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MASTER

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REPORT NO. CASD-LLL-79-004

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**EVALUATION OF TWO POLYURETHANE
RESINS FOR INJECTION SHIMMING
OF THE MFTF MAGNET**

GENERAL DYNAMICS
Convair Division

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EVALUATION OF TWO POLYURETHANE RESINS FOR INJECTION SHIMMING OF THE MFTF MAGNET

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SUMMARY

This work was conducted to support selection of a filler for the injection shimming of the MFTF Magnet (Reference 1). Two candidate polyurethanes (Polycast RG256-57-3 and Polycast 1009-78 manufactured by CPR Upjohn Co.) were evaluated with respect to their physical and mechanical properties. Physical properties examined include thermal contraction, viscosity, and cure shrinkage. Mechanical properties include thermal shock resistance, compression modulus, and compression strength at LH₂ temperature.

Based on the results from the above tests, Polycast RG256-57-3 was chosen as the superior filler material over Polycast 1009-78 for injection shimming of the MFTF magnet. It is recommended that (1) further evaluation of Polycast RG256-57-3 be conducted through injection of the material into a scaled representative segment of a simulated MFTF magnet assembly and (2) procedures be developed to ensure the reliability of injection shimming for the magnet.

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INTRODUCTION

Welded construction of the thick plate for the MFTF magnet case requires a relatively large tolerance to accommodate stock flatness, forming accuracy, and weld distortion. The resulting tolerance buildup can create an uneven internal clearance between the case and the winding pack, which must be uniformly filled to attain a continuity which ensures uniform distribution of cooldown and magnetic load effects.

Based on the given geometry of the magnet and the associated design requirements, two room-temperature curing polyurethane resins were selected to be evaluated as candidate filler materials. This is based on the fact that urethanes are generically more reliable at cryogenic temperatures than other materials pertinent to our application. Addition of fillers (such as glass beads or chopped fibers) to the organic resin to lower the cure shrinkage and thermal contraction was considered. However, arbitrary incorporation of fillers would actually invite processing difficulties and possibly porosity in the filled material. Thus, a filled resin was not pursued in this program.

Since "urethanes" embrace a variety of polymers varying in physical and mechanical properties, it is important that the candidate filler materials be evaluated for their handling characteristics and performance properties under the representative service conditions.

The main selection criteria were:

- 6000 psi minimum in compressive strength and 1×10^6 psi minimum in compressive modulus at LHe temperature (to endure the high electromagnetic operating forces from the coil, Reference 1)
- Low thermal contraction from 300°K to 4°K (to minimize any losses in pack-to-case continuity due to contraction of the filler upon cooling)
- Negligible dimensional change upon full cure of the material
- Low viscosity and long work life of the mixed material (i.e., resin mixed with curing agent) for ease of injection of the material into the space between the winding pack and the case.

This report covers the evaluation of two candidate urethanes based on the above criteria. The evaluation included the following:

1. Thermal contraction of the urethanes from 300°K to 20°K with an intermediate point at 80°K using a quartz tube dilatometer.
2. Thermal cycling of the urethanes 50 times from 20°K to 300°K by alternately submerging in liquid hydrogen and gaseous hydrogen at 300°K.
3. Compressive strength and the modulus of the urethanes at 20°K. (Both thermally cycled and control specimens were tested to determine the effect of thermal cycling).
4. Viscosity of the urethane resin mixture (for both urethanes).
5. Volume contraction upon full cure of the urethanes.

The low temperature tests were performed at 20°K (LH₂ temperature) rather than the magnet service temperature of 4°K (LH₂ temperature) to minimize costs and still produce usable data.

Very little information on the thermal contraction characteristics is gained extending the testing from 20°K to 4°K. Most materials experience very small changes in length at cryogenic temperatures. Thus, the change in length of the material below 20°K could be safely extrapolated.

The thermal cycling effects on the material are the result of stresses caused by the rate of cooling rather than the exact final temperature to which the material is cooled. The cooling rates produced by directly submerging the specimen in LH₂ were far greater than those predicted for the actual service.

The compressive properties of the materials are expected to be very similar between 20°K and 4°K, since mechanical properties at cryogenic temperatures generated for organic resin based composites have shown similar trends (Reference 2).

