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Thermal Expansion of an E-Glass/Vinyl Ester Composite from 4 to 293 K

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We have measured the thermal expansion of the three principal orthogonal directions of an E-glass/vinyl ester structural composite from liquid helium temperature, 4.2 K, to room temperature, 293 K. The linear thermal expansion at 4.2 K ranged from -0.23 to -0.71 %, referenced to zero expansion at 293 K. We fitted the linear thermal expansion data from 4.2 to 293 K with a cubic polynomial for each of the three principal orthogonal directions.

KEY WORDS: composite; thermal expansion; vinyl ester.

1. INTRODUCTION

The linear thermal expansion, $\Delta l/l_0$, of a glass–polymer composite from liquid helium temperature (4.2 K) to room temperature (293 K) was measured. The composite has E-glass fibers in a vinyl ester matrix. The composite is a candidate material for the coil pack of the Superconducting Magnetic Energy Storage (SMES) system. Because composites generally exhibit anisotropic linear thermal expansion, thermal expansion data for specimens oriented in the three principal orthogonal directions are required. These orthogonal directions are designated longitudinal (parallel to the primary fibers), short-transverse (perpendicular to the primary fiber planes), and longitudinal-transverse (perpendicular to the primary fibers but in the fiber plane).

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2. APPARATUS

A modified commercial quartz-tube dilatometer was used for the measurements. The measurement method is consistent with ASTM E228-85 [1]. Figure 1 is a schematic drawing of the apparatus. The upper end of the specimen supports a fused-quartz measurement tube that is hemispherical on the contacting end. The lower end of the specimen rests on a brass support plate. A spring holds the plate rigidly against the bottom of an outer fused-quartz support tube. The end of the quartz support tube is ground flat and perpendicular to the specimen axis. The average coefficient of thermal expansion of fused quartz is $5.5 \times 10^{-7}\,\mathrm{K}^{-1}$ [2], which is negligibly small compared to that of the composite material tested here. The top end of the support tube is anchored to a rigid frame that supports a linear variable differential transformer (LVDT). The top of the measurement tube has a flat fused-quartz end cap perpendicular to the specimen axis, which ensures consistent contact with the LVDT. The output of this LVDT was calibrated with a micrometer capable of resolving $0.6\,\mu$m,

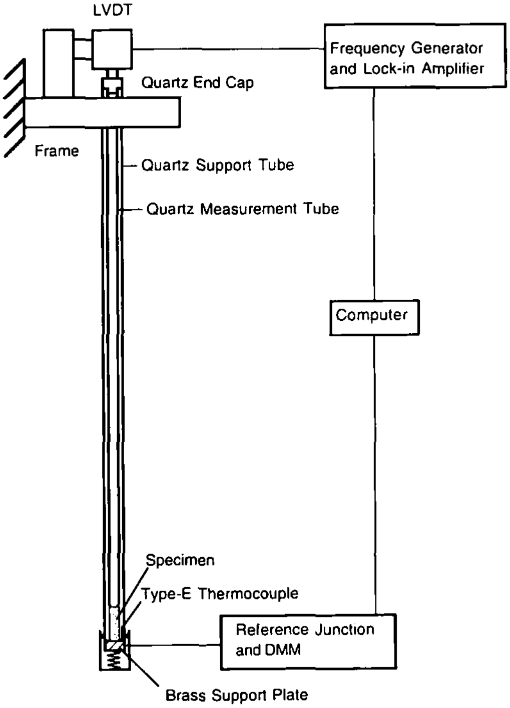

Fig. 1. Schematic drawing of the thermal expansion apparatus.

