Study of Viscoelastik Behavior of Polyethylene and Mixture of Polyethylene Depending on the Degree of Crystallinity

Polietilen ve Polietilen Karışımının Kristalinite Derecesine Bağlı Olarak Viskoelastik Davranışının İncelenmesi

Salâhaddin ANIK *)
Selma AKKURT **)

SUMMARY

In this study viscoelastic behavior of polyethylene with regard to the degree of srystallinity was investigated in view of statio tension and creep, shapes of tension diagrams as well as variations occuring in mechanical properties were explained within the framework of a certain theory and a relation between the tensile modulus of elasticity and the degree of crystallinity was found.

Furthermore, depending on creep tests performed under different loads and temperatures, definite results were obtained about the creep behavior of polyethylene, creep equations were determined in view of these results for polyethylene having high and low densities and creep modulus of elasticity was studied with regard to load, temperature and time. In addition, viscoelastic behavior of the mixture of polyethylene, being obtained from two kinds of polyethylene with different degrees of crystallinity was investigated within the framework of the above stated points.

ÖZET

Bu çalışmada, statik çekme ve sürünme bakımından polietilenin kristalinite derecesine bağlı olarak viskoelastik davranışı incelenmiş, çekme diyagramlarının şekilleri ile mekanik değerlerde meydana gelen farklılıklar belirli bir teori çerçevesinde izah edilmiş ve çekme elastiklik modülü ile kristalinite derecesi arasında bir bağıntı kurulmuştur.

*) Faculty of Mechanical Engineering, Technical University of Istanbul, Prof.
**) Faculty of Mechanical Engineering, Technical University of Istanbul, Dr.

Ayrıca çeşitli yüklerde ve sıcaklıklarda yapılan sürünme deneylerine dayanarak, polietilenin sürünme davranışı hakkında belirli sonuçlara varılmış, bu sonuçlara göre yüksek yoğunluk polietileni ve alçak yoğunluk polietileni için sürünme denklemleri tayin edilmiş ve yüke, sıcaklığa ve zamana bağlı olarak sürünme elastiklik modülü incelenmiştir. Bunun yanısıra, farklı kristalinite sahip olan iki çeşit polietilenden elde edilen karışımın, yukarıda belirtilen hususlar çerçevesinde viskoelastik davranışı incelenmiştir.

1. — INTRODUCTION

In investigations that have been conducted to date, viscoelastic behavior of polyethylene has been mainly investigated in view of relaxation modulus of elasticity (1), viscosity and molecular weight (2), changes occuring in crystalline region of structure (3) or dependence on creep regardless of structure (4). In this study, viscoelastic behavior of polyethylene was investigated in view of static tension and creep with regard to the degree of crystallinity. Besides, viscoelastic behavior of the mixture, obtained from two kinds of polyethylene, having different degrees of crystallinity, was studied under the same conditions.

2. — THEORETICAL CONSIDERATIONS

A polymer (5) is a long - chain molecules constructed from many smaller structural units called monomers, covalently bonded together in any conceivable pattern. Chemically polymers may be linear, branchad and crosslinked. Besides, there are various arrangement kinds of units forming chains that are called stereoregularity and are described in configuration and conformation terms. The term configuration is reserved in polymer science to refer to the arrangement of units along the axis of the chain. Bonding kind of the units may be head to tail, head to head and tail to tail or random and arrangement kind isotactic, syndiotactic and atactic. The bonds connecting the repeat units of a polymer chain are generally flexible enough to permit a degree of rotational freedom about the bonds. This freedom of rotation allows the molecule to assume any of a number of conformations· The limiting conformations are the random coil and the full extended chain. Polymeric masses can be built of either coiled, nearly independent molecules (amorphous polymers) or of weel - alligned chains that bond with each other and exhibit crystaline order. If a number of similar isotactic or syndiotactic chains can simultaneously become extended and aligned, it is possible for van

der Waals or hydrogen bonds to form between the structural units of adjacent chains and a state of crystalline order is thus constructed. However, a polymers is rarely completely crystalline, generally the polymers are a mixture of crystalline and amorphous regions. These structures are called spherulites. Examination of thin sections of semicrystalline polymers reveals that the crystallites themselves are not arranged randomly, but form regular birefringent strucis called the degree of crystallinity. The general mechanical properties of a polymer are strongly affected when crystallites are present. For this reason the mostuseful and practical concepts in the characterization of semicrystalline polymers is the degree of crystallinity.

3. — EXPERIMENTS AND EXPERIMENTAL DATA

3.1.0. — Experimental materials and conditions

As a material for test specimen, high density polyethylene (HDPE) having a linear structure and low density polyethylene (LDPE) having a branched structure was used. Raw materials for both test specimens were obtained from Petkim Petrokimya A.Ş. and were molded by Plastaş A.Ş. Besides these two materials a third material called 0,5 KPE and composed of 50 % HDPE and 50 % LDPE as a result of molding was used in order to study viscoelastic behavior of the mixture of polyethylene This material is essentially a physical mixture.

First of all, in special prepared molds test specimens were obtained dimension of which are shown in Figure 1 a· Test specimens were manufactured according to ASTM D 638 standard for tension tests and with a very slight change according to ASTM D 674 standard for creep tests. Tension tests were conducted on Instron test machine which automatically recorded force - strain diagrams and creep tests were performed on a test machine for plastic materials. A furnace equiped with a thermostat that kept a constant temperature with a tolerance of $\pm 0,1$ C was used for temperature tests. Two different speeds, namely $v_1 = 2$ mm/min and $v_2 = 20$ mm/min were employed in tension tests. Considering principles of statistics and propability every test was repeated using at least five test specimens. Creep tests were performed using temperature levels of $T = 20°C$, $T = 50°C$ and $T = 80°C$. Tests were performed for every temperature level at five constant stresses on the average and every test was repeated using three test specimens. Duration of tests was limited to 1000 hours at most.

Principles of statistics and probability (6) were applied both in conducting tests and evaluating test results. The test model was set up,
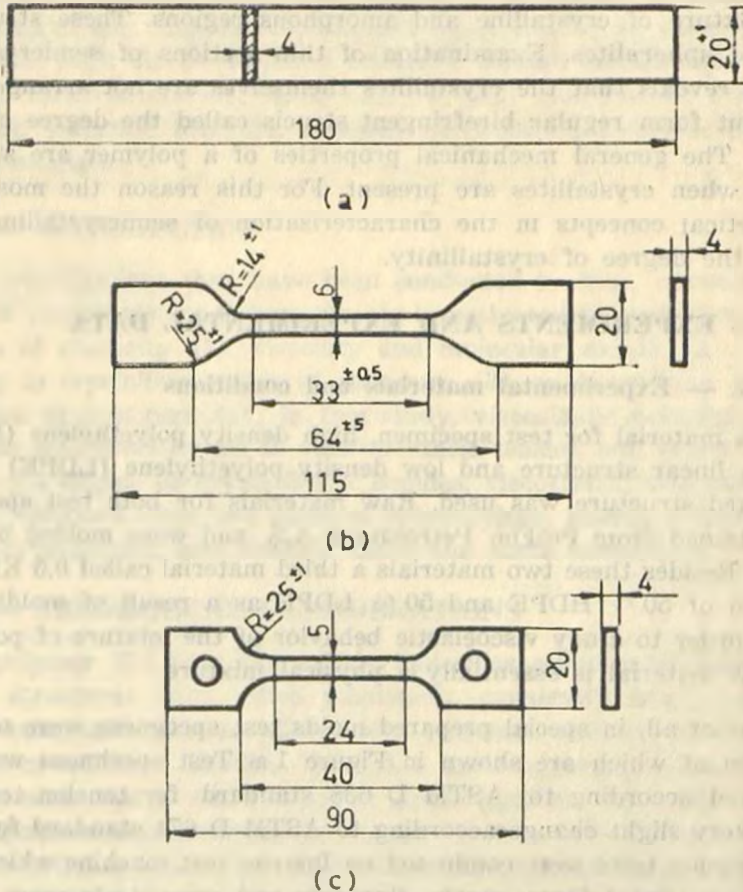

Fig. 1. — Test specimen.
(a)   As molded specimen
(b)   Tension test specimen
(c)   Creep test specimen

tests were planned and test results were evaluated with respect to these principles. The obtained results were represented diagrammetically by the method of least squares and analytical expressions for the drawn curves were determined by the method of regression. Computations were carried out according to a prepared program to be used in the computer.

