Stretching of PET Films under Constant Load.
I. Kinetics of Deformation

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Synopsis

The deformation of amorphous and isotropic polyester film is studied using a device which simulates on a laboratory scale the continuous stretching between rolls by a drawing under constant load. The kinetics of deformation is compared to the kinetics involved in a conventional drawing at constant speed of elongation. The results of drawing under constant load are applied to the industrial case of stretching between rolls.

INTRODUCTION

In film processing and low speed fiber processing, polymeric materials are submitted to a stretching between rolls, at least during one of the steps of the process.[1] This drawing is performed between two rolls whose tangential speeds are different from each other.

The main characteristic of this kind of stretching is the drawing force which is constant if air drag, mass, and inertia forces are neglected.[2] This implies that a good laboratory simulation of a continuous stretching between rolls is an experiment of drawing under constant load, i.e., a creep experiment at high deformation.

This type of stretching has been studied in the past for various fibers like PA 66,[3-5], PA 6,[6,7] PP,[8] and PET.[8-10] More recently, this technique has been used to produce high modulus PET film or fiber with the so called zone-annealing method.[11,12]

The present work reports a study of the kinetics of deformation involved in a drawing under constant load of PET film whose width is kept constant. A comparison is done with the drawing at constant speed of elongation. The results at constant force are used to describe the industrial stretching between rolls.

EXPERIMENTAL

Specimen Preparation

Amorphous isotropic PET films (thickness 150 μm) were supplied by Rhône-Poulenc Films. T_g was 76°C as measured by DSC at 10 K/mn with a Mettler analyzer.

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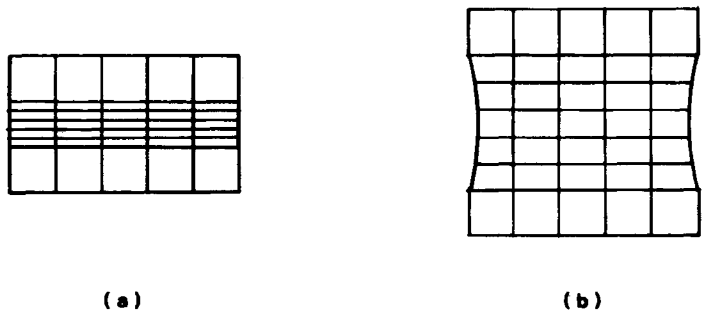

Fig. 1. Sample before (a) and after (b) stretching.

Rectangular samples (1 cm long × 5 cm wide) were cut from the sheet. A lattice (2 × 10 mm) has been drawn on the samples to measure accurately the deformation [Fig. 1(a)].

Drawing under Constant Load

The experiments of stretching under constant load have been performed in a blown air oven with a specific device of dropping load. A schematic representation of the apparatus is given in Figure 2.

The sample was clamped between jaws with low thermal inertia. A wire was suspended on the lower jaw and different weights were hung on this wire. The drawing was triggered by an electromagnetic device and was stopped by shifting a moving plate.

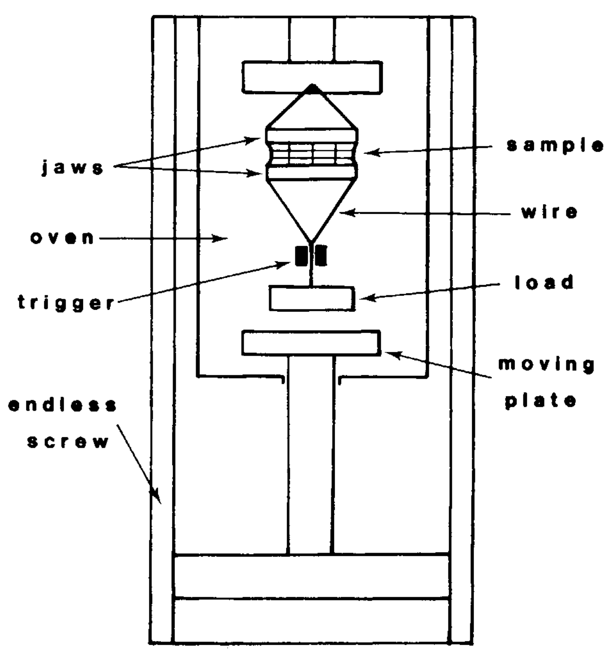

Fig. 2. Schematic description of the apparatus of stretching under constant load.