which results in a sensitivity of 6.35 μm · mV^{-1}. The specimen expansion is measured by recording the output of a lock-in amplifier that senses an excitation signal (2500 Hz) sent through the LVDT. The temperature is monitored by a type E thermocouple in close proximity to the specimen and thermally anchored to the brass support plate. The thermocouple therefore experiences the same environment as the specimen. The quartz tube assembly is lowered into a dewar of liquid helium to cool the specimen to 4.2 K. After complete cooling is achieved, the assembly is slowly raised out of the dewar; we controlled this process to produce specimen heating at a rate of approximately 1 K · min^{-1}. Data were taken as the temperature rose from 4 K to room temperature. The thermocouple used in the system was calibrated to a 0.5 K accuracy. Calibration of the LVDT showed a linearity of better that 1%. The total systematic uncertainty in the linear thermal expansion measurements is estimated as 4% at 4.2 K.

A specimen of oxygen-free, high-purity copper was used to verify the proper operation of the apparatus. Figure 2 shows experimental data as circles and corresponding handbook data for high-purity copper as triangles [3]. The data agree well, demonstrating the validity of the method and apparatus. Figure 3 shows a deviation plot of the experimental data versus the literature data. Since linear thermal expansion is a quantity that has, by convention, a zero-expansion reference point at 293 K, relative deviation cannot be calculated due to the singularity at 293 K. We therefore normalized the deviation between experimental and literature values by dividing the absolute difference between these values by the literature value for linear thermal expansion at 4.2 K. Figure 3 shows that the

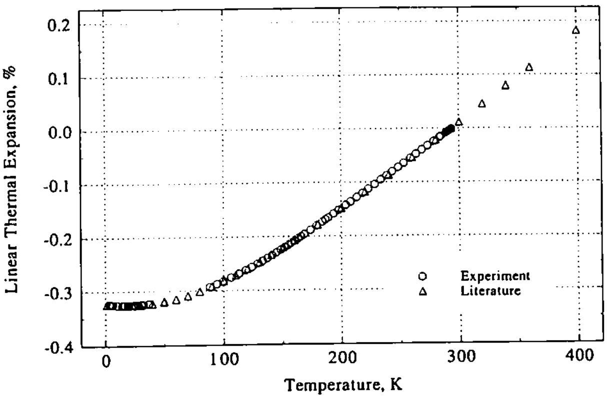

Fig. 2. Thermal expansion data for high-purity copper used to verify the apparatus.

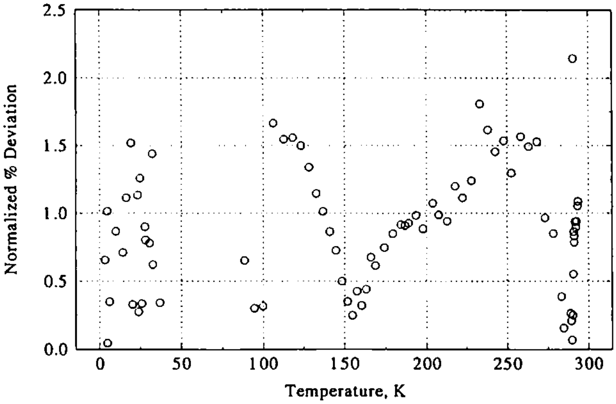

Fig. 3. Normalized deviation plot of high-purity copper thermal expansion data compared to literature values.

maximum normalized deviation is less than 2.4%, which is smaller than our estimated uncertainty.

3. RESULTS

Three specimens, designated A, B, and C, from each of the three principal orthogonal directions, were tested. The material from which the specimens were cut was a single large plank of E-glass/vinyl ester composite with a fiber volume fraction of 57%. The vinyl ester was 10% clay-filled. A pultrusion process was used in manufacturing the plank. The fibers were laid in planes, with 40% at 0°, 40% at ±45°, and 20% at 90° to the long axis of the plank (the direction of the primary fibers).

