MECHANICAL PROPERTIES OF HELIUM IMPLANTED SILICON CARBIDE – L. L. Snead (Oak Ridge National Laboratory), R. Scholz (JRC Ispra), A. Frias Rebelo (Whereabouts Unknown)

Abstract

It has been long apparent that ceramics will undergo significant gas production, transmutation and material "burn-up" under high energy neutron irradiation. For example, the original calculations [1] for pure SiC exposed to 1 MW-a/m^2 of 14.1 MeV monoenergetic neutrons yielded 1596 appm helium, 440 appm hydrogen, 458 appm magnesium, 234 appm beryllium and 72 appm aluminum, as well as some less significant transmutants. Clearly, helium is the largest product and, because of its limited diffusivity in SiC [2-4], may cause significant swelling and/or stress in the material. This paper will give preliminary results of the effect of high levels of helium on the mechanical properties of CVD SiC.

Discussion

There are many possible mechanical property changes possible due to the fast-neutron transmutation of SiC. The production of the metallic impurities may increase creep as they will tend to migrate to grain boundaries and will, in most cases, be above their melting points. Also, the effect of material burn-up needs to be addressed, especially considering more than one percent of the atoms in the material have been displaced or transmuted at 1 MW-a/m^2, possibly leaving behind gas, vacancies or other fugitive elements in their lattice sites. Helium and hydrogen production (and corresponding production of Mg, Al and Be) is also dependent on the neutron energy spectrum. This is demonstrated in Figure 1, which gives the gas production as a function of the distance through the ARIES IV blanket [5]. Also included in this figure is the gas/DPA ratio in the blanket. From these data, it is seen that helium production is approximately 2000 appm at the first wall, (for 1 MW-a/m^2) and drops to less than 20 appm at the end of the blanket region. From inspection of the He/DPA curve of the same figure it is seen that the helium production is quite dependent on the neutron energy ranging from 130 appm/DPA near the first wall to about 30 appm/DPA at the end of the blanket region. Because of the neutron energy dependence of gas production it is useful to compare the helium production to DPA ratio of SiC with that of the metallic systems. Specifically, the He/DPA ratios for type 316 stainless steel is approximately 20 appm/DPA at the first wall and changes by less than a factor of two at the end of the blanket region [6].

There has been limited study of high levels of helium on the mechanical properties of SiC. In these studies [7-9] helium has been uniformly injected into a CVI SiC/Ceramic Grade Nicalon™ fiber composite material. A decrease in bend strength was observed in all cases. However, this degradation (~30-40%) is consistent with what would be expected given the displacment dose associated with the helium implantations.

