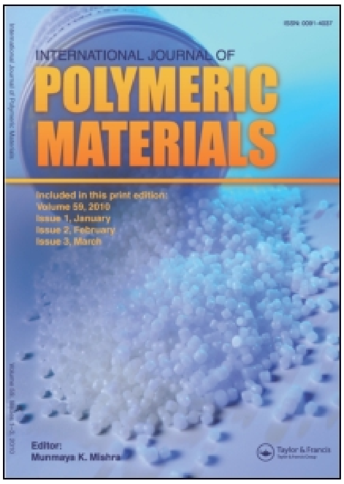

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Mechanical Properties and Plastic Deformation of PA-6 and of some of PA-6 Composites and Blends

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Mechanical Properties and Plastic Deformation of PA-6 and of some of PA-6 Composites and Blends

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It has been followed the short-term mechanical behaviour (tensile strength, elongation at break, modulus of elasticity at 100% elongation, flexural strength, flexural modulus, Charpy impact strength) and creep strain under long-term load, of the commercial polyamide 6, talc-filled polyamide and short glass fiber reinforced polyamide 6 over a large temperature and speed of testing range. The mechanical behaviour of a new polyamide 6/polyethyleneterephtalate blend has been presented.

The graphical and mathematical dependence obtained describing the behaviour of materials under conditions of service and the limits of resistance for PA-6-based materials under various stresses (thermal, mechanical).

Keywords: Polyamide 6; mechanical behaviour; filled polyamide; glass-fiber-reinforced polyamide; polyethyleneterephthalate; blends

1. INTRODUCTION

Mechanical behaviour of plastic materials has an important role in the determination of their applications. Plastics offer an impressive range of attractive properties in use where they are exposed to the outdoor environment in a broad range of conditions. There is always a great concern regarding the durability of polymeric materials because if the

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* Corresponding author.

useful time of these materials can be predicted their maintenance or replacement can be properly planned. Under extreme outdoor exposure conditions a plastic sample reaches temperatures can vary from −70° to 170°C and various mechanical stresses may be applied for different periods of time.

A new polymeric material is generally put on the market with a comprehensive account of its physical and mechanical properties. However, there is seldom sufficient information available to assess the behaviour and length of time the material will fulfill its function satisfactory before the need repair or replacement. It is imperative to determine the basic experimental data on durability of a material so that the designers, formulators and researchers have enough information for correctly formulating a polymer product for its serivce life. The functional requirements of engineering applications usually include both short-term properties such as strength, stiffness, toughness and flexibility (elongation) and long term properties, such as creep resistance and retention of toughness during service.

Mechanical properties (tensile strength, elongation at break, flexural strength or impact strength) are usually determined in extreme conditions at the breaking point when the specimens are destroyed.

Several standard methods have been proposed but the conditions imposed by these methods do not always correspond with practical needs to characterize each material in specific conditions of its service. The polymeric materials could exhibit a viscous, viscoelastic or brittle fracture depending on the testing (or service) speed and temperature.

Both, the design of new products including new molds and also during the utilization of the products are necessary information on the behaviour of materials under various actions as heat and stress. Thermoplastic structural composites offer the designer improved damage tolerance and environmental resistance; inertness, dimensional stability and wear resistance [1].

The overwhelming number of polymer blends, modified resins and composites are produced to improve the mechanical properties of a given dominant component with already useful properties [2]. The capacity of a material to absorb mechanical energy depends on the testing parameters which determine the actual value of each mechanical characteristics. Mechanical properties are influenced by molecular architecture, polymer microstructure, polymer homogeneity. Needless to mention that sample geometry (especially notches), experimental parameters as rate of stress or strain, temperature and environment can also influence a material to such an extent as to provoke tough-brittle transition.

Polyamides rank fourth in worldwide consumption of plastic materials after polyolefins, polyvinylchloride and styrenic polymers [3]. For polyamides the most wide application fields are microelectronics, aeronautics, automotive industry, engine building, where the dimensional stability and mechanical properties of the parts must be approximately constant during the entire service life.

The properties of polyamide-6 (PA-6) depend on its chemical and morphological structure. The crystallites have small dimensions and are randomly distributed into the spherulites or dendrites of the paracrystalline domains. It has also been proposed that the polyamide 6 is a single phase paracrystalline system with a glass transition of 49°C.

Due to this particular morphology it is possible to incorporate into PA-6 homopolymer matrix a large variety of additives of various chemical nature to enhance some properties or reduce cost [4, 5]. So, PA-6 can be plasticized to increase flexibility and processability by diminishing intermolecular forces. It can be also loaded with up to 25 wt% mineral fillers as TiO_2, micronized talc, graphite, metallic powders, carbon black, *etc.*

The mineral fillers increase dimensional stability, improve surface properties, decrease water absorption and shrinkage expansion coefficient at high temperature and also, reduce creep and increase the temperature resistance under load.

Short-fiber reinforced PA is commonly used in automotive industry under-the-hood applications such as radiator and caps and air intake manifolds. The radiator end-cap is one of the earliest large under-the-hood components manufactured from heat-stabilized glass-fiber-reinforced polyamide. The radiator in automobiles transfers the heat of the coolant from the engine to the ambient air. During serivce, the material in a radiator end-cap is exposed to coolant in addition to high temperatures and mechanical loading. Thus the functional requirements include resistance to hydrolysis at elevated temperatures. Normal operating temperature of the coolant ranges from 85°C to 110°C. The coolant usually consists of a 1:1 mixture of water and ethylene glycol, which is the most common antifreeze [6]. Owing to a lack a knowledge concerning the durability of polyamides and the critical requirements regarding reliability, a better understanding is needed about their performance during service at both high and low temperature and under various mechanical stresses.

