SAND2022-13213C

Radiation Sensitivity of Adhesives and Potting Materials for Space Environments

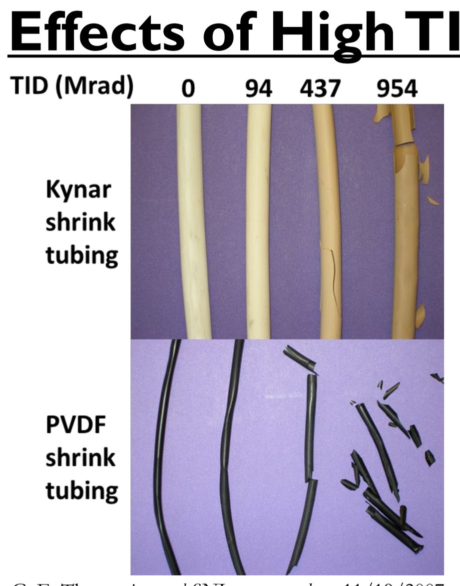
Jamie M. Kropka, Catherine Groves, Shianne Carroll, Estevan Martinez, Robert W. Hammerstein, Paul M. Thelen and Elliott J. Leonard

Sandia National Laboratories, Albuquerque, NM

Background

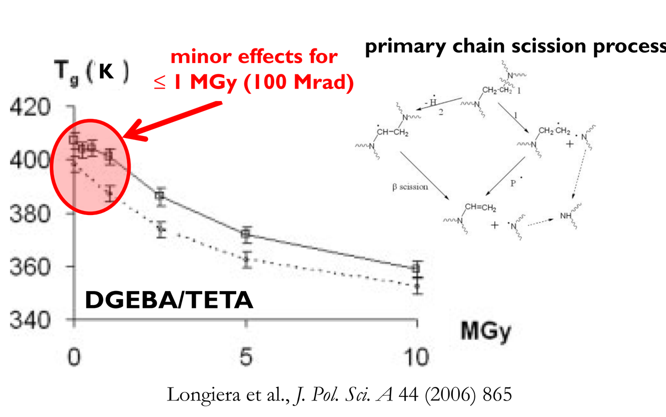
Many materials used in space applications are of known good physical properties, but what is documented can be lacking when it comes to exposure to naturally occurring ionizing radiation. This is particularly true for the environment in medium earth orbit, typified by the operational orbits of Global Positioning System (GPS) satellites (approximately, 12,550 miles). Although GPS satellites, generally, orbit in the "slot region" between the Van Allen belts, their long service life and fluctuation in trapped particle flux in this region - due to magnetic and solar disturbances - can subject the satellites and their payloads to an appreciable total ionizing dose (TID). This paper examines several common materials used in satellite assembly and the effects on these materials from progressively accumulated TID.