Here the important problem is to determine the degree of crystallinity of the material used in tests. From the comparison of present analytical and axperimental methods (Figure 2) the relation to determine the degree of crystallinity of polyethylene was found; namely (7),
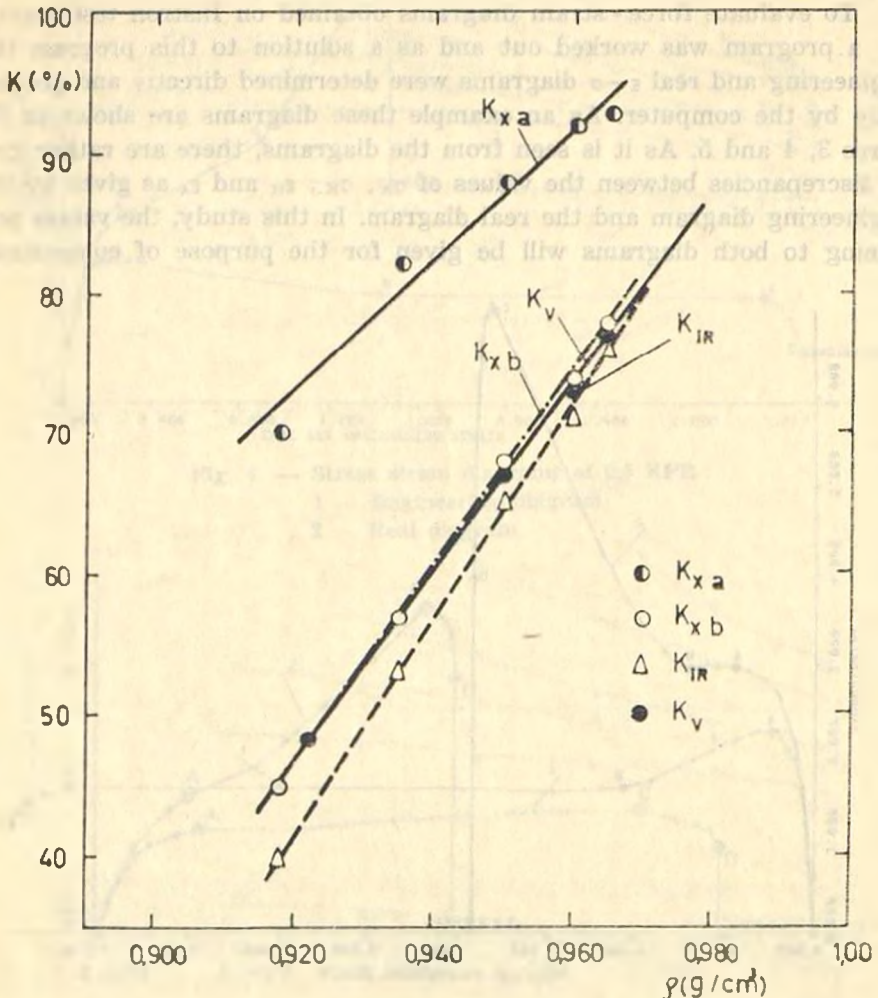
$$K_v = -5,67 + 6,67 \, \rho \qquad\qquad (1)$$

Fig. 2. — Comparison of experimental and analitical metods to determine the degree of crystallinity (8) :

    Kxa X - ray method according to Matthews;

    Kxb X - ray method according to Hendus;

    K_{IR} Infrared Absorption;

    K_v According to the relation (1):

which is an a good agreement. (ρ - density of the polymer). According to this relation the degree of crystallinity of the tested HDPE was determined as $K_v = 73{,}32 \%$ and the degree of crystallinity of the tested LDPE was determined as $K_v = 48 \%$.

3.2.0. — Experimental data

To evaluate force - strain diagrams obtained on Instron test machine, a program was worked out and as a solution to this program the engineering and real $\varepsilon - \sigma$ diagrams were determined directly and graphically by the computer. As an example these diagrams are shown in Figures 3, 4 and 5. As it is seen from the diagrams, there are rather great discrepancies between the values of σ_A, σ_K, ε_K and ε_A as given by the engineering diagram and the real diagram. In this study, the values pertaining to both diagrams will be given for the purpose of comparison,
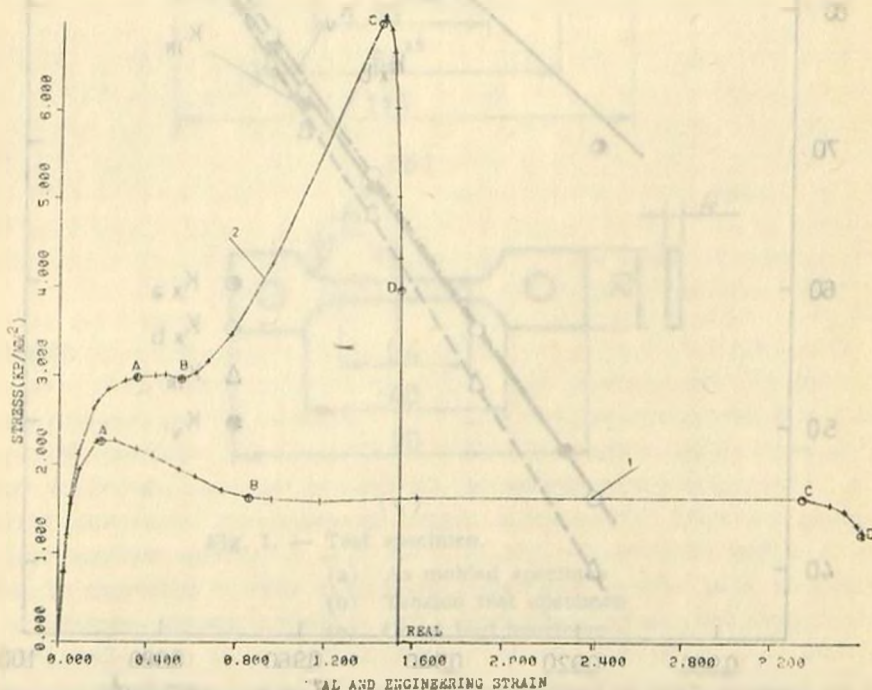

Fig. 3. — Stress - strain diagrams of polyethylene having a high degree of crystallinity.
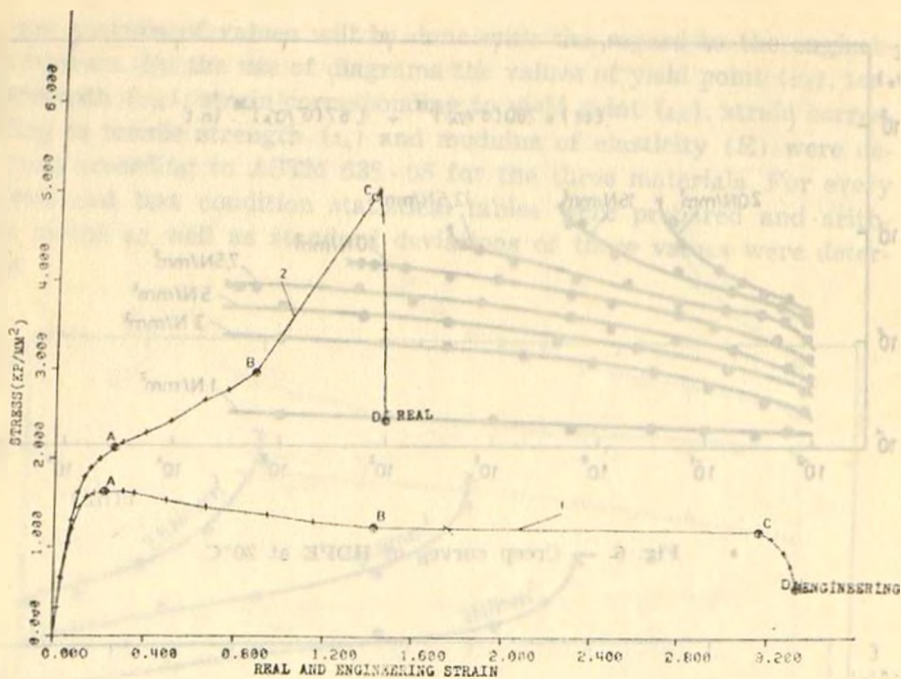1 — Engineering diagram;
2 — Real diagram.

