



VİSKOPLASTİSİTE MODELLERİDE DENGE GERİLMESİ: GÖZLEMLER VE OVERSTRESS KONSEPTİ İLE KULLANIMI

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ÖZET

Birçok malzeme modeli, "denge gerilmesi", "artık gerilmesi" ve "etkili gerilme" olarak da adlandırılan özel bir durum değişkeni içerir. Uzun süreleri içeren malzeme davranışı çözüm için kinematik sertleşme ve / veya Bauschinger etkisini modelleyebilen çok kullanışlı bir değişkendir. Denge gerilmesi ölçülebilir ve kontrol edilemez teorik bir değişkendir, ancak bu değişken dolaylı test ve model simülasyonlarının karşılaştırılması ile çıkarılabilir. Denge gerilimi kavramına dayanan viskoplastik modelin çalışma prensibi; gevşeme testindeki gerilme değişiminin yeterli bir süre sonra sabit bir değere ulaşmasından esinlenme şeklindedir. Denge gerilmesini deneysel olarak belirlemek için, iki basit yöntem vardır, bunlardan biri konvansiyonel gevşeme testi ve diğeri ise, özel sık tekrarlı yükleme testleri içeren daldırma testidir. Bu tip testler kullanılarak farklı denge gerilmesi belirleme türleri sunulmuştur, ancak bu çalışmaların sonuçları, hiçbir malzeme için denge gerilme seviyelerini tam olarak göstermemiştir.

Bu çalışmada oda sıcaklığından 100 °C'ye kadar sıcaklıklarda boşaltma eğrisi üzerindeki noktalarda PP'nin zamana bağlı cevabı ile ilgili deneysel çalışmalar ortaya konulmuştur. Makalenin amacı; i) Çeşitli maksimum gerilme seviyelerine kadar yüklenen ve daha sonra oda sıcaklığından 100 °C'ye kadar geniş sıcaklık aralıklarında çeşitli gerilme seviyelerinde gevşeme testine tabi tutulan PP numunelerinin deneysel verileri rapor etmek. ii) deneysel verileri kullanılarak denge gerilmesinin varlığını göstermek.

ABSTRACT

Many of material models include a special state variable that is called "equilibrium stress", "back stress" and "effective stress" also. This is very useful variable which is able to model kinematic hardening or/and Bauschinger effect for long time solution. Equilibrium stress cannot be measured and controlled, but this variable can be deduced from transient test and comparison of model simulation. Basis of the model based on equilibrium stress concept are that change of stress in relaxation test will reach constant value after a sufficient time. To experimentally determine equilibrium stress, there are two simple methods, one of this is conventional relaxation test and another is dip test that include special cyclic loading tests. Different types of attempting were presented using these tests, but the results of these studies did not precisely show equilibrium stress levels for none of materials.

This paper focuses on the experimental investigation that is related to time dependent response of PP after strain reversal in wide interval temperatures. The aim of the paper is; i) to report experimental data in tensile relaxation test on PP specimens subjected to stretching up to various maximum strains and subsequent retraction down to various strains in wide interval

of temperatures ranging from room temperature to 100 °C ii) to demonstrate existence of equilibrium stress using experimental data of PP.

Keywords: Equilibrium stress, relaxation, temperature effect, viscoplasticity, polypropylene.

1. Introduction

In the materials science and constitutive modeling effort, observation of relaxation behavior is important, because stress evolution on elapsed time is used to determine characteristic properties of a material. Unusual relaxation of polymeric materials has interesting properties that give some clue about equilibrium stress of materials. In this study, extensive experimental results are reported. Polypropylene (PP) specimens were imposed uniaxial tension loading in the interval of temperature ranging from room temperature up to 100 °C. The main aim of the study is to reveal mysteries of unusual relaxation of both polymers. In order to investigate these materials behavior, relaxation test after strain reversal are performed at strain rate of 10 mm/min at various stress levels under various temperature. The following properties are observed: strain amount after the reversal influences unusual relaxation behavior of the polymers at all temperature and stress levels.

