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Temperature Dependent Uniaxial Stress Relaxation in Polyamide 6

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SUMMARY

Stress relaxation phenomena in polyamide 6 are presented in this paper. Tests were carried out on the tensile testing machine Fu 10 000e, using oar-shaped samples. Dependence of uniaxial stress changes in time, temperature or initial load was observed and measured. Analytical and mechanical models of stress relaxation in polyamide 6 were developed and are presented here.

INTRODUCTION

Time dependence in properties of polymers manifests itself in several forms, for example: creep - a progressive increase in deformation under a constant load, and stress relaxation - a gradual decrease in stress under a constant deformation. Both these phenomena influence, and in many cases limit, possibilities of using plastics for load bearing applications. The property of creep and stress relaxation do not preclude the use of plastics for these applications, but a knowledge of these phenomena is essential for design purposes. That is why many scientists took interest in stress relaxation, namely: structural changes character and description of the phenomena. They were: [HAWTHORNE 1981] - in polyethylene, [ADACHI 1981] - in polypropylene, [KATO and YANAGIMARA 1981] - in polymethacrylate. Analytical and mechanical models of stress relaxation were presented by [BERGER and WOLSTENHOLM 1960].

EXPERIMENTAL

Experiments were carried out on the tensile testing machine Fu 10 000e at 293, 303, 313, 323, 338 and 353 K. Uniaxial initial stresses of 30, 40 and 50 MPa were applied by axial tension. Polyamide 6 which was obtained from anionic polymerisation of caprolactam was examined. In experiments standard samples for tension tests were used. The samples were cut out from plates produced by casting. The thick-

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ness of the plates corresponds with the width of samples. The samples were seasoned in climatic chamber MYTRON in constant temperature 293±1 K and relative humidity 65±1.5%. The experiments applied a strain at constant speed in an elastic range of material. All test were conducted in heating chamber T 250 at a designated temperature checked with accuracy ± 1 K. On the grounds of previous research we ascertained that a soaking period of 5 minutes at a designated temeperature was necessary. A special control system checked constant elongation and absence of deformation in force measurement assembly. The preliminary experiments assumed the stress relaxation time equal to 45 min, afterwhich in the majority of experiments the relaxed stress curve ceased its decline and leveled off. Only for T=293 K and initial load σ_{max} = 30 MPa, σ_{max} = 40 MPa relaxation time increased to 75 min, and for temperature T=353 K and initial load σ_{max} = 50 MPa was reduced to 20 min. Five tests were made at each temperature and initial load. At constant intervals of time stress levels were read from received stress/time curves. The first reading was taken after the straining phase was completed. Average values, standard errors and confidence intervals were calculated for this series of experiments.

RESULTS AND DISCUSSION

Figure 1 shows schematically a typical stress/time curve recorded during experiments.

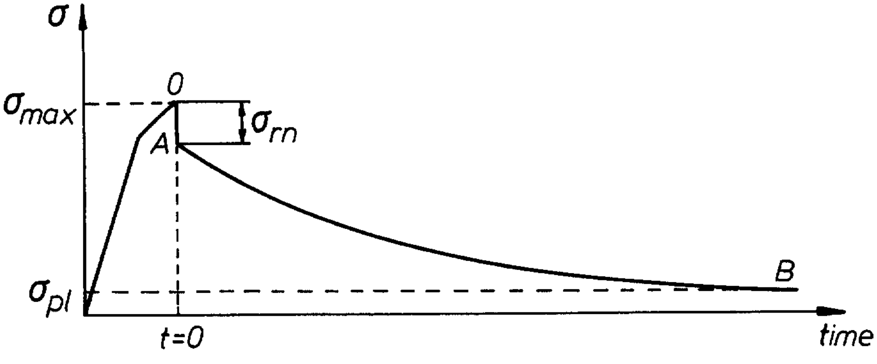

Fig.1 Typical curve of stress relaxation in polyamide 6
OA - immediate elastic recovery
AB - retarded elastic recovery
B∞ - plastic flow

It was evident from the stress/time curves, that the following expression can be used to describe satisfactorily the stress relaxation behaviour of polyamide 6.

