BPX INSULATION IRRADIATION PROGRAM TEST RESULTS*

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Abstract

The toroidal field coil insulation for the Burning Plasma Experiment (BPX) is expected to receive a radiation dose of nearly 10^{10} rad and to withstand significant mechanical stresses. An irradiation test program was performed at the Idaho National Engineering Laboratory (INEL) using the Advanced Technology Reactor (ATR) for irradiations to doses on the order of 3×10^{10} rad. The flexure and shear strength with compression of commercially procured sheet material were reported earlier [T. J. McManamy et al., "Insulation interlaminar shear strength testing with compression and irradiation," in *Proceedings of the 13th Symposium on Fusion Engineering*, 1990, pp. 342-347; "Insulation irradiation test programme for the Compact Ignition Tokamak," *Cryogenics*, vol. 31, pp. 277-281, April 1991]. A second series of tests has been performed to slightly higher dose levels with vacuum-impregnated materials, glass strand material, and Spaulrad-S sheet samples. Vacuum impregnation with a Shell 9405 resin and 9470 hardener was used to produce bonded copper squares and flexure samples of both pure resin and resin with S-glass. A new test fixture was developed to test the bonded samples in shear without applied compression. The Spaulrad-S flexure samples demonstrated a loss of strength with irradiation, similar to previous results. The pure resin lost nearly all flexibility, while the S-glass-reinforced samples retained between 30% and 40% of the initial flexure strength. The S-glass strands showed a 30% loss of strength at the higher dose level when tested in tension. The bonded copper squares had a low room-temperature shear strength of approximately 17 MPa before irradiation, which was unchanged in the irradiated samples. Shear testing of unirradiated bonded copper squares with ten different types of surface treatment revealed that the low shear strength resulted from the polyurethane primer used. In the later series of tests, the epoxy-based primers and DZ-80 from Ciba-Geigy did much better, with shear strengths on the order of 40 MPa. These samples also demonstrated a resistance to cryogenic shock. One irradiated bonded sample was tested up to 210 MPa in compression, the limit of the test fixture, without failure.

Introduction

A series of tests was carried out to evaluate the shear strength of bonded copper samples after exposure to radiation doses in excess of the the end-of-life requirement for BPX.

Two capsules were irradiated, one for 20 days and one for 40 days. Each capsule contained 50 bonded samples, a combination of flexure samples of Spaulrad-S sheet material and samples fabricated by impregnating S-glass with the Shell 9405 resin/9470 hardener system, and also ten strands of silane-treated S-glass. The capsules were approximately 1 in. in diameter and packed with aluminum powder for heat transfer to the outer surface, which was water cooled. The capsules were welded shut with a normal air atmosphere inside. Surrounding the capsule was a lead shield approximately 1.6 in. thick to reduce the gamma heating. The dose to the insulation was on the order of 2×10^{10} rad for capsule 1 and twice that for capsule 2 with roughly 40% of the dose from neutrons. The fast fluence (> 1.0 MeV) was approximately 1.7×10^{18} n/cm² and 3.4×10^{18} n/cm² for the two dose levels.

Material Selection

A manufacturing study was performed by the Advanced Technology Division (ATD) at Oak Ridge National Laboratory (ORNL), and vacuum impregnation was selected as the most promising technique to meet the BPX design requirements. Eight different combinations of epoxy resin and hardener were screened in laboratory tests to evaluate handling characteristics, compressive strength, and cryogenic shock resistance. The Shell system was selected because it had the best cryogenic shock resistance with the pot life and low viscosity required for impregnation. In addition, the hardener was an aromatic amine, which was expected to give good radiation resistance. The system is carcinogenic, however, which complicates handling.

Sample Fabrication

The bonded samples were fabricated by ATD. The copper was normal silver-bearing hard copper, approximately 3.63 mm thick, cut into 12.7-mm squares. The surfaces to be bonded were grit blasted using a procedure developed by Princeton Plasma Physics Laboratory (PPPL) for the Tokamak Fusion Test Reactor (TFTR). After grit blasting, the surfaces were cleaned and coated with a polyurethane-based primer, Conastic AD-20. The primer was cured at 80°C for six hours. S-2 glass cloth with a silane treatment was cut into 12.7-mm squares. Each sample had five layers of cloth between copper pieces, giving a bond thickness of approximately 1 mm. A mold was developed and used for vacuum impregnating 66

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samples at a time. Figure 1 shows the central mold plate with squares to hold the samples. Upper and lower cover plates with a series of grooves were used to seal the mold and provide a path for resin impregnation. This sample geometry was also chosen to allow future testing of combined compression and shear on bonded chips. The Shell 9405 resin was used with the Shell 9470 curing agent in a stoichiometric mix ratio of 100/28. The samples were allowed to gel at 80°C for 8 to 12 h and then cured at 120°C for 14 h. Three batches were fabricated for testing at INEL. A fourth batch was also fabricated with only four layers of glass; these samples were used at PPPL to evaluate the shear test device described below.