Effects of High TID

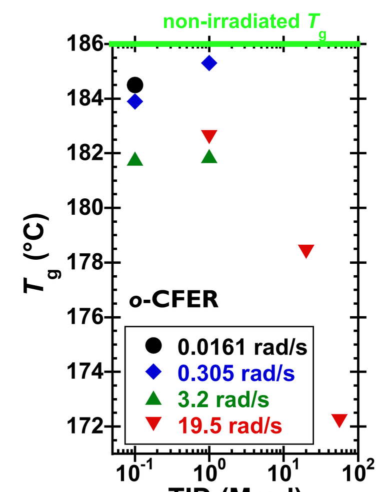


G. E. Thayer, *internal SNL memorandum*, 11/19/2007.

Observations for Epoxy Thermosets



Longiera et al., *J. Pol. Sci. A* 44 (2006) 865



Liu et al., *ACS Appl. Mater. Int.* 14 (2022) 5959

Relatively small effects for ≤ 100 Mrad TID, typically scission dominated

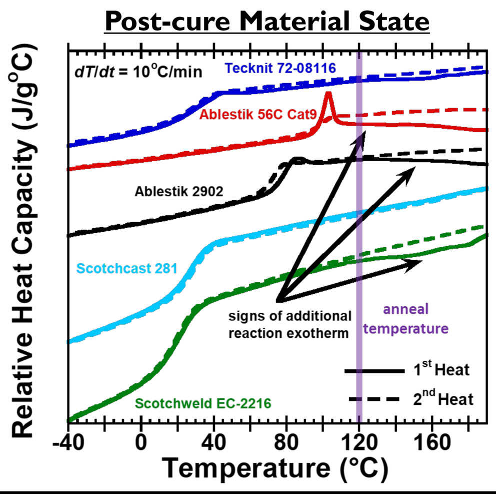
Experimental Materials and Procedure

Materials

Material	Mix Ratio in Parts by Weight Resin:Curative
Scotchweld EC-2216	5:7
Scotchcast 281	2:3
Ablestik 2902	100:6
Ablestik 56C Cat9	100:2.5
Tecknit 72-08116	1:1

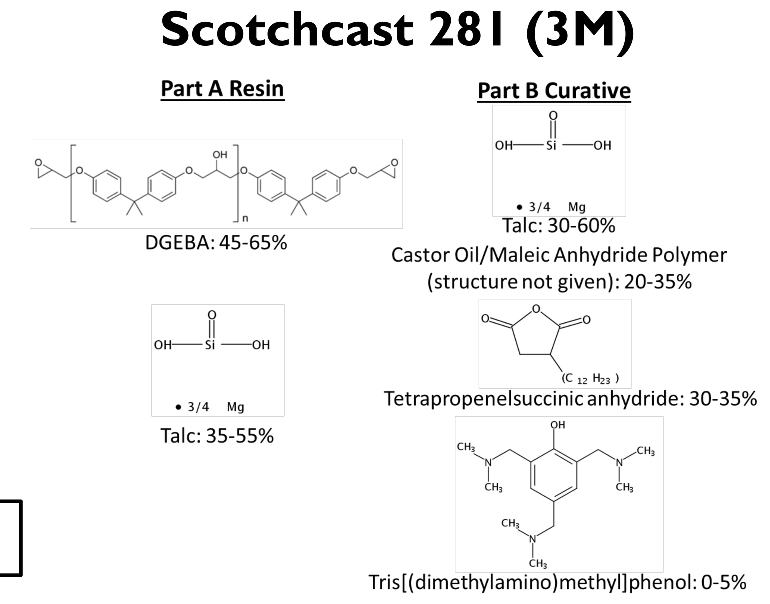
Processing for all materials:
- cured 24 hours at 80°C
- cut to dimensions
- thermal anneal 30 min at 120°C followed by cooling at 1°C/min to ambient

Post-cure Material State



Small amounts of continued cure in 2216, 2902 and 56C Cat9 that is extinguished during anneal

Scotchcast 281 (3M)



