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MASTER

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MECHANICAL PROPERTIES CONSIDERATIONS FOR USE OF EPOXY INSULATORS AND BONDED JOINTS IN NEUTRAL BEAM ION SOURCES*

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Abstract

In the Doublet III (D-III) neutral beam injectors, cast, rigid-epoxy insulators are joined to the AISI 304 stainless steel corona rings with semi-rigid epoxy adhesive. Selected mechanical properties of these materials were measured between 11°C and 65°C, well below the material temperature limits, to identify the trends and to confirm adequate mechanical strength for the insulators. Significant creep deformation was measured at 22°C. Empirical relationships were developed to predict long term strain over a range of stress and temperature of design interest. Delayed failure was observed in bonded specimens at stress levels well below the ultimate strength. In order to protect the D-III neutral beam ion source epoxy from elevated temperature effects, a chill was installed in the cooling water circuit. Outgassing measurements of the insulator epoxy were made and found to be low and primarily H_2O.

Introduction

Cast epoxy high voltage insulators have generally been specified for use in neutral beam ion sources used for hydrogen plasmas. Difficulties and expense in fabricating sources using ceramic insulators have opened reconsideration of epoxy insulators for the pending D-T plasma devices. The ease of casting epoxy to any desired shape, its machinability, and low cost makes cast epoxy attractive. Further, joining is a straightforward procedure using semi-rigid epoxy adhesive. These features of the epoxy focus attention on the need for measurement of strength and long-term creep properties at moderately elevated temperature.

The D-III insulators are rectangular shaped rings, which are epoxy bonded to AISI 304 stainless steel components (Fig. 1). Stresses occur from dead-weight loading in the cantilevered held assembly and from evacuation. Calculated stresses are low when compared with published 22°C strength data by the suppliers of the epoxy formulations. Useful applications up to 80°C for cast, rigid epoxy (Bisphenol-A) and up to 121°C for a semi-rigid epoxy adhesive are implied. Since the current designs of ion sources do not provide temperature control of the insulators, an investigation was therefore conducted to measure property trends and to confirm adequate mechanical strength and creep properties for the insulators and bonded joints. Creep strain was measured as the insulator structure that supports the ion accelerator must not undergo substantial time-dependent strain or else the grid-spacing dimensions cannot be held within the critical dimensional tolerance of <0.2 mm. Empirical relationships were developed for predicting creep deformation.

A supplementary investigation was conducted to determine the degradation of epoxy and outgassing species at elevated temperatures.

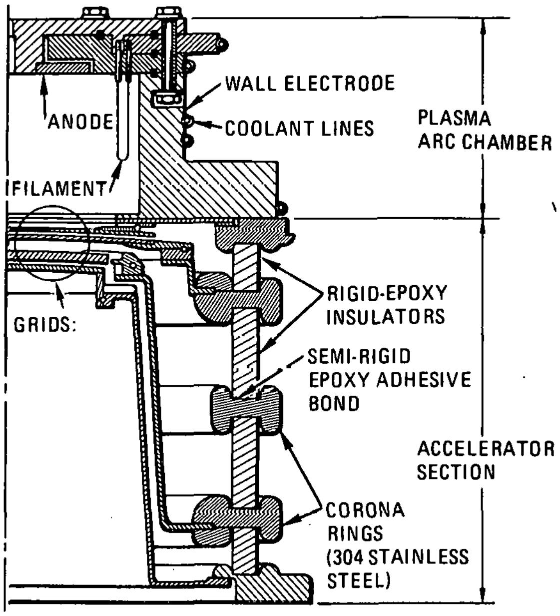

Fig. 1. Adhesive Bonded Insulator Joints in D-III Neutral Beam Ion Source

Experimental Approach and Procedure

Tensile-pull tests were conducted on specimens of dog-bone shape excised from a production insulator. The rigid-epoxy was of composition [75 wt. % Epon® 826 resin (Shell Chemical Co.); 15 wt. % Furane D-40 catalyst; 10 wt. % Dow corning 736, "flexibilizer"]. The cast epoxy insulator was cured at 60°C for 15 h. The pertinent tensile specimen dimensions were 17 cm length by 6 cm thickness. The gage region was 6 cm long by 1.2 cm wide. Marks were penciled at 5 cm intervals on the gage region to permit determination of % elongation. Also, an extensometer with 2.5 cm gage length was attached and load versus applied strain was recorded during load application at a constant rate-of-travel. The modulus of elasticity was calculated using the linear region of the load versus strain curve.

