

# Effects of Prior Aging at 288°C in Air and in Argon Environments on Creep Response of PMR-15 Neat Resin

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**ABSTRACT:** The creep behavior of PMR-15 neat resin, a polyimide thermoset polymer, aged in air and in argon environments at 288°C for up to 1000 h was evaluated. Creep tests were performed at 288°C at creep stress levels of 10 and 20 MPa. Creep periods of at least 25-h in duration were followed by 50-h periods of recovery at zero stress. Prior isothermal aging increased the elastic modulus and significantly decreased the polymer's capacity to accumulate creep strain. The aging environment had little influence on creep and recovery behaviors. However, aging in air dramatically degraded the tensile strength of the material. Dynamic mechanical analysis revealed an increase in the glass transition temperature from ~330°C to ~336°C after 1000 h in argon or in air at 288°C. The rise in the glass transition temperature with aging time is

attributed to an increase in the crosslink density of the PMR-15 polyimide. Increase in the crosslink density due to aging in both air and argon environments is likely behind the changes in the elastic modulus and the decreased capacity for inelastic straining. A visibly damaged surface layer of ~0.16 mm thickness was observed in specimens aged in air for 1000 h. Results indicate that the unoxidized core material governs the overall mechanical response, whereas the oxidized surface layer causes a decrease in tensile strength by acting as a crack initiation site and promoting early failures. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 111: 228–236, 2009

**Key words:** aging; creep; high temperature materials; polyimides

## INTRODUCTION

Thermosetting polymers are widely used as matrix materials in fiber-reinforced composites in a broad range of applications, including aerospace, automotive, and oil and gas industries.<sup>1</sup> The growing interest in the use of polymer matrix composites in critical load-bearing structures mandates extensive knowledge of the mechanical behavior as well as of the durability of these materials. To analyze or predict the behavior of the composite material, it is necessary to evaluate the contribution of the matrix to the overall response of the composite. Among thermosetting polyimide resins, PMR-15 is of particular interest because of its superior high-temperature properties and ease of processing.<sup>1–3</sup> Designed for use at temperatures near its glass transition temperature ( $T_g$ ), the PMR-15 resin is extensively used as a matrix material in high-temperature structural com-

posites for aerospace applications. The carbon/PMR-15 composites, capable of service temperatures up to 300°C, are excellent candidates for use in structural components such as aircraft engines and engine exhaust washed structures, as well as high-speed aircraft skins.<sup>4–9</sup> These applications require extended exposures to temperatures approaching the  $T_g$  of the polymer matrix and to oxidizing environments, which cause chemical changes as well as thermal degradation of the glass forming matrix material.<sup>5,6,10</sup> The chemical changes occurring in the polymer matrix result in changes in its mechanical response and properties. To assure long-term durability and structural integrity of the polyimide composite components, a thorough understanding of the effects of the exposure to elevated temperature in oxidizing environment on the mechanical behavior of the polyimide matrix is essential.

Significant progress has been made in the understanding of the effects of isothermal aging on the thermooxidative stability of PMR-15 neat resin and PMR-15-based composites.<sup>5–8,10–12</sup> Bowles and co-workers<sup>5–7</sup> aged PMR-15 specimens in air at 288, 316, and 343°C for up to 4000 h. It was found that while thermal degradation occurred throughout the material, the oxidative degradation occurred mainly within a thin surface layer where oxygen diffused into the material. It was observed that the oxidized surface layer, which had different properties than

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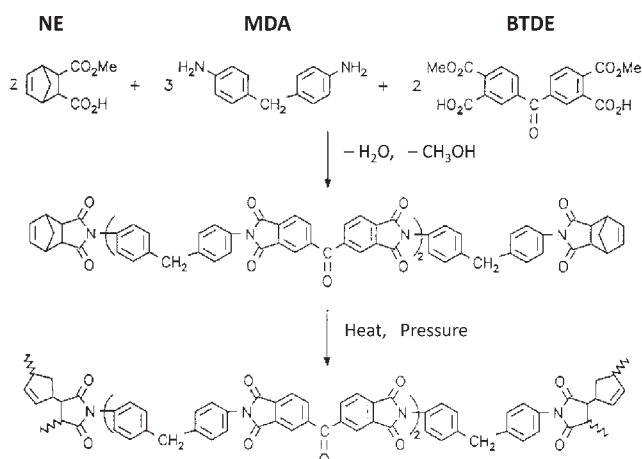


Figure 1 PMR-15 chemistry.

the unoxidized interior material, developed and grew during thermal aging. However, at all aging temperatures the thickness of the oxidized layer asymptotically approached a constant value of about 170  $\mu\text{m}$  as the aging time increased. Recent studies<sup>13–18</sup> document the growth of the thermooxidative layer and the changes in the elastic moduli and chemical composition resulting from isothermal aging. Ripberger et al.<sup>15</sup> reported that the thickness of the oxidation layer was approximately 55  $\mu\text{m}$  after 50 h of aging at 343°C and varied between 107 and 129  $\mu\text{m}$  after 342 h at 343°C. Furthermore, the aforementioned studies revealed that the interior core of the PMR-15 specimens was protected from oxidative degradation by the surface layer and remained relatively unchanged during thermal aging.

