

# Direct Curing of Polymer Construction Material in Simulated Earth's Moon Surface Environment

Alexey Kondyurin\*

University of Sydney, Sydney 2006, Australia

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The Earth's industrial exploitation of the moon requires a hermetically sealed structure large enough for all services: the life-support systems, energy-producing units, the storage of air and water, a greenhouse, and so on. The best way to achieve this is a light inflatable structure, which is transported to the moon in a packed position. The construction material has to be soft at deployment and made rigid at exploitation. One way of making the deployed structure rigid is a chemical reaction that forms a hard frame for the construction. However, a previous project based on an external hermetically sealed pocket experienced problems as a result of high pressure due to evaporation of the low-molecular-weight mass fractions of the uncured matrix and the resultant uncontrolled deployment of the inflatable structure. A structure is proposed in which the frame wall is external to the wall of the structure, which would eliminate the problem of uncontrolled inflation but requires that the structure be cured in a free-space environment.

## Nomenclature

$C$	=	actual concentration of active groups in epoxy matrix
$C_0$	=	initial concentration of active groups in epoxy matrix
$k$	=	rate of reaction
$M$	=	molecular mass of vapor
$P$	=	equilibrium vapor pressure of component
$T$	=	temperature, K
$W$	=	rate of evaporation
$\alpha_0$	=	constant of acceleration of the reaction
$\beta$	=	conversion of reaction
$\xi$	=	constant of deceleration of the reaction

## I. Introduction

THE Earth's moon is our nearest celestial body, and its exploitation for humanity is of considerable interest to the space programs of both national space agencies and industry. Not only is there considerable scientific and industrial interest in our nearest celestial neighbor, but the establishment of an artificial human colony on the moon as prototype for space bases on Mars, Venus, and other planets. The transportation of the ready-in-state moon base from Earth is impossible because of limited capacity of space carriers. On the other hand, a moon base of telephone-booth size, as in the Apollo Program, is impossible for the long-duration presence of crews on the moon's surface. Human activity on the moon requires a structure with a sufficiently large volume to house life-support systems, water, food, air, crew living and working rooms, and scientific and industrial equipment. The creation of a large-sized base on the moon is feasible only by the way of deployed construction on the moon surface after landing. Such projects are generally based on an inflatable structure, which can be transported from Earth in a compact container. After landing, the container is opened and the construction is inflated.

The history of inflatable space structures started from the Echo, Explorer, Big Shot, and Dash balloon satellites in the 1960s [1]. Based on the success of balloon satellite flights, new projects using inflatable structures for antennas, reflectors, lunar and Mars houses and bases, air locks, and modules based on light polymer films were

proposed from these years [1–5]. From these times, the inflatable structures based on new materials were developed and used for space application [2,6–19]. The world leaders of space inflatable structure production are American companies ILC Dover and L'Carde, Inc., which worked with support from NASA [20]. However, inflatable structures do not have a wide application in space exploitation because of high risk of damage of the soft inflatable shell. The use of an inflatable construction in a high-vacuum environment needs a more durable frame for the wall. Since the first inflatable constructions flights, several methods of rigidization have been discussed: rigidization due to chemical reaction of a soft polymer matrix by thermal initiation of reaction, by UV-light initiation, and by inflation of gas reaction; mechanical rigidization due to a stressed aluminum layer in the deployed shell; foam inflation; passive cooling below  $T_g$  of material; and evaporation of liquid from gel [3,21–27]. In some cases a combination of hard and rigidizable structures was developed [28,29]. All of these methods were tested in Earth laboratory experiments. Only one real mechanism of rigidization was successfully tested in real space conditions: aluminum stressed layers [2,30,31]. The best way of rigidization is a chemical reaction in polymer matrix impregnated by fiber filler, which gives a durable composite material tested and used for a wide number of space constructions. Such chemical reaction for rigidization process must be used for a moon base.