Measurements of creep behavior in air were obtained by averaging the strain values from gages bonded on both sides of the specimen. Temperature was measured with a thermocouple attached to the specimen. Uniaxial load was applied using dead-load weight affixed to a flexible chain linkage. Both ends of the specimen were held with a free-to-rotate cylindrical pin and clevis.

Tests for the adhesive bonded specimens were conducted in the same manner as described above for the tests of epoxy. A spacer was incorporated in each grip to enable the loading direction to be parallel to the bond line of the single-lap shear specimen. The different types of bonded specimens utilized are shown in Fig. 2. The bonded specimens were 17 cm in length and had the same cross sectional dimensions and were the same

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material as the rigid-epoxy tensile specimens. The cylindrical, butt-joint tensile specimens were 1.5 cm in diameter. The bonded area for the single-lap shear specimens were 1.3 cm × 1.6 cm (2.1 cm^2) and twice the amount for the double-lap shear specimen. The metallic member was AISI 304 stainless steel. The adhesive was a semi-rigid epoxy of the formulation 50 wt. % Eccobond® #45 resin and 50 wt. % Eccobond® catalyst #15 (Emerson and Cumming Co.).

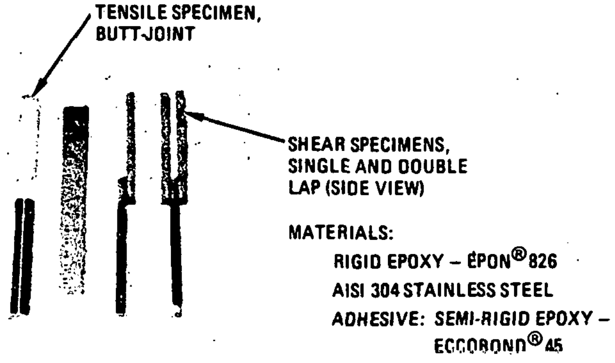

Fig. 2. Test Specimens for Evaluating Bond Strength of Epoxy-Adhesive Joints

The procedure used for surface preparation and adhesive bonding was selected to ensure consistent, optimum adhesion and was the same procedure used for bonding the insulators and corona rings for the D-III neutral beam ion source. Surfaces were appropriately cleaned and roughened by grit blasting. The stainless steel surface was also chemically cleaned and passivated and dried. The adhesive was mixed and entrapped air was evacuated. Adhesive was applied to both mating surfaces. The adhesive bonded specimens were cured at 44°C in air for 22 h with dead load applied to minimize porosity and to develop a uniform bond thickness of 0.03 mm ±0.01 mm.

The evaluation of the outgassing was conducted initially by measuring material weight loss using a thermogravimetric analyzer. The test sample was held in a Pt holder and exposed to a stream of flowing 99.999% purity argon at 1 atm pressure. A relationship was derived which enabled weight loss due to outgassing to be predicted at the lower anticipated D-III service temperatures. The volatiles were identified using mass spectrometry methods.

Test Results

The test results are presented in three parts. Tensile-pull and creep-test results for the rigid epoxy are reported first, followed by the adhesive bond strength results, and last, the investigation of volatiles emanating from the rigid epoxy.

Rigid-Epoxy Tensile Properties. The strength of the Epon 826 epoxy decreased sharply with increasing temperature as shown in Fig. 3. At 23°C the tensile strength was 60.3 MPa (8,750 psi) and decreased abruptly from 50.0 MPa (7,250 psi) at 48°C to 22.1 MPa (3,200 psi) at 56°C; an accompanying increase in ductility was observed. The modulus of elasticity at 25°C was calculated to be 3,078 MPa (0.45 × 10^6 psi) and at 48°C and 56°C was reduced to 2,627 (0.38 × 10^6 psi) and 1,882 (0.27 × 10^6 psi), respectively.

