# Effects of Prior Aging at 288°C in Argon Environment on Time-Dependent Deformation Behavior of a Thermoset Polymer at Elevated Temperature, Part 2: Modeling with Viscoplasticity Theory Based on Overstress

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**ABSTRACT:** The viscoplasticity based on overstress (VBO) is augmented to model the effects of prior isothermal aging in an argon environment on the inelastic deformation behavior of PMR-15 neat resin, a high-temperature thermoset polymer. VBO is a unified state variable theory with growth laws for three state variables: the equilibrium stress, the kinematic stress and the isotropic stress. A systematic model characterization procedure based on a limited number of well defined experiments is employed to determine the VBO parameters. Experimental findings presented in Part I reveal the equilibrium stress and the kinematic stress to be affected by prior aging. Based on the experimental results, the isotropic stress is developed as a function of prior aging time. In addition, several VBO model parameters are made dependent on prior aging time. Comparison with experimental data demonstrates that the modified VBO successfully predicts the inelastic deformation behavior of the PMR-15 polymer subjected to prior isothermal aging for up to 2000 h. © 2009 Wiley Periodicals, Inc.† J Appl Polym Sci 114: 3389–3395, 2009

**Key words:** aging; creep; high-temperature materials; viscoelastic properties; polyimides

## INTRODUCTION

The experimental results presented in a companion paper, Part 1,[1] showed that the PMR-15 solid polymer subjected to prior isothermal aging at 288°C in argon exhibited rate (time)-dependent mechanical response at elevated temperature. These deformation behaviors have considerable design implications. Time-dependent behavior can cause dimensional changes in polymeric structural components throughout their service life that must be accounted for in design. Current design practice for metallic components operating at elevated temperatures ensures reliable service by performing inelastic analysis and life prediction in the design stage long before the component is built. The revolutionary growth of computing power and development of advanced finite element software make the use of inelastic analysis in the design phase possible. The same approach can be applied for polymeric components and polymer-matrix composite (PMC) structures, provided the proper constitutive equations to accurately describe the time-dependent deformation behavior of the constituent materials are available. In the case of PMC structures it is particularly important to account for the time-dependent deformation behavior of the matrix material since the fibers can generally be considered to remain linear elastic within their application range.[2,3]

Various types of constitutive formulations have been developed to describe the mechanical behavior of polymeric materials.[4–12] Recently, Krempl and Khan[13] demonstrated that the unified viscoplastic models originally developed for engineering alloys show the capability to model the features of material behavior exhibited by solid polymers at room temperature. One such model is the viscoplasticity based on overstress (VBO) developed by Krempl et al.[14–17] Kitagawa et al.[18,19] and Bordonaro and Krempl[20] applied VBO to polymers. Krempl and Ho[21] developed a specialization of viscoplasticity based on overstress for polymers (VBOP), which Khan and Krempl[22,23] and Khan[24] employed to represent the mechanical behavior of various polymers at room temperature. Other specializations of VBO have also been applied to polymers at room temperature by Khan,[25] Colak,[26] and Colak and Dusunceli.[27]

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Recently, McClung and Ruggles-Wrenn[28] reported results of strain-controlled tests investigating the rate (time)—dependent mechanical behavior of the PMR-15 neat resin at 288°C. In this study, a piecewise linear deformation history, involving strain-controlled tensile loading and unloading with intermittent periods of relaxation during loading and during unloading, or periods of creep during loading, was used to explore the effect of strain rate on the inelastic behavior of the polymer. The results of this study revealed several qualitative features of the inelastic behavior of the PMR-15 polyimide that strongly suggested the usefulness of the overstress viscoplastic constitutive model in predicting the inelastic behavior of this material. McClung and Ruggles-Wrenn[29,30] demonstrated that the VBOP was capable of accurately reproducing the inelastic deformation behavior of the PMR-15 neat resin at 288°C. In addition, McClung and Ruggles-Wrenn[29,30] developed and validated a systematic experimentally based procedure for determining the VBOP model parameters.

