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Introduction

The effect of irradiation on materials properties is an important field of study for materials usage in both fission and fusion systems for energy production. Neutron flux and energy spectrums are well understood; however, the irradiation temperature can be more difficult to determine. Since the early 1960s, Silicon Carbide (SiC) has been used as a passive post-irradiation temperature monitor because the irradiation defects anneal out above the irradiation temperature. Irradiation temperature is determined by measuring a property change after isochronal annealing or during a continuously monitored annealing process. There are many properties that may be measured including electrical resistivity, bulk density, dimensions, thermal diffusivity, or lattice spacing. Electrical resistivity is accepted as a robust measurement technique; however, such method is time-consuming since the steps involved must be performed in a serial manner. The use of thermal expansion from continuous dilatometry is an automated process requiring minimal setup time. As part of a Nuclear Science User Facilities (NSUF) project, low dose silicon carbide monitors were irradiated in the BR2 material test reactor at SCK, Belgium. These samples were then evaluated at the Idaho National Laboratory (INL) High Temperature Test Laboratory (HTTL) to determine their peak temperature achieved during irradiation. The technical significance of this work is that total dose of the irradiated monitors is significantly less than the recommended in published literature. This paper will discuss the evaluation processes available at HTTL to read peak irradiation temperature of passive monitors.

Figure 1 - SiC temperature monitors available for use in irradiation testing include small rods and discs. Monitors photographed with US cent for size perspective.

Methodology

In the first method, HTTL used resistivity measurements to find the peak irradiation temperature. The SiC monitors are heated in the annealing furnace using isochronal temperature steps. After each isochronal annealing, the specimens are placed in a resistance measurement fixture located in the constant temperature chamber (maintained at 40°C) for a minimum of 30 minutes. An ohmic response curve is generated for each monitor prior to heating. The peak irradiation temperature, using an electrical resistivity technique, can be taken as the point where the resistivity begins and consistently remains, above the error band. For this evaluation, the error band was established as the ±2σ value based on a sample size of the first five data points taken below 150°C.

In the second method, INL researchers developed a method aimed at using electrical resistance measured during a two-pass heating – cooling cycle as a means of recovering the irradiation temperature of a SiC monitor. A fully automated means of using continuous measurement of resistance of SiC monitors during heating/cooling has been developed and involves relatively inexpensive resistance measuring equipment. To minimize thermal perturbations, which increase uncertainty in the temperature measurement, a constant heating rate is applied during the measurements above 150°C. Results indicate that the relationship between resistance and temperature of a SiC monitor shows a significant change in resistance difference slope when the peak irradiation temperature is reached.

Finally, the last method uses thermal expansion from continuous dilatometry that is an automated process requiring minimal setup and run time. This method uses an optical dilatometer that requires no contact with samples, eliminates the need for measurement calibrations and corrections, and requires only one measurement run to determine irradiation temperatures. Moreover, there has been only very limited reports on the effect of irradiation on the thermal expansion behavior of SiC. The historical inability to implement this process was most likely due to limited resolution of the dilatometers and lack of statistical analysis methods. This dilatometer has a very high resolution that produces continuous measurement of length/diameter. In order to achieve similar anticipated error range in determining irradiation temperature as previous methods discussed, ramp rates smaller than ~2.5 K/min for the dilatometry-based thermometry is recommended. Limited yet significant improvement in accuracy may be achieved by further decreasing the ramp rate.

Conclusion

SiC temperature monitors were irradiated in BR2 as part of an NSUF project and were evaluated at the HTTL using multiple evaluation methods to determine their peak irradiation temperatures. The peak irradiation temperature of each monitor was evaluated using the resistance measurement method, an automated resistivity method, and dilatometry-based thermometry method. Deviations between the calculated temperature and the evaluated temperature were within or near published limits for all methods. A significant finding from this evaluation is that it is possible to evaluate SiC temperature monitors at dose levels much less than 1 dpa. SiC monitors were successfully evaluated that were irradiated to 0.5 dpa with temperatures ranging from 240 – 380°C.

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