Most published research is focused on thermal oxidation of neat polymer systems. There are limited studies that consider the effects of thermal aging on the mechanical performance of high temperature polymers. While several research efforts evaluated the effects of aging on the tensile and compressive properties of PMR-15 based composites,<sup>7,8,12,18</sup> the effects of isothermal aging on time-dependent mechanical behavior of PMR-15 neat resin at elevated temperatures remain largely unexplored. For successful application of PMR-15 based composites in advanced aerospace system, the long-term durability characteristics of the constituent materials must be established under realistic service environments. In particular, the effect of thermal aging on high-temperature creep response of the PMR-15 polyimide needs to be thoroughly understood. The objective of this study is to investigate the influence of thermal aging at a temperature near the  $T_g$  of the PMR-15 neat resin on creep behavior of this polymer at elevated temperature. Creep tests at stress levels of 10 and 20 MPa were conducted at 288°C on specimens subjected to prior thermal aging at 288°C in an air or in argon environment. Results reveal that prior

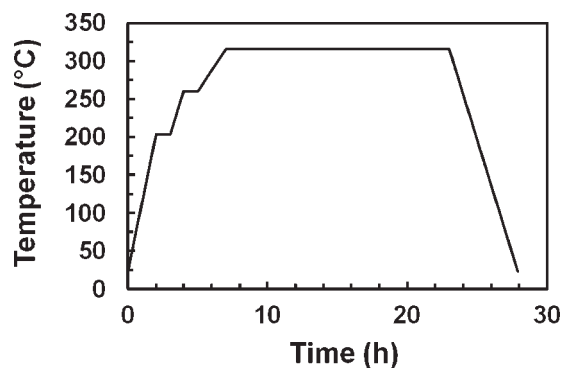


Figure 2 Freestanding postcure cycle.

aging has a noticeable effect on  $T_g$ , as well as on high-temperature tensile properties and tensile creep performance of the PMR-15 polyimide.

#### MATERIAL AND EXPERIMENTAL ARRANGEMENTS

The material studied was PMR-15 solid polymer, a thermosetting polyimide used as a matrix material for high-temperature polymer–matrix composites. Chemical structure of the PMR-15 polymer is shown in Figure 1. The PMR-15 polymer has a  $T_g$  of  $\sim 340^\circ\text{C}$  and a long-term use temperature of 288°C. The PMR-15 neat resin panels were supplied by HyComp, Inc. (Cleveland, OH) and were postcured at the Air Force Research Laboratory Materials and Manufacturing Directorate (Wright-Patterson Air Force Base, OH). The free-standing postcure cycle is shown in Figure 2. Dogbone-shaped test specimens shown in Figure 3 were machined from the panels using diamond grinding. All specimens were washed with a common household soap and thoroughly rinsed with distilled water to remove contaminants from the machining process. The specimens were then dried in a vacuum oven at 110°C for at least 24 h and were subsequently stored in a nitrogen-purged desiccator until aging.

Specimens were aged at 288°C, which is the design maximum sustained service temperature of PMR-15-based composites in aerospace applications.

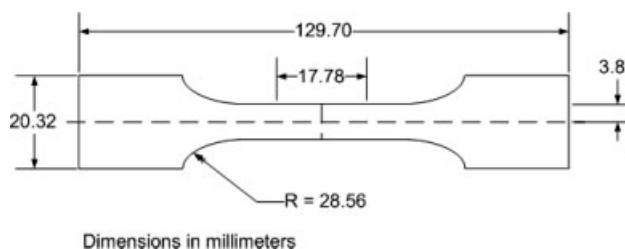
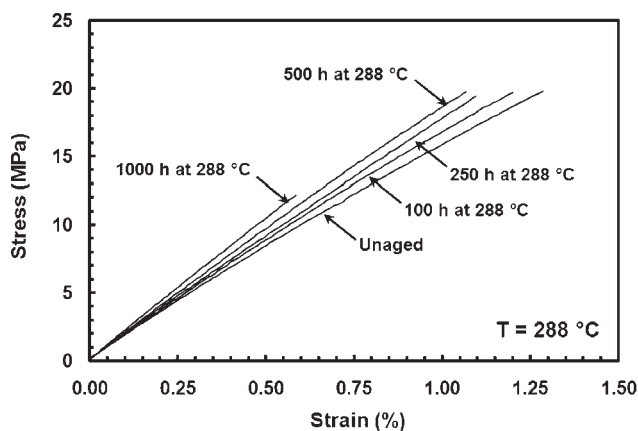


Figure 3 Test specimen (dimensions are given in millimeters).

The isothermal aging in air was accomplished in a Blue M model 7780 air-circulating oven that provided continuous replenishment of oxygen in ambient air by convection through the oven inlet. Air was supplied to the Blue M oven via a compressed air line with an inline filter and a desiccator. The measured relative humidity of the air fed to the oven chamber was less than 5% at room temperature. To isolate the effect of oxidation on material behavior, specimens were also aged in an inert gaseous environment. In this case, high purity argon gas (99.999% pure) was supplied to the Blue M oven from a liquid argon tank. When specimens were taken out for periodic inspection and/or testing, the oven was opened without cooling and closed immediately. Then the oven automatically entered the 18-min purge cycle to flush out any ambient atmosphere that had entered the chamber. The flow rate of argon (dry air when aging in air) was ~30 SCFH during the steady state operation and 150 SCFH during the purge cycle.

Each group of test specimens isothermally aged for a given duration in a given environment included a rectangular sample for the purpose of monitoring weight change and oxidized surface layer growth with aging time in air and in argon. The aged rectangular specimens were removed from the oven and allowed to cool in the desiccator prior to obtaining weight measurement. To observe the growth of the oxidized surface layer, a section of the aged rectangular specimen was cut, mounted in epoxy, polished and examined with an optical microscope (Nikon EPIPHOT digital microscope).

A servocontrolled MTS mechanical testing machine equipped with hydraulic water-cooled wedge grips, a compact resistance-heated furnace, and a temperature controller were used in all tests. An MTS Test-Star II digital controller was employed for input signal generation and data acquisition. Strain measurement was accomplished with an MTS high-temperature air-cooled uniaxial extensometer of 12.5-mm gage length. For elevated temperature testing, thermocouples were attached to test specimens using Kapton tape to calibrate the furnace on a periodic basis. The furnace controller (using a non-contacting thermocouple exposed to the ambient environment near the test specimen) was adjusted to determine the power setting needed to achieve the desired temperature of the test specimen. The determined power setting was then used in actual tests. Thermocouples were not attached to the test specimens after the furnace was calibrated. All mechanical tests were performed in laboratory air environment. In all elevated temperature tests, a specimen was heated to 288°C at a rate of 2°C/min and held at 288°C for an additional 30 min prior to testing. In all creep tests the specimens were loaded



**Figure 4** Tensile stress–strain curves for PMR-15 neat resin specimens aged at 288°C in air obtained during loading to the 20 MPa stress level with a stress rate of 1 MPa/s at 288°C.

to the creep stress level at the stress rate of 1 MPa/s. Fracture surfaces of failed specimens were examined using an optical microscope (Zeiss Discovery V12).