The moon conditions are terrible for polymer matrices. It includes high-vacuum ( $10^{-3}, \dots, 10^{-7}$  Pa) space plasma as a combination of different kinds of irradiations (UV middle and short wavelength irradiations, x-ray irradiation,  $\gamma$  irradiation, and high-energy electrons and ions), sharp temperature changes ( $-150, \dots, +150^\circ\text{C}$ ), depending on the sunlight, low gravity, and meteorite flux. The degradation of polymer materials in free-space environment is significant. Real space experiments during space stations and satellite missions were carried out with solid polymer materials. The effects of free-space environment on polymer materials were analyzed during the flights when polymers were exposed by atomic oxygen, VUV light, x-rays, electron and ion flows, thermal cycling, and high vacuum, and after the flights (SETAS, LDEF, MEEP, SARE, AORP, DSPSE, ESEM, EuReCa, HST, MDIM, MIS, and MPID missions), as well as under laboratory conditions where the separate factors or their combination were simulated in high-vacuum chambers with UV, x-ray, electron, and ion sources [32–71]. As was shown, the most degradation effect in polymer is caused by high density flux of atomic oxygen on low Earth orbit (LEO). The investigation of polymer material in far space exposure on orbit over Van Allen radiation belts (at an altitude of more than 65,000 km) is not known. There was an experiment of polymer exposure on satellites in

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\*Senior Researcher, School of Physics.

geostationary Earth orbit at 40,000 km of altitude, but the samples of polymer environment were not returned to Earth after exposure in space, and analysis of the polymer structure was not done.

An uncured polymer matrix is expected to give rise to significant problems in such an environment that the liquid components can evaporate into the space vacuum. In present inflatable structures projects, the uncured polymer matrix is isolated from the space environment by a hermetic shell, but a problem arises during the inflation process. As was shown in a number of real space flight experiments, the pressure for inflation may be so low that the vapor pressure of the low-molecular-weight fraction of the cured polymer wall inside the hermetic shell (outgassing) may be sufficient to cause inflation. The outgassing from polymer materials occurs continuously during long time flight to the moon and the collected gases can reach quite high pressure inside the hermetic shell. In this case the inflation becomes uncontrolled. The presence of uncured polymer matrix should significantly increase the vapor pressure inside the shell that should increase instability in deployment. This significantly restricts the use of uncured compositions inside the hermetic shell.

Kondyurin [72] proposed that the uncured polymer matrix be located on the external side of the inflating shell. In this case, the evaporation of low-molecular-weight components of the matrix occurs freely in space and outgassing does not break the inflation process. The matrix must be stable and curable under a free-space environment.

The curing processes were not investigated under free-space conditions. There were some experiments on curing of acrylamide gel onboard Mir, shuttle, and International Space Station under microgravity conditions [73]. There were attempts to cure the epoxy matrix composites under free-space conditions during LEO orbital flights such as Consort-02 (1989), Consort-03 (1990), and Consort-04 (1992) missions and the Polymer Composite Curing Experiment [Joust-01 (1991)]. Difficulty in curing limits the repair technology available for the thermo-resistant protection of the shuttle and is referred to in a NASA report [74]. However, detailed investigation of the curing processes under real free-space conditions were not given.

The influence of free-space environment on the polymerization process of epoxy matrix of the composite materials was investigated under laboratory conditions [72,73,75–83]. The free-space factors were simulated in high-vacuum chamber, plasma chamber, and ion beam implanter. Experiments in simulated free-space environment showed that the polymerization process is sensitive to space conditions, especially to high-vacuum, space plasma, and temperature variations. In the present publication a possible polymerization technique for inflatable and curable construction on the moon's surface is considered.

## II. Experiment

### A. Sample Preparation

Epoxy resins based on Bisphenol A (average molecular mass 374, viscosity at 25°C 1 Pa·s, Aldrich) and commercial epoxy resin based on Bisphenol F (UPPC AG, Germany) were used in our experiments. Triethyltetraamine (TETA) (Aldrich) hardening agent was used. The ratio of resin and hardening agent was 10:1 in weight parts. Glass fabric of satin weave was impregnated by liquid epoxy resin with hardener.

In experimental viscosity measurements, the epoxy resin was mixed with hardener and placed between measuring plates of a viscosimeter and for spectroscopic measurements, the epoxy resin was mixed with hardener and placed on KBr windows of 5 mm diameter.