Fig. 4. — Stress strain diagrams of 0.5 KPE :
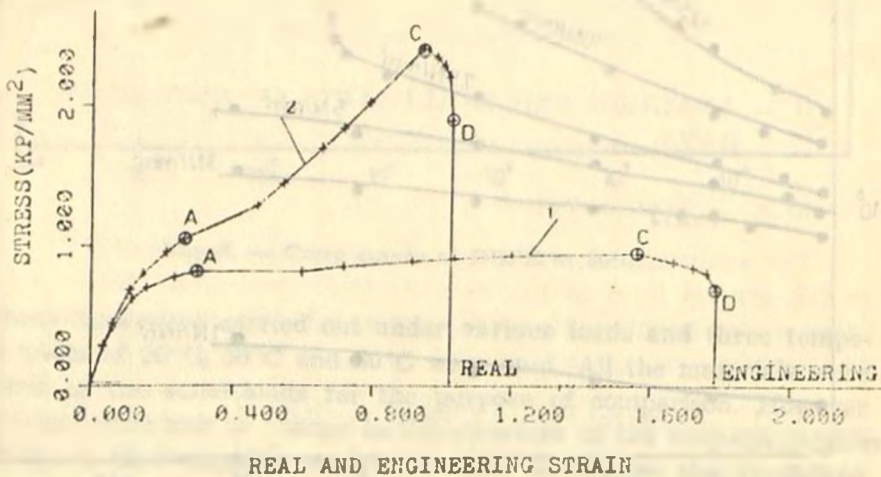   1 — Engineering diagram;
   2 — Real diagram.

REAL AND ENGINEERING STRAIN

Fig. 5. — Stress - strain diagrams of polyethylene having a low degree of
   crystallinity :
   1 — Engineering diagram;
   2 — Real diagram.

$$\varepsilon(t) = 7{,}80(\sigma/\sigma_A)^{1{,}18} + 1{,}67(\sigma/\sigma_A)^{1{,}29} \cdot \ln t$$
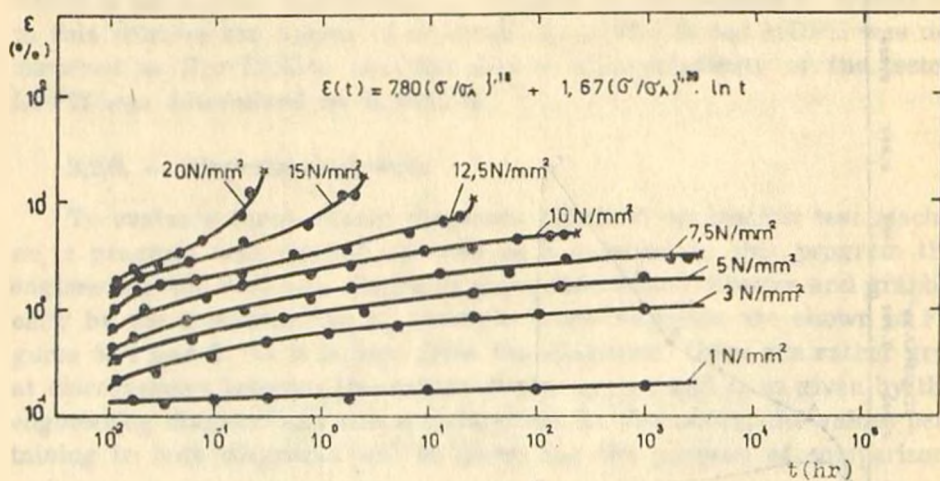
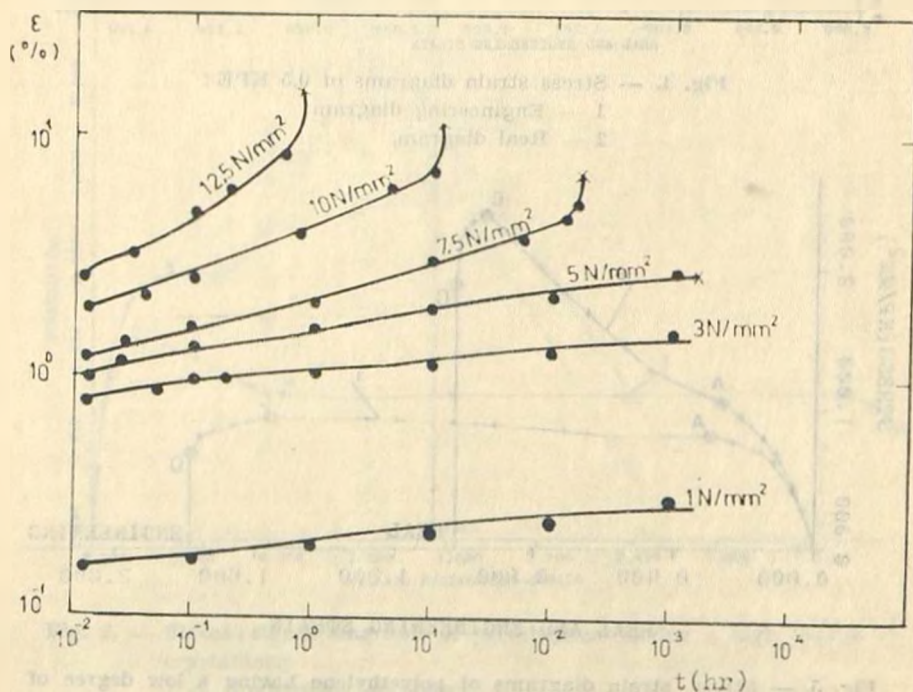Fig. 6. — Creep curves of HDPE at 20°C.

Fig. 7. — Creep curves of HDPE at 50°C.

but the analysis of values will be done with the regard to the engineering diagram. By the use of diagrams the values of yield point (σ_A), tensile strength (σ_K), strain corresponding to yield point (ε_K), strain corresponding to tensile strength (ε_A) and modulus of elasticity (E) were determined according to ASTM 638 - 68 for the three materials For every material and test condition statistical tables were prepared and arithmetic means as well as standard deviations of these values were determined.
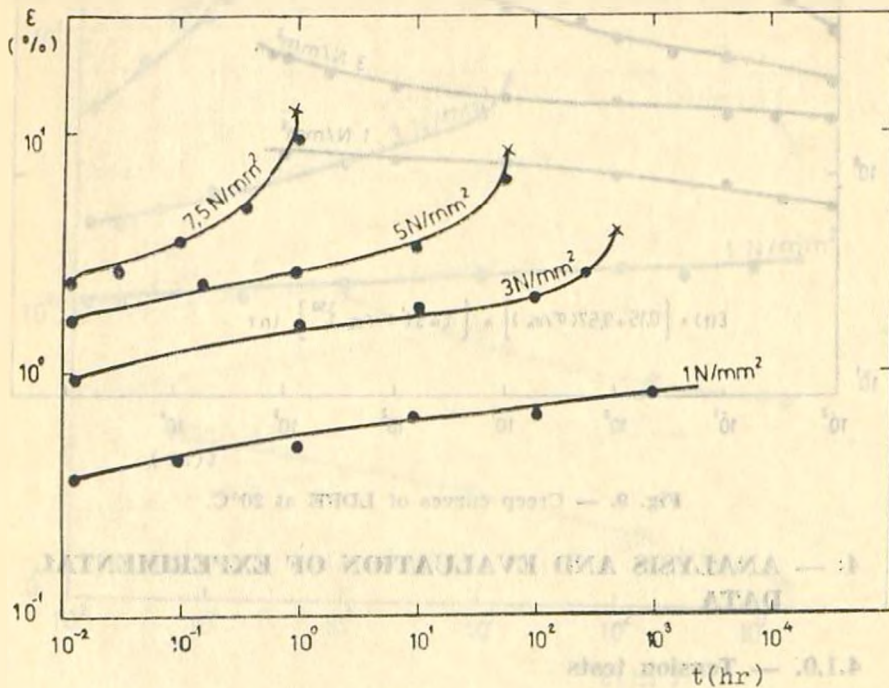

Fig. 8. — Creep curves of HDPE at 80°C.