This paper focused on experimental investigation of the time-dependent behavior of semicrystalline polymers (PP); observed in relaxation tests on specimens subjected to loading and partial retraction. Relaxation tests at various high temperatures at various stress levels for two polymers are performed on the unloading segment of the stress–strain. Under these tests conditions, the relaxation stress can increase, decrease, or decrease then increase during the test intervals.

1.1 Materials

To investigate these behavior, firstly, Isotactic polypropylene Moplen HP 400R (density 0.905 g/cm³, melt flow rate 25 g/10 min) was purchased from Basell Polyolefins (Switzerland). Dumbbell specimens for tensile tests (ASTM standard D-638) with cross-sectional area 10.2 mm x 4.2 mm were molded by using injection-molding machine Arburg 320C.

DSC (differential scanning calorimetry) measurements were performed by means of Mettler Toledo DSC 823E apparatus. PP samples with mass of about 20 mg were scanned at the heating rate 12 K/min under nitrogen flow. Melting peak of polypropylene corresponded to the temperature $T_m=161$ °C. The specific enthalpy of melting H_m was calculated as 99 J/g, which resulted in degree of crystallinity 47% (by using the specific enthalpy of melting 209 J/g for purely crystalline polypropylene [8]).

1.2 Mechanical Tests

Mechanical tests were conducted by means of universal testing machine Instron-5568 equipped with an electro-mechanical sensor for control of longitudinal strains in the active zone of samples. Tensile force was measured by 5 kN load cell.

The experimental program included two series of tests performed at room and various high temperatures. Each test was conducted on a virgin specimen.

The first series involved six relaxation tests performed after tension up to a fixed maximum elongation d_{\max} and retraction down to various stresses σ_{\min} . In each test, a specimen was stretched with cross-head speed 10 mm/min (which corresponded to strain rate $=2 \times 10^{-3}$ 1/s) up to $d_{\max} = 9$ mm (which corresponded to maximum strain $\epsilon_{\max} = 0.09$), and unloaded with the same cross-head speed down to a required stress σ_{\min} . Afterwards, the strain ϵ was preserved constant, and stress σ was monitored as a function of time t . Following the ASTM protocol E-328 for short-term relaxation tests, duration of relaxation tests $t_{\text{rel}} = 20$ min was chosen. Experiments were carried out with minimum stresses under retraction $\sigma_{\min} = 1, 5, 10, 15, 20$ and 25 MPa.

Next, relaxation tests after strain reversal were performed at 1 MPa at various high temperatures. These series involved tensile tests with cross-head speed 10 mm/min and maximum strain $\epsilon_{\max} = 0.15$. Each test was conducted on at least two virgin specimens. Test temperatures are 23 °C, 40 °C, 50 °C, 60 °C, 70 °C, 80 °C, 90 °C and 100 °C. In experiments at high temperature, each specimen was pre-heated in a thermal chamber required temperature for at least half an hour, and then equilibrated in the testing machine for 10 minutes before loading.

1.3 Results

The stress-strain curves and the change of stress as a function of time at room temperature are given in Figs. 1 and 2 respectively.

