Dimensional Isotropy of 6H and 3C SiC Under Neutron Irradiation

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ABSTRACT

This investigation experimentally determines the as-irradiated crystal axes dimensional change of the common polytypes of SiC considered for nuclear application. Single crystal α -SiC (6H), β -SiC (3C), CVD β -SiC, and single crystal Si have been neutron irradiated near 60°C from 2×10^{23} to 2×10^{26} n/m² ($E > 0.1$ MeV), or about 0.02 to 20 dpa, in order to study the effect of irradiation on bulk swelling and strain along independent crystalline axes. Single crystal, powder diffractometry and density measurement have been carried out. For all neutron doses where the samples remained crystalline all SiC materials demonstrated equivalent swelling behavior. Moreover the 6H-SiC expanded isotropically. The magnitude of the swelling followed a ~ 0.77 power law against dose consistent with a microstructure driven by single interstitial (carbon) mobility. Extraordinarily large $\sim 7.8\%$ volume expansion in SiC was observed prior to amorphization. Above $\sim 0.9 \times 10^{25}$ n/m² ($E > 0.1$ MeV) all SiC materials became amorphous with an identical swelling: a 11.7% volume expansion, lowering the density to 2.84g/cm³. The as-amorphized density was the same at the 2×10^{25} and 2×10^{26} n/m² ($E > 0.1$ MeV) dose levels.

1. Introduction

Irradiation-induced anisotropic swelling of the hexagonal close packed crystal of α -SiC-based ceramics has been postulated historically[1-3], and recently implicated in, the irradiation-induced degradation of SiC ceramics. The root of this speculation has been the observed difference in the irradiation-induced properties (including strength) of irradiation-stable pyrolytic SiC (a faulted form of cubic β SiC) and certain SiC ceramics processed with hexagonal α -SiC. Such anisotropy and resulting degradation in properties does occur in many ceramics possessing trigonal crystal structures such as alumina (sapphire)[4,5] and hexagonal structures such as silicon nitride,[6] aluminum nitride,[7] and beryllium oxide.[8] It is therefore reasonable that α -SiC and other ceramics with a hexagonal crystal structure may display irradiation-induced differential expansion on the $\langle a \rangle$ and $\langle c \rangle$ axes leading to microcracking and loss of ceramic physical integrity.

For the case of single crystal alumina a modest $\sim 0.1\%$ dilation in the $\langle a \rangle$ axis occurs upon irradiation at $\sim 400^\circ\text{C}$, negatively trending as irradiation temperature is increased. A more exaggerated dilation in the $\langle c \rangle$ axis occurs as a function of dose occurs for the same 400°C irradiation, resulting in just under 0.2% dilation, substantially increasing with irradiation temperature. This anisotropic expansion in the $\langle a \rangle$ and $\langle c \rangle$ direction has been implicated in the decreased mechanical strength that occurs by $1 \times 10^{26} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$) for alumina.[1] Similar, modest reductions in strength for polycrystalline alumina have been reported elsewhere in the literature.[4,5] For lower temperature irradiation (near 80°C),[9] swelling along the $\langle a \rangle$ and $\langle c \rangle$ axes of alumina are essentially identical until about $2.5 \times 10^{25} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$), at which point the $\langle c \rangle$ axis exhibits slightly greater swelling. The anisotropic swelling of BeO at similar irradiation temperatures occurs in a more exaggerated fashion beginning at a lower irradiation dose. While the $\langle a \rangle$ axis monotonically expands to approximately 0.1% by $1 \times 10^{25} \text{ n/m}^2$ ($E > 1 \text{ MeV}$) the $\langle c \rangle$ axis expands to approximately 3.5% over the same dose range. The result of this large anisotropic expansion for BeO is the near complete loss of mechanical strength by $1 \times 10^{24} \text{ n/m}^2$ ($E > 1 \text{ MeV}$).[8]