$$\sigma(t) = \varkappa\,\sigma_{rn} + C e^{at} + \sigma_{pl} \qquad [1]$$

where: $\varkappa$ - Heavisede`a function = $\begin{cases} 1 \text{ for } t = 0 \\ 0 \text{ for } t \neq 0 \end{cases}$

$$C = [(\sigma_{max} - \sigma_{rn}) - \sigma_{pl}]$$

From the analysis of all curves it was found that each of values in equation [1] is a function of temperature and initial load. This is why general equation of stress relaxation can be expressed in the form:

$$\sigma(t) = f_1(T,\sigma_{max})\varkappa\sigma_{rn} + f_2(T,\sigma_{max})Ce^{f_3(T,\sigma_{max})at} + f_4(T,\sigma_{max})\sigma_{pl} \qquad [2]$$

For detailed calculation of coefficients "C" and "a" a least squares method was used. Results obtained in calculations show Fig. 2 and Fig. 3.

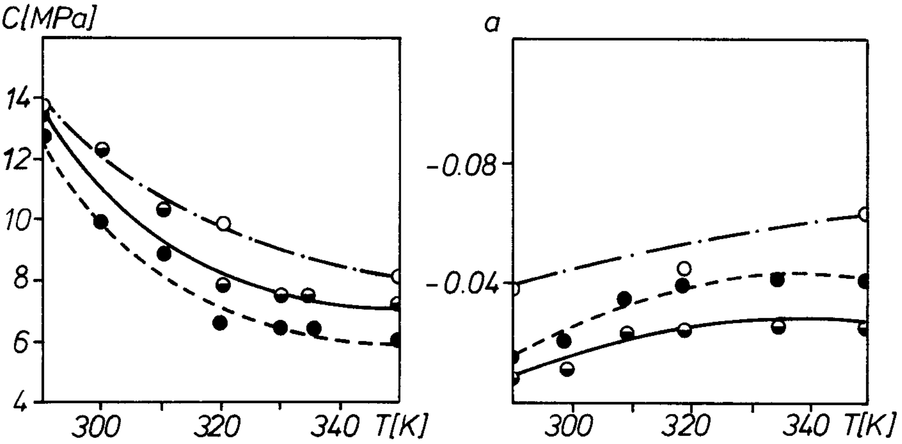

Fig.2 Diagrams of coefficients "C" and "a" in depend on temperature

$-\,-\bullet-\,-$ $\sigma_{max} = 30$ MPa $\quad$ —◒— $\sigma_{max} = 40$ MPa $\quad$ $-\cdot-\circ-\cdot-$ $\sigma_{max} = 50$ MPa

From Fig. 2 and 3 we can see that coefficient "C" nonlinearly decreases with increasing of temperature and increases linearly with the increase of the initial load. The dependence of coefficient "a" on temperature and initial load is more interesting, because it tells us about speed of relaxation [the greater the value of coefficient "a", the quicker the progress of relaxation]. The inverse of coefficient "a" is called relaxation time. The analysis of the dependence of coefficient "a" on temperature and initial load gives us dependence of speed of relaxation on two previously mentioned elements. Coefficient "a" increases with

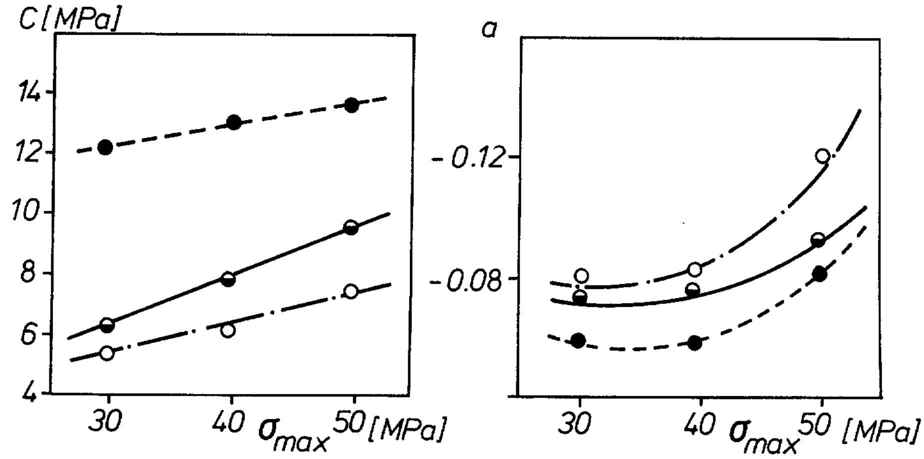

Fig.3 Diagrams of the variation of coefficients "C" and "a" in the function of initial load

- - ● T = 293 K —— ◒ T = 323 K —·— ○ T = 353 K

the increase in temperature to a certain level. On the other hand when the initial load increases, the level of coefficient "a" remains constant until 40 MPa, after which it increases. For a more conclusive illustration of results we also calculated components in equation [1], which are illustrated in Fig. 1. Results which were obtained are showned on Fig. 4 and 5.

