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GRAPHITE SURFACE EROSION AND BLISTERING

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ABSTRACT

Bulk reactor grade graphite and WCA graphite cloth were exposed to an atom and ion beam produced in a Hall accelerator. Samples exposed to hydrogen received a total dose of 10^{20} atoms/cm² of 580 eV mean energy H⁰ and 5×10^{18} ions/cm² of 875 eV mean energy H⁺. Samples exposed to helium received a total dose of 10^{20} atoms/cm² of He⁰ and 5×10^{18} ions/cm² of He⁺. During irradiation, the pressure in the sample region was governed by the partial pressure of the source gas (H₂ or He), and was at 0.35 mTorr. The samples were then examined with a scanning electron microscope and an optical interference microscope to determine the surface damage. Upon exposure to H⁰ and H⁺, the bulk graphite and the graphite cloth samples showed substantial surface erosion. The amount of erosion was found to be strongly dependent on the sample surface temperature. Upon exposure to He⁺, blistering was observed on the graphite cloth sample. No measurable surface erosion of the samples exposed to He was observed with the scanning electron microscope.

Introduction

Graphite has been proposed as a nonstructural first wall material for a tokamak fusion reactor.⁽¹⁾ A major energy loss mechanism for a fusion reactor plasma is line radiation from partially stripped high atomic number impurities. Low Z impurities would rapidly be fully stripped, and therefore not emit line radiation. A reactor could therefore operate with a larger percentage of low Z material as an impurity than with high Z impurity.⁽²⁾ A graphite liner for a tokamak fusion reactor has also been proposed to shift the energy spectrum of neutrons emitted from the plasma, thereby increasing the lifetime of the structural wall.⁽¹⁾

Two types of experiments have been performed previously with graphite. The first involves the bombardment of graphite by cold atomic hydrogen and H^+ ions in a background of H_2 at a pressure of the order 1 Torr.^(3,4) In this first type of experiment, volatile hydrocarbons, such as methane, were produced which then desorb from the graphite surface. Carbon is therefore "chemically sputtered" from the surface. The amount of sputtering is a strong function of the surface temperature of the graphite. The sputtering yield, or ratio of the number of carbon atoms leaving the surface to the number of incident H atoms, (in units of atoms/incident particle, either H^0 or H^+) increases with increasing surface temperature up to about 800°K, and then sharply decreases to at least 1250°K. This peak in the chemical sputtering yield is thought to be due to the enhanced reaction rate for dissociation of methane, the volatile hydrocarbon, at high temperatures.⁽⁴⁾ The chemical sputtering yield also depends on the degree of crystallinity of the target surface. Sputtering yields are about a factor of ten higher for samples with randomly oriented crystal

structure compared to samples with a high degree of crystallinity.⁽³⁾ At temperatures of about 800°K, the chemical sputtering yield ranges from 10^{-4} to 10^{-3} atoms/incident particle depending on the crystalline structure of the surface.⁽³⁾

The second type of experiment involves the bombardment of graphite by high energy (10-20 keV) H^+ ions. This type of experiment has been performed with a very low background pressure (10^{-6} Torr) of H_2 gas. The sputtering yield observed in these experiments ranges in magnitude from 10^{-2} to 10^{-1} atoms/ion, and is dependent on the surface temperature.^(5,6,7) No change in the sputtering yield was observed when the energy of the incident ion was varied from 10-20 KeV⁽⁶⁾, leading to the conclusion that "chemical sputtering" is responsible for these high yields.

Neither of these types of experiments accurately reproduces the conditions expected in a tokamak fusion reactor environment. Present tokamak reactor designs envision a flux of about $10^{13} \text{ cm}^{-2} \text{ sec}^{-1}$, 100 eV to 1 KeV H atoms and ions impinging on the first wall. The neutral pressure of hydrogen at the first wall is expected to range from 10^{-5} to 10^{-3} Torr.⁽⁸⁾ A tokamak fusion reactor environment is therefore likely to have a relatively high background pressure of neutral hydrogen, in addition to a large flux of energetic H atoms, with energies up to about 1 KeV.

Experimental Procedure

The present experiment uses a Hall source⁽⁹⁾ to produce a high flux of energetic atoms and ions. The Hall source produced a flux of $2 \times 10^{19} H^0$ atoms/ $\text{cm}^2 \text{ sec}$ at the sample, ranging in energy (full width, half maximum) from 200 to 750 eV, with a mean energy of 580 eV. The source simultaneously produced

a flux of $10^{18} \text{ H}^+ \text{ ions/cm}^2 \text{ sec}$ at the sample, ranging in energy from 500 to 1100 eV, with a mean energy of 875 eV.⁽¹⁰⁾ The base pressure is 5×10^{-6} Torr, but during irradiation the pressure at the sample location rises to 0.35 mTorr due to the increase in partial pressure of the source gas. The pulse of energetic atoms and ions is 5 msec long, with a repetition rate of one pulse every 80 seconds. Each sample is exposed to 1000 pulses, resulting in a total fluence of $10^{20} \text{ H}^0 \text{ atoms/cm}^2$ plus $5 \times 10^{18} \text{ H}^+ \text{ ions/cm}^2$, over a 24 hour period.