For constant stress values of 1.72 MPa (250 psi) and 4.14 MPa (600 psi) strain was measured at 11°C, 22°C, and 39°C and are plotted in Fig. 4. To enable an analytical prediction of long-term strain due to creep, assumptions were made in which the measured strain prior to one hour was considered to be elastic and subsequent strain to be creep strain, and a series of equations were developed using a least-squares curve fit of a power law model,

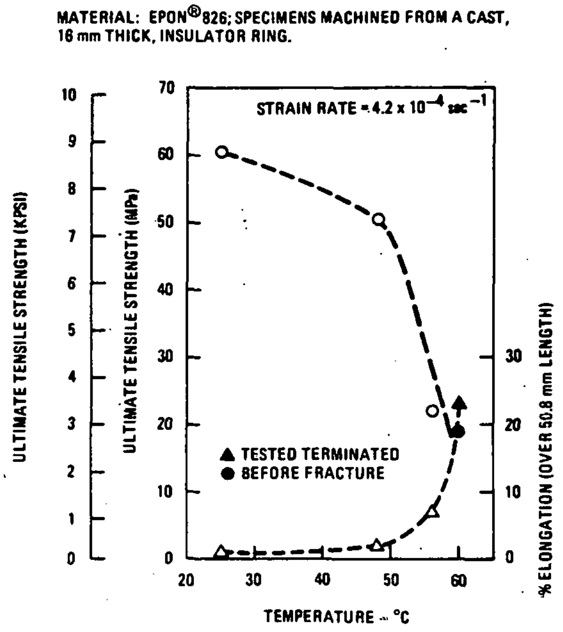

Fig. 3. Tensile Strength and Ductility at Elevated Temperatures for a Rigid Epoxy

$$\varepsilon = At^B \quad (1)$$

and for t = 1 hr., ε = A

$$\varepsilon_{creep} = A(t^B - 1) \quad (2)$$

Curves predicting creep strain to 12 × 10^3 hours are presented in Fig. 5. Using the same analytical method and all the strain data , a series of equations were developed for total strain. Curves of predicted total strain were generated as shown in Fig. 6. The equations and conditions for each are listed in Table 1.

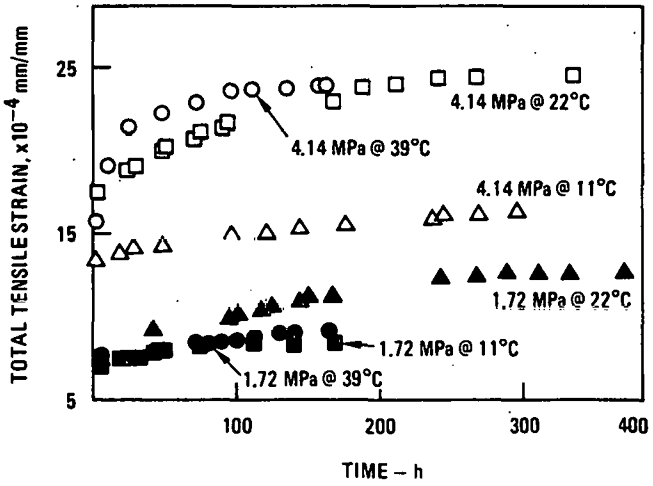

Fig. 4. Temperature and Stress Effects on Tensile Strain vs Time Behavior of Rigid Epoxy Epon® 826 During Constant Stress Tests