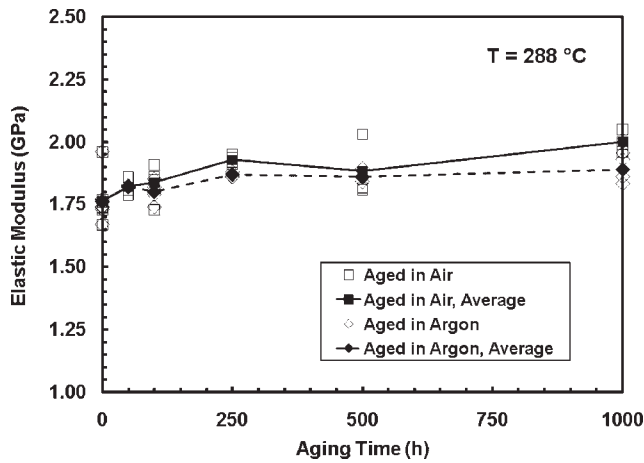
A Rheometric Scientific ARES 3A1 dynamic mechanical analyzer with a nitrogen-purged test chamber was used to measure the  $T_g$  values of the unaged specimens, specimens aged in air, and specimens aged in argon.

## RESULTS AND DISCUSSION

The influence of prior thermal aging on the mechanical behavior of the PMR-15 neat resin was explored in creep and recovery tests conducted at 288°C on specimens aged for various durations in air and in argon. The aging times were 10, 50, 100, 250, 500, and 1000 h for both environments.

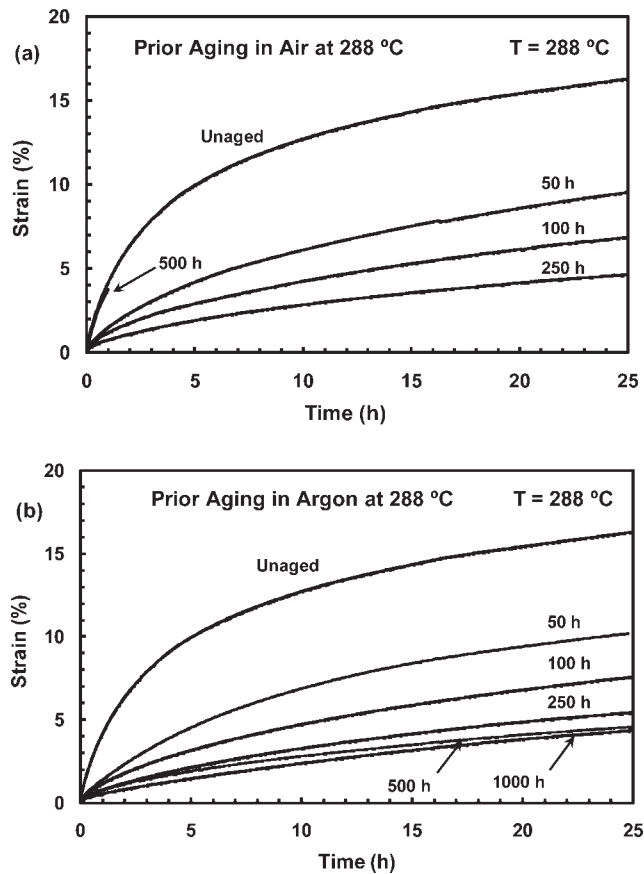
### Monotonic stress–strain behavior–effect of prior aging

Previous work<sup>19</sup> showed the average ultimate tensile strength of the PMR-15 polymer supplied for this study to be ~30 MPa at 288°C. Therefore, creep stress levels of 10 and 20 MPa were selected for the experimental effort. The load-up segments of the 20 MPa creep tests were used to evaluate the effects of prior aging on the monotonic tensile stress–strain behavior and on the tensile modulus. The stress–strain response obtained during loading to 20 MPa at a stress rate of 1 MPa/s of specimens aged in air is typified in Figure 4. It is seen that the qualitative stress–strain behavior is unaffected by prior aging. Similar observation was made for the specimens aged in argon. The effect of prior aging on the elastic modulus is illustrated in Figure 5. The elastic modulus increases with prior aging time. Modulus increase is somewhat more pronounced for

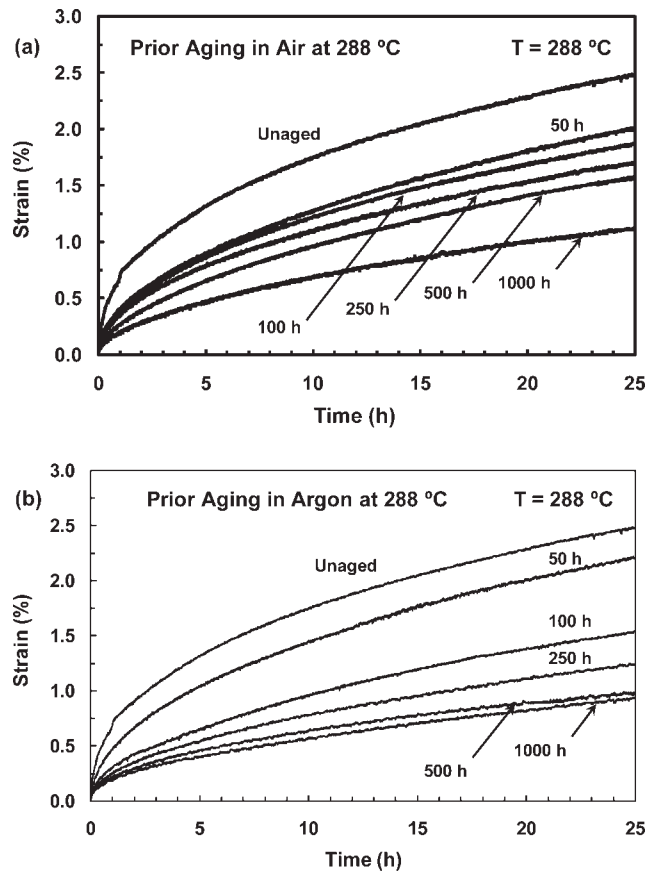


**Figure 5** Elastic modulus at 288°C versus aging time for PMR-15 neat resin specimens aged at 288°C in air and in argon.

