Attempts to Enhance the Properties of EPON 830–Methylene Dianaline Epoxy Resin Systems by Exposure to Magnetic Fields during Thermal Curing

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ABSTRACT: This effort's objective was to determine whether enhancements to the properties of fully conventionally cured epoxy resin systems (ERSs) consisting of EPON 830, a diglycidylether of bisphenyl A epoxy resin, and methylene dianaline could be found by thermally curing the resins while simultaneously exposing them to economically generated magnetic fields (MFs). Stoichiometric mixes were thermally cured with one of the following profiles: 5 at 121°C or 4 h at 149°C. While being thermally cured, they were also exposed to MFs of strengths between 0.1290 and 0.8810 Tesla. Exposed and control specimens were simultaneously generated from the same ERS mix in each run. The resulting specimens were mechanically and thermally tested. This effort determined that under these conditions there were no modifications to the properties of MF-exposed, fully thermally cured, ERSs relative to their associated controls. © 1998 John Wiley & Sons, Inc.* J Appl Polym Sci 70: 2539–2568, 1998

Key words: magnetic; field; epoxy; EPON 830; methylene dianaline

INTRODUCTION

During the early and mid 1990s research was conducted to enhance the properties of conventional high-temperature epoxy resin systems (ERSs). Research efforts attempted to enhance these properties by thermally curing them to full cure while simultaneously exposing them to economically generated magnetic fields (MFs). Previous efforts to partially cure ERSs while simultaneously exposing them to MFs of the same strength as those used in this effort generated 34 to 300% improvements in the resultant ERS's mechanical properties.[1–5] An independent effort by Dr. Mallon, then at the Aerospace Corp., to fully cure at elevated temperatures a stoichiometric mPDA-based ERS while simultaneously exposing it to a 9 Tesla (T) MF enhanced the resin's glass transition temperature (T_g) by 25°C.[6] The specific property enhancements, ERSs used, and processing conditions associated with these three efforts are depicted in Figure 1. Also the foreign literature, primarily written by researchers in the former Soviet Union, is replete with hundreds of their efforts to enhance, by processing in an MF, almost every conceivable permutation of property, polymer, processing technique, and end product.[7–135] These previous efforts indicated that the potential to economically enhance particular properties of ERSs by processing them with conventional production techniques into end items while simultaneously exposing them to economical MFs was highly probable.

In 1986 the author conducted research which verified the existence of enhancements to the mechanical properties of an ERS when it is simulta-

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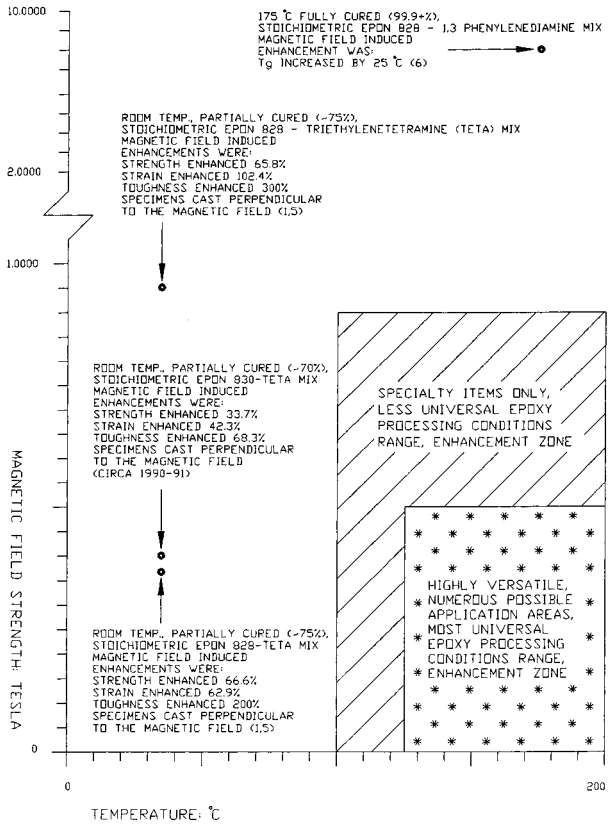


Figure 1 Property enhancement versus magnetic field strength versus polymer processing temperature.[1,5,6]

neously exposed to an MF while being partially cured at room temperature.[1,5] This 1986 effort verified many of the Soviet claims. They claimed that the mechanical properties of a similarly cured ERS could be substantially enhanced. The results of that effort demonstrated that at selected MF strengths the mechanical properties of the partially cured ERSs could be improved by 63 to 300%. They also indicated that the effect had an orientation bias. They reported that the effect depended upon both the orientation in which the specimen was generated in the MF and the orientation relative to that in which it was tested.[110–29] The results of that effort verified the existence of the orientation dependence. Enhanced mechanical properties were found in specimens that were cast and tested such that the angle of the specimen's testing axis was perpendicular to the angle of the MF during its cure. Specimens cast parallel to the MF showed no signs of enhancement. They also indicated that the magnitude of the enhancement varied sinusoidally with the MF's strength, as depicted in Figure 2.[2,81–115] The results of that effort did not decisively verify, but did imply the existence of, a sinusoidal relationship between the MF's strength and the degree of enhancement.

Between 1990 and 1991 the results of the 1986 effort were reconfirmed by the author.

As indicated in Figure 1 and demonstrated by the author's and Dr. Mallon's efforts, the MF effect does exist and can generate enhancements in the properties of ERSs so processed. Unfortunately, the combination of the thermal conditions of these ERSs' cure profiles and the MF strengths under which they were cured are not directly transferable to an economically viable production setting. The author's early efforts were directed at verifying and confirming the existence of the effect, and generated only partially cured ERSs at economically attainable MF strengths; whereas Dr. Mallon's effort generated fully cured ERSs in a superconducting electromagnet whose cost was equivalent to or greater than most epoxy end-item production equipment.

In order for the MF effect to be more than just a curiosity, it was necessary to find thermal processing profiles versus MF strengths which generated desirable enhancements in the so-processed ERSs and which were economically viable in a production setting. If enhancements to the properties of ERSs could be attained by processing them in the temperature and MF conditions found in the starred zone depicted in Figure 1, then the results of this effect could be easily incorporated into existing production setups. The ordinate of the starred zone in Figure 1 represents temperatures commonly found in the thermal cure profiles used in the production of cured ERSs. The abscissa of this zone represents MF strengths that are routinely generated by permanent magnets, electromagnets,

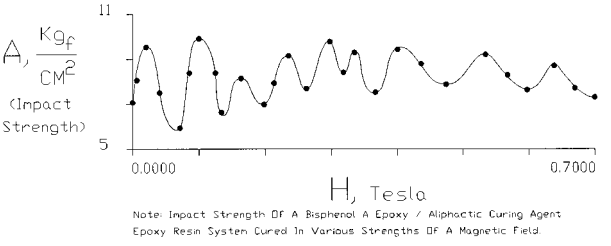


Figure 2 Sinusoidal correlation: property enhancement versus magnetic field strength.[82]

and MFs created by large currents. With the many ways of creating them, the necessary MFs could be effectively generated in whatever configuration was necessary to complement and not force the redesign of existing production setups. Overall, if enhancements to the properties of ERSs could be found to occur in this zone, then presently available production devices could be economically and unobtrusively modified to generate these enhancements.

The hatched zone depicted in Figure 1 represents temperature and MF conditions which could be incorporated into some existing specialty ERS production setups, if the enhancements found are both significant and robust enough. Most of the MF strengths encompassed by this zone can only be generated, with sufficient working volume, by electromagnets. This limits the dimensions of the end item which could be enhanced by the effect. Also, uniform MFs that encompass large working volumes of the strengths seen in the high end of the zone are very difficult to generate. Normally, very steep gradients are associated with the MFs seen at the high end of this zone. Due to these gradients, the effect would need to be seen over a large span of MF strengths so as to provide for sufficient working volume and controllability. Overall, if substantial-enough enhancements to the properties of ERSs could be found to occur in this zone, then it could be economically justifiable to modify some presently available specialty production devices.

As of mid-1992, no group had conducted any work concerning the existence or nonexistence of property enhancements in epoxies that had been MF-coprocessed under conditions outlined by the zones described in Figure 1. The only work then available was the very sketchy and unreproducible work published by the various Soviet research groups. To resolve these deficiencies, the objective of this effort was sequentially twofold. The prime objective was to conduct research to find a condition at which the effect generated a sufficient and robust enough degree of enhancement in the desirable properties of an ERS so as to be an economically viable addition to an existing production setup. The secondary objective, if the primary was unattainable, was to conduct a sufficient amount of research to be able to confidently dismiss the economically viable existence of the effect in the zones depicted in Figure 1.

EXPERIMENTAL

Research Tools

Table I lists the substantial equipment and chemicals used to accomplish this effort.

Resin Selection

The results of earlier efforts indicated that exposure to a MF completely damped out selected rotational motions of molecules with aromatic rings in them.[1–5] It also indicated that the larger the rigid length between the aromatic rings and the larger the number of aromatic rings in the molecules, the faster the damping would occur. If an ERS that had been oriented by some other mechanism were exposed to a MF of the correct strength and orientation, the molecular orientation in the ERS could possibly be maintained through to full cure in spite of thermally induced randomizations. The best candidate epoxy resin for the MF to induce property enhancements in was EPON 830.

EPON 830 is a diglycidylether of Bisphenyl A (DGEBA) epoxy resin[136] and is the largest epoxy resin molecule available in the DGEBA class that is also liquid at room temperature. EPON 830 DGEBA molecules will be the most aligned of all the liquid DGEBA type molecules by the shear field generated in the resin flowing through a constrained casting cavity.[2]

4,4′-Methylene dianaline (MDA) was selected as the curing agent. Table II lists the (effectively stoichiometric) MDA concentrations used. It is representative of the type of aromatic curing agents commonly used in the aerospace industry as matrix materials in composites. It was also selected because its core structure, the molecule minus amine reactant groups, is representative of the core structures of most available epoxy resin curing agents and other resins that use thermosetting reaction mechanisms. The highly aromatic nature of this group would also cause it to be effected by the MF and so both the EPON 830 epoxy resin and this curing agent would be influenced by exposure to the MF.

Magnetic Field Generation, Measurement, and Mapping

The MFs used in this effort were generated with two different electromagnet power supply systems. Both electromagnets were operated while a

Table I Research Tools

Magnetic field measurement
- *Probe:* Type: transverse hall effect; MB: Walker Scientific Inc.; capability: 0 to 1 T; linearity of reading: +/− 0.1% from 0 to 1 T; MN: HP-73R; design type: T-640859; style: I-10X
- *Gaussmeter:* Type: hall effect; MB: Walker Scientific Inc.; NBS traceable calibration; range: 0.0010 T to 10 T; resolution: +/− 0.1% or 1 microT; accuracy: +/− 0.1% or 1 microT; MN: MG-3D-4
- *Calibration:* Type: transverse reference magnets; MB: Walker Scientific Inc.; MN: MR-10T-2 (calibrated to 1.0099 T), MR-5T-1 (to 0.5043 T), MR-3T-1 (to 0.3010 T), MR-2T-1 (to 0.1991 T), MR-1T-1 (to 0.09868 T); accuracy: +/−0.25% NBS traceable
- *Zero Gauss chamber:* MB: Walker Scientific Inc.; MN: ZG-1

Mechanical testing equipment
- *Load:* Type: Lebow load cell; MB: Eaton Corp.; MN: 3132-149; SN: 10436; load capacity: 0 to 2224.1 N; calibration value: 1619.6 N; accuracy: 0.5% of reading or 0.25% of load range, whichever is higher from 5% to 100% of rated capacity
- *Strain:* Type: Extensometer; flat blade contacts; MB: MTS Systems Corp.; MN: 632.13B − 20; SN: 503; gauge length: 12.700 +/− 0.051 mm; accuracy: +/−0.5% of indicated strain from 100% to 5% of the extensometer's range; max travel: +/−1.905 mm or +/−15%; linearity: +/−0.15%; hysteresis: +/−0.10%
- *Strain Calibration:* Type: precision micrometer fork; MB: Measurements Technologies, Inc.; accuracy: +/−0.0025 mm; MN: CAL-01; SN: 861002-01
- *Fixtures and jigs:* Drafting: 04-11-91; X9119705 A; fixture, tensile test assembly
- *Frame:* Type: tabletop computer integrated testing system; MB: Sintech Div., MTS Systems Corp.; MN: SINTECH/1; drive mechanism: precision ball screw drive; position measurement: precision glass optical encoder; resolution: 2.540 μm; load range: 0–4448.2 N; frame stiffness: 35,020 N/mm; crosshead speed: 0.508 mm/min to 508.0 mm/min continuously variable (accurate to +/−0.1% of set speed for all speeds)
- *Mechanical data analysis and testing equipment control:* Runs 65-70: MB: Sintech Corp.; software: TESTWORKS (TM) 1989; ver.: 1.35. Runs 81-114: MB: Sintech Div., MTS Corp.; software: TESTWORKS II (TM) 1991; ver.: 2.11a

Experimental Data Recorder
- Type: MRL 488 Series multipoint recorder/logger; MB: Esterline-Angus, Esterline Co.; MN: MRL488-5-BD-RC-64--C4-Y

Thermal Analysis
- Type: differential scanning calorimeter; MN: DuPont Thermal Analyst 2000; program type: DuPont DSC calibration data analysis program ver. 5.0; accuracy: +/−1.1°C; heating rate: 10°C/Min; pan type: aluminum, hermetic, sealed in air; atmosphere: nitrogen, 50 mL/min; calibration materials: indium, tin

Positioner
- *3-Axis linear positioner:* MB: Daedal positioning tables and controls
  - *Z axis:* Type: rail table standard grade; MN: 506121S-LH; travel: 304.8 mm; positional repeatability: +/−0.00508 mm; accuracy: positional, +/−0.00025 mm/mm; linear, +/−0.0002 mm/mm
  - *Z axis bracket:* MOC: aluminum; *Z* Axis to *X-Y* axis perpendicularity: +/−0.025 mm
  - *X–Y axis:* Type: Series 300000, open frame linear table; MN: 318122S-20E-LH; travel: 304.8 mm by 304.8 mm; squareness: 60 Arcsec; positional repeatability: +/−0.0254 mm; accuracy: positional, +/−0.0002 mm/mm; linear: +/−0.0005 mm/mm
- *Rails:* Type: linear motion rail table system; MB: Thomson Industries, Inc.; MN: 1CB-24-FAO-S X 96.00; rail length: 2438.40 mm; shaft diameter: 38.1 mm; shaft hardness: Rockwell 60-65C; rail straightness: +/−0.0254 mm
- *Mobile rail car:* MOC: support channels: 152.4 mm 1020 steel channel; alignment bracket and leveling pad receiver blocks: 4340 Steel
- *3 axis positioner to Hall probe extension arm:* Drafting: 13-09-91; nonmagnetic Hall probe extension: assembly