Figure 4 shows data from tests of three specimens cut in the longitudinal direction. In this, and in all of the following figures, data are normalized to zero expansion at 293 K. At liquid helium temperature, the expansion of these three specimens averaged −0.23%. Hartwig and Knaak [4] measured a unidirectional E-glass/epoxy composite containing 70% volume fraction fiber. They report a value of −0.16% expansion at 4.2 K in the longitudinal direction. Due to the high fiber content and the unidirectional weave, the −0.16% expansion would be very close to that of the E-glass fiber itself. This corresponds well with the −0.23% average expansion observed here because our material has a lower fiber-volume fraction, a fiber weave that results in fewer fibers oriented in the direction of expansion, and a vinyl ester resin that expands at a higher rate than most epoxies [5].

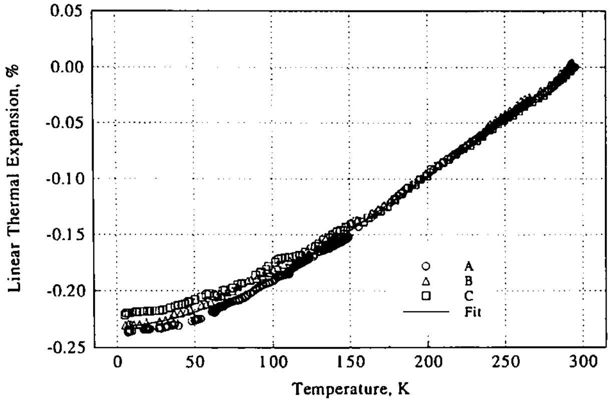

Fig. 4. Thermal expansion data and fitted curve for longitudinal specimens A, B, and C.

The result of fitting a cubic polynomial to all three sets of longitudinal data is shown in Fig. 4 and the coefficients are given in Table I. The fitted curve is shown in Fig. 4. The longitudinal data are consistent in form but have a ±4% scatter at 4.2 K. This difference is probably due to differences in the fiber weave or makeup from one specimen to the next.

Figure 5 shows data for three specimens cut in the short-transverse direction. Since all fibers in the weave are perpendicular to the short-transverse direction, the expansion of these three specimens should reflect primarily the expansion of the vinyl ester matrix. The average measured value for the thermal expansion of these three specimens at 4.2 K is −0.71%. For comparison, a literature value for the thermal expansion at 4 K of an unfilled vinyl ester was −1.30% [5]. The vinyl ester in the

Table I. Linear Thermal Expansion Results and Fitted Equations[a]

	Expansion at 4.2 K (%)	*a*	*b*	*c*	*d*
Longitudinal	−0.23	−0.232	6.31E−5	4.38E−6	−6.55E−9
Longitudinal-transverse	−0.28	−0.282	−1.40E−4	6.20E−6	−8.15E−9
Short-transverse	−0.71	−0.712	4.87E−4	1.10E−5	−1.50E−8

[a] Form of fitted equation: linear thermal expansion $= a + bT + cT^2 + dT^3$, with T in K and reference length at 293 K.

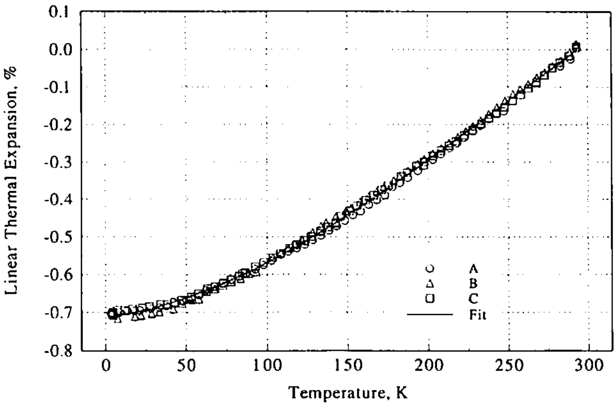

Fig. 5. Thermal expansion data and fitted curve for short-transverse specimens A, B, and C.