Samples of polycrystalline reactor grade (AGOT) graphite and WCA graphite cloth were exposed to the flux from the Hall source. The reactor grade bulk graphite samples were heated by resistance heating to examine the temperature dependence of the sputtering yield. Before firing the Hall source, the samples were at room temperature, 650°K, and 920°K. These temperatures were calculated by balancing the electrical power input and the radiated power output. The heated samples were electrically in series with a fourth sample at higher temperature. The temperature of the fourth sample was measured using an optical pyrometer and this temperature was used to normalize the calculated temperatures of the other heated samples. Assuming that the energetic atoms and ions deposit all their energy on the surface of the bulk graphite samples, the surface temperature is calculated to rise ~150°K during each pulse. With the exception of the sample initially at room temperature, the samples return to their initial temperature by radiative cooling during the 80 second interval between pulses. After the third pulse, the room temperature sample is calculated to radiatively cool to a temperature of 330°K before each succeeding pulse.

The bulk graphite samples were prepared by polishing with progressively finer grades of emery paper, and were finished on a polishing cloth with a solution of 0.3 μm alumina particles in water. The samples were then partially covered by a 0.5 mil tungsten wire. The wire shadowed part of the surface from the beam, and therefore provided a "step" on the surface. This step height was measured using an optical interference microscope⁽¹¹⁾, and the magnitude of surface erosion could therefore be determined. A small amount of tungsten was sputtered onto the surface of the graphite on each side of the wire. The distribution of the tungsten deposit was examined using an electron probe. No interference fringe shifts were observed due to this deposit. Therefore the tungsten deposit did not influence the sputtering measurements.

WCA graphite cloth samples were also exposed to the flux of energetic particles from the Hall source. The graphite fibers are about 8 μm in diameter, and have a turbostratic crystalline structure. The cloth samples were prepared by heating in vacuo. They were then removed from the vacuum chamber and placed in the Hall source vacuum system. The temperature of the surface fibers is calculated to rise to 3300°K during each shot. The temperature rise is calculated by assuming that the energetic atoms and ions deposit all their energy in the first layer of fibers. Conductivity is negligible in graphite cloth, and radiation cooling limits the temperature. The surface erosion was measured by using a scanning electron microscope (SEM) to compare the size of the fibers before and after exposure to the energetic atoms and ions. Photographs of the fiber were taken with three different angular views, $\theta = 0^\circ, 45^\circ, 60^\circ$ measured from the surface normal.

Results

The sputtering yields for the bulk graphite samples are presented in Figure 1. The yields are given in terms of atoms/incident particle, adding together the atom and ion fluences. The error bars are placed at the average temperature of each of the three samples. The large magnitude of the error bars is due to uncertainties in the total fluence hitting the samples. The samples were exposed simultaneously so the relative magnitude of the sputtering yields as a function of average surface temperature is accurate to within +50%.

The sputtering yield for the graphite cloth sample was determined to be 0.23 atoms/incident particle. Figure 2 shows a diagram of the shape of the fiber before and after bombardment. The actual fiber is not circular in shape, but has ridges parallel to the fiber axis. Figure 3 shows an SEM photograph of a fiber that has been partially shadowed by another fiber during bombardment. The reduction in diameter due to bombardment can be clearly seen.

To test that source impurities were not responsible for the observed erosion, samples of WCA graphite cloth were exposed to energetic helium atoms and ions produced in the Hall source. The fluxes and energy distribution of the helium atoms and ions were the same as for the hydrogen atoms and ions. The samples were exposed for 1000 pulses, resulting in a total fluence of 10^{20} He⁰ atoms/cm² plus 5×10^{18} He⁺ ions/cm². No measurable surface erosion was observed by using the SEM to compare the fiber diameters before and after exposure. The absence of measurable erosion verifies that neither source impurities nor background contaminants are responsible for the large amount of erosion observed under bombardment with hydrogen. Surface deformation, in the form of

blisters or ridges perpendicular to the fiber axis, was observed. Figure 4 shows an SEM photograph of a fiber exposed to helium. There are about 5 ridges per micron of fiber length.

Discussion

The observed sputtering yield for the bulk graphite samples in this experiment agrees quite well with the sputtering yield measured for 10-20 KeV H^+ ions. (5,6,7) The dependence of the yield on surface temperature is qualitatively similar to that observed both for room temperature H^0 atoms and for 10-20 KeV H^+ ions. (4,6) The magnitude of the sputtering yield, however, is more than two orders of magnitude higher than the sputtering yield measured with room temperature H^0 atoms. (3) This suggests that the high rate of erosion is due to chemical sputtering enhanced by the energetic ions.

The observed sputtering yield for the WCA graphite cloth is two to three times the maximum yield of the bulk graphite samples. This may be due to the formation of other volatile hydrocarbons, such as acetylene, at the very high temperature of the graphite fibers.