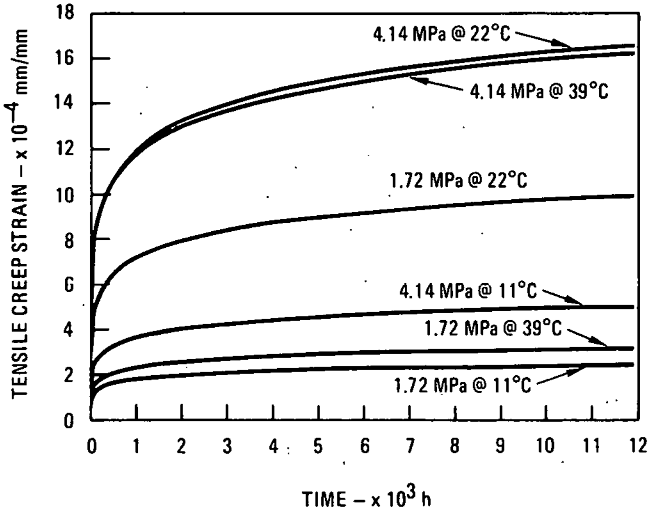

Fig. 5. Predicted creep strain at 1.72 and 4.14 MPa Stress Using Empirical Relationship

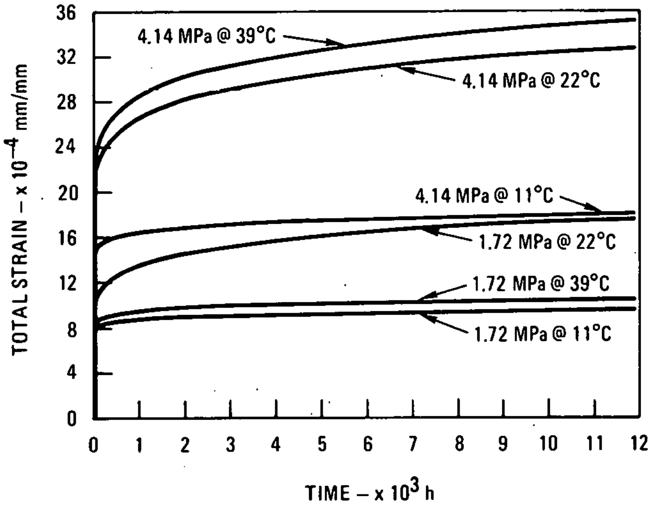

Fig. 6. Predicted Total Strain with Time at 1.72 and 4.14 MPa Stress Using Empirical Relationship

TABLE 1
EMPIRICAL EQUATIONS FOR PREDICTING TIME DEPENDENT STRAIN AT STRESSES OF 1.72 MPa AND 4.14 MPa AT TEMPERATURES OF 11°C, 22°C, AND 39°C

Temperature (°C)	Creep Strain Equation(1) (ε_{creep} =)	Total Strain Equation(2) (ε_{total} =)
At stress of 1.72 MPa (250 psi):		
11	$5696\ (t^{0.0045067} - 1)$	$698.62\ t^{0.033458}$
22	$7600\ (t^{0.013098} - 1)$	$646.49\ t^{0.10724}$
39	$9710\ (t^{0.003423} - 1)$	$713.54\ t^{0.4172}$
At stress of 4.14 MPa (600 psi):		
11	$6482\ (t^{0.0079378} - 1)$	$1289.50\ t^{0.035882}$
22	$8424\ (t^{0.01919} - 1)$	$1495.43\ t^{0.083277}$
39	$10575\ (t^{0.01521} - 1)$	$1590.30\ t^{0.084194}$

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(1)Empirical relationship $\varepsilon_{creep} = A\ (t^B - 1)$, curve fit through all strain data for time > 1 h (t = hours; ε = strain; A, B, C, and D are constants).

(2)Empirical relationship $\varepsilon_{total} = Ct^D$, curve fit through all strain data.

As shown in Figs. 4-6, the strain increase between 11°C and 22°C was greater than the increase between 22°C and 39°C. While scatter may be a factor, the most likely explanation is that at 39°C, additional curing and strengthening of the epoxy was taking place.

Strength of Adhesive Bonded Joints. Results for the uniaxial pull tests on the single-lap shear specimens and the butt-joint specimens are presented in Fig. 7. Failure consistently occurred by separation at the stainless steel/epoxy adhesive interface.