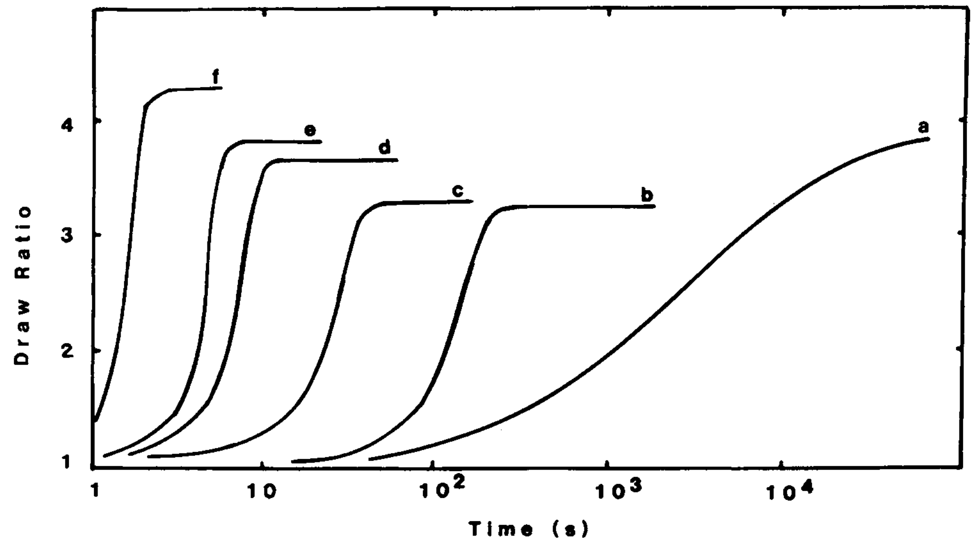

Fig. 3. Kinetics of deformation at 80°C for various loads expressed in kg/mm^2: (a) 0.15; (b) 0.38; (c) 0.50; (d) 0.63; (e) 0.75; (f) 1.05.

In order to describe the kinetics of deformation, the change of the lattice was recorded during the drawing, using a high speed video camera NAC (200 pictures/s). By playing subsequently the video tape, it was possible to build the deformation curve as a function of time by step of 5 ms.

The measurements were done at the center of the sample where the width remains constant [Fig. 1(b)]. If DR is the applied draw ratio, this implies that the final thickness e_f becomes:

$$e_f = e_i/\mathrm{DR} \qquad (e_i = \text{initial thickness})$$

That means that the symmetry is uniaxial-planar and not uniaxial, for which $e_f = e_i/\sqrt{\mathrm{DR}}$.

RESULTS AND DISCUSSION

Kinetics of Deformation

Figure 3 shows the kinetics of deformation obtained with various loads at 80°C. The important properties of these curves are the following:

—All the curves end by a plateau where the polymer is in equilibrium under the applied tension.
—The lower possible draw ratio for a plateau is $\mathrm{DR}_p = 3.2$ at a temperature of 80°C. This value constitutes the natural draw ratio (NDR) at 80°C (curves b and c).
—Under very low tension (curve a), the plateau deformation corresponds to a draw ratio much higher than the NDR. In this case, the kinetics of stretching is very slow and the chains have time enough to slip, which induces a higher DR_p. This phenomenon is well known for drawings at constant strain rate[13] or