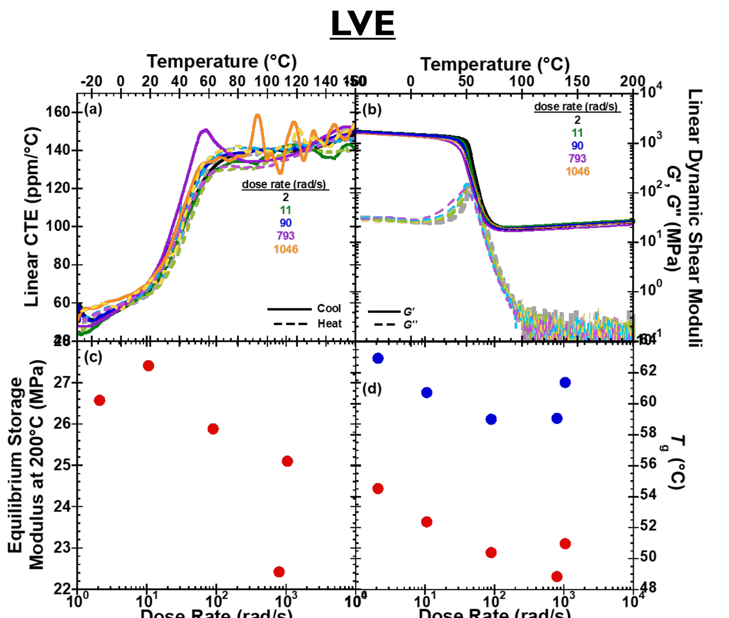
Radiation Exposures

Irradiation of the epoxy coupons was performed at Sandia National Laboratories' (SNL) Gamma Irradiation Facility (GIF) and Low Intensity Cobalt Array (LICA). The facilities utilize the gamma emissions from arrays of Colbalt-60 pins. Samples were irradiated to Total Ionizing Dose (TID) levels at dose rates ranging in magnitude from approximately 1-1000 rad/s in an inert (nitrogen or argon) atmosphere. Specific dose rates depended upon the configuration of the facility and were verified using either a cavity ionization chamber or CaF_2 Thermoluminescent Dosimeters (TLD). Most TIDs were obtained at the highest dose rate. Insensitivity of the results to dose rate was demonstrated through the lower rate irradiations.

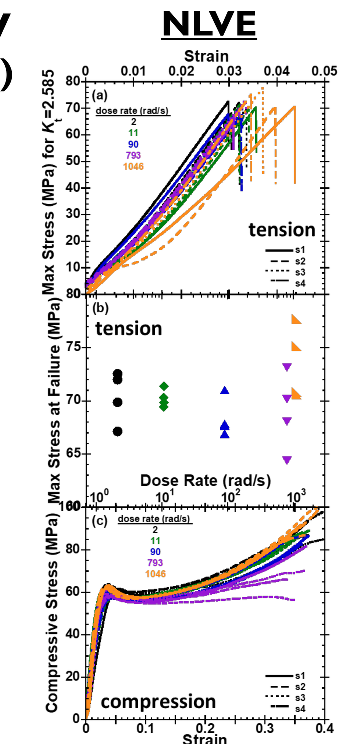
Dose Rate (rad/sec)	σ Dose Rate (rad/sec)	Dwell Time (dd:hh:mm:ss)	Total Dose (Mrad)	σ Total Dose (Mrad)
1046	16	various	1, 5, 10, 30, 70	
793	12	00:03:30:02	10.00	0.15
90	1	01:07:02:12	10.0	0.15
10.6	0.2	10:22:03:16	10.0	0.15
2.1	0.1	55:17:19:00	10.0	0.15

Dose Rate Insensitivity (Scotchcast 281 @ 10 Mrad TID)

LVE



NLVE



differences amongst dose rates comparable to typical sample-to-sample variability for these measurements

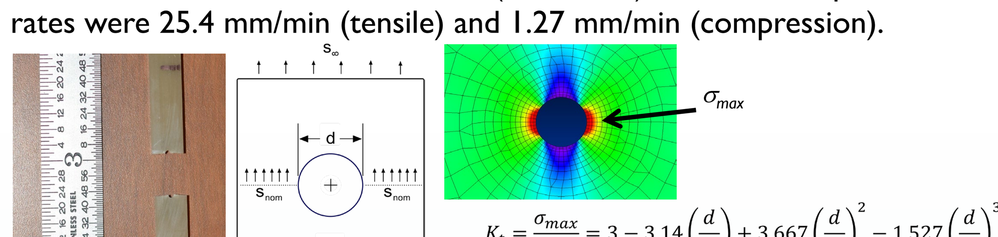
Thermal-Mechanical Testing

Measurements Spanning the Full Width of T_g

- Heat capacity assessed with a Q2000 DSC on ~10 mg samples in Tzero aluminum pans with a heat-cool-heat profile at 10°C/min
- Thermal expansion assessed with a Q400 TMA on bulk (6x6x12mm³) samples with a heat-cool-heat profile at 1°C/min under a small (0.05 N) compressive load from a flat-tipped standard expansion probe
- Linear viscoelastic shear moduli assessed with an ARES G2 DMA on thin (40x6x1mm³) samples in the torsion rectangular geometry by applying a small (0.01%) sinusoidal strain at a frequency of 1 Hz when heating the sample at 1°C/min

