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Thermal Expansion of Epoxy–Fiberglass Composite Specimens[1]

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The thermal expansion behavior of three epoxy–fiberglass composite specimens was measured from 20 to 120°C (70 to 250°F) using a fused quartz push-rod dilatometer. Billets produced by vacuum-impregnating layers of two types of fiberglass cloth with an epoxy were core-drilled to produce cylindrical specimens. These were used to study expansion perpendicular and parallel to the fiberglass layers. This type of composite is used to separate the copper conductors that form a helical field coil in the Advanced Toroidal Facility, a plasma physics experiment operated by the Fusion Energy Division at Oak Ridge National Laboratory. The coil is operated in a pulsed mode and expansion data were needed to assess cracking and joint stresses due to expansion of the copper–composite system. The dilatometer is held at a preselected temperature until steady state is indicated by stable length and temperature data. Before testing the composite specimens, a reliability check of the dilatometer was performed using a copper secondary standard. This indicated thermal expansion coefficient (α) values within $\pm 2\%$ of expected values from 20 to 200°C. The percentage expansion of the composite specimen perpendicular to the fiberglass layers exceeded 0.8% at 120°C, whereas that parallel to the fiberglass layers was about 0.16%. The expansion in the perpendicular direction was linear to about 70°C, with an α value of over 55×10^{-6} °C^{-1}. Anomalous expansion behavior was noted above 70°C. The expansion in the direction parallel to the fiberglass layers corresponds to an α value of about 15×10^{-6} °C^{-1}. The lower α values in the parallel direction are consistent with the restraining action of the fiberglass layers. The α values decreased with the specimen density and this is consistent with literature data on composite contraction from 20 to -195°C.

KEY WORDS: anisotropic expansion; composites; dilatometry; epoxy; fiberglass; thermal expansion coefficient.

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1. INTRODUCTION

The copper conductors that form the helical field coil in the Advanced Toroidal Facility are mechanically separated by a fiberglass-reinforced epoxy composite for electrical insulation. The Fusion Energy Division, Oak Ridge National Laboratory, conducts plasma physics experiments in this facility [1]. The coil is operated in a pulsed mode. The differential expansion of the copper–epoxy system resulting from the expected temperature excursion due to a pulse could stress joints to failure and cause cracks to develop in the epoxy. This concern led to the need to measure the thermal expansion behavior of a set of epoxy–fiberglass composite specimens over a limited temperature range above room temperature.

Glass-reinforced epoxy laminates are used for structural supports and for electrical and thermal insulation in superconducting magnets. Mechanical, electrical, and thermal property characterizations have been reported for such laminates over the 300 to 4 K temperature range and these provide a basis for thermal expansion expectations above 300 K.

For example, Kasen et al. [2] and Fujii et al. [3] report thermal contraction data that show a directional dependence and indicate that the epoxy was a dominant factor in CR-grade glass–epoxy laminates with densities of 1.90 and 1.95 $g \cdot cm^{-3}$. Total contractions between room temperature and 4 K were 0.6% for the laminate with a density of 1.95 $g \cdot cm^{-3}$ for the perpendicular direction and 0.2% for the parallel direction. The lower-density laminate had about 10% greater contraction.

Wang et al. [4] report integral thermal contraction values ($\Delta L/L$) for micarta from 300 to 77 K of 0.85% in the perpendicular direction and 0.3 to 0.6% in the parallel direction. For comparison, $\Delta L/L_0$ values for OFHC copper and 304SS are 0.31 and 0.26%, respectively.

Hamelin [5] measured contraction from 200 to 77 K of glass–cloth-reinforced epoxy laminates parallel and perpendicular to the glass–cloth layers. The observed $\Delta L/L_0$ values decreased linearly with density and were significantly greater in the perpendicular direction. The epoxy filling fraction is responsible for these results and Hamelin found an increased curing pressure reduced $\Delta L/L_0$ by a factor of 3. These results provided a means to keep laminates under compression in external casings.

