Radiolytic Hydrogen Generation at Silicon Carbide – Water Interfaces

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Abstract

While many of the proposed uses of SiC in the nuclear industry involve systems that are assumed to be dry, almost all materials have dissociated chemisorbed water associated with their surface, which can undergo chemistry in radiation fields. Silicon carbide α -phase and β -phase nanoparticles with water were irradiated with γ -rays and 5 MeV ^4He ions followed by the determination of the production of molecular hydrogen, H_2 , and characterization of changes in the particle surface. The yields of H_2 from SiC – water slurries were always greater than expected from a simple mixture rule indicating that the presence of SiC was influencing the production of H_2 from water, probably through an energy transfer from the solid to liquid phase. Although the increase in H_2 yields was modest, a decrease in the water mass percentage led to an increase in H_2 yields, especially for very low amounts of water. Surface analysis techniques included diffuse reflectance infrared Fourier transform spectroscopy (DRIFT), nitrogen absorption with the Brunauer – Emmett – Teller (BET) methodology for surface area determination, X-ray diffraction (XRD), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS). Little change in the SiC surface was observed following radiolysis except for some conversion of β -phase SiC to the α -phase and the formation of SiO_2 with He ion radiolysis.

1. Introduction

Silicon carbide (SiC) has been suggested for many different applications in the nuclear industry, including use as the clad for new nuclear fuels for light water reactors [1], incorporation into the matrix of Gen III+ fuel materials [2], and the encapsulation of high level waste for long term storage [3]. This popularity is due to its high temperature stability [4], good thermal conductivity [5, 6], and oxidation resistance at high temperatures [7]. SiC exists as 3 main polytypes, 6H-SiC (SiC α -phase), 3C-SiC (SiC β -phase) and 4H-SiC. Much work has been done on examination of SiC properties in the context of its use as a nuclear material [8, 9]. There have been reports on α -phase and β -phase SiC polytypes for nuclear industry applications, including their behavior under irradiation [10] and transformation between types [4], however, the study of SiC has very much remained in the domain of material science and little work has been published on its radiolysis in realistic environments [1].

In the absence of a long term disposition solution, the nuclear industry is facing extended periods of interim storage for used fuel and consequently dry storage following cooling in a water pond is being investigated and under serious consideration for adoption. Many of the proposed uses of SiC in the nuclear industry involve systems that are relatively dry [1]. However, almost all materials have dissociated chemisorbed water associated with their surface [11]. In addition, accidents and other mishaps can lead to the introduction of water onto nuclear materials. Waste storage containers may be designed to be dry, but the probability of such containers coming into contact with ground water at some point in the future is not negligible.

Several studies have shown that the radiolysis of water in contact with solid interfaces has very different radiation chemistry than observed in bulk water [12-17]. The transfer of energy, charge, or mass through the solid – water interface may lead to significant modifications to the

surface as well as enhanced degradation of the water. Systematic studies on the radiolysis of SiC – water interfaces will aid future nuclear engineering and used fuel management endeavors.

This work investigated the radiolysis of water in association with the surface of SiC nanoparticles to determine how each component affected radiolytic changes in the other. Adsorbed water on SiC and SiC – water slurries were examined in order to obtain the relative dependence of radiolytic yields on the amount of each phase. Water radiolysis was monitored by observing the variation in the yield of molecular hydrogen, H₂. The surfaces of SiC were characterized before and after radiolysis using diffuse reflectance infrared Fourier transform spectroscopy (DRIFT), nitrogen adsorption using the Brunauer – Emmett – Teller (BET) methodology for surface area determination, X-ray diffraction (XRD), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS). Irradiations were performed with γ -rays and accelerated 5 MeV ⁴He ions to mimic the radiation typically associated with nuclear materials.