Ambient Testing Through Yield/Failure

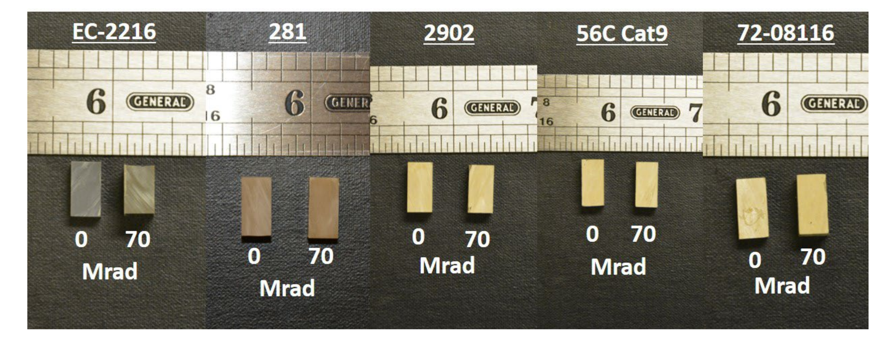
Tensile failure and compressive stress-strain response assessed with a 5882 loadframe (Instron) on thin (40x6x1mm³) and bulk (6x6x12mm³) samples, respectively. Tensile samples had a ~1mm hole drilled in the center to affect a stress localization and failure locus (see below). Constant displacement rates were 25.4 mm/min (tensile) and 1.27 mm/min (compression).



$$K_t = \frac{\sigma_{max}}{\sigma_{nom}} = 3 - 3.14\left(\frac{d}{W}\right) + 3.667\left(\frac{d}{W}\right)^2 - 1.527\left(\frac{d}{W}\right)^3$$

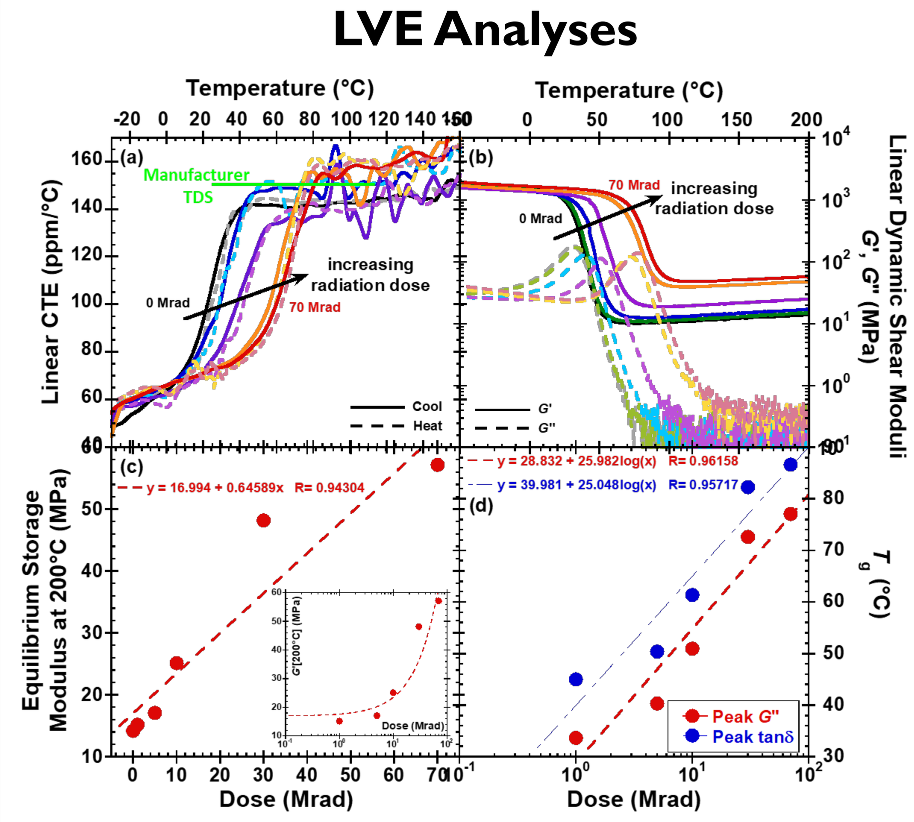
https://www.fracturemechanics.org/hole.html
Kirsch, E.G., *Zeit.Ver. Ingen.* 42 (1898) 797

