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Thermal Expansion of Epoxy-Fiberglass Composite Specimens*

by

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Abstract

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 resin 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 both a pulsed and steady state mode, and expansion data were needed to assess the induced loads at the coil joints 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 ±2% of expected values from 20 to 200°C.

The percent 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 $55 \times 10^{-6}\,°C^{-1}$. 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 $15 \times 10^{-6}\,°C^{-1}$. The lower α-values in the parallel direction are

consistent with the restraining action of the fiberglass layers. The α-values decreased with specimen density and this is consistent with literature data on composite contraction from 20° to -195°C.

Introduction

The copper conductors that form the helical field coil in the Advanced Toroidial Facility are mechanically-separated by a fiberglass reinforced epoxy composite to take the compressive loads of the conductors which are induced by thermal and magnetic forces during operation. The Fusion Energy Division, Oak Ridge National Laboratory, conducts plasma physics experiments in this facility (1). The coil is operated in both a pulsed and steady-state mode. The differential expansion of the copper-epoxy system resulting from the expected temperature excursion due to electrical heating could stress joints to failure. 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 the epoxy was a dominant factor in CR-grade glass-epoxy laminates with densities of 1.90 and 1.95 g/cm^3. Total contractions between room temperature and 4 K were 0.6% for the laminate with a density of 1.95 g/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 $\Delta L/L_o$ values for OFHC copper and 304SS are 0.31% and 0.26%, respectively.

J. Hamelin (5) measured contraction from 300 to 77 K of glass-cloth-reinforced epoxy laminates parallel and perpendicular to the glass-cloth layers. The observed $\Delta L/L_o$ values decreased linearly with density and were significantly greater in the perpendicular direction. The epoxy filling fraction is responsible for these results and he found an increased curing pressure reduced $\Delta L/L_o$ by a factor of 3. These results provided a means to keep laminates under compression in external casings.

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, 7, 8). Figure 1 is a cross section of the apparatus. The specimen is placed in a cylindrical copper holder to provide 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:

$$\alpha = \frac{1}{L_o} \cdot \frac{\Delta L}{\Delta T} = \frac{1}{L_o} \cdot \frac{L(T_2) - L(T_1)}{T_2 - T_1} \qquad (1)$$

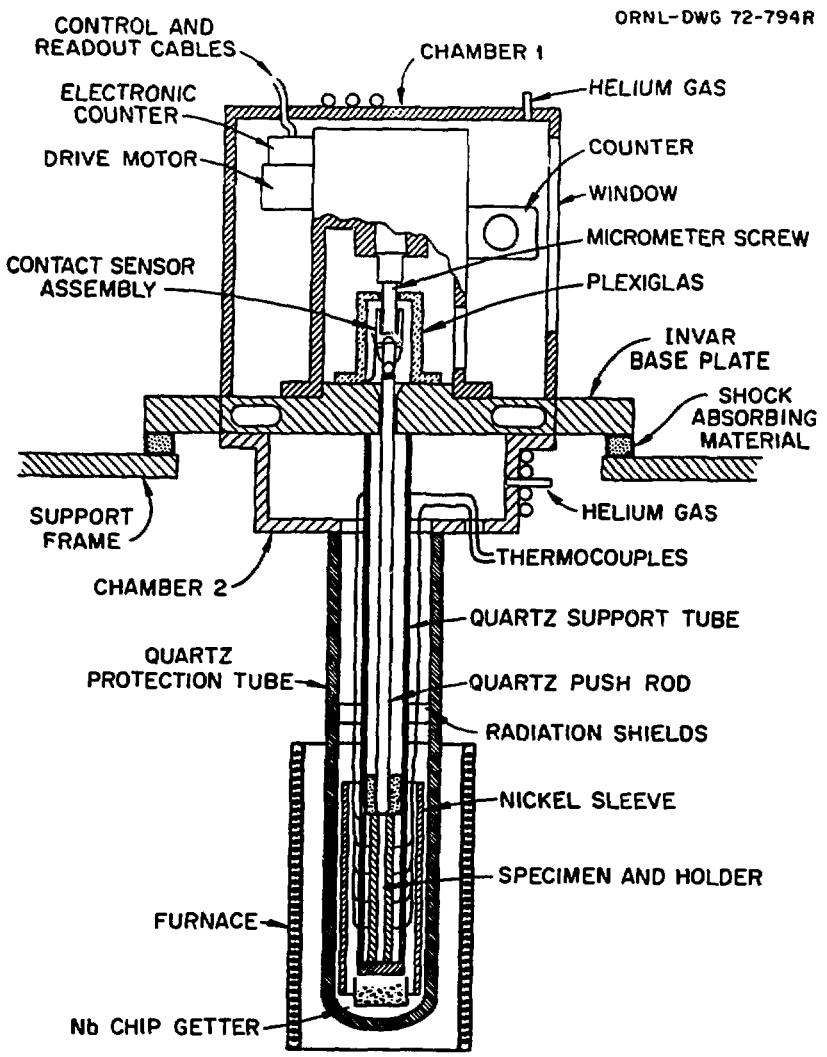

Figure 1. Cross Section of the Fused Quartz Differential Dilatometer Showing the Major Components of the Apparatus

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 ±0.8% ($\pm 0.1 \times 10^{-6}\ K^{-1}$) for copper using a 100 K temperature change and ±1.4% ($\pm 0.2 \times 10^{-6}\ K^{-1}$) for a 50 K temperature change. The uncertainty for tungsten was approximately triple these values because of its lower α-value ($Cu \sim 18 \times 10^{-6}\ K^{-1}$, $W \sim 5 \times 10^{-6}\ K^{-1}$). Consequently we have estimated uncertainties in $\Delta L/L_0$ and α at less than ±1 and ±2%, respectively, for a temperature change of 25 K.

Materials

Two billets were produced by vacuum impregnating layers of fiberglass cloth with an epoxy resin. Table Ia shows the epoxy resin included four components: resin, hardener, flexibilizer, and promoter. Table Ib shows the epoxy impregnation and cure cycle. The Textoglass billet included layers of 7 mil thick J. P. Stevens (9) "Textoglass" tape and the Burlglass 1200 billet was made using a randomly woven batt of fiberglass manufactured by Burlington Industries (10). Right-circular cylinders were machined from these billets to allow expansion tests perpendicular and parallel to the Textoglass tape layers and random direction for the Burlglass 1200. Table Ic shows the dimensions and densities of the expansion specimens. The measured density of the two Textoglass specimens (1.81 g/cm^3) agreed to ±0.2% indicating good billet uniformity. The Burlglass 1200 specimen density was 1.3 g/cm^3 or about 30% less than that of the Textoglass specimens.

