by the vibration of the microtome blade.

ANALYSIS

The propagation of a single shear band, under the assumption that its thickness grows at a constant rate, was considered theoretically [28]. Heat transfer to the rolls was neglected and only the thermal conductivity of the polymer was taken into account. It was assumed that the shear flow is localized at the front of the strip, where heat is released. Solving the thermal conductivity equation showed that the temperature is maximum



Fig. 8. Side cut of a sample at rolling degrees Λ equal to (a) 1.12, (b) 1.19, and (c) 1.27.

at the front of the strip, and its increase is described by the formula

$$T = \frac{\beta \varepsilon \tau}{\rho c} \operatorname{erf} z, \qquad (1)$$

where *T* is the heating value, ρ is the density, *c* is the specific heat capacity of the polymer, β is the fraction of mechanical work converted into heat, $\varepsilon = \Delta/h$ is the shear strain in the strip, Δ is the shear value, *h* is the strip thickness, τ is the shear stress, $\operatorname{erf} z = \frac{2}{\sqrt{\pi}} \int_0^z \exp x$

 $(-t^2)dt$ is the probability integral, $z = \sqrt{\frac{uh\rho c}{4k}}$, k is the thermal conductivity coefficient, u is the strip thickness growth rate related to rolling speed v (roll speed)

by the formula $u = \frac{\Lambda - 1}{2\sqrt{2N\epsilon}}V$, where *N* is the number of sawtooth zones.

Factor $\frac{\beta \epsilon \tau}{\rho c} = T_o$ in front of the probability integral

in formula (1) is the value of adiabatic heating, when heat does not escape from the strip either due to the thermal conductivity of the polymer or into the external environment. The T/T_o ratio values corresponding to heating in the bands shown in Figs. 1 and 9, are given in Table 1. The condition for the strip to be adiabatic is satisfied for $z \gg 1$. For $z \ll 1$, the error integral



Fig. 9. Top view of a two-layer PET sample, between the layers of which benzyl powder was poured, after rolling at a speed of 1300 mm/min. The thickness of the strips is $110-150 \,\mu$ m.

is described by the formula $\operatorname{erf} z = \frac{2}{\sqrt{\pi}} z$. Taking into account $z = \sqrt{\frac{uh\rho c}{4k}}$ and $u = \frac{\Lambda - 1}{2\sqrt{2N} \varepsilon} V$ [28], we have

$$T = \frac{\beta \varepsilon \tau}{\rho c} \sqrt{\frac{h \rho c (\Lambda - 1) V}{8 \sqrt{2} k N \varepsilon}}.$$
 (2)

The amount of heating depends on several parameters: rolling speed v, the number of bands N, their thickness h, and shear deformation ε . While the degree and speed of rolling are external parameters set by the technologist, the deformation in the bands ε and the thickness h are not known in advance and require measurement. The thickness of the bands can be measured or estimated if the number of bands and the shear deformation ε are known.

The results of the heating temperature calculations are presented in Table 1 for $\varepsilon = 0.78$ [28]. It is noted that to form a regular structure of three to four sawtooth zones, strong heating is not required. A heating of 10% to 7% of the adiabatic limit is sufficient. Assuming that in a double-layer structure, the temperature increase is approximately 80°C, this corresponds to a heating of only 6–8°C. Such a small amount of heating is sufficient to suppress the formation of competing shear bands.

RESULTS AND DISCUSSION

During deformation, mechanical work is converted into heat [24, 25]. Polymer flow is non-uniform and localized within shear bands. Consequently, heat is also localized within these bands. Shear bands have been referred to as "adiabatic" in previous work [29], a term that has become established but requires clarification. It is important to distinguish between the adiabatic deformation of the sample and the shear bands. In the latter case, heat remains within the bands. Although the term "adiabatic bands" has been used for some time [29], it initially referred to homogeneous deformation of the material and essentially related to adiabatic deformation of the sample rather than the shear bands themselves.