specimens aged in air than for those aged in argon. The average increase in modulus after 1000 h at 288°C was ~13% for aging in air and 7% for aging in argon. It is noteworthy that prior aging in air for ≥500 h also caused a drastic decrease in the tensile

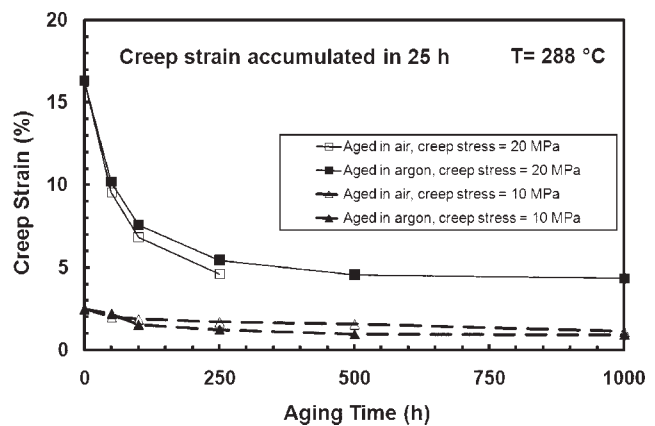


**Figure 6** Creep strain versus time at 20 MPa and 288°C for PMR-15 specimens aged at 288°C (a) in air and (b) in argon. The effect of prior aging time on creep strain is apparent.



**Figure 7** Creep strain versus time at 10 MPa and 288°C for PMR-15 specimens aged at 288°C (a) in air and (b) in argon. The effect of prior aging time on creep strain is apparent.

strength. Of the three specimens aged in air for 500 h one failed at 19.7 MPa and two failed shortly after reaching 20 MPa. Three specimens aged in air for 1000 h failed at even lower stress levels of 12.2, 13.1, and 13.8 MPa.



**Figure 8** Creep strain accumulation as a function of prior aging time for PMR-15 specimens aged at 288°C in air and in argon. Creep stress levels = 10 and 20 MPa. Creep test temperature = 288°C.

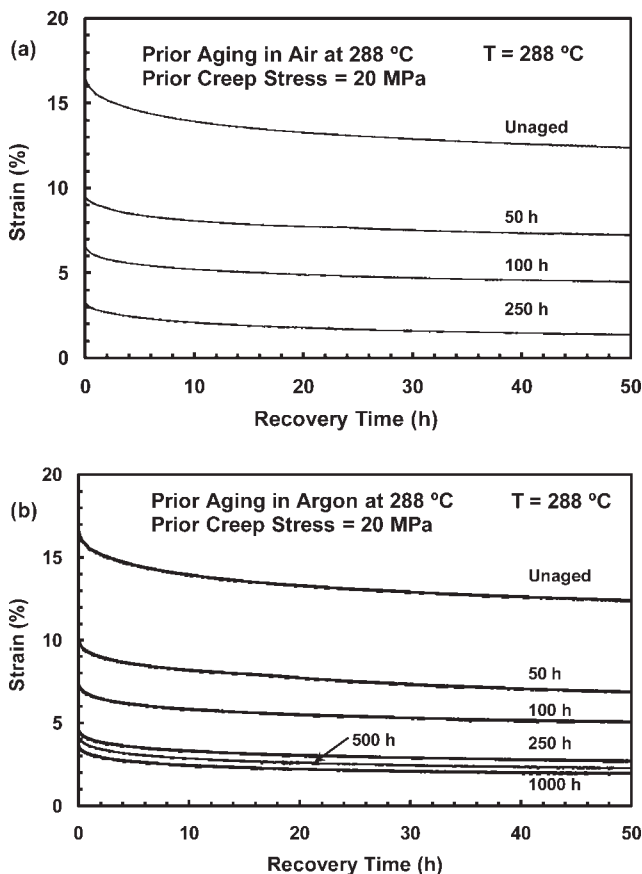
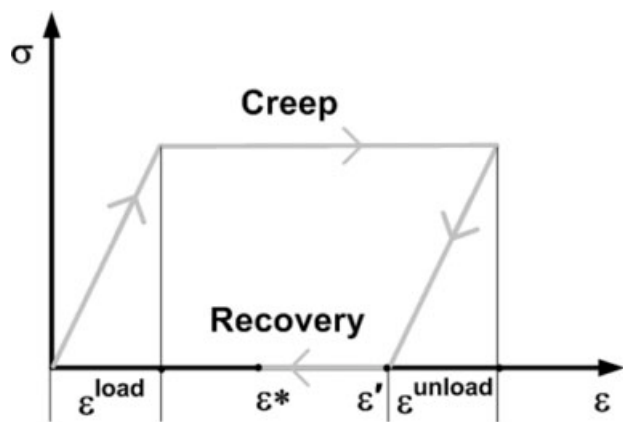


Figure 9 Strain versus time curves obtained during recovery at zero stress preceded by creep at 20 MPa at 288°C for PMR-15 specimens aged at 288°C (a) in air and (b) in argon.



Creep strain:  $\epsilon^{creep} = \epsilon' + \epsilon_{unload} - \epsilon_{load}$   
 Recovered strain:  $\epsilon^r = \epsilon' - \epsilon^*$   
 Percent Creep Strain Recovered =  $\frac{\epsilon^r}{\epsilon^{creep}} \times 100\%$

Figure 10 Schematic of the stress-strain behavior during the creep and recovery test.

### Creep behavior–influence of prior aging

Effect of prior aging on creep behavior was explored in creep tests at 10 and 20 MPa conducted on specimens subjected to prior aging of different durations in air and in argon. Creep time was set to 25 h. Results of the 20 MPa creep tests are presented in Figure 6(a,b) for specimens aged in air and in argon, respectively. Creep strain versus time curves obtained at 10 MPa are shown in Figure 7(a,b) for specimens aged in air and in argon, respectively. Effect of prior aging time and environment on creep strain accumulation is illustrated in Figure 8.