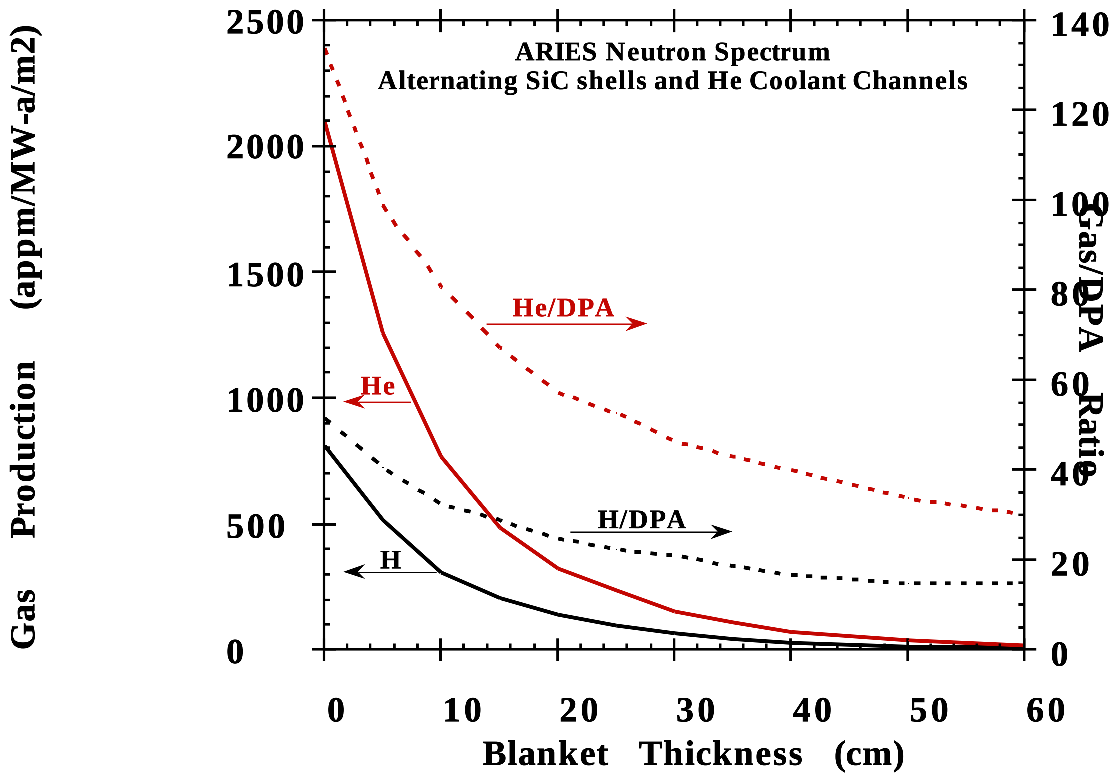

Figure 1: Effect of fusion neutrons on the gaseous production in SiC.

Experimental

Samples of Morton CVD SiC were machined into 1 x 1 x 25 mm bend bars and polished and chamfered on one surface. This middle section of this surface was then implanted uniformly over a depth of 0.46 mm with helium at 600°C using the cyclotron at JRC Ispra. The width of the implantation was 10 mm symmetric about the center of the sample. The helium implantation levels were 100 and 1000 appm. Samples were tested in four-point bending with a load and support span of 10 and 20 mm, respectively. The implanted side was placed in tension. Indentation hardness and toughness were measured using a Bueller Micro-Vickers hardness tester at a load of 500 g. Fracture toughness follows the Evans-Davis Model [10]. Density was measured using a density gradient column technique. Elastic modulus was measured using a Nanoindenter 2™ in the continuous stiffness mode.

Results

Figure 2 shows the Weibull failure probability for the non-irradiated, 100 appm and 1000 appm implanted SiC. It is important to note that the number of tests is limited, and therefore the standard deviation fairly high. However, from the data it is clear that the stress required to break the samples was increased with increasing helium implantation. It is worth noting, however, that due to the swelling of the helium implanted material (~0.5 %) the region to be tested was under compression prior to testing. The effect of this has not been analyzed. From the limited data, the effect of helium on the Weibull modulus can not be determined.

Weibull Analysis of He Implanted CVD SiC

	Mean ± St. Dev. (MPa)	Tests	Weibull Modulus	Size Parameter (MPa)
Non-Implanted	353 ± 72	29	13	368
100 appm	474 ± 127	10	7	508
1000 appm	532 ± 120	9	10	560

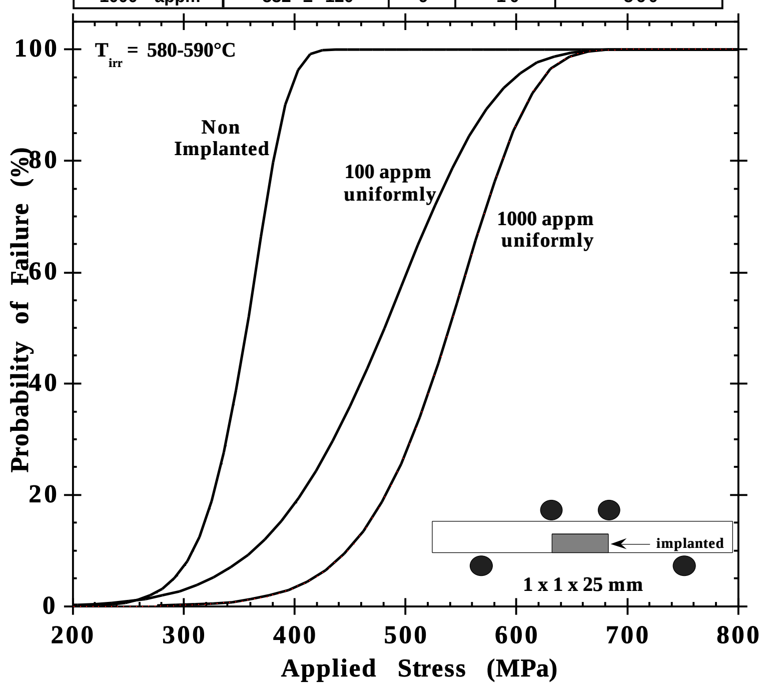

Figure 2: Bend Test resuts for non-implanted and He implanted CVD SiC.

From the data in Table 1 it is seen that there is a trend towards higher strength, higher hardness, and lower indent fracture toughness. The elastic modulus as measured by nanoindentation shows a slight decrease with irradiation.