Creep tests were carried out under various loads and three temperature levels of 20°C, 50°C and 80°C were used. All the materials were subjekted to the same loads for the purpose of comparison. However since temperature rise or change in the structure of the material causes a decrease in load carrying capacity, all the loads were not applied to the materials. So the only material that sustained all the loads at the temperature of 20 C was HDPE. Obtaining results this way they were treated according to statistical principles and then they were presented

diagrammatically. Creep diagrams are given in Figure 6, Figure 7, Figure 8 pertaining to HDPE; in Figure 9, Figure 10, Figure 11 pertaining to LDPE and in Figure 12, Figure 13, Figure 14 pertaining to 0,5 KPE.
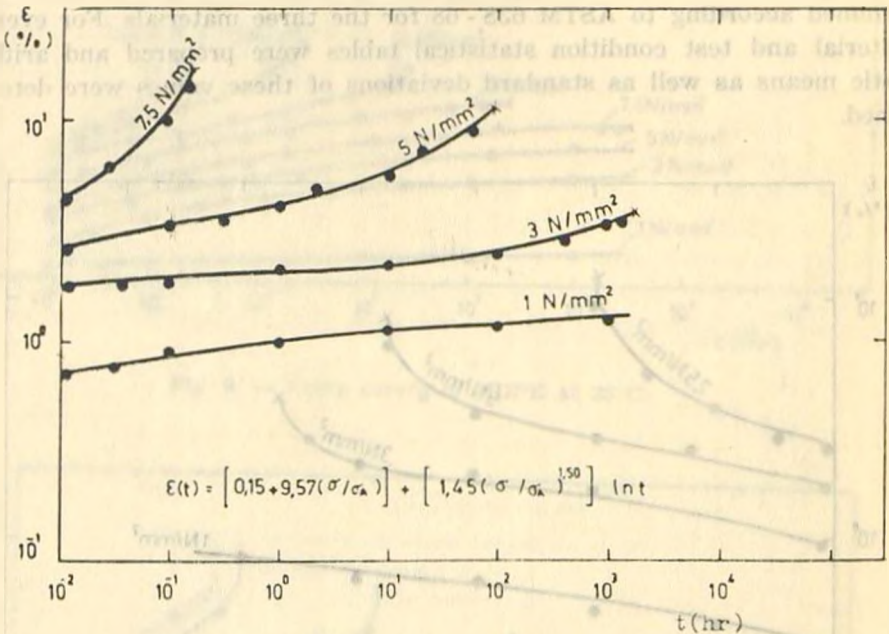

$$\epsilon(t) = \left[0{,}15 + 9{,}57(\sigma/\sigma_A) \right] + \left[1{,}45(\sigma/\sigma_A)^{1{,}50} \right] \ln t$$

Fig. 9. — Creep curves of LDPE at 20°C.

4 — ANALYSIS AND EVALUATION OF EXPERIMENTAL DATA

4.1.0. — Tension tests

The analysis and evaluation of test results will be done in respect to the general form of the $\sigma - \epsilon$ diagram, yield point, tensile strength, strains corresponding to yield point and tensile strength and modulus of elasticity.

4.1.1. — General form of the $\sigma - \epsilon$ diagram

The upper (figure 3) and lower yield points and the horizontal region up to the tensile strength at break are seen in the engineering diagram in tension for the HDPE that has a high degree of crystallinity. It was seen that at the upper yield point a neck was developed in the

test specimen and as the tension proceeded, the neck spreand along the
length of the test specimen. It was also observed that the deformations
produced during this period were rather uniform. At a certain magni-
tude of strain, the test specimen contracts in one or two places and fi-
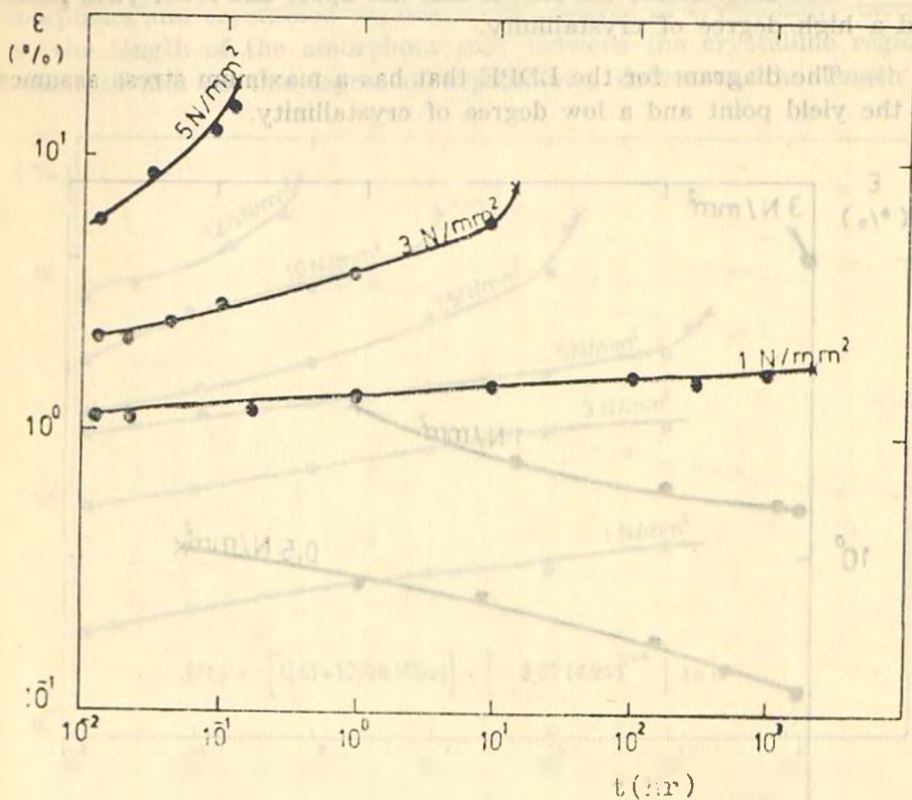nally it fractures at one of the contractions.

Fig. 10. — Creep curves of LDPE at 50 C.

Studying the $\sigma-\varepsilon$ diagram (Figure 5) for the LDPE which has a
low degree of crystallinity it is seen that the stresses remain constant
after a maximum is reached and the strains occur in a uniform manner.
A neck was not developed as was in the case of the HDPE at the point
A corresponding to the yield point. However, at a certain strain, a cont-
raction takes place and a fracture occurs at this point.

The $\sigma-\varepsilon$ diagram (Figure 4) for the 0.5 KPE exhibits a rather in-
teresting aspect. After the stresses reach a maximum, there is a slight

drop and then a horizontal region begins where the stresses are constant.

Consequently, there are two kinds of the $\sigma - \varepsilon$ diagram in relation to the degree of crystallinity of polyethylene.

— The diagram for the HDPE that has upper and lower yield points and a high degree of crystallinity.