Flow is considered adiabatic if the time of plastic deformation *t* is much shorter than the characteristic heat transfer time τ . For the sample, the heat transfer time τ is estimated by the formula [29]:

$$\tau = H^2/a,\tag{3}$$

where *H* is the sample thickness, $a = \sqrt{\frac{k}{\rho c}}$ is the thermal diffusivity coefficient. We estimate the deformation time *t* by dividing the length of the flow region L

POLYMER SCIENCE, SERIES A 2024

Fig. 10. Optical image of a side cut of a two-layer PET sample after rolling at a speed of 1300 mm/min. The thickness of the strips is $110-150 \mu m$.

by the roll speed *v*: t = L/V. The transition from isothermal to adiabatic flow occurs at $\tau = t$:

$$V = \frac{La}{H^2}.$$
 (4)

During rolling, not the entire sample is deformed, but only the area under the roll, the size of which at small degrees of rolling Λ [30] is described by the formula $L \approx \sqrt{2HR(\Lambda - 1)}$, where *H* is the film thickness, and *R* is the radius of the roll. At H = 0.9 mm, R =50 mm, $\Lambda = 1.06$, k = 0.14 W/(m K), $\rho = 1330$ kg/m³, and c = 1/13 J/(kg K), deformation rate during transition to adiabatics (formula (4)) is estimated at 1 m/s.

Table 1. Ratio T/T_{o} values

Rolling speed <i>v</i> , mm/min	Ν	h, μm	$T/T_{\rm o}$
1300*	1	110-150	0.76-0.83
1300	1	45	0.55
416	2	22.5	0.155
310	3	15	0.099
260	4	11	0.068
26	10	4.5	0.0084

h is the thickness of the stripes, T_0 is the adiabatic limit of heating of the polymer, *N* is the number of simultaneously growing shear bands (in Fig. 1a it is the density of stripes per 1 mm). *Double film shown in Fig. 10.



Note that in industry, rolling of metal sheets is carried out at speeds of up to 5 m/s.

Similarly, the conditions for adiabaticity of shear bands are analyzed. In this case, the characteristic dimension is the thickness of the band h. not the sample thickness H. Based on the film thickness shown in Fig. 1a, the number of microbands per millimeter is approximately 50, and for $\varepsilon \approx 1$, the thickness of a microband is estimated to be 1 µm. This is three orders of magnitude smaller than the film thickness. Consequently, the rolling speed required for achieving adiabatic conditions (4) must be six orders of magnitude higher. This means that adiabatic micro-shear bands are not feasible not only in metals but also in polymers, whose thermal conductivity is two to three orders of magnitude lower. However, during rolling, sawtooth mesobands with thicknesses up to 100 µm appear (Fig. 10), and the conditions for adiabaticity become practically achievable, as indicated by the data in Table 1. Thus, adiabaticity is realistic for mesobands but not for micro-shear bands. According to Table 1, even for microbands with a thickness of 4.5 um, the heating is less than a percent of the adiabatic limit.

Several sawtooth zones of flow create an ordered structure, with the spacing between the sawtooth zones being practically uniform. In contrast, at low rolling speeds, microbands appear randomly. This may be explained by the fact that they are initiated by randomly located defects. However, with increased rolling speed, a transition from disorder to order occurs. Similar phenomena are observed in the intensive deformation of viscoelastic fluids [31]. Sawtooth flow zones form two to seven flow zones, creating an ordered structure. This transition is associated with heating in the shear bands, but the mechanism of the transition to order remains unexplained.

CONCLUSIONS

As the rolling speed increases, the mechanism of plastic flow transitions changes from shear in irregular microbands to several regularly spaced sawtooth meso-zones of flow, and eventually to the formation of a single sawtooth zone. An increase in the degree of rolling also leads to a rise in the number of simultaneously developing sawtooth zones and the homogenization of the rolling process. The appearance of regular meso-shear bands is explained by the heating of the polymer during shear, which suppresses the formation of competing shear bands.

FUNDING

This work was supported by ongoing institutional funding. No additional grants to carry out or direct this particular research were obtained.

CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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Translated by V. Avdeeva

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