Magnetic field generators
- *Small electromagnet:* rebuilt by Alpha Scientific Magnetics Inc.; MN: 1290; diameter flat pole faces: 101.6 mm; variable air gap: 0–127 mm
- *Small generator power supply:* MB: Kepco Inc.; MN: ATE36-30M; SN: F31926; current: 0 to 30 A; Power: 0 to 1 KWA; current regulation: 0.25% of I_o max; ripple: 0.3% of I_o max; drift: 0.02% of I_o max
- *Large electromagnet:* MB: Walker Scientific Inc.; MN: HV-7H; SN: 501591; diameter flat pole faces: 177.8 mm; variable air gap: 0–184.15 mm
- *Large generator power supply:* MB: Walker Scientific Inc.; MN: HS-1365-3A; SN: X-4644; power: 0–8.5 KWA; power regulation: +/−0.01%; current: 0–65 A; current stability: +/−0.001%

Table I *Continued*

Ovens *Type 1:*
Lid: Dimensions: thick: 6.35 mm; long: 285.75 mm; wide: 69.85 mm; bevel on all lower edges: 45 deg × 3.175 mm; MOC: optically clear, clouding, pitting, and heat-resistant, fire-polished Pyrex flat plate glass
Insulation: MOC: fiberglass non-woven felt sheet
Type 2: Drafting: 10-12-93; X936161 A; oven, curing, assembly
Oven Assembly: Dimensions: long: 234.950 mm; high: 171.450 mm; wide: 82.550 mm
Support Assembly: Dimensions: long: 254.00 mm; high: 82.677 mm; wide: 95.250 mm
Lid: Dimensions: 6.35 mm thick; 206.375 mm long; 85.725 mm wide; bevel on all lower edges: 45 deg by 3.175 mm; MOC: optically clear, clouding, pitting, and heat-resistant, fire-polished Pyrex flat plate glass
Insulation: MOC: fiberglass nonwoven felt sheet
Mold clamps
Type 1: Clamping plates: MOC: 6-6-2 titanium. Threaded rods: size: 1/4–20 by 50.0 mm; MOC: brass. Spacers: MOC: brass. Nuts: size: 1/4–20 jam and full; MOC: brass. Washers: type: 18L flat washers; MOC: brass. Compression springs: wave springs; gap type; MB: Smalley Steel Ring Co; MOC: X-750 Inconel; capability: 60.0 N–73.4 N at 0.762 mm working height
Type 2: Overall assembly: drafting: X936158; Type 2, mold pack: assembly. Clamping plates: MOC: 6-4 titanium. Nuts, spacers, compression springs, and washers: same as those used in the Type 1 clamps. Threaded rods: size: 1/4–20 by 76.2 mm; MOC: brass
Chemicals
4,4'-Methylene Dianaline: Purity: 99+%; MB: Aldrich Chemical Co.; LN: 03209LV
EPON 830: MB: Shell Chemical Co.; distributed by and procured from E. V. Roberts; LN: 7HHJ401; DOM: 7-86; LN: 01LHJ402; DOM: 1-89
Nitrogen: Form: gas; purity: 999995 ppm +/− 1 ppm; water content: 5 ppm +/− 1 ppm
GE 664 RTV: MB: General Electric Co.; material constituent: vinyl silicone rubber. Distributed by and procured from E. V. Roberts: LN: KM705; DOM, 1-91 and 2-91. Distributed by and procured from R. S. Hughes: LN: BC733; DOM: prior to 7/92

MB, made by; MN, model number; PN, part number; LN, lot number; MOC, material of construction; DOM, date of manufacture.

constant flow of clean, nonconductive coolant with a feed temperature of −1 +/−1°C passed through them. The clean, nonconductive coolant was necessary to eliminate the MF strength drift that electromagnets tend to exhibit over time from repeatable power settings, due to leakage currents through a conductive coolant.

Both electromagnets were anchored in place on stands which themselves were bolted to an overall equipment support scaffold, as depicted in Figure 3. A single MF mapping system was positioned above the electromagnets, on the scaffold, to measure and map the MFs.

Measurement and mapping of the MFs was accomplished with a Hall Probe–Gaussmeter–Three Axis Positioner setup. The Hall Probe–Gaussmeter combination was used to measure the MFs. The Three Axis Positioner was used to move the Hall probe within and to map the MFs.

Table II MDA Concentration

Run	Concentration (PHR)	Run	Concentration (PHR)	Run	Concentration (PHR)	Run	Concentration (PHR)
65	26.98	66	28.02	67	26.04	68	26.01
69	25.51	70	25.54	81	25.49	87	25.55
90	25.52	99	25.50	101	25.48	105	25.49
106	25.49	109	25.48	110	25.48	113	25.51
114	25.51						

Measurement accuracy: +/−0.01 parts per hundred resin (PHR)

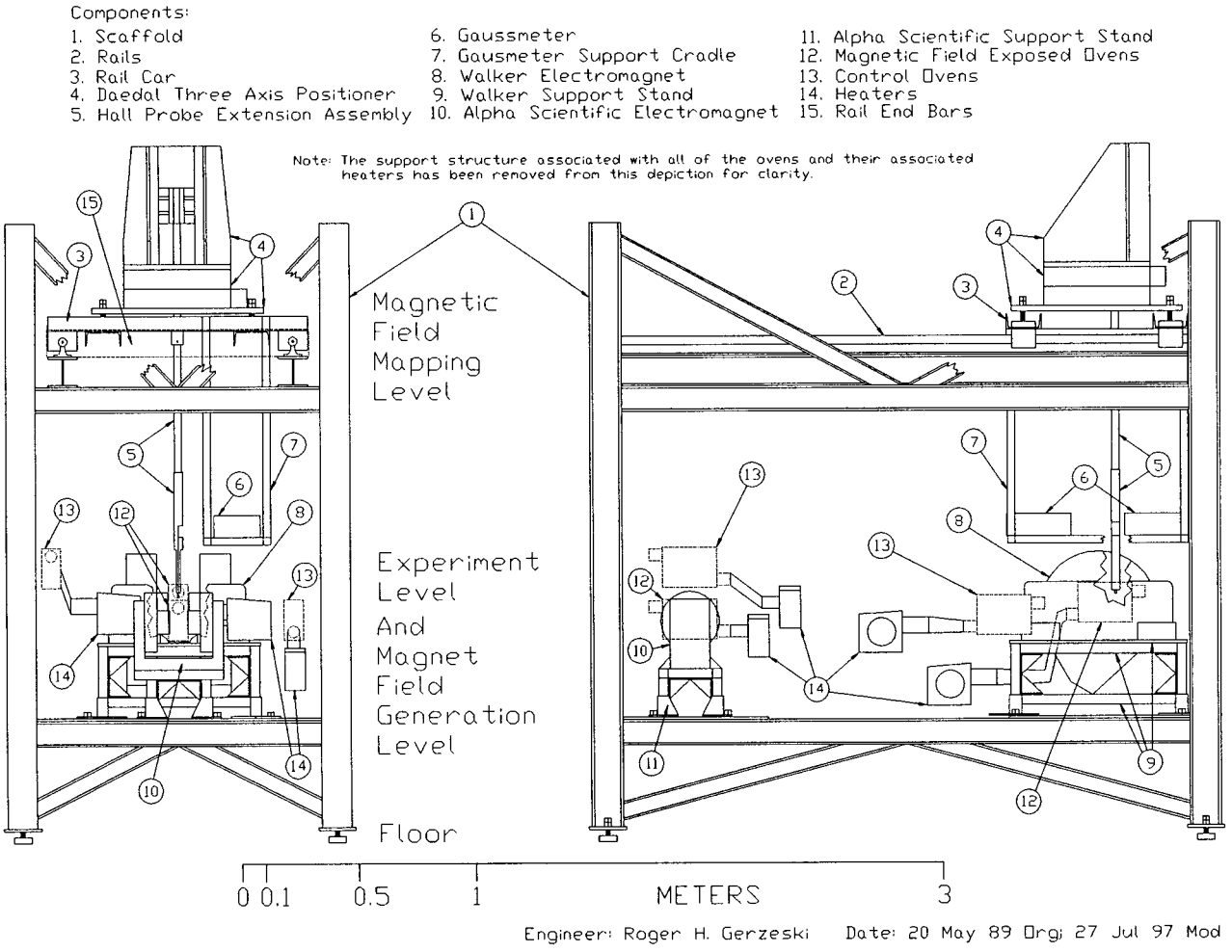


Figure 3 Supporting scaffold overall.

A transverse-style Hall Probe was used to measure the MF's strengths. The Hall Probe–Gaussmeter setup could accurately measure a MF's strength to 0.1%; its accuracy was +/−0.0009 T or better for all of the MFs generated. The Hall Probe was inserted into the relevant zone of an MF used for a run at specifically selected, accurately measured, and repeatable points; the strength of the MF was sampled at those points. The average of those points was then determined and adjusted as calibration requirements dictated. The resultant average and its associated extremes were then reported as the MF strength and range used in a run.

The Hall Probe–Gaussmeter setup was calibrated by measuring its readout when the Hall Probe was measuring two known NBS-calibrated MFs. The average MF strength and its extremes used for a run were then adjusted accordingly by linear interpolation.

To map the MFs the Hall Probe was moved in the fields via an anchored, positionally repeatable, three-axis positioner with a stiff and very low magnetic susceptibility extension arm. The Hall Probe was attached to an extremely low magnetic susceptibility clamp at the end of the extension arm. Figure 4 depicts the overall extension arm assembly and this clamp. The clamp was designed to be readily removable and reattachable to the extension arm while also being positionally repeatable. The overall positional repeatability of the extension arm assembly was +/−0.127 mm.

The precise and repeatable movement of the Hall Probe in and through the various MFs was achieved by using a three-axis positioner. The positioner was capable of moving the extension arm and attached Hall Probe from a set zero point to any and all necessary locations in the MFs with a repeatability of +/−0.0254 mm and a measure-

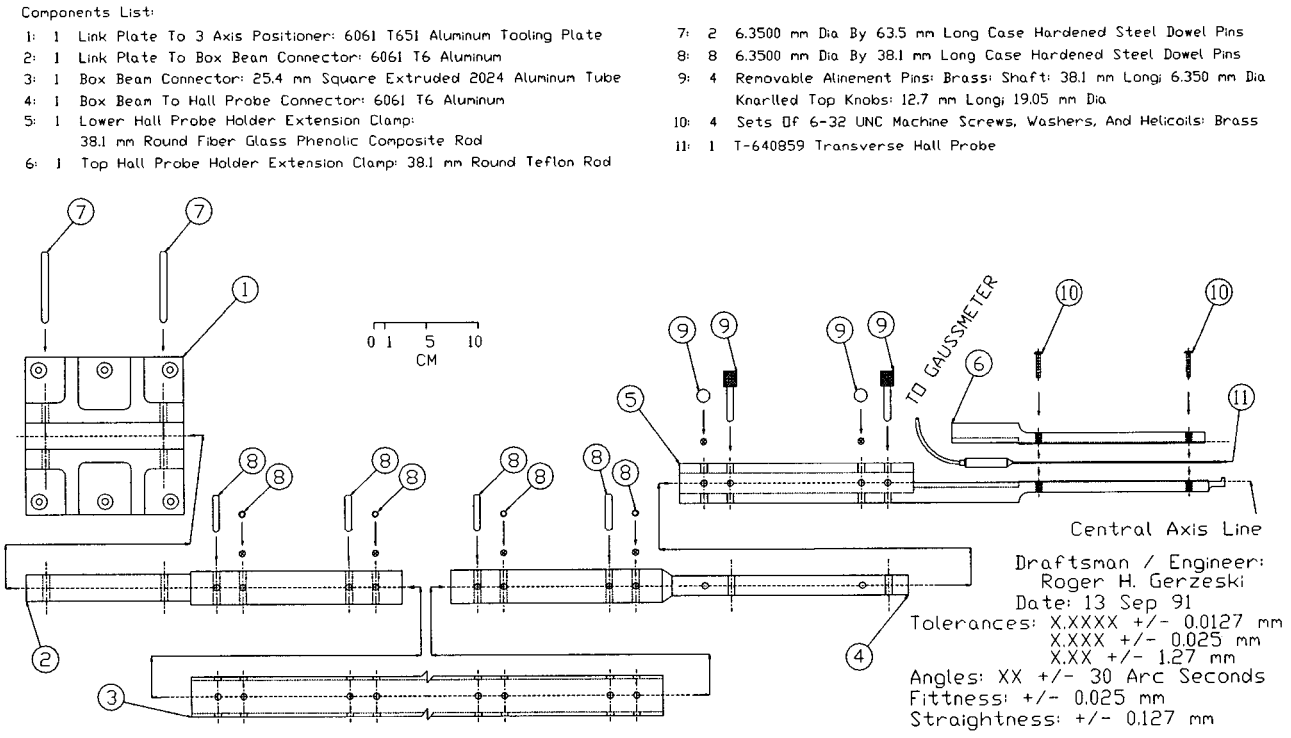


Figure 4 Hall probe extension: assembly.

ment accuracy of +/−0.0508 mm. Overall this equipment and its precision allowed for the sampling of enough points within the small-volume MFs used that an average MF strength and a statistically significant standard of deviation (std) could be accurately determined. It also allowed the determination of the extremes (i.e., the range) of the MFs used.

The positioner and all of the items attached to it were positionally anchored to a mobile railcar. This allowed the positioner and its associated mapping equipment to be moved from a repeatable point over one magnet to a repeatable point over the other magnet, and vice versa.

During the actual measuring and mapping of any particular MF, the car, positioner, and attached subcomponents were bolted to one of two bars that were permanently attached to each end of the rails. This bolt was torqued to 22.6×10^3 +/− 0.6×10^3 N mm and pulled the railcar and the associated measuring and mapping equipment into a stable and repeatable position over an electromagnet of choice.

All of the MF generating, measuring, and mapping equipment associated with this effort was either permanently or rigidly attached to the scaffold depicted in Figure 3. The objective of this design and of permanently anchoring all of the MF-related equipment to the scaffold was to rigidly link the zones in which MFs were created to the mechanisms which would measure and map them. Except for the railcars' ability to move from one position over one of the magnets to another position over the other magnet, all components associated with the MFs were permanently fixed. Overall, all MFs generated for this effort were mapped and all specimens were positioned for MF exposure with a repeatable positional accuracy of +/−0.635 mm or less.