— The diagram for the LDPE that has a maximum stress assumed as the yield point and a low degree of crystallinity.
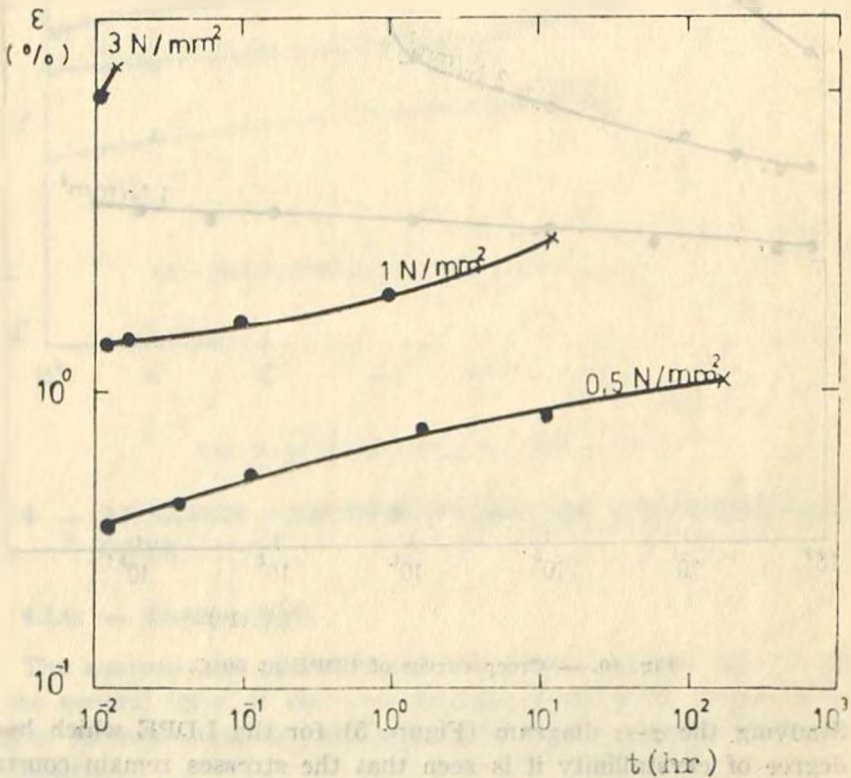

Fig. 11. — Creep curves of LDPE at 80°C.

This behavior of polyethylene that has a semicraystalline structure is attributed by some researchers (9) to the orientation of the original semicrystalline structure with respect to the direction of tension while others (10) maintain that the spherulites being the most important

morphological unit of the semicrystalline polymer deform into an ellipsoidal shape. Taking a base these theories, the different behavior of the semicrystalline polymer in tension can be explained with respect to the degree of crystallinity as follows. It will be done taking into account that one of the chains of the semicrystalline structure is composed of amorphous and crystalline regions As the degree of crystallinity increases, the length of the amorphous part between the crystalline regions decreasses and as the degree of crystallinity decreases, the length of
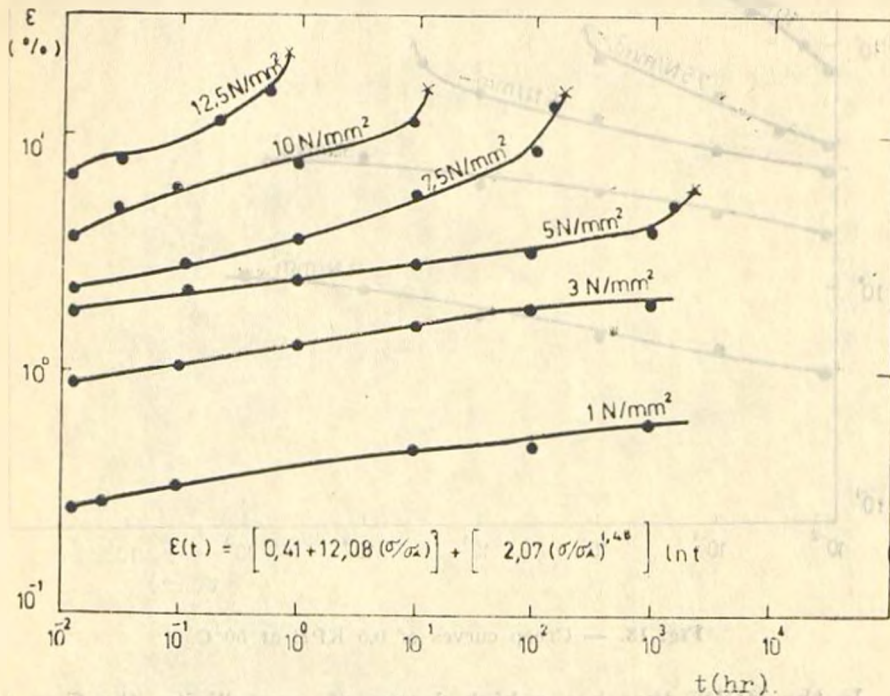

Fig. 12. — Creep curves of 0,5 KPE at 20°C.

the amorphous part increases. According to this structure, the first deformation starts at the amorphous part where the chains are softer. This is followed by the crystalline regions where the chains are bonded. As the tensile load is increased, the structural change occurs in the crystalline regions as is decribed above. The most important feature of this structural change is the orientation of the structure with respect to the direction of tension. The orientation continues up to the yield point and at this point the possibility of the orientation ceases. During the

orientation some micro - cavities are formed in the structure of the material and the most explicit matter of this fact on the macroscopic level is the fading of the color of the material. However the phenomena stated above happen in a different manner for the HDPE and LDPE with respect to the degree of crystallinity.
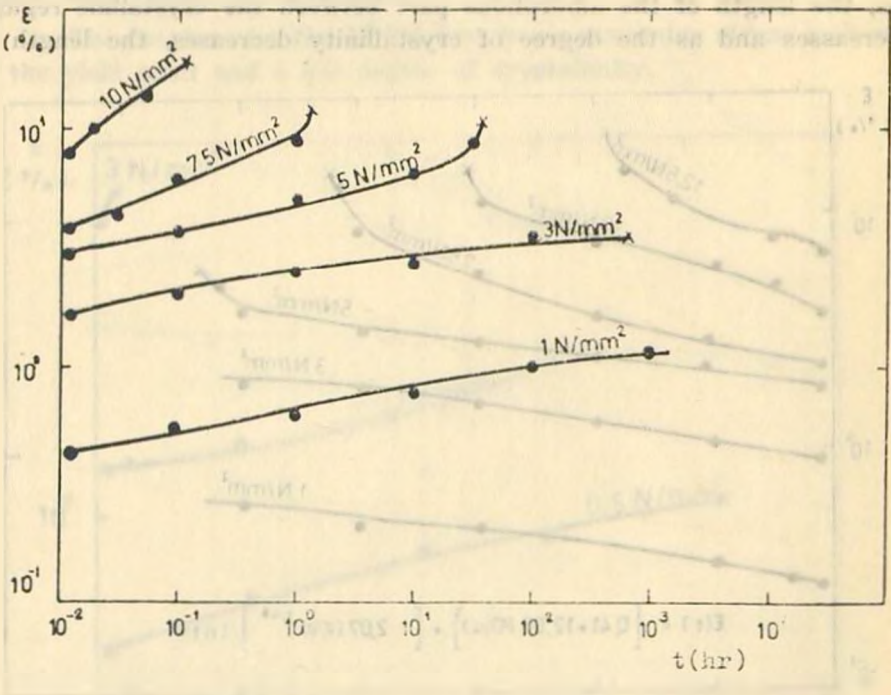

Fig. 13. — Creep curves of 0,5 KPE at 50°C.

In the HDPE that has a high degree of crystallinity, the first deformations of the amorphous parts last very little and immediately the crystalline parts begin to change. Therefore, it is controlled by the crystalline regions including the first deformations. If it is assumed that the orientation is the resistance against the axternal forces, the cessation of the orientation will correspond to the maximum tensile force, in other words, the upper yield point. As it is claimed by Kramer (11) the crystalline units start to deteriorate locally that produce softness in the material and cause a neck develope. This phenomenon reduces the resistance of the material and causes the lower yield point to be produced.

In the LDPE that has a low degree of crystallinity, the first defor-

mations start at the amorphous part and they continue for a while in this way. When the structural changes start in the crystalline parts, the orientation is not finished in the amorphous parts. Thus the phenomena up to the yield point are controlled by the amorphous and also by the crystalline regions.