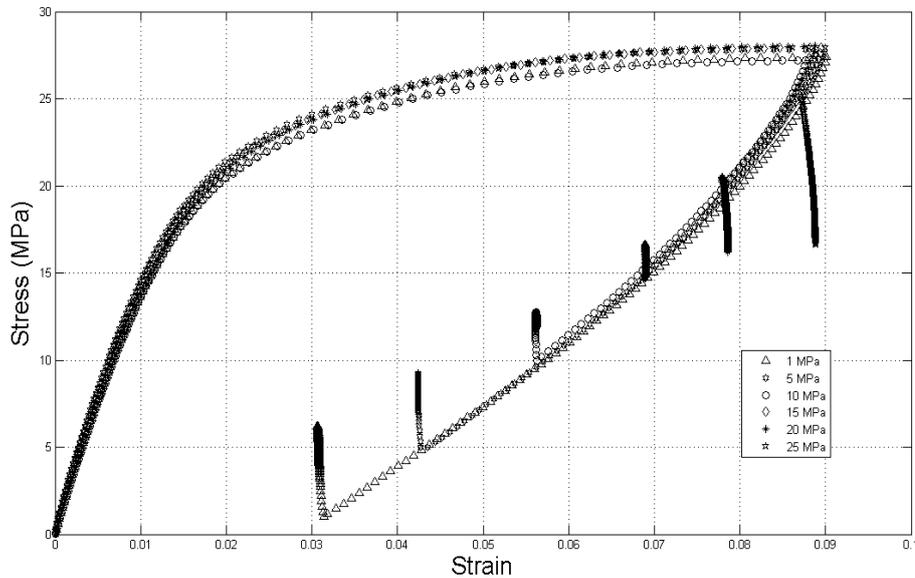


Fig. 1. Stress versus strain for PP. Symbols: experimental data in cyclic tests with strain rate 2×10^{-3} 1/s, maximum strain $\epsilon_{\max} = 0.09$, and various minimum stresses σ_{\min} . MPa.

Observations from the Fig. 2 are that evolution of the relaxation curves are depend on the amount of strain reversal, ($\Delta\epsilon = \epsilon_{\max} - \epsilon$). When amount of strain reversal is large, it continues to increase with increasing time until it reaches an asymptotic value. If $\Delta\epsilon$ is intermediate value, stress at first increase up to a level and then decreases continuously with time until it approaches a stable value asymptotically.

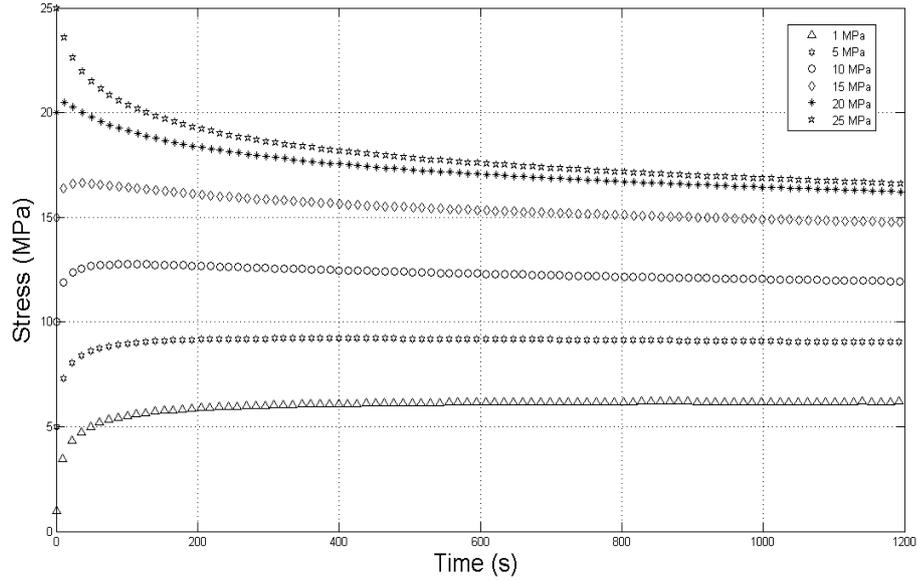


Fig. 2. Stress versus relaxation time for PP. Symbols: experimental data in relaxation tests on specimens subjected to tension up to maximum $\epsilon_{max} = 0.09$ and subsequent unloading down to various minimum stresses σ_{min} MPa.

Stress-strain curves of PP at various temperatures are given in Fig. 3. As can be seen in Fig. 3, stress-strain curves are nonlinear and stress monotonically decreases with increasing temperature maximum stress at 15% strain decreases approximately by 3 times, from 25.8 MPa at room temperature to 21 MPa at 40, 18.2 MPa at 50, 15,6 MPa at 60, 12.8 MPa at 70, 11.3 MPa at 80, 10.2 MPa at 90, and 9 MPa at 100 °C. Residual strain after unloading is practically same for all temperatures and this value is close to 0.06 and independent of temperature.