2. Experimental

2.1 Sample Preparation and Analysis

Silicon carbide, α -phase SiC (99.8%) and silicon carbide, β -phase SiC (99.8%) were purchased from Alfa Aesar and used as received. The density of both polytypes is 3.21 g/cm³. The Brunauer-Emmett-Teller (BET) methodology was used to determine the surface area and estimated particle size of the powders using a Quantachrome Autosorb-1. Water and other contaminants present on the surfaces were analyzed using two complimentary techniques: temperature programmed desorption (TPD) and temperature dependent diffuse reflectance infrared Fourier transform spectroscopy (DRIFT). The TPD measurement was performed on a sample of between 50 - 100 mg deposited in a custom cell. The cell was heated from room temperature to 500°C at a rate of 2°C/min, while the gases desorbing from the sample were

monitored using a Pfeiffer Prisma quadrupole mass spectrometer. The mass to charge ratios measured were 14, 15, 16, 18, 28, 32, 40, and 44. A background scan was taken using an empty cell and was subtracted from the sample measurement. The DRIFT was obtained using *in-situ* measurements of samples heated up to 400°C using a Bruker Vertex 70 with a Harrick Praying Mantis high temperature cell. Prior to DRIFT measurements samples of SiC were mixed with KBr. Two mixtures were measured for each phase, one with 10% and one with 1% SiC by mass. Mixtures of KBr had to be used because the pristine samples absorbed too much light.

Surface analysis was performed using Raman spectroscopy and X-ray photoelectron spectroscopy (XPS). The Raman spectroscopy measurements were taken using a Jasco Micro-Raman Spectrometer MRS-5100. In this configuration, the beam diameter on the sample is estimated to be around 1 μm with a 10 μm sampling depth. A 532 nm laser was used for the measurements with a power of 0.2-0.3 mW and resolution of 3.53 cm^{-1} . XPS measurements were taken using a PHI VersaProbe II X-ray photoelectron spectrometer with a monochromatic Al-K α X-ray source, photon energy of 1486.6 eV, and a hemispherical electron energy analyzer. Wide scans over the binding energy range from 1486 – 0 eV were taken with a pass energy of 187.85 eV and an energy step of 0.4 eV. High resolution scans were taken for each element of interest with a pass energy of 23.5 eV and energy steps of 0.05 eV. Multiple 200 μm diameter spots were analyzed on each sample and the sampling depth is estimated to be less than 10 nm. Charge neutrality was maintained on the surface by flooding the sample chamber with 10 eV Ar²⁺ ions and low-energy electrons during measurement. Under these conditions, the resolution for the wide range scans was 2.43 eV and was 0.58 eV for the elemental regions scans as determined using the full width at half maximum, FWHM, for the Ag 3d_{5/2} peak from a sputtered silver foil.

X-ray diffraction (XRD) measurements were taken using a Bruker D8 Advance DAVINCI Powder X-Ray Diffractometer equipped with a Cu-K α X-ray source. Scans were taken over a range of two-theta values from 20° to 80°, with a 0.05° step. To prevent radiation damage to the surface during the measurement, samples were rotated at a rate of 15 revolutions per minute.

2.2 H₂ Production and Gamma Irradiation

Silicon carbide does not adsorb water easily onto its surface and no H₂ could be observed under normal irradiation conditions. Therefore these carbides were not baked to remove adsorbed water and slurries were investigated instead. The amount of water was varied as a function of the mass from 10 to 97.5%. Water was obtained from an Elga Water system, and mixed with 1 mM KBr. The addition of KBr is used to scavenge •OH radicals that are produced during the radiolysis of water, which would otherwise react with H₂, resulting in a lower yield.

Radiolysis with γ -rays was performed using a self-contained Foss Therapy Services Inc. 812 ⁶⁰Co source located within The University of Manchester Dalton Cumbrian Facility. Temperatures inside the source reach 35 °C so little macroscopic heating was observed. The dose rate was nominally about 107 Gy/min as determined using the Fricke dosimeter. This dosimeter is strictly valid for only water or aqueous solutions; however, both the elements Si and C have relatively low atomic numbers and the absorption of gamma rays is expected to be very similar to that of the O in water and no further correction was made to the dosimetry. The sample cells were 10 cm long and 10 mm diameter Pyrex tubes. Samples were prepared by purging the SiC powders under argon to positive pressure and then flame sealing the tubes. In order to ensure homogeneous mixing of the slurries, samples were irradiated at a constant distance from the source while turning at 3 rotations/min, which was sufficient to ensure complete mixing.