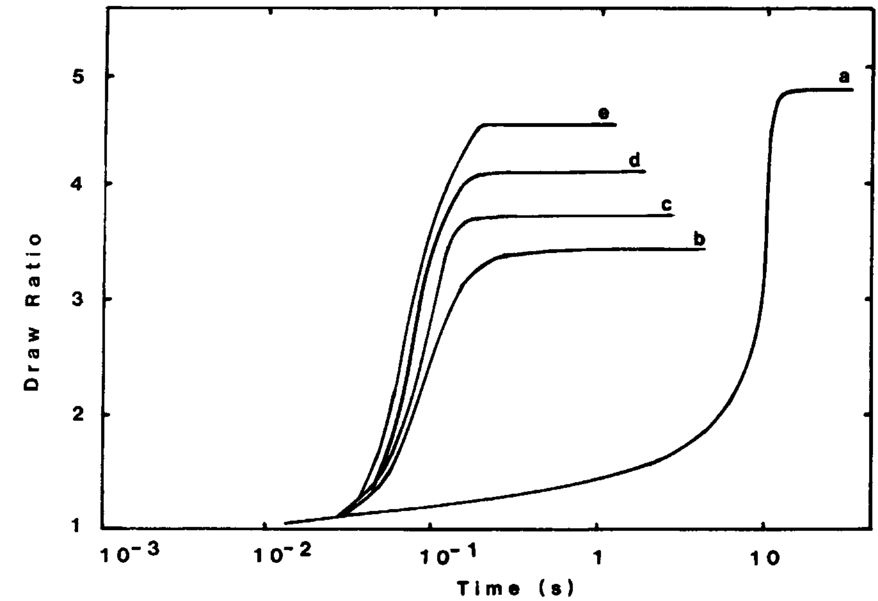

Fig. 4. Kinetics of deformation at 97°C for various loads expressed in kg/mm^2: (a) 0.13; (b) 0.25; (c) 0.38; (d) 0.50; (e) 0.63.

at constant speed of elongation,[14] for which the NDR increases when the rate decreases.

—Under high tension (curves d and e), the plateau deformation increases with the applied load. This evolution corresponds to the stress hardening of conventional stretching at constant rate.

—In all the cases, the higher is the tension, the shorter is the time t_p needed to reach the plateau.

The kinetics of deformation at 97°C are reported in Figure 4. The results are similar to the case of a drawing at 80°C: existence of a plateau for each drawing force, existence of an NDR. The same influence of the tension is observed.

The effect of an increase of the drawing temperature is to accelerate the kinetics of deformation. As shown in Figure 5, the time needed to reach the plateau is about 100 times shorter at 97°C than at 80°C. The natural draw ratio increases with the temperature (3.4 at 97°C), which is a well-known result for drawings at constant rate.[13,14]

Evolution of the Strain Rate

The main parameter which controls the chains relaxation during a stretching at a given temperature is the strain rate $\dot{\epsilon} = (1/l)(dl/dt)$. The quantity $1/\dot{\epsilon}$ is similar to time and can be considered as a characteristic time to be compared to the relaxation times of the polymer.[15] Therefore, the value of $\dot{\epsilon}$ determines the efficiency of the stretching to generate chains orientation.

From the deformation curves of Figures 3 and 4, the evolution of the strain rate during the stretching has been calculated. At 80°C (Fig. 6), after 10% of