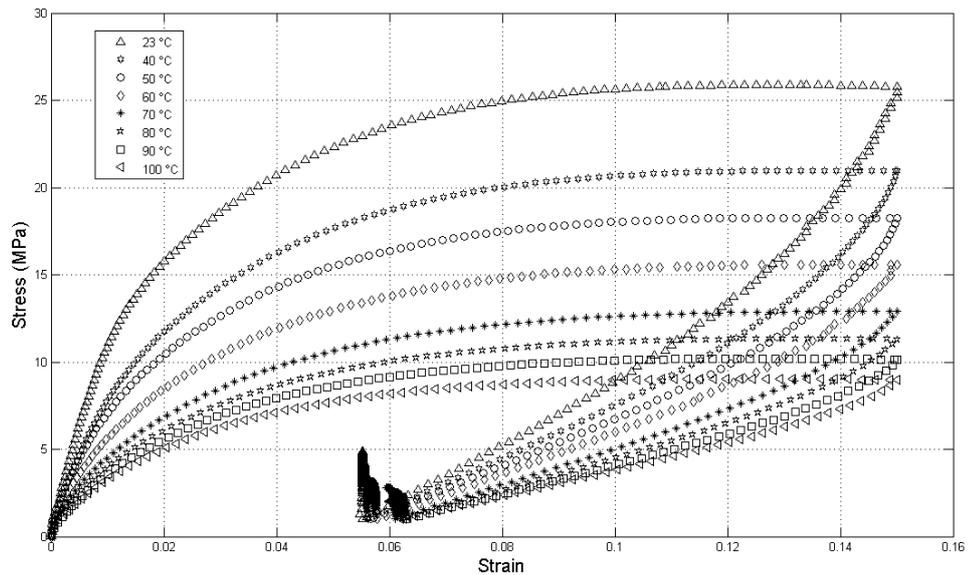


Fig. 3. Stress versus strain for PP. Symbols: experimental data in cyclic tests with strain rate 2×10^{-3} 1/s, maximum strain $\varepsilon_{\max} = 0.15$, minimum stresses $\sigma_{\min.} = 1$ MPa. and various temperature levels.

Experimental results in relaxation tests at 1 MPa at various temperatures are shown in Fig. 4. Strain reversal was very large level in these cases, for evolution of relaxation behavior at high temperature can be observed. For all temperatures, tensile stress monotonically increases with elapsed time. Especially, when temperatures are bigger than 40 °C, stress level reaches asymptotic values and then it seems to be practically constant.

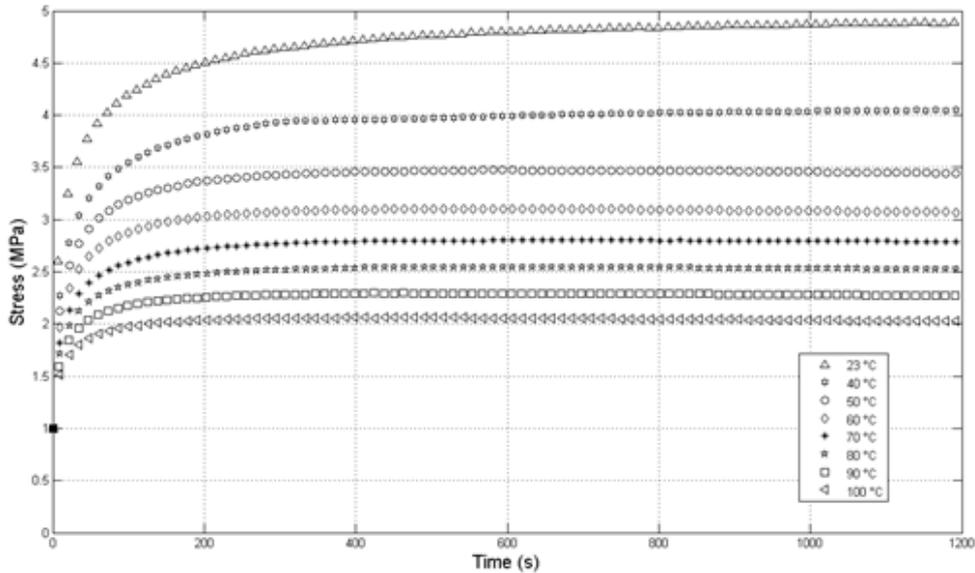


Fig. 4. Stress versus relaxation time for PP. Symbols: experimental data in relaxation tests on specimens subjected to tension up to maximum $\varepsilon_{\max} = 0.15$ and subsequent unloading down to minimum stresses $\sigma_{\min} = 1$ MPa at various temperature levels.