Table Ia. Epoxy Resin Formulation

Resin:	100 parts by weight DOW #DER-332 Epoxy Resin
Hardener:	90 parts by weight Nadic Methylanhydride (NMA)
Flexibilizer:	10 parts by weight Union Carbide "Carbowax" #400
Promoter:	0.5 to 0.8 parts by weight Benzyldimethylamine (BDMA)

Table Ib. Epoxy Impregnation and Cure Cycle

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

Table Ic. Thermal Expansion Specimen Characteristics

Specimen	Dimensions (in.) Length (L_o)	Diameter	Weight (g)	Density (g/cm^3)
Textoglass Perpendicular	3.0623 (3.0622)*	0.3985	11.305	1.806
Textoglass Parallel	3.0624 (3.0622)*	0.3992	11.400	1.814
Burlglass 1200 Random	3.0595 (3.0578)*	0.3993	8.2723	1.318

*Length after test.

Results

A reliability check of the dilatometer was made immediately before testing the composite specimens (11). This test used a 3-in. copper specimen (Q82-1) as a secondary standard and the length-temperature data obtained are indicated in Table II. The α values are within ±2% of the expected values. This indicated the dilatometer system was functioning within the estimated uncertainty limits.

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

Temperature (°C)	Micrometer Length (microinch)	$\alpha \cdot 10^6$ (K^{-1})	$\frac{T_1 + T_2}{2}$ (°C)	Expected $\alpha \cdot 10^6$ (K^{-1})	Difference %
24.0	69778				
		17.4	62.9	17.1	+1.7
101.8	73841				
		17.7	151.6	17.9	-1.1
201.5	79138				
		17.8	111.5	17.6	+1.1
21.6	69509				

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

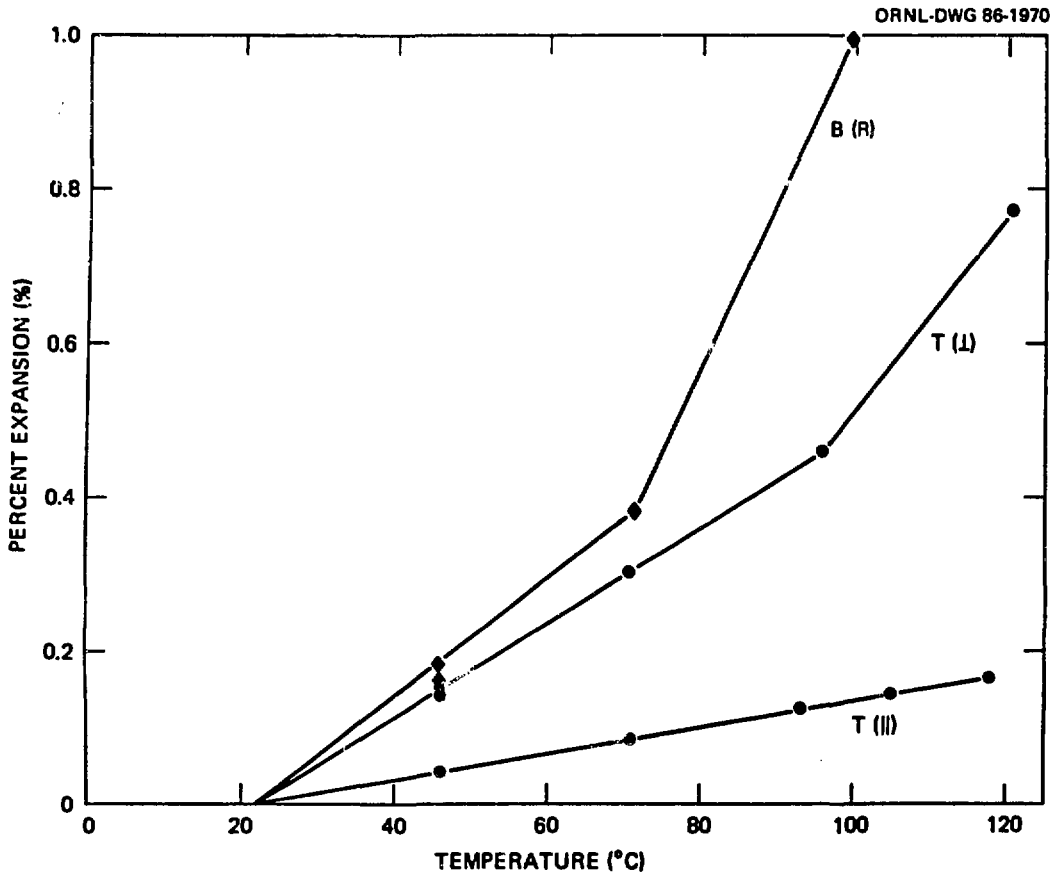

Figure 2. Percent Thermal Expansion as a Function of Temperature for Three Epoxy-Fiberglass Composite Specimens in the Perpendicular and Parallel Directions T(⊥) Textoglass Perpendicular, T(11) Textoglass Parallel, and B(R) Burglass 1200 Random.