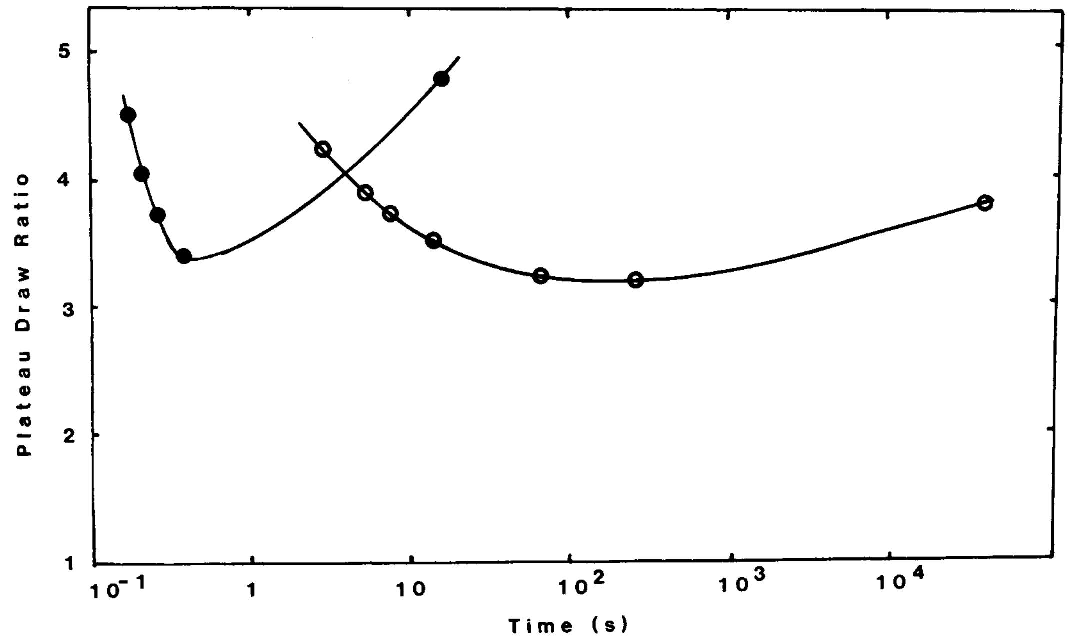

Fig. 5. Time to reach the plateau deformation DR_p at 80°C (○) and 97°C (●).

deformation, the strain rate varies between 0.1 s^{-1} and a maximum value around 1 s^{-1} under high drawing forces. At 97°C (Fig. 7), in the same way, $\dot{\epsilon}$ varies between 1 s^{-1} and around 30 s^{-1}. Thus, the drawings under constant load can be characterized by strain rates spectra which depend on the stretching force and the temperature.

It is interesting to compare these results with those of a drawing at constant speed of elongation. The latter is equivalent to an industrial drawing in a stenter of the flat film process. The evolutions of the strain rate and the deformation are reported in Figure 8 for a stretching at a rate of elongation of 4000%/min, which is a typical industrial value. For this kind of drawing:

$$V = \frac{dl}{dt} = \text{const}$$

Thus,

$$l = V_t + l_0 \qquad \text{and} \qquad \dot{\epsilon} = 1/(t + l_0/V)$$

At 4000%/min, the strain rate is always lower than 1 s^{-1}.

It is to be noted that, at 80°C, the strain rates involved in a drawing under constant load are of the same order of magnitude as those generated at constant speed of elongation (less than 1 s^{-1}). However, at higher temperatures, the strain rates of a stretching at constant force are much larger than the case $V =$ const (at least 10 times higher at 97°C). That means that a drawing under constant load is very efficient at high temperatures to generate chains orientation and this point has been enhanced by Kunugi and co-workers, who made high-modulus PET films by this kind of stretching.[11,12]