In addition to the bonded samples, sheet sections of resin and sheets of resin with S-glass were fabricated and cut into flexure samples 50.8 mm × 5 mm × 1.5 mm. Spaulrad-S 1-mm-thick sheet material was also cut into flexure samples of the same size.

Shear Test Fixture and Preliminary Testing

A shear test device for one sample was developed and is shown in Fig. 2. One edge of the sample is supported on a ledge, and the load is applied on the opposite edge through a pusher block. This block is supported on the face away from the sample by a piece of low-friction material that prevents rotation but allows vertical motion. Variations in sample thickness are accommodated by a wedge. The wedge surface is at a shallow angle and is designed to lock by friction due to the compressive loads generated by the restraint forces against rotation. A vacuum line is used to hold the sample while the wedge and pusher block are installed. This configuration allows for very easy insertion of a sample, which is important for use with activated chips.

Flexure Testing

The span length for the tests was 31.8 mm measured between the centers of 3-mm-radius supports. Six samples of each material were contained in both capsules. The extreme fiber stress at the highest load for control and irradiated samples of Spaulrad-S is shown in Fig. 3. It was noted that the results depended on the orientation of the sample. Higher strengths and slightly lower modulus values were found for control samples with the fill side on the bottom tensile surface. This orientation was used for the irradiated sample testing. The asymmetric construction was a result of an odd number of layers (5). The average peak stress of the three control samples with the fill side on the bottom was 870 MPa. The average loss of strength was 44% for capsule 1 (20-day exposure) and 54% for capsule 2 (40-day exposure).

The maximum flexure stress test results for Shell resin with S-glass are also shown in Fig. 3. Some of the irradiated sample failures were in interlaminar shear, rather than flexure. The average control group maximum stress was 558 MPa and the average reduction in strength was 69% for capsule 1 and 57% for capsule 2.

Flexure samples of pure resin were tested. The control samples showed a very large strain before failure. The irradiated samples, however, lost nearly all flexibility. Five of the six samples in each capsule fractured either during irradiation or during gentle cleaning after irradiation. The samples had turned completely black and the fracture surfaces appeared glassy. One sample from each capsule was available and was tested in flexure in addition to five control samples. The control group had an average flexure strength of approximately 100 MPa, while the flexure strengths of the two samples that survived irradiation were only about 7 and 15 MPa.

Fig. 1. Central mold plate.

(20-day exposure), which had been out of the reactor for six weeks, was approximately 1 R. The activity of samples from capsule 2 (40-day exposure) was approximately 3 R. The dominant contribution to the activation was from cobalt.

The test results are shown in Fig. 4. The average shear strength for the control group was 17 MPa. This was also the average for capsules 1 and 2. On average, no reduction due to radiation was noted. All failures were at the copper-epoxy interface. Control and irradiated samples were also tested after being "dunked" in liquid nitrogen and warmed to room temperature. No significant differences were found.

The irradiated epoxy changed color from amber to black. There was no consistent difference between the three fabrication batches.

One bonded sample from capsule 2 was tested in pure compression with the load perpendicular to the flat faces. The plot of deflection versus load was smooth, with no indications of failure up to 275 MPa, the limit of the test fixture.

Fig. 2. Shear test device.

Fig. 4. Shear strength.

S-Glass Tensile Strength

Strands of plain S-2 glass with silane treatment were irradiated in each capsule. Ten strands from each capsule and a control group of ten were tested in tension. Each strand was wrapped around cylinders approximately 16 mm in diameter separated by approximately 75 mm on a Tinius Olson tensile test machine. The ultimate stress at failure is shown in Fig. 5. The cross-sectional area was estimated from the measured weight per unit length and the published S-glass density of 2.48 g/cm³. The average 31% loss of strength at the higher dose indicates that a threshold for damage has been passed. The activity of the samples was about 0.4 mR for both capsules.

Fig. 3. Maximum flexure stress for Spaulrad-S and Shell resin with S-glass.

Shear Testing

Five samples from each of three fabrication batches were tested in shear from an unirradiated control group and from the two capsules. The contact activity of samples from capsule 1

comparison. Two groups were fabricated using the DZ-80 primer; one of these sets was cured at 250°F prior to impregnation as recommended by the vendor, and the second set was cured at 284°F, as recommended by PPPL based on the experience from FTIR testing.

Fig. 5. S-glass strand tensile strength.