2. DILATOMETER

Thermal expansion measurements were made in the range 20 to 120°C (70 to 250°F) using a modification of a fused quartz push-rod dilatometer described by Kollie et al. [6–8]. Figure 1 is a cross section of the apparatus. The specimen is placed in a cylindrical copper holder to provide

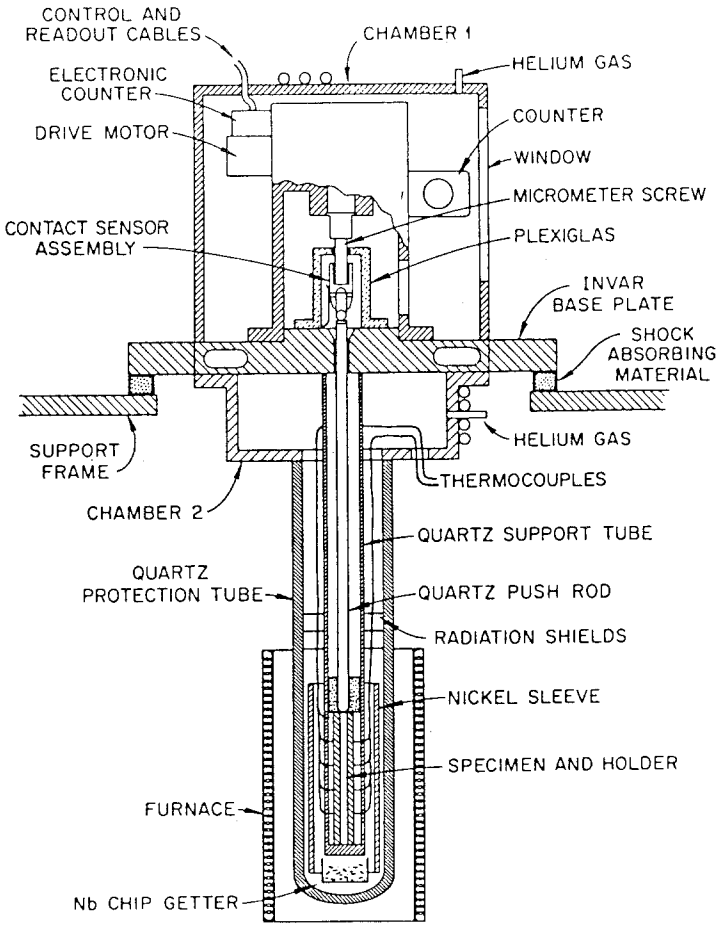

Fig. 1. Cross section of the fused quartz differential dilatometer showing the major components of the apparatus.

vertical stability and to reduce temperature gradients along its length. The holder rests on the platform of the quartz support tube. The support tube is attached to an Invar base plate which serves as the reference plane for length (L) measurements of the dilatometer. The L is the difference between the expansion of the support tube and the summed expansions of the specimen and the push rod. An automated micrometer, Carson-Dice Electronic Micrometer Model 71-A-9, is used to measure L and consists of a motor-driven micrometer screw readable to 1×10^{-6} in. and accurate to 10×10^{-6} in. Calibrated Pt/Pt–10 Rh thermocouples are used to measure temperature changes to ± 0.27 K. A vertically mounted tube furnace is used

to heat the dilatometer. To minimize temperature gradients in the specimen and its environs, the specimen and the lower portion of the support tube and push rod were placed inside a nickel sleeve. A quartz protection tube is used to maintain a pure helium atmosphere around the lower portion of the dilatometer. The operation of the dilatometer includes controlling the furnace temperature, measuring the specimen thermocouple emfs, and measuring the specimen length. The dilatometer is heated in succession to a series of temperatures and a thermal steady state is established at each. This yields reproducible temperature profiles in the dilatometer, which are necessary for accurate L measurements. The micrometer is read at least 40 times at each thermal steady state. These 40 readings typically exhibit a range of L values of less than $\pm 5 \times 10^{-6}$ in. The accuracy of the dilatometer has been assessed by measurements on NBS-certified copper from 300 to 800 K and NBS-certified tungsten from 300 to 1000 K. The thermal expansion coefficient, α, is calculated from length data (L) at two temperatures, T_1 and T_2, which can be selected to differ by any desired amount:[5]

$$\alpha = \left(\frac{1}{L_0}\right)\left(\frac{\Delta L}{\Delta T}\right) = \left(\frac{1}{L_0}\right)\left[\frac{\mathrm{L}(T_2) - \mathrm{L}(T_1)}{T_2 - T_1}\right] \tag{1}$$