The production of H₂ from empty glass vials after purging was lower than the detection limit when irradiated for times comparable with those of the samples. The yield of H₂ with aqueous KBr solutions using this methodology gives a value of 0.46 molecules/100 eV, which agrees well with previous results [18]. The SiC powder irradiated without water (purged with argon) gave no detectable H₂, which indicates clean surfaces on the powders.

Samples were irradiated up to 12.5 kGy with the H₂ produced measured using a SRI 8610C gas chromatograph coupled with a thermal conductivity detector (TCD) [12]. Carrier gas, 99.999% argon, was passed through a constant flow regulator, an injection septum, a four-way valve connected to a sample injection loop, and into a column consisting of 13× molecular sieves. Following irradiation, the sample cells were inserted into Tygon tubing, connected in-line to the GC, flushed with the argon carrier gas, and then cracked open allowing any gases produced during the irradiation to be swept away and detected by the GC. Calibration was performed by injecting pure gases with a gas-tight microliter syringe. The sensitivity for detection of H₂ was 1 μL and all errors associated with H₂ determination are about 5%.

2.3 Irradiations with γ -rays and He Ion for Surface Analysis

Irradiations of SiC with γ -rays to produce samples for surface analysis were performed using a self-contained Shepard ⁶⁰Co source located at the University of Notre Dame Radiation Laboratory, which had a nominal dose rate of 179 Gy/min as determined using Fricke dosimetry. Sample cells consisted of 10 mm diameter Pyrex tubes about 10 cm long that were flame sealed following freezing, pumping with vacuum and thawing 3 times, to remove any air from the samples. Following irradiation, the sample vials were cracked open and the samples were deposited on SEM stubs for surface analysis.

Heavy ion irradiations were performed with 5 MeV ^4He ions obtained using the facilities of the Nuclear Science Laboratory at the University of Notre Dame. The beam diameter was approximately 6.4 mm, with a beam current of 1.5 nA charge. Energy loss to the machine exit window was determined using standard range tables and fully stripped ions were used for irradiations [19]. Samples for He ion irradiation were prepared by pressing SiC powders into a carbon tab attached to an SEM stub and were irradiated to 1×10^{15} ions/cm². The SEM stub and exit window were connected electrically, and current from them was integrated to give the ion fluence.

3. Results and discussion

3.1 Characterization of SiC Surfaces

Water adsorbed on the surface of solid oxides typically dissociates to form OH groups [11, 16]. The characterization of the surface of the SiC powders was undertaken to determine how water adsorption occurs on SiC. The specific surface area for α -phase SiC was 10.3 ± 0.6 m²/g and for β -phase was 16.3 ± 0.1 m²/g using the BET methodology. Multiple scans were used to determine the standard deviations. From these surface areas, the average particle diameters were estimated as 183 and 115 nm for the α -phase and β -phase SiC powders, respectively, assuming spherical particles. An isotherm for each phase is shown in Figure 1. The shape of the isotherms as well as the lack of hysteresis between the adsorption/desorption curves suggest that the particles are either macroporous or nonporous.

TPD and DRIFT measurements were made to analyze the water and other contaminants present on the SiC surface. The TPD measurements for water are shown in Figure 2. Curves for the other gases monitored were orders of magnitude less than that for the desorption of water. The desorption curves are broad but three peaks at 135°C, 261°C, and 480°C can be fit for α -phase SiC, and two peaks at 155°C and 384°C can be fit for β -phase samples. The desorption energies