Consequently, the behavior of polyethylene in tension is controlled by the crystalline regions having a high degree of crystallinity and by the amorphous and also by the crystalline regions having a low degree of crystallinity.
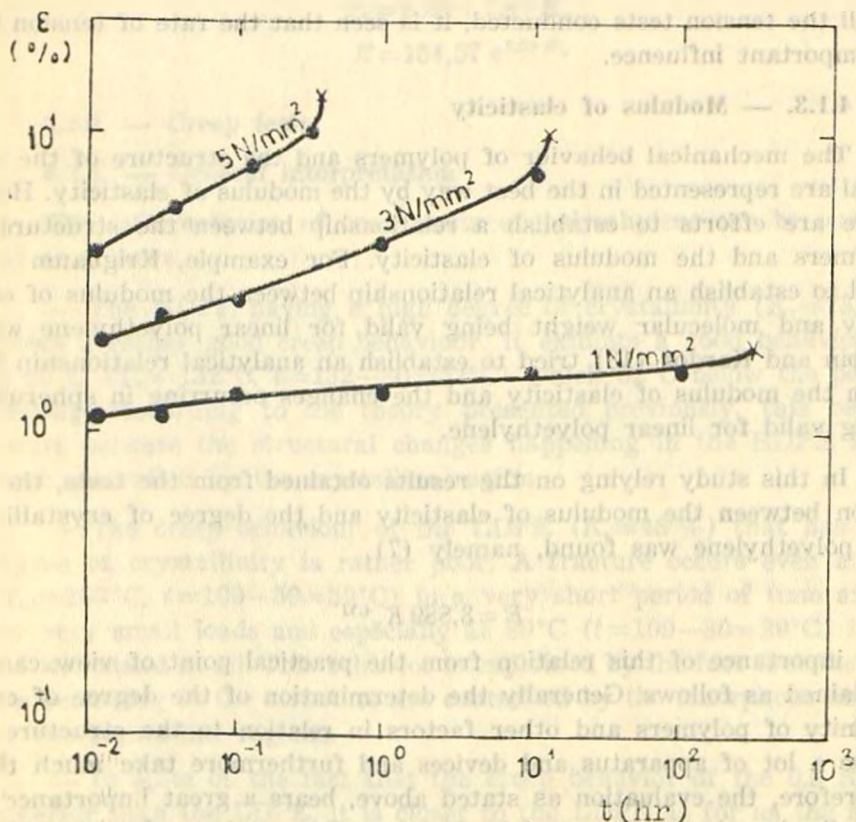

Fig. 14. — Creep curves of 0,5 KPE at 80°C.

4.1.2. — Strains corresponding to yield point and tensile strength

According to this theory the mechanical behavior of the semicrys-talline polymers depends mainly upon the change of the crystalline re-

gions. Since the changes represent the resistance against the external forces, the factors such as σ_A, σ_K and E will attain higher magnitudes in polymers having a higher degree of crystallinity. Thus, according to the results obtained from the tests, the following relations were estaslished, namely

$$\sigma_{A\ HDPE} \approx 2{,}78\ \sigma_{A\ LDPE} \tag{2}$$

$$\sigma_{K\ HDPE} \approx 1{,}91\ \sigma_{K\ LDPE} \tag{3}$$

$$E_{HDPE} \approx 6{,}20\ E_{LDPE} \tag{4}$$

In all the tension tests conducted, it is seen that the rate of tension has an important influence.

4.1.3. — Modulus of elasticity

The mechanical behavior of polymers and the structure of the material are represented in the best way by the modulus of elasticity. Hence there are efforts to establish a relationship between the structure of polymers and the modulus of elasticity. For example, Krigbaum (12) tried to establish an analytical relationship between the modulus of elasticity and molecular weight being valid for linear polyethylene while Halpin and Kardos (13) tried to establish an analytical relationship between the modulus of elasticity and the changes occurring in spherulites being valid for linear polyethylene.

In this study relying on the results obtained from the tests, the relation between the modulus of elasticity and the degree of crystallinity for polyethylene was found, namely (7),

$$E = 3{,}889\ K_v{}^{4.32} \tag{5}$$

The inportance of this relation from the practical point of view can be explained as follows. Generally the determination of the degree of crystallinity of polymers and other factors in relation to the structure require a lot of apparatus and devices and furthermore take much time. Therefore, the evaluation as stated above, bears a great importance espacially in science of materials. It is possible to determine the degree of crystallinity of the material using relation (5) if the modulus of elasticity being determined in simple tension tests, is computed. On the other hand, if the density of the material is known, it is possible to find the degree of crystallinity of the material using relation (1) and the modulus of elasticity using relation (5).

4.1.4. — Mixture of polyethylene

In spite of the fact that the material of the 0,5 KPE is composed of equal amounts of the HDPE and LDPE, the values for this material are not the average values obtained for the HDPE and LDPE. Therefore, the evaluation of the mechanical factors of the 0,5 KPE is performed in relation to the anount of the mixture ($K_a = V_{HDPE}/V$). Relying on the test results, the following relations are found, namely (7),

$$\sigma_A = 8,22 + 15,11\, K_a \qquad (6)$$

$$\sigma_K = 8,34 + 7,47\, K_a \qquad (7)$$

$$E = 154,57\, e^{1,82\, K_a} \qquad (8)$$

4.2.0. — Creep tests

4.2.1. — General interpretation

The interpretation of creep tests of polyethylene can be summarized as follows :

— The HDPE having a high degree of crystallinity ($K_v = 73,32\,\%$) shows a rather good creep behaviour. It exhibits a good behavior even at 80°C ($T_m = 132$°C, $t = 132 - 80 = 52$°C) which 52°C below the point of melting. According to the theory presented previously, this behavior occurs because the structural changes happening in the HDPE are almost controlled by the crystalline region.

— The creep behaviour of the LDPE ($K_v = 48\,\%$) that has a low degree of crystallinity is rather poor. A fracture occurs even at 50°C ($T_m = 109$ C, $t = 109 - 50 = 59$ C) in a very short period of time and under very small loads and especially at 80°C ($t = 109 - 80 = 29$°C) it does not withstand at all. This behavior is explained by the fact that the changes occurring in the structure are controlled by the amorphous and also by the crystalline regions.

— In spite of the fact that the creep behavior of the 0,5 KPE is superior than the LDPE, it is closer to the LDPE as for us the magnitude is concerned.

4.2.2. — Equations of creep

The creep behavior of the materials subjected to the tests varies according to the applied load and only under a certain loading called a

low loading it exhibits the principal creep behavior. Taking into account the strain (ε) and the ratio of (σ/σ_A) as a criterion to express these loads, it was established that the low loading is valid where

$$\text{at } 20°C \quad \sigma \leq 0,3\,\sigma_A \quad \text{and} \quad \varepsilon \leq 5\%$$
$$\text{at } 50°C \quad \sigma \leq 0,2\,\sigma_A \quad \text{and} \quad \varepsilon \leq 5\%$$
$$\text{at } 80°C \quad \sigma \leq 0,125\,\sigma_A \quad \text{and} \quad \varepsilon \leq 5\%$$

Theoretically the creep phenomenon can be expressed by Voight - Kelvin model and its related by equation.

$$\varepsilon(t) = \frac{\sigma_0}{E}\,[1 - e^{-(t/t_R)}]$$

However, both Voight - Kelvin model and the more developed model of four elements do not reflect the properties of creep behavior of plastic materials. Therefore, some researchers such as Findley (14), Overath and Menges (4), Zapas and Crissman (3) proposed empirical equations to express the creep behavior. However, these equations are very complex and do not take into account the degree of crystallinity of polyethylene and thus the variation of the creep behavior. Therefore, in this study taking into account that the degree of crystallinity of polyethylene plays an important role in the creep behavior, the following equations are found, namely (7)

$$\varepsilon(t) = 7,80\,(\sigma/\sigma_A)^{1.18} + 1,67\,(\sigma/\sigma_A)^{1.39}\,\ln t \qquad (9)$$

for the HDPE that has a high degree of crystallinity.

$$\varepsilon(t) = 0,15 + 9,57\,(\sigma/\sigma_A) + 1,45\,(\sigma/\sigma_A)^{1.50}\,\ln t \qquad (10$$

for the LDPE that has a low degree of crystallinity and

$$\varepsilon(t) = 0,41 + 12,08\,(\sigma/\sigma_A) + 2,07\,(\sigma/\sigma_A)^{1.48}\,\ln t \qquad (11)$$

for the 0,5 KPE that is a mixture of the two. These equations are valid for T=20°C.