Other Tests

Because different equipment and personnel were used before and after irradiation for dimensional measurements of flexure samples, two available control samples were remeasured at the hot cell. The results indicated that there was a systematic difference of approximately 0.02 mm between the measurements made before loading and those made at the hot cell. This difference would cause an apparent shrinkage in thickness on the order of 2%. A correction factor was added to all hot cell measurements for comparison with measurements taken before irradiation. The changes in width, thickness, length, and volume for Spaulrad-S and resin with glass were on the order of 1% or less and probably within the experimental error. The pure resin volume change on average was -5% for capsule 1 and -3.5% for capsule 2.

One flexure sample of each type was also used to measure density for control and irradiated conditions. The density was measured by weighing the samples in air and in water. Figure 6 shows the results.

The Spaulrad-S and resin densities agree well with published data. The density for the resin + glass implies a glass volume fraction of 40% based on the S-glass density of 2.48 g/cm³.

PRIMER EVALUATION

After the shear test results and manner of failure were reviewed, a set of shear samples was fabricated by ATD, using the same resin and procedures as before with a variety of different primers. With one impregnation of the mold, 60 samples were fabricated; there were 10 groups of samples, each containing 6 samples of each type of surface preparation. One group used the AD-20 primer for comparison with the RAD II results. Another group used no primer for a control

Fig. 6. Density measurements.

One set of samples received a surface treatment from PDA Engineering, which developed a method of improved copper-epoxy bonding under a DOE SBIR contract. The other five types of primers were selected by ATD after a literature survey. One primer type was the EBANOL-C chemical treatment based on the ASTM procedure. The other four were different types of epoxy-based primers from American Cyanamid, Dexter Hysol, and 3M. A summary is given in Table 1.

After curing, one of the unprimed samples and three of the samples primed with the PDA process failed when they were removed from the mold. The remaining samples were tested in shear on a duplicate of the shear test device used at INEL. Prior to testing, two samples from each group (except for the PDA and unprimed samples) were dunked in liquid nitrogen and then warmed to room temperature to see if cryogenic shock influenced the shear strengths. The test results are shown in Fig. 7. Shear strengths as high as 48 MPa were measured with the average of some on the order of 35 to 40 MPa. Most samples still failed at the copper-epoxy interface, but several of the samples that failed at the highest loads did have an interlaminar shear failure mode. The epoxy-based primers as a group did well. There was a significant amount of scatter in the data. Examination of samples indicated that the glass did not go to the edges, and this may be part of the reason for the scatter. Cryogenic shock did not appear to have a significant effect.

CONCLUSIONS

The measured shear strengths after irradiation were limited by the adhesive strength of the copper-epoxy bond to approximately 17 MPa with the polyurethane primer used.

The cohesive shear strength of the epoxy systems was >20 MPa and the compressive strength was >275 MPa, even after a dose on the order of 4×10^{10} rad. There was a significant amount of scatter in the data, but the results appeared to be independent of radiation dose level within the range tested. Subsequent testing indicates that the interlaminar shear strength of the Shell system is on the order of 40 to 50 MPa prior to irradiation. Cryogenic shock does not appear to have a significant effect. The Shell resin showed a great loss of flexibility due to irradiation damage. The flexure samples of Shell resin with S-glass did not fail at the strain limit of the pure resin and went to significantly higher strains, although there was a large loss of strength compared to the unirradiated samples.

There appears to be a threshold for damage to the S-glass, and a 31% loss of strength at the high dose level was observed.

The data on dimensional changes are inconsistent. There is no direct agreement between measured changes in dimensions and measurement of volume change based on density calculations from wet and dry weight measurement. There is some indication of shrinkage, but the amount appears to be less than reported previously.

Acknowledgment

This report is the result of the work of a large number of people, including those at ATD who designed the mold and did the screening tests, the hot cell technicians at INEL who performed all the irradiated sample testing, and the Princeton technicians who developed the shear tester and evaluated the primers. Space does not permit a complete list, but special thanks are due J. Dooley, J. Hubrig, and C. Janke at ATD, D. Sparks at INEL, and R. Majeski and C. Lindemuth at PPPL.

Table 1. Summary of primer types

Identifier	Name	Vendor	Type
1	AD-20	Conastic, Inc.	Polyurethane-based
2	Ebanol-C	Ethone, Inc.	ASTM D2651 Method E
3	DZ-80	Ciba-Geigy (U.K.)	Phenol formaldehyde and methylated melamine in methylated solvent, cured at 250°F
4	BR127	American Cyanamid	Epoxy phenolic with corrosion inhibitors
5	EA9210H	Dexter Hysol	Epoxy based with corrosion inhibitors
6	EC3924B	3M Co.	Epoxy based with corrosion inhibitors
7	EC3960	3M Co.	Epoxy based with corrosion inhibitors
8			No primer
U	PDA	PDA	Chemical process for copper-epoxy bonding
X	DZ80	Ciba-Geigy (U.K.)	Phenol formaldehyde and methylated melamine in methylated solvent, cured at 284°F

Fig. 7. Bonded sample shear strength with different primers.

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