Magnetic Field Strengths: Selection and Use

Four factors were considered in the selection of the number, strengths, and range of MFs to be used in this effort. The first factor concerned making the steps between one MF strength and the next small and frequent enough to avoid synchronizing with the troughs of the sinusoidal function indicated in the Introduction. The second factor took into account the author's previous efforts. A third factor involved the practical aspects associated with generating sufficiently large and ade-

quately uniform MFs. The final factor concerned the practicality of investigating a sufficient number of MFs to reasonably locate or dismiss the effect.

Soviet publications imply a sinusoidal correlation between the magnitude of a properties' enhancement versus MF strength.[2,81–115]From their publications, the wavelength of this sinusoidal correlation has an average of 0.0928 +/− 0.0550 T, with a range of between 0.0121 and 0.2520 T between 0 and 0.9000 T. If this sinusoidal aspect does exist, then runs must be conducted in MFs whose differences will assure locating any tangible resultant of the effect. In essence, the step size needs to be small enough to assure that the sampled MFs do not succeed in just hitting the bottoms of the troughs of this prospective sinusoidal correlation. Based on the published Soviet efforts, the author determined that an appropriate step size should be either the Soviet average minus or plus one std: i.e., 0.0378 or 0.1478 T.

Previous work indicated that the effect was not apparent below 0.1000 T.[1–5] Therefore, MFs below 0.1000 T were not investigated.

There are two distinct MF strength ranges which, if the property enhancement were adequate, would make the incorporation of this effect into existing production processes feasible. As Figure 1 indicates, the first range was between 0 and 0.5000 T; the second between 0.5000 and 0.9000 T. Below 0.5000 T, reasonably large-volume MFs with a uniformity of roughly +/−0.0100 T can be repeatably generated with fairly simple, low-cost MF generation devices and positional controls.[1–5] Between 0.5000 and 0.9000 T, only small-volume MFs with a uniformity of roughly +/−0.0100 T can be repeatably generated with complicated, costly MF generation devices and positional controls. Overall, the attributes associated with MFs below 0.5000 T allow for the effective operational determination of the effect over small spans of the MF on the order of a few hundredths of a Tesla; whereas the attributes associated with fields over 0.5000 and under 0.9000 T require larger spans of the MF, on the order of tenths of a Tesla, to detect the effect.

Based on the above, the author decided to use MFs below 0.5000 T that were separated by a rough average of 0.0400 T steps. The author also decided to use MFs above 0.5000 T that were separated by an average of roughly 0.1100 T steps. Fifteen different MFs were generated, mapped, and had EPON 830–MDA ERS specimens cast in them. These fifteen MFs and the range that they span are listed and further correlated to the particular run in which they were used in Table III.

Specimen Configuration, Orientation, and Generation

The dimensions of the specimens generated in this effort are depicted in Figure 5. These specimens were miniaturized ASTM tensile test specimens.

Table III lists the exact number of MF-exposed and control specimens that were generated in each run. The generation of so many specimens was necessary to assure that at least three or more mechanically testable exposed specimens and another three or more mechanically testable control specimens were created. Also, one additional exposed and one additional control specimen were required for thermal analysis.

Based on the results of earlier efforts, all specimens generated while exposed to the various MFs were oriented with their tensile load axis perpendicular to the MF's major overall vector.[1–5] Figure 6 depicts this major overall vector and the perpendicular orientation of the specimens to it. Actual specimen tensile load axis orientations relative to the MFs they were cast in for each run are listed in Table III. The worst orientation, with a desired perpendicular orientation being 90 degrees, was 86.61 degrees in run 69. The average orientation using Type 1 ovens and mold clamps was 89.10 +/− 0.87 degrees for the 13 runs using them. (See Ovens and Mold Clamps section, below, for definition of Type 1.) The worst-case orientation for the remaining four runs using Type 2 ovens and clamps was 89.80 degrees.

All specimens were generated by being cast into RTV-664 silicone rubber mold cavities. Sheets of these molds were created by pouring the freshly mixed silicone rubber system over aluminum mold negatives. Individual casting cavities where cut from these sheets and stacked together as depicted in Figure 7. These silicone rubber mold stacks were then clamped together and placed into the ovens. The ERS mixes were then cast into these molds and cured. The specimens were then extracted from the cured mass by peeling the rubber molds away and then trimming away the flash and excess left in the sprues.

Table III Field Strength, Specimen Orientation Within, and Specimens Cast

Run	Field (Tesla) Mean +/− Std	Min	Max	[Data Points]	Specimen Orientation (Degrees) Min	Max	Specimens Cast Exposed	Control
69	0.8810 +/− 0.0011	0.8785	0.8829	[384]	86.61	90.00	8	8
67	0.8637 +/− 0.0011	0.8610	0.8655	[315]	89.23	90.00	8	8
65	0.7640 +/− 0.0033	0.7611	0.7668	[273]	88.92	90.00	8	8
81	0.6871 +/− 0.0020	0.6822	0.6907	[385]	89.80	90.00	8	8
87	0.6871 +/− 0.0020	0.6822	0.6907	[385]	89.69	90.00	8	8
68	0.5533 +/− 0.0071	0.5396	0.5653	[385]	87.98	90.00	8	8
70	0.5533 +/− 0.0071	0.5396	0.5653	[385]	89.12	90.00	8	8
66	0.4841 +/− 0.0098	0.4684	0.5010	[231]	89.07	90.00	8	8
109	0.4474 +/− 0.0010	0.4456	0.4491	[75]	89.80	90.00	10	10
90	0.3965 +/− 0.0052	0.3827	0.4079	[385]	89.95	90.00	8	8
113	0.3741 +/− 0.0008	0.3727	0.3756	[75]	89.80	90.00	10	10
99	0.3301 +/− 0.0006	0.3290	0.3310	[75]	89.80	90.00	10	10
105	0.2918 +/− 0.0006	0.2907	0.2927	[75]	89.80	90.00	10	10
101	0.2748 +/− 0.0037	0.2686	0.2815	[385]	89.62	90.00	8	8
106	0.2313 +/− 0.0036	0.2248	0.2357	[72]	89.54	90.00	8	8
114	0.1773 +/− 0.0028	0.1721	0.1809	[72]	89.12	90.00	8	8
110	0.1290 +/− 0.0021	0.1251	0.1318	[72]	89.64	90.00	8	8

Measurement accuracy: +/−0.0009 Tesla; +/−0.01 degrees.

Elevated Temperature Curing

Ovens and Mold Clamps

The ERSs used in this effort required exposure to elevated temperatures for many hours in order to be fully (i.e., 99+% of theoretical maximum) cured. Elevated-temperature curing equipment was fabricated that allowed this to take place between the poles of the electromagnets. The curing equipment was designed and built to not thermally modify the temperature sensitive electromagnets; which, if so modified, would shift the strengths of the MFs they generated. It was also designed and built to not modify the strengths of the MFs that the cast ERS specimens experienced. To meet these requirements various pieces of equipment were needed, such as oven-like structures, rubber mold stack clamping apparatus, heat generation devices, and means to transport the heat to the ERS to cure it.

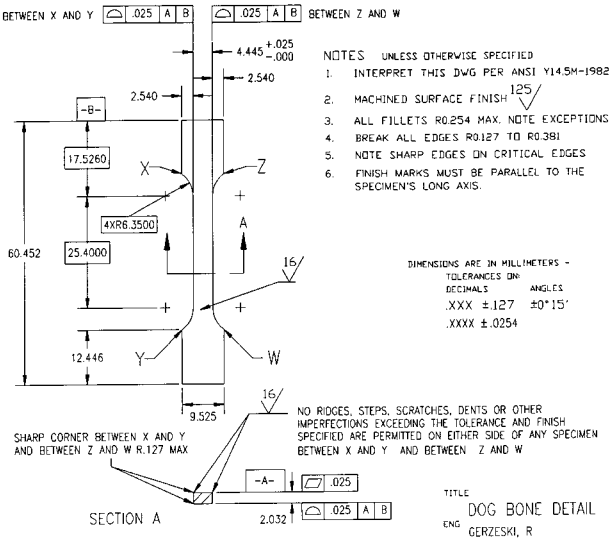


Figure 5 Tensile test specimen.

Two generations of matched ovens and clamping assemblies were designed and used. The first-generation set was designated Type 1; the second, Type 2. Both ovens and clamping assemblies were fabricated from very low-magnetic susceptibility materials that did not measurably modify the strength of the MFs that the curing specimens experienced.

The ovens were also thermally isolated from the electromagnets. The surfaces of the electromagnets experienced a temperature rise from the heated ovens of no more than 8.3°C, and the cool-

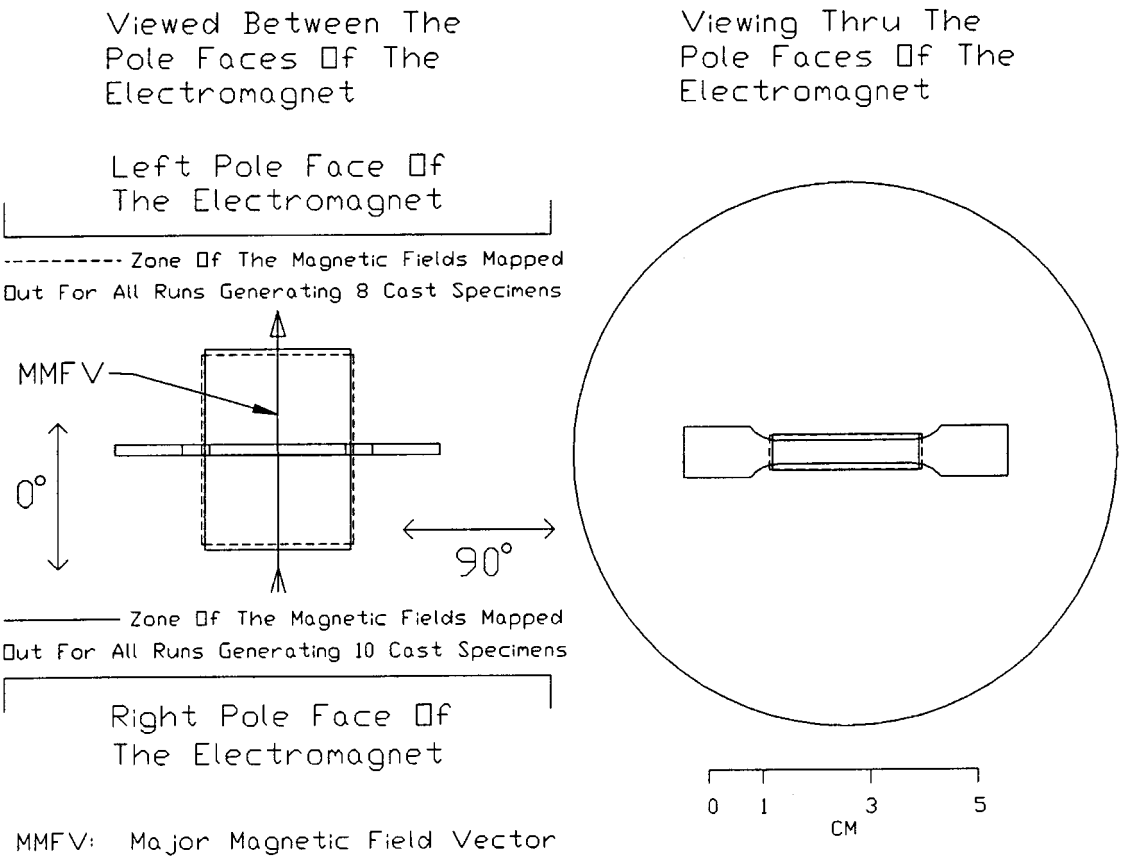


Figure 6 Perpendicular specimen orientation: within magnetic field.

ant leaving the various electromagnets registered a rise of no more than 2.7°C. The constant-current power supplies of the electromagnets were more than capable of compensating for the resistivity increase in the electromagnet's current-conducting coils resulting from this temperature rise and of maintaining constant and repeatable MF strengths.

The ovens were bolted to the stands upon which the magnets were anchored. This forced their position, and subsequently the position of the specimens cast within them, to be repeatably set relative to the entire MF generation and mapping system.

The mold stacks required an external clamping assembly. The prime function of the assembly is to compress the individual rubber casting cavities and to seal them together without distorting the shape of the casting cavities. For this effort the second function of the assembly was to accurately locate and maintain the location of the casting cavities throughout a run. This was required to determine the exact MF zone in which the specimens were cast for that MF's later measurement and mapping. The third function of the clamping assembly was to be able to expand and contract as the rubber molds did in a predictable fashion to avoid inducing a twist or deformation into the cast specimen's shape and to keep to a minimum the zone or volume of the MF that needed mapping.

The mold pack is the result of placing the mold stack into a clamping assembly. Figure 8 depicts an exploded view of a Type 2 mold pack. Both Type 1 and Type 2 clamping assemblies used the same connecting rods, spacers, fasteners, and compression springs. The springs used were wave-type compression springs, designed to provide a compression force on the mold pack over a large range of travel in a small space. All of the clamping assemblies' subcomponents were fabricated of very low-magnetic susceptibility materials. Therefore their presence near the mold stack would not measurably distort the strength of the MF to which the curing ERS would be exposed.