Creep curves produced in all tests exhibit primary and secondary creep regimes, regardless of the prior aging history. However, creep strain accumulation is strongly influenced by the prior aging. The amount of creep strain accumulated in 25 h decreases with increasing prior aging time. For aging times  $\leq 250$  h, the aging environment has little effect on creep response. However, for aging times exceeding 250 h, aging in air dramatically degrades tensile strength of the PMR-15 polymer. The tensile strength of specimens aged in air for 500 h was reduced to  $\sim 20$  MPa.

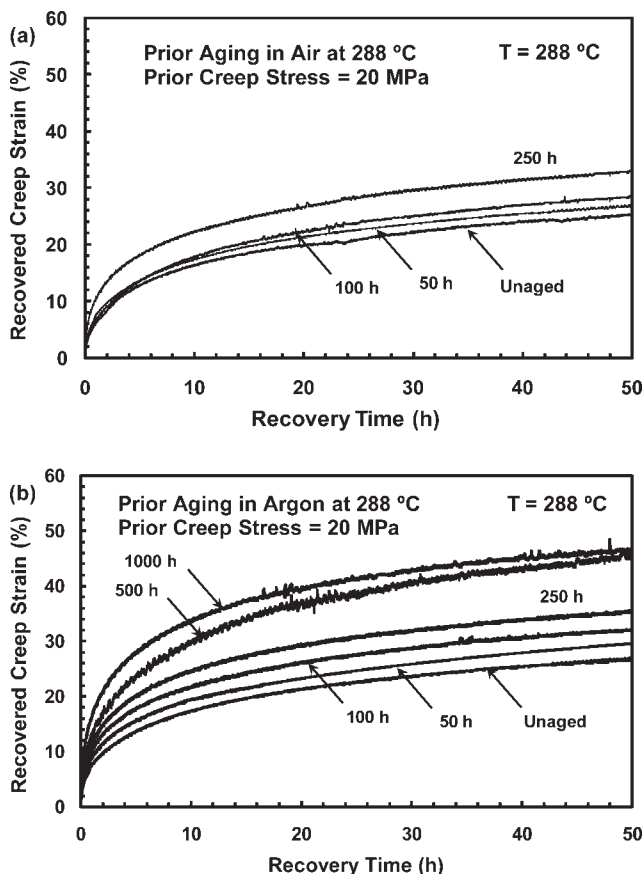
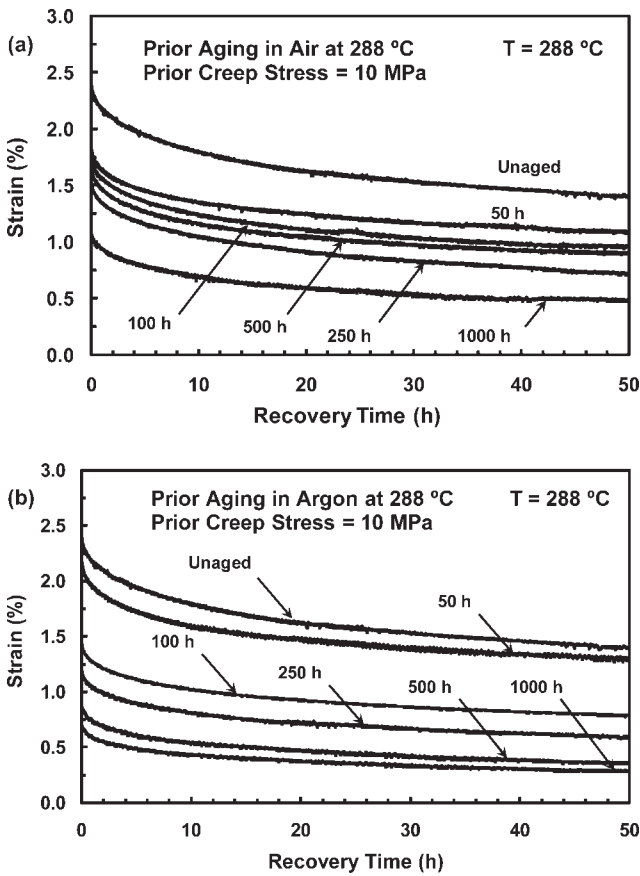


Figure 11 Percent creep strain recovered as a function of recovery time for PMR-15 specimens aged at 288°C (a) in air and (b) in argon. Prior creep stress = 20 MPa.

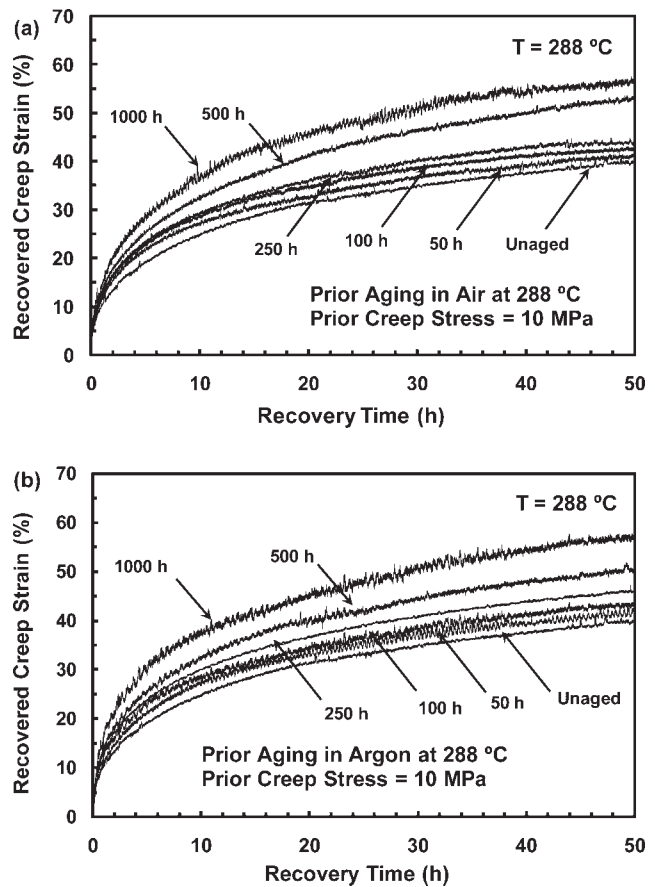


**Figure 12** Strain versus time curves obtained during recovery at zero stress preceded by creep at 10 MPa at 288°C for PMR-15 specimens aged at 288°C (a) in air and (b) in argon.