Among the thermosetting polyimide resins, PMR-15 is of particular interest because of its superior high-temperature properties and ease of processing.[31–33] Designed for use at temperatures near its $T_g$, the PMR-15 resin is extensively used as a matrix material in high-temperature structural composites for aerospace applications. The key features of the deformation behavior of the PMR-15 neat resin subjected to prior isothermal aging identified from experimental results presented in Part I suggest the VBOP as a promising candidate constitutive model to represent the inelastic behavior of the aged material. The objective of this effort is to extend the VBOP to capture the effects of prior aging on the inelastic behavior of the PMR-15 polymer at elevated temperature. Based on experimental results, the phenomenological indicators of aging and implications for modeling are assessed. The isotropic stress and several model parameters are developed into functions of prior aging time. The capability of the modified VBOP to account for the observed effects of prior aging on the rate-dependent behavior is evaluated by comparing model predictions with the test data.

## VISCOPLASTICITY BASED ON OVERSTRESS FOR POLYMERS

The experimental results reported by Falcone and Ruggles-Wrenn,[34] and McClung and Ruggles-Wrenn[28–30] reveal features of the inelastic deformation behavior of the PMR-15 polymer that are qualitatively similar to those exhibited by metals[35–39] as well as by several other polymers.[13,22–24]

The experimental observations of inelastic behavior of metals and alloys presented in[35–37] served as the foundation for developing the VBO, a constitutive, state variable model where the inelastic strain rate depends on the overstress.[15,16] The VBO is based on the hypothesis that the test specimen serves as an integrator of all internal deformation events and/or mechanisms. Kitagawa et al.[18,19] and Bordonaro and Krempl[20,40] observed that at room temperature solid polymers exhibited deformation behaviors similar to those exhibited by engineering alloys. While similarities were numerous, several key differences were also noted. It was concluded that VBO could be employed to model the behavior of polymers provided modifications could be made to capture such key differences in behaviors. As a result, a specialization of the VBOP was developed.[21]

The full three-dimensional formulation of the VBOP is given by Ho.[41] For brevity only the uniaxial form is introduced here. The central flow law of the model is expressed in an overstress format as the sum of elastic and inelastic strain rates,

$$\dot{\varepsilon} = \dot{\varepsilon}^{el} + \dot{\varepsilon}^{in} = \frac{\dot{\sigma}}{E} + \frac{\sigma - g}{Ek}, \tag{1}$$

where $E$ is the elastic modulus, $g$ is the equilibrium stress, and $k$ is the viscosity function with dimension of time. Note that this form of the flow law is analogous to the governing equation for the standard linear solid (see Krempl and Ho[21] and Krempl and Khan[13]). Within the VBO and the VBOP, the equilibrium stress $g$ represents the stress–strain behavior in response to loading at an infinitesimally slow strain rate. The growth law for the equilibrium stress is given by

$$\dot{g} = \Psi \frac{\dot{\sigma}}{E} + \Psi \left[ \frac{(\sigma - g)}{Ek} - \frac{(g - f)}{A} \left| \frac{(\sigma - g)}{Ek} \right| \right] + \left[ 1 - \frac{\Psi}{E} \right] \dot{f}. \tag{2}$$

Here $\Psi$ is the positive shape function that governs the transition from quasi-linear elastic behavior to inelastic flow, i.e. the shape of the "knee" in the stress–strain diagram, as well as the curvature of the unloading curve. The kinematic stress $f$ sets the tangent modulus at the maximum strain of interest and is given by the growth law,

$$\dot{f} = \left[ \frac{|\sigma|}{\Gamma + |g|} \right] E_t \frac{(\sigma - g)}{Ek}, \tag{3}$$

where $E_t$ is the tangent modulus and $\Gamma$ is the overstress invariant which, in the uniaxial case, equals $|\sigma\text{-}g|$. The isotropic stress $A$ is responsible for modeling cyclic hardening or softening. The evolution of the isotropic stress $A$ is given by

$$\dot{A} = A_c [A_f - A] \left| \frac{\sigma - g}{Ek} \right|. \tag{4}$$

The evolution of the state variables $g$, $f$, and $A$ in a tensile test is schematically depicted in Figure 1. In

the case of the PMR-15 polymer, cyclically neutral behavior is assumed. Therefore, the isotropic stress is set to $A = \text{const}$. The form of the shape function $\Psi$ for the VBOP is given by Khan and Krempl[22,23] and Khan[24] as

$$\Psi = C_1 + (C_2 - C_1)e^{-C_3|\varepsilon^{in}|}, \tag{5}$$

where $C_1$, $C_2$, and $C_3$ are material constants. This function is positive, with $E_t < \Psi < E$. The nonlinear viscosity function has the following form[23]:

$$k = k_1\left[1 + \frac{\Gamma}{k_2}\right]^{-k_3}, \tag{6}$$

where $k_1$, $k_2$, and $k_3$ are material constants.