calculated using Redhead's method are 1.3, 1.7, and 2.4 eV for the α -phase samples and 1.3 and 2.1 eV for the β -phase samples [20]. This result suggests that there are multiple bonding sites available on the SiC surfaces for chemisorbed water, which has a desorption energy greater than 0.35 eV [21]. DRIFT spectra for each sample were measured at room temperature; following heating to 100°C, 200°C, 300°C, and 400°C; and again after cooling to room temperature giving the results shown Figure 3. For comparison, a reference curve of water ice at 10K is also displayed [22]. Neither SiC phase shows peaks indicating the presence of surface water at room temperature. However, following heating a broad inverse reflection peak around 3475 cm^{-1} is present, suggesting that water is being lost from the surface. The water peak at room temperature is not visible due to the high amount of KBr present, which was required because pure SiC absorbs almost completely in the infrared. The peak at 3475 cm^{-1} disappears when the sample is cooled to room temperature, indicating that the water is reabsorbed on the surface. This water must be the chemisorbed water observed in the TPD spectra that desorbs at the higher temperatures. The α -phase SiC sample has a main peak at 870 cm^{-1} from the Si-H bond, with a shoulder at 790 cm^{-1} from the Si-C bond and at 950 cm^{-1} . Other peaks are also visible at 1300, 1540, and 1620 cm^{-1} . Previous work on SiC films have identified vibrations from the Si-C bond [23-29] at 780 cm^{-1} , the Si-H bond [23, 25] at 860 cm^{-1} , the Si-CH₃ bond [25, 29] at 980 cm^{-1} , the Si-O bond [28] at 1020 cm^{-1} , and from work on silicon nitride films, the Si-O bond [26, 30] at 1040 - 1060 cm^{-1} . Additional modes which were reported in the previous works [23-28] were not observed in these samples. The β -phase SiC sample had a main peak at 920 cm^{-1} , with smaller peaks at 760 and 620 cm^{-1} . A previous study [31] of β -phase SiC nanowires identifies the transverse optic mode of the Si-C bond at 792 cm^{-1} with a longitudinal optic mode shoulder at 954 cm^{-1} . The location and sizes of the

peaks are not observed to change during the heating experiments, suggesting that except for the loss of water the SiC surfaces are stable during heating.

3.2 Production of H_2 with γ -ray Radiolysis

The most obvious probe of the variation in the radiolysis of water in the presence of SiC is the production of H_2 . This gaseous product is relatively unreactive and easy to measure in mixed-phase systems. Systems of physisorbed water would not give observable amounts of H_2 so slurries with various amounts of water were used. The yields for H_2 with 70% water by mass are shown in Figure 4 as a function of the dose. These results show that the yields of H_2 are not dependent on the total dose to the system. Variation of the dose rate also did not affect the production of H_2 . The presence of the solid SiC interface does not lead to any spontaneous or catalytic water decomposition.

Variation in the relative amount of SiC and water gives information on the interactions occurring at the interface in radiolysis. The yields of H_2 are shown in Figure 5 as a function of the water mass percentage in the system. Each experiment was performed four times and the error bars represent one standard deviation. Errors in H_2 determination are sometimes more dependent on sample reproducibility than in H_2 measurement. Fewer data were collected for the β -phase since this system was not expected to behave much differently than the α -phase. Yields can be reported with respect to the total energy deposited in the whole system (water and SiC) as in the upper plot or with respect to the water alone, as in the lower plot. In all cases, the yields of H_2 with SiC present are higher than would be expected for pure water. This result implies either a transfer of energy from the carbide into the water for a given sample or modification of the sensitivity of adsorbed water to radiation. Although the yields of H_2 are small, they are comparable to recent studies using iron [14] and copper oxide [15] slurries with water. These small yields suggest that

the carbide surfaces are relatively inefficient at transferring “radiation damage” to the water. Similar results were observed with iron oxides [14]. Very little is known about the mechanism for the transfer of energy through the bulk and interface, but previous studies with ZrO_2 suggested that it is due to an exciton. [13] Calculations seem to indicate that exciton energies in SiC are only a few eV, which would be too small to break a typical water bond. [32]

Observations of the H_2 yields with respect to the energy deposited only in the water give a better representation of the effect of SiC on the water radiolysis. The yields of H_2 with α -phase SiC increases from 0.46 molecules/100 eV for pure water to about 0.87 molecules/100 eV at 60% water and then remains constant until 10% water. This result suggests that there is a relatively constant amount of energy being transferred from the SiC to the water phase and the yield appears to increase because the amount of energy directly deposited in the water is decreasing. However, there appears to be a rather large increase in H_2 yields as the amount of water decreases below 10%. This lower limit is quite reproducible. Experimental difficulties precluded the extension of the data to much lower water content, but the trend in the data with decreasing water content suggests that a high yield of H_2 is expected from SiC with a few associated water layers.