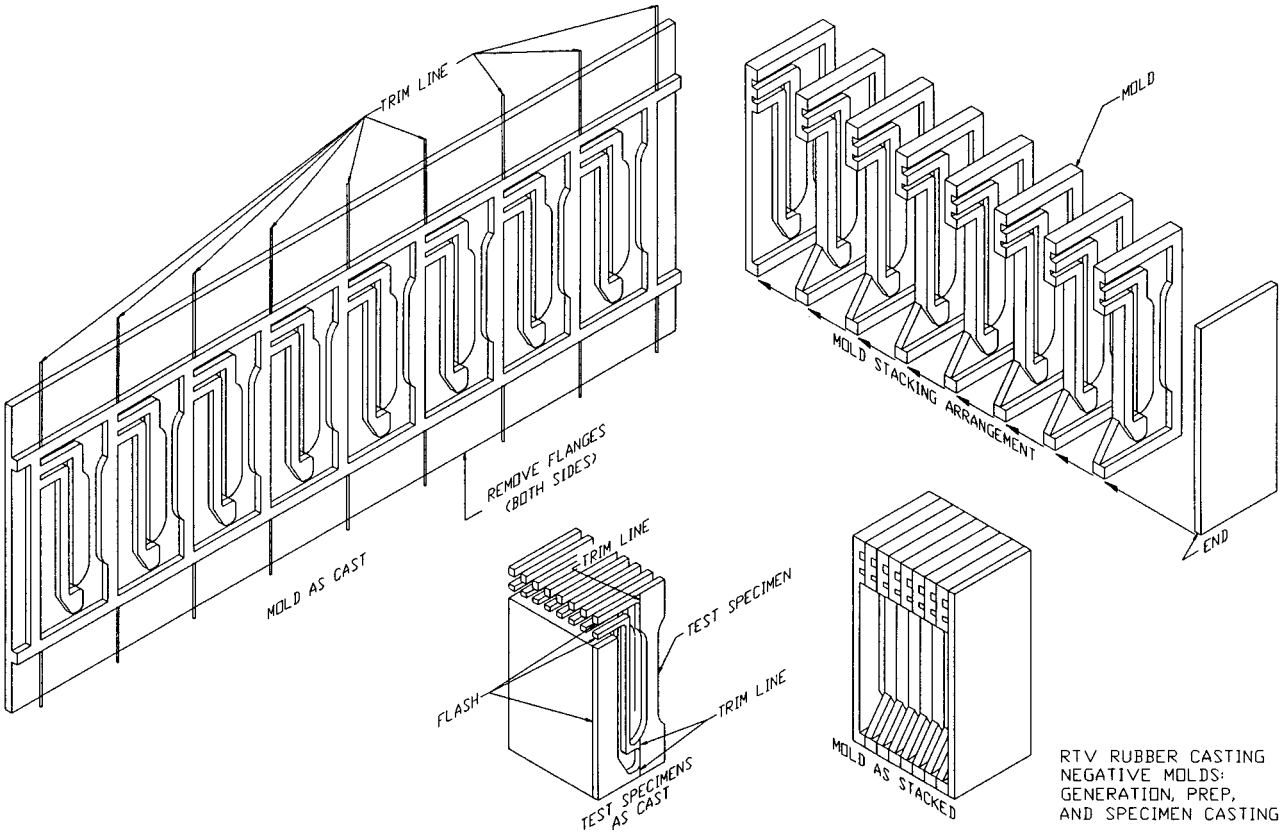


Figure 7 Rubber molds prep and stacking.

The Type 1 oven and the Type 1 clamp were used in those 13 runs in which eight exposed and eight control specimens were generated. Figure 9 is a drafting of the Type 1 oven with a Type 1 mold pack in it. The oven was made from 304 stainless steel. These steels do over time take on a slight ferromagnetic set, but because the MF zone of interest was inside the oven and could be mapped with the oven in place, this was acceptable.

There were a series of problems with the Type 1 set. First, it was very difficult to match and stabilize the temperature of the exposed set to that of an identical control set. Second, during a typical run the temperatures of the mold packs within the paired ovens had to be continuously monitored and adjusted because the two tended to drift. Third, the Type 1 mold pack rode on an inverted-U pedestal, and over the duration of a typical run it would shift its position randomly in the oven by up to 6.35 mm. Along with this random walk, no provision was made in these Type 1 sets to accurately and repeatably position the Type 1 mold pack in the Type 1 oven. This degraded the accuracy of the reported MF strength under which the run was generated by requiring an expansion of the zone required to map the MF.

To resolve the problems associated with the Type 1 set of ovens and clamps, the Type 2 ovens and clamps were designed. All of the Type 2 oven's subcomponents were made from very low-magnetic susceptibility materials. The Type 2 set of ovens and clamps were used in those four runs in which 10 exposed and 10 control specimens were generated.

The Type 2-style oven and clamp set resolved all of the thermal problems exhibited by the Type 1 set. This was accomplished by directing the flow of the gas heating medium directly into the bottom of the mold stack in the mold pack, as depicted in Figure 10, instead of splitting the flow as was done in the Type 1 set. Also, the Type 2 set did not require the intense temperature control oversight during a run that the Type 1 set did. The Type 2 ovens and mold packs were specially designed to exactingly control the initial position and direct the mold stack-induced expansion and

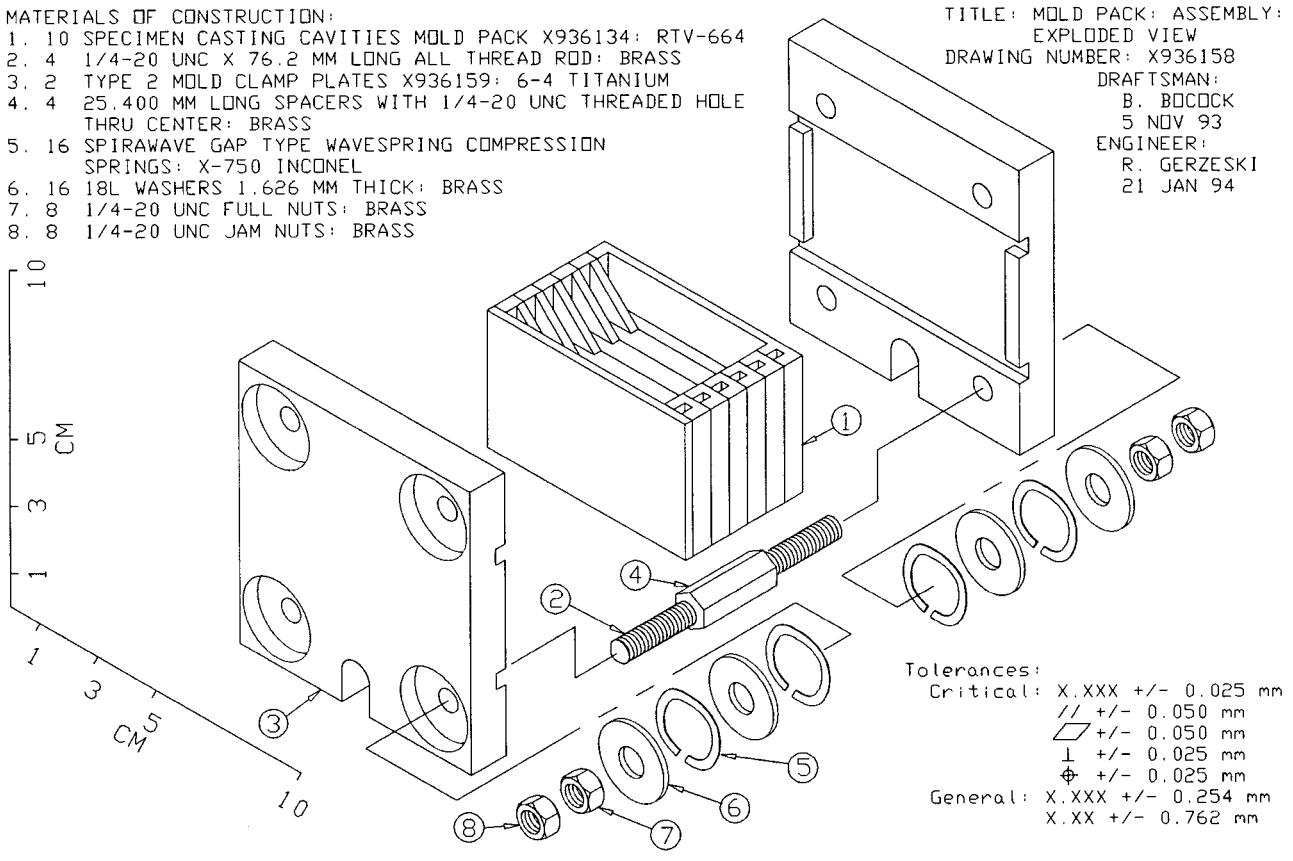


Figure 8 Mold pack assembly.

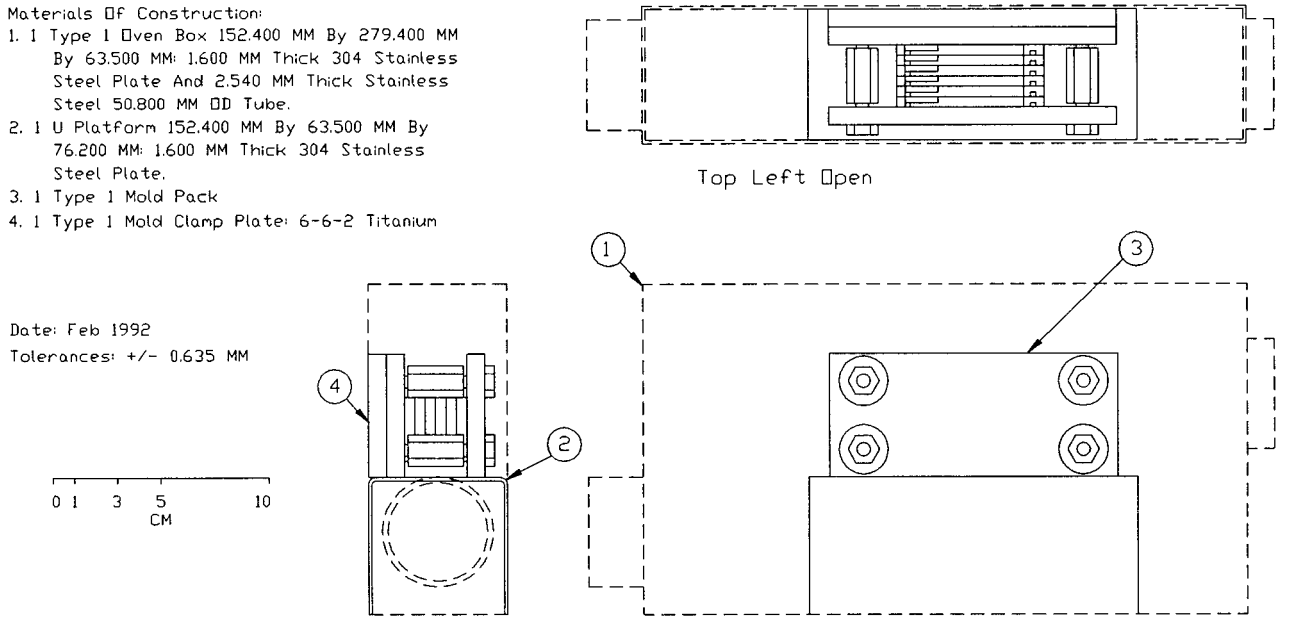
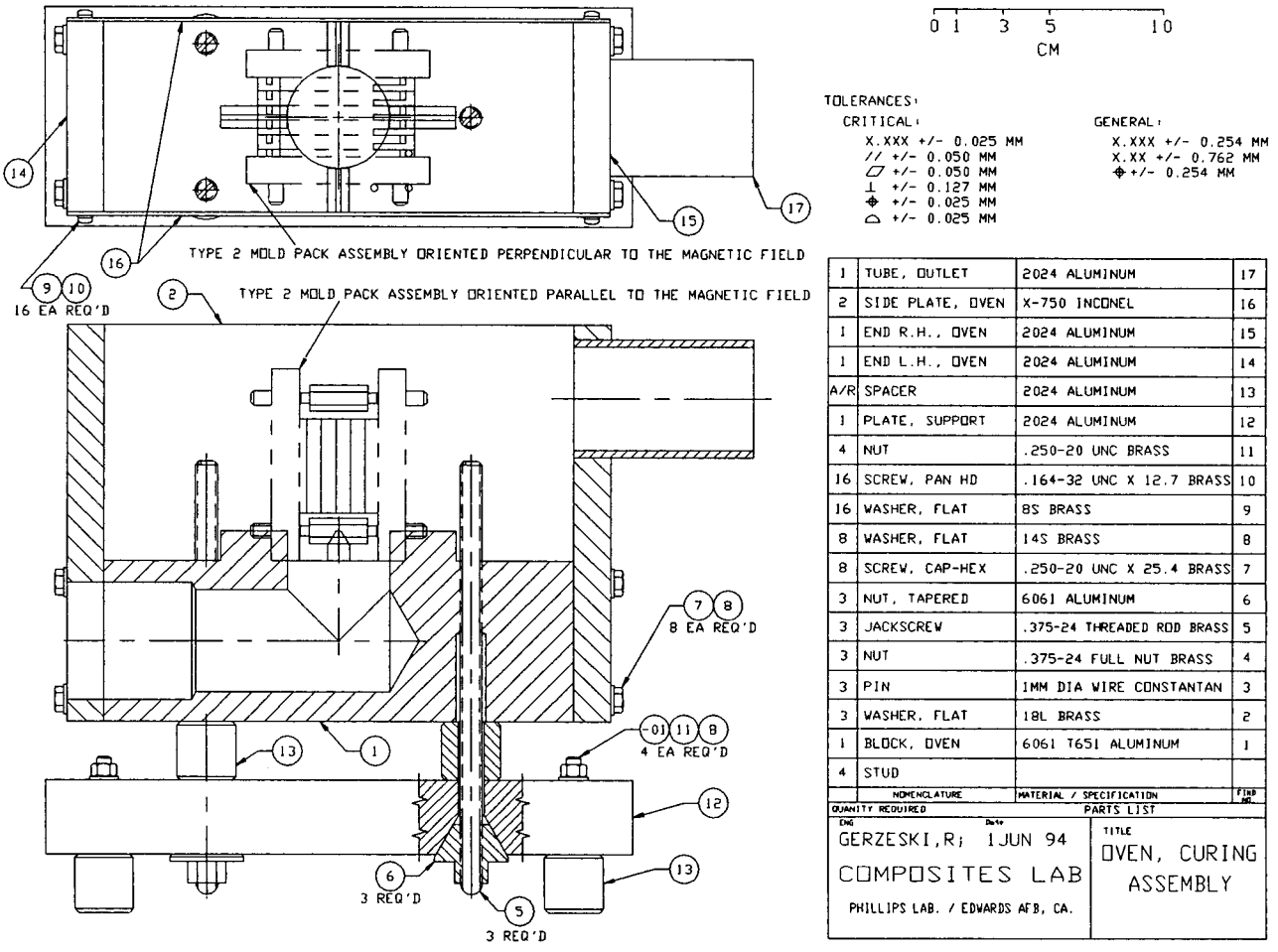


Figure 9 Type 1 oven: assembly.



QUANTITY REQUIRED	NOMENCLATURE	MATERIAL / SPECIFICATION	FIND NO.
1	TUBE, OUTLET	2024 ALUMINUM	17
2	SIDE PLATE, OVEN	X-750 INCONEL	16
1	END R.H., OVEN	2024 ALUMINUM	15
1	END L.H., OVEN	2024 ALUMINUM	14
A/R	SPACER	2024 ALUMINUM	13
1	PLATE, SUPPORT	2024 ALUMINUM	12
4	NUT	.250-20 UNC BRASS	11
16	SCREW, PAN HD	.164-32 UNC X 12.7 BRASS	10
16	WASHER, FLAT	8S BRASS	9
8	WASHER, FLAT	14S BRASS	8
8	SCREW, CAP-HEX	.250-20 UNC X 25.4 BRASS	7
3	NUT, TAPERED	6061 ALUMINUM	6
3	JACKSCREW	.375-24 THREADED ROD BRASS	5
3	NUT	.375-24 FULL NUT BRASS	4
3	PIN	1MM DIA WIRE CONSTANTAN	3
3	WASHER, FLAT	18L BRASS	2
1	BLOCK, OVEN	6061 T651 ALUMINUM	1
4	STUD		

PARTS LIST

Figure 10 Type 2 oven: assembly.

contraction travel of the mold pack. This was accomplished by machining alignment slides and press-fitting alignment pins into the main base block of the oven and cutting slide notches into the clamp plates. By mating the cut notches of the mold clamps depicted in Figure 8 to the machined slides on the oven blocks depicted in Figure 10, and by placing one of the two mold clamp plates between the press-fit alignment pins and over the machined slides depicted in Figure 10, the initial position and movement of a mold pack could be controlled. Overall, this arrangement anchored one plate to within less than +/−0.127 mm of a set position, and rigidly constrained the range of movement of the remainder of the pack to within +/−0.127 mm along the specimen axis and +/−0.635 mm along the mold pack's expansion–contraction axis.