4.2.3. — Creep behavior depending on the degree of crystallinity

In this study the creep behavior of polyethylene in relation to the

creep modulus of elasticity as given by the equation of elasticity as given by the equation of $E_K = \sigma/\varepsilon(t)$ was also investigated. Using the values of $\varepsilon(t)$ (Figure 6 to Figure 14) obtained under various loads and temperatures, E_K was computed and represented diagrammatically in Figure 15 to Figure 18 in relation to the loading (σ), Temperature (T) and time (t). As it is seen from the diagrams, the creep modulus of elasticity varies significantly with the temperature. This variation is accounted for the fact that the degree of crystallinity varies with the temperature. As the temperature increases, the deterioration starts in the crystalline region, at first slowly and then proceeds more rapidly and finally at the melting temperature the crystalline structure is completely deteriorated.

Furthermore, the variation of the creep modulus of elasticity exhibits a different manner of variation depending on the degree of crys-
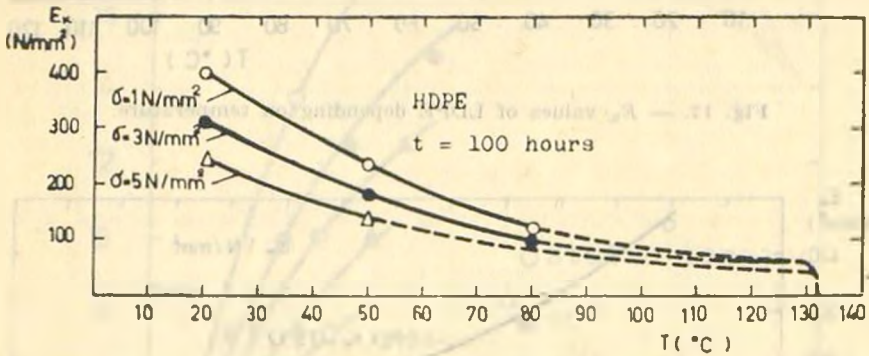

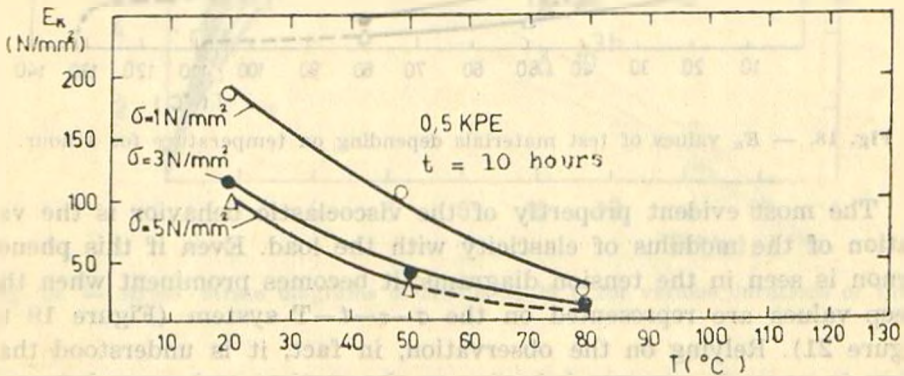Fig. 15. — E_K values of HDPE depending on temperature.

Fig. 16. — E_K values of 0,5 KPE depending on temperature.

tallinity of the material. This different behavior according to the theory previously estaslished occurs because the creep behavior of polyethylene that has a high degree of crystallinity are completely controlled by the crystalline regions and the creep behavior of polyethylene that has a low degree of crystallinity are controlled by the amorphous and also by the crystalline regions.
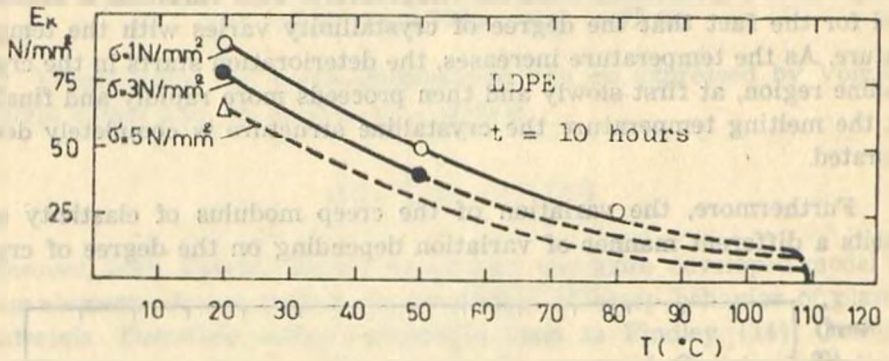

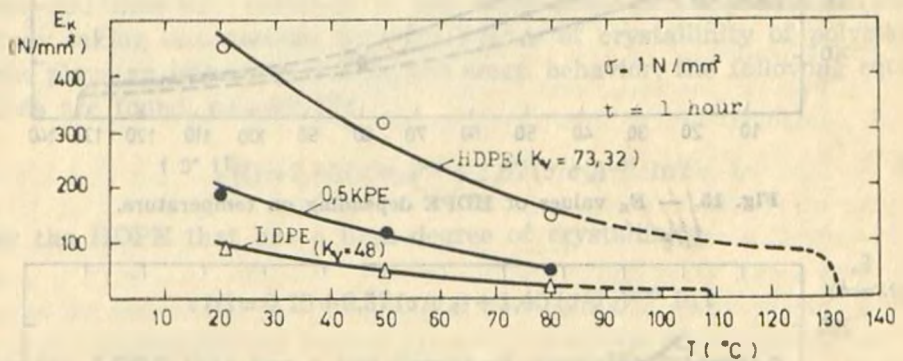Fig. 17. — E_K values of LDPE depending on temperature.

Fig. 18. — E_K values of test materials depending on temperature for 1 hour.

The most evident propertly of the viscoelastic behavior is the variation of the modulus of elasticity with the load. Even if this phenomenon is seen in the tension diagrams, it becomes prominent when the creep values are represented on the $\sigma - \varepsilon - t - T$ system (Figure 19 to Figure 21). Relying on the observation, in fact, it is understood that there is no such separate behavior as the statical and creep behavior