The literature on the irradiation effects of SiC includes a wide array of SiC ceramics including those fabricated from hexagonal (α) or cubic (β) phases. A detailed discussion of the effects of irradiation on SiC properties can be found elsewhere.[10] Generally, the irradiation-stability of the β -phase SiC is founded on a relatively solid body of work on chemically vapor deposited (CVD) SiC, a highly-faulted form of pure β -SiC which can be of high purity. The stability of CVD SiC is generally contrasted with the instability of α and β SiC ceramics formed through conventional processing methods, which demonstrate rapid irradiation-induced degradation of engineering properties. For example, the initial 1972 work of Matthews and Hutchings[11] discusses the behavior of reaction-bonded α and β -SiC-derived ceramics irradiated to a few tenths of a dpa at 400 and 700°C , observing significant

strength loss. In that paper the ceramic degradation was attributed to differential swelling of the α -SiC and β -SiC as well as the Si phase present at the grain boundaries. In follow-on work Matthews[12] studied irradiated α -SiC powder, β -SiC powder, Si powders and their respective sintered ceramics finding little difference between the lattice-dilation of the two SiC polytypes at irradiation temperature above 400°C and up to a few dpa. Neutron irradiation studies carried out by Matheney[2] , Iseki[13] , Price [14] , Dienst[15] and Correlli[16] all report significant strength decrease in conventionally processed α -SiC ceramics and with the general consensus of internal strain and lack of grain boundary cohesion as the driving force behind degradation. Li and coworkers [17] have carried out Si ion irradiation at 1000°C of cubic 3-C SiC, reporting anisotropic crystal swelling, though attributed this anisotropy to a Si-substrate effect.

In one study on very low dose H implantation into β -SiC films an initial contraction along the $\langle a \rangle$ axis was observed by Jiang et al.[18] Constantini [19] and Kerbiroui[20] provide a comparison of the accumulation of lattice disorder and resulting mechanical properties in Cree single crystal α -6H-SiC and β -3C-SiC epitaxial layers indicating similar layer expansion. Pramono et al[21] studied the effect of implanted helium on the swelling behavior of irradiated α -6H-SiC ceramics, providing data on the dilation of the $\langle a \rangle$ and $\langle c \rangle$ axes, showing them to be essentially equivalent for irradiation at 300°C to 0.28×10^{25} ($E > 0.1$ MeV.)

By comparing the available ion irradiation data with neutron irradiation data such as that of Matthews[12] and Price[22] it could be reasonably argued that no differential swelling exists between the most common α -6H and β -3C SiC polytypes. Moreover, it could be similarly put forward that no large difference exists in irradiation-dilation between the major axes of the 6H alpha polytype of SiC. This lack of an anisotropic swelling response to irradiation, in contrast to the hexagonal crystals above, could have a number of explanations including the lack of mobility for both Si and C vacancies as well as the Si interstitial at these irradiation temperature. Regardless, the absolute comparison of the relative swelling difference under neutron irradiation, if any, between cubic and hexagonal forms of SiC has not been directly demonstrated. As this question is of current practical interest to the community this work has been carried out to provide such a direct comparison.

2. Experimental

Materials selected for this comparative study were single crystal Hoya β -3C SiC, single crystal Cree Systems α -6H SiC, polycrystalline Rohm-Haas CVD SiC (a highly faulted β -SiC), and single crystal Si. All materials were highly pure, electronic-grade materials. Irradiation was carried out in the hydraulic tube or peripheral target tube position of the High Flux Isotope Reactor (HFIR) within a perforated “rabbit” capsule. The rabbit capsule consists of an aluminum tube (the rabbit) which has