where L_0 is the specimen length at room temperature. The value of α is assigned to the average temperature between readings, $0.5\ (T_2 + T_1)$. Kollie estimated the maximum uncertainty in α to be $\pm 0.8\%$ ($\pm 0.1 \times 10^{-6}\ \mathrm{K}^{-1}$) for copper using a 100 K temperature change and $\pm 1.4\%$ ($\pm 0.2 \times 10^{-6}\ \mathrm{K}^{-1}$) for a 50 K temperature change. The uncertainty for tungsten was approximately triple these values because of its lower α value (Cu, $\sim 18 \times 10^{-6}\ \mathrm{K}^{-1}$; W, $\sim 5 \times 10^{-6}\ \mathrm{K}^{-1}$). Consequently we have estimated uncertainties in $\Delta L/L_0$ and α at less than ± 1 and $\pm 2\%$, respectively, for a temperature change of 25 K.

3. MATERIALS

Two billets were produced by vacuum impregnating layers of fiberglass cloth with an epoxy resin. Table I shows that the epoxy resin

[5] Definitions of the terms are given under Nomenclature.

Table I. Epoxy Resin Formulation

Resin	100 parts by weight DOW No. DER-332 epoxy resin
Hardener	90 parts by weight nadic methylanhydride (NMA)
Flexibilizer	10 parts by weight Union Carbide Carbowax No. 400
Promoter	0.5 to 0.8 part by weight benzyldimethylamine (BMDA)

Table II. Epoxy Impregnation and Cure Cycle

Function	Time (h)	Temperature, °C (°F)
Impregnation and soak	4	54 (130)
Gel and initial cure	20	82 (180)
Final cure	30	121 (250)

Table III. Thermal Expansion Specimen Characteristics

Specimen	Dimension (in.)		Weight (g)	Density ($g \cdot cm^{-3}$)
	Length (L_0)	Diameter		
Textoglass perpendicular	3.0623 (3.0622)[a]	0.3985	11.305	1.806
Textoglass parallel	3.0624 (3.0622)	0.3992	11.400	1.814
Burlglass 1200 perpendicular	3.0595 (3.0578)	0.3993	8.2723	1.318

[a] Length after test given in parentheses.

Table IV. Reliability Test on 3-in. Copper (Q82-1)

Temperature (°C)	Micrometer length (μ in.)	$10^6\alpha$ (K^{-1})	$\frac{T_1+T_2}{2}$ (°C)	Expected $10^6\alpha$ (K^{-1})	Expected Difference (%)
24.0	69,778	17.4	62.9	17.1	+1.7
101.8	73,841	17.7	151.6	17.9	−1.1
201.5	79,138	17.8	111.5	17.6	+1.1
21.6	69,509				

included four components: resin, hardener, flexibilizer, and promoter. Table II shows the epoxy impregnation and cure cycle. The Textoglass billet included layers of 7-mil-thick J. P. Stevens Company (Aberdeen, N.C.) Textoglass tape, and the Burlglass 1200 billet was made using layers of fiberglass manufactured by Burlington Industries (Greenboro, N.C.). Right-circular cylinders were machined from these billets to allow expansion tests perpendicular and parallel to the Textoglass tape layers and perpendicular to the Burlglass 1200 layers. Table III shows the dimensions and densities of the expansion specimens. The measured density of the two Textoglass specimens ($1.81\ g \cdot cm^{-3}$) agreed to $\pm 0.2\%$, indicating good billet uniformity. The Burlglass 1200 specimen density was $1.3\ g \cdot cm^{-3}$, or about 30% less than that of the Textoglass specimens.

4. RESULTS

A reliability check of the dilatometer was made immediately before testing the composite specimens [9]. This test used a 3-in. copper specimen (Q82-1) as a secondary standard and the length–temperature data obtained are indicated in Table IV. The α values are within $\pm 2\%$ of the expected values. This indicated that the dilatometer system was functioning within the estimated uncertainty limits.

For the expansion tests on the composite spcimens, the dilatometer was held at the temperatures indicated in Tables V, VI, and VII for at least 24 h and until steady state was obtained as indicated by stable length and temperature data. The test sequence typically included data near room temperature, 45, 70, and 45°C (a checkpoint), one or two points above 75°C (95 and 120°C), and room temperature. The behavior observed for each specimen is described below.