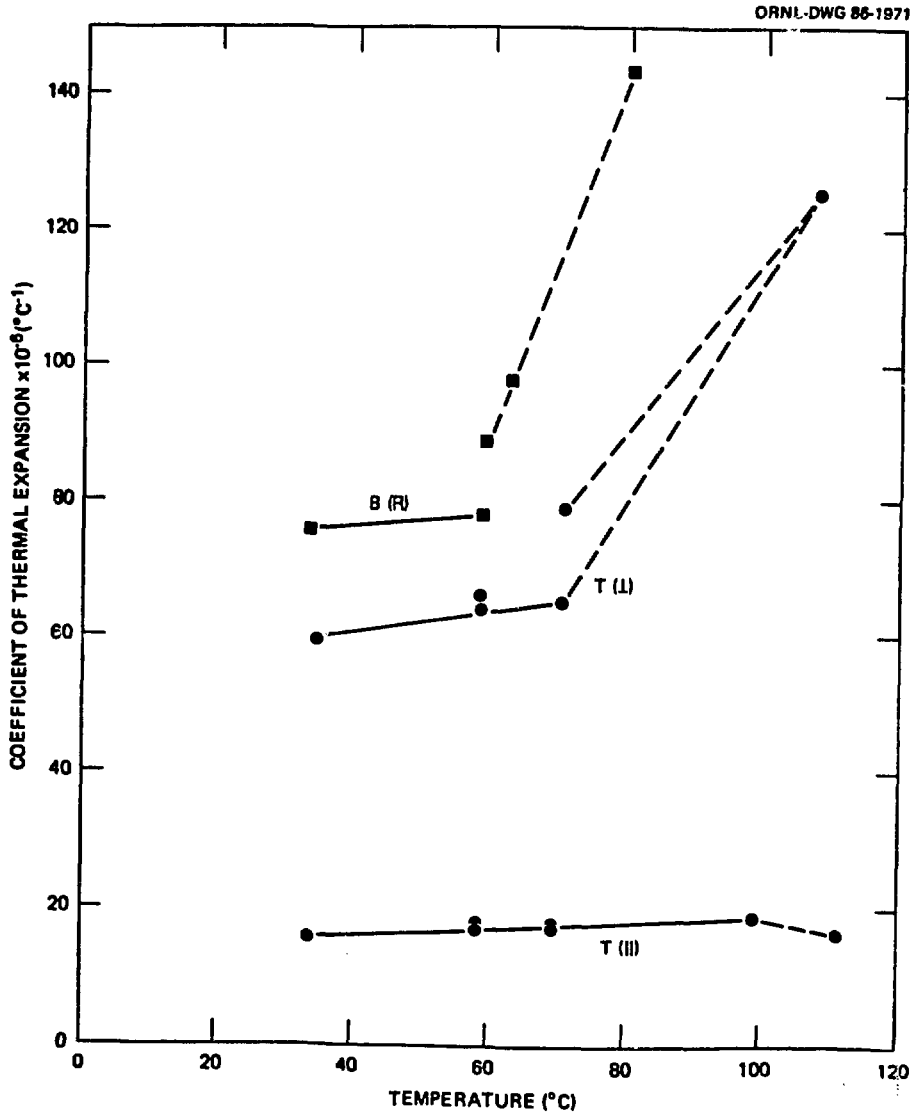

Figure 3. Coefficient of Thermal Expansion as a Function of Temperature for Three Epoxy-Fiberglass Composite Specimens in the perpendicular and Parallel Directions. T(⊥) Textoglass Perpendicular, T(11) Textoglass Parallel, and B(R) Burglass 1200 Random.

Textoglass Perpendicular — Figure 2 shows the percent expansion increased uniformly to 0.45% at 95°C and then increased to about 0.8% at 120°C. This suggests a change in expansion behavior occurs above 95°C and this is shown by the α-values in Figure 3. The four α-values are described to about ±3% by

$$\alpha \times 10^6 = 54.8 + 0.163\ T\ (°C) \quad (20 < T < 70°C). \qquad (2)$$

The length at room temperature after test showed no change.

Textoglass Parallel — Figure 2 shows the percent expansion was about 0.16% at 120°C, which is about 20% of that found for the Textoglass perpendicular specimen. This is a clear indication of the restraining effect of the fibers aligned along the cylinder axis. The α-values shown in Figure 3 are described to about ±3% by

$$\alpha \times 10^6 = 14.80 + 0.039\ T\ (°C) \quad (20°C < t < 100°C). \qquad (3)$$

The α-values for the Textoglass parallel specimen are about 1/4 of those given by Equation (2) for the Textoglass perpendicular specimen. Good agreement was found for the length check points.

Burlglass 1200 Random — Figure 2 shows this specimen exhibited the largest percent 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 x 10^{-6} $°C^{-1}$. This is about 15% greater than that of the Textoglass perpendicular specimen.

Table III. Thermal Expansion Data for Textoglass Perpendicular

Temperature (°C)	Length (microinch)	$\frac{L(T) - L(T_o)}{L_o}$ (%)	α 10^6 °C^{-1}	$\frac{T_1 + T_2}{2}$ (°C)
22.08	26798	---		
			59.78	34.16
46.23	31219	0.144		
			64.17	58.54
70.84	36055	0.302		
			66.17	58.49
46.14	31049	0.139		
			65.21	70.90
95.65	40936	0.462		
			125.36	107.97
120.29	50391	0.770		
			78.96	70.97
21.66	26544	---		

Table IV. Thermal Expansion Data for Textoglass Parallel

Temperature (°C)	Micrometer Length (microinch)	$\frac{L(T) - L(T_o)}{L_o}$ (%)	α (10^6 °C^{-1})	$\frac{T_1 + T_2}{2}$ (°C)
21.86	99956	---		
			15.91	33.81
45.76	101121	0.038		
			17.14	58.33
70.89	102440	0.081		
			17.67	58.45
46.02	101094	0.037		
			17.58	69.43
92.84	103615	0.119		
			18.65	98.88
104.93	104306	0.142		
			16.66	111.43
117.93	104969	0.164		
			17.06	69.71
21.49	99932	---		

Table V. Thermal Expansion Data for Burlglass 1200 Random

Temperature (°C)	Length (microinch)	$\frac{L(T) - L(T_o)}{L_o}$ (%)	$10^6\ \alpha\ °C^{-1}$	$\frac{T_1 + T_2}{2}$ (°C)
21.85	98571	---		
			75.91	33.89
45.93	104164	0.183		
			78.06	58.74
71.56	110284	0.383		
			88.83	58.74
45.92	103317	0.155		
			143.08	75.26
104.60	129004	0.995		
			97.82	63.09
21.58	104160	---		

Discussion

As noted in Table Ic the Burlglass 1200 specimen density (1.3 g/cm^3) is less than that of the Textoglass specimens (1.8 g/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/cm^3, he observed $\Delta L/L$ of 7×10^{-3} and 2.5×10^{-3}, which correspond to average α-values of $31 \times 10^{-6}\ °C^{-1}$ 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.