Results



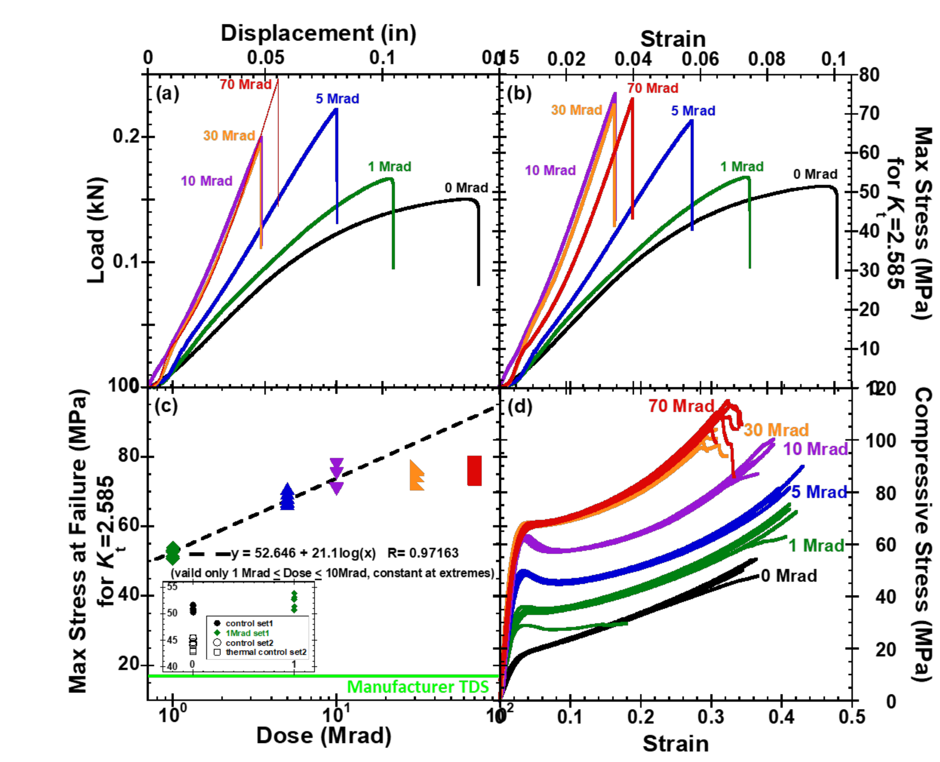
Scotchcast 281 (3M)

LVE Analyses

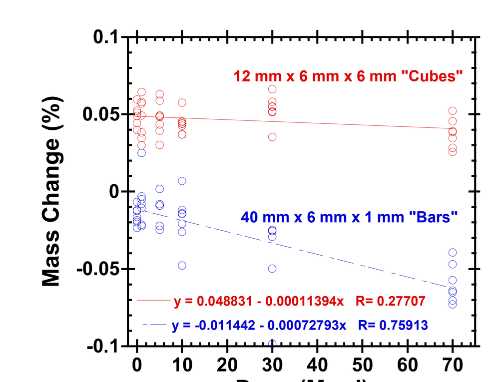


The increases in T_g and equilibrium modulus with dose suggest additional network crosslink formation. A minimum critical dose, ≥ 5 Mrad, may be required for significant effects, and effects may "plateau" beyond a maximum critical dose, > 30 Mrad

