Viscoelastic Characterization of Polymers for Deployable Composite Booms

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Abstract

Deployable space structures have been built from thin-walled fiber-reinforced polymer composite materials due to their high specific strength, high specific stiffness, and bistability. However, the inherent viscoelastic behavior of the resin matrix can cause dimensional instability when parts are stored under strain. The extended time of stowage between assembly and deployment in space can result in performance degradation and in the worst case, mission failure. In this study, the viscoelastic properties of candidate commercial polymers for deployable boom structures of solar sails were evaluated. Stress relaxation master curves of the candidate polymers were used to predict the relaxation that would occur in 1 year at room temperature under relatively low strains of ~0.1%. A bismaleimide (BMI) showed less stress relaxation (about 20%) than the baseline Novolac epoxy (about 50%). Carbon fiber composites fabricated with the BMI resin showed a 44% improvement in relaxation compared to the baseline epoxy composite. Other difunctional and tetrafunctional epoxies, thermoplastic, and thermosetting polyimides were also evaluated.

Keywords: Deployable Structure, Relaxation, Viscoelastic, Polymer

Nomenclature

T	Temperature
E_a	Activation energy
R	*Boltzman constant*
f	*Frequency*
TGDDM	4,4′-Methylenebis(*N*,*N*-diglycidylaniline)
DDS	4,4′-Diaminodiphenyl sulfone
DMA	Dynamic mechanical analyser
TTS	Time-temperature superposition principle
BMI	Bismaleimide

Subscripts

g	Glass transition
a	*Activation*

1. Introduction

Deployable structures have been used to erect solar sails, solar arrays, antennas, payload booms, planetary decelerators, from the confined volume of launch vehicles [1]. The large space structure is packaged prior to launch, and deployed to the designed configuration at the mission location. Deployable space structures have been built from fiber reinforced polymer composite materials due to their low mass, high specific strength and high specific stiffness [2], but dimensional instability is a critical issue. The inherent viscoelastic behavior of polymers and the extended time of compacted stowage between assembly and deployment in space can result in performance degradation and, in the worst case, mission failure. An example of the

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reduction in boom cross section after 1 month of stowage at room temperature is shown in Figure 1. The boom becomes flattened, losing flexural strength. The area moment of inertia of a thin beam depends on the distance of material from the bending axis cubed. For example, a 25% reduction in total boom width will result in a loss of approximately 33% of the area moment of inertia [3], and this can result in the deployment failure.

The composites' relaxation originates from the viscoelastic behavior of polymeric materials. Under stress, parts of molecular chains or entire chains rearrange and slide past each other. Generally, thermosetting polymers can be expected to show less creep and stress relaxation compared to thermoplastic polymers due to restriction of chain motions by cross-linking [4]. Particulate additives like silica may also reduce creep [5], but only if the chains are securely bonded to the filler [6].

In this study, we investigated the viscoelastic properties of commercial polymers that are candidates for fabricating deployable booms. A quantitative study of stress relaxation information can provide design guidelines to increase reliability of deployable space structures such as deployable booms for solar sail systems.

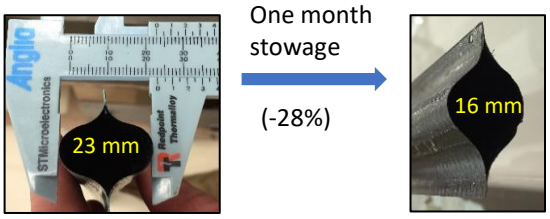

Fig. 1. Dimension change of boom due to relaxation during storage [3].

