**HELIUM EFFECT ON IRRADIATED MICROSTRUCTURES AND PROPERTIES IN SiC/SiC COMPOSITES**—Y. Katoh (Oak Ridge National Laboratory), S. Kondo, K. Ozawa, K. H. Park, and A. Kohyama (Kyoto University)

## OBJECTIVE

The objective of this report is to summarize recent efforts for understanding the effects of helium on irradiated microstructures and properties in silicon carbide and its composites in fusion environments by using a technique of dual-beam charged particle irradiation.

## SUMMARY

Synergistic effects of displacement damage production and helium implantation are being extensively studied. Recent progress can be highlighted:

- Major progress is achieved in comprehensive understanding of microstructural development in SiC during irradiation to very high doses up to very high temperatures.
- Substantial understanding of void swelling behavior of SiC in the presence of helium is provided.
- Superior irradiation stability of an advanced fiber-matrix interphase is demonstrated.
- Helium-related potential issues for the Generation-III SiC fibers are identified.
- Understanding of irradiation-induced toughening and the underlying mechanism is advanced.

## PROGRESS AND STATUS

Technical issues related to potential helium effects in SiC and SiC/SiC composites for fusion in-vessel structural applications are identified and summarized in Table 1 [1]. Among the anticipated issues, potential helium-assisted enhancement in point-defect swelling and thermal conductivity degradation may force up the low temperature application limit of the materials of these classes. On the other hand, the enhanced cavity swelling may lower the limits of highest temperature and lifetime fluence in high temperature regimes of design window. Potential detrimental effects on interphase stability, irradiation creep and matrix fracture toughness may affect the materials performance at all temperatures.

### Microstructural Development under Dual-beam Irradiation

The experiment was performed using chemically vapor deposited (CVD) SiC (Roam & Haas Co., Philadelphia, PA) as samples at DuET dual beam static accelerators facility, Kyoto University, Japan. Irradiation temperatures, displacement damage rate, and helium co-implantation rates were 873~1673K, ~$1x10^{-3}$ dpa/s, and 0/60 appm He/dpa, respectively.

At 1273K, 'black spot' defects and small dislocation loops developed at very high density. In the dual-beam irradiation case, the initial 'black spot' microstructure appeared substantially finer than in the single-beam case. As the number density of 'black spot' defects increased with the increasing dose, they tended to align to form planer agglomerates, and eventually collapsed into planer defects. At 1673K, without helium, the initial 'black spot' features developed into large planer defects on <111> family planes before the fluence level reached 30dpa. When helium was present, dislocation networks and fine helium bubbles developed instead of the planer defects by 30dpa.

Table 1. Potential helium effects in irradiation properties of SiC and SiC/SiC composites

| | |
|---|---|
| Enhanced point-defect swelling | - Potentially significant at intermediate temperatures.<br>- Comprehensive data available from a dual-beam ion irradiation experiment. |
| Cavity-swelling | - Potentially significant at >1000C at very high fluence levels.<br>- Cavity formation occurs in high purity crystalline SiC when helium atoms are present. |
| Enhanced thermal conductivity degradation | - Potentially significant at intermediate temperatures.<br>- Experimental data not published. |
| Fracture toughness modification | - Potentially significant at all temperatures.<br>- Fracture toughness of SiC may either be enhanced or be degraded and may consequently influence composite strength. |
| Enhanced interphase instability | - Potentially significant at all temperatures.<br>- Microstructural damage accumulation and amorphization are promoted by helium in PyC interphase.<br>- Helium cavity precipitation on PyC-SiC boundaries. |
| Enhanced irradiation creep | - Potentially significant at all temperatures.<br>- Very significant dislocation evolution acceleration and/or modification by helium. |
| Accelerated amorphization | - Anticipated only at < ~150C. |

Cavities (voids and bubbles) were not observed in CVD-SiC irradiated with self ions only at 1273~1673K. During dual-beam irradiation at T>~1273K, relatively large cavities developed preferably on grain boundaries, subgrain boundaries and twin boundaries. These cavities grew in size with the increasing dose. Tiny bubbles became visible within matrix at very high concentration at T>~1473K. Size distribution of the matrix bubbles remained unchanged up to ~100dpa. Large coarse cavities were found, both on grain boundaries and within matrix, in the subsurface regions of 1673K dual-beam irradiated samples. This indicates that a long-range helium transport has occurred toward the free surface by diffusion on grain boundaries and/or along network dislocations.