3.3 Changes to the SiC Surface

Powder X-ray diffraction, Raman spectroscopy, and X-ray photoelectron spectroscopy were used to analyze the SiC powder surfaces and bulk before and after irradiation with He ions and γ -rays. The XRD patterns (not shown) confirmed the correct diffraction patterns for α -phase and β -phase SiC following comparison with the particle diffraction file database [33] as well as with previously reported diffraction patterns for α -phase SiC [31, 34, 35] and β -phase SiC [36, 37]. No significant changes in the diffraction patterns were observed following irradiation, indicating that the bulk crystalline structure is unchanged at the doses used in this work.

Samples of SiC were also characterized using Raman spectroscopy and the spectra are shown in Figure 6. The pristine α -phase sample has peaks at 154, 244, 769, 791, 950, 972, 1515 cm^{-1} , while the β -phase pristine sample has peaks at 793, 1354, 1513, 1586, and 1615 cm^{-1} . The peaks at 1354 and 1586 cm^{-1} are due to graphitic or amorphous carbon present on the β -phase SiC sample [38, 39]. These peaks are in good agreement with previously reported Raman spectra of α -phase [29, 39-41] and β -phase [31, 39, 40, 42, 43] SiC samples [26, 44]. No peak around 480 cm^{-1} , due to the presence of the Si-Si bond [26] was observed for either sample. Following irradiation with γ -rays and He ions, no changes are observed in the α -phase SiC sample. For the β -phase sample, the γ -ray irradiated sample shows an increase in the relative intensity of the graphitic carbon peaks and one of the sampled locations on the He ion irradiated samples showed additional peaks at 152, 243, 764, and 965 cm^{-1} suggesting that it was converted to α -phase SiC. The near similarity in H_2 yields observed for both phases suggests that this phase conversion has little to do with H_2 production, but further experiments may offer more clarity. A previous study where SiC was irradiated with 4 MeV Au ions reported the loss of crystalline structure in the SiC samples and the formation of Si-Si bonds, which was found to be dependent on the dose given to the sample. However, none of the He ion irradiated samples showed the formation of the Si-Si bond, which could be due to the lower stopping power of the He ion than the Au ion leading to less local damage to the SiC surfaces.

X-ray photoelectron spectroscopy is an extremely surface sensitive technique, providing analysis of the chemical composition and chemical bonding occurring in the top 10 nm of a sample. The lower resolution wide scans showed the presence of carbon, oxygen, and silicon atoms in the SiC samples. High resolution scans for those elements for α -phase SiC are shown in Figures 7-9 and for both the α -phase and β -phase SiC. The atomic concentrations of carbon, silicon, and

oxygen were calculated based on the relative areas of the C 1s, Si 2p, and O 1s peaks and relative sensitivity factors for the PHI VersaProbe at those photoelectron energies. The calculated atomic concentrations are given in Table 1. For both the α -phase and β -phase samples, the amount of carbon decreases following irradiation while the relative amounts of silicon and oxygen increase. The C 1s spectra was fit with up to four peaks, one that was set at 282.6 eV for the C-Si bond, one at around 284.7 eV for the C-C bond, and ones at 286.5 eV and 288.9 eV due to carbon-oxygen bonds [45, 46]. These fittings were based on earlier studies that characterized SiC powders using XPS [23, 34, 45-49]. The C-Si bond was used to charge correct the samples instead of the normal procedure using the C-C bond. The C-C bond was low in concentration and appeared as a shoulder, making it difficult to locate the exact position for charge calibration. The other spectra, O 1s and Si 2p, were shifted accordingly. For both the α -phase and β -phase samples, the C-C peak had the largest contribution to the C 1s spectrum in the pristine sample; however, following irradiation the contributions dropped dramatically as can be seen in Table 2. This decrease suggests that irradiating the surfaces with both γ -rays and He ions removed some of the carbon contamination present on the SiC surfaces. The C-Si contribution either increased or stayed the same, as was the case for He ion irradiated α -phase SiC. The contributions of the carbon-oxygen peaks were initially less than 5% of the total, and remained as such following radiolysis.