Heaters and Heat Transfer

Bone-dry nitrogen gas was used as a heat-transfer fluid to get heat to the curing resins. With this technique a fluid was heated outside the influence of the MF and transported into the ovens to subsequently heat the mold packs and thermally cure the resins cast within them. This also assured that the cured resins would not be contaminated with any foreign matter, particularly water.

A one-pass system was used in which the heat-transfer fluid was heated and then transported to the ovens, where it heated the mold packs and the resins within them and then was dumped into the surrounding atmosphere. It also facilitated the removal of undesirable substances emanating from the curing resin by constantly using clean heat-transfer fluid which swept them out with its passage.

Table IV Cure Profile

Run	Cure Profile
65 and 66	149°C for 4 h, heated from initial temp[a]
67 thru 114	121°C for 5 h, heated from initial temp

[a] "Heated From Initial Temp" means that the resin was heated from an initial temperature corresponding to the minimum overall cure temperature recorded for this run.

Initially, a single heater was used to heat the heat-transfer fluid, which was then split into two streams: one delivered heated gas to the exposed and one to the control ovens. After each successive run, it became increasingly more difficult to thermally equalize the exposed and control oven and mold pack set's temperatures at the start of each run with this system.

An independent heater and gas delivery system was, as depicted in Figure 3, eventually settled upon and built for each oven. A single feed of nitrogen gas was used to provide the heat-transfer fluid for both ovens. With these two heat-transfer fluid heating and transport systems it was possible to finely regulate both the volume and temperature of the gas delivered to each oven.

Cure Profile

The EPON 830–MDA ERS cast in each run was cured according to one of the temperature profiles described in Table IV. The cure profiles used in all of the runs started out with the resin being cast into both exposed and control molds at roughly room temperature. Bone-dry nitrogen gas was already flowing through the ovens as the resin was cast. The heaters were then turned on to a predetermined setting. The mold pack temperature, and all other needed temperatures, were all measured using type T thermocouples and recorded on an Esterline–Angus data recorder. The temperature of the mold packs was then allowed to rise naturally to a selected temperature. The mold packs were then maintained within +/−5.6°C of the predetermined curing temperature, and the temperature of the exposed and control mold packs associated with any particular run were also maintained within +/−5.6°C of each other. At the end of the elevated temperature cure profile, the heaters were powered down and the unheated nitrogen gas was allowed to flow over the mold packs to cool them.

Throughout the entire forced heating sequence of the cure, for each run, temperature measurements using type T thermocouple wires were made at the bottom of the feed trough in the exposed and the control mold stacks. The type T thermocouples were selected because they are made of very low susceptibility materials that did not measurably modify the strength of the MFs to which the curing ERSs were exposed. This temperature sampling point was arbitrarily selected to be the temperature of all of the curing resins at any particular sampling time. These temperatures were sampled and logged on an Esterline–Angus data recorder, as were all of the other temperatures taken in this effort, every 10 min, at the start of a run (i.e., just after the resin was cast), at the powering down of the heaters during a run, and whenever a temperature reading was needed to make a decision. The maximum and minimum temperatures experienced by a thermocouple were also recorded. Statistics were generated with all of these temperature readings for the curing resins in the exposed and control mold packs for each run. Their averages, stds, and extremes appear in Table V.

In addition to specific cure-temperature statistics, a measure of exactly how well the temperature of the exposed mold pack for any particular run was maintained relative to the temperature of its corresponding control mold pack was determined. For each run, at each temperature sampling time, the difference between the exposed mold pack's temperature and the control mold pack's temperature was determined. This was defined to be the "delta." The average, stds, and extremes associated with these delta for each run were also determined and also appear in Table V.

All of the statistics associated with the exposed and control mold packs and their relative deltas were determined for two different ranges of the cure profile. One set was determined for the entire forced heating duration associated with the curing of each run's ERS. These statistics are listed in Table V in the Overall Cure column. Another set was determined for the temperatures of the ERS's cure profile after that cast resin had attained the preselected cure temperature and continuing through to the end of the forced heating section of their cures. These statistics are listed in Table V in the Post Heat-up column.

An examination of the cure temperature profile statistics reveals the following. All specimens cast in each run, whether exposed or control, experi-

Table V Cure Temperatures

	Overall Cure (°C)				Post Heat-up (°C)			
Run	Mean +/− Std	Min	Max	[Data Points]	Mean +/− Std	Min	Max	[Data Points]
65 Exposed	38 +/− 33	23	156	[19]	151 +/− 1	149	156	[16]
65 Delta	2 +/− 3	−1	+9	[19]	2 +/− 2	0	+8	[16]
65 Control	136 +/− 34	24	151	[19]	149 +/− 1	147	151	[16]
66 Exposed	136 +/− 32	23	151	[20]	147 +/− 12	99	151	[17]
66 Delta	−1 +/− 3	−11	+7	[20]	−2 +/− 3	−11	+1	[17]
66 Control	137 +/− 33	24	155	[20]	148 +/− 10	111	155	[17]
67 Exposed	113 +/− 24	30	131	[34]	122 +/− 2	121	131	[28]
67 Delta	−1 +/− 3	−4	+9	[34]	−2 +/− 1	−4	+2	[28]
67 Control	114 +/− 27	27	133	[34]	124 +/− 2	123	133	[28]
68 Exposed	115 +/− 26	22	132	[34]	125 +/− 2	123	132	[28]
68 Delta	1 +/− 4	−4	+12	[34]	−1 +/− 2	−4	+1	[28]
68 Control	114 +/− 28	21	132	[34]	126 +/− 2	123	132	[28]
69 Exposed	119 +/− 16	47	129	[30]	123 +/− 2	121	129	[26]
69 Delta	−1 +/− 4	−7	+14	[29]	−2 +/− 1	−7	−2	[25]
69 Control	119 +/− 19	37	136	[30]	126 +/− 3	123	136	[26]
70 Exposed	114 +/− 21	20	128	[31]	121 +/− 2	119	128	[26]
70 Delta	1 +/− 3	−2	+11	[30]	−1 +/− 1	−2	+1	[25]
70 Control	114 +/− 23	18	130	[31]	122 +/− 2	120	130	[26]
81 Exposed	113 +/− 23	17	123	[38]	122 +/− 1	121	123	[30]
81 Delta	1 +/− 3	−9	+9	[38]	1 +/− 1	−4	+2	[30]
81 Control	113 +/− 24	14	125	[38]	121 +/− 2	119	125	[30]
87 Exposed	118 +/− 23	23	148	[31]	126 +/− 6	122	148	[27]
87 Delta	−1 +/− 2	−9	+4	[31]	0.2 +/− 1	−1	+4	[27]
87 Control	119 +/− 22	23	143	[31]	125 +/− 5	122	143	[27]
90 Exposed	113 +/− 28	26	146	[33]	124 +/− 5	118	146	[27]
90 Delta	0.1 +/− 2	−2	+10	[33]	−0.02 +/− 2	−2	+10	[27]
90 Control	113 +/− 27	26	136	[33]	124 +/− 3	120	136	[27]
99 Exposed	112 +/− 27	20	126	[33]	122 +/− 1	121	126	[28]
99 Delta	1 +/− 4	−16	+7	[33]	3 +/− 2	+2	+7	[28]
99 Control	111 +/− 24	23	121	[33]	119 +/− 1	117	121	[28]
101 Exposed	124 +/− 10	NA	159	[33]	127 +/− 7	124	159	[29]
101 Delta	3 +/− 4	−2	+23	[33]	3 +/− 4	+1	+23	[29]
101 Control	116 +/− 22	25	136	[36]	124 +/− 3	121	136	[29]
105 Exposed	115 +/− 28	16	124	[31]	124 +/− 1	123	124	[28]
105 Delta	1 +/− 4	−11	+4	[29]	2 +/− 4	−11	+4	[26]
105 Control	114 +/− 27	19	134	[31]	123 +/− 4	120	134	[28]
106 Exposed	114 +/− 27	17	126	[31]	123 +/− 1	122	126	[28]
106 Delta	−1 +/− 3	−7	+4	[29]	−1 +/− 3	−7	+3	[26]
106 Control	115 +/− 28	18	133	[31]	124 +/− 4	120	133	[28]
109 Exposed	112 +/− 24	21	126	[24]	122 +/− 2	117	126	[19]
109 Delta	−7 +/− 10	−30	+4	[24]	−7 +/− 11	−30	+4	[19]
109 Control	119 +/− 27	22	148	[24]	129 +/− 9	119	148	[19]
110 Exposed	115 +/− 26	22	142	[24]	122 +/− 1	121	123	[14]
110 Delta	6 +/− 12	−8	+41	[24]	−1 +/− 4	−8	+3	[14]
110 Control	109 +/− 24	22	129	[24]	122 +/− 4	119	129	[14]
113 Exposed	119 +/− 23	19	141	[32]	126 +/− 4	124	141	[28]
113 Delta	1 +/− 3	−9	+8	[32]	2 +/− 2	+1	+8	[28]
113 Control	118 +/− 21	22	133	[32]	124 +/− 3	122	133	[28]
114 Exposed	118 +/− 24	19	152	[32]	126 +/− 7	122	152	[28]
114 Delta	−1 +/− 3	−7	+4	[31]	0.2 +/− 2	−4	+4	[27]
114 Control	119 +/− 23	22	148	[32]	126 +/− 7	121	148	[28]

Measurement accuracy: +/−1°C.

enced a transient temperature spike of up to 36.1°C higher than the average temperature at which they were intended to be cured. This was due to a partial runaway of the curing reaction which usually occurred just after the ERS's temperature had reached the desired overall cure temperature. Nothing could be done with the available equipment to correct for this transient temperature spike. The ERS's temperature quickly dropped back to the desired cure temperature and, roughly, remained there. Additionally, the vast majority of the curing profile experienced by the ERS in each run is represented in the Post Heat-Up column in Table V. As seen from those results, the average temperature for each mold pack in each run was routinely kept to within +/−5.6°C of the desired cure temperature. Also, the delta between the exposed and control mold packs for each run throughout their elevated temperature cure profiles was usually less than +/−5.6°C. The cure temperature profile statistics in Table V indicate that the ERSs cast in the corresponding exposed and control mold packs associated with the various runs of this effort experienced effectively the same cure temperature profile. Also, the cure temperature profile actually experienced by the ERSs in this effort was, for all intents and purposes, the desired cure profile listed in Table IV.

Experimentation Sequence

Each of the steps delineated in Table VI was sequentially followed for each run. The temperatures of EPON 830 and MDA at addition appear in Table VII.

Characterization

Relevant tensile mechanical properties were determined from specimens generated in each run of this effort. Also, the T_g and a measure of the degree of cure of the ERSs cured in each run was determined.

Mechanical Characterization

Uniaxial tension tests were conducted on the specimens generated in this effort. Each specimen was tested using a tension test with a constant extension rate of 0.508 mm/min. All of the strain data acquired throughout this entire effort was measured with the same MTS extensometer. From the stress-versus-strain curve data generated from each successfully tension-tested specimen, tensile stress and strain at yield were determined (if that specimen had a yield point, as were the ultimate tensile stress and strain, the Young's modulus, and the gauge cross-sectional area normalized, energy to failure toughness.

The extremely tight toleranced jigs, fixtures, and clevis depicted in Figure 11 were required for this effort. According to ASTM standards, failures in the tab and or neck-down regions of a specimen require the rejection of that test from any final reported results.[137–139] As a result of this, the reject failure rate attributed to this equipment was less than one in 200.

Thermal Characterization

Differential scanning calorimetry (DSC) scans were conducted on three to five specimens cut out of one of the dog bone specimens generated in each condition of each run in this effort. Each DSC scan used the same profile. Except for the first scan in any day's continuous effort, which started at room temperature, the scan was begun at a sample temperature of between 50 and 100°C, the temperature was then ramped up at a rate of 10°C/min, and the run was terminated when the specimen reached 200°C. All specimens were air-sealed in hermetic aluminum pans and heated under a dry nitrogen flow of 50 ml/min.

From the heat flow-versus-temperature curve data acquired from a DSC scan, the T_g's were determined. T_g was taken to be the temperature found at the lowest point of the heat-flow spike associated with the heat-flow change generated by a second-order transition in an almost (but not completely)-cured thermoset material.

An estimate of the residual heat of reaction (H_{res}) of the material was hand-calculated from selected DSC curves that were extreme cases for each different type of thermal cure profile used. The area under the curve was measured from the T_g point on the curve until the curve stopped climbing and straightened out. From this area and the mass of the DSC specimen, the H_{res} could be determined.