The experiments on evaporation were done with uncured matrix (mixture of epoxy resin and hardener), as well as with pure epoxy resin and pure hardener separately. In these experiments, one layer of glass fabric of 40 × 40 × 0.2 mm<sup>3</sup> size impregnated by matrix component (epoxy resin or hardener or their mixture) with 1:1 fabric/matrix weight ratio as prepreg was prepared. One side of the prepreg was covered by Teflon foil (in the case of curing matrix) or by aluminum foil (in the case of separate component evaporation) to

simulate the hermetic shell. The uncured prepreg was placed in preheated vacuum thermobox and pumped down to measuring pressure. The rate of pumping was about 0.5–1 min, depending on measuring pressure. The prepreg was kept under vacuum for 15, 30, 45, and 60 min. Then it was taken out and the prepreg was weighted with analytical balance. The rate of evaporation was calculated by the following equation:

$$W = \frac{m_o - m_i}{S \cdot \Delta t} \quad (1)$$

where  $m_o$  is the mass of the initial matrix,  $m_i$  is the mass of the matrix after evacuation,  $S$  is the surface area of the prepreg, and  $\Delta t$  is the time for which the samples were under vacuum. The evaporation from the edges of the prepreg was neglected because of the small thickness of the prepreg. The mass loss was analyzed only up to 75–80% to exclude any change of evaporation rate due to decrease of evaporating matrix area with time.

In curing experiments, a prepreg of 100 × 100 × 0.2 mm<sup>3</sup> size was prepared as described above and cured under vacuum and plasma conditions at room temperature. A control sample of the prepreg was cured at room temperature and atmospheric pressure.

For dynamical mechanical analysis (DMA) measurement of curing kinetics, the prepreg was prepared as described above and fixed between sample holders of a DMA analyzer. Then the sample was mechanically loaded by DMA analyzer and cured with continuous measurement at room temperature and atmospheric pressure.

### B. Simulation of Moon Surface Environment

The Earth's moon vacuum environment and temperature of the structure on the moon surface were simulated in a vacuum thermobox. The thermobox was a Binder VD23 (Binder GmbH, Germany) was used for heating and vacuum treatment. The thermobox was evacuated from normal atmosphere to 30 Pa, which is lower than the vapor pressure of all the resin components (about 100 Pa at room temperature), and the temperature was varied from 25 to 160°C, with accuracy of ±1°C. Loss of sample mass was measured on a Sartorius balance (weight measurement accuracy of ±0.1 μg).

A high-vacuum chamber with a turbomolecular vacuum pump was evacuated down to a pressure of 10<sup>−3</sup> Pa for the high-vacuum experiments. This pressure is close to the pressure on the moon's surface near space carriers transported from the Earth atmosphere. High-vacuum treatment was done at room temperature only (25°C).

### C. Material Analysis Methods

Fourier transform infrared (FTIR) transmission spectra were recorded on a Bruker IFS-66 spectrometer with a DTGS detector, and spectral resolution of 2 cm<sup>−1</sup>. Bruker's OPUS software package was used for spectral analysis. The thickness of the sample was determined by weight measurements on the balance and from the absorbance of characteristic lines in the FTIR transmittance spectra. Analysis of the curing kinetic by FTIR transmittance spectra was made on the Bruker IFS-66 FTIR spectrometer. Samples for FTIR analysis were placed on KBr windows. Prepreg samples were used for mass-loss measurements.

For analysis of the mechanical properties of the composite, a bending test was selected because it is more sensitive to the matrix than a tensile test. The three-point bending test was carried out on a Zwick tensile testing machine under test conditions similar to the ISO-178-1992 (E) standard [84]. The test standard could not be completely adhered to, because the thickness of all the samples was less than the lowest sample thickness permitted by this standard. The distance between ground holders ( $L$ ) was 16 mm, and the speed of the upper holder was 1 mm/min. The elastic modulus  $G'$  was determined by the linear part of the stress–strain curve between 0.01 and 1% elongation. The thickness and width of every sample were measured before the tests.