EXPERIMENTAL

Two polyurethane resins were received from CPR Upjohn on 19 August 1979. These were: Polycast RG256-57-3, Part A lot 908-1155

Part B lot 809-1545

Polycast 1009-78

Part A lot 809-1538

Part B lot 809-1545

2.1 SPECIMEN PREPARATION

The test specimens were mixed in accordance with CPR Upjohn's recommended mixing ratio (Polycast RG256-57-3 at 49.4 of A to 50.6 of B by weight and Polycast 1009-78 at 42.6 of A to 57.4 of B by weight) and degassed until bubble-free at room temperature prior to casting. The test specimen molds were coated with release agent (Frekote-33) and baked at 250°F for 30 minutes prior to casting the specimens. During casting, it was noted that Polycast RG256-57-3 was more prone to bubble formation than Polycast 1009-78. This could indicate that Polycast RG256-57-3 is more sensitive to moisture.

For thermal cycling and compression tests, the specimens were cast in 5/16 inch I.D. glass tubing. Upon full R.T. cure, the specimens were obtained by shattering the glass tubing. A 5-inch long cylinder was cut for thermal cycling. Compression specimens were machined from the thermally cycled as well as an uncycled rod to approximately 5/8 inch in height (height and diameter of the specimen ratioed 2:1 per Reference 3). The ends of the specimens were carefully machined to a smooth surface with sharp and clean edges. The ends were flat and parallel, and perpendicular to the long axis of the specimen to within 0.001 inch.

Thermal contraction specimens measuring approximately 3.5 inch x 1 inch x 0.085 inch were cut from cast sheets of the urethanes.

2.2 TEST PROCEDURE

2.3 THERMAL CYCLING

Thermal cycling of the specimen was accomplished by suspending the 5-inch long specimen from a stainless steel tube. The tube passed through a

concentric tubular electric heater (maintained at $300 \pm 2^\circ\text{K}$) into a cryostat containing LH_2 . The specimen was held at the heater temperature and LH_2 temperature for three minutes each alternatively to achieve a total of 50 cycles. The temperature of the test specimen was monitored by a thermocouple taped at the center of the specimen. The cooling rate produced by this procedure ranged from 50°F to 500°F .

2.4 THERMAL CONTRACTION

Measurements on the urethanes were made on a General Dynamics quartz tube dilatometer shown in Figure 1. The test specimen was held in tension by Invar grips attached to concentric fused silica tubes. Changes in the relative positions of the upper ends of the tubes, representing changes in specimen length, were recorded by a .0001 inch dial indicator.

The lower end of the tube assembly was inserted into an environmental chamber which can be stabilized at any temperature between -320°F and $+400^\circ\text{F}$. The chamber was sealed around the outer tube and a dry helium purge was maintained inside the tubes. Temperature in the chamber was controlled by electrically balancing heat flow to a LN_2 bath. Specimen temperature was monitored by three thermocouples.

Effects of dimensional changes in the low expansion Invar grips on the measurement were further minimized by the self-compensating design. The upper ends of the tubes and the indicator mechanism were thermally stabilized by an ambient temperature helium flow.

Tension load on the specimen during test was approximately 4 oz.

Normal test procedure is to adjust the chamber temperature and hold the specimen temperature constant. Accuracy of the measurement is approximately $\pm 5 \times 10^{-6}$ in/in over the full temperature range. This is less than 1% of the $\Delta l/l$ over the full temperature range of a specimen with a coefficient of 7×10^{-6} in/in $^\circ\text{F}$ or greater.

2.5 COMPRESSION

For modulus measurements, two strain gages (WK-13-062 AP-350 by Micromasurements, Inc.) were attached to each specimen. The gages were centered, positioned lengthwise at 180° from each other, and bonded onto the specimen with Micromasurements, Inc. AE10 epoxy cement (cured at room temperature).

FIGURE 1. Tube Dilatometer for Thermal Contraction Measurement

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The specimen was positioned in the test fixture (see Figure 2) which was placed in the cryostat. Strain gage leads were attached to the Portable Digital Strain Indicator Model 1200 data acquisition system. The lid was placed on the cryostat. The system was purged with gaseous helium to remove any oxygen in the cryostat. The cryostat was then filled with liquid hydrogen and allowed to equilibrate for approximately two minutes. The LH₂ was maintained at a level above the test fixture.