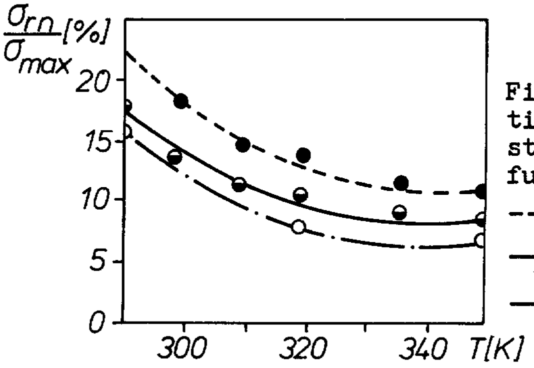

Fig.4a Variation in the participation of immediate elastic recovery in eq.[1] in function of temperature

- - ● σ_{max} = 30 MPa
—— ◒ σ_{max} = 40 MPa
—·— ○ σ_{max} = 50 MPa

The results were calculated twice: the first time for constant initial load and variable temperature (Fig.4), the second for constant temperature and variable levels of initial load (Fig.5). From Fig.4 we can see that participation in immediate and retarded elastic recovery in eq.[1], in applied temperature range of 293-353 K decreases with the increase of temperature and with the increase of applied level of initial load. The second correlation is linear (Fig.5). The participation of stress which corresponds with plastic strain increases nonlinearly with the increase of temperature (Fig.4).

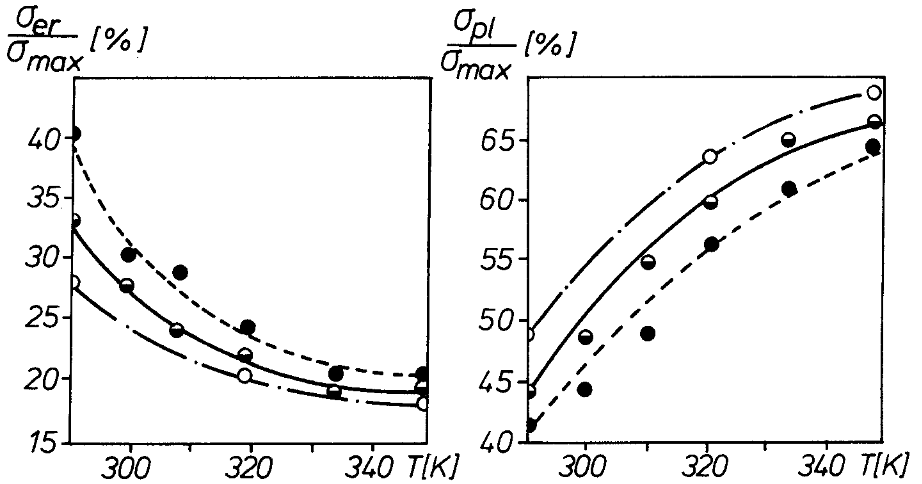

Fig.4b Variation in the participation of retarded elastic recovery and plastic flow in eq.[1] in function of temperature