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

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Key Words

Thermal Expansion Coefficient

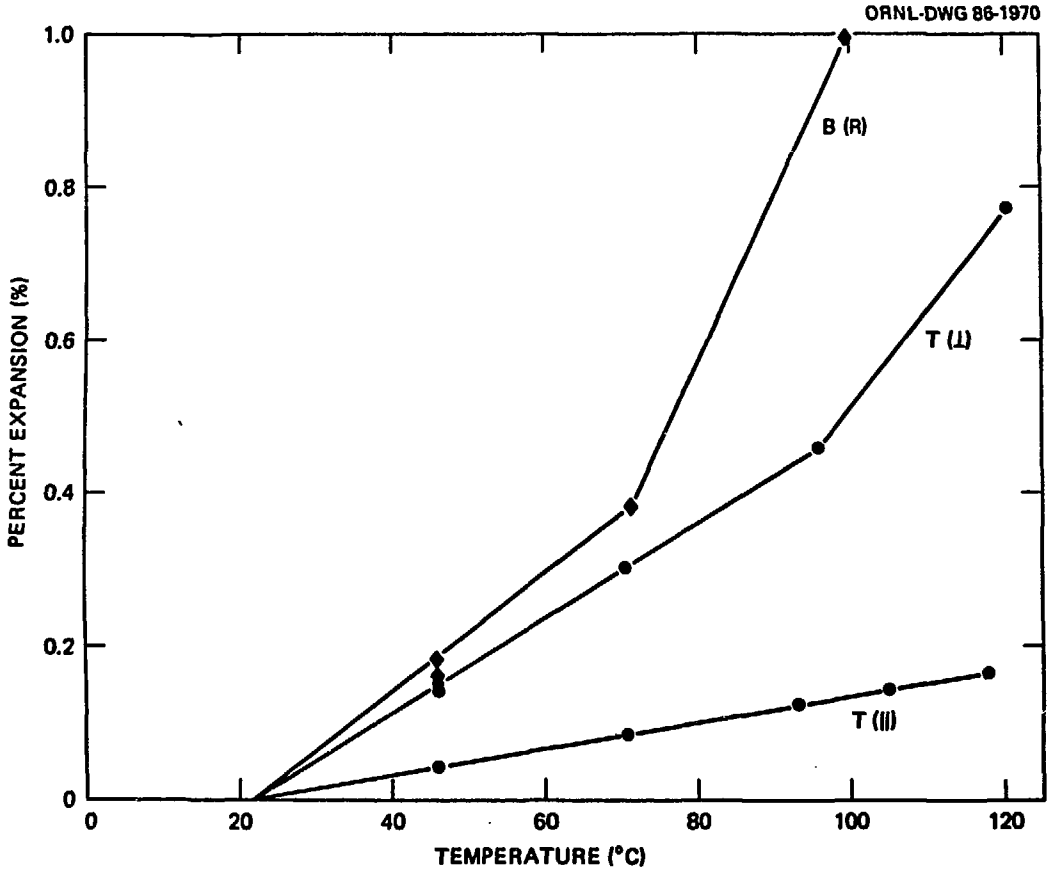
Epoxy-Fiberglass Composites

Anisotropic Expansion

Temperature Dependency