Results of the test were analyzed from the recorded diagrams. Strength was calculated from the following formula:

$$\sigma = \frac{3 \cdot F \cdot L}{2 \cdot b \cdot d^2} \quad (2)$$

where  $L$  is distance between ground holders,  $F$  is applied force,  $b$  is the sample width, and  $d$  is the sample thickness. Elongation was calculated from the following formula:

$$\varepsilon = \frac{600 \cdot d \cdot x}{L^2} \quad (3)$$

where  $x$  is a position of the upper holder. The modulus was calculated by the following formula:

$$E_f = \frac{\sigma_1 - \sigma_2}{\varepsilon_1 - \varepsilon_2} \quad (4)$$

where  $\sigma_1$  and  $\sigma_2$  are stresses, and  $\varepsilon_1$  and  $\varepsilon_2$  are elongations in points of 1 and 0.01% elongation on the diagram. Because of the woven structure of the prepreg, the measured thicknesses of the prepreg are not the relevant thicknesses required for stress-strain curve analysis, and the strength and modulus results presented here are effective moduli.

The sol fraction of the cured matrix was determined by dissolving in toluene. The cured composite samples were weighed, swollen in toluene for 24 h, dried for 24 h in air, and weighed. The sol fraction was calculated as the difference between the initial dry weight of the matrix and dried weight after swelling in toluene.

Dynamic mechanical analysis was made with an ARES DMA analyzer (Rheometric Scientific, Inc.). Sample plates of  $50 \times 10 \times 0.3$  mm of cured prepreg were used. The rate of heating was  $5^\circ\text{C}/\text{min}$ ; strain of 0.05 rad with load frequency of 1 Hz was applied. A sample was heated and cooled continuously in a dry  $\text{N}_2$  gas flow. The temperature range was  $-50$ – $+150^\circ\text{C}$ . The  $G'$  and  $G''$  moduli and  $\tan \delta$  were recorded. Orchestrator software was used for analysis.

Kinetic measurements of the curing mixture were made at room temperature ( $25^\circ\text{C}$ ) for the epoxy matrix and also for the prepreg with the same matrix. The viscosity measurements of the epoxy resin and hardener mixture during curing were done with the same ARES analyzer with parallel plate (disk) geometry. The diameter of the disks was 25 mm, the gap between disks was 2.5 mm, and a strain of 20% at 1 Hz frequency was applied. The DMA measurements during curing were done for the prepreg with the liquid epoxy matrix and hardener for the same conditions and time scale. Sample plates of  $50 \times 10 \times 0.3$  mm were used. A strain of 0.05 rad with load frequency of 1 Hz was applied. The temperature of the prepreg during measurement was stabilized at  $25^\circ\text{C}$ . The prepreg was heated and cooled continuously under dry  $\text{N}_2$  gas flow.

### III. Results and Discussion

#### A. Evaporation of Epoxy Resin and Hardener

Evaporation of the uncured resin and hardener starts immediately after the pressure reduction resulting from opening the container with the inflatable construction in a free-space environment. The evaporation occurs due to high vapor pressure of the epoxy resin and hardener in comparison with the low pressure of space. The vapor pressure of most liquid epoxy resin is about 100 Pa and higher at room temperature. For the most used hardeners (such as polyamines) the vapor pressure is higher than for epoxy resins.

The rate of evaporation into high-vacuum  $W$  in  $\text{g cm}^{-2} \text{s}^{-1}$  for space materials is given by the Langmuir formula:

$$W = \frac{P}{17.14} \cdot \sqrt{\frac{M}{T}} \quad (5)$$

where  $M$  is the molecular mass of vapor,  $T$  is the temperature (in degrees Kelvin), and  $P$  is the equilibrium vapor pressure of component from Clausius–Clapeyron equation [85]. Note that the evaporation rate depends on the mass and the equilibrium vapor pressure of the substance, which also depends on the temperature. The dependence on temperature of the evaporation rates for epoxy resin based on Bisphenol A with molecular mass of 374 and TETA hardener are presented in Fig. 1. When the pressure of the