PA-6 was also blended with other polymers as polyamide 6,6, polyamide 6,10, polycarbonate, polyethyleneterephtalate or ABS copolymers [7, 8].

A detailed analysis of the mechanical characteristics of polyamide 6 and PA-6 based materials must also take into account besides the effect of additives, the influence of testing parameters as temperature, speed of testing, loading time that are close to conditions which exist during the serivce life of the products [9–16].

Such a detailed study is presented in this paper for PA-6, talc-filled PA-6, glass–fiber reinforced PA-6 and a PA-6/polyetheleneterephthalate (PET) blend.

2. EXPERIMENTAL

2.1. Materials

The characteristics of the studied materials are given in Table I.

TABLE I Characteristics of the studied materials

No.	*Characteristic*	*PA-6-Relon P*	*PA-6-25%T*	*PA-6-30%GF*	90% *PA-6*/10%*PET*
1	Density (g/cm^3)	1.12	1.31	1.28	1.24
2	Melting interval (°C)	215–218	220–230	218–221	225–232
3	Relative viscosity at 20°C in H_2SO_4	2.6–3.0	–	2.25	–
4	Tensile strength (Kgf/cm^2) STAS 6642-73	717	670	1060	663
5	Elongation at break (%) STAS 6642-73	180–275	0	0	0–20
6	Flexural strength (Kgf/cm^2) STAS 5874-83	450–500	560–590	1500	700–750
7	Bending modulus (Kgf/cm^2) STAS 5874-83	16000–18000	25000	69000	23000
8	Notched Charpy impact resistance (Kgf·cm/cm^2) STAS 5801-90	12–27	12–14	10–11	**11–13**

Polyamide 6 (PA-6)-Relon P type with characteristics given in the Table I are produced by FIBREX – Savinesti-Romania. The polyethyleneterephthalate (PET) was supplied by TEROM-Fibers Factory-. Iaşi, Romania. PET had a $Tg = 60°C$ and an average crystallinity index of ~60%

A 25 wt% talc-filled PA-6 (PA-6 25%*T*) and a 30 wt% glass fibers reinforced PA-6 (PA-6 30% GF) have been obtained on a KO-BUSS MDK/40 compounding device using the conditions from the Table II.

We used untreated micronised talc (2 mμ size) supplied by "Synthesis" Oradea Chemical Work – Romania and short glass fibers with a diameter of 5 mμ.

The granulator was equipped with a knife acting under water flow with 2000 rpm.

TABLE II Temperatures for the compounding PA-6 with 25% talc, 30% glass fibers and 10% PET

No.	*Zone*	*Temperature (°C)*
1	Heating zone of screw	240
2	Feeding zone – comixer	250
3	Reinforcement zone	260
4	Extrusion zone	230
5	Die	260–280

The resulted granules had a length of 3 mm and a diameter of 1.5 mm. They were passed through sieves to remove the agglomerates and by a drying cyclone with air at 120°C so that, the residual humidity was maximum 0.1 wt%.

The mechanical behaviour of the two composites with PA-6 and PA-6/PET blend has been comparatively studied with that of PA-6 in various testing (service) conditions on the injection-molded testing bars obtained by injection procedure using a Kuassy 100/25 injection machine in the conditions presented in Table III.

2.2. Methods

Tensile and impact tests were performed on dry (as molded) samples.

Starting from the standard conditions metnioned in Table I for mechanical properties determination, the range of study was extended to various temperatures, (from –20 to 100°C), speed of testing

TABLE III Conditions for the obtainment by the injection-molded test bars

No.	*Injection parameters*	*Values*
1	Feeding zone temperature (°C)	230
2	Precompression zone temperature (°C)	240
3	Compression zone temperature (°C)	250
4	Mold temperature (°C)	80
5	Injection pressure (Mpa)	120
6	Injection speed (mm/s)	16
7	Injection time (s)	0.7–1

(from 2 to 50 mm/min) and long duration under load (more than 1000 h). The variation of tensile strength, elongation at break, modulus of elasticity, flexural strength and modulus, notched Charpy impact resistance and creep strain has been followed.

The tensile strength, elongation at break and modulus of elasticity were determined by means of a FP 10/1 HECKERT VEB THURINGER dynamometer, flexural strength and modulus on a KARL FRANK GmbH Mannheim instrument and for the impact resistance a CEAST TORINO apparatus was used.

All results presented are the average values for 10 determinations. Minimum and maximum values have been plotted to preserve the clarity of the diagrams and to obtain information on the risk limits.

3. RESULTS AND DISCUSSION

3.1. Short-term Mechanical Properties

3.1.1. Influence of the Temperature on Mechanical Behaviour

The testing of the temperature influence has been made at a constant tensile speed of 50 mm/min. in standard conditions according to STAS 9238-89.

For the four studied cases the tensile strength decreases with increasing temperature – Figure 1. The incorporation of talc decreases tensile strength values in respect to PA-6 and the values at 100°C is 50% lower than initial values.