The Si 2p spectra show contributions from the Si-C bond at 100.5 eV and the Si-O bond in SiO₂ at 102.2 eV [34, 45]. No lower binding energy peak for a Si-Si bond was present in any of the samples. There is good agreement between the calculated concentrations of the C-Si bond and the Si-C bond, listed in Table 2; however there is a discrepancy in the comparison of the Si-O and O-Si bonds with the values calculated in the O-Si bonds being higher. Both phases of SiC had about a 10 % increase in the Si-C bond following irradiation with γ -rays, and had only slight

increases for He ion irradiation. The contribution of the Si-O peak was observed to increase, particularly for the He ion irradiated samples suggesting that following the ion irradiation that parts of the surface are oxidized to SiO₂.

The O 1s spectra are fit with two peaks, a main one at 532.0 eV for the O-Si oxide layer [34, 45], and a higher binding energy shoulder around 533.4 eV, due to H₂O present on the surface [46], which is only present in the pristine samples. Following irradiation, the H₂O peak is no longer visible in the spectrum suggesting the loss of the water layer during irradiation with both γ -rays and He ions. The concentration of the O-Si peak is observed to increase during irradiation, showing the formation of SiO₂, especially following He ion irradiation.

4. Conclusions

Changes in α -phase and β -phase SiC nanoparticle surfaces were observed following radiolysis with γ -rays and with 5 MeV He ions. Adsorbed water was characterized using TPD and DRIFT to show that little physisorbed water was on the SiC surfaces and the chemisorbed water desorbed on heating. Slurries of SiC with varying amounts of water were also examined and the yield of H₂ was found to be higher than that expected using a simple mixture rule, and its yield increases with decreasing amount of water. This result indicates that there is some transfer of energy between the SiC and water in radiolysis. Bulk crystal structure was not observed to change following irradiation with γ -rays or He ions. However, Raman spectroscopy of the SiC surface suggests that the β -phase can be converted to the α -phase in He ion radiolysis. XPS analysis of the SiC surfaces indicated that following irradiation the contribution from the C-Si bonds had increased and that there was a loss of the H₂O peak suggesting that water and impurities were removed from the surfaces during the irradiations. With He ion radiolysis, the formation of SiO₂ oxide was observed on both α -phase and β -phase SiC.

Acknowledgements

The authors thank Prof. Michael Wiescher for making available the facilities of the Notre Dame Nuclear Structure Laboratory, which is supported by the U.S. National Science Foundation. The authors also acknowledge the Center for Sustainable Energy at Notre Dame (cSEND) Materials Characterization Facilities for the use of the PHI VersaProbe II X-ray Photoelectron Spectrometer, Jasco Micro-Raman Spectrometer NRS-5100, and Bruker D8 Advance DAVINCI Powder X-Ray Diffractometer. Support of the National Science Foundation through MRI award 1126374 is acknowledged for the XPS data in this paper. The work reported here was supported by the Division of Chemical Sciences, Geosciences and Biosciences, Basic Energy Sciences, Office of Science, United States Department of Energy through grant number DE-FC02-04ER15533 and by the EPSRC and the Dalton Cumbrian Facility project, a joint initiative of the Nuclear Decommissioning Authority and The University of Manchester. J. Schofield is supported by a studentship from the EPSRC Nuclear FiRST Doctoral Training Centre. This contribution is NDRL-5085 from the Notre Dame Radiation Laboratory.

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Table 1. Atomic composition for α -phase and β -phase SiC samples.

	Carbon (%)	Silicon (%)	Oxygen (%)
α -phase SiC - pristine	55	26	19
α -phase SiC - γ -ray irradiated	45	37	18
α -phase SiC – He irradiated	37	34	29
β -phase SiC - pristine	64	19	17
β -phase SiC - γ -ray irradiated	54	32	14
β -phase SiC – He irradiated	41	34	25

Errors in atomic concentration are estimated at $\pm 5\%$

Table 2. Peak atomic concentrations.