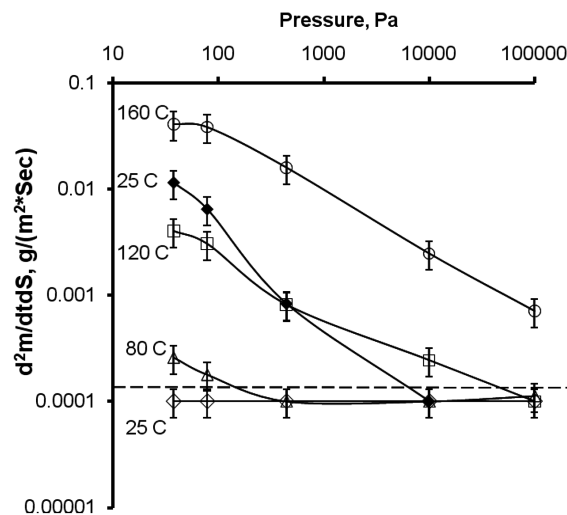


Fig. 1 Evaporation rate of the epoxy resin at  $160^\circ\text{C}$  (circle),  $120^\circ\text{C}$  (cubic),  $80^\circ\text{C}$  (triangle), and  $25^\circ\text{C}$  (empty rhombus), and the hardener agent at  $25^\circ\text{C}$  (full rhombus) into vacuum. Dashed line shows experimental limit of evaporation-rate measurement.

environment is lower than equilibrium vapor pressure, the rate of evaporation increases sharply. At high temperature of  $160^\circ\text{C}$  the evaporation rate is strongly dependent on pressure. At the relatively low temperature of  $80^\circ\text{C}$  the evaporation is weak and at room temperature the evaporation rate could be neglected. The evaporation rate for hardener is much higher than for epoxy resin at the same temperature.

This formula works for one-component liquid. In the case of a mixture of different evaporation-rate components, the diffusion of the components in the matrix plays a role. In a complex mixture the evaporation rates of the various components are different, so that means the ratio of the concentration of the component is changing both with time and distance from the composite surface during of the evaporation process. If the epoxy resin and hardener mixture are dissolved in a low-molecular-weight solvent, the evaporation process can occur not only from the surface of the composite, but the low-molecular-weight component of the mixture could form a separate phase as a gas bubble within the composite. The evaporation process can result in the formation of foam within the liquid matrix. We have observed the formation of foam structure in a liquid epoxy resin mixed with hardener and exposed to vacuum. After curing, a foam structure with a random distribution of the size and location of the bubbles was observed for solvent-applied epoxy matrices and for epoxy matrices with low-molecular-weight hardeners.

In the case of high-molecular-weight components, the evaporation process is slower and the formation of bubbles is not observed. The application of high-molecular-weight components is difficult, however, as the high-molecular-weight components have high viscosity and the mixture will not penetrate through fabric to fill all the spaces between the individual fibers. To exclude foam formation in a mixture with a low-molecular-weight component, the uncured matrix should be restricted to a thin layer on the surface of the fabric. In this case, the low-molecular-weight components can evaporate freely from the liquid matrix into vacuum without the formation of foam. Such a prepreg with a thin epoxy matrix layer was cured in the vacuum chamber at  $10^{-3}$  Pa pressure without foam forming.

#### B. Kinetics of the Curing Reaction

The curing kinetics of the epoxy resin with amine hardeners can be described by the following formula:

$$\frac{\partial \beta}{\partial t} = k \cdot (1 - \beta) \cdot (1 + \alpha_0 \beta) \cdot (1 - \xi \beta) \quad (6)$$

where  $k$  is a rate of reaction,  $\alpha_0$  is a constant of acceleration,  $\xi$  is a constant of deceleration, and  $\beta$  is the fraction of the reactionable groups that have reacted (the conversion):

$$\beta = \frac{[C_0] - [C]}{[C_0]} \quad (7)$$

where  $C_0$  is the start concentration of reactionable groups, and  $C$  is the actual concentration of reactionable groups [86].

The conversion was calculated from absorbance of 915  $\text{cm}^{-1}$  line, which is associated with the concentration of the epoxy group:

$$A = \varepsilon \cdot C \cdot d \quad (8)$$

where  $A$  is an absorbance at the 915  $\text{cm}^{-1}$  line of FTIR spectra,  $\varepsilon$  is an extinction coefficient,  $C$  is a concentration of epoxy groups, and  $d$  is the thickness of the matrix layer.

The reaction of epoxy matrix curing has periods of acceleration and deceleration. The curve of the reaction conversion for epoxy resin with amine hardener is presented in Fig. 2.

The  $\beta$  curve is consistent with Eq. (6); initially, the reaction proceeds slowly, then increases, and finally slows and stops before all the groups have reacted. Because of the reaction, the viscosity of the epoxy matrix increases with time (Fig. 2); the rate of change of viscosity is observed to increase sharply after the conversion reaches 0.2, and at 0.4 the matrix becomes nonfluid, corresponding to the formation of a gel in the epoxy matrix.