The specimen was then incrementally loaded at 100 pound intervals. The load and the strain were recorded at each interval. The loading was continued until a strain reading reached greater than 15000 microstrain ($\text{in/in} \times 10^{-6}$). At that point, strain readings were discontinued and the specimen was loaded uniformly to failure at a crosshead speed of 0.1 inch/minute.

2.6 VISCOSITY

Viscosity was measured using a viscosimeter (Brookfield Synchro-lectric Model LVF). The sample was heated separately to 90°F, poured into a beaker kept in a 90°F water bath, and viscosity of the material was measured as a function of time. The viscosity was measured with spindle #4, rotating at 6 RPM.

2.7 VOLUME CONTRACTION

Materials were poured into Pyrex test tubes (#9826). The meniscus levels were marked with tape. After cure, the volumetric contraction of each specimen was measured. This measurement was made under the assumption that the meniscus shape had undergone no change during cure.

The volume contraction was obtained with an optically aligned vernier caliper. The caliper was held next to the tube while reading. Since the tape was aligned by eye it was necessary to do the same with the caliper to keep the refraction error to a minimum.

By estimating the bottom of the tube to be a hemisphere and the rest to be a cylinder the initial volume of the material was obtained. The percentage of volumetric contraction was calculated based on the initial volume and the final volume (after full cure) of the material (see Figure 3).

FIGURE 2. Compression Test Fixture

FIGURE 3. Volume Contraction Test Specimen

RESULTS AND DISCUSSION

3.1 THERMAL CYCLING

Polycast RG256-57-3 (henceforth referred to as RG256) withstood the thermal cycling without fracturing. However, the length of the specimen that contained small air bubbles turned cloudy, possibly evidence of micro-cracks within the material (see Figure 4).

Polycast 1009-78 (henceforth referred to as 1009) was more susceptible to thermal shock. Two specimens were actually tested. The first fractured and broke into pieces after 10 cycles. The second specimen showed a conchoidal fracture pattern at the center of the specimen (where the thermocouple was taped) after 12 cycles. After an additional 13 cycles, the material showed crazing at the tip of the specimen (see Figure 5).

The thermal shock imposed on the test specimens using this thermal cycling procedure is many times more severe than that anticipated in the real service condition. The maximum thermal shock occurred upon cooling when the skin reached LH_2 temperature while the core was still warm by comparison. The outer surface was contracting faster than the core, thus creating a large thermal stress in the material. The cooling rate produced by this thermal cycling procedure ranged from 56° to $500^\circ F$ per second.

Thermal shock was much less of a problem upon warming since the heat transfer medium was a gas, which has a much lower heat transfer capability compared to the liquid cooling medium. Thus, the temperature change rates were much lower.

3.2 THERMAL CONTRACTION

Results of thermal contraction measurements of RG256 and 1009 are presented in Table 1, and Figures 6 and 7 respectively. RG256 showed a larger thermal contraction than 1009. Assuming the thermal expansion of the material remained a linear function of the temperature, the coefficient of thermal expansion for RG256 is approximately 32×10^{-6} in/in $^\circ F$ from R.T. to LH_2 and 36×10^{-6} in/in $^\circ F$ from R.T. to LN_2 . For 1009, the coefficient of thermal expansion is approximately 15×10^{-6} in/in $^\circ F$ from R.T. to LH_2 and 19×10^{-6} in/in $^\circ F$ from R.T. to LN_2 .

FIGURE 4. Thermally Cycled Polycast RG256-57-3

FIGURE 5. Thermally Cycled Polycast 1009-78

FIGURE 6. Thermal Contraction, Polycast RG256-57-3

FIGURE 7. Thermal Contraction of Polycast 1008-78

Coefficients of thermal contraction of both materials were greater between R. T. and LN_2 than between R. T. and LH_2 . (In the case of 1009, the contraction which occurred between LN_2 and LH_2 was extremely small.) This change in contraction slope could have occurred at a temperature higher than LN_2 ; molecular activity begins decreasing rapidly at low temperatures causing noticeable property changes.

3.3 COMPRESSION

Due to the partial internal fracturing of 1009 upon thermal cycling, insufficient compression specimens were obtained from the thermally cycled rod. There were also difficulties initially in bonding strain gages onto the specimens. As a result, some of the thermally cycled specimens were rendered useless.