2. Experimental

2.1. Materials

Cured novolac epoxy (PMT-F7), the baseline for this study, was obtained from Patz Materials and Technology (CA, USA) and used without further treatment. Thermoplastic polyimides, LaRC™ SI and LaRC™ IA, and thermosetting polyimides, LaRC™ RP-46 and LaRC™ PETI-5 were obtained from Imitec and processed according literature recommendations [7-10]. Thermoplastic polyetherimide, ULTEM 1000 was obtained from GE plastics (MA, USA). Bismaleimides (BMI), Cycom® 5250-RTM and Compimide® 353RTM were obtained from Cytec Industries Inc. (NJ, USA) and Evonik Industries AG (North Rhine-Westphalia, Germany) and cured according to manufacturers' recommendations. Tetrafunctional epoxy, equivalent to Araldite® MY 720 (Huntsman Co., UT, USA), was prepared from 4,4′-Methylenebis(*N*,*N*-diglycidylaniline) (TGDDM, Sigma Aldrich, MO, USA) cured with 4,4′-Diaminodiphenyl sulfone (DDS, Sigma Aldrich, MO, USA) at a weight ratio of 1:0.38 (or 1:0.44). Resin and hardener were mixed at 110°C to get a homogenous mixture and degassed in a vacuum oven before curing. The mixed resin was cured at 120°C for 1 hour, 150°C for 2 hours and 177°C for 2 hours at a heating rate of 3°C/min. Post cure condition was 204°C for 4 hours, all under ambient pressure. The

Spread-tow, carbon fiber reinforced epoxy composite fabric (Toray M30S plain weave/PMT-F7) was obtained from Patz Materials and Technology (CA, USA) and used as the baseline material. Carbon fiber reinforced BMI composites were prepared using spread-tow Mitsubishi carbon fiber (MR60H, 24P carbon fiber, 24000 filament) from Sakai Ovex Co., Ltd. (Sakai, Japan) and Cycom's 5250-4 BMI resin. Overall, the fabrication process was similar to that used in the previous study [11]. A 50 wt% solution was prepared by dissolving the Cycom 5250-4 resin in a mixture of methyl ethyl ketone and cyclohexanone (1:1). The predetermined amount of BMI solution was painted onto the carbon fiber fabric to fabricate a "pre-infused" carbon fiber/BMI prepreg sheet with desirable weight fraction (about 50 wt%) of carbon fiber after drying in a convection oven at 110°C for 3 hours and in a vacuum oven at 60°C overnight. The dried prepreg was stacked according to the predetermined layup configuration and cured in an autoclave at 121°C for 1 hour and 177°C for 6 hours at the heating rate of 3°C/min. Pressure of 0.7 MPa was applied at the end of cycle of 121°C and released at the end of cure cycle. Post cure condition was 227°C for 6 hours at ambient pressure.

2.2. Viscoelastic Properties Test

Viscoelastic properties of candidate materials at low strain regimes (0.1%) were characterized from storage modulus and loss modulus at a heating rate of 1°C/min and a frequency of 1Hz using a dynamic mechanical analyser (DMA, Q800, TA Instruments). A dual cantilever fixture (for polymers) and 3-point bending fixture (for composites) were utilized. Stress relaxation behavior was characterized from relaxation in modulus at 20°C to 130°C in increments of 10°C. At each temperature, load was applied to get a strain of 0.1%. Relaxation was observed for 60 minutes and then the load was removed to allow 10 minutes for strain recovery. From the raw data, a master curve was created using time-temperature-superposition (TTS), and the percent decrease of relaxation modulus at 1 or 2 years was calculated. Complementary to this study, a recent research effort has evaluated, using the newly-developed Column Bending Test (CBT) method [12] for

large deformation bending of thin flexures, the viscoelastic behaviour of the boom's baseline matrix material (PMT-F7) and thin-ply composite laminates of interest under high strain regimes dictated by the boom application (> 1%) [13].

3. Results and Discussion

3.1. Stress Relaxation

In order to predict relaxation in a certain period of time, an accelerated stress relaxation test was performed using the TTS to create a master curve [14]. An example of creating a master curve is shown in Fig. 2. Individual stress relaxation data for the tetrafunctional epoxy sample were obtained at certain temperatures (Fig. 2) and the individual curves were shifted horizontally to form a continuous master curve as shown in Fig. 3. The credibility of the master curve was confirmed by a linear relationship between the natural log of the shift factor and inverse temperature (Arrhenius law). The predicted relaxation modulus at 20°C in 1 year (5.26×10^5 min) decreased by 26% from the initial modulus.