It is easy to note that in spite of the fact that both PA-6 and PA-6-25%*T* exhibit the same tensile strength at low temperatures, the samples behave differently with increasing temperature. The difference

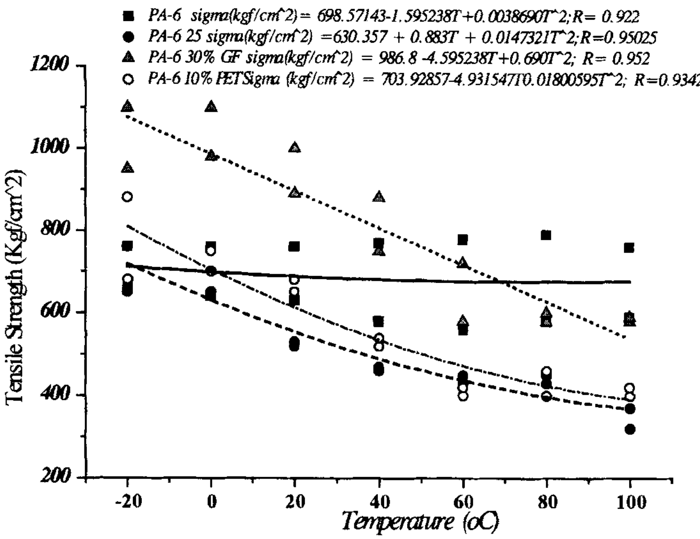

FIGURE 1 Tensile strength *versus* temperature.

between PA-6 and filled PA-6 consist in the data scatter. For PA-6 the scatter of data is greater at $T > 40°C$ and the decrease in the tensile strength at 100°C is only 16% from initial value.

The glass fibers reinforced PA-6 had 1.5 fold higher values of the tensile strength than PA-6 and the values begin to decrease only for $T > 20°C$ while at $T > 80°C$ the values seem to remain constant. This composite material can be used with satisfactory performance even at 100°C because the corresponding tensile strength at this temperature is approximately equal to that of PA-6 at 20°C.

The dependence of tensile strength on temperature follows mainly a parabolic law, regression coefficient being improved for the filled and reinforced PA-6.

The PA-6-10% PET exhibits superior values at the same temperature in respect to PA-6 and at $T > 60°C$, tensile strength remains approximately constant with a low scatter of the values.

As expected, the elongation at break varies in all three cases contrary to the variations in tensile strength, it increases with increasing temperature (Fig. 2), and the increase is logarithmic. Again, the scatter of data is higher for PA-6 and lower for filled and reinforced PA-6.

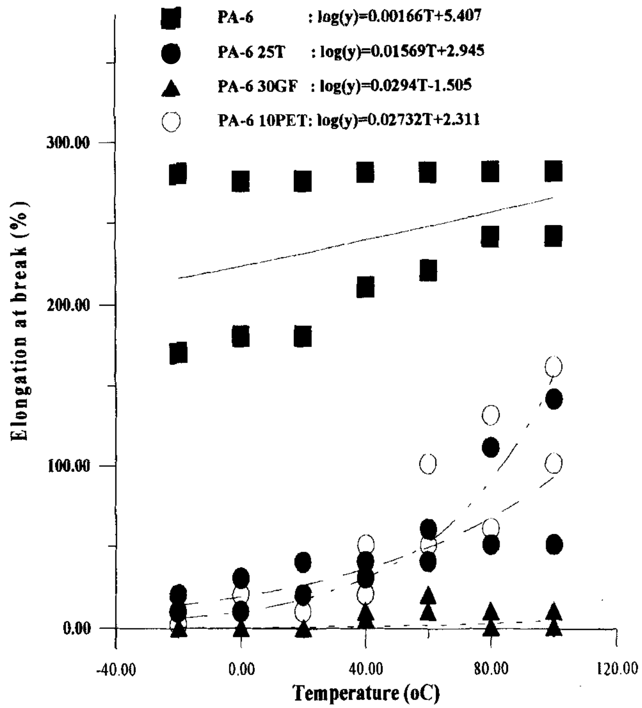

FIGURE 2 Elongation at break *versus* temperature.

The glass fibers reinforced PA-6 does not have an elastic component with the exception of the narrow range from 40°C to 60°C where the elogation at break increases up to 10–20%.

Only in the case of PA-6 having elongation at break above 100%, a variation in the modulus of elasticity with temperature can be studied (Fig. 3). In the –20 to 100°C temperature range the elastic modulus decreases by 50%.

It can be concluded that the talc decreases the temperature resistance of PA-6 while glass fibers increase it, but the elastic component is sacrificed.

The PA-6 10% PET blend exhibits a spectacular increase of the elongation at break having 10% elongation at break at –20°C and 130% at 100°C.

The variations of the flexural strength and modulus with temperature are illustrated in Figures 4 and 5 respectively.

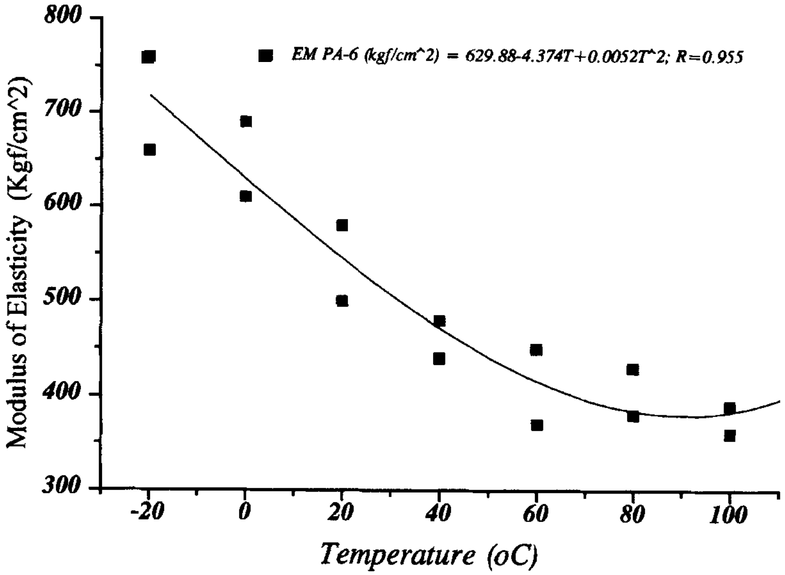

FIGURE 3 Modulus of elasticity of PA-6 *versus* temperature.