As a conclusion, helium co-implantation at 60 appm He/dpa significantly modified the irradiated microstructure in cubic SiC. The observation is summarized in Figure 1. Helium influenced evolution of all types of defects as well as introduced new type of defects in irradiated SiC. Drastic microstructural modifications by helium occurred at T>~1073K, at which vacancies are mobile. Void swelling, high temperature creep, and thermal conductivity shall be affected by helium through vacancy-related mechanisms. Many of the underlying mechanisms for the observed helium effects on microstructural defect evolution are not sufficiently understood. Nature of some defect features is not yet known, either. The interaction of helium with development of dislocation microstructures seems to be the key issues for better understanding the helium effects. Details will be published in Reference [2].

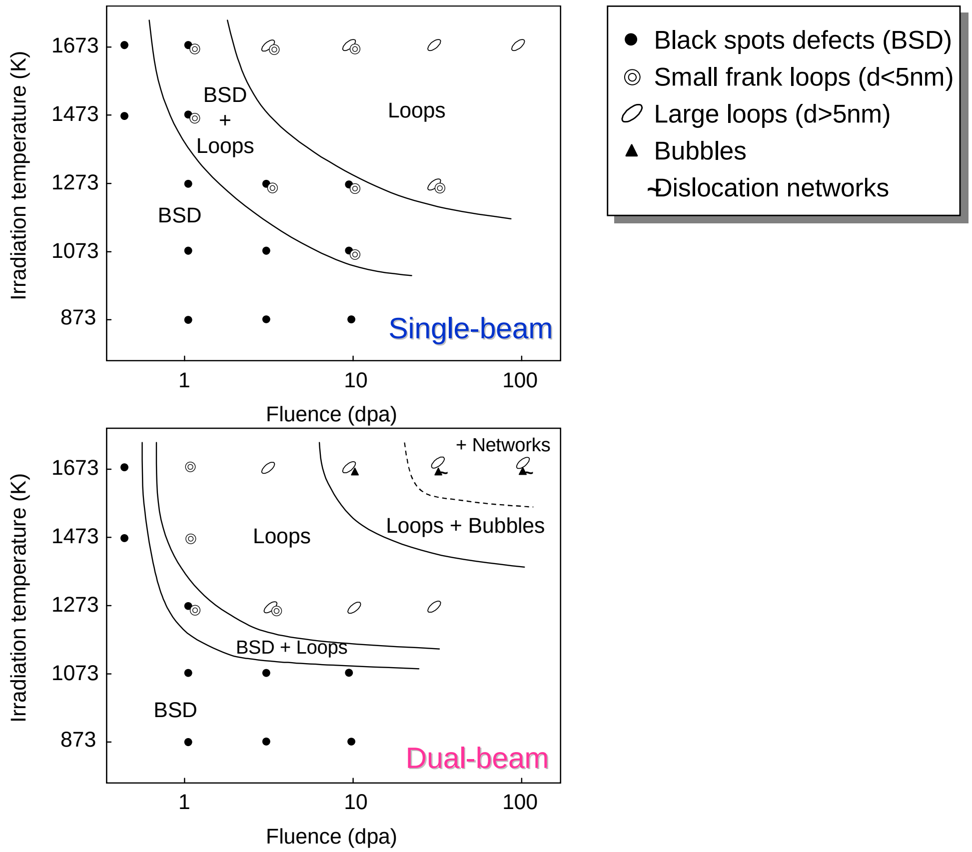


Figure 1. A preliminary overview of helium effect on microstructural development in CVD-SiC during ion irradiation.

**Helium Effect on Swelling Behavior**

The experiment was performed in a way similar to the previous section, while irradiation temperatures and helium co-implantation rates were 333~1673K and 0/60/1000 appm He/dpa, respectively, for the purpose of swelling determination. Macroscopic swelling was measured by precision surface profilometry [3]. Void swelling was determined based on graphical analysis to cavity images on transmission electron micrographs.

In the 'point defect swelling' regime, saturated ion-induced swelling agreed well with recent neutron data and post-neutron irradiation annealing data at T<~1273K. At T>~1273K, ion irradiation causes enhanced swelling due probably to dose rate effect and implantation effect. Helium seems to have enhanced swelling significantly at 673~1073K, as shown in Figure 2. The swelling enhancement observed in this temperature range seems to be too significant for the implanted amount of helium. It is preferred that this result is confirmed by additional experiments.