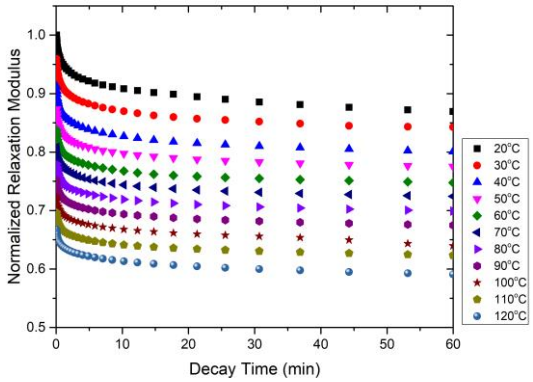

Fig. 2. Stress relaxation data of an MY720 epoxy sample at different temperatures

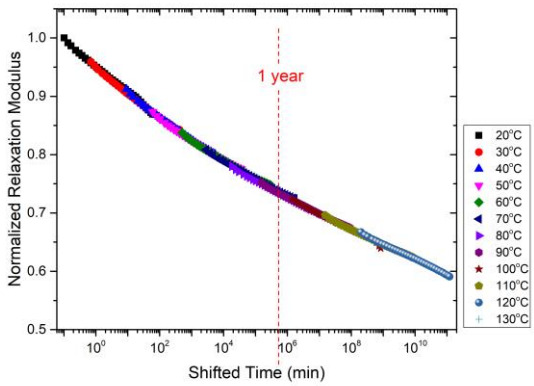

Fig. 3. A master curve of relaxation modulus created by the TTS method.

Various candidate commercial polymers were selected based on the feasibility of fiber reinforced composite fabrication for applications involving confined storage prior to deployment. The predictions for relaxation due to confined stowage over a moderate time period are summarized in Table 1.

Table 1. Percent relaxation of modulus of candidate polymers for deployable structures.

Sample	Specification	Manufacturer	% Relaxation at 1 year, 20°C
PMT-F7	Baseline, Novolac epoxy	Patz Materials and Technology	49
D.E.R. 332	Difunctional Epoxy, D.E.R. 332	Dow Corning	68
MY720-1	Tetrafunctional epoxy1 (1:0.38 ratio), Araldite® MY720	Huntsman	34
MY720-2	Tetrafunctional epoxy2 (1:0.44 ratio), Araldite® MY720	Huntsman	26
Ultem® 1000	Thermoplastic Polyetherimide	GE Plastics	73
LaRC™ IA	Thermoplastic Polyimide	Imitec, NASA LaRC	98
LaRC™ SI	Thermoplastic Polyimide	Imitec, NASA LaRC	29
LaRC™ RP46	Thermoset Polyimide, RP46	NASA LaRC	23
LaRC™ PETI-5	Thermoset Polyimide	Imitec, NASA LaRC	22
Compimide 353RTM	Bismaleimide (BMI)	Evonic	27
Cycom 5250-4	Bismaleimide (BMI)	Cytec	20

The baseline novolac epoxy material, PMT-F7, has a predicted 49% relaxation in modulus in 1 year at 20°C. A difunctional epoxy (DF epoxy) synthesized with 4,4'-isopropylidenediphenol diglycydyl ether (D.E.R. 332) showed higher relaxation of about 68% due to its lower crosslinking density. However, tetrafunctional epoxy synthesized with N,N'-tetraglycidyl diaminodiphenyl-methane (Araldite® MY720) showed less relaxation (26%) compared to the novolac epoxy, PMT-F7, because of its highly crosslinked molecular structure. Thermoplastic polyetherimide, ULTEM® 1000 and thermoplastic polyimide, LaRC™ IA have flexible molecular structures (ether linkages) and showed higher relaxations of about 73~98%. Thus, neither the ULTEM® nor LaRC™ IA is a good candidate for