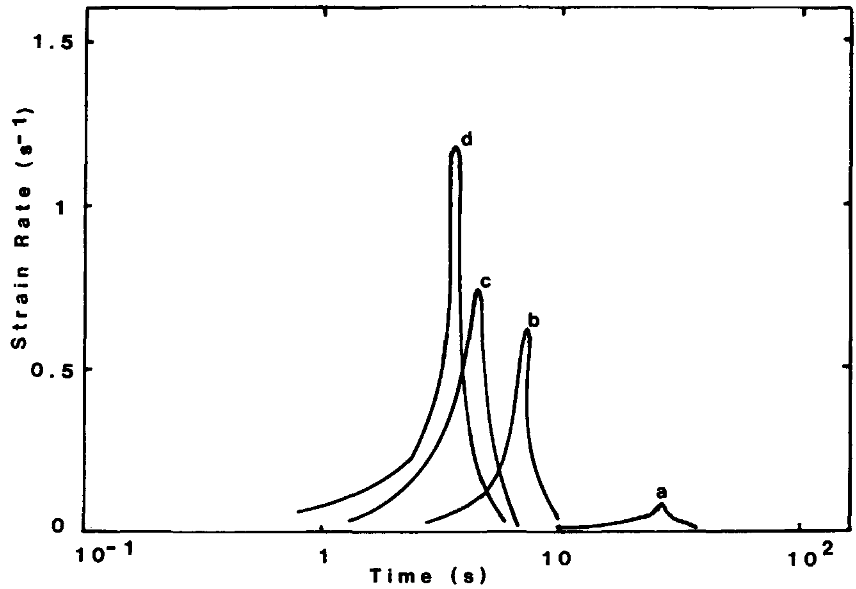

Fig. 6. Evolution of the strain rate during drawings at 80°C under various loads expressed in kg/mm^2: (a) 0.50; (b) 0.63; (c) 0.75; (d) 0.88.

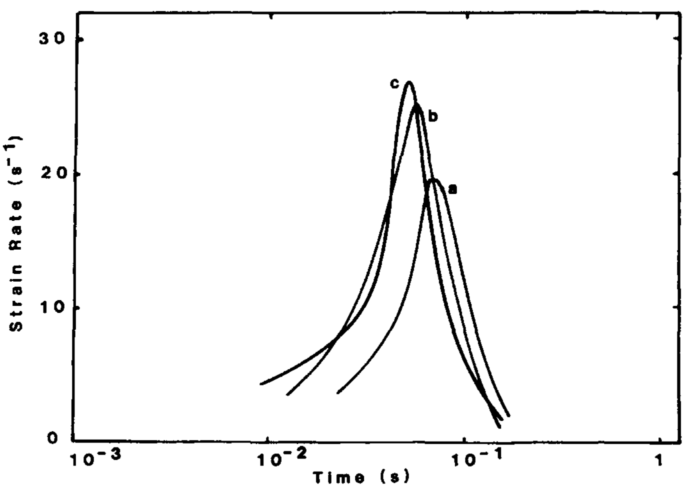

Fig. 7. Evolution of the strain rate during drawings at 97°C under various loads expressed in kg/mm^2: (a) 0.38; (b) 0.50; (c) 0.63.

Application to the Stretching between Rolls

A schematic representation of a stretching between rolls is given in Figure 9. The drawing force F remains constant between the two rolls and the draw ratio DR is defined by the ratio of the fast speed V_2 over the slow speed V_1.

In the case of a stretching between rolls, the force F cannot be chosen. However, we apply a draw ratio DR, which implies a minimum residence time t_{rm} and a maximum residence time t_{rM} of a film element in the stretching

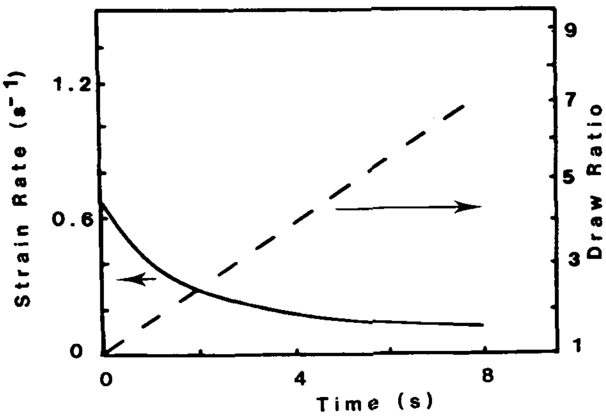

Fig. 8. Evolution of the deformation (dashed line) and the strain rate (solid curve) during a stretching at $V = 4000\%/\text{mn}$.