Consequently the two specimens that reached the creep stress of 20 MPa produced short creep lifetimes of <1 h. The specimens aged in air for 1000 h failed before reaching the creep stress of 20 MPa. In contrast, all specimens aged in argon survived the 25 h of creep at 20 MPa. Note that all specimens regardless of the prior aging history survived 25 h at 10 MPa. For a given prior aging time in argon environment, lower creep strains were accumulated at 10 MPa than at 20 MPa. Prior aging in air for over 250 h, which severely decreased specimen lifetimes in the 20 MPa creep tests, had no influence on creep lifetimes at the lower stress of 10 MPa. In the case of specimens aged in air for  $\leq 250$  h, creep strains obtained at 10 MPa are lower than those obtained at 20 MPa for a given prior aging time.

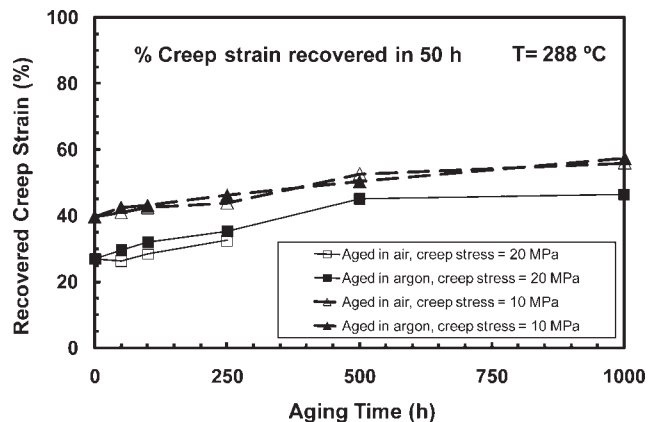
**Recovery at zero stress—influence of prior aging**

All specimens that survived 25 h at creep stress were unloaded to zero stress at the rate of 1 MPa/s and allowed to recover at zero stress for 50 h. Results of the recovery tests following creep at 20 MPa are summarized in Figure 9(a,b) for speci-

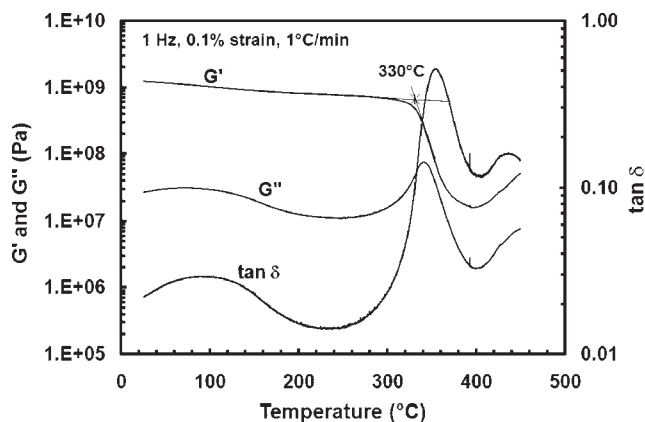


**Figure 13** Percent creep strain recovered as a function of the recovery time for PMR-15 specimens aged at 288°C (a) in air and (b) in argon. Prior creep stress = 10 MPa.

mens aged in air and in argon, respectively. Permanent strain is observed in all tests after 50 h of recovery. Furthermore, results demonstrate that aging environment has little qualitative effect on the recovery response. To gain a better insight into the recovery behavior, percent creep strain recovered,



**Figure 14** Percent creep strain recovered as a function of the prior aging time for PMR-15 specimens aged at 288°C in air and in argon. Prior creep stress levels = 10 and 20 MPa.



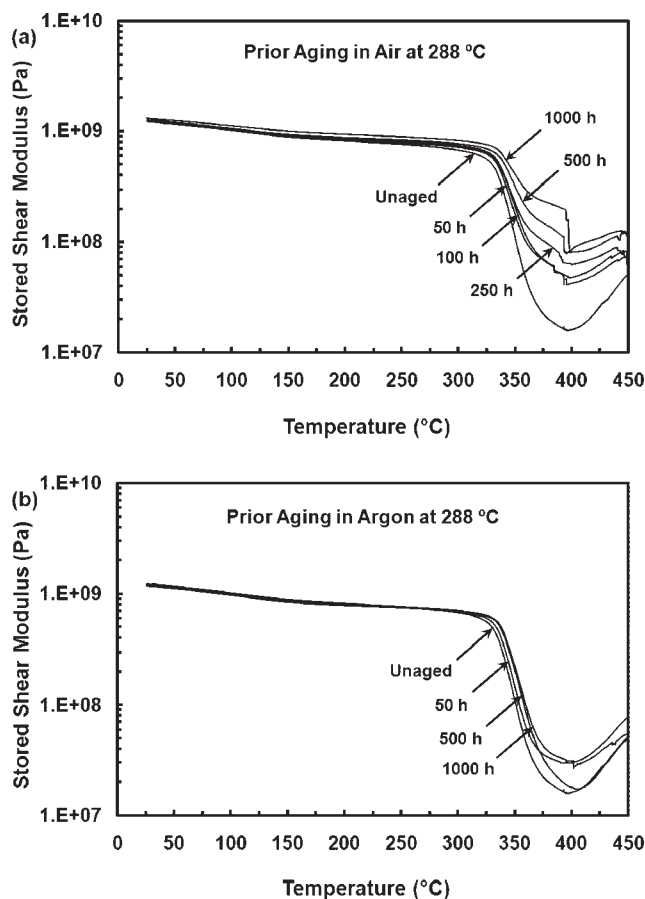
**Figure 15** Components of the complex shear modulus as functions of temperature for the unaged PMR-15 neat resin ( $G'$  = storage modulus;  $G''$  = loss modulus).