large holes drilled through the wall to allow HFIR coolant water to flow through and contact its contents. The HFIR coolant water enters the core at 49°C and exits at 69°C. Samples were located mid-core such that the exterior of the samples should be just above 60°C. Samples were irradiated in a fast neutron dose range from 2×10^{23} to 2×10^{26} n/m² ($E > 0.1$ MeV) at a fast neutron flux of approximately 1×10^{19} n/m²s corresponding to approximately 10^{-6} dpa/s. For this paper an equivalence of 1×10^{25} n/m² ($E > 0.1$ MeV) = 1 displacement per atom (dpa) is assumed. Square samples of approximately 6 mm on edge were used. The CVD SiC materials were nominally 2.8 mm thick while the α -SiC, β -SiC and Si samples were cut from wafers of 0.2 mm, 0.1 mm, and 0.5 mm thickness, respectively.

Following neutron irradiation the samples were removed from the rabbit container and ultrasonically cleaned first with ethyl alcohol and then immersed in hydrofluoric acid for approximately 30 minutes to remove any glassy surface layer that may have been present. Prior to x-ray examination samples were wrapped in kapton for radioactive contamination control. Lattice parameters of single crystals were measured using x-ray diffraction. X rays were generated using a Rigaku R200 generator with a Cu target operating at 45 kV and 100 mA. A Ge (220) crystal monochromator with slits selected for Cu K $_{\alpha 1}$ incident beam. The sample was mounted in reflection geometry on a 4-circle diffractometer; the angle of diffracted x-rays was determined using a Ge (220) crystal analyzer. X rays were counted using a NaI(Tl) scintillation detector. Instrumental resolution is 0.005° in 2θ . 2θ scans were taken for a series of Bragg reflections: (111), (002), (202), (113), (311), and (004) for SiC(3C) and Si; (0 0 0 6), (0 0 0 12), (1 0 -1 2), (1 0 -1 5), (1 0 -1 7), (1 0 -1 10), (1 1 -2 6), and (2 0 -2 4) for SiC(6H). The center of each Bragg reflection was found by least-squares fitting to a Gaussian profile. The lattice parameters were determined using a least-squares refinement of angles calculated using Bragg's law to measured Bragg angles. Swelling magnitude, determined based on X ray data, was quantified using the changes in these lattice parameters (5 reflections for the cubic materials and 7–9 reflections for the hexagonal materials) as a function of dose.

The microstructure of certain specimens were observed using transmission electron microscopy (field emission type JEM2100F at 200kV). TEM foils were prepared using a focused ion beam system (FEI Versa 3D Dual Beam) operated at 30 kV for initial milling and at 2-5 kV for final thinning.

Physical dimensions were measured before and after irradiation using calibrated micrometers. However, a density gradient column (DGC) was utilized to determine (and report) immersion bulk density with calibrated floats and mixtures of methylene iodide and tetrabromomethane with much greater accuracy. It is noted that density calculated from physical dimensions and dry weight were in agreement. A representative column section is provided in Figure 1. The denser, non-irradiated SiC samples are provided in the left of the figure. As observed, the non-irradiated α -6H, β -3C and CVD faulted β exhibit a very close grouping as did the irradiation-darkened 0.5 dpa samples of the right figure. Measurements were repeatable

through multiple columns. A typical density gradient as shown for the column of figure 1 was 0.002 (g/cm³) per centimeter of column length. Error associated with the density measurement, assuming no sample surface contamination, for such a column is less than 0.001 g/cm³ or < 0.03%.

Figure 1 : Density gradient column with calibrated floats and groupings of non-irradiated and 0.5 dpa irradiated SiC samples.

3. Results and Discussion

The dilation of the $\langle a \rangle$ and $\langle c \rangle$ axes of α -6H-SiC as compared to non-irradiated is shown in Figure 2. From the figure a monotonic expansion occurs along both axes to a dose level of 0.5×10^{25} n/m² ($E > 0.1$ MeV). Above each irradiated sample data point within the figure the percent expansion is provided. By comparing the $\langle a \rangle$ and $\langle c \rangle$ axes percent expansion and noting that the differences are within the combined x-ray technique and computational error it is clear that the irradiation-induced lattice dilation for each orientation of the α -6H-SiC is identical.