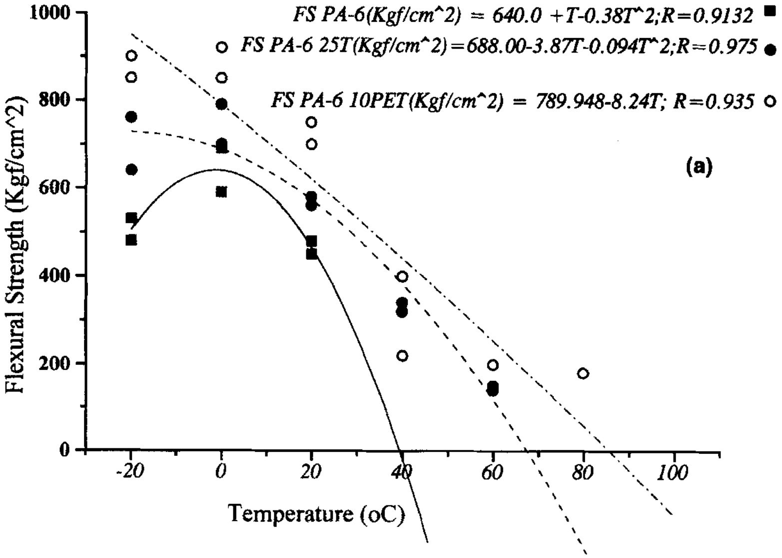

FIGURE 4 Flexural strength *versus* temperature for PA-6; PA-6 25%*T*; PA-6 10% PET (a) and PA-6 30% GF (b).

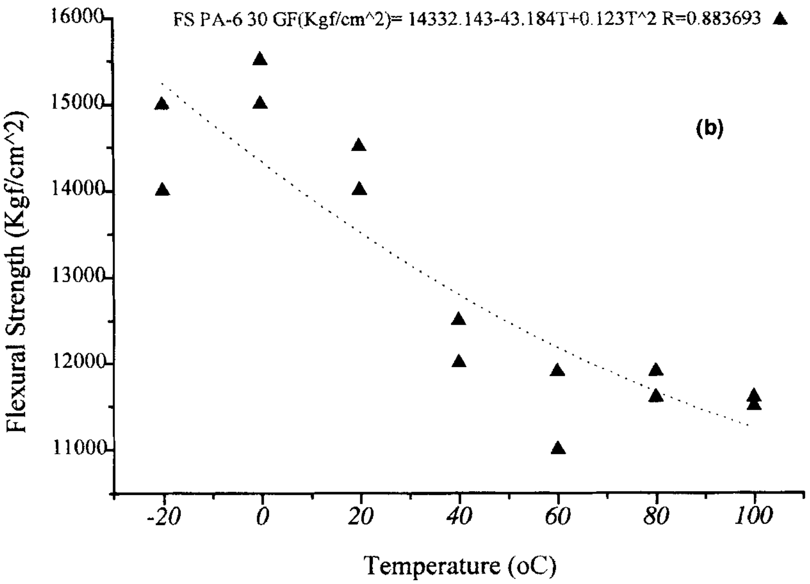

FIGURE 4 (Continued).

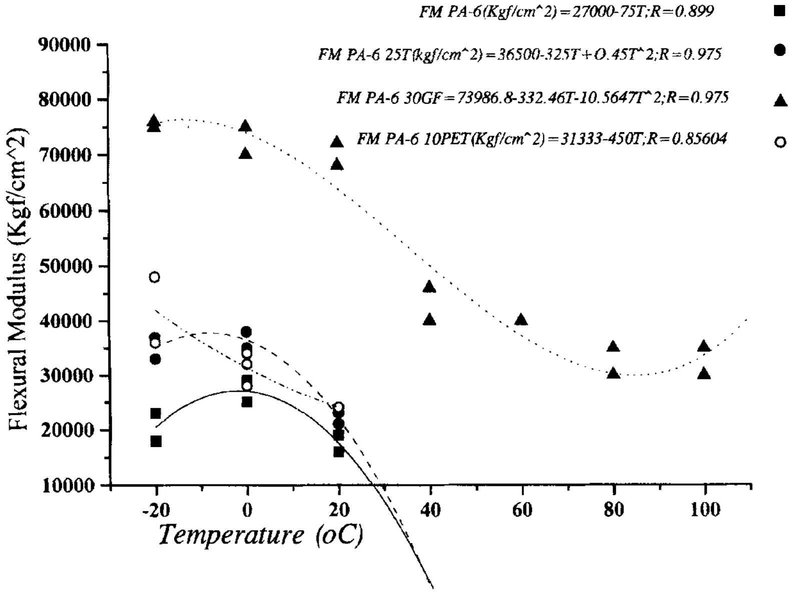

FIGURE 5 Flexural modulus *versus* temperature.