NLVE Analyses

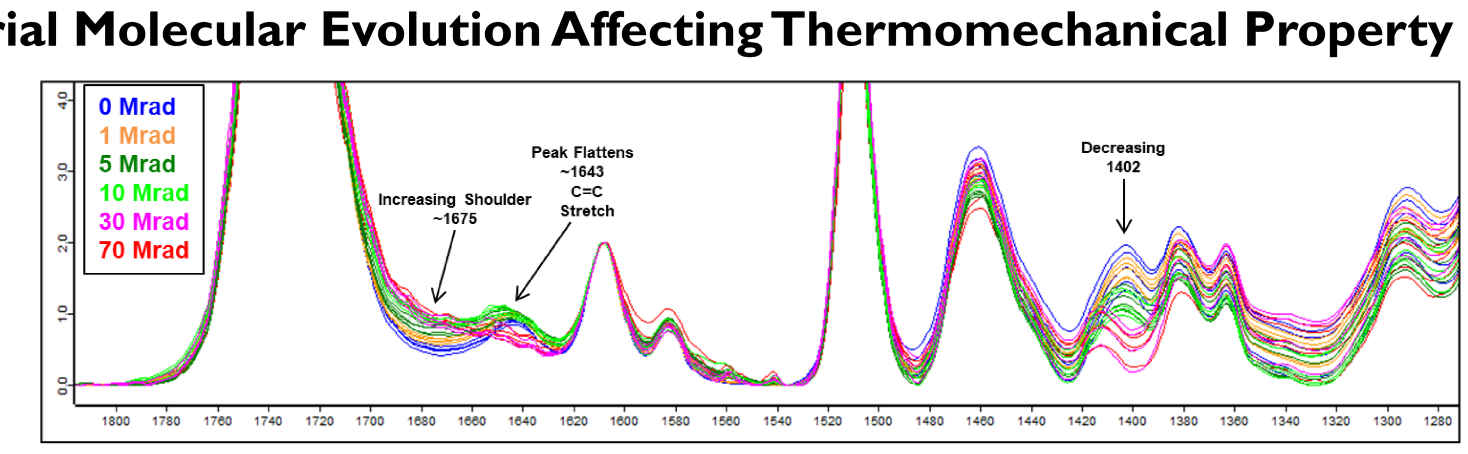


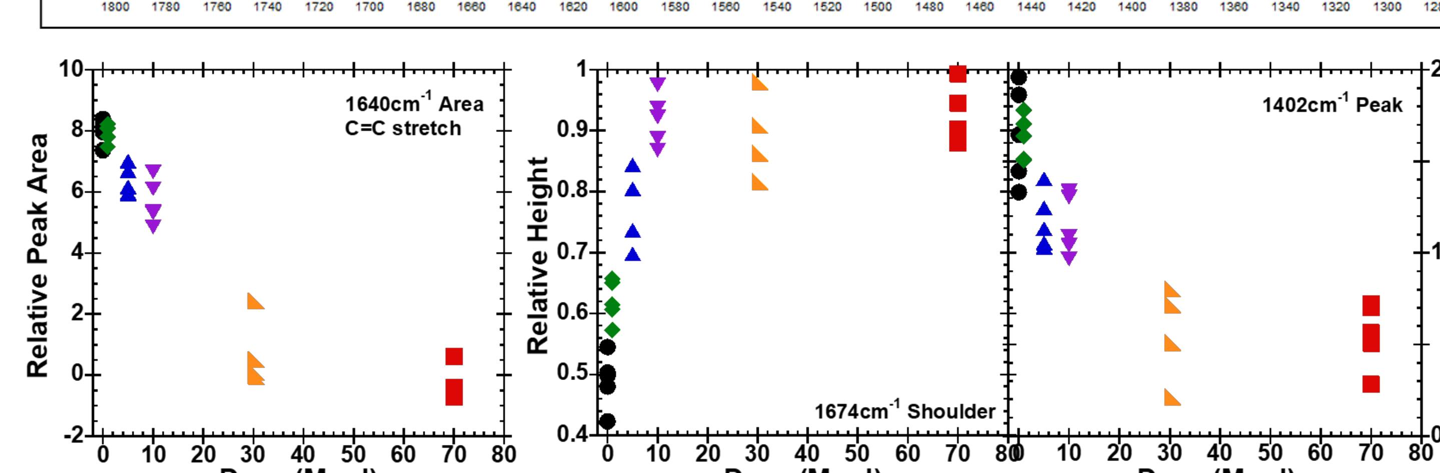
Ambient temperature stiffening and strengthening (higher yield) at low (≤ 10 Mrad) doses, with a "plateau" of effects beyond a maximum critical dose, > 30 Mrad. The changes are consistent with the increase in material T_g. The maximum tensile stress at failure tracks compressive yield.