material we tested was 10% clay-filled, with a 57% volume fraction of E-glass fibers that also act somewhat like a filler. A first-order approximation that treats thermal expansion of the filler as negligible by comparison to the matrix can be used to estimate the thermal expansion of a filled system [6]:

$$\text{Expansion}_{\text{filled}}/\text{Expansion}_{\text{unfilled}} = 1 - V_{\text{f}}$$

where V_{f} is the volume fraction of filler. According to this simplistic relation, the 10% clay-filled vinyl ester without any fibers should result in a thermal expansion of −1.17% at 4 K. Our experimental value of −0.71% corresponds to the fibers acting like a 35% filler in addition to the 10% clay fill. This is reasonable, because fibers may not act as effectively as a conventional fill material and the assumption of the filler having effectively zero thermal expansion may not be valid, particularly at this high value (57%) of volume fraction.

The short-transverse data are in excellent agreement with each other (i.e., within the systematic uncertainty at 4.2 K), which is expected because these specimens are essentially like filled vinyl esters. Inhomogeneities in the fiber weave would not be as apparent in these specimens. A cubic equation for thermal expansion as a function of temperature using all of the short-transverse specimen data is shown in Table I. The curve for the fitted equation is also shown in Fig. 5.

Specimens tested in the longitudinal-transverse direction have an average linear thermal expansion at 4.2 K of −0.28%. Data for the three tests are shown in Fig. 6. This direction is similar to the longitudinal direction. The

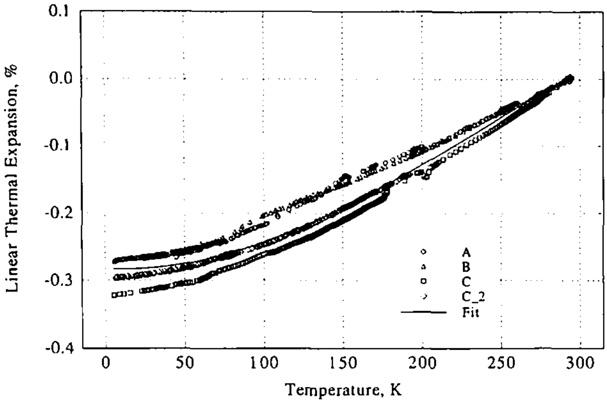

Fig. 6. Thermal expansion data and fitted curve for longitudinal-transverse specimens A, B, and C.

fiber weave results in 40% of the fibers being at 45° to both the longitudinal and the longitudinal transverse directions. However, the longitudinal-transverse direction has only half as many (20%) fibers in parallel as the longitudinal direction. Therefore, it is not surprising that the thermal expansion along the longitudinal-transverse direction is slightly higher in magnitude than it is along the longitudinal direction.

The result of fitting all of the data for longitudinal-transverse specimens to a cubic polynomial is shown in Fig. 6 and the coefficients are given in Table I. The tests on specimens A and B yielded consistent data. Specimen C had a thermal expansion much lower than that of specimens A and B. We tested specimen C again to verify the 15% difference in thermal expansion at 4.2 K. The retest data gave a difference of over 10% between specimen C and specimens A and B. This is most likely due to a variation in fiber layup or fiber makeup from one specimen to another.

4. CONCLUSIONS

The linear thermal expansion of an E-glass/vinyl ester composite at 4.2 K in the short-transverse direction is 2.5 to 3 times greater than that for the longitudinal direction. This is because the fiber weave dominates the thermal expansion in the longitudinal direction, whereas the vinyl ester matrix dominates in the short-transverse direction.

The data for the longitudinal-transverse direction demonstrate that this material is not entirely homogeneous, which is typical of most composites. Contributors to inhomogeneity can be differences in the fiber weave, slight changes in layup or makeup, variations in fiber fraction from specimen to specimen, or fiber inhomogeneities. The sizes of the specimens used for thermal expansion testing can also contribute to data inconsistency. For a woven composite, larger specimens will tend to yield more consistent test results, as inhomogeneities may be averaged out.

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