Table V. Thermal Expansion Data for Textoglass Perpendicular

Temperature (°C)	Length (μ in.)	$\frac{L(T)-L(T_0)}{L_0}$ (%)	$10^6\alpha$ ($°C^{-1}$)	$\frac{T_1+T_2}{2}$ (°C)
22.08	26,798	—		
			59.78	34.16
46.23	31,219	0.144		
			64.17	58.54
70.84	36,055	0.302		
			66.17	58.49
46.14	31,049	0.139		
			65.21	70.90
95.65	40,936	0.462		
			125.36	107.97
120.29	50,391	0.770		
			78.96	70.97
21.66	26,544	—		

Table VI. Thermal Expansion Data for Textoglass Parallel

Temperature (°C)	Micrometer length (μ in.)	$\frac{L(T)-L(T_0)}{L_0}$ (%)	$10^6\alpha$ ($°C^{-1}$)	$\frac{T_1+T_2}{2}$ (°C)
21.86	99,956	—		
45.76	101,121	0.038	15.91	33.81
70.89	102,440	0.081	17.14	58.33
46.02	101,094	0.037	17.67	58.45
92.84	103,615	0.119	17.58	69.43
104.93	104,306	0.142	18.65	98.88
117.93	104,969	0.164	16.66	111.43
21.49	99,932	—	17.06	69.71

4.1. Textoglass Perpendicular

Figure 2 shows that the percentage expansion increased uniformly to 0.45% at 95°C and then increased to about 0.8% at 120°C. This suggests that a change in expansion behavior occurs above 95°C and this is shown by the α values in Fig. 3. The four α values are described to about $\pm 3\%$ by

$$10^6\alpha = 54.8 + 0.163\ T\,(°C) \qquad (20°C < T < 70°C) \tag{2}$$

The length at room temperature after the test showed no change.

4.2. Textoglass Parallel

Figure 2 shows that the percentage expansion was about 0.16% at 120°C, which is about 20% of that found for the Textoglass perpendicular

Table VII. Thermal Expansion Data for Burlglass 1200 Perpendicular

Temperature (°C)	Length (μ in.)	$\frac{L(T)-L(T_0)}{L_0}$ (%)	$10^6\alpha$ ($°C^{-1}$)	$\frac{T_1+T_2}{2}$ (°C)
21.85	98,571	—		
45.93	104,164	0.183	75.91	33.89
71.56	110,284	0.383	78.06	58.74
45.92	103,317	0.155	88.83	58.74
104.60	129,004	0.995	143.08	75.26
21.58	104,160	—	97.82	63.09

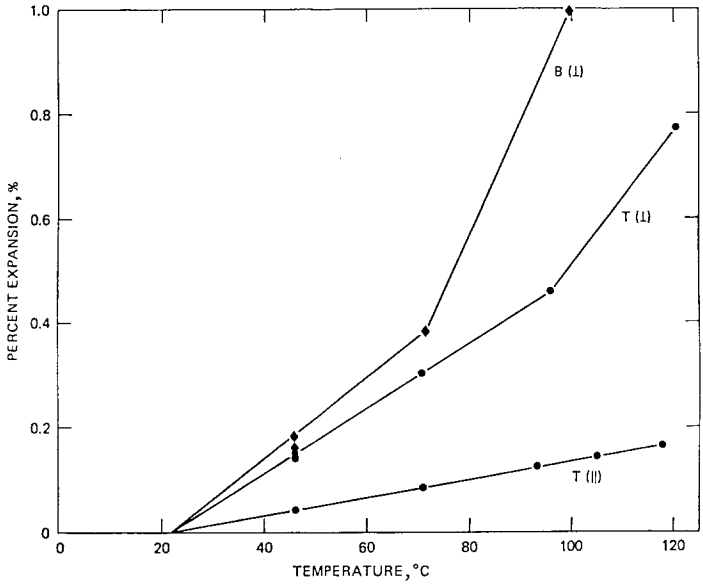

Fig. 2. Percentage thermal expansion as a function of temperature for three epoxy–fiberglass composite specimens in the perpendicular and parallel directions. T(⊥) Textoglass perpendicular, T(||) Textoglass parallel, and B(⊥) Burglass 1200 perpendicular.

specimen. This is a clear indication of the restraining effect of the fibers aligned along the cylinder axis. The α values shown in Fig. 3 are described to about $\pm 3\%$ by

$$10^6\alpha = 14.80 + 0.039\, T\,(°\mathrm{C}) \qquad (20°\mathrm{C} < T < 100°\mathrm{C}) \tag{3}$$

The α values for the Textoglass parallel specimen are about one-fourth of those given by Eq. (2) for the Textoglass perpendicular specimen. Good agreement was found for the length check points.