Only four distinct combinations of cure-temperature profiles and MDA concentrations were used. Table VIII delineates these experimental cure-style combinations. To determine a measure of the degree to which the specimens were cured, the heat of reaction (H_{rxn}) of the cure style at the cure temperature for the EPON 830–MDA ERS

Table VI Experimentation Sequence

Step	Action Accomplished
1	An electromagnet is turned on and stabilized for at least 12 h at a preselected current.
2	The control and MF-exposed ovens are loaded with dummy mold packs, heated to the desired operating temperature, and stabilized for long-duration use to within 2.8°C of each other and the desired operating temperature.
3	Mold packs are assembled from cleaned and waxed mold clamps and cleaned and dry RTV-664 silicone rubber mold cavity negatives.
4	The packs are heated to the desired curing temperature plus 28°C for 30 min to 1 h, then placed in a vacuum desiccator and cooled to room temperature while being vacuum degassed overnight.
5	The mold packs are removed from the vac chamber and precisely placed into the control and exposed ovens.
6	The mold pack's initial position in the MF is mapped.
7	The balances are leveled, turned on, and stabilized.
8	The EPON 830 and the MDA are removed from their desiccated and dry nitrogen atmosphere storage environments and added to cleaned flasks to be heated.
9	The balances are electronically calibrated and all timekeeping and/or timed data recording devices are synchronized.
10	The EPON 830 and the MDA are heated to a low-viscosity condition.
11	A predetermined amount of heated EPON 830 is added to a clean Pyrex beaker. The temperature of the EPON 830 at its addition to this beaker is listed in Table VII.
12	A stoichiometric amount of preheated MDA is then added to the resin in the beaker. The temperature of the MDA added to the beaker is also listed in Table VII.
13	The EPON 830 and the MDA are vigorously mixed for approximately 1 min.
14	The resulting ERS is immersed into an ice-water cooling vat until its temperature is reduced to the desired casting temperature.
15	Bone-dry unheated nitrogen gas is set flowing through the ovens.
16	The cooled ERS is degassed for 15 min.
17	The degassed ERS is cast into the MF-exposed mold pack and then within 1 min into the associated control mold pack.
18	Within 1 min of casting the control mold pack, the heaters are turned on and heated, bone-dry nitrogen gas is set flowing through the ovens and around the exposed and control mold packs.
19	The data recorder is initiated and a recording of the casting's temperatures is logged within 1 min of the start of heated gas flow.
20	The mold packs are naturally heated to the desired curing temperature and once at that temperature, manually maintained to within 5.6°C of it and each other for the desired cure duration.
21	One minute prior to the end of the desired curing duration, a final resin temperature is logged and the peak and valley temperatures measured by and retained in the data recorder's memory are logged.
22	After the desired curing duration has elapsed the heaters are shut down and cold, bone-dry nitrogen gas is allowed to flow over the mold packs until they have cooled to room temperature.
23	The mold pack's final position in the MF is mapped.
24	The mold packs are removed from their respective ovens and placed into polyethylene Ziploc® storage bags along with packs of indicating Drierite desiccant.
25	The mold packs are removed from their Ziploc bags and the exact position of the rubber mold cavity negatives is determined relative to their position in the mold pack.
26	The rubber mold cavities are removed from the mold clamps and the individual cast specimens are stripped from their rubber negative molds, trimmed, inspected under a 10× magnifier for defects, and then sorted out as mechanically testable, thermally testable, or untestable. The individual specimens are then placed in individual polyethylene Ziploc bags along with packs of indicating Drierite desiccant. All of these bags, for each condition, are then put into larger polyethylene Ziploc bags with more indicating Drierite desiccant. Finally, all of the specimen bags, for each condition of each run, are placed into a final, larger polyethylene Ziploc bag with more indicating Drierite desiccant and stored.

Table VI *Continued*

27	The MF strength measuring system is turned on, zeroed, and checked for stability by being run overnight.
28	The three-axis positioner is turned on and rezeroed. The maximum volume of the zone depicted in Figure 7 of the MF in which the resin specimens could possibly have been positioned at any time while they were being cured is determined from the initial and final positions of the mold pack in the ovens and from the position of the rubber mold cavity negatives in the mold packs; and then mapped out by measuring the MF at between 72 and 385 points within and at the edge of that zone.
29	Specimens suitable for mechanical testing are removed from their desiccated storage bags and measured.
30	The Sintech mechanical testing machine is calibrated for a continuous session of tensile testing.
31	Specimens are mechanically tested and the results from those specimens not falling in an unacceptable location or on a previously undetected flaw are retained.
32	Specimens suitable for thermal analysis are removed from their desiccated storage bags, cut, trimmed to suitable sizes, and returned to their bags.
33	Thermal analysis specimens are weighed and hermetically sealed in analysis cans.
34	The DSC is calibrated for a continuous session of thermal analysis.
35	DSC runs are conducted on the sealed specimens and the results are retained.

used was calculated based on the works of Sourour and Kamal[140] and Horie and colleagues[141] for amine cured epoxies. One result of their work was that small increases in the concentration of the curing agent above stoichiometric had little if no measurable effect on the base ERS's H_{rxn}. Based upon their work and the experimental findings of this paper's author in his previous efforts, there were in fact only two H_{rxn}s relevant to the various cure styles used in this effort. Table IX lists those calculated H_{rxn}s for those two more-simplified cure styles. Worst-case degrees of cure for the different cure styles used in this effort were calculated from a percentage ratio of the H_{res} to the H_{rxn} calculated for that specific cure style.

Table VII EPON 830 and MDA Temperatures at Addition

	Addition Temp (°C)			
	EPON 830		MDA	
Run	Begin	End	Begin	End
65	RT	RT	125	NA
66	RT	RT	135	NA
67	RT	RT	115	NA
68	RT	RT	135	NA
69	RT	RT	125	NA
70	RT	RT	120	NA
81	RT	RT	119	107
87	RT	RT	139	115
90	RT	RT	130	138
99	93	NA	110	130
101	100	NA	120	128
105	95	86	112	122
106	95	86	112	122
109	97	93	128	117
110	97	93	128	117
113	95	93	130	120
114	95	93	130	120

Measurement accuracy: +/−0.5°C; RT, room temperature; NA, not available.

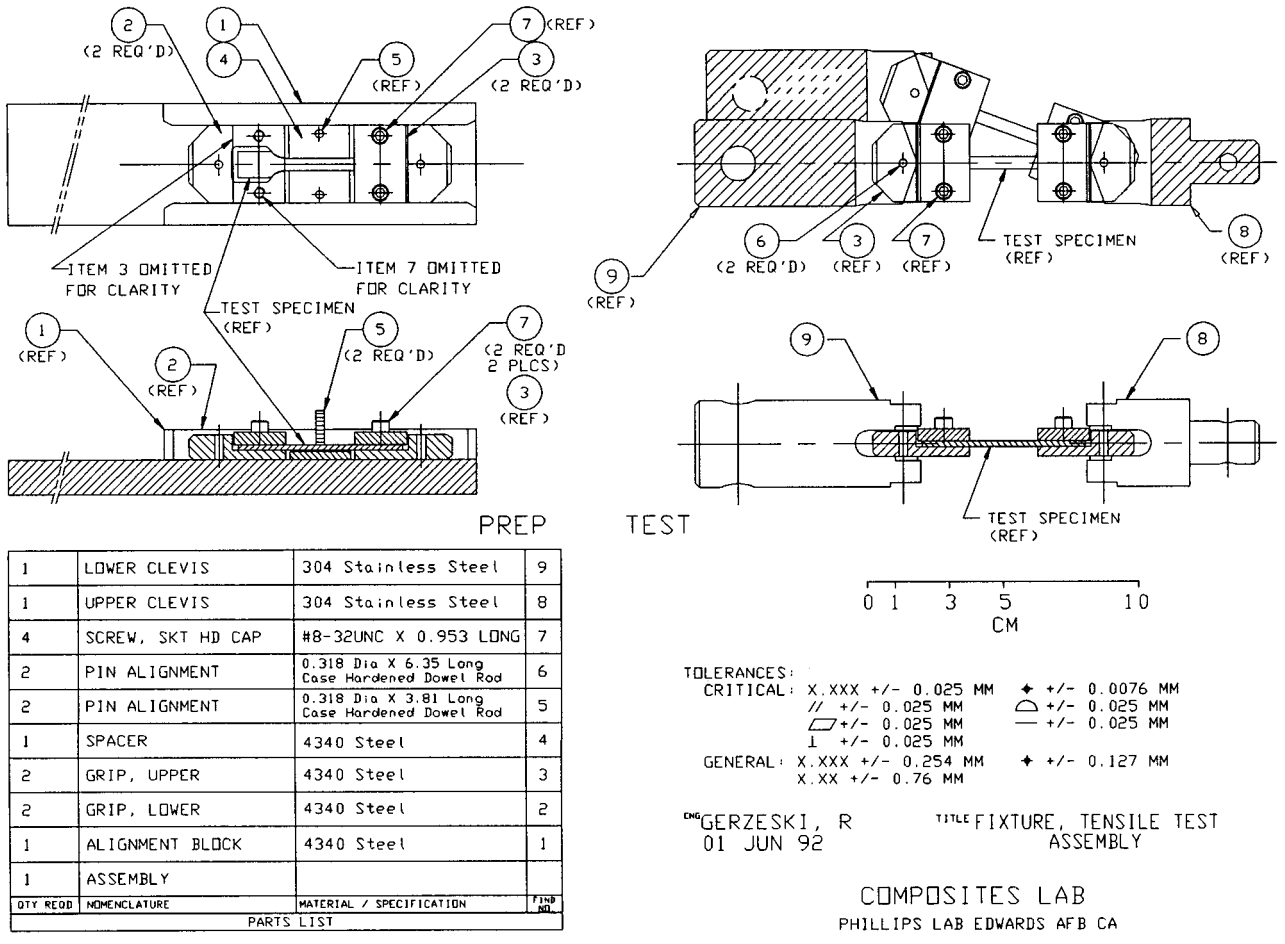
RESULTS

Mechanical Studies Results

Core Mechanical Properties Results

Table X lists the tensile stress at yield and the ultimate tensile stress of the exposed and corresponding control specimens measured for the runs in this effort. There are no discernible differences in the measured peak stresses between those specimens generated under any MF strength and their corresponding controls. Also, there are no discernible differences in the measured break stresses between those specimens generated under any MF strength and their associated controls.

Table X also lists the strain to yield (STY) and the strain to failure (STF) of the MF-exposed and corresponding control specimens measured for



QTY REQD	NOMENCLATURE	MATERIAL / SPECIFICATION	FIND NO.
1	LOWER CLEVIS	304 Stainless Steel	9
1	UPPER CLEVIS	304 Stainless Steel	8
4	SCREW, SKT HD CAP	#8-32UNC X 0.953 LONG	7
2	PIN ALIGNMENT	0.318 Dia X 6.35 Long Case Hardened Dowel Rod	6
2	PIN ALIGNMENT	0.318 Dia X 3.81 Long Case Hardened Dowel Rod	5
1	SPACER	4340 Steel	4
2	GRIP, UPPER	4340 Steel	3
2	GRIP, LOWER	4340 Steel	2
1	ALIGNMENT BLOCK	4340 Steel	1
1	ASSEMBLY		

PARTS LIST

Figure 11 Tensile test fixture assembly.

the runs in this effort. With two possible exceptions, there are no discernible differences in the peak strains between those specimens generated under any MF strength and their corresponding controls. Also, there are, with two possible exceptions, no discernible differences in the break strains between those specimens generated under any MF strength and their associated controls.

Runs 99 and 114 are the only runs to exhibit a difference in the STY and STF results between their MF-exposed and control specimens. The STY and the STF results for the control specimens of these runs are larger than the same results for the corresponding MF-exposed specimens. Also, the range of these strain results for the control specimens is marginally larger than and does not overlap the range of the strain results for the associated exposed specimens. Student's *t*-test analysis of the raw strain numbers is listed in Table XI. Based on these numbers, there is a high degree of confidence in the difference between the raw number-generated mean of Run

Table VIII Cure Styles

Concentration PHR	Thermal Cure Profile
25.5	121°C 5 h
26.0	121°C 5 h
27.0	149°C 4 h
28.0	149°C 4 h

PHR, parts per hundred resin.

Table IX Heat of Reaction, H_{RXN}

Cure Temperature (°C)	H_{rxn} (J/gm)
121	417
149	460

These heats of reaction are derived from the works of Sourour and Kamal[140] and Horie and associates.[141]