2. Modeling

Many models use the “equilibrium stress”, also sometimes known as “the back stress”, in characterizing the response of both polymeric and non-polymeric materials [1-4]. Several researchers attempted to determine characteristics of the equilibrium stress. Their assumption is that there exist loading conditions under which the relaxation processes stop so the load may be held at constant strain indefinitely, and which the material response would normally tend toward this equilibrium [5-7].

In the literature, there are two types of experimental to determine equilibrium stress. The first method is similar to our test on PP that the specimen is partially unloaded and then waited for zero stress rate at constant strain level. The second method is dip test that include cyclically loading, firstly, specimen is load and then rapidly is unload, and then stress is fixed while observation of strain. On the other hand, there is modified version of dip test that is conducted using deformation history. The method is called rapid load-unload test also [8-10].

Fig. 2 and 4 show that at constant strain the stress either decrease or increases toward the equilibrium response, depending on which side of the equilibrium response the process starts from, and then indefinitely stays there. But these tests take very long time to reach equilibrium stress.

Viscoplasticity theory based on overstress (VBO) model was developed by Krempl based on the observations of the behaviour of metals and alloys at high temperatures [11]. [12 and 13] observed that the mechanical behaviour of the solid polymer at room temperature has a number of similarities with the mechanical behaviour of metallic materials. In order to model the mechanical behaviour of solid polymers, [14] developed the viscoplasticity theory based on overstress for polymers (VBOP) by modifying the VBO model.

In this study, as the experiment is uniaxial tension test, one-dimensional equations of VBOP model used. The flow law for small strain, incompressibility and isotropy is given by;

$$\dot{\epsilon} = \dot{\epsilon}^{el} + \dot{\epsilon}^{in} = \frac{\dot{\sigma}}{RE} + F \frac{\sigma - g}{\Gamma} \quad (1)$$

where E is Young's modulus, and F is the viscosity function. The superposed dot represents the material time derivative. The difference between the Cauchy and the equilibrium stress is "the overstress". $\Gamma = |\sigma - g|$ is the overstress invariant. It can be seen that evolving of the overstress in Fig. 5.

The evolution of the equilibrium stress is as follows;

$$g = \psi \frac{\dot{\sigma}}{RE} + \psi F \left(\frac{\sigma - g}{\Gamma} - \frac{g - k}{A} \right) + \left(1 - \frac{\psi}{E} \right) \dot{k} \quad (2)$$

the ψ represent the positive shape function. The Shape function is influential on the transition from the quasi-linear region to the fully inelastic flow region. It controls the modelling of the neck region, as well as the modelling of the unloading curve. It is defined for polymeric materials as follows [15].

$$\psi = \psi_1 + \left| \frac{C_2 - \psi_1}{\exp(C_3 |\epsilon^{in}|)} \right|$$

$$\psi_1 = C_1 \left(1 + C_4 \left(\frac{|g|}{A + |k| - \Gamma} \right) \right) \quad (3)$$

where C_1, C_2, C_3, C_4 are the material parameters. Relation between Cauchy stress and equilibrium stress are given in Fig. 5.

Kinematic stress adjusts the tangent module at the maximum strain of interest. It also enables the modelling of the Bauschinger effect.

$$\dot{k} = \frac{|\sigma|}{\Gamma + |g|} \bar{E}_t \dot{\epsilon}^{in} \quad (4)$$

where $\bar{E}_t = \frac{E_t}{1 - \frac{E_t}{E}}$ and E_t is the tangent modulus. The isotropic stress (A) is used for the

modelling of the hardening and softening behaviour. It was taken as a constant value in this study. The positively increasing form of the nonlinear viscosity function is;

$$F = B \left(\frac{\Gamma}{D} \right)^m \quad (5)$$

B is the universal constant, m is the material constant, and D is the drag stress. In order to model the nonlinear features of the unloading curve of polymeric materials, The R parameter is added to the elastic part of Eq. 1 [16 and 17]. It is defined as $R = 1 - t \left(\frac{|g - k|}{A} \right)^n$, where t and n are the material parameters.