The VBO was successfully applied to modeling the inelastic behavior of Nylon 66[21] and several other semicrystalline and amorphous polymers[22–24] at room temperature. More recently the VBOP together with the newly developed model characterization procedure were used to predict the response of the PMR-15 polymer under various test histories at 288°C.[30] Predictions were in excellent agreement with experimental data.

## VBOP EXTENDED TO CAPTURE THE EFFECTS OF PRIOR AGING

### Phenomenological indicators of aging and implications for modeling with VBOP

The experimental results reported in Part I reveal the following key features of the deformation behavior of the PMR-15 polymer subjected to prior aging in argon at 288°C:

i. Results obtained in tests conducted with the strain rates ranging from $10^{-6}$ $s^{-1}$ to $10^{-3}$ $s^{-1}$ reveal that the aged PMR-15 neat resin exhibits significant nonlinear strain rate sensitivity in monotonic loading. For a given prior aging time, the flow stress increases nonlinearly with increase in the loading rate. A ten-fold increase in strain rate does not cause a ten-fold increase in the flow stress level. A unique stress–strain curve is obtained for a given strain rate. Note that a qualitatively similar rate-dependent stress–strain response was observed for the unaged PMR-15 polymer[28] that was successfully modeled with the VBOP.[30]
ii. The flow stress increases with prior aging time. Notably the increase in flow stress is independent of the strain rate. Recall that in the context of the VBOP the equilibrium stress $g$ is representative of the stress–strain behavior in response to loading at an infinitesimally slow strain rate. Thus, in the inelastic flow region, the equilibrium stress $g$ represents the flow stress produced at an infinitesimally slow strain rate. Hence, in the inelastic flow region, the equilibrium stress must also increase with increasing prior aging time. Within the VBOP the isotropic stress represents the difference between the equilibrium stress and the kinematic stress in the region of inelastic flow[13] (see schematic in Fig. 1). Consequently the increase in equilibrium stress with prior aging time can be achieved by increasing the isotropic stress $A$. In the next section the isotropic stress is established as a function of prior aging time based on experimental data.
iii. Prior aging has no effect on relaxation behavior. As in the case of the unaged PMR-15 polymer, relaxation behavior of the aged material is strongly influenced by prior strain rate. Stress drop in relaxation depends only on time and prior strain rate and is independent of stress and strain at the beginning of relaxation. This result is particularly important because it indicates that prior aging has no effect on the viscosity function $k$ included in the VBOP formulation.
iv. Departure from the quasi-linear behavior is delayed as the prior aging time increases. Departure from the quasi-linear behavior corresponds to the beginning of the "knee" in the stress–strain diagram. The shape function $\Psi$ defines the shape of the "knee". Parameters of the shape function are obtained from the tensile test results.[30] Within the shape function, $C_2$ is the specific parameter that governs the departure from quasi-linear behavior. Therefore a delayed departure from quasi-linear behavior can be achieved by making $C_2$ a function of prior aging time.
v. Both the elastic modulus and the tangent modulus increase with prior aging time. The values of the elastic modulus and of the tangent modulus are obtained from the tensile test results.[30]

### VBOP applied to PMR-15 subjected to prior aging

The observations outlined above suggest that to extend the VBOP to capture the effects of prior

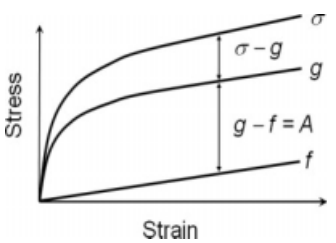


**Figure 1** Schematic of a stress–strain path generated by the VBOP. The equilibrium stress ($g$) and kinematic stress ($f$) curves are also shown.