Table X Core Mechanical Properties

Run	Tensile Stress at Yield[a] (Peak Stress) (MPa)				Ultimate Tensile Stress (Break Stress) (MPa)				Strain to Yield[b] (Peak Strain) (%)				Strain to Failure (Break Strain) (%)			
	Mean +/− Std	Min	Max	[Data Points]	Mean +/− Std	Min	Max	[Data Points]	Mean +/− Std	Min	Max	[Data Points]	Mean +/− Std	Min	Max	[Data Points]
65 Exposed	76.7 +/− 1.1	75.8	78.2	[4]	76.7 +/− 1.1	75.8	78.2	[4]	9.1 +/− 1.0	8.3	10.3	[4]	9.1 +/− 1.0	8.3	10.3	[4]
65 Control	76.8 +/− 1.9	74.4	79.2	[6]	76.4 +/− 1.6	74.4	78.4	[6]	9.3 +/− 1.2	7.7	10.6	[6]	10.1 +/− 2.2	7.7	13.1	[6]
66 Exposed	78.1 +/− 4.2	75.3	84.5	[4]	76.6 +/− 4.2	72.8	82.6	[4]	9.5 +/− 0.2	9.2	9.5	[4]	11.2 +/− 1.2	10.2	12.9	[4]
66 Control	75.4 +/− 2.8	70.3	82.6	[6]	73.6 +/− 3.0	69.2	76.6	[6]	9.0 +/− 0.5	6.7	11.0	[6]	9.6 +/− 2.9	6.7	13.9	[6]
67 Exposed	78.2 +/− 2.4	75.9	81.7	[5]	78.0 +/− 2.0	75.9	80.7	[5]	6.4 +/− 0.9	5.5	7.9	[5]	6.6 +/− 1.4	5.5	9.0	[5]
67 Control	79.3 +/− 1.6	77.2	81.1	[5]	79.1 +/− 1.6	77.2	80.5	[5]	7.6 +/− 0.9	6.1	8.6	[5]	7.9 +/− 1.3	6.1	9.7	[5]
68 Exposed	78.0 +/− 3.5	75.4	83.0	[4]	77.9 +/− 3.4	75.4	82.9	[4]	6.8 +/− 1.1	5.6	8.3	[4]	6.8 +/− 1.1	5.6	8.3	[4]
68 Control	72.0 +/− 4.0	69.5	77.8	[4]	71.9 +/− 3.9	69.5	77.8	[4]	5.4 +/− 1.7	4.5	8.0	[4]	5.5 +/− 1.9	4.5	8.3	[4]
69 Exposed	82.9 +/− 1.5	80.9	85.3	[6]	82.8 +/− 1.5	80.8	85.3	[6]	7.3 +/− 1.0	5.3	8.3	[6]	7.5 +/− 1.2	5.3	8.5	[6]
69 Control	79.6 +/− 4.5	72.3	84.4	[6]	79.5 +/− 4.5	72.3	84.4	[6]	6.8 +/− 1.6	5.1	8.5	[6]	6.9 +/− 1.7	5.1	8.5	[6]
70 Exposed	80.1 +/− 3.4	77.7	82.5	[2]	80.1 +/− 3.4	77.7	82.5	[2]	7.1 +/− 1.2	6.3	7.9	[2]	7.1 +/− 1.2	6.3	7.9	[2]
70 Control	79.8 +/− 2.5	76.4	81.8	[4]	79.7 +/− 2.5	76.4	81.7	[4]	6.9 +/− 0.9	6.2	8.2	[4]	7.0 +/− 1.1	6.2	8.5	[4]
81 Exposed	79.1 +/− 4.0	75.5	83.4	[3]	79.1 +/− 4.0	75.5	83.4	[3]	6.2 +/− 1.9	4.8	8.4	[3]	6.2 +/− 1.9	4.8	8.4	[3]
81 Control	81.8 +/− 2.3	80.0	84.4	[3]	81.8 +/− 2.3	80.0	84.4	[3]	6.7 +/− 1.4	5.8	8.3	[3]	6.7 +/− 1.4	5.8	8.3	[3]
87 Exposed	82.2 +/− 1.9	79.8	84.4	[5]	81.8 +/− 1.8	79.8	84.4	[5]	7.2 +/− 0.4	6.5	7.7	[5]	7.5 +/− 0.6	6.5	8.0	[5]
87 Control	81.9 +/− 3.1	75.3	84.5	[7]	81.4 +/− 2.9	75.3	84.0	[7]	7.3 +/− 1.1	5.3	8.4	[7]	7.9 +/− 1.8	5.3	9.9	[7]
90 Exposed	82.8 +/− 1.1	81.3	84.2	[6]	81.3 +/− 2.3	77.4	84.1	[6]	7.9 +/− 0.6	7.2	8.8	[6]	9.1 +/− 2.0	7.3	12.6	[6]
90 Control	84.2 +/− 1.2	82.1	85.4	[6]	82.0 +/− 1.3	80.6	83.6	[6]	8.0 +/− 0.5	7.0	8.5	[6]	9.5 +/− 1.2	7.4	11.0	[6]
99 Exposed	81.6 +/− 2.6	78.7	83.5	[3]	81.6 +/− 2.6	78.7	83.5	[3]	5.9 +/− 0.4	5.5	6.2	[3]	5.9 +/− 0.4	5.5	6.2	[3]
99 Control	85.7 +/− 0.2	85.5	85.8	[2]	85.7 +/− 0.2	85.5	85.8	[2]	7.3 +/− 0.4	7.0	7.5	[2]	7.3 +/− 0.4	7.0	7.5	[2]
101 Exposed	82.7 +/− 1.6	80.7	85.2	[6]	82.0 +/− 1.8	79.5	84.2	[6]	7.9 +/− 0.7	6.9	8.8	[6]	8.6 +/− 1.3	6.9	10.4	[6]
101 Control	83.7 +/− 2.5	79.0	85.7	[6]	83.4 +/− 2.3	79.0	85.7	[6]	7.7 +/− 1.0	5.8	8.7	[6]	8.4 +/− 1.5	5.8	10.1	[6]
105 Exposed	83.7 +/− 1.1	81.8	84.6	[5]	83.7 +/− 1.1	81.8	84.5	[5]	6.8 +/− 0.7	5.7	7.4	[5]	6.8 +/− 0.7	5.7	7.4	[5]
105 Control	80.5 +/− 2.8	76.1	83.7	[5]	80.4 +/− 2.8	76.1	83.7	[5]	6.6 +/− 1.2	5.0	8.1	[5]	6.8 +/− 1.4	5.0	8.6	[5]
106 Exposed	84.1 +/− 2.4	82.1	86.7	[4]	84.1 +/− 2.3	82.1	86.7	[4]	6.5 +/− 0.9	5.7	7.4	[4]	6.6 +/− 1.0	5.7	7.5	[4]
106 Control	83.6 +/− 1.5	81.4	84.5	[4]	83.2 +/− 1.5	81.4	84.5	[4]	7.3 +/− 0.8	6.4	8.3	[4]	7.6 +/− 0.9	6.4	8.5	[4]
109 Exposed	82.4 +/− 3.9	77.7	86.5	[4]	81.4 +/− 3.4	77.7	85.9	[4]	7.1 +/− 1.7	5.3	9.0	[4]	8.0 +/− 2.7	5.3	10.5	[4]
109 Control	83.0 +/− 0.6	82.6	83.7	[3]	83.0 +/− 0.5	82.6	83.6	[3]	8.4 +/− 1.2	7.0	9.1	[3]	8.4 +/− 1.2	7.0	9.1	[3]
110 Exposed	85.3 +/− 1.0	84.2	86.5	[4]	84.3 +/− 0.6	83.7	85.2	[4]	8.2 +/− 0.3	7.7	8.5	[4]	9.6 +/− 2.0	8.0	12.4	[4]
110 Control	83.2 +/− 3.5	78.1	86.0	[4]	83.0 +/− 3.4	78.1	85.9	[4]	8.0 +/− 1.6	5.6	8.9	[4]	8.5 +/− 1.9	5.6	9.7	[4]
113 Exposed	81.2 +/− 4.2	76.4	84.1	[3]	81.2 +/− 4.2	76.4	84.1	[3]	7.3 +/− 1.7	5.4	8.8	[3]	7.3 +/− 1.8	5.4	9.0	[3]
113 Control	81.6 +/− 7.1	71.0	85.9	[4]	81.6 +/− 7.1	71.0	85.9	[4]	7.3 +/− 2.0	4.4	8.7	[4]	7.6 +/− 2.2	4.5	9.6	[4]
114 Exposed	83.1 +/− 3.2	78.3	85.6	[4]	83.1 +/− 3.2	78.3	85.6	[4]	6.9 +/− 1.2	5.4	8.2	[4]	6.9 +/− 1.2	5.4	8.2	[4]
114 Control	84.7 +/− 0.9	84.0	85.6	[3]	84.1 +/− 1.3	83.0	85.5	[3]	8.3 +/− 0.4	8.0	8.7	[3]	8.9 +/− 0.5	8.5	9.4	[3]
Avg Runs 69, 70, 81, 87, 90, 99, 101, 105, 106, 109, 110, 113, 114																
Ov[c] Control	82.6 +/− 1.9	71.0	86.0	[57]	82.2 +/− 1.8	71.0	85.9	[57]	7.4 +/− 0.6	4.4	9.1	[57]	7.8 +/− 0.9	4.5	11.0	[57]
Avg Runs 67–70, 81, 87, 90																
Ov[c] Control	79.8	69.5	85.4		79.3	69.5	84.4		7.0	4.5	8.6		7.3	4.5	11.0	

[a] Measurement accuracy: +/−0.6 MPa.
[b] Measurement accuracy: +/−0.5%.
[c] Overall.

Table XI Strain Discrepancies

Contrasting Runs	Property	Magnitude	t^c	Confidence
99 Exposed versus All Like Cured Controls	STF[a]	24% Reduction	1.880	95%
99 Exposed versus All Like Cured Controls	STY[b]	20% Reduction	1.867	95%
99 Exposed versus 99 Control	STF	19% Reduction	4.138	97.5%
99 Exposed versus 99 Control	STY	19% Reduction	4.138	97.5%
114 Exposed versus All Like Cured Controls	STF	11% Reduction	0.892	80%
114 Exposed versus All Like Cured Controls	STY	6% Reduction	0.535	70%
114 Exposed versus 114 Control	STF	25% Reduction	2.622	95%
114 Exposed versus 114 Control	STY	19% Reduction	1.861	90%

[a] STF: Strain to failure.
[b] STY: Strain to yield.
[c] Student's *t*-test analysis of raw strain numbers.

99 Exposed and its associated Control and all like cured controls. For Run 114 there is a sufficiently high degree of confidence in the difference between the raw number-generated mean of Run 114 Exposed relative to its associated Control, but only an 80% confidence relative to all like cured controls for the STF results and a 70% confidence for the STY results. Since a 90% confidence level is required in all comparisons if we are to accept the hypothesis that there is a difference between these raw number means, Run 114's differences are not significant on statistical grounds alone.

Based on the following reasoning the differences between Run 99's Exposed and Control STY and STF results are also not significant. The range of the exposed strain values is well within the range of the overall average of the controls. Also, the raw number statistics in Tables X and XI do not take into account the measurement inaccuracies of the extensometer used to measure strain during the mechanical testing. These add an additional +/−0.5% inaccuracy to the exposed strain measurements and another +/−0.5% to the control strain measurements, for a total additional measurement inaccuracy of +/−1.0%. Based on all of the above, and most particularly on the additional +/−1.0% strain inaccuracy resulting from the extensometer, the differences between Run 99's Exposed and Control STY and STF values are not significant. It is more reasonable to attribute these differences to the inaccuracy contribution of the extensometer than to MF exposure.

Derived Mechanical Properties Results

Table XII lists the Young's modulus (modulus) of the MF-exposed and corresponding control specimens for the runs in this effort. There are no effective differences in the modulus of those specimens generated under any MF strength exposure relative to their associated controls.

Table XII also lists the toughness, as measured from the areas under the stress-versus-strain curves, of the MF-exposed and corresponding control specimens for the runs in this effort. With two exceptions, there are no resolvable differences between the toughness exhibited by those specimens generated under any MF strength exposure and their associated controls.

Runs 99 and 114 are the only runs to exhibit a difference between the toughness of specimens generated in an MF relative to their associated controls. The average toughness results for the control specimens are larger than the same results for the MF-exposed specimens. Also, the range of these toughness results for the two control specimens are marginally larger than and do not overlap the range of the toughness results for the corresponding exposed specimens. Student's *t*-test analysis of the raw toughness numbers are listed in Table XIII. Based on these numbers, there is a high degree of confidence in the difference between the raw number-generated mean of Run 99 Exposed and its associated Control and all like cured controls. For Run 114 there is a high degree of confidence in the difference between the raw number-generated mean of Run 114 Exposed and its associated Control but only an 80% confidence relative to all like cured controls. Since a 90% confidence level is required in both comparisons if we are to accept the hypothesis that there is a difference between these raw number means,

Table XII Derived Mechanical Properties

	Modulus (GPa)				Toughness (Energy to Break/Area) (J/cm^2)			
Run	Mean +/− Std	Min	Max	[Data Points]	Mean +/− Std	Min	Max	[Data Points]
65 Exposed	2.25 +/− 0.10	2.15	2.38	[4]	142.0 +/− 21.9	123.3	168.9	[4]
65 Control	2.34 +/− 0.12	2.19	2.45	[6]	165.1 +/− 51.7	113.8	231.7	[6]
66 Exposed	2.16 +/− 0.14	2.04	2.36	[4]	193.0 +/− 37.7	164.5	247.8	[4]
66 Control	2.11 +/− 0.10	2.03	2.26	[6]	151.7 +/− 65.4	86.6	250.7	[6]
67 Exposed	2.62 +/− 0.19	2.36	2.80	[5]	102.0 +/− 32.5	76.6	158.7	[5]
67 Control	2.59 +/− 0.06	2.50	2.65	[5]	130.3 +/− 29.7	89.6	172.8	[5]
68 Exposed	2.52 +/− 0.16	2.41	2.75	[4]	104.0 +/− 27.7	77.3	142.6	[4]
68 Control	2.45 +/− 0.16	2.30	2.61	[4]	74.4 +/− 38.3	53.9	131.8	[4]
69 Exposed	2.61 +/− 0.46	2.33	3.53	[6]	124.4 +/− 24.7	79.5	148.2	[6]
69 Control	2.53 +/− 0.14	2.39	2.79	[6]	108.8 +/− 40.4	64.8	151.3	[6]
70 Exposed	2.76 +/− 0.41	2.48	3.05	[2]	112.9 +/− 28.9	92.5	133.3	[2]
70 Control	2.54 +/− 0.21	2.29	2.81	[4]	109.5 +/− 26.0	90.1	146.3	[4]
81 Exposed	2.54 +/− 0.01	2.54	2.56	[3]	95.6 +/− 43.3	63.7	145.0	[3]
81 Control	2.56 +/− 0.14	2.41	2.68	[3]	107.3 +/− 31.7	86.1	143.8	[3]
87 Exposed	2.43 +/− 0.09	2.30	2.52	[5]	122.8 +/− 12.7	101.7	134.8	[5]
87 Control	2.47 +/− 0.06	2.38	2.54	[7]	134.5 +/− 42.6	71.0	179.7	[7]
90 Exposed	2.42 +/− 0.12	2.25	2.59	[6]	159.6 +/− 44.2	119.4	238.8	[6]
90 Control	2.45 +/− 0.13	2.29	2.61	[6]	171.1 +/− 29.2	122.1	205.6	[6]
99 Exposed	2.50 +/− 0.11	2.37	2.57	[3]	86.9 +/− 13.2	77.6	96.3	[3]
99 Control	2.49 +/− 0.12	2.41	2.57	[2]	120.7 +/− 8.1	114.9	126.4	[2]
101 Exposed	2.29 +/− 0.12	2.18	2.50	[6]	148.3 +/− 32.0	107.0	194.7	[6]
101 Control	2.38 +/− 0.12	2.23	2.59	[6]	147.3 +/− 36.5	82.8	188.3	[6]
105 Exposed	2.53 +/− 0.09	2.46	2.66	[5]	110.9 +/− 15.1	86.4	124.5	[5]
105 Control	2.56 +/− 0.08	2.49	2.67	[5]	108.3 +/− 31.5	68.1	152.9	[5]
106 Exposed	2.62 +/− 0.04	2.59	2.66	[4]	109.3 +/− 25.9	86.2	132.8	[4]
106 Control	2.52 +/− 0.12	2.39	2.65	[4]	129.4 +/− 23.1	97.3	148.7	[4]
109 Exposed	2.41 +/− 0.12	2.34	2.59	[4]	137.1 +/− 64.3	72.6	197.0	[4]
109 Control	2.31 +/− 0.14	2.20	2.46	[3]	139.3 +/− 24.1	111.5	154.1	[3]
110 Exposed	2.32 +/− 0.07	2.23	2.39	[4]	175.2 +/− 50.8	137.0	245.8	[4]
110 Control	2.30 +/− 0.04	2.27	2.36	[4]	145.6 +/− 45.7	77.6	175.4	[4]
113 Exposed	2.30 +/− 0.06	2.25	2.37	[3]	117.3 +/− 42.0	72.0	155.1	[3]
113 Control	2.36 +/− 0.12	2.27	2.53	[4]	126.2 +/− 51.9	53.2	172.9	[4]
114 Exposed	2.36 +/− 0.10	2.23	2.44	[4]	111.6 +/− 27.7	76.2	140.6	[4]
114 Control	2.34 +/− 0.08	2.25	2.39	[3]	158.5 +/− 12.5	145.1	169.8	[3]
Avg Runs 69, 70, 81, 87, 90, 99, 101, 105, 106, 109, 110, 113, 114								
Ov[a] Control	2.45 +/− 0.10	2.20	2.81	[57]	131.2 +/− 20.6	53.2	205.6	[57]
Avg Runs 67–70, 81, 87, 90								
Ov[a] Control	2.51	2.29	2.81		119.4	53.9	205.6	

[a] Overall.

Run 114's differences are not significant on statistical grounds alone.