PA-6 and PA-6-25%*T* exhibit a parabolic dependence with the maximum at 0°C where the respective flexural strength is higher by 30% and 40% than the initial value. The values decrease drastically with the temperature increse, at $T > 20°C$ for PA-6 and $T > 50°C$ for PA-6-25%*T*, which can be considered temperature limits for the resistance of this materials.

At higher temperatures, the flexural strength can not be determined. The increase of flexural strength of PA-6-30% GF in a −20−0°C temperature interval is insignificant but, above this temperature the material presents a linear decrease with a plateau at $T > 60°C$.

Flexural modulus of the PA-6-30% GF is three times higher than that of PA-6, this composite having superior properties while for the PA-6-10% PET blend both flexural strength and modulus decrease with increasing temperature.

Notched Charpy impact resistance – Figure 6 – has been determined on notched specimens either with an impact of 1J or 4J depending on the behaviour of the tested material.

The scattering of the values is higher at low temperature and it decreases for $T > 40°C$. In the case of PA-6, Charpy impact resistance can be determined only below 40°C the increase being approximately of the exponential type. Above 40°C, PA-6 does not break. For the PA-6-25%*T* PA-6-30%GF and PA-6-10%PET the impact strength can be determined in the entire temperature interval, the talc filled PA-6 having higher values.

3.1.2. Influence of the Speed of Testing (or under Service Conditions)

The speed of testing below 50 mm/min (standard speed of testing according to STAS 9234-89) are usually met in the application of PA-6-based materials. Therefore, a dependence of mechanical properties on this parameter is important to be known.

The samples have a tensile strength at low speed of testing a little higher than that at 50 mm/min (Fig. 7). Generally, the values are close to the average values. All tensile strength values of the samples containing talc are below those of PA-6 while the glass fiber reinforced

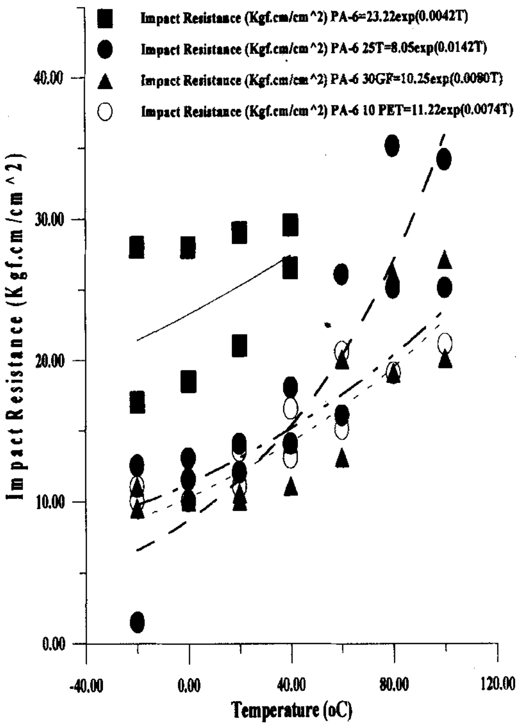

FIGURE 6 Charpy impact strength *versus* temperature.

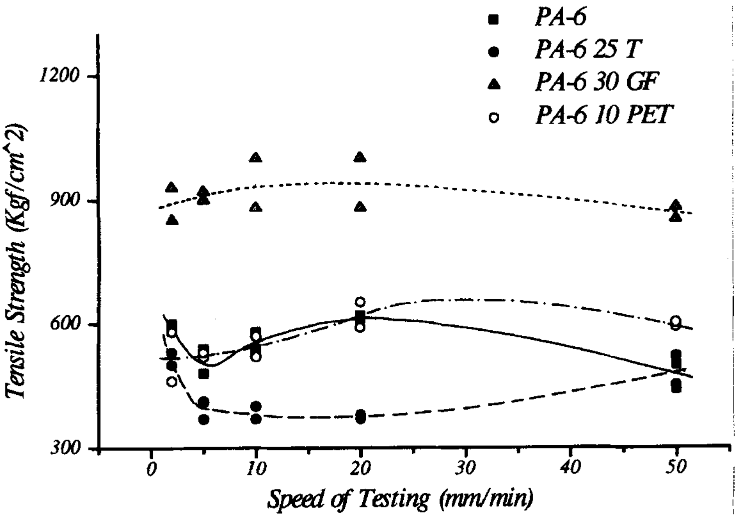

FIGURE 7 Tensile strength *versus* speed of testing.

PA-6 show higher tensile strength, the variation with composition being the same as in Figure 1.

The elongation at break (Fig. 8), exhibits a particular dependence on the speed of testing for each studied material. The PA-6 shows large scattering of points between 5 – 10 mm/min speed of testing, at higher values of speed of testing, the elongation at break remains approximately constant.

The variation of the elongation at break *versus* speed of testing for the PA-6-25%*T* shows a minimum corresponding to 10 mm/min where the tensile strength shows a twofold and elongation at break threefold decrease in respect to the values at both ends of studied range of speed of testing.