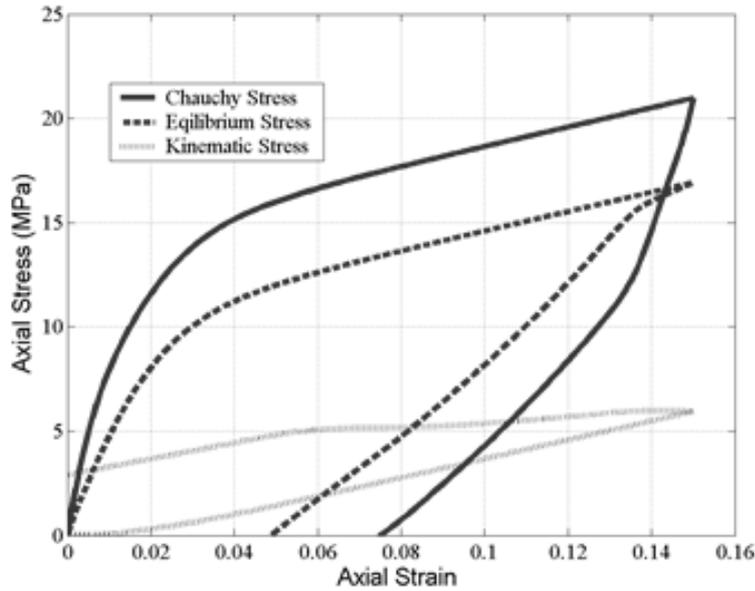


Fig. 5. "Overstress" model's Cauchy-Equilibrium-Kinematic Stress Relation.

The model predictions were calculated using these determined material parameters. The following simulations were carried out to investigate the modeling capability of VBOP for 20 min relaxation at five different stress levels (20 MPa, 15 MPa, 10 MPa, 5 MPa and 1 MPa) on unloading path.

Stress-strain curves of PP under uniaxial tensile loading up to 9%, afterwards unloading down to 20, 15, 10, 5, and 1 MPa respectively, with strain rate of $2 \times 10^{-3} \text{ s}^{-1}$ and model predictions are given in Fig. 6. Each stress-strain curves include 20-min relaxation experiments at all stress levels. Model prediction and the experimental results are compatible with each other. Especially unloading curves are captured quite well for 0.006 and 0.009 strains; however, there is a little bit deviation at 0.0027 strains.