Figure 2: Irradiation-induced dilation of the <a> and <c> axis of α -6H-SiC.

As understood from a large body of previous work[10] the initial irradiation-induced swelling of SiC for irradiation temperatures less than $\sim 1000^\circ\text{C}$ increases per a power law and then approaches a saturation level by a few dpa ($\sim 2 \times 10^{25} \text{ n/m}^2$ $E > 0.1 \text{ MeV}$). This relative volume change is highly dependent on temperature, exhibiting enhanced swelling at lower temperature irradiation due to a larger fraction of residual interstitial complexes remaining following neutron-induced cascade events at the lower temperatures.[23] Data on the effect of irradiation on bulk swelling of CVD faulted β -SiC as measured by DGC is provided in Figure 3 as solid square data points. For the irradiation temperature of this study, $\sim 60^\circ\text{C}$, the exaggerated swelling at lower temperature is clearly observed. For the 0.02, 0.1 and 0.5 dpa doses, the single crystal α -6H and β -3C SiC bulk swelling was essentially identical to that of the faulted- β CVD SiC (depicted in Figure 1, not plotted in Figure 3 for the sake of clarity.) The power-law rate constant of swelling with dose, approximately 0.77, agrees well with 0.81 found in the previous work of Katoh et al.[24] for Ni-ion irradiation (at $\sim 100^\circ\text{C}$.) For this temperature range it is assumed that there is no vacancy mobility on either the silicon or carbon sublattice and that only the carbon interstitial is mobile. Under these conditions Katoh has previously discussed the power law rate constant for swelling accumulation[25] as being either $2/3$ for the formation of two-dimensional defects or $3/5$ for the formation of three-dimensional defect agglomerates. In the more limiting case where the irradiation temperature is reduced and the carbon interstitial also become immobile the rate constant would approach unity.

Swelling data calculated from the single crystal diffractometry measurements is provided next to the first three dose data points of Fig 3. As can be seen from the figure the irradiation-induced volume change measured through bulk (DGC) means or by x-ray density are essentially identical and the equivalence of the α -6H and β -3X SiC swelling is also observed through comparison of the X-ray data.

Figure 3: Irradiation-induced swelling of faulted β CVD SiC inset with effect of dose rate on threshold dose for Si ion irradiation on α -6H SiC[26] .

Also of note from Fig. 3 is the significant swelling that occurs in SiC (7.8%) at 0.5 dpa. Though these samples undergo impressive irradiation-induced lattice strain, X-ray and TEM analysis indicates that the remains crystalline at that dose. Figure 4 provides a high-resolution TEM image of the 0.5 dpa irradiated single crystal 6H-SiC. Close inspection of the micrograph does suggest the formation of discontinuous pockets of highly damaged areas that may evolve into amorphous regions. Moreover, the diffraction profiles for the 0.5 dpa α -6H-SiC suggests a level of complexity in the α -6H microstructure. While the (0 0 0 6), (1 1 -2 6), and (1 0 1 -5) reflections are reasonable well fit by a Gaussian profile, the (0 0 0 12), (1 0 -1 2), (1 0 -1 7), and (1 0 -1 10) reflections have a double-peak profile, with a splitting $\Delta d/d \sim 4 \times 10^{-4}$. This is shown in Figure 5, in particular for the (1 0 -1 10) and (1 0 -1 7) reflections, giving the progression from non-irradiated, 0.1 dpa, to 0.5 dpa, clearly

showing a peak splitting at the 0.5 dpa irradiation damage level. Lattice parameters were determined by fitting line-shapes to a profile with two Gaussian peaks and including both peaks in the lattice parameter refinement, giving some sort of average lattice parameter. Peak splitting in this sample is accompanied by lower intensities: the (2 0 -2 4) reflection was too weak to be measured. Line-shapes for all other samples are reasonably fit by a single Gaussian. It is currently unclear what defects give rise to this splitting. However, it has been previously noted[19] [27] in heavy ion irradiation studies that a two-stage progression occurs in the amorphization regime for SiC whereby the swelling progresses from point-defect formation (Stage 1) into an amorphization regime (Stage 2) at a dose slightly lower than the 0.5 dpa where the Bragg-peak splitting was observed in this study. Unfortunately the gap between the 0.1 and 0.5 dpa was too large to directly observe or confirm this previous observation.