Nomenclature

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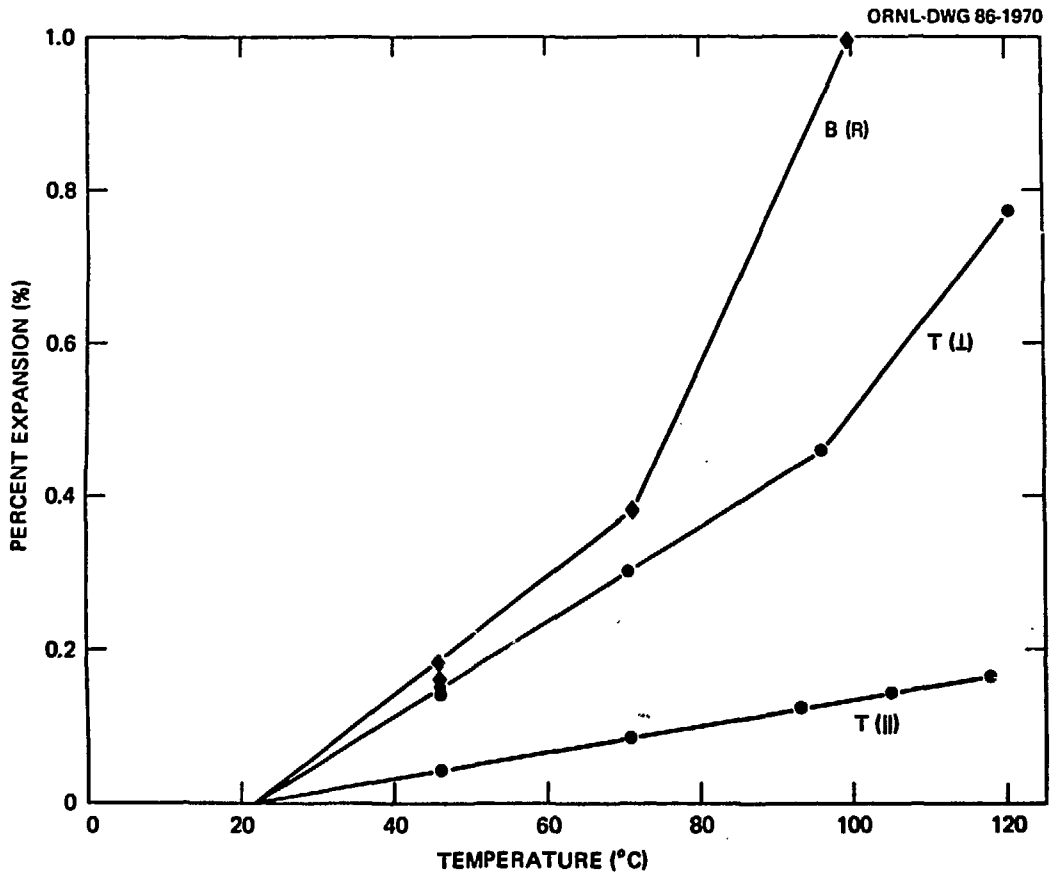
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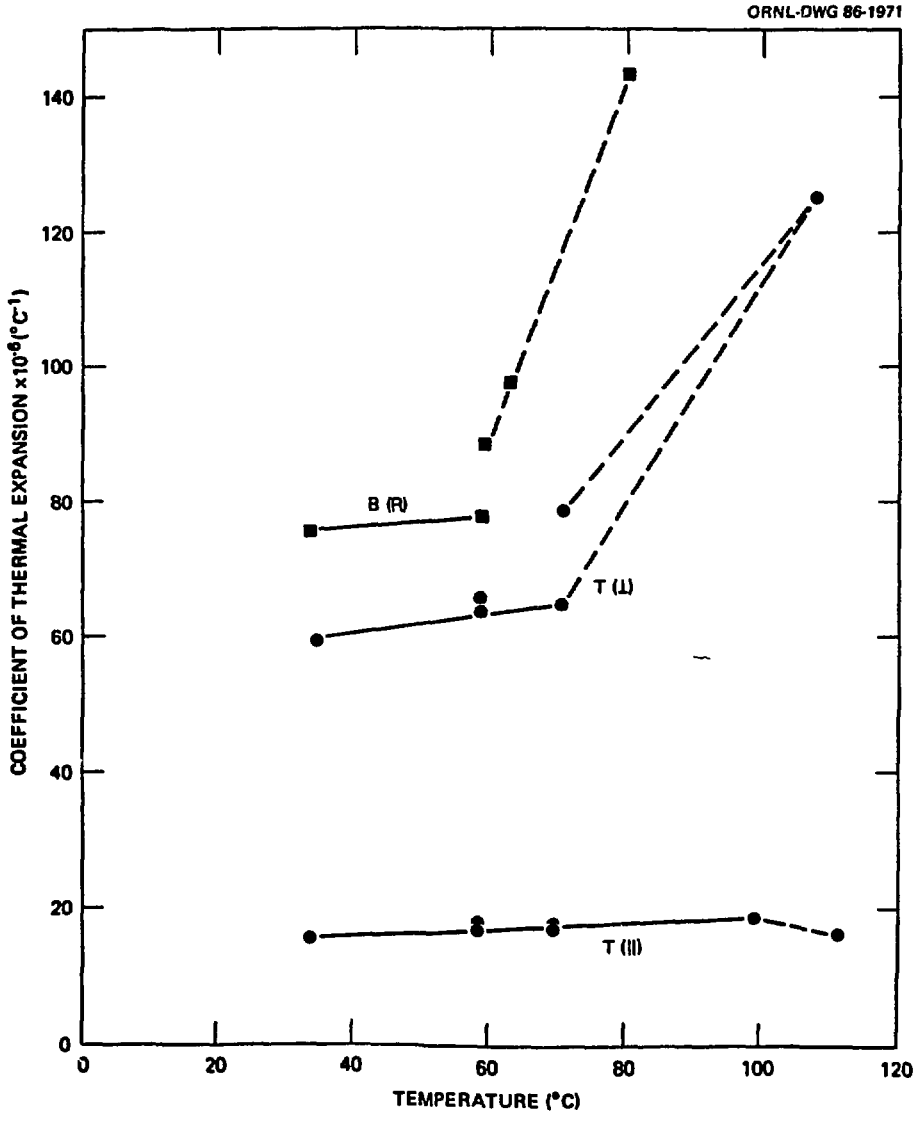
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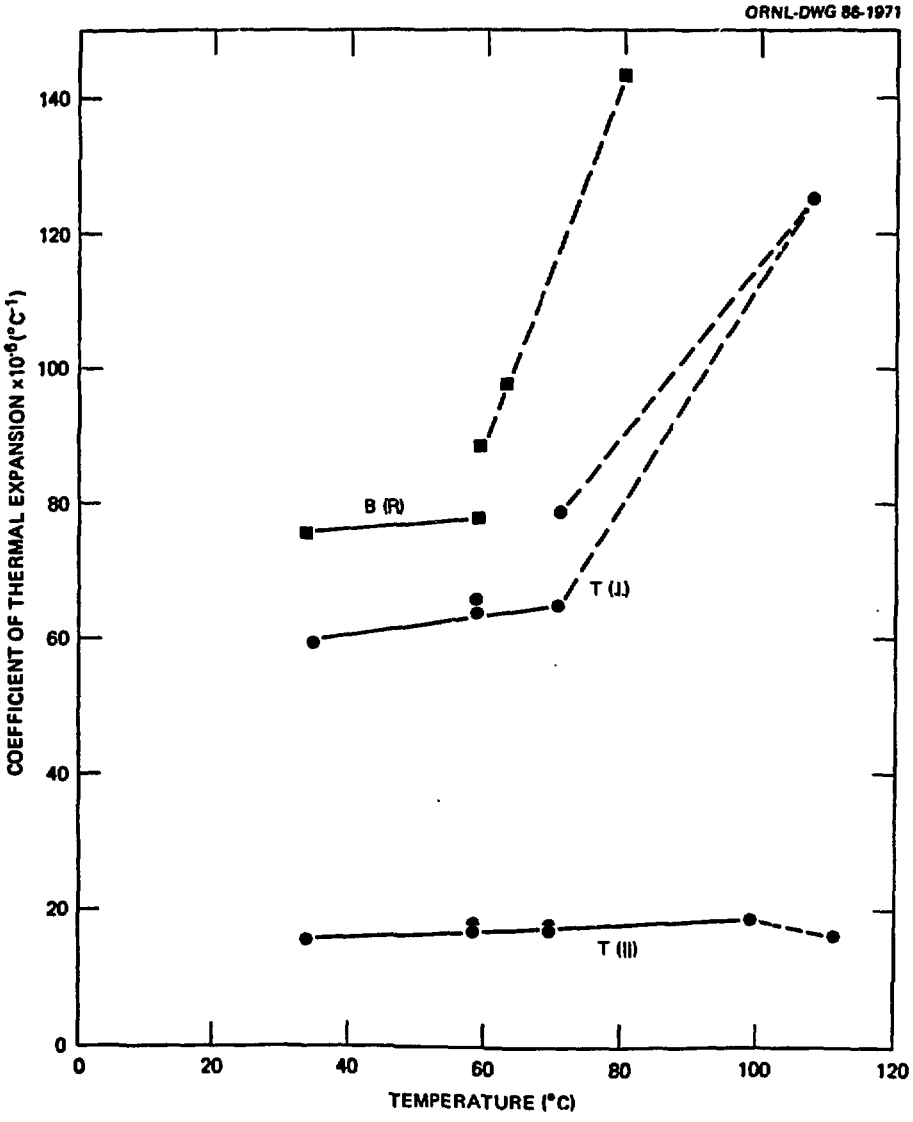