The elongation at break of the PA-6 30%GF, significantly increases with the speed of testing. The PA-6-10%PET blend exhibits a maximum of the tensile strength at 20 mm/min – Figure 7 and

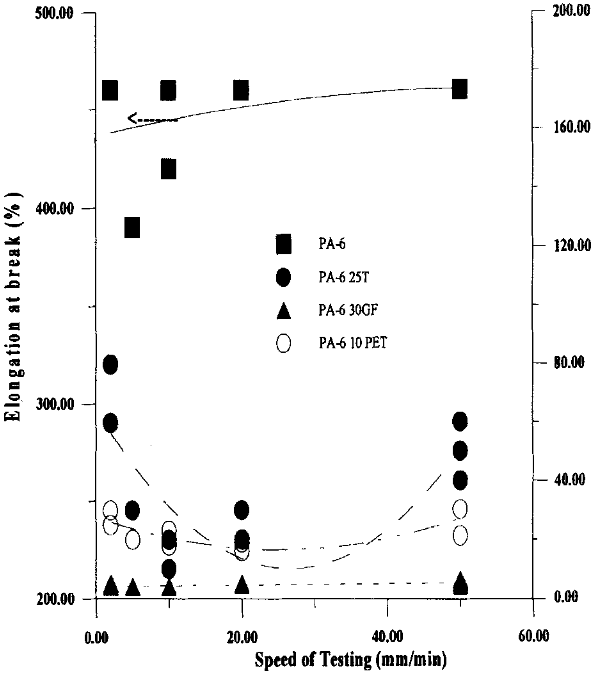

FIGURE 8 Elongation at break *versus* speed of testing.

approximately constant values of the elongation at break from 2 to 50 mm/min – Figure 8.

On the basis of these results, it can be concluded that with the exception of talc-filled PA-6, the maximum values for mechanical properties are found at a speed of testing of 20 mm/min.

It is well known that the dependence of the deformation speed is given by the equation [9]:

$$\sigma_2 = \sigma_1 + K\, lg\, \varepsilon_2/\varepsilon_1 \tag{1}$$

where σ_1 and σ_2 are the tensile strength values corresponding to the deformation speed ε_1 and respectively ε_2. The deformability factor K has the same units as the modulus of elasticity.

For the PA-6-based material studied, the K values obtained are given in Table IV.

The incorporation of 25% talc into PA-6, lowers the K value by a factor of seven, therefore, it has a negative influence on the elastic component of PA-6. This effect is much more pronounced with the 90%PA-6/10%PET blend.

The K values of PA-6 −30%GF are negative hence the elastic component did not exist and deformability of material results in deterioration of the structure.

TABLE IV Deformability factor-K for the studied polymeric systems

Material	$K = (\sigma_2 - \sigma_1)/lg\, \varepsilon_2/\varepsilon_1$ (kgf/cm^2)
PA-6	1446,5
	1399,1
	1365,7
PA-6-25%T	207,6
	197,2
	186,4
PA-6-30%GF	−586,4
	−325,3
	−696,2
PA-6-10%PET	5.25
	5.01
	4.71

3.2. Long Term Mechanical Behaviour – Creep Strain under Long-term Load

Creep strain under prolonged load simulates the service conditions of polymeric materials.

A measuer of the relative deformation of a solid under a constant load, temperature and humidity is the elongation at break $\varepsilon_t = (l_t - l_0)/l_0$. The other three important characteristics are:

(i) – creep speed k_c evaluated by relation:

$$k_c = (l_2 - l_1)/(t_2 - t_1)[\mathrm{cm.\ h^{-1}}] \qquad (2)$$

is the increase of relative elongation ($\varepsilon = \Delta l/l_0 \cdot 100$) in a certain time interval;

(ii) – creep index, measuring the increase of relative elongation in a certain time interval (in percentages):

$$K'_c = k_c(t_2 - t_1)/l_1 \cdot 100 = (l_2 - l_1)/l_1 \cdot 100[\%] \qquad (3)$$

(iii) – limit of creep resistance or normal yield stress that produces breakdown of the specimen or relative elongation for a certain duration of testing where l_1 and l_2 are the lengths of the specimen at time t_1 and t_2 of the experiment; and l_0–initial length specimen.

The longest duration of creep strain experiment was 42 days (or 1008 h).

The creep strain determination were performed on an apparatus provided with 10 testing positions conforming to the standard ISO 899-81. One of them is schematically presented in Figure 9.

The creep strain measurements have been made at constant temperature and humidity conditions (STAS 5794-86) on 10 testing specimens for each material studied. The loads applied listed in Table V were 10 to 50% of the tensile strength at break presented in Table I.

The creep diagrams – Figure 10 – give useful information on the applications of each material under long-term load. Another objective of creep tests was the determination the load corresponding to a

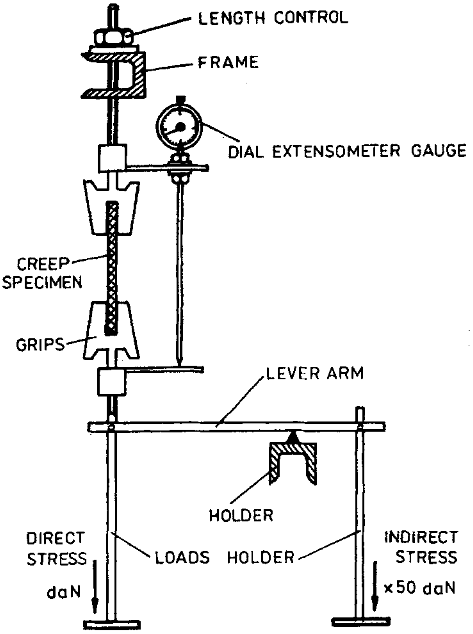

FIGURE 9 Scheme of one position of measurement of creep strain long-term load diagram.