**TABLE I**
**VBOP Parameters at 288°C for Unaged PMR-15 Neat Resin and for PMR-15 Neat Resin Subjected to Prior Aging at 288°C in Argon**

| Aging time (h) | Elastic modulus (GPa) | Tangent modulus (MPa) | Isotropic stress (MPa) | Viscosity function | | | Shape function | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | $k_1$ (s) | $k_2$ (MPa) | $k_3$ | $C_1$ (MPa) | $C_2$ (GPa) | $C_3$ |
| Unaged | 2.08 | 18 | 20.0 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.00 | 10 |
| 50 | 2.09 | 18 | 22.3 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.10 | 10 |
| 100 | 2.18 | 30 | 22.9 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.19 | 10 |
| 250 | 2.20 | 40 | 24.0 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.20 | 10 |
| 500 | 2.23 | 44 | 25.4 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.29 | 10 |
| 1000 | 2.27 | 49 | 26.0 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.30 | 10 |
| 2000 | 2.32 | 60 | 27.7 | $1.0 \times 10^4$ | 35 | 12 | 100 | 1.37 | 10 |

aging on inelastic deformation behavior, it may be sufficient to make the elastic modulus, $E$, the tangent modulus, $E_t$, the isotropic stress, $A$, and the parameter $C_2$ of the shape function $\Psi$ dependent on prior aging time. Note that the viscosity function $k$ remains unaffected by prior aging.

The systematic model characterization procedure developed by McClung and Ruggles-Wrenn[29,30] was used to obtain the VBOP model parameters from experimental data reported in Part I. Elastic modulus and tangent modulus were found directly from the results of the tensile tests. The equilibrium stress and the isotropic stress were determined from the results of the constant strain rate tests with intermittent periods of relaxation. The viscosity function parameters were also obtained from the results of the relaxation tests conducted with various prior strain rates. Finally results of the tensile tests representing