The compression modulus and strength determined at LH_2 for both RG256 and 1009 are presented in Table 2. The compression modulus for both materials is approximately 1.4×10^6 psi. The compression strengths are quite comparable as well. All test specimens fractured and failed in a mode similar to a brittle glassy material. One thermally cycled RG256 specimen (#1-2) was included to determine the extent to which the compression properties of the material were affected by the presence of air bubbles and, possibly, microcracks (see Figure 4). While there was almost no difference detectable by modulus measurement, the compressive strength of that specimen was approximately 75% of the unfractured specimens.

The one thermally cycled 1009 specimen (#3), which had a visible crack, had a lower modulus as well as lower strength. One specimen (#2) machined from the middle section of the thermally cycled rod (see Figure 5) failed while being installed into the test fixture.

The compressive modulus and strength of both materials appeared unaffected by thermal cycling where there was no visible damage such as cracks, air bubbles, or microcracks. Both materials more than met the MFTF performance requirements of 1×10^6 psi compression modulus and 6,000 psi compression strength at LH_2 .

3.4 VISCOSITY

The viscosity of both materials was monitored up to 100,000 cps, whereupon the consistency was hard to stir, hard to pour, and definitely difficult to inject. The viscosity at 90°F of both materials as a function of time is tabulated in Table 3 and a comparison is shown in Figure 8.

The viscosity of 1009 increased much faster than RG256. In less than 60 minutes, 1009 had reached close to 100,000 centipoise, while RG256 took greater than 150 minutes to reach 100,000 centipoise.

The importance of this time duration is only affected by the injection rate and temperature present at the time of injection. However, with the injection equipment available today, successful injection filling of either material into the MFTF assembly should not be a problem. Nevertheless, the longer work life of RG256 will ease the processing parameters with respect to injection rate and temperature.

3.5 VOLUME CONTRACTION

Table 4 shows the volume contraction measured for RG256 and 1009. The contraction upon full cure (or cure shrinkage) of 1009 is approximately twice that of RG256 (i.e. approximately 3.5% as opposed to 1.9% for RG256). The variance in the amount of contraction measured was in the order of $\pm 7\%$.

Figure 8. Viscosity of Polycast RG256-57-C And 1009-78 at 90°F

CONCLUSIONS

An overall assessment of the test results showed Polycast RG256-57-3 to be preferred over Polycast 1009-78 for injection shimming of the MFTF magnet.

The compression modulus and strength at LH_2 temperature of both materials were comparable. Polycast 1009-78 had advantages over Polycast RG256-57-3 in terms of lower thermal contraction and shorter cure time. Other work has also shown Polycast 1009-78 to be less susceptible to moisture. However, these are quickly overshadowed by the superior thermal shock resistance and lower cure shrinkage (which more than make up for the larger coefficient of thermal contraction) of Polycast RG256-57-3. The longer work life of Polycast RG256-57-3 will also ease the processing parameters.

RECOMMENDATIONS

Further evaluation of Polycast RG256-57-3 should be conducted through injection of the material into a scaled representative segment of a simulated MFTF magnet assembly. Procedures should also be developed in order to attain a reliable injection shimming system for the MFTF magnet.

REFERENCES

1. Structural Analysis of the Magnet System for Mirror Fusion Test Facility (MFTF), GD/C Report No. CASD-LLL-78-003, by J. W. Wohlwend, et al, October 1978.
2. Mechanical Characterization of G-10CR and G-11CR Glass Cloth/Epoxy Laminates Between Room Temperature and 4K, presented at Cryogenic Engineering/Materials Conference, Madison, Wisconsin, by M. B. Kasen, et al, August 21-24, 1979.
3. Standard Test Method for Compressive Properties of Rigid Plastics, ASTM D 695, 1977 Annual Book of ASTM Standards.

Table 1. Thermal Contraction of Polycast RG256-57-3 and Polycast 1009-78

Polycast Material	Spec. No.	Spec. Length (inches)	Temp.	$\Delta L/L$	$\alpha = \Delta L/L/^{\circ}F$
			$^{\circ}F$	$10^{-3}/in/in$	$10^{-6} in/in/^{\circ}F$
RG256-57-3	1	3.65	67.3	0.0	—
			-310.2	-13.59	35.8
			-395.8	-15.01	32.4
	2	3.62	56.3	0.0	—
			-311.0	-14.90	40.6
			-403.0	-16.30	35.5
	3 *	3.78	74.0	0.0	—
			-314.0	-13.36	34.5
			-409.0	-14.70	30.5
	4 *	3.84	58.0	0.0	—
			-320.0	-13.40	35.3
			-416.0	-15.80	33.3
	5 *	3.78	71.0	0.0	—
			-314.0	-12.88	33.5
			-409.0	-14.18	29.6
1009-78	1	3.60	71.3	0.0	—
			-316.0	-7.28	18.8
			-414.4	-7.25	14.9
	2	3.60	79.2	0.0	—
			-316.7	-6.44	16.3
			-414.9	-6.53	13.2
	3	3.61	74.4	0.0	—
			-318.3	-8.44	21.5
			-419.0	-8.14	16.5