	C 1s				Si 2p		O 1s	
	C-Si (%) 282.6 eV	C-C (%) 284.7 eV	C-O (%) 286.5 eV	C=O (%) 288.9 eV	Si-C (%) 100.5 eV	Si-O (%) 102.2 eV	O-Si (%) 532.0 eV	H ₂ O (%) 533.4 eV
α -phase SiC - pristine	24	26	3	2	21	5	14	5
α -phase SiC - γ -ray irradiated	32	12	0	0	31	6	18	0
α -phase SiC – He irradiated	23	13	0	1	24	10	29	0
β -phase SiC - pristine	14	46	2	0	16	3	11	6
β -phase SiC - γ -ray irradiated	21	31	3	0	27	5	14	0
β -phase SiC – He irradiated	24	15	1	2	23	11	25	0

Figure Legends

Figure 1. N₂ adsorption (■) and desorption (●) isotherms for α -phase (black) and β -phase (red) SiC nanoparticles.

Figure 2. Temperature programmed desorption curves for water on α -phase (black) and β -phase (red) SiC surfaces. Desorption energies calculated using Redhead's method [20].

Figure 3. DRIFT spectra of α -phase SiC powder (upper) and β -phase SiC powder (lower) from room temperature to 400°C. Spectra are offset for clarity. The spectra for water ice are shown for comparison [22].

Figure 4. Yield of H₂ with α -phase SiC and 70% water mass as a function of dose. Yields on the upper plot are calculated with respect to the entire slurry sample (SiC + water), and yields on the lower plot right are calculated only with respect to the mass of water in the sample.

Figure 5. Yields of H₂ from α -phase and β -phase of SiC in water slurries as a function of the percentage of water mass. Yields on the upper plot are calculated with respect to the entire slurry sample (SiC + water), and yields on the lower plot right are calculated only with respect to the mass of water in the sample. The dashed line represents the yield expected from the law of mixtures.

Figure 6. Normalized Raman spectra of pristine (top), γ -ray irradiated (middle), and He ion irradiated (lower) for α -phase SiC samples (upper plot) and β -phase SiC (lower plot). Spectra are offset for clarity. The lower plot for β -phase SiC includes spectra for two different regions following He irradiation.

Figure 7. High resolution XPS scan with peak fitting of the C 1s photoelectrons from a pristine (top), γ -ray irradiated (middle), and He ion irradiated for α -phase SiC (upper plot) and β -phase SiC (lower plot). Spectra are offset for comparison.

Figure 8. High resolution XPS scan with peak fitting of the O 1s photoelectrons from a pristine (top), γ -ray irradiated (middle), and He ion irradiated for α -phase SiC (upper plot) and β -phase SiC (lower plot). Spectra are offset for comparison.

Figure 9. High resolution XPS scan with peak fitting of the Si 2p photoelectrons from a pristine (top), γ -ray irradiated (middle), and He ion irradiated for α -phase SiC (upper plot) and β -phase SiC (lower plot). Spectra are offset for comparison.

Figure 1. N₂ adsorption (■) and desorption (●) isotherms for α -phase (black) and β -phase (red) SiC nanoparticles.

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Figure 3. DRIFT spectra of α -phase SiC powder (upper) and β -phase SiC powder (lower) from room temperature to 400°C. Spectra are offset for clarity. The spectra for water ice are show for comparison [22].

Figure 4. Yield of H_2 with α -phase SiC and 70% water mass as a function of dose. Yields on the upper plot are calculated with respect to the entire slurry sample (SiC + water), and yields on the lower plot right are calculated only with respect to the mass of water in the sample.

Figure 5. Yields of H_2 from α -phase and β -phase of SiC in water slurries as a function of the percentage of water mass. Yields on the upper plot are calculated with respect to the entire slurry sample (SiC + water), and yields on the lower plot right are calculated only with respect to the mass of water in the sample. The dashed line represents the yield expected from the law of mixtures.

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