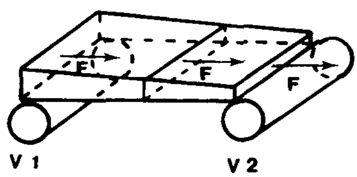

Fig. 9. Schematic description of a drawing between rolls (slow speed V_1 and fast speed V_2).

zone. These three quantities are determined by the stretching distance d and the rolls speeds V_1 and V_2:

$$\text{DR} = V_2/V_1, \qquad t_{rm} = d/V_2 \text{ and } t_{rM} = d/V_1$$

In fact, the true residence time t_r is such that

$$t_{rm} < t_r < t_{rM} \text{ and } t_r = \int_0^d \frac{dx}{V(x)}$$

Therefore, the film will choose its kinetics of deformation so that the associated force takes a minimum value.

For a given temperature and a given draw ratio, there is a minimum time t_p needed to reach the plateau deformation DR_p. This time t_p is defined by the curves of Figure 4. If the residence time between rolls t_r is longer than t_p, the polymer has time enough to reach the plateau deformation DR_p under the minimum drawing force F [Fig. 10(a)]. If the residence time t_r is shorter than t_p, the film will be strained faster under a larger force F', which would lead to a plateau deformation higher than the applied draw ratio DR. By quenching on the cold fast roll, the stretching is stopped at the applied draw ratio [Fig. 10(b)]. According to the residence time t_r between the rolls, we can distinguish three types of drawings by their kinetics:

—stretching with quenching
—stretching with a short plateau
—stretching with a long plateau

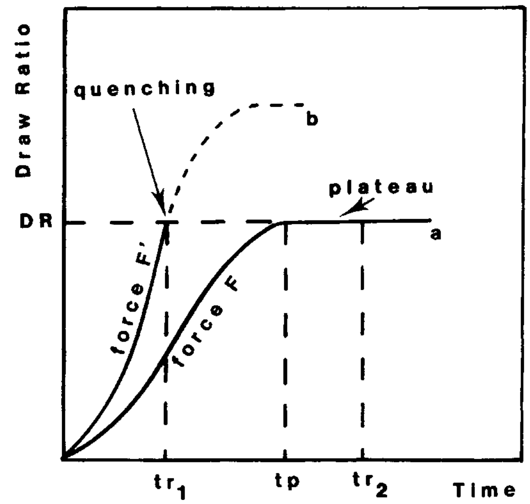

Fig. 10. Typical kinetics of deformation between rolls.