*thickness for these specimens = 0.013 inch.
all others = 0.085 inch.

Table 2. Compression Properties of Polycast RG256-57-3 and Polycast 1009-78

Material	Specimen No.	Specimen Conditioning	Compressive Modulus, $\times 10^6$ psi	Compressive Strength, ksi
Polycast RG256-57-3	1-1	Uncycled	1.40	80.5
	1-2		1.43	86.4
	1-3		1.58	79.2
	1-4		1.54	80.1
			Avg 1.49	Avg 81.6
	1-1	Cycled	1.45	92.0
	1-2*		1.43*	61.6*
	1-3		1.42	76.9
	1		1.68	95.9
			Avg 1.50	Avg 81.8
Polycast 1009-78	6	Uncycled	1.33	77.3
	7		1.46	80.3
			1.41	52.1
			1.58	83.8
			Avg 1.45	Avg 73.4
	2**	Cycled	---	---
	3***		0.99***	30.5***
	4		1.56	89.5
			Avg -	Avg -
MFTF Design Requirement		-	1.00	6.0

*The specimen was machined from the cloudy section of thermally cycled rod (see Figure 4)

** The specimen was machined from the center, fractured section of thermally cycled rod (see Figure 5) and failed during installation of the specimen into the test fixture.

***The specimen had a small fracture within the material.

TABLE 3. Viscosity Measurements at 90°F of Polycast RG256-57-3 and Polycast 1009-78

Viscosity (centipoise) at 90°F

Time Minutes	Polycast RG256-57-3 Runs			Polycast 1009-78 Runs		
	1	2	3	1	2	3
0	0	4,500	4,500	-	-	-
10	6,800	-	-	-	1,700	3,300
15	7,600	5,500	-	6,700	4,000	6,400
20	-	-	-	-	8,650	11,000
25	11,000	9,000	-	15,600	16,100	17,200
30	-	-	11,000	24,000	27,000	25,000
35	-	12,500	-	36,000	40,000	35,000
40	-	-	-	51,000	59,000	47,000
45	18,200	15,000	16,000	64,000	81,000	60,000
50	-	-	-	77,000	98,000	77,000
55	-	19,500	-	97,000	-	98,000
60	22,000	-	22,000	-	-	-
65	-	23,000	-	-	-	-
75	30,000	27,000	28,000	-	-	-

TABLE 3 (Cont.)

Time Minutes	Polycast RG256-57-3 Runs			Polycast 1008-78 Runs		
	1	2	3	1	2	3
85	-	-	-			
90	37,000	32,000	33,000			
95	40,500	36,000	-			
105	-	41,000	41,000			
115	-	47,500	-			
120			49,000			
122	62,500	-	-			
125	-	52,500	-			
135	-	60,000	58,500			
142	82,000	-	-			
145	-	68,000	-			
150	-	-	69,000			
155	96,000	75,000	-			
165		84,000	83,000			
175		94,000	-			
180		-	95,000			

Table 4. Volume Contraction Upon Full Cure of Polycast
RG 256-57-3 and Polycast 1009-78

Material	No.	Change in Meniscus Level	Percentage Volumetric Contraction
Polycast 1009-78	1	0.153 $\pm$ 0.005 in.	3.2 $\pm$ 0.2%
	2	0.185 $\pm$ 0.005 in.	3.7 $\pm$ 0.2%
	3	0.178 $\pm$ 0.005 in.	3.5 $\pm$ 0.2%
Polycast RG-256-57-3	1	0.103 $\pm$ 0.005 in.	1.9 $\pm$ 0.2%
RG-256-57-3	2	0.096 $\pm$ 0.005 in.	1.9 $\pm$ 0.2%
RG-256-57-3	3	0.100 $\pm$ 0.005 in.	1.9 $\pm$ 0.2%