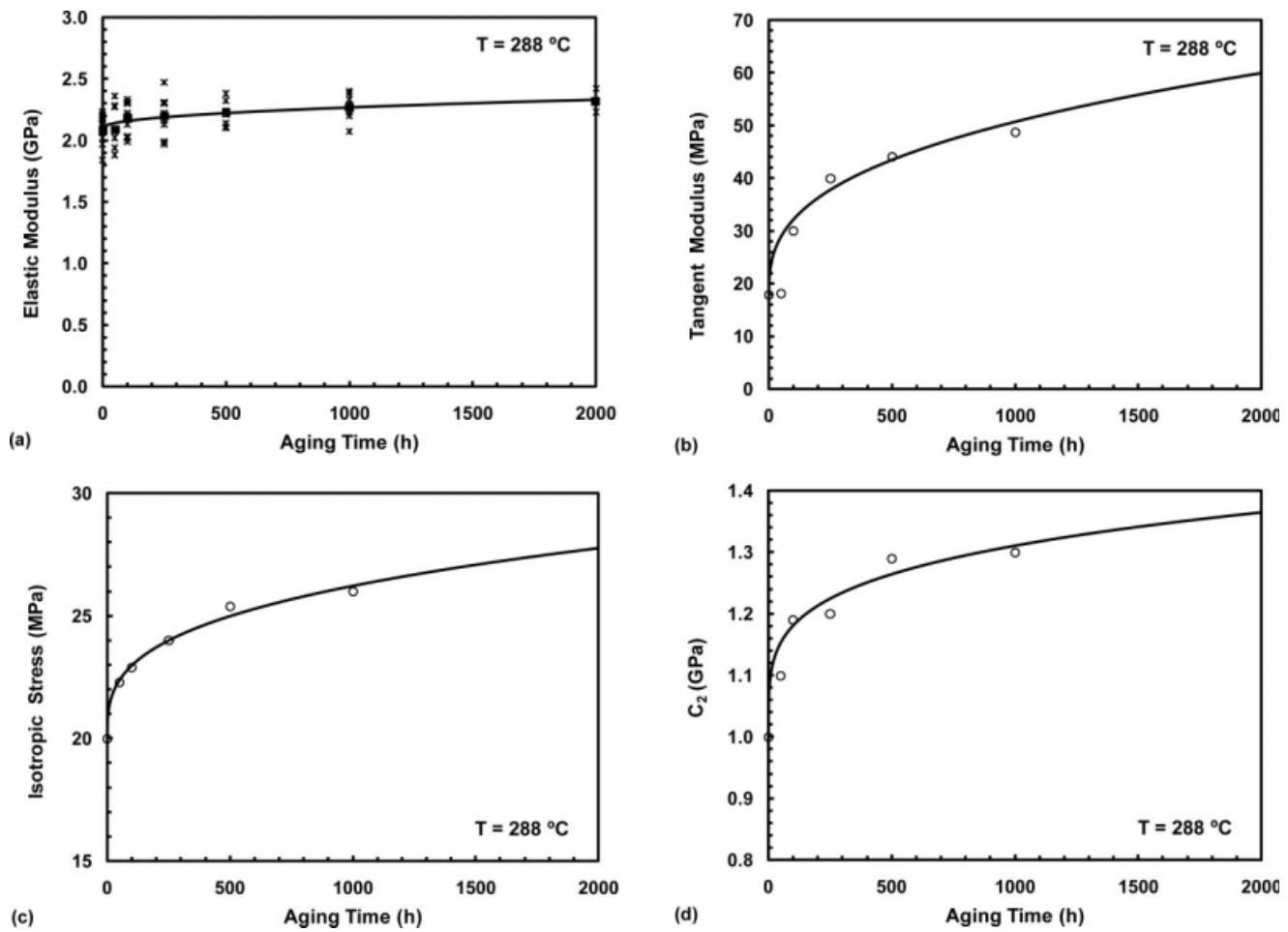


**Figure 2** VBOP model parameters as functions of prior aging time at 288°C in argon: (a) elastic modulus vs. aging time, (b) tangent modulus vs. aging time, (c) isotropic stress vs. aging time, (d) parameter $C_2$ vs. aging time.

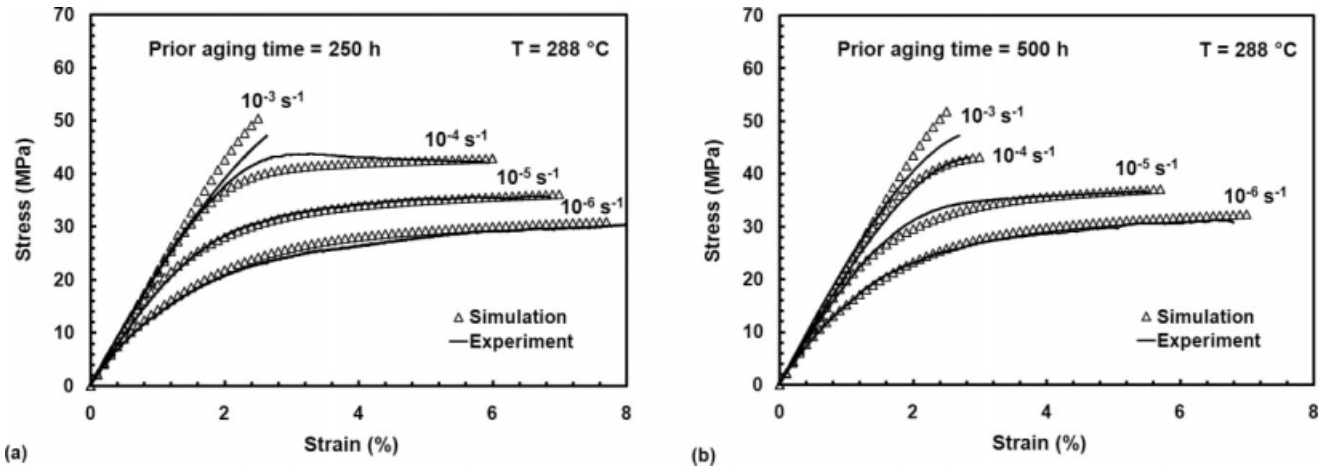


**Figure 3** A comparison between experimental and simulated stress–strain curves obtained in tensile tests to failure conducted at constant strain rates of $10^{-6}$, $10^{-5}$, $10^{-4}$, and $10^{-3}$ $s^{-1}$ at 288°C for PMR-15 polymer aged at 288°C in argon for: (a) 250 h and (b) 500 h. The VBOP model successfully represents the strain rate dependence of the aged material.

the regions where the quasi-linear stress–strain behavior transitions to the inelastic flow were used to calculate the parameters of the shape function. The experimental data for the PMR-15 specimens subjected to prior aging of various durations at 288°C in argon were utilized to produce the VBOP model parameters for various prior aging times. The results are summarized in Table I. Elastic modulus, tangent modulus, isotropic stress, and parameter $C_2$ are also plotted vs prior aging time in Figures 2(a–d), respectively. Based on these results the VBOP model parameters were established as functions of prior aging time,