Table 1: Mechanical Properties of non and He-implanted CVD SiC

Property	Virgin	100 appm	1000 appm
Swell (%)	**0**		**~0.5**
Bend Strength MPa	**353 ± 72**	**474 ± 127**	**532 ± 122**
Indent Fracture Toughness (MPa/m$^{1/2}$)	**1.4**	**1.2**	**0.9**
Vickers Hardness (Kg/m^2)	**2257 ± 103**	**2381 ±120**	**2516 ±180**
Elastic Modulus (GPa)	**527**	**515**	**490**

An interesting observation related to the toughness is the state of the surface following the Vicker's hardness measurement. It appears that the propagation of cracks and spalling of the surface is more pronounced in the as-implanted condition. An example of this can be seen in Figure 3, which shows a non-implanted and 1000 appm helium implanted example. In many cases, the spalling which is evident in the 1000 appm micrograph occurs. This sort of spalling was not seen for the non-implanted samples. Again, it should be noted that there was likely a compressive stress state in the implanted area due to the heterogeneous swelling which may cause or exacerbate spalling. Also of note is that non-implanted data was taken in non-implanted regions of the implanted samples, ruling out material non-uniformity or environmental factors which might cause property variation.

Future Work

The purpose of this work was to provide a baseline for identical materials, both implanted and non-implanted, which were irradiated in the 14J HFIR irradiation experiment. This irradiation provided ~8 dpa at 800°C, yielding a fusion-relevant He/dpa ratioup to 125. Following the post-irradiation examination data should be available on non-irradiated, neutron irradiated, as-implanted, and as-implanted and neutron irradiated specimens. This should allow a clean comparison of the effects of helium and helium + dpa on the mechanical properties of CVD SiC.

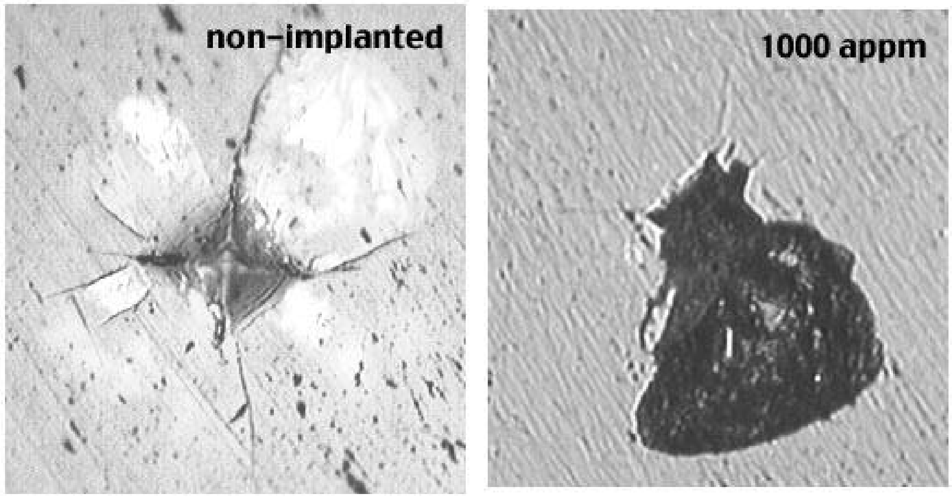

Figure 3: Optical images of Vicker's Indentations.

References

1. Rovner, L.H. and G.R. Hopkins, *Ceramic materials for fusion.* Nucl. Tech., 1976. **29**: p. 274-302.
2. Jung, P., *Diffusion and retention of helium in graphite and silicon carbide.* Journal of Nuclear Materials, 1992. **191-194**: p. 377-381.
3. Causey, R.A., J. Nucl. Mater., 1993. **203**: p. 196-205.
4. Sasaki, K., et al., *Helium release and microstructure of neutron-irradiated SiC ceramics.* Journal of Nuclear Materials, 1991. **179-181**: p. 407-410.
5. El-Guebaly, L., *Personally communicated data.*. 1995.
6. W M Stacey, J., *Fusion*. 1981: John Wiley and Sons.
7. Scholz, H.W., P. Fenici, and A.F. Rebelo. in *Miniturized Specimens for Testing of Irradited Material*. 1995. Juelich.
8. Rebelo, A.J.F., et al., *Comparison of the mechanical beaviour of SiC/SiC composites following neutron irradiation and helium implantation.* Journal of Nuclear Mateerials, 1998.
9. Hasegawa, A., et al., Journal of Nuclear Materials, 1998. **253**: p. 31.
10. Evans, A.G. *Fracture Toughness: The Role of Indentation Techniques*. in *Proceedings of the Eleventh National Symposium on Fracture Mecahnics Part II.* 1979: ASTM, Philadelphia.