STATUS OF LITHIUM-FILLED SPECIMEN SUBCAPSULES FOR THE HFIR-MFE-RB-10J EXPERIMENT — J. P. Robertson, M. Howell, and K. E. Lenox (Oak Ridge National Laboratory)

OBJECTIVES

The objective of this research is to irradiate a sufficient variety and number of mechanical properties specimens of V-4Cr-4Ti at 420 and 480°C in order to study the flow and fracture behavior and to establish a mechanism-based approach to modeling the effects of external parameters on fracture.

SUMMARY

The HFIR-MFE-RB-10J experiment will be irradiated in a Removable Beryllium position in the HFIR for 10 reactor cycles, accumulating approximately 5 dpa in steel. The upper region of the capsule contains two lithium-filled subcapsules containing vanadium specimens. This report describes the techniques developed to achieve a satisfactory lithium fill with a specimen occupancy of 26% in each subcapsule.

PROGRESS AND STATUS

The HFIR-MFE-RB-10J experiment, conducted under the U. S. DOE/Japan Atomic Energy Research Institute Collaborative Testing Program, will be irradiated in a Removable Beryllium position in the HFIR for 10 reactor cycles, accumulating approximately 5 dpa in steel. The upper region of the capsule contains two lithium-filled subcapsules containing vanadium specimens. One subcapsule will operate at 420°C and one will operate at 480°C. The lower region of the capsule contains austenitic and ferritic steel specimens operating at 250°C. The capsule will be surrounded by a Eu_2O_3 shield in order to harden the spectrum and prevent unwanted transmutations.

The vanadium specimens in this experiment were encapsulated in lithium-filled subcapsules to control the vanadium absorption of the interstitial impurities carbon, nitrogen, and oxygen, and to provide temperature uniformity between specimens.

There were five major issues to be resolved prior to filling the subcapsules with lithium and each of these is briefly discussed here. A complete report is in progress.

Specimen loading: Each subcapsule was made out of type 316 stainless steel. The interior diameter was 2.62 cm and the interior height was 9.75 cm. Fifty-one SS-3 type flat tensile specimens, 23 one-third size pre-cracked Charpy V-notch specimens, six creep tubes, one DC(T) fracture toughness specimen, and one 1.52 cm-long tube containing 25 TEM disks were put into each subcapsule. A loading arrangement had to be devised such that all the specimens fit and were surrounded by lithium, and that they stayed in place during the various welding and inspection procedures. The specimens were arranged into three layers such that the top of the upper layer was 5.59 cm high in the capsule, leaving a gas gap at the top of the capsule of 4.17 cm. This gas gap was required to be at least 1.45 cm in order to accommodate all of the gas contained in the pressurized tubes, should they burst. A sleeve of high purity vanadium sheet metal was fit inside the capsule to minimize the exposure of the lithium to the steel wall and to serve as a getter material during irradiation. Of the total internal subcapsule volume, including the necessary gas gap, vanadium specimens occupy 26%. For comparison, the specimen packing fraction in the EBR-II X530 experiment ranged from about 11 (for a mixture of SS-3 tensile and TEM disk specimens) to 33% (for a mixture of DCT-B specimens and 1/3-size Charpy specimens) for the various subcapsules [1].

handled in a flowing argon glovebox and specimens taken for analyses were triply contained in glass bottles, plastic zipper bags, and mason jars. After purification, samples were taken by forcing a small amount of the Li into a "dead-end" tube that was then welded closed. The metals analyses were performed in the Chemical and Analytical Sciences Division of ORNL. The oxygen content was analyzed by neutron activation analysis in the Analytical Services Organization (ASO) at Y-12. The ASO also performed nitrogen analyses using a modified Kjeldahl procedure. Twin samples were also sent to the Analytical Chemistry Laboratory of Argonne National Laboratory for analysis via the modified Kjeldahl procedure. In each instance, the receiving laboratory was notified of the sample shipment and was prepared to take the appropriate actions to maintain the cleanliness of the sample. The on-site sample shipments were made by special courier and the shipments to ANL were by overnight express mail.