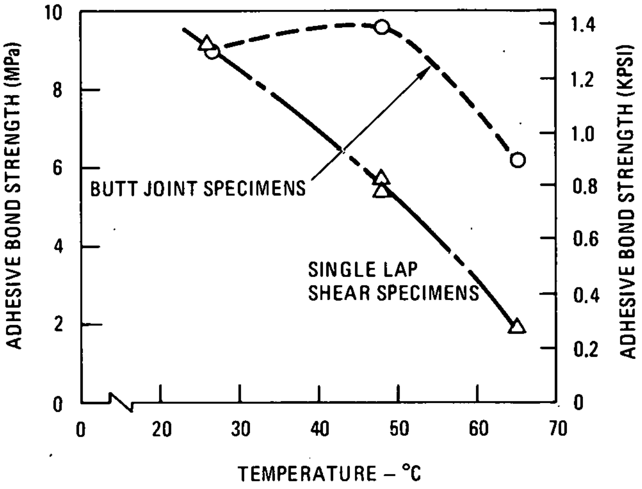

Fig. 7. Adhesive Bond Strength at Elevated Temperatures for a Cast, Rigid Epoxy Bonded to 304 Stainless Steel with a Semi-Rigid Epoxy Adhesive

The stress rupture results and parameters are presented in Table 2. In contrast with the fracture strength values of the short-term pull tests (Fig. 7), the delayed-failure phenomenon at lower stresses substantially limits the ability to place bonded joints under constant stress service at even moderately elevated temperatures.

TABLE 2
DELAYED-FAILURE PHENOMENON (STRESS RUPTURE) IN EPOXY-BONDED JOINTS

(Materials: rigid epoxy member bonded to 304 stainless steel with a semi-rigid epoxy adhesive)

Type of Bonded Joint	Test Temp. & Constant Stress (°C)	(MPa)	Duration of Test (hour)	Event at Termination of Test
Double-lap shear(1)	11	1.72	152	No failure
Double-lap shear(1)	22	1.72	117	No failure
Single-lap shear	48	4.14	2.33	Failure(2)
Butt(3,4)	11	1.72	50	No failure
Butt(3,4)	22	1.72	504	No failure
Butt(3,4)	39	1.72	165	Failure(2)
Butt(3)	48	1.72	0.72	Failure(2)
Butt(3)	48	4.14	0.0042	Failure(2)

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(1)Same specimen used in both tests. The 11°C test was conducted first.

(2)At interface of 304 SS and epoxy adhesive.

(3)Interface under tensile stress.

(4)Same specimen used in all tests. The 11°C test was conducted first.

Measurement of Volatiles from Epon 826 Epoxy.

Weight loss measurements were made for specimens of Epon 826 epoxy at 175°, 190°, 200°, 215°, and 225°C as shown in Fig. 8. The weight loss data plotted versus the square root of the time gives a reasonably straight line function after the first few hours of exposure. A relationship, useful in predicting weight losses for the epoxy, is obtained when the slopes of weight loss curves are plotted versus the reciprocal of absolute temperatures as shown in Fig. 9. The least square equation for the line shown is:

$$\log \alpha = \frac{-4.334 \times 10^3}{T} + 7.0553 \qquad (3)$$

where T is the absolute temperature and α is the slope value, and

$$\%\ \text{weight loss} = \alpha \sqrt{t} \times 100 \qquad (4)$$

where t is time in hours.