defined according to the schematic in Figure 10, is plotted as a function of recovery time for each test (see Fig. 11). It is seen that percentage of recovered creep strain increases with increasing prior aging time in both air and argon. Similar observations can be made regarding recovery at zero stress preceded by creep at 10 MPa. Strain versus recovery time curves presented in Figure 12 as well as the percent creep strain recovered versus recovery time curves in Figure 13 reveal that a greater percentage of creep strain was recovered for specimens with longer prior aging time. These results are further illustrated in Figure 14, where the percent creep strain recovered is presented as a function of prior aging time. Notably aging environment has negligible influence on the recovery behavior.

### Dynamic mechanical analysis

$T_g$  values of the unaged material and of the specimens subjected to prior aging were measured by dynamic mechanical analysis. The test pieces were stressed in torsion across the specimen width. The heating rate was 1°C/min, the frequency was 1 Hz, and the shear strain was 0.1%. The two components of the complex shear modulus were measured as a function of temperature (see Fig. 15 depicting the results obtained for the unaged material).  $T_g$  was determined by measuring the intersection of the two tangents to the linear portions of the stored shear modulus curve where it abruptly drops to a minimum. The average  $T_g$  value for the unaged material was 330°C.

Effect of prior aging on stored shear modulus is presented in Figure 16(a,b) for specimens aged in air and in argon, respectively.  $T_g$  as a function of prior aging time is shown in Figure 17. It is seen that the aging environment has only negligible influence on  $T_g$ .  $T_g$  increases with increasing prior aging time in

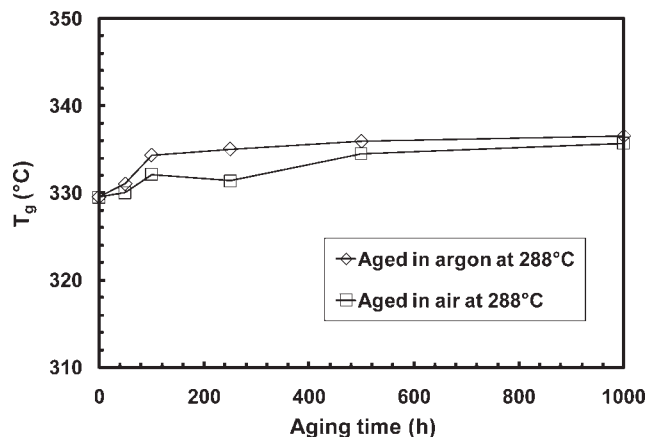


**Figure 16** Stored shear modulus as a function of temperature for PMR-15 specimens aged for various durations at 288°C (a) in air and (b) in argon.

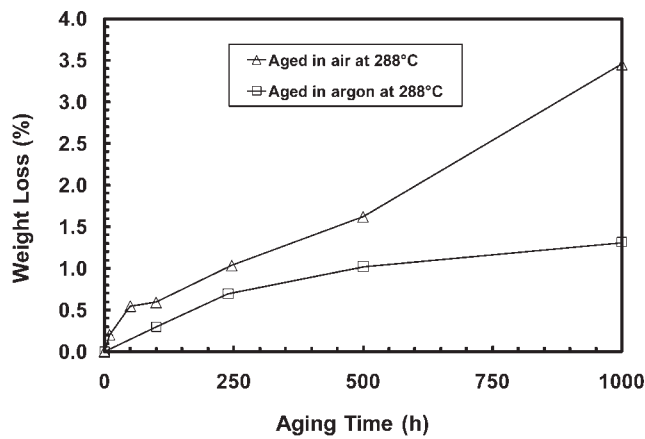
both air and argon, indicating that crosslinking continues during aging at 288°C.

### Thermooxidative stability

The average weight loss as a function of prior aging time is shown in Figure 18. As expected, the weight decreases with increasing prior aging time. The



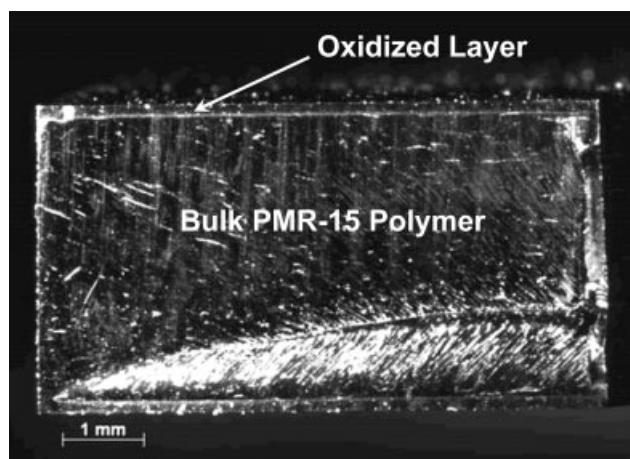
**Figure 17**  $T_g$  as a function of prior aging time for PMR-15 specimens aged at 288°C in air and in argon.



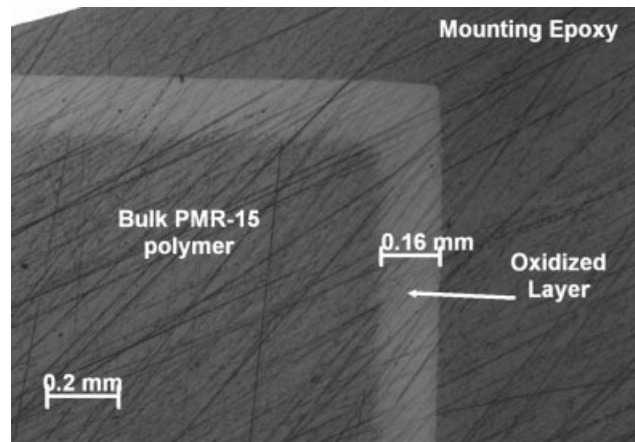
**Figure 18** Weight loss as a function of prior aging time for PMR-15 specimens aged at 288°C in air and in argon.

aging environment has a significant influence on the weight loss. The weight loss for specimens aged for 1000 h in air is three times that for specimens aged for 1000 h in argon. While the weight loss in argon may be attributed to loss of low molecular weight volatiles, the weight loss in air occurs due to the loss of volatiles as well as due to the chemical/oxidative degradation.