$$E = 0.0072\ t_a^{0.456} + 2.10 \tag{7}$$

$$E_t = 2.89\ t_a^{0.354} + 17.40 \tag{8}$$

$$A = 0.7022\ t_a^{0.3168} + 19.97 \tag{9}$$

$$C_2 = 0.0685\ t_a^{0.224} + 0.989 \tag{10}$$

Simulations generated with the eqs. (1)–(6) and model parameters in Table I are shown in Figures 3 and 4. It is seen that simulations of strain-controlled monotonic loading for PMR-15 polymer aged for 250 and 500 h in Figure 3 are in good agreement with the experimental data. Likewise the simulations of the relaxation response produced for the PMR-15 specimens aged for 250 and 500 h (Fig. 4) compare reasonably well with the experimental results. The model somewhat over-predicts the stress drop in the early stages of relaxation. However, after ~4 h of relaxation, model predictions and experimental results begin to converge. The simulations generated for the material subjected to prior aging for 50, 100, and 1000 h agree with the experimental data equally well.

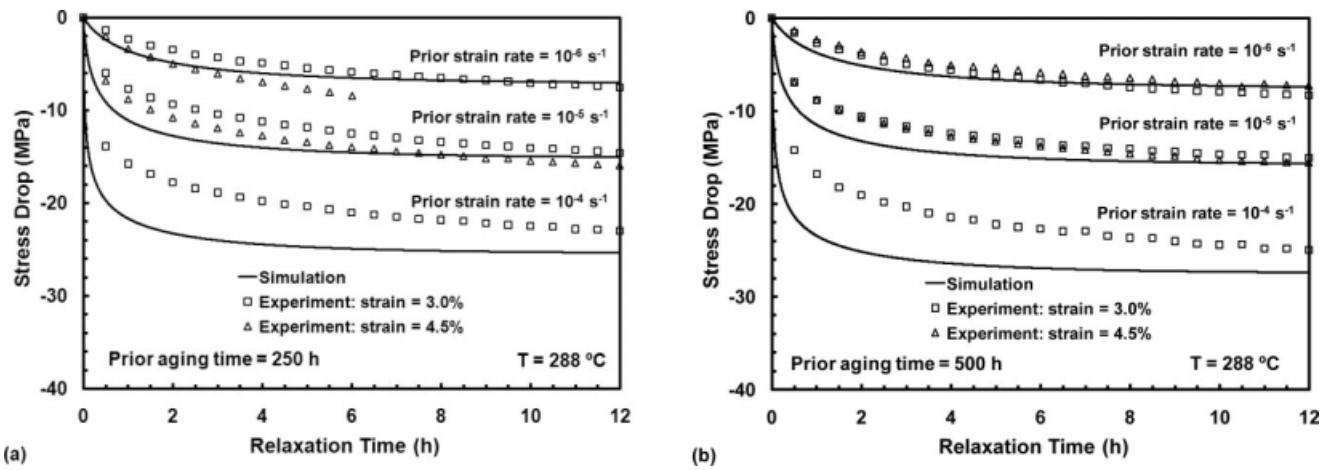


**Figure 4** A comparison between experimental and simulated stress decrease vs. relaxation time curves obtained in relaxation tests at $\varepsilon = 3\%$ and 4.5% for PMR-15 polymer aged at 288°C in argon for: (a) 250 h and (b) 500 h. The VBOP model successfully represents the relaxation response of the aged material.

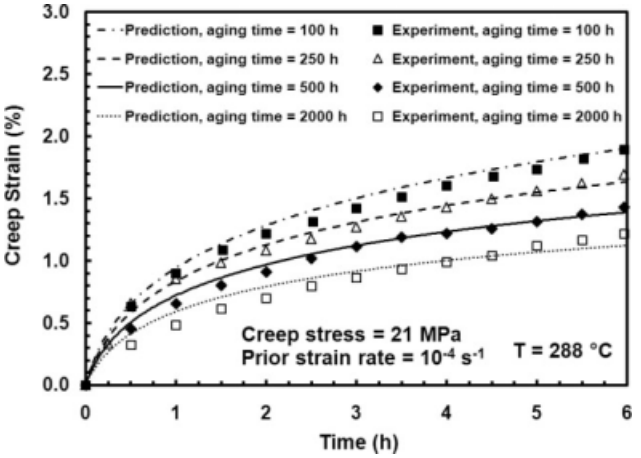


**Figure 5** A comparison between experimental and predicted creep strain vs. time curves obtained at 21 MPa for PMR-15 polymer aged at 288°C in argon for various durations. Effect of prior aging on creep behavior is predicted well by the model.