— — ● σ_{max} = 30 MPa ——— ◒ σ_{max} = 40 MPa —·— ○ σ_{max} = 50 MPa

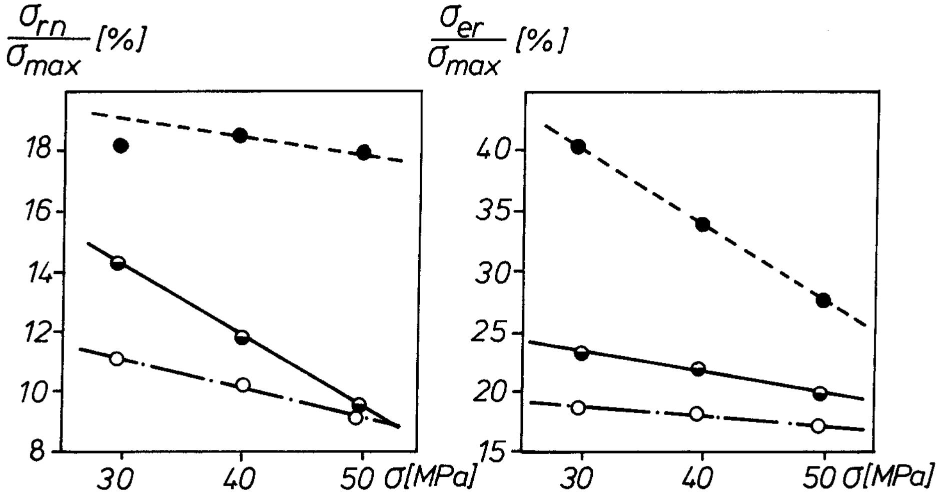

Fig.5a Variation in the participation of the immediate and retarded elastic recovery in eq.[1] in function of initial load

— — ● T = 293 K ——— ◒ T = 323 K —·— ○ T = 353 K

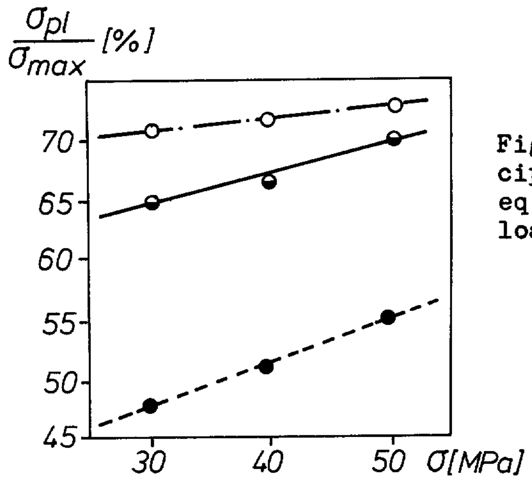

Fig.5b Variation in the participation of plastic flow in eq.[1] in function of initial load

- - - ● T = 293 K
——— ◒ T = 323 K
—·— ○ T = 353 K

The participation of stress corresponds with the plastic strain in eq.[1], also display the just as immediate and retarded elastic recovery linear relationship with applied level of initial load. In comparison with former components there is a linear increase (Fig.5). Analysis of the dependence of stress corresponds with plastic strain in eq.[1] provides us with changes in the degree of relaxation (i.e. value of relaxed stress) in functions of temperature and initial load. These changes are then inverse for the stress corresponding with plastic strain, and so the degree of relaxation decreases non-linearly with the increase of temperature and asymptotic tend to the certain level. The degree of relaxation also decreases but linearly with the increase of applied level of initial load. It was also ascertained, that mechanical model which best describes uniaxial stress relaxation phenomena in polyamide 6 is a model created by series connection of: Hook`s, Kelvin-Voigt`s and Newton`s models (Fig.6).

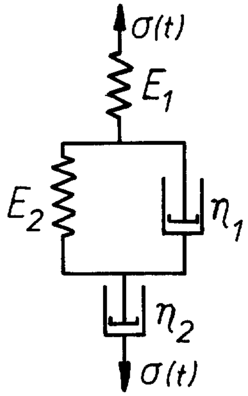

Fig.6 Mechanical model corresponding with uniaxial stress relaxation phenomena in polyamide 6

CONCLUSIONS

It has been shown that uniaxial stress relaxation phenomena in polyamide 6 described analytical model including dependence of immediate and retarded elastic recovery and plastic flow in functions of temperature and initial load. The mechanical model, which best corresponds with the analytic equation, is a series connection of Hook`s, Kelvin-Voigt`s and Newton`s models. Experiments proved, that speed of relaxation increases with the increase of temperature, but simultaneously the degree of relaxation decreases (i.e. value of relaxed stress). These two values stabilize on certain levels dependent on initial load. Also ascertained was, that the value of relaxed stress (degree of relaxation) linearly decreases with increasing of initial load, and that speed of relaxation (value of coefficient "a") increases next to exceed the value of 40 MPa.

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