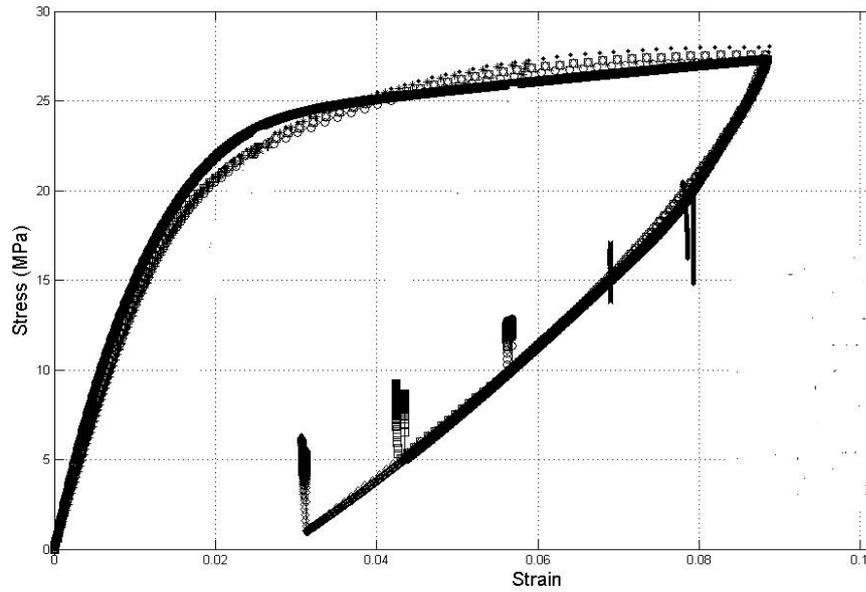


Fig. 6. Stress versus strain. Specimens are subjected to tensile loading to maximum strain $\epsilon_{\max} = 9\%$ and subsequent unloading to various minimum stresses at 2^{-3} s^{-1} , over a 20 min. relaxation test.

VBOP model relaxation predictions for loading up to 0.009 strain and unloading down to five different stress levels (20 MPa, 15 MPa, 10 MPa, 5 MPa and 1 MPa) are given in Fig. 7. Mixed relaxation is observed at 20 and 15 MPa. In these stress level, there are a little bit deviation between VBOP model prediction and experimental data for long time, but evolution of prediction and experimental data have same characteristics. If the stress level is below 15 MPa, the material shows inverse relaxation. Good agreement between the inverse relaxation of PP and predictions of the model are observed.

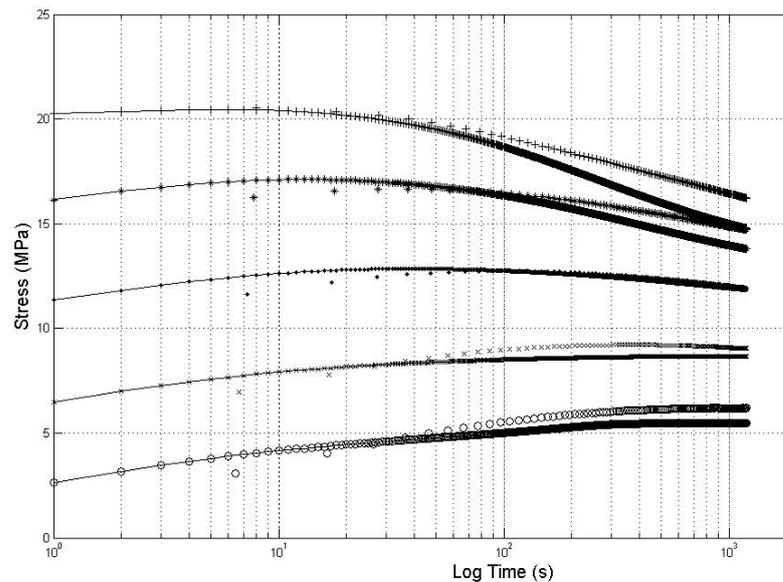


Fig.7. Stress versus logarithmic relaxation time. Specimens are subjected to tensile loading to maximum strain $\epsilon_{\max}=9\%$ and subsequent unloading to various minimum stresses at $2 \times 10^{-3} \text{ s}^{-1}$, over a 20 min. relaxation test.

3. Conclusions

A comprehensive study is reported on isotactic polypropylene in uniaxial tensile test with $2 \times 10^{-3} \text{ 1/s}$ strain rate, tensile relaxation test with various stresses on unloading segments at room temperature. In this study, it is observed that unusual relaxation behaviour is affected by the difference between the maximum strain level and the current strain level. As the strain increment increases, the relaxation behaviour changes from simple relaxation behaviour to inverse relaxation behaviour. Additionally, Unusual relaxation behavior of PP under uniaxial tensile loading at various temperatures is experimentally investigated. Following observations are concluded: Unusual relaxation behavior not only depends on strain amount after strain reversal but also on temperature.

The modified VBOP is applied to predict unusual creep/relaxation behavior of polymer at various stresses. In addition, multiple creep behavior of polypropylene at various stress levels at room temperature is predicted using the modified VBOP model. Comparisons between the VBOP model prediction and experimental data of polypropylene reveal that the VBOP model has good capability to predict unusual relaxation behaviour of polypropylene.

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