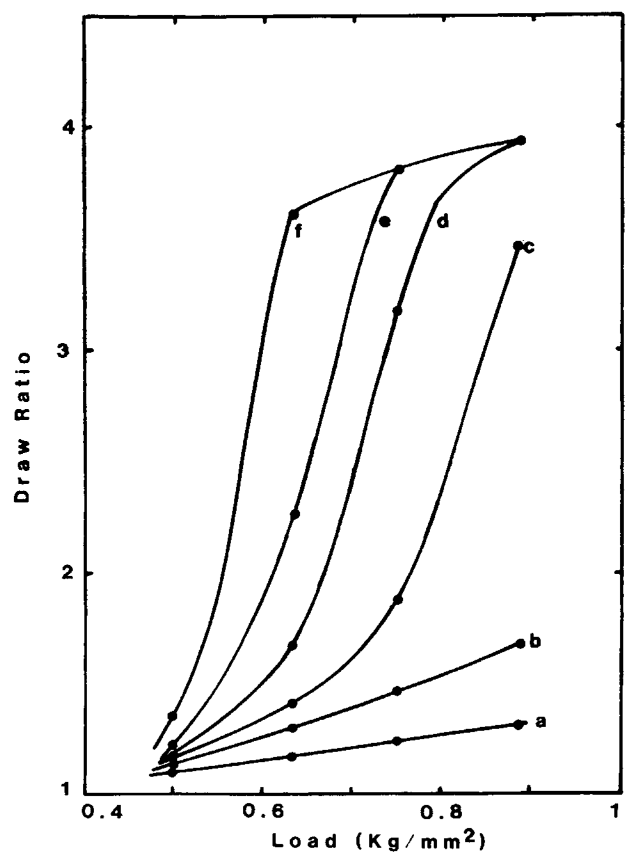

Fig. 11. Evolution of the draw ratio with the stretching force at 80°C for various drawing times: (a) 2 s; (b) 3 s; (c) 4 s; (d) 5 s; (e) 7 s; (f) 10 s.

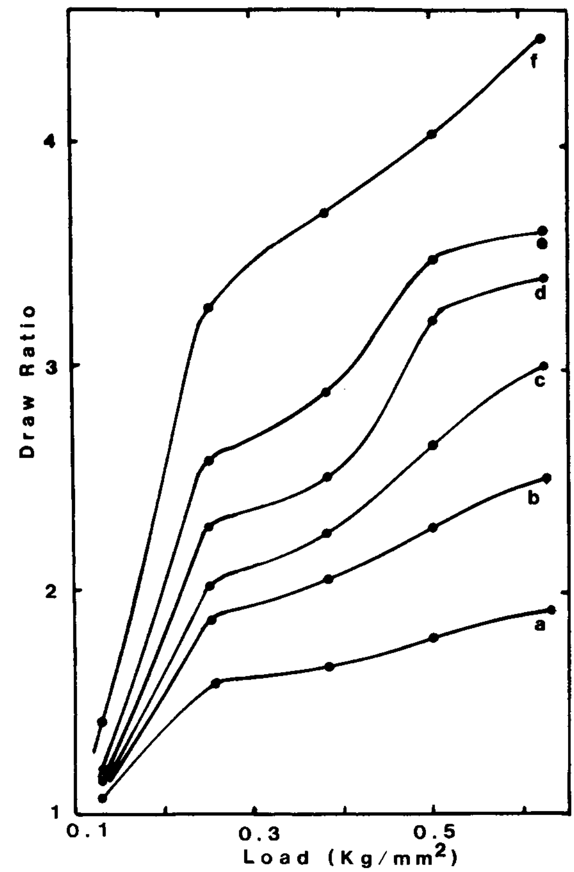

Fig. 12. Evolution of the draw ratio with the stretching force at 97°C for various drawing times: (a) 0.06 s; (b) 0.07 s; (c) 0.08 s; (d) 0.09 s; (e) 0.1 s; (f) 0.2 s.

From the laboratory experiments of drawing under constant load (Figs. 3 and 4), the evolution of the draw ratio with the applied load at different stretching times has been reported in Figures 11 and 12. With these curves, knowing the residence time of the film between rolls, it is possible to deduce the drawing force associated to a given stretching between rolls.

CONCLUSIONS

In the investigation reported in this paper, the kinetics of deformation at constant force have been described in detail. From the results, some main conclusions can be drawn:

1. The deformation curves end by a plateau.
2. At a given temperature, there is a minimum value of the plateau deformation (Natural Draw Ratio).

3. Under low drawing tension, the plateau deformation increases when the load decreases. Under high tension, this plateau deformation increases with the load.
4. The time needed to reach a plateau always decreases when the load increases.
5. The effect of a higher stretching temperature is to raise the natural draw ratio and to accelerate the kinetics of deformation.
6. At temperatures far from T_g, the strain rates involved in a stretching under constant load can reach very high values like 30 s^{-1} at 97°C.
7. These laboratory experiments enhanced three typical cases of stretching between rolls:

—drawing with quenching
—drawing with a short plateau
—drawing with a long plateau

A structural analysis of PET films stretched under constant load will be reported in Part II of this paper.

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