deployable structures even though they have good processability. Another thermoplastic a soluble polyimide, LaRC™ SI, showed lower relaxation of about 29% due to the lower flexibility of its main chain. The molecular ordering and free volume might affect the relaxation, but further investigation is required. The thermosetting polyimides such as LaRC™ 46 polyimide and LaRC™ PETI-5 polyimide showed less relaxation (about 22~23%) due to hindrance of the molecular chain mobility by chemical crosslinking [4]. The bismaleimide (BMI) polymers such as Cycom 5250-4 and Compimide 353RTM showed low relaxation (20% for Cycom 5250-4, 26% for Compimide 353RTM) due to their higher glass transition temperatures and low-flexibility of molecular chains. The cross-linking density might affect the relaxation and further study is required. Among the tested materials, Cycom 5250-4 BMI and PETI-5 showed the lowest relaxation and are good candidates to be incorporated as matrix resins into composites for deployable structures.

3.2. Viscoelasticity

Viscoelastic dynamic mechanical spectra of the selected polymers were analysed at different frequencies (0.1, 0.32, 1, 3.16, 10, 31.6 and 100 Hz) to gain an understanding of their relaxation behavior. Fig. 4 shows the isochronal temperature scans of LaRC™ SI polyimide. Storage modulus gradually decreased as a function of temperature and rapidly decreased through the polymer glass transition temperature. Tan δ showed clear β- and α-relaxations which are responsible for reorganizations of molecular chain conformations (bending/stretching, side groups) and gross segmental movement (glass transition), respectively. The cooperative molecular motion is a kinetic transition (depending on temperature and frequency) [15]. Thus, as the frequency increases, the relaxation occurs at higher temperatures.

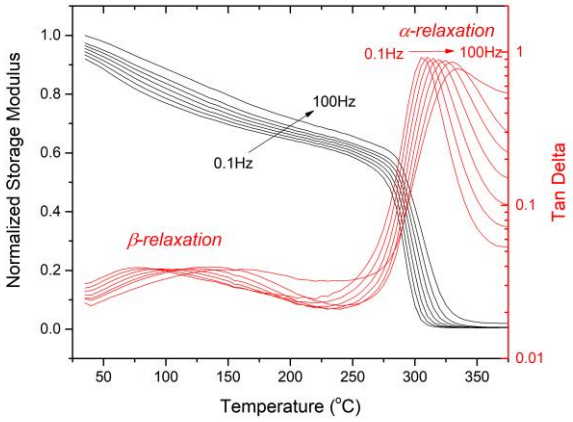

Fig. 4. Storage modulus and tan δ of LaRC™ SI polyimide as a function of frequency and temperature.

The activation energies of the α- and β-relaxations can be calculated with Arrhenius plots (Fig. 5-6). From the relationship between the natural log of frequency at the peak of Tan δ and inverse temperature, the activation energy was calculated by the following equation (1):

$$E_a = -R \left[\frac{d \ln f}{d1/T}\right] \qquad (1)$$

where E_a is activation energy, R is the Boltzmann constant, T is the temperature and f is the frequency of peak of relaxation. The activation energies are summarized in Table 2. Tetrafunctional epoxy (MY720) and Cycom 5250-4 showed abnormally large activation energies - over 1000 kJ/mol. This may have been caused by additional remnant curing during the DMA test because the sample was not fully cured by the manufacturer's recommendation. Thus, those values were not used for analysis. In addition, the β-relaxation peak of Cycom 5250-4 was too weak to determine the activation energy. Even though the entire data set was not obtained, there does seem to be a correlation of lower stress relaxation with higher activation energy of the β-relaxation. For example, compared to D.E.R. 332 epoxy (difunctional epoxy), LaRC™ RP 46 polyimide required higher energy for molecular chain rearrangement near room temperature and it also showed lower relaxation in modulus. This relationship is undergoing further study, as are other factors such as free volume.