Figure 4 : High resolution TEM of high-swelling 0.5 dpa 6H-SiC .

Figure 5 : Diffraction peaks for non-irradiated, 0.1, and 0.5 dpa materials indicating progression toward peak splitting for the (1 0 -1 10) and (1 0 -1 7) reflections.

The measured 7.8% swelling for the neutron irradiated SiC of figure 3, while a remarkably high strain for the crystal to manage, and to the authors knowledge the highest neutron-induced swelling of SiC reported, has been previously reported for ion irradiated SiC. Katoh et al.[24] carried out a series of 4 MeV Ni ion and Ni-He dual ion implantation experiments on β -3C SiC to determine the effects of He on swelling. In that work, carried out at approximately the same temperature as the present work, the material remained crystalline up to approximately 1.07 dpa, which was defined as the threshold amorphization temperature above which the materials made the amorphous transition with a resulting $\sim 12.8\%$ swelling. In that work the maximum reported swelling prior to amorphization (at 1.07 dpa) was approximated as 7%, consistent with the current work.

In contrast with the swelling of the SiC, the Si samples of the present study showed essentially no change for the irradiation conditions studies. The Si sample irradiated to a dose of 20 dpa yielded a bulk density through DGC nearly identical to non-irradiated while single crystal diffractometry for the sample irradiated to 2 dpa yields an $\langle a \rangle$ axis changing from 5.43115 to 5.43126 Angstrom, or an approximate 0.002% expansion. Such a modest expansion is not surprising with previous authors reporting on essentially no change for a 380-700°C range irradiation[28] [12] in the dose range of the current study.

Also seen in Figure 3, at some point above the 0.5 dpa dose level a transition from crystalline to amorphous SiC occurs. At the 2 dpa dose level all three SiC materials indicate complete amorphization, as determined through x-ray diffraction. Moreover, within experimental error the density for all SiC materials studied have equivalent density as measured by DGC for the 2 and 20 dpa dose levels, or $\sim 11.7\%$ volume expansion (2.84 g/cm^3): for a dose of greater than 10x the threshold amorphization dose no change in density occurs. It is noted that for previous work where neutron-amorphized SiC was annealed slightly above the irradiation temperature significant changes in a range of properties including density occurred[29,30], indicating that amorphous SiC is a continuum of states depending on the variables by which amorphization is achieved. However, steady state density and by inference its short-range order appears to establish itself soon after amorphization occurs.

Amorphization of SiC has been widely studied and it is now understood that the dose required for the crystal-to-amorphous transition has a relatively strong dependence on temperature and a weaker dependence on irradiation spectrum, starting microstructure (whether α or β initial crystal or impact of grain structure,) and chemical purity. The temperature dependence for amorphization is due to the onset of species mobility such as the carbon and silicon interstitials as indicated by a number of subtle dose inclines prior to onset of a rapid, asymptotic increase in dose with temperature. The minimum temperature-dependent dose at which amorphization occurs is referred to as the critical dose, having a minimum of ~ 0.2 dpa near liquid nitrogen temperature. The effect of temperature on critical dose has been studied for a number of irradiating species including 2 MeV electrons,[31] [32] [33] 360 keV argon [34] or 1.5 MeV xenon ions[35,36] [34,37,38] and 0.56 MeV silicon-ions[39] [40] [41]. The temperature dependence for critical dose for these previous studies were somewhat different, depending primarily on the ion, being in the range of 20°C and 70°C for electrons[31,32,42], $\sim 150^\circ\text{C}$ for Si ions [40] [33] and $\sim 220^\circ\text{C}$ for Xe ions.[36] The displacement damage rates for all of those studies were similar, on the order of $\sim 1 \times 10^{-3}$ dpa/s or about three orders of magnitude greater than the current neutron irradiation. It is noted that both Weber [35,36] and Matsunaga's[43] work did not observe a significant difference in amorphization threshold between α - and β -SiC, whereas Inui did report a threshold temperature $\sim 50^\circ\text{C}$ higher for faulted β -SiC.[31]