The elastic modulus of the prepreg increases with curing time, in a manner similar to, but not as dramatically as, the viscosity of the epoxy matrix. Increase of the elastic modulus is not sharp as for viscosity, but at the gel point the modulus has also changed. The modulus does not increase beyond the gel point, when the composite becomes hard.

### C. Curing in Vacuum Environment

Prepregs cured under atmospheric pressure and under vacuum were examined in a similar manner: there were no bubbles or foam, and the samples were hard and not sticky. The mass loss of the sample cured under vacuum was less than the detectable limit (0.2%). The bending test gave a modulus of  $527 \pm 21$  MPa for the control sample, cured under atmospheric pressure and  $499 \pm 19$  MPa for the sample cured under a vacuum of 30 Pa.

The thermomechanical properties of the composite after curing were analyzed by determining the elastic and nonelastic modulus of the composite from DMA data. The thermomechanical curves for the composite cured in vacuum at  $10^{-3}$  Pa residual pressure are presented in Fig. 3. In negative temperature region the composite behavior corresponds to a solid material with a high-elastic modulus. As the temperature increases, the elastic modulus decreases, the nonelastic modulus increases, the viscous mechanical losses increase, and the material becomes softer. At 43°C the composite

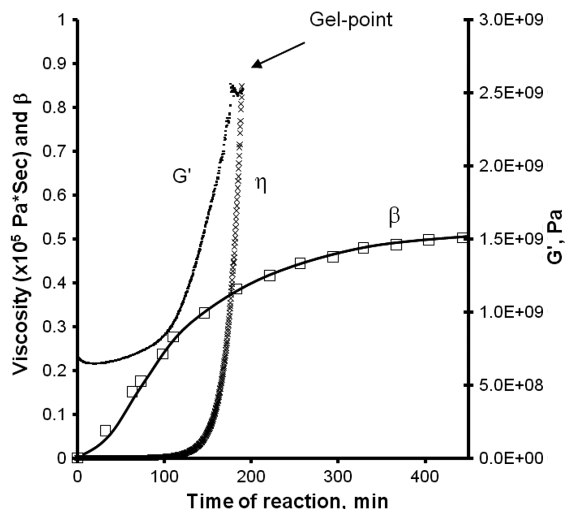


Fig. 2 Conversion fraction  $\beta$  of the curing reaction as determined from FTIR spectra, viscosity of epoxy matrix  $\eta$ , and elastic modulus  $G'$  of the glass-fiber-filled composite vs of curing time.

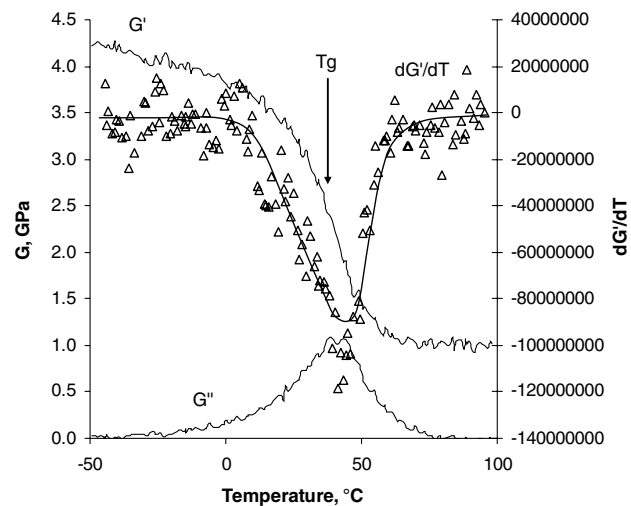


Fig. 3 Thermomechanical properties of a composite cured in vacuum, showing the elastic and inelastic shear coefficients  $G'$  and  $G''$  plotted as a function of temperature, and the first derivative of  $G'$  as a function of temperature. Arrow indicates phase transition temperature  $T_g$ .

curves have a specific point; the elastic modulus drops down and the nonelastic modulus is maximal. The extreme point (glass-transition temperature) of the elastic modulus is more clearly observed if the first derivative of the elastic modulus with respect to temperature is plotted against temperature (Fig. 3). The position of the changes corresponds to the phase transition of the epoxy matrix, which becomes fluid at temperatures higher than 43°C. The phase transition temperature depends on the density of the polymer network and the completeness of the curing reaction.