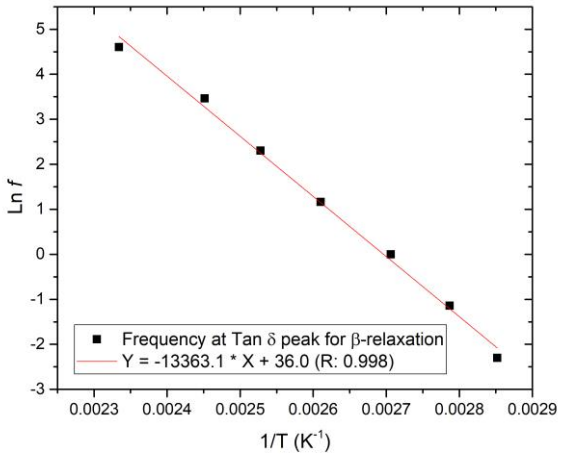

Fig. 5. Arrhenius plot of tan δ peak for β-relaxation of LaRC™ SI polyimide.

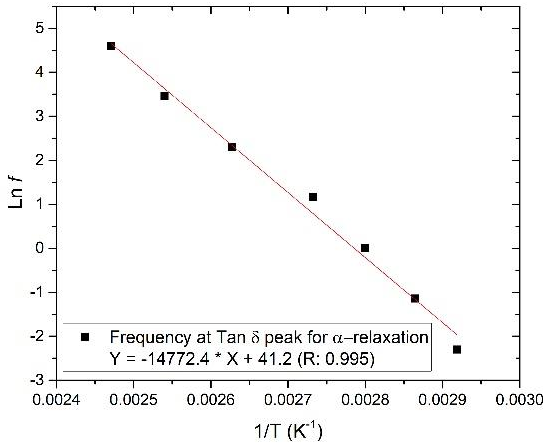

Fig. 6. Arrhenius plot of tan δ peak for α-relaxation of LaRC™ SI polyimide.

Table 2. Glass transition temperature and transitional activation energy.

Sample	Tg (Tan δ)	Activation Energy (KJ/mol)	
		β-relax	α-relax
D.E.R. 332	173	66	647
PMT-F7	248	70	601
MY720-2	270	-	-
LaRC™ SI	319	111	700
LaRC™ RP46	332	131	823
Cycom 5250-4	343	N/A	-

3.3. Carbon Fiber Reinforced Polymer Composites

The relaxation behavior of carbon fiber reinforced polymer composites was characterized for two polymer matrices, PMT-F7 epoxy as a baseline and Cycom 5250-4 BMI as a low-relaxation polymer. Two different layup configurations of thin-ply, spread-tow carbon fiber plain weave fabric (Toray M30S / PMT-F7 epoxy) were used as baselines: [+45°$_{pw,2}$/-45°$_{pw,2}$] , representative of a highly flexible boom (CF45/Epoxy) and [0°$_{pw,2}$/90°$_{pw,2}$] to represent a highly stiff boom (CF0/Epoxy). Sakai Ovex Co.. Ltd. (Sakai, Japan) spread and wove a thin-ply carbon fiber plain weave fabric (MR60H), which was used for the BMI resin. Its properties are very similar to those of the Toray M30S fabric they also spread and wove. In addition to the cross-ply and off-axis configurations, two additional layups were fabricated from the BMI resin: [+45°$_{pw}$/-45°$_{pw,}$/0°$_{pw}$/90°$_{pw}$] to represent a small size boom application [e.g. NASA's Advanced Composite Solar Sail System (ACS3) project [16]] and [+45°$_{pw}$/-45°$_{pw}$/0°$_{ud,2}$/+45°$_{pw}$/-45°$_{pw}$] for a large size boom application [e.g. NASA-DLR (the German Aerospace Center) deployable composite boom project [17]]. The carbon fiber/BMI composites were prepared by NASA LaRC's composite fabrication technique [11]; photos are shown in Fig. 7.