4.3. Burglass 1200 Perpendicular

Figure 2 shows that this specimen exhibited the largest percentage expansion, nearly 1% at 100°C, and the checkpoint lengths showed significant disagreements. The first three $L:T$ data suggest that up to 60°C the α value is about $77 \times 10^{-6}\ °\mathrm{C}^{-1}$. This is about 15% greater than that of the Textoglass perpendicular specimen.

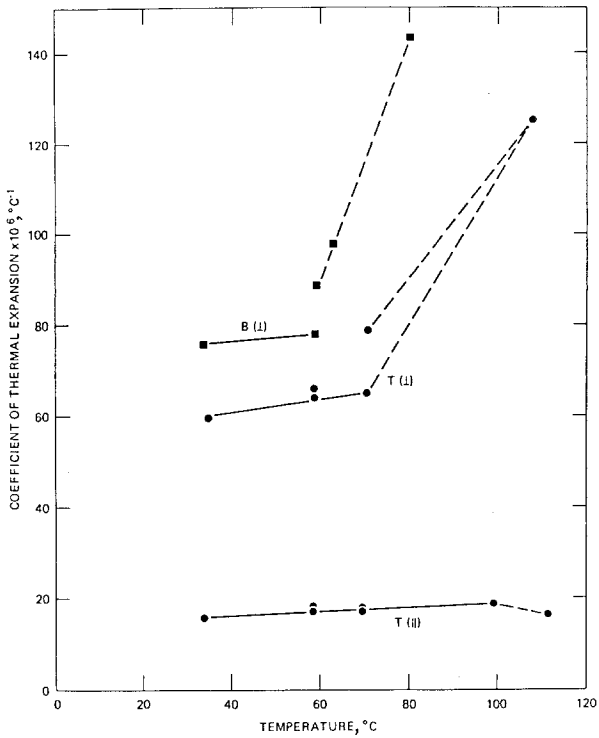

Fig. 3. Coefficient of thermal expansion as a function of temperature for three epoxy–fiberglass composite specimens in the perpendicular and parallel directions. T(⊥) Textoglass perpendicular, T(||) Textoglass parallel, and B(⊥) Burglass 1200 perpendicular.

5. DISCUSSION

As noted in Table III the Burlglass 1200 specimen density ($1.3\ g \cdot cm^{-3}$) is less than that of the Textoglass specimens ($1.8\ g \cdot cm^{-3}$). The lower-density specimen exhibits a greater expansion. This observation is consistent with that of Hamelin [5], who reported thermal contraction values from 300 to 77 K that are anisotropic (perpendicular exceeds parallel) and increase with decreasing density. At $1.8\ g \cdot cm^{-3}$, he observed $\Delta L/L$ of 7×10^{-3} and 2.5×10^{-3}, which correspond to average α values of 31×10^{-6} and $11 \times 10^{-6}\ °C^{-1}$, for perpendicular and parallel, respectively. The lower α values would be expected at the lower average temperature of 188 K (−85°C).

The lower α values for the parallel specimen are consistent with the restraining action of the fiberglass in the parallel direction. This restraint does not occur in the perpendicular direction and leads to high α values, near that of epoxy without fiberglass. High stresses due to differential thermal expansion between the epoxy and the fiberglass probably exist in each of these specimens and thermal cycling could lead to anomalous growth and cracks.

Finally, it is noteworthy that as the temperature is increased, major length changes occur for the low-density Burlglass 1200 perpendicular specimen first, Textoglass perpendicular second, and Textoglass parallel last.

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NOMENCLATURE

α	Thermal expansion coefficient, $°C^{-1}$
ΔL	$L(T_2) - L(T_1)$, cm
ΔT	$T_2 - T_1$, °C
L_0	Length at room temperature, cm
$L(T_i)$	Length at temperature T_i, cm
T_i	Temperature, °C
T_0	Room temperature, °C

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