Lithium source and purification: "High purity" Li-7 was obtained from the Y-12 Plant in Oak Ridge. This isotope of lithium was chosen in order to reduce the production of tritium by transmutation reactions in the HFIR. The lithium was 99.99% Li-7. Because of its cost and rarity, extreme care was used to minimize the volumes left behind as "scrap" in the processing steps. The Li-7 was provided in the form of irregular chunks, approximately 125 cm^3 each. The chunks were melted, slagged, and then gettered with chromium chips and then zirconium chips and titanium foil. The as-melted material had a nitrogen content of 540 μg/g (540 wt ppm), an oxygen content of 0.17%, and only trace amounts of metallic impurities. The lithium was pushed through a 40 μm filter to remove any remaining slag material and the larger oxide particles but no attempt was made to getter for oxygen. The as-gettered material that went into the experimental capsules had a nitrogen content of 26 μg/g (26 wt ppm) and an oxygen content of 0.16 wt%.

Capsule filling: The capsules were filled using a technique known as the "blowback" method. In this technique, the specimens are placed inside the capsule and the capsule head is welded in place. This head has two penetrating tubes: lithium flows in one, fills the capsule, and overflows out the exit tube. After the capsule is completely full, high purity helium is blown back through the exit tube and the excess Li is blown back through the fill tube to the reservoir. Once the Li level recedes down to the height of the fill tube, only helium gas flows back up the fill tube and the Li level is set. For the fill process, the Li was pushed under high purity He gas pressure through clean stainless steel tubing. All connections were made with stainless steel swagelock fittings. In preparation for capsule filling, the lithium temperature was raised to 400°C. This temperature was a compromise based on two factors: as the temperature increases, lithium is more likely to wet the steel capsule and the vanadium specimens and the lithium flows easier in the transfer tubes. Above 500°C, however, the lithium begins to leach nickel from the stainless steel. Prior to filling, the capsule was evacuated to a vacuum on the order of 10^{-1} torr. A valve on the exit tube was closed and the Li was forced in from the reservoir; the pressure was gradually increased to 69 kPa. The lithium was then blown back with both minimum pressure and flow rate. The capsule was then left to soak at temperature (400°C) and a vibration applied. After a soak/vibration of about 45 minutes, the fill and blowback processes were repeated. After the capsule was filled, the fill and exit tubes were crimped closed using a specially designed tool to ensure a good seal. The exterior portion of these tubes were then cut to the design length and welded.

Detection of fill level: The nature of this fill process required the establishment of a non-destructive technique to measure the lithium fill level. A number of techniques were attempted but only two proved very useful. Both were performed on the capsules while they were still attached to the fill system so that, if the fill was not adequate, the capsules could be filled again. Once the fill tubes are crimped and the capsule removed from the fill system, no more lithium can be added. Standard x-ray radiographs using a portable x-ray source were taken of the capsules while they were still attached to the system. The lithium level showed up as a faint contrast change against the black of the gas gap at the top of the capsule. The steel wall, the steel fill tubes, and the vanadium specimens appeared very bright and the lithium contrast was not visible in these regions. The second useful technique was a variation on ultrasonics. Through experimentation, it was determined that a relationship existed between the presence of Li and the wall ringing induced by an ultrasonic transducer. Even though several interfaces existed because of the capsule liner, the lithium dampened the ringing significantly. This damping was used to estimate the fill level to within about 0.3 cm. This same method could was also used to locate and size any significant (greater than 1.3 cm diameter) internal voids within the capsule.

CONCLUSIONS

The techniques outlined above resulted in the successful filling of the two subcapsules required for the HFIR-MFE-RB-10J experiment. The fill level was confirmed by x-ray and by ultrasound and the fill uniformity was confirmed by ultrasound and by a post-weld weight measurement. The shape of the lithium meniscus seen in the x-ray implies that the lithium is wetting the vanadium liner and therefore is also wetting the vanadium specimens .

FUTURE WORK

A complete report detailing all aspects of this project is in progress.

REFERENCES

1. H. Tsai, R. V. Strain, A. G. Hins, H. M. Chung, L. J. Nowicki, and D. L. Smith, in Fusion Materials Semiannual Progress Report (March 31, 1995), DOE/ER-0313/18, U.S. DOE Office of Fusion Energy, July 1995, p. 85.