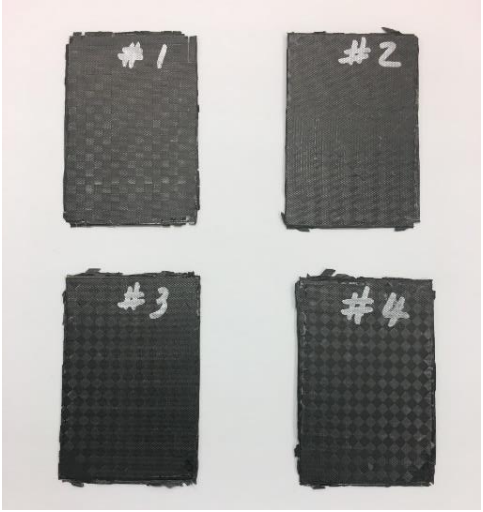

Fig. 7. Carbon fiber reinforced BMI composite specimen (2"x3" size). (1) [0°$_{pw,2}$/90°$_{pw,2}$] layup, (2) [+45°$_{pw,2}$/-45°$_{pw,2}$] layup, (3) [+45°$_{pw}$/-45°$_{pw}$/uni$_2$/+45°$_{pw}$/-45°$_{pw}$] layup, and (4) [+45°$_{pw}$/-45°$_{pw,}$/0°$_{pw}$/90°$_{pw}$] layup.

The relaxation moduli of carbon fiber reinforced composites were characterized by the same technique used in section 3.1 (Fig 8 and 9), and the results are shown in Table 3 and Fig. 10. CF45/Epoxy showed about 27% modulus relaxation in 1 year at 20°C and about 36% relaxation in 2 years at 40°C. However, CF0/Epoxy showed only about 2% relaxation because the resin viscoelasticity contributes relatively little to that fiber-dominated layup. In comparison, the low relaxation polymer composite, CF45/BMI showed much less relaxation in modulus: 16% at 20°C in 1 year and 20% at 40°C in 2 years. This was a 41-44% improvement relative to the baseline composite. Other layups relaxed about 3% to 17% as shown in Table 2 and Figure 10.

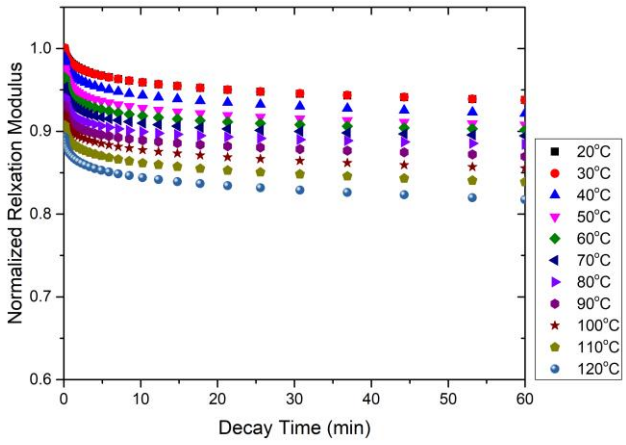

Fig. 8. Stress relaxation data of carbon fiber/BMI composite ([+45°$_{pw}$/-45°$_{pw}$/0°$_{ud,2}$/+45°$_{pw}$/-45°$_{pw}$]) at different temperatures.

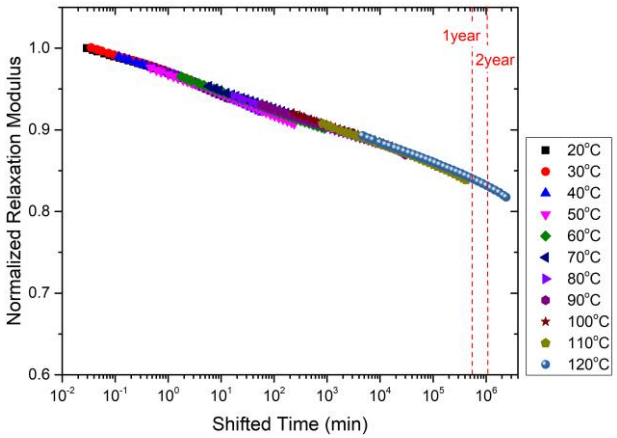

Fig. 9. A master curve of relaxation modulus of carbon fiber/BMI composite ([+45°$_{pw}$/-45°$_{pw}$/uni$_2$/+45°$_{pw}$/-45°$_{pw}$]) created by the TTS method.