Figure 19 shows the fracture surface of a PMR-15 specimen aged for 1000 h at 288°C in air. A micrograph of a sectioned surface of an untested PMR-15 specimen aged for 1000 h at 288°C in air is presented in Figure 20. An oxidized surface layer is clearly visible in both Figures 19 and 20. The thickness of the damaged layer in both specimens is fairly uniform. Average growth of the oxidized layer is shown in Figure 21 as a function of aging time at 288°C in air. The oxidized layer growth data were collected for specimens aged for a given duration, then failed in mechanical tests. In addition, the oxidized layer measurements were recorded for specimens aged for a given duration, then sectioned



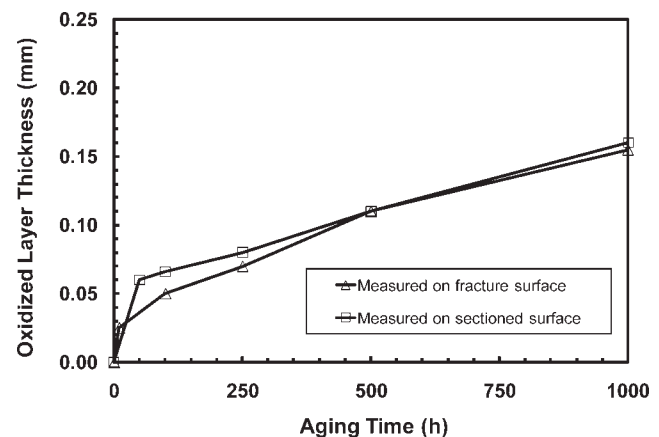
**Figure 19** Fracture surface of a PMR-15 specimen aged for 1000 h at 288°C in air (stereo-optical microscope).



**Figure 20** Sectioned surface of an untested PMR-15 specimen aged for 1000 h at 288°C in air (optical microscope, bright-field imaging).

without being subjected to mechanical testing. It is seen that for a given prior aging time, the oxidized layer measurements obtained on the fracture surfaces of tested specimens are consistent with those obtained on the sectioned surfaces of untested specimens. After 1000 h of aging at 288°C in air, the thickness of the oxidized layer approaches 0.16 mm. Note that a pathological layer was not observed in specimens aged in argon.

Results of the present study reveal that the mechanical behavior of the PMR-15 specimens aged in air for  $\leq 250$  h is essentially the same as that of the specimens aged in a nonoxidizing environment. The oxidation in high-temperature polymers is primarily a surface reaction phenomenon controlled by the diffusion of oxygen into the polymer and the rate of oxidative chemical reactions.<sup>20</sup> The oxidation process is generally limited by the amount of oxygen that can diffuse into and react with the polymer. As a result, the oxidized layer develops near the free surfaces, while the interior of the specimens aged in air



**Figure 21** Thickness of the oxidized surface layer in PMR-15 neat resin as a function of aging time at 288°C in air.



remains unoxidized. As the thickness of the oxidized surface layer is very small compared to the overall cross-sectional dimensions of the specimen, the unoxidized core material governs the mechanical response of the specimens aged in air. These findings confirm the assumption of Tsuji et al.<sup>6</sup> that the mechanical properties of specimens aged in a nonoxidizing environment are similar to those of the unoxidized core material of specimens aged in air.

In the case of the PMR-15 neat resin specimens aged in air for  $\geq 500$  h, continued surface oxidation leads to a decrease in local density and shrinkage of the oxidized layer producing surface cracks.<sup>20</sup> Because inward diffusion of oxygen proceeds slower than the outward diffusion of degradation byproducts, voids form in the oxidized layer concentrating near the surface.<sup>14,20</sup> It is likely that the severely damaged surface layer acts as a crack initiation site, causing early failures and dramatically degrading the tensile strength of the specimens aged in air for  $\geq 500$  h.

## CONCLUSIONS

Monotonic tensile stress-strain behavior and tensile modulus of PMR-15 neat resin aged at 288°C in air and in argon for various durations were evaluated at 288°C. Prior aging has little effect on the qualitative stress-strain behavior. The elastic modulus increases with increasing prior aging time. Prior aging in air for over 250 h dramatically degrades the tensile strength of the material.

Effects of prior aging on creep and recovery response were also evaluated. Creep strain accumulation decreases with increasing prior aging time. Aging environment has little influence on creep strain accumulation. Greater percentage of creep strain is recovered for specimens subjected to prior aging of longer duration. Aging environment has negligible effect on recovery of strain. Permanent strain after 50 h of recovery at zero stress was observed in all tests.

$T_g$  increases with increasing prior aging time, indicating an increase in crosslink density. The aging environment has no effect on changes in  $T_g$ . Higher values of  $T_g$  are likely behind the increased elastic modulus and reduced creep strains produced by aged specimens.

The thermooxidative stability of the PMR-15 neat resin was assessed. The average weight loss after 1000 h at 288°C in air was 3.5%, while the average weight loss after 1000 h at 288°C in argon was limited to 1.3%. For specimens aged in argon, the weight loss is attributed to a loss of low-molecular-weight volatiles. The chemical/oxidative degradation and the loss of low volatiles are behind the weight loss of specimens aged in air. A visibly damaged surface layer is observed in specimens aged in

air, while no pathological surface layer is observed in specimens aged in argon. The average thickness of the oxidized layer in specimens aged in air for 1000 h at 288°C is  $\sim 0.16$  mm. It is believed that while the unoxidized core material governs the mechanical response of the PMR-15 neat resin, the oxidized surface layer promotes early failures by acting as a crack initiation site.

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