of polyethylene and in more general manner of polymers, but only one mechanical behavior. The difference in appearance happens because of the different rate of the deformation in the tension and creep tests.
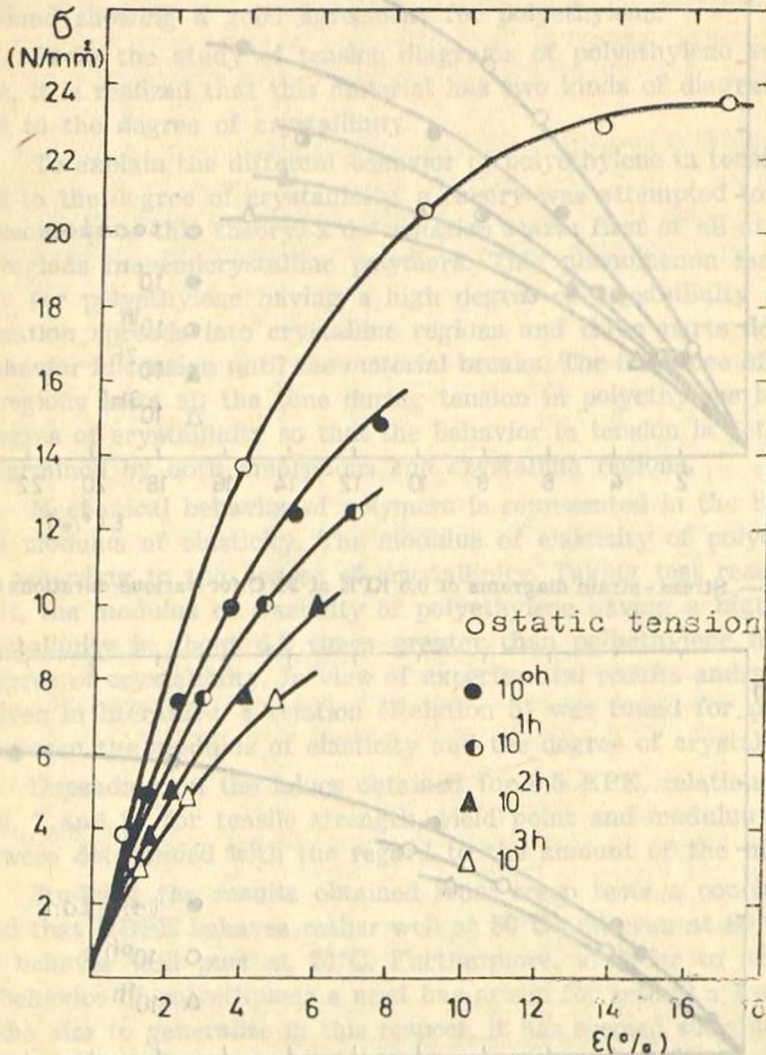

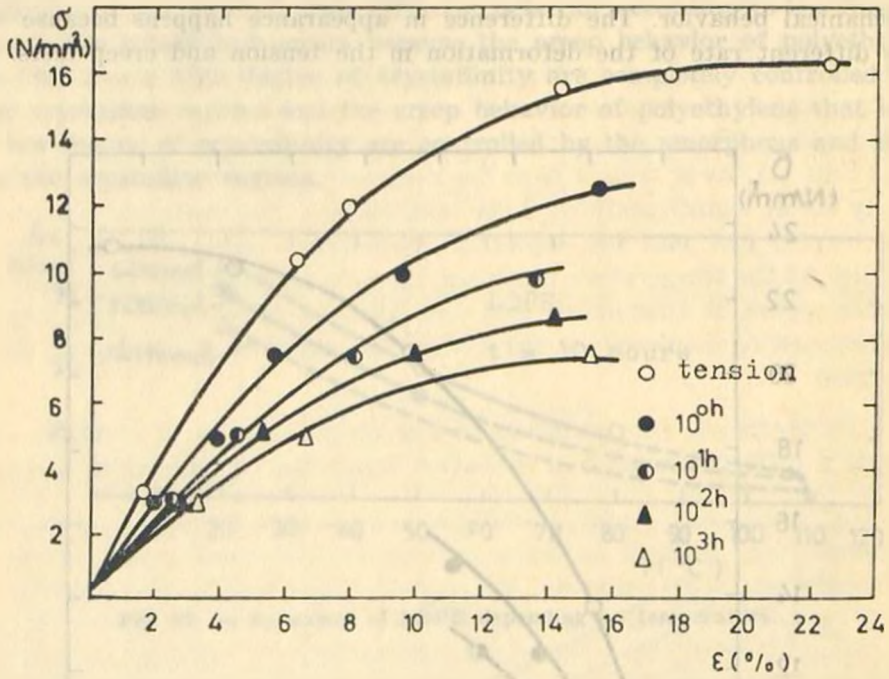Fig. 19. — Stress - strain diagrams of HDPE at 20°C for various durations of time.

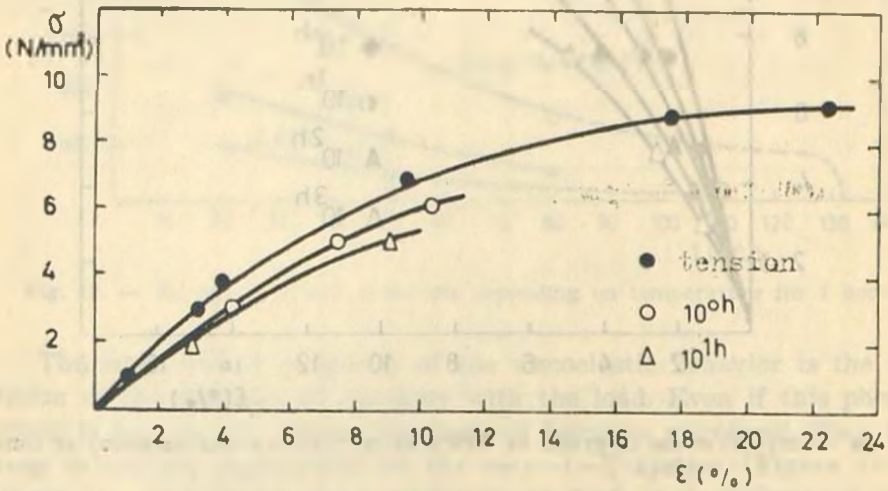Fig. 20. — Stress - strain diagrams of 0,5 KPE at 20°C for various durations of time.

Fig. 21. — Stress - strain diagrams of LDPE at 20°C various durations of time.

5. — CONCLUSIONS

1. An important factor that affects the mechanical behavior and properties of semicrystalline material is the degree of crystallinity. By comparing available analytical and experimental metods (Figure 2) for the determination of the degree of crystallinity, a relation (Relation 1) was found showing a good agreement for polyethylene.

2. From the study of tension diagrams of polyethylene subjected to test, it is realized that this material has two kinds of diagrams with regard to the degree of crystallinity.

3. To explain the different behavior of polyethylene in tension with regard to the degree of crystallinity, a theory was attempted to be seet up. According to this theory, a deformation starts first of all at amorphous regions in semicrystalline polymers. This phenomenon lasts very shortly for polyethylene having a high degree of crystallinity and the deformation spreads into crystalline regions and these parts determine the behavior in tension until the material breaks. The influence of amorphous regions lasts all the time during tension in polyethylene having a low degree of crystallinity so that the behavior in tension is determined by determined by both amorphous and crystalline regions.

4. Mechanical behavior of polymers is represented in the best way by the modulus of elasticity. The modulus of elasticity of polyethylene varies according to the degree of crystallinity. Taking test results into account, the modulus of elasticity of polyethylene having a high degree of crystallinity is about 6,2 times greater than polhethylene having a low degree of crystallinity. In view of experimental results and some values given in literature, a relation (Relation 5) was found for polyethylene between the modulus of elasticity and the degree of crystallinity.

5. Depending on the lalues obtained for 0,5 KPE, relations (Relations 6, 7 and 8) for tensile strength, yield point and modulus of elasticity were determined with the regard to the amount of the mixture.

6. Studying the results obtained from creep tests a conclusion is reached that HDPE behaves rather well at 50°C and even at 80°C while LDPE behaves well pust at 20 C. Furthermore, in order to determine creep behavior of polyethylene a need has arisen for setling a load limit. With the aim to generalize in this respect, it has seemed suitable to express the load limit as σ/σ_Λ and from manipulation of test results only for $\sigma/\sigma_\Lambda \leqslant 0,3$ polyethylene has shown creep behavior within practically acceptable duration of time.

7. All three materials tested within the limit determined above have shown different creep behaviors and for this reason, these beha-

viors cannot be expressed by a single equation. Therefore, starting from the test results, separate creep equations are given for HDPE (Relation 9), LDPE (Relation 10) and 0,5 KPE (Relation 11).

8. If $\sigma-\varepsilon-t-T$ diagrams of tested materials are considered, it is realized that there are not distinct behaviors such as static and creep for polyethylene or in a more general manner for polymers but only one mechanical behavior. The difference in appearance happens to be the results of different rate of deformation in the tension and creep tests.

9. In this study a conclusion is reached that it is right to study static (high rate of deformation) and creep (low rate of deformation) behaviors together in order to become acquainted with the mechanical behavior of viscoelastic material such as polyethylene thoroughly.

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