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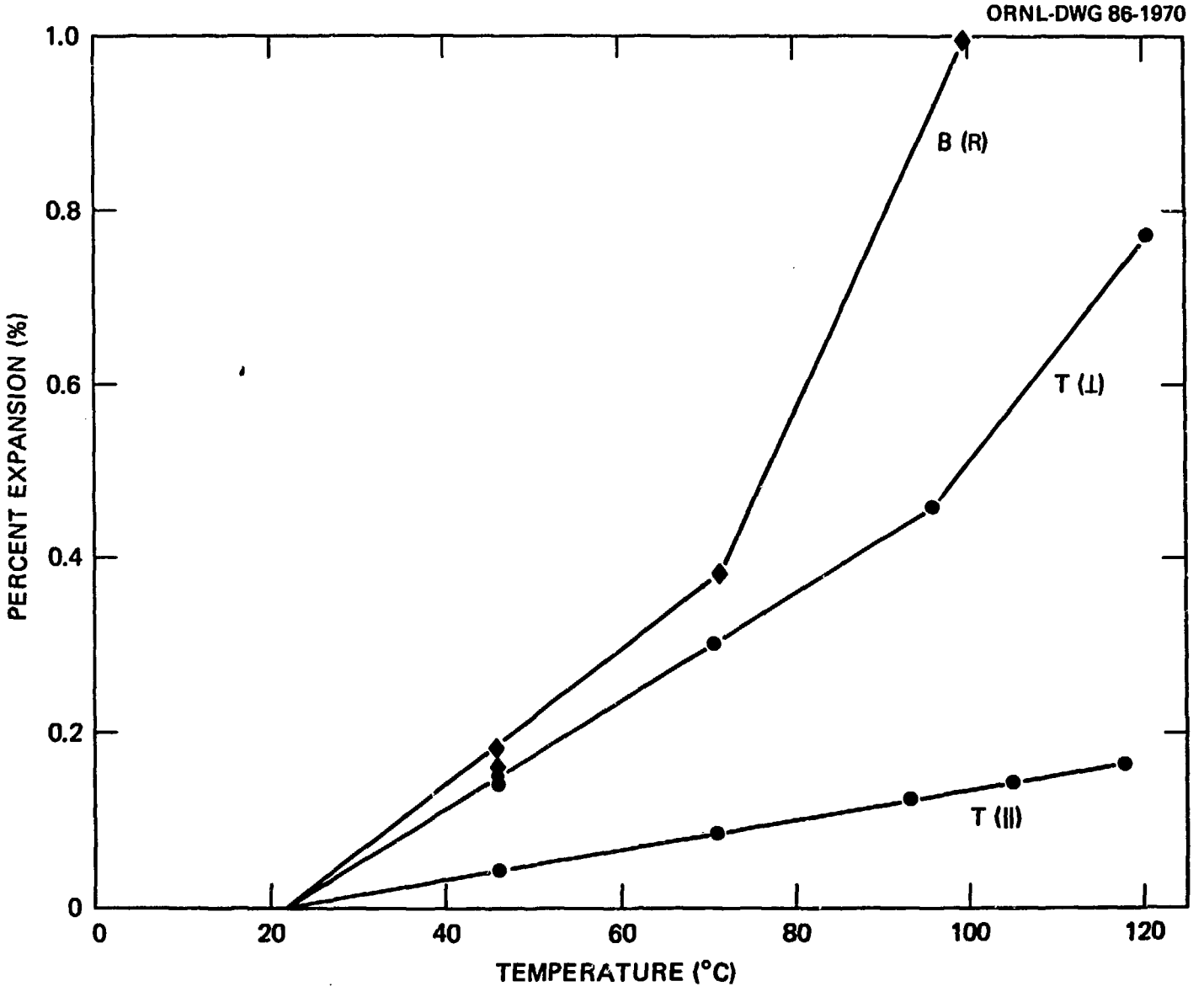
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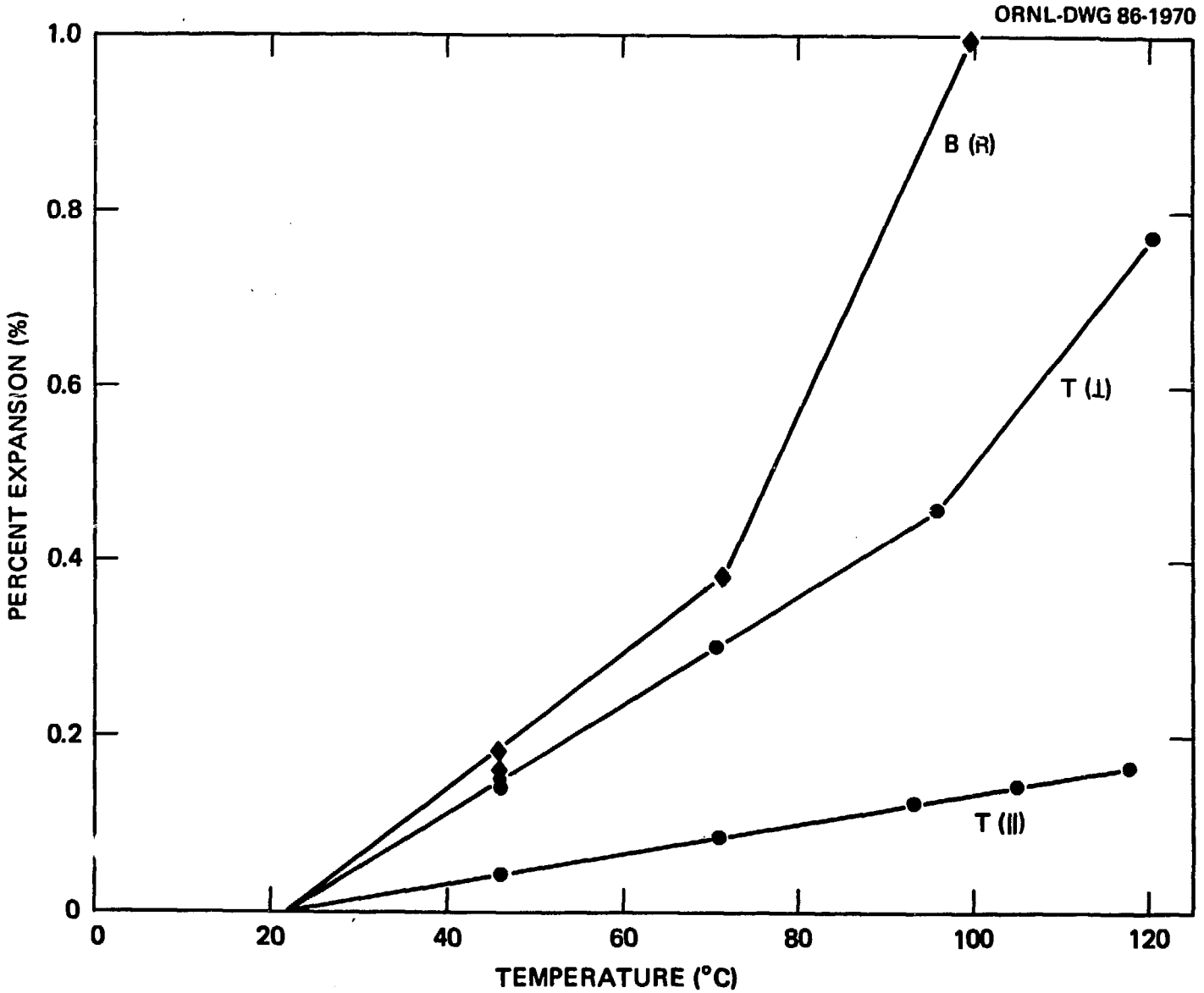
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