Table 3. Carbon fiber/polymer composite layup configuration and relaxation in modulus.

Sample ID	Carbon Fiber	Fiber layup	Matrix Resin	% Relaxation in modulus	
				20°C, 1year	40°C, 2year
CF45/ Epoxy	Toray M30S	[+45°$_{pw,2}$/-45°$_{pw,2}$]	PMT F7	27	36
CF0/ Epoxy	Toray M30S	[0°$_{pw,2}$/90°$_{pw,2}$]	PMT F7	2	2
CF45/ BMI	Mitsubishi MR60H	[45°$_{pw,2}$/-45°$_{pw,2}$]	Cycom 5250-4	16	20
CF0/BMI	Mitsubishi MR60H	[0°$_{pw,2}$/90°$_{pw,2}$]	Cycom 5250-4	3	3
CF45U45/ BMI	Mitsubishi MR60H	[+45°$_{pw}$/-45°$_{pw}$/ 0°$_{ud,2}$/ +45°$_{pw}$/-45°$_{pw}$]	Cycom 5250-4	15	17
CF45-0/BMI	Mitsubishi MR60H	[+45°$_{pw}$/-45°$_{pw}$/0°$_{pw}$/90°$_{pw}$]	Cycom 5250-4	11	12

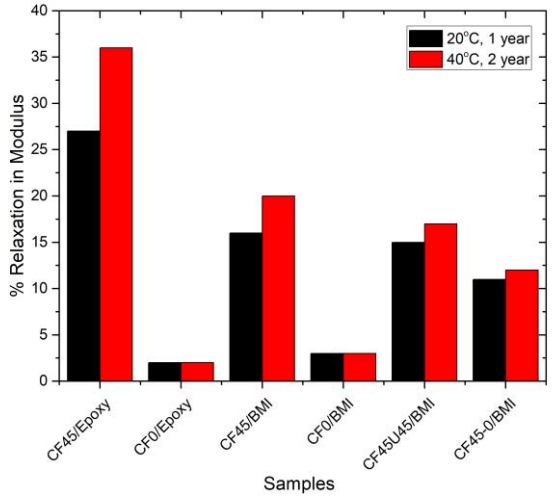

Fig. 10. Relaxation in modulus of baseline epoxy composite and low relaxation BMI composite.

4. Conclusion

The quantitative study of stress relaxation of the commercial polymers at relatively low strains (0.1%) can provide design guidelines to increase reliability of deployable space structures. Tetrafunctional epoxy (Araldite® MY720) showed lower relaxation than difunctional epoxy (D.E.R 332) or Novolac epoxy (PMT-F7). A polyimide with less flexible molecular chains (LaRC™ SI) showed lower relaxation compared to highly flexible polyimides (LaRC™ IA and ULTEM® 1000). Thermosetting polyimides (LaRC™ RP46, LaRC™ PETI-5, Cycom 5250-4, and

Compimide 353RTM) showed lower relaxation than either thermoplastic polyimides or epoxies. Carbon fiber composites fabricated from BMI resin (Cycom 5250-4) showed lower relaxation than the baseline epoxy composite (PMT-F7) and are good candidate materials for deployable composite space structures with better dimensional stability during extended stowage. Viscoelastic analysis suggests that higher activation energies of the β-relaxations can explain the lower relaxation. Further studies should include free volume analysis. Other factors worthy of study are the effect of the interface between carbon fibers and the polymer resin on relaxation, the effect of carbon fiber stiffness (graphite fiber), different fibers such as boron fiber, and the material nonlinearities at high strain regimes (> 1%).

5. References

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