Void swelling was identified at temperatures higher than ~1273K. The fluence-dependent void swelling curves, which can be seen in Figure 3, seem to be consisting of incubation, transient and steady-state

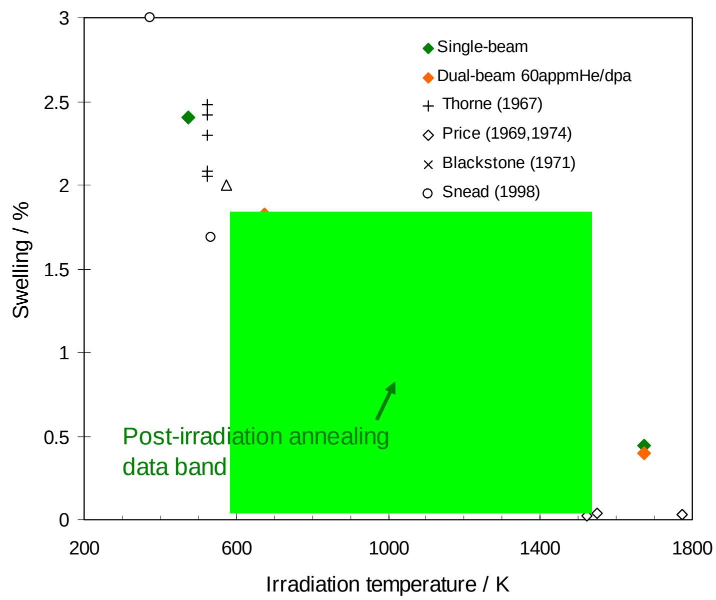


Figure 2. Irradiation temperature dependence of macroscopic swelling of ion-irradiated CVD-SiC with and without helium co-implantation at 60 appm He/dpa. The amount of swelling was measured by surface profilometry.

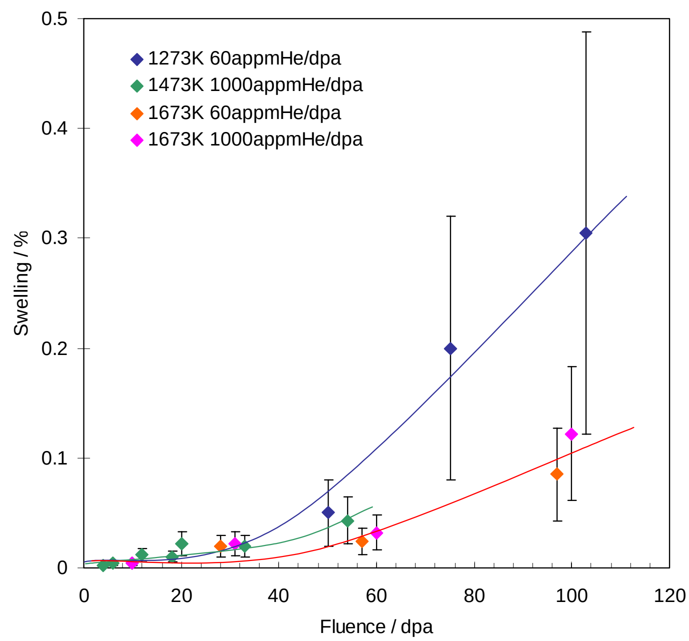


Figure 3. Fluence dependence of cavity swelling in CVD-SiC during dual-beam ion irradiation. The amount of swelling was estimated from TEM micrographs.

regimes, as is common in metals and metallic alloys. The extent of void swelling appeared relatively small. Temperature dependence of steady-state swelling rate can not be discussed from these data, primarily because the transport of helium to free surfaces must significantly be affecting data for 1673K, as discussed in the previous section.

**References**

[1] Y. Katoh, A. Kohyama, T. Hinoki, and L. L. Snead, Fusion Science and Technology 44 (2003) 155.

[2] S. Kondo, Y. Katoh, and A. Kohyama*, Interaction of Dislocation Development with Helium in Silicon Carbide*, presented at the 11th International Conference on Fusion Reactor Materials, December 7-12, 2003, Kyoto, Japan.

[3] Y. Katoh, H. Kishimoto, and A. Kohyama, J. Nucl. Mater. 307-311 (2002) 1221.