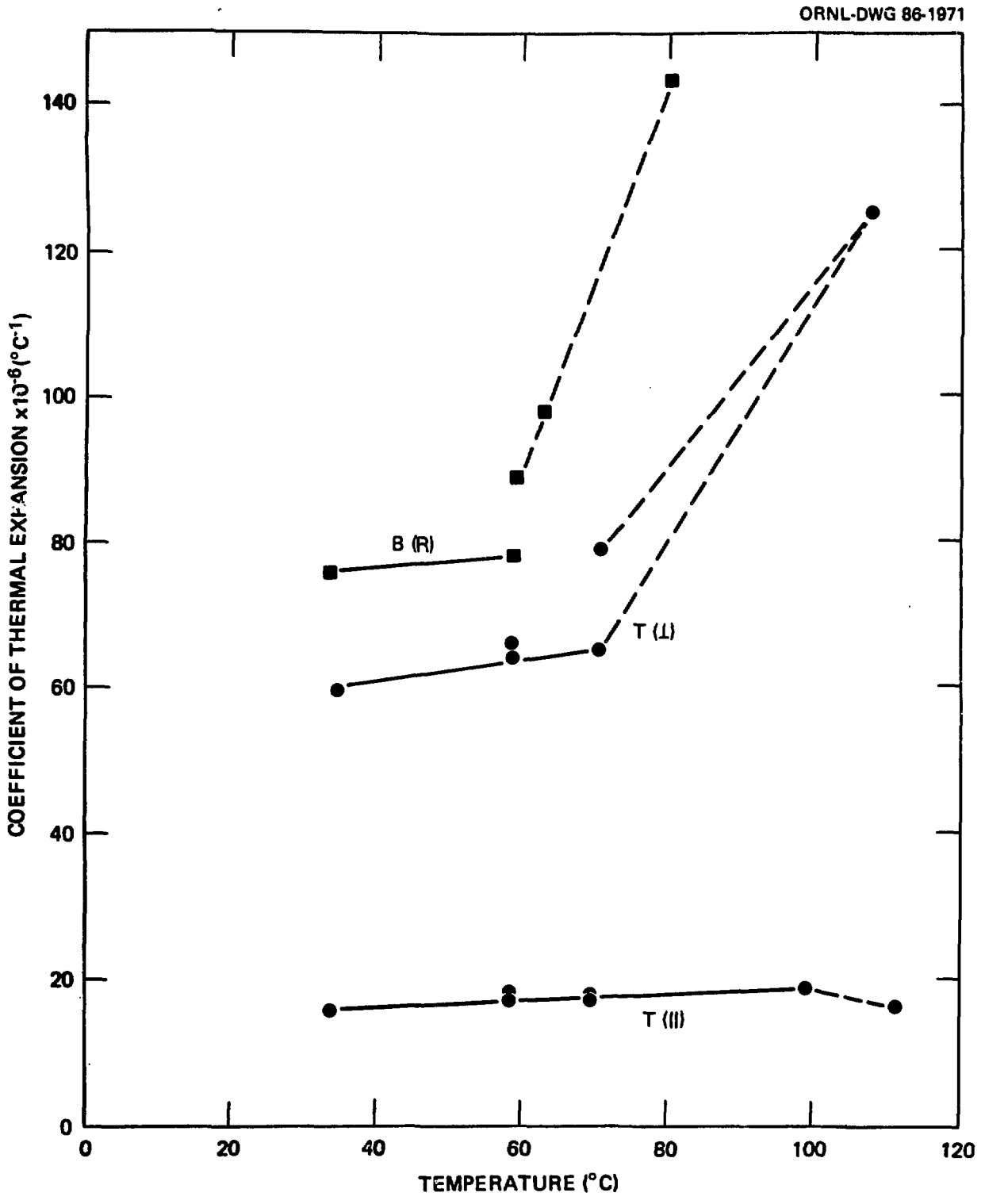
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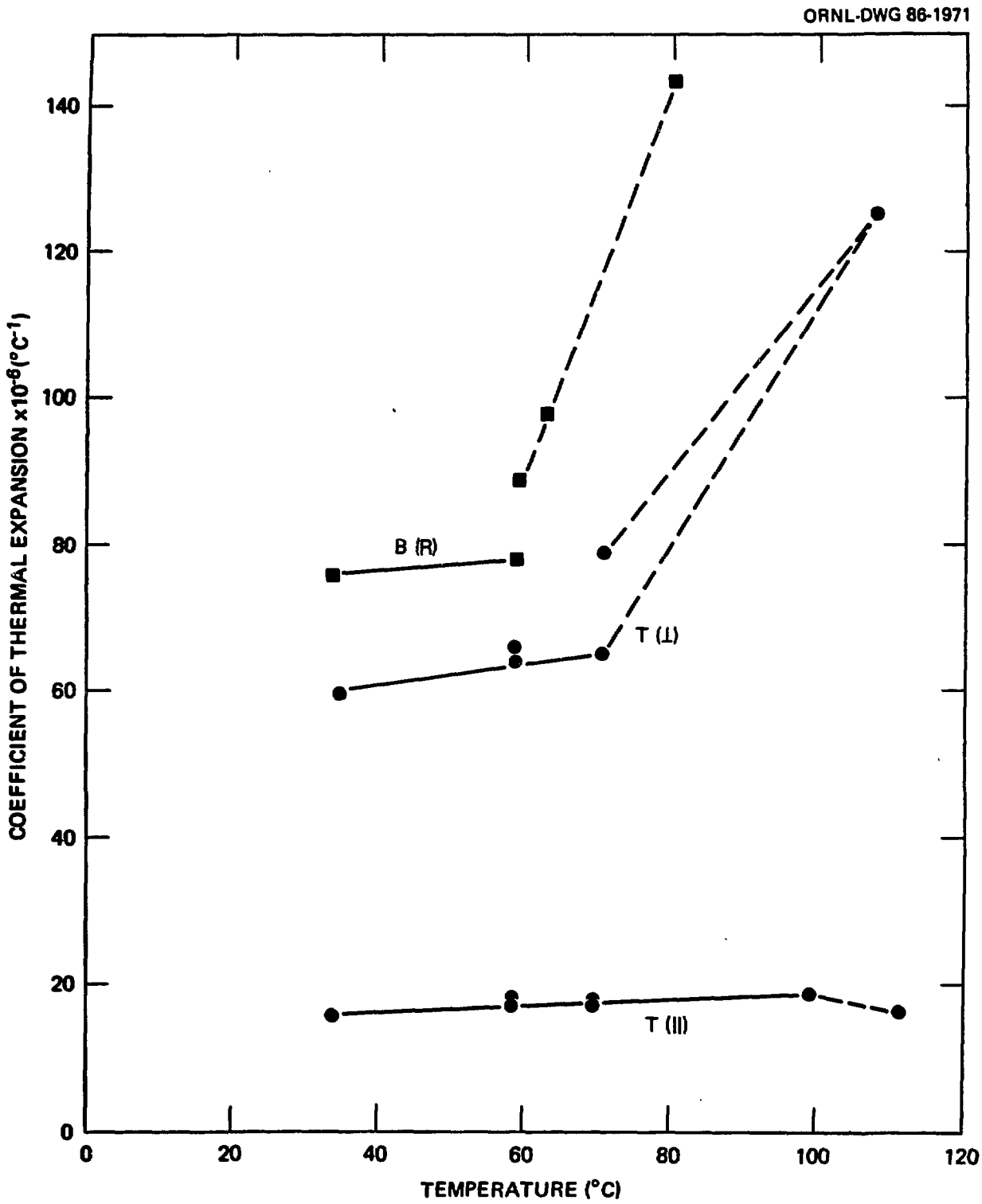
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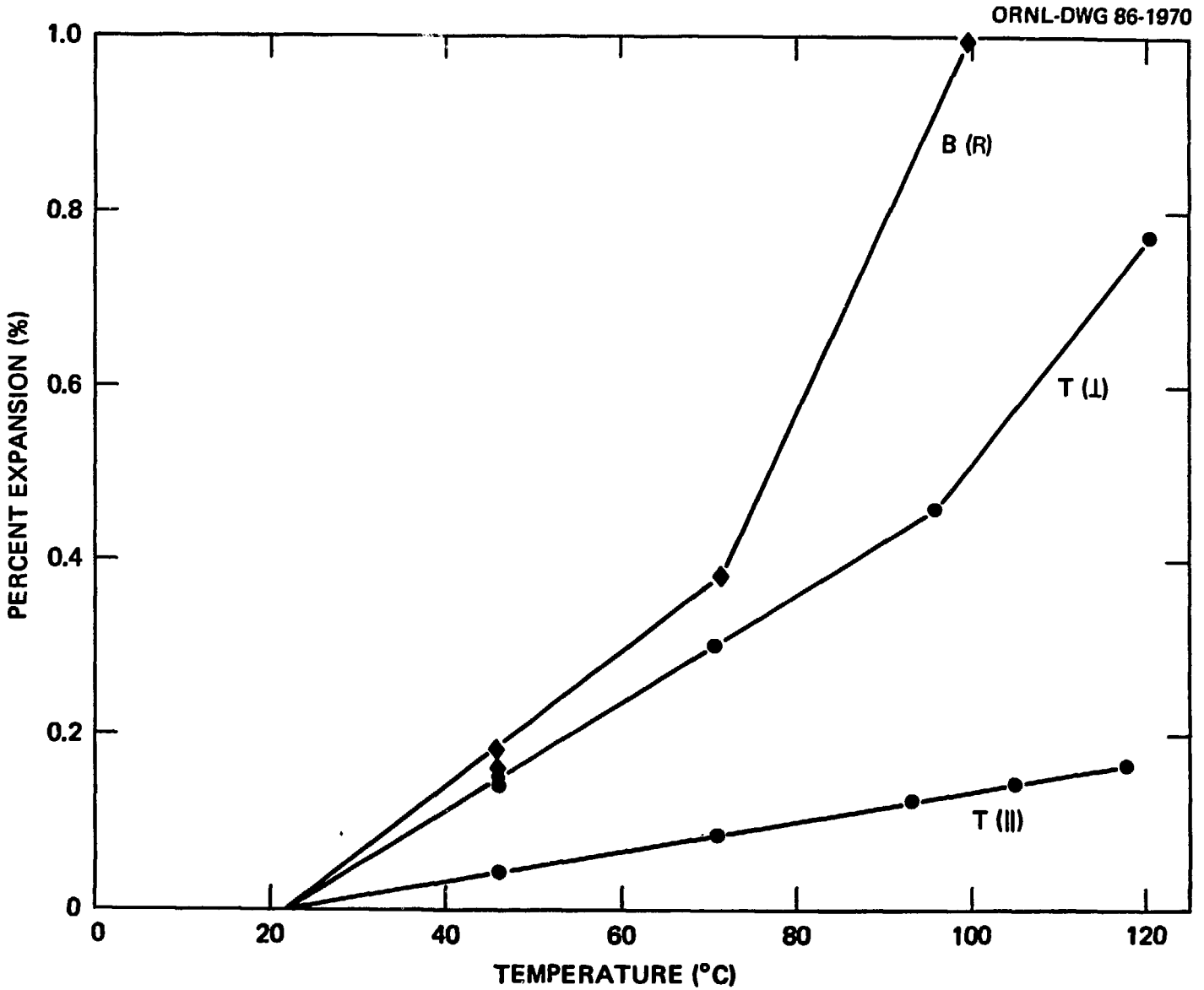
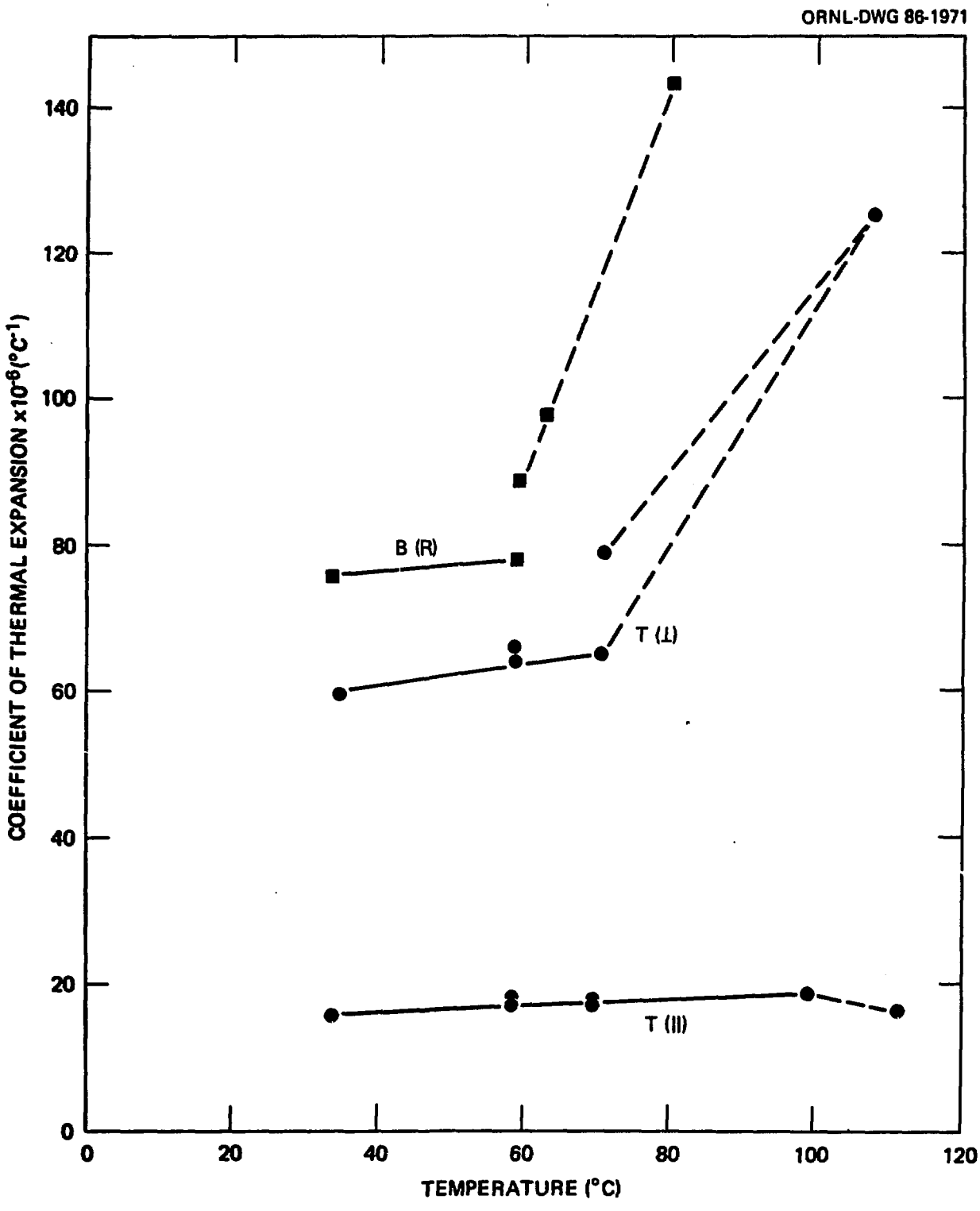

ORNL-DWG 86-1970
1.0
0.8
0.6
0.4
0.2
0
PERCENT EXPANSION (%)
0
20
40
60
80
100
120
TEMPERATURE (°C)
B (R)
T (⊥)
T (||)


ORNL-DWG 86-1971
COEFFICIENT OF THERMAL EXPANSION x10⁻⁶(°C⁻¹)
TEMPERATURE (°C)
0
20
40
60
80
100
120
140
B (R)
T (⊥)
T (||)