TABLE V Loads applied for creep strain measurements

Nominal tensile stress *(kgf/cm^2)*	*PA*-6	*PA*-6-25%*T*	*PA*-6-30%*GF*	*PA*-6-10%*PET*
Tensile strength at break σ-(kg/cm^2)	717	670	1060	663
Load applied 10% of σ	71,7	67	106	66.3
Load applied 20% of σ	143,1	134	212	132.6
Load applied 30% of σ	–	201	318	198.9
Load applied 50% of σ	–	335	–	–

minimum strain of 1% which means a very good dimensional stability for a long period of time.

PA-6 has been tested at two values of stress load amounting to 10 and 20% of the tensile strength at break. In the first case, the normal strain (Fig. 10) increases linearly during 300 h of testing than the creep strain remains constant of 1%. If the stress load is twice higher (20% of σ), normal strain increase logarithmic up to a limit of 5% of strain.

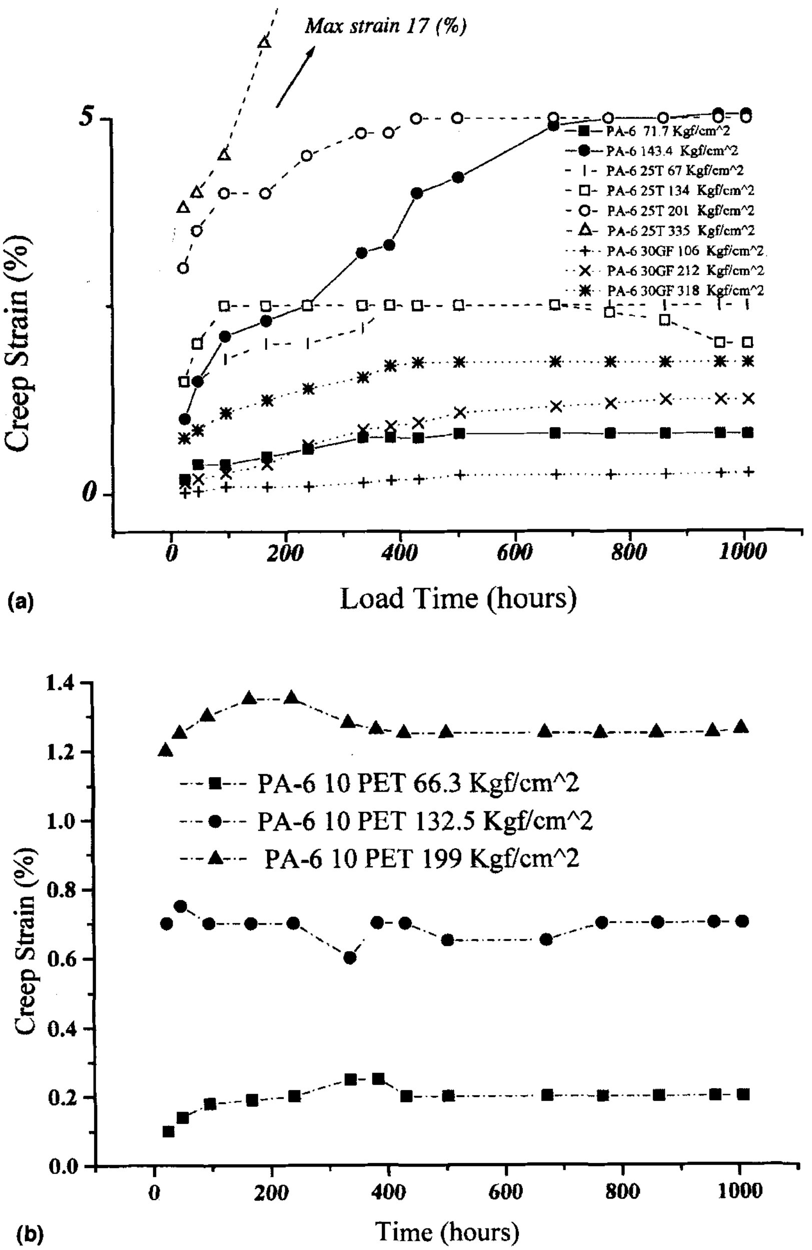

FIGURE 10 Creep strain *versus* testing time diagram for PA-6; PA-6 25%*T*; PA-6 30%GF (a) and PA-6 10%PET (b).

It can be appreciated that PA-6 are useful only for applications requiring working under load below 10% of σ for a strain under 1%, that means a nominal tensile stress of 72 Kgf/cm^2 for 1000 h.

Incorporation of 25% talc in PA-6 had a negative effect on the creep strain behaviour under load for long time duration even at very small loads. For 10 and 20% of tensile strength at break σ, normal strain is already 1% after 300 h testing while for 1000 h, the normal strain is 2% and respectively 2.5%. At higher loads of 30% σ and 50% σ, the strain of 1% is reached in only 24 h of testing time and after 1000 h the strains reach 5 and 18% respectively.

The PA-6-25% *T* exhibits irreversible "cold flow" strain under loads higher than 10% σ (67 Kgf/cm^2).

The PA-5-30% GF composite has a very good creep resistance – Figure 10. For a load of 10% σ, (106 Kgf/cm^2) the nominal strain is 0.3% after 1000 h of testing, while for a double load – 20% σ (212 Kgf/cm^2) the strain reached 1% after 500 h of testing and remained constant at 1.2% for the entire duration of testing up to 1000 h. The strain is 1% even if the load is threefold higher (318 Kgf/cm^2) during 96 h testing and increase at 1.5% at 300 h and respectively 1.75% after 1000 h. The "cold flow" takes place between 100 and 300 h and remains approximately constant up to 1000 h before crack formation.