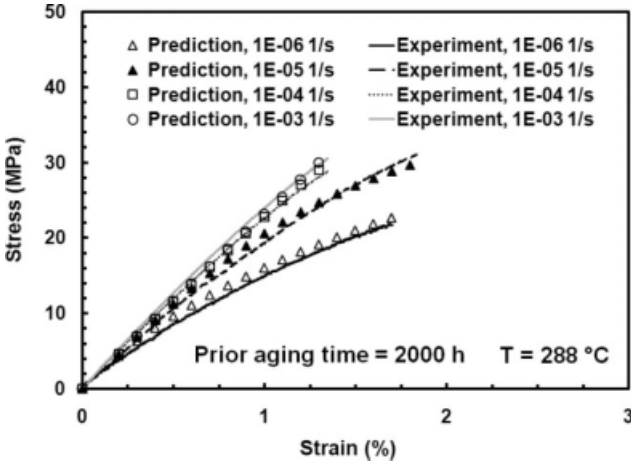


**Figure 6** A comparison between experimental and predicted stress–strain curves obtained in tensile tests to failure conducted at constant strain rates of $10^{-6}$, $10^{-5}$, $10^{-4}$, and $10^{-3}$ $s^{-1}$ at 288°C for PMR-15 polymer aged at 288°C in argon for 2000 h. The model successfully predicts the strain rate sensitivity of the aged material.

The VBOP constitutive model and the model parameters determined in the characterization procedure for the aged PMR-15 neat resin were further validated by comparing the model predictions with experimental results obtained in tests that differ in kind from those used for model characterization. Because the model characterization procedure employs tests conducted in strain control, prediction of the stress-controlled experiments is a more rigorous test of the modeling capabilities of the VBOP and the associated model parameters. Figure 5 compares predictions with the experimental results produced in creep tests of 6 h duration conducted at 21 MPa (naturally, in stress control), preceded by strain-controlled loading at $10^{-4}$ $s^{-1}$. Figure 5 presents predictions and data for PMR-15 neat resin subjected to prior aging for 100, 250, 500, and 2000 h. In the case of the material aged for 100, 250, and 500 h, the model characterization procedure was employed to determine the model parameters from experimental data. In contrast, eqs. (7)–(10) were employed to calculate the model parameters for the material aged for 2000 h (see Table II). Thus the results in Figure 5 provide means for a more thorough verification of the VBOP formulation extended to account for the effects of prior aging. It is seen that the predictions in Figure 5 are in excellent agreement with the experimental data. The model predicts the effect of prior aging time on creep behavior well.

**TABLE II**
**VBOP Parameters at 288°C for PMR-15 Neat Resin Subjected to Prior Aging at 288°C in Argon for 2000 h**

| | | | |
|---|---|---|---|
| Moduli | $E = 2.32$ GPa | $E_t = 60$ MPa | |
| Isotropic stress | $A = 27.7$ MPa | | |
| Viscosity function | $k_1 = 1.0 \times 10^4$ s | $k_2 = 35$ MPa | $k_3 = 12$ |
| Shape function | $C_1 = 100$ MPa | $C_2 = 1.37$ GPa | $C_3 = 10$ |

The model parameters calculated for the PMR-15 polymer aged for 2000 h were also used to generate predictions of stress–strain response to monotonic loading at various strain rates in Figure 6. The VBOP extended formulation predicts experimental results in Figure 6 very well. The effect of prior aging for 2000 h on the rate-dependent deformation behavior of the material is accurately captured by the model.

## CONCLUDING REMARKS

The VBOP was extended to model the deformation behavior of the PMR-15 neat resin subjected to prior isothermal aging. Based on experimental observations, the elastic modulus, the tangent modulus, the isotropic stress, and the parameter $C_2$ of the shape function were made dependent on prior aging time. The viscosity function remained unaffected by prior aging. The modified VBOP was used to reproduce the deformation response of the PMR-15 polymer aged for up to 1000 h during monotonic loading at various strain rates as well as during relaxation tests. The simulations were in good agreement with experimental data. A more rigorous verification of the extended VBOP formulation was accomplished by comparing model predictions with experimental results produced in tests that differ in kind from those used to characterize the model parameters. The model predictions were compared with the experimental results obtained for PMR-15 neat resin subjected to 2000 h of prior aging in both strain-

controlled tension to failure tests and stress-controlled creep tests. The model extended to capture the effects of prior aging accurately predicted the inelastic behavior of the material subjected to prior isothermal aging for up to 2000 h.

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