The neutron-amorphization of SiC and associated properties was first described by Snead et. al. [29,44,45]. As with the current work, materials were irradiated in contact with coolant water, though through inspection of large diameter specimens a critical amorphization temperature of $\sim 125^\circ\text{C}$ was inferred,[46] in reasonable agreement with the Si ion results. [40] [33] This would be expected given the relative similarity of the sub-cascades produced by both fast neutrons and 2 MeV Si ions. However, in comparing the Si-ion beams and neutron irradiation experiments the effect of dose rate must also be considered. Inset into Fig. 3 is included a data-set from work of Snead et al[26] who investigated the effect of damage rate on

critical amorphization dose for Cree α -6H-SiC using 2 MeV Si ions implanted at 68°C (similar to the irradiation temperature of this study.) Of interest from the inset is the extrapolation of the dose dependent critical dose from that of the Si ion experiments to that of the current neutron irradiation experiments. With decreasing dose rate the critical dose is seen to increase linearly on the log dose plot, excepting the highest dose rate point that appears anomalous and was attributed to beam heating at the extremely high ion flux.[26] By extrapolating the previous Si-ion data back to the dose rate appropriate for the current neutron irradiation experiment ($\sim 1 \times 10^{-6}$ dpa/s) a critical threshold for amorphization of ~ 0.9 dpa is suggested. It is cautioned that whenever comparing neutron and ion irradiation data comparison using dpa must be considered approximate for a number of reasons including those associated with the use of SRIM as discussed recently by Stoller. [47] Further investigation to more precisely define the amorphization transition and to better define the flux dependence is an area for further research.

4. Conclusion

As demonstrated clearly in this work the irradiation-induced lattice strain for the most common crystal structures of interest for nuclear application, α -6H (alpha) and β -3C (beta) silicon carbide, have near identical irradiation swelling behavior near 60°C. Single crystal alpha and beta SiC swelling was also equivalent to that of highly pure CVD SiC (a highly-faulted polycrystalline form of β -SiC.) Results of single crystal X-ray diffraction were confirmed by precise bulk density measurements of all materials. Moreover, the swelling along the $\langle a \rangle$ and $\langle c \rangle$ axis of SiC polytypes demonstrates the isotropic swelling behavior of both alpha-6H and beta-3C SiC. Parallel irradiation of single crystal Si to 20 dpa indicated essentially no change in lattice spacing or bulk swelling.

At a level of 0.5 dpa all SiC materials underwent an extraordinarily large swelling ($>7.8\%$) while remaining crystalline. At some dose beyond this point, assumed to be ~ 0.9 dpa, the SiC crystal transitions to an amorphous structure with a resulting 11.7% (2.84 g/cm^3) swelling. Alpha-6H, beta-3C, and polycrystalline CVD SiC all resulted in a similar density upon amorphization with a similar density measured at the 2.0 and 20 dpa, suggesting that the short-range order of SiC saturates soon after amorphization occurs.

The overall findings of this study, combined with understanding that can be gleaned from the literature, suggests that the common SiC polytypes and the ceramics derived from them are similar in the irradiation stability and for the purposes of engineering ceramics should be treated as equivalent for low temperature irradiation.

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