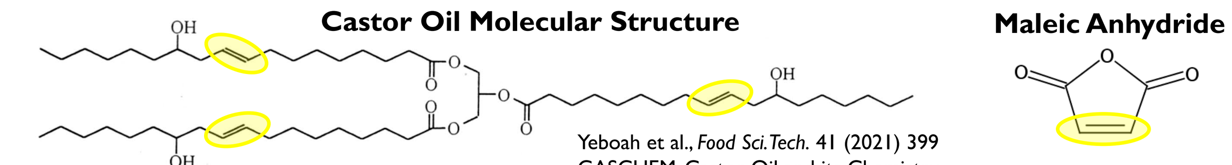
Total mass changes for both sample geometries ≤ 0.05%, which is on the order of the scatter in the measurements

Material Molecular Evolution Affecting Thermomechanical Property Changes





Castor Oil Molecular Structure **Maleic Anhydride**



Yeboah et al., *Food Sci.Tech.* 41 (2021) 399
CASCHEM, Castor Oil and its Chemistry

(while the extent of the functionalization/polymerization of castor oil with maleic anhydride in the Scotchcast 281curative is unknown, the unsaturated nature of the product will be preserved)

The unsaturated nature of castor oil and maleic anhydride (used in the curative for the epoxy) provide an avenue for addition crosslinking under irradiation. Diminishing of C=C stretch absorption with TID correlates strongly with thermomechanical property changes.

Machi, S., *J. Pol. Sci. A.* 4 (1966) 821

Conclusions

- The radiation sensitivity of some common epoxy adhesives and potting materials utilized in space environments was examined to TIDs of 70 Mrad in an inert atmosphere. While some materials exhibit signs of chain scission (not shown) and others exhibited signs of additional cross-linking, the magnitude of change in thermomechanical properties at these dose levels were relatively small in all materials except the Scotchcast 281, which exhibited thermomechanical property changes of magnitude well beyond any known reports under similar conditions.
- The additional cross-linking is proposed to be associated with polymerization of C=C bonds that remain in the epoxy network after cure. The cross-linking has been confirmed NOT to be associated with the following: (1) remaining reaction potential associated with the epoxy-curative functionality, (2) thermal heating associated with the irradiation and (3) oxidation during irradiation associated with any "loss" of the inert environment
- Changes in thermomechanical properties are insensitive to the dose rate at which the irradiation is performed and hence depend only on TID

Further Interests

- The details of the cross-linking process under irradiation are unknown as are the relative contributions of the castor oil and maleic anhydride C=C consumption to the resulting thermomechanical property changes. Systematic substitution of the maleic anhydride with other anhydrides (e.g., with and without C=C) in the curative would help to discriminate relative contributions to the changes in the material associated with irradiation.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.
SAND No. ____________

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.