The curves of the  $G''$  modulus for various composites cured in vacuum are shown in Fig. 4. The first sample was precured in air to 0.15 of the conversion limit and then it was cured under high-vacuum conditions ( $10^{-3}$  Pa). At this stage of the reaction the epoxy matrix is fluid and the low-molecular-weight hardener can evaporate easily. The second sample was precured in air up to 0.3 of the conversion limit. After that it was cured in vacuum at the same pressure. At this stage of the reaction, the epoxy matrix has a higher viscosity and the hardener evaporates more slowly. The third sample was treated under vacuum after complete curing (the reaction is stopped itself at a conversion fraction of about 0.5 at room temperature). Glass transition temperature is shifted from 40°C for first sample to 64°C for third sample. Lower glass-transition temperature corresponds to lower final conversion fractions of the curing reaction.

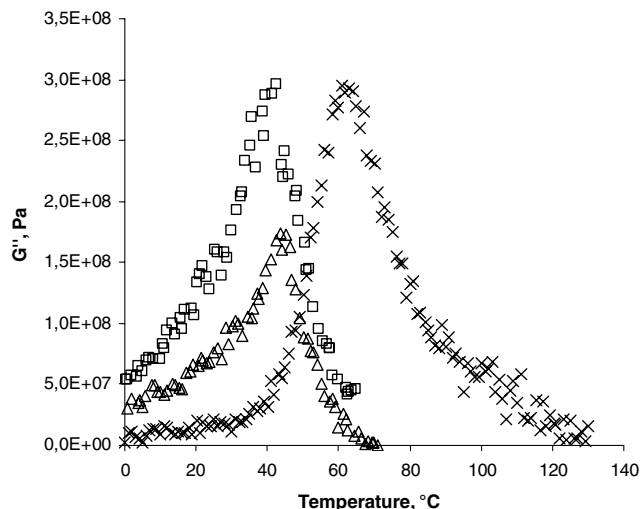


Fig. 4 Nonelastic modulus  $G''$  of composites cured in vacuum at  $10^{-3}$  Pa for precuring conversion fractions of  $\beta = 0.15$  (square data points),  $\beta = 0.3$  (triangles), and  $\beta = 0.5$  (crosses).

The stopping of the curing reaction in vacuum was determined from FTIR spectra. The final conversions of the reaction components after three days of curing at room temperature is 0.45 for the control sample (the first sample), 0.41 for the precured second sample, and 0.31 for the sample for which precuring step was omitted. The incompleteness of the reaction was observed by high concentration of the non-cross-linked part of the matrix, which was measured by solubility in toluene. The soluble part of the matrix after curing is 1.3% for the sample cured under vacuum and 0.8% for the control sample. The reason for stopping is the evaporation of hardener into vacuum at the beginning stage of the curing reaction.

The low glass-transition temperature observed for composite cured in vacuum and the cessation of the curing reaction confirm a weakness of the composite caused by the evaporation of active components of polymer matrix into vacuum. Thus, the precured composite is preferable for space application. However, the precured composite has a higher-elastic modulus, and the deployment of such a space structure would require high inflation pressure. Therefore, the development of an inflating and rigidizing space structure needs an optimization of epoxy matrix composition in the evaporation process of low-molecular-weight active components, together with inflating properties of the composite.

#### IV. Conclusions

The investigation of the polymerization processes in an epoxy matrix under vacuum conditions showed that curing of the epoxy resin with highly evaporating hardening agent is possible. The evaporation of the active components depends on pressure and temperature of the composition. The curing kinetics and evaporation kinetics occur in the same time scale and influence each other. The thermomechanical properties of the composite depend on evaporation rate of the components during the curing reaction. Increasing the epoxy matrix viscosity by precuring before evacuation decreases the evaporation, but the composite becomes harder and inflation could become more difficult. The optimization of the composition of the epoxy matrix based on a study of the evaporation and inflation processes is essential.

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T. Minton  
Associate Editor