The maximum load accepted by this composite for 1% strain is 212 Kgf/cm^2, which is threetimes higher than that of PA-6.

It seems that the dependence of normal strain on time is a logarithmic one for all three materials – Figure 11.

The glass fiber reinforcement increases the mechanical performance of PA-6. The enhanced performance of reinforced-PA-6 is achieved by transferring applied stresses from the matrix to the supporting fibers.

The PA-6-10%PET exhibits in the entire studied time interval a very low creep strain, so this material is able to endure for more than 1000 h under 132.6 kgf/cm^2 load – Figure 10b. This behaviour is similar with that of other PET-containing blends [17].

In order to evaluate the precise load to obtain a certain strain, isochronous stress–strain curves are plotted in Figure 12. These curves are useful to graphically establish the limit values of constant tensile stress or the creep resistance of materials for a certain acceptable deformation.

The obtained data allow us to compare different materials.

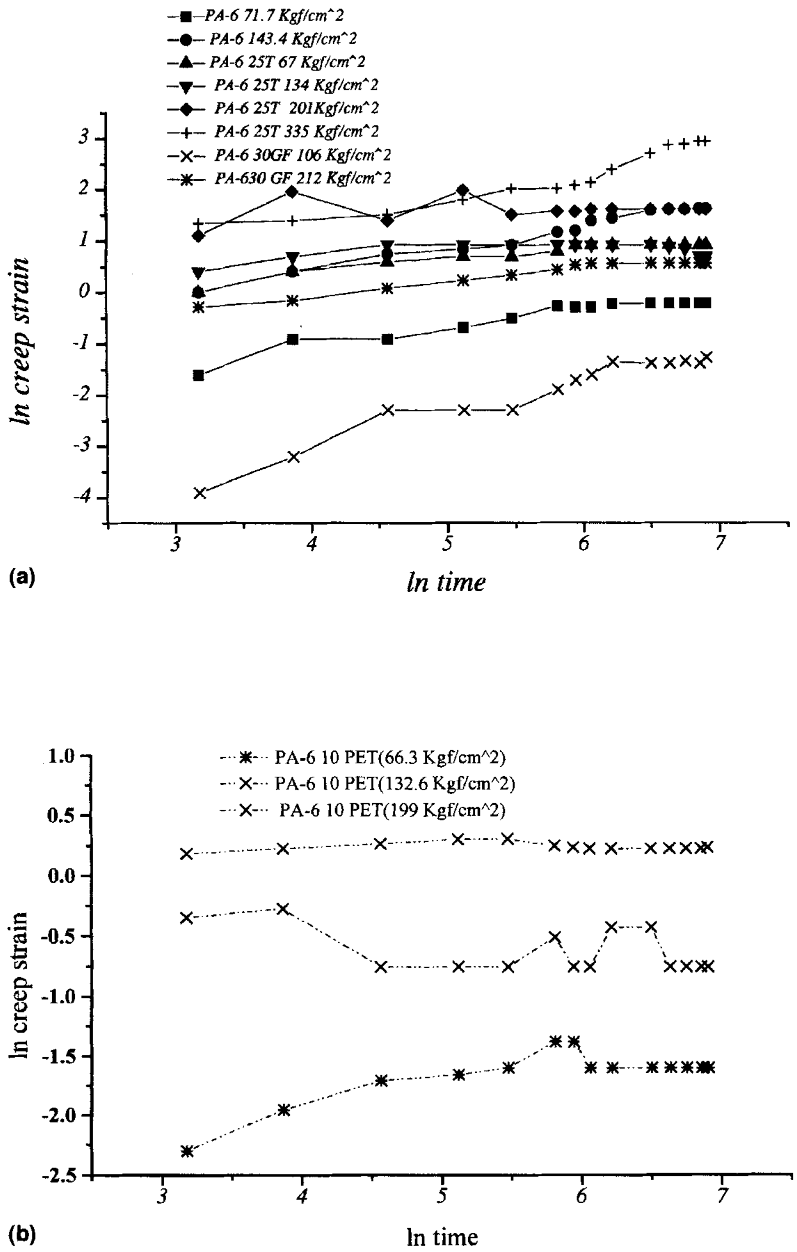

FIGURE 11 Logarithmic diagrams of creep strain for PA-6; PA-6 25%*T*; PA-6 30%GF (a) and PA-6 10%PET (b).

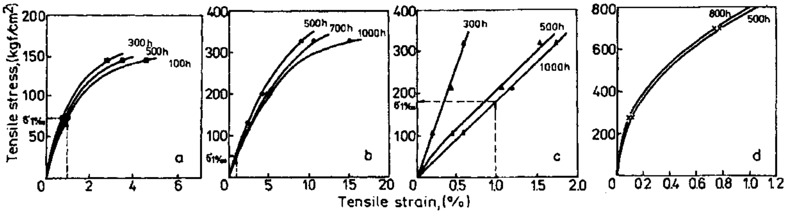

FIGURE 12 Isochronous stress–strain curves for PA-6(a); PA-6 25%*T* (b); PA-6 30%GF(c) and PA-6-10%PET(d).

CONCLUSIONS

A set of data necessary for design of various molds and parts have been obtained.

The comparative mechanical behaviour of four different materials: PA-6, talcum filled PA-6, glass fiber reinforced PA-5 and PA-6-10%PET under heating and long-term mechanical stresses permit the selection of the appropriate material for various service conditions.

On the basis of these data it is possible to predict with a reasonable degree of accurance the variation of mechanical properties under temperature, deformation speed loading.

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