Based on the following reasoning, the differences between Run 99's Exposed and Control toughness are also not significant. The toughness values for Run 99, Exposed and Control, are less than the overall average toughness values. This indicates that the specimens, both Exposed and Control, generated from this batch of resin were marginal to begin with. Also, the range of the exposed toughness values for Run 99 is well within the range of the overall average for all of the controls. Based on these points, the difference between Run 99's Exposed

Table XIII Toughness Discrepancies

Contrasting Runs	Magnitude	t[a]	Confidence
99 Exposed versus All Like Cured Controls	34% Reduction	1.819	95%
99 Exposed versus 99 Control	28% Reduction	4.089	95%
114 Exposed versus All Like Cured Controls	15% Reduction	0.855	80%
114 Exposed versus 114 Control	30% Reduction	2.684	97.5%

[a] Student's t-test analysis of raw toughness numbers.

and Control toughness values are not significant.

Figures 12 and 13 show the stress-versus-strain curves of Runs 69 and 87, respectively. These curves are representative of the stress-versus-strain curves resulting from the mechanical testing of specimens from the runs in this effort. As seen from these curves, there is no discernible difference between those for specimens generated while exposed to a MF and those for their simultaneously generated controls.

Thermal Studies Results

T_g Results

Table XIV lists the T_gs of the MF-exposed and corresponding control specimens measured for the runs in this effort. With one exception, there are no discernible differences in the T_gs exhibited by those specimens generated under any MF strength exposure relative to their controls.

Run 110 is the only run which exhibits a minutely discernible difference in the T_g results between MF-exposed and control specimens. The T_g average results for the control specimens are marginally larger than the same results for the MF-exposed specimens. Also, the range of these T_g results for the control specimens is larger than and does not overlap the range of the T_g results for the exposed specimens. The Student's t-test confidence statistics conducted on the raw T_g numbers are listed in Table XV. They indicate that the very minute T_g reductions in the exposed specimens relative to the various controls are statistically significant. These differences in the exposed and control T_gs are, in fact, *not* significant due to the following: Run 110's control average and range values are high in comparison to the overall control average and range; whereas, Run 110's exposed average and range are well within the overall control's range. Also, the cure profile collected on this run was incomplete. As seen in Table V, only 12 temperature samplings were collected during the Post Heat-up section of the cure, when in fact 27 samplings should have been collected over this 4-h period. Due to a mistake by the author the first 2 h of temperature sampling in this section of the cure were not collected. When collection was resumed the ovens had stabilized, with the control oven operating 8°C hotter than the exposed oven. Based upon the author's experience with the Type 1 ovens used to generate these specimens, it is reasonable to assume that this temperature difference had been sustained for most of the lost sampling time. Redetermining the cure profile with this assumption indicates that the control specimens were cured at an overall temperature 4°C higher than the exposed, and this shift should also be seen in the relative T_gs. Also, the statistics in Tables XIV and XV do not take into account the measurement inaccuracies of the

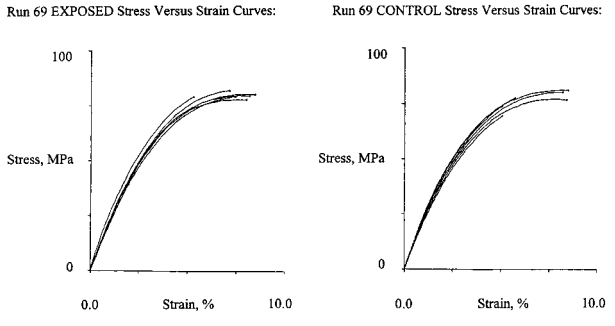


Figure 12 Run 69 stress-versus-strain curves.

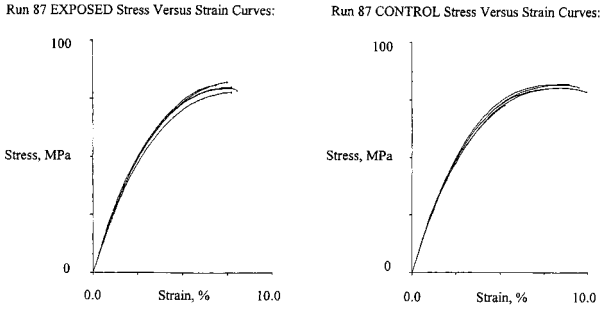


Figure 13 Run 87 stress-versus-strain curves.

Table XIV Glass Transition Temperatures (T_gs)

	T_g (°C)					T_g (°C)			
Run	Mean +/− Std	Min	Max	[Data Points]	Run	Mean +/− Std	Min	Max	[Data Points]
65 Exposed	167 +/− 1	166	169	[4]	66 Exposed	168 +/− 0.4	168	169	[4]
65 Control	168 +/− 1	166	169	[4]	66 Control	168 +/− 0.1	168	169	[4]
67 Exposed	155 +/− 1	154	156	[4]	68 Exposed	154 +/− 3	149	156	[4]
67 Control	158 +/− 1	157	158	[4]	68 Control	156 +/− 2	155	158	[4]
69 Exposed	153 +/− 1	152	154	[3]	70 Exposed	151 +/− 1	150	152	[4]
69 Control	156 +/− 0.4	156	157	[2]	70 Control	154 +/− 1	153	155	[4]
81 Exposed	150 +/− 1	149	151	[3]	87 Exposed	156 +/− 0.2	156	157	[3]
81 Control	151 +/− 0.3	151	151	[3]	87 Control	154 +/− 0.4	154	154	[3]
90 Exposed	150 +/− 1	149	151	[3]	99 Exposed	150 +/− 1	150	151	[3]
90 Control	146 +/− 1	145	146	[3]	99 Control	145 +/− 1	144	146	[3]
101 Exposed	152 +/− 1	152	153	[3]	105 Exposed	153 +/− 4	148	156	[3]
101 Control	145 +/− 1	144	147	[3]	105 Control	152 +/− 2	150	153	[3]
106 Exposed	148 +/− 3	146	151	[3]	109 Exposed	148 +/− 1	148	150	[3]
106 Control	154 +/− 1	153	155	[3]	109 Control	159 +/− 1	158	160	[3]
110 Exposed	146 +/− 1	145	147	[3]	113 Exposed	156 +/− 1	155	157	[3]
110 Control	154 +/− 0.4	154	154	[3]	113 Control	155 +/− 1	154	155	[3]
114 Exposed	153 +/− 1	152	153	[3]					
114 Control	154 +/− 2	152	156	[3]					
Avg Runs 69, 70, 81, 87, 90, 99, 101, 105, 106, 109, 110, 113, 114									
Ov[b] Control	152 +/− 4	141	160	[40]					
Avg Runs 67–70, 81, 87, 90									
Ov[b] Control	154	146	158						

Measurement accuracy: +/−1.1°C.
[b] Overall.

thermocouples used to measure the temperature of the ovens or the DSC. These inaccuracies add an additional +/−1°C to each of the cure profile temperatures and an additional +/−1.1°C to each of the T_g measurements, for a total additional measurement inaccuracy of +/−4.2°C. Based on all of the above, with emphasis on the cure profile temperature sampling mistakes, the difference between Run 110's Exposed and Control T_g values is not significant. It is more reasonable to attribute these T_g differences to the combination of these sampling errors, the more probable cure-temperature profile differences, the measurement inaccuracies, and the statistical number inaccuracies listed in Tables V and XIV, than to exposure to the MF.

Degree of Cure Results

Table XVI lists the worst-case degrees of cure found for the exposed and control specimens generated in the various runs associated with each listed cure style. All specimens generated in this effort were cured to 99+% of full cure for that curing temperature. Also, there was no effective

Table XV Glass Transition Temperature Discrepancies

Contrasting Runs	Magnitude	t[a]	Confidence
110 Exposed versus All Like Cured Controls	4% Reduction	2.637	99%
110 Exposed versus 110 Control	5% Reduction	12.235	99.5%

[a] Student's t test confidence statistics.

Table XVI Degrees of Cure

Concentration Range (PHR)	Thermal Cure Profile	Worst-case Degree of Cure	
		Control (%)	Exposed (%)
25.5 to 26.0	121°C 5 h	99.5	99.5
27.0 to 28.0	149°C 4 h	99.9	99.9

PHR, parts per hundred resin.

difference between the degree of cure found in specimens cast while exposed to MFs relative to those cast as their associated controls.

Figures 14 and 15 show the heat flow-versus-temperature DSC curves of Runs 69 and 87, respectively. These curves are representative of the DSC curves resulting from the thermal testing of specimens from all of the runs and the curing styles used. As seen from these curves, there is no discernible difference between those for specimens generated while exposed to a MF and those for their associated controls.

DISCUSSION

Control Properties Comparisons

Table XVII lists the various relevant control specimen-derived mechanical and thermal properties for the 25.5 parts per hundred resin 99+% MDA 121°C 5-h curing style used in this effort. Table XVIII lists the published values of these same properties for similar-base ERSs cured with analogous concentrations of MDA, but with 150°C multi-hour cures. When the information in these two tables is compared, three facts can be deduced: (1) The stress values and the T_g values are effectively identical. (2) The modulus values for this effort's control specimens are only slightly reduced from the referenced values. (3) The strain values are as much as twice as great for this effort's control-generated specimens as compared with the referenced values.

The slightly reduced values of the control's modulus relative to the reference's values, and the substantially increased control strain values relative to those of the reference, are direct results of the cure temperatures used. The 121°C temperature profile used to generate the specimens and their subsequent values listed in Table XVII resulted in the generation of moderately highly crosslinked, and subsequently less stiff and substantially tougher, cured ERSs than the referenced ERSs listed in Table XVIII. The referenced values in Table XVIII were reported to be generated from specimens which were cured at 29°C hotter temperatures. This modest increase in cure temperature obviously resulted in more brittle, but stiffer, cured ERSs.

When the results tabulated in Table XVII for the control specimens of this effort are contrasted and compared with the reference values listed in Table XVIII, they are effectively equivalent. This equivalence indicates that the techniques used to generate the specimens and then test them are reasonable. It also indicates that any lack of difference between the properties of the MF-exposed specimens of any one run relative to its associated control's properties is the result of the nonexistence of any property enhancement resulting from the processing and not an unanticipated resultant of the experimental technique. In essence, the technique produced reasonable and usable control specimens which, when tested, subsequently produced reasonable and valid control results. It therefore follows that this technique would have clearly indicated any enhancements to the properties of specimens generated while simultaneously exposed to a MF if there had been any enhancements to find.

Mechanical Properties

Overall, the mechanical properties of stoichiometric EPON 830–MDA ERSs that have been fully

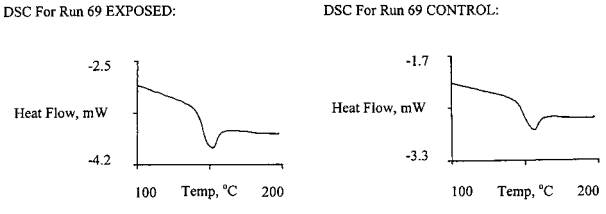


Figure 14 RUN 69 DSCs.

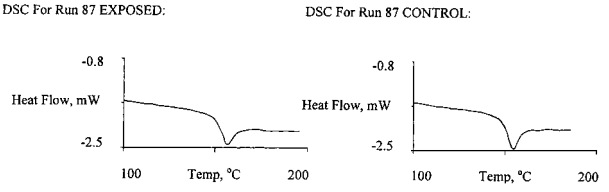


Figure 15 RUN 87 DSCs.

Table XVII Overall Observed Control

Property	Units	Mean +/− Std	Min	Max	Mean +/− Std	Min	Max
Stress	MPa	Tensile stress at yield (peak stress)			Ultimate tensile stress (break stress)		
		82.6 +/− 1.9	71.0	86.0	82.2 +/− 1.8	71.0	85.9
		Measurement accuracy: +/− 0.6 MPa					
Strain	%	Strain to yield (peak strain)			Strain to failure (break strain)		
		7.4 +/− 0.6	4.4	9.1	7.8 +/− 0.9	4.5	11.0
		Measurement accuracy: +/−0.5%					
Modulus	GPa	2.45 +/− 0.10	2.20	2.81			
T_g	°C	152 +/− 4	141	160			
		Measurement Accuracy: +/−1.1°C					

cured while simultaneously exposed to the MF strengths and associated ranges delineated in Table III are not enhanced by MF exposure. Enhancements to the mechanical properties of these ERSs may exist when selected, very tightly controlled MF strengths within this overall range are used, but these enhancements are not apparent from this experimental effort's results and appear to not be viable for incorporation into actual production devices.

These mechanical results do not corroborate the extensive list of mechanical property enhancement results published by the Soviets.[7–135] The Soviets indicated that they were able to achieve numerous enhancements in the mechanical properties of similar ERSs cured in similar ways while simultaneously exposed to similar MF strengths.

Thermal Properties

Overall, the thermal properties of ERSs that have been fully cured while simultaneously exposed to the MF strengths and associated ranges delineated in Table III are not enhanced by MF exposure. Enhancements to the thermal properties of ERSs may exist when selected, very tightly controlled MF strengths are used within this overall range, but they are not indicated from this experimental effort's results.

These thermal results do not corroborate the thermal property enhancement results published by the Soviets.[7–135] The Soviets indicated that they were able to achieve enhancements of up to 12°C in the T_gs of similar ERSs cured in similar ways while being simultaneously exposed to similar MF strengths.

CONCLUSION

This experimental effort's objective was to determine whether enhancements to the properties of stoichiometric mixes of MDA, an aromatic epoxy curing agent, and EPON 830, a DGEBA-based

Table XVIII Reference Reported Control[136]

Property	Units	Mean +/− Std	Min	Max	Mean +/− Std	Min	Max
Stress	MPa	Tensile stress at yield (peak stress)			Ultimate tensile stress (break stress)		
				88			
Strain	%	Strain to yield (peak strain)			Strain to failure (break strain)		
							5.8
Modulus	GPa			2.8			
T_g	°C			160			
			150	160			

epoxy resin, could be generated by fully thermally curing them while simultaneously exposing them to MFs whose strengths could be economically generated. Previous efforts by the author and an independent effort by Dr. Mallon, then at Aerospace Corp., suggested that the potential to enhance particular properties of ERSs (by processing them with conventional production techniques into end items while simultaneously exposing them to MFs of strengths that could be generated by permanent magnets and conventional electromagnets) was highly probable.

This effort decisively determined that under most conditions of conventional elevated temperature cure and economically generated MF strengths there was no modification to the important properties of fully cured stoichiometric EPON 830–MDA ERSs relative to their associated controls.

From the results of previous experimental efforts, the author concludes that this effect is real. From the results of this experimental effort, the author concludes that this effect is not economically viable for incorporation into conventional EPON 830–MDA ERS-based production devices.

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