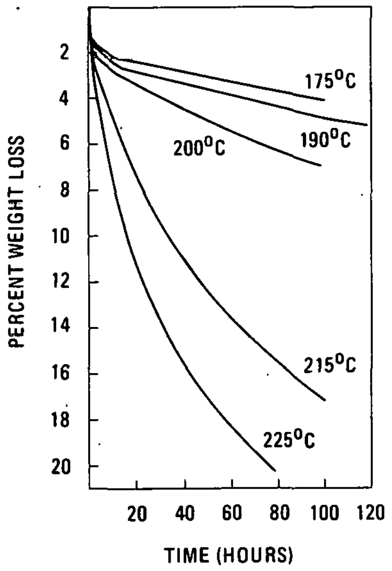

Fig. 8. Weight Loss of Epon® 826 Rigid-Epoxy at Elevated Temperature

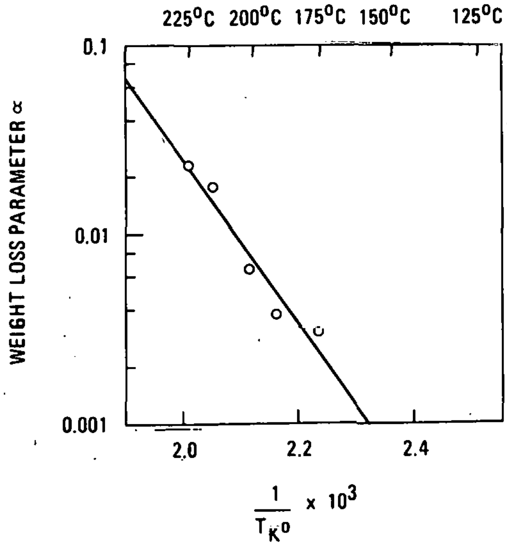

Fig. 9. Slope α Versus Reciprocal Absolute Temperature

Equations 3 and 4 can be used to predict percent weight losses for the Epon 826 epoxy to within ±15% for the temperature range 175° to 225°C. The equations should not be used above approximately 250°C, but should be appropriate for predicting long-term weight losses for temperatures at or below 225°C. Above ~250°C rapid weight loss occurs which indicates gross degradation has commenced. In Table 3 predicted values are presented of weight loss and outgassing rate for various times at temperatures of 21°C and 38°C.

TABLE 3

PREDICTED WEIGHT LOSS AND RATE OF OUTGASSING FOR EPON® 826 RIGID EPOXY AT 21°C AND 38°C

Time (h)	Weight Loss(1) (%)		Rate of Weight Loss(2) $\left(\frac{\text{Torr-liters}}{\text{cm}^2\ \text{sec}}\right)$	
	21°C	38°C	21°C	38°C
1,000	6.5×10^{-5}	4.1×10^{-4}	1.8×10^{-11}	1.2×10^{-10}
5,000	1.5×10^{-4}	9.3×10^{-4}	8.0×10^{-12}	5.4×10^{-11}
20,000	2.9×10^{-4}	1.9×10^{-3}	4.0×10^{-12}	$2.7\times10\text{-}11$

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(1) Calculated using Equations 3 and 4.

(2) Calculated using time differential of the weight loss prediction and conversion to Torr-liter/cm² sec using H_2O as the outgassing specie and a density of 1.17 g/cm³ for the epoxy.

The mass spectrometer measurements at 75°C identified outgassing species as primarily H_2O, with CO_2 (~2% by volume) and unidentified hydrocarbons (<1%) comprising the remainder. At 38°C H_2O was the only volatile specie.

Discussion and Conclusions

It is apparent that deformation by creep should be accounted for in using rigid-epoxy as a structural material in designs even at temperatures near 21°C. Further, the overall investigation has revealed alarmingly temperature-dependent strength limits for cast-rigid epoxy and/or epoxy adhesive joints. The delayed failure of bonded joints indicates that deformation by creep was appreciable, even at temperature and stress levels relatively low as compared to the material's upper temperature limit and short-term fracture strength values. It is therefore advisable, that for components supporting constant force or requiring maintenance of precise tolerances, both stress and thermal analyses should be conducted and supported by stress-rupture testing to account for deformation due to creep and the possibility of delayed-failure in bonded joints.

In order to protect the epoxy adhesive joints in the D-III neutral beam ion source from the elevated temperature effects, a chill was installed in the cooling water circuit and set for a regulated temperature of 20°C.

Since the outgassing of the insulator epoxy material below ~38°C was found to be primarily H_2O and at a sufficiently low rate, satisfactory operation of the ion source should not be impaired due to volatiles emanating from the insulators.

Acknowledgements

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