The first wall environment in a fusion reactor is presently expected to consist of a low flux ($10^{13} \text{ cm}^{-2} \text{ sec}^{-1}$) of energetic (100-1000 eV) hydrogen atoms and protons in a relatively high (10^{-5} - 10^{-3} Torr) background pressure of H_2 . The present experiment indicates that bombarding samples with 200-750 eV H^0 atoms with a relatively high background pressure of H_2 results in sputtering yields comparable to that measured for much higher energy incident ions, and orders of magnitude higher than that measured for room temperature hydrogen atoms and ions.

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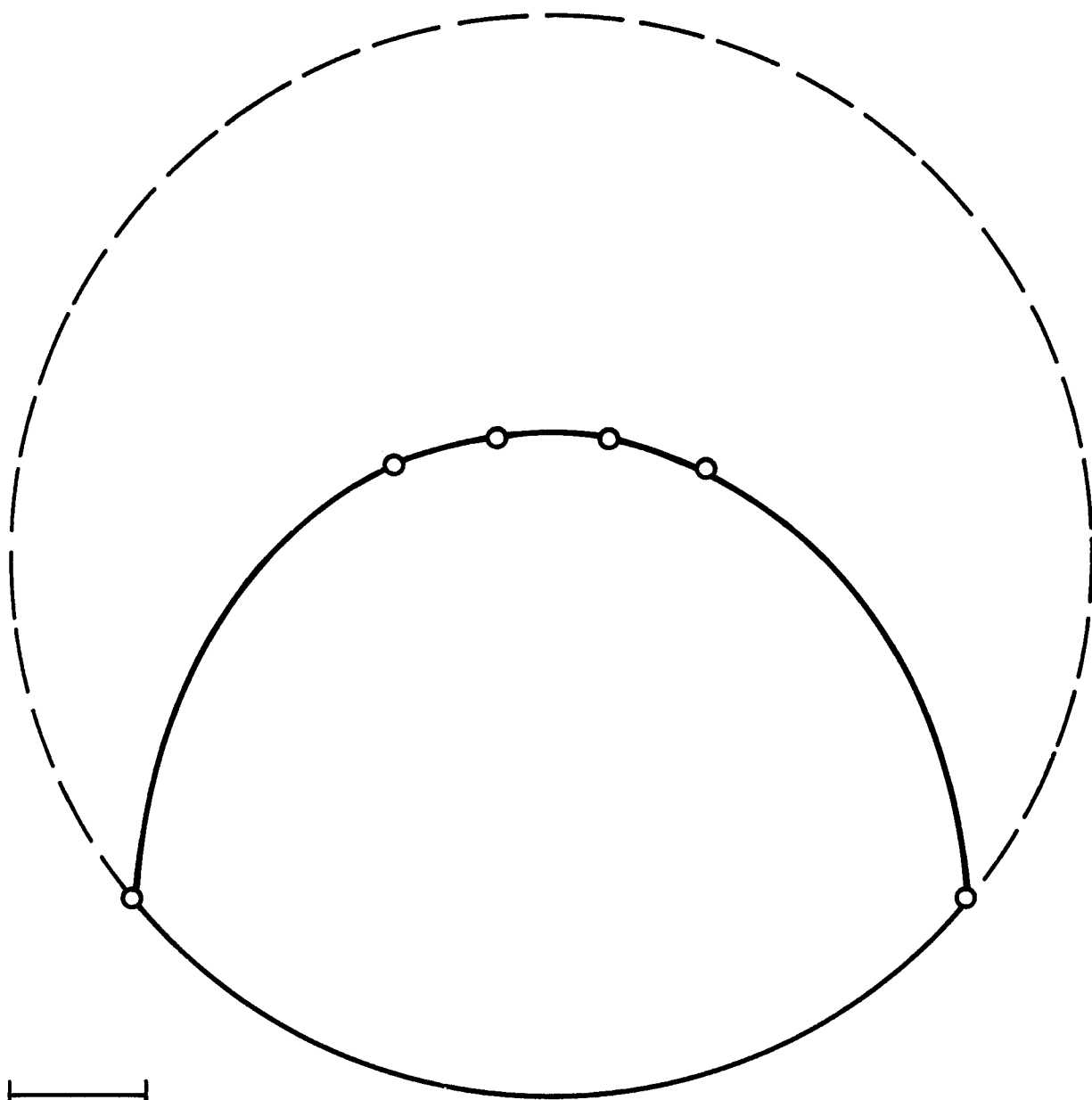
FIGURE CAPTIONS

Figure 1 - The sputtering yield as a function of surface temperature, for polycrystalline reactor grade (AGOT) graphite samples exposed to 10^{20} cm^{-2} hydrogen atoms and ions.

Figure 2 - The dashed circle indicates the WCA graphite fiber shape before bombardment. The solid line indicates the fiber shape after exposure to 10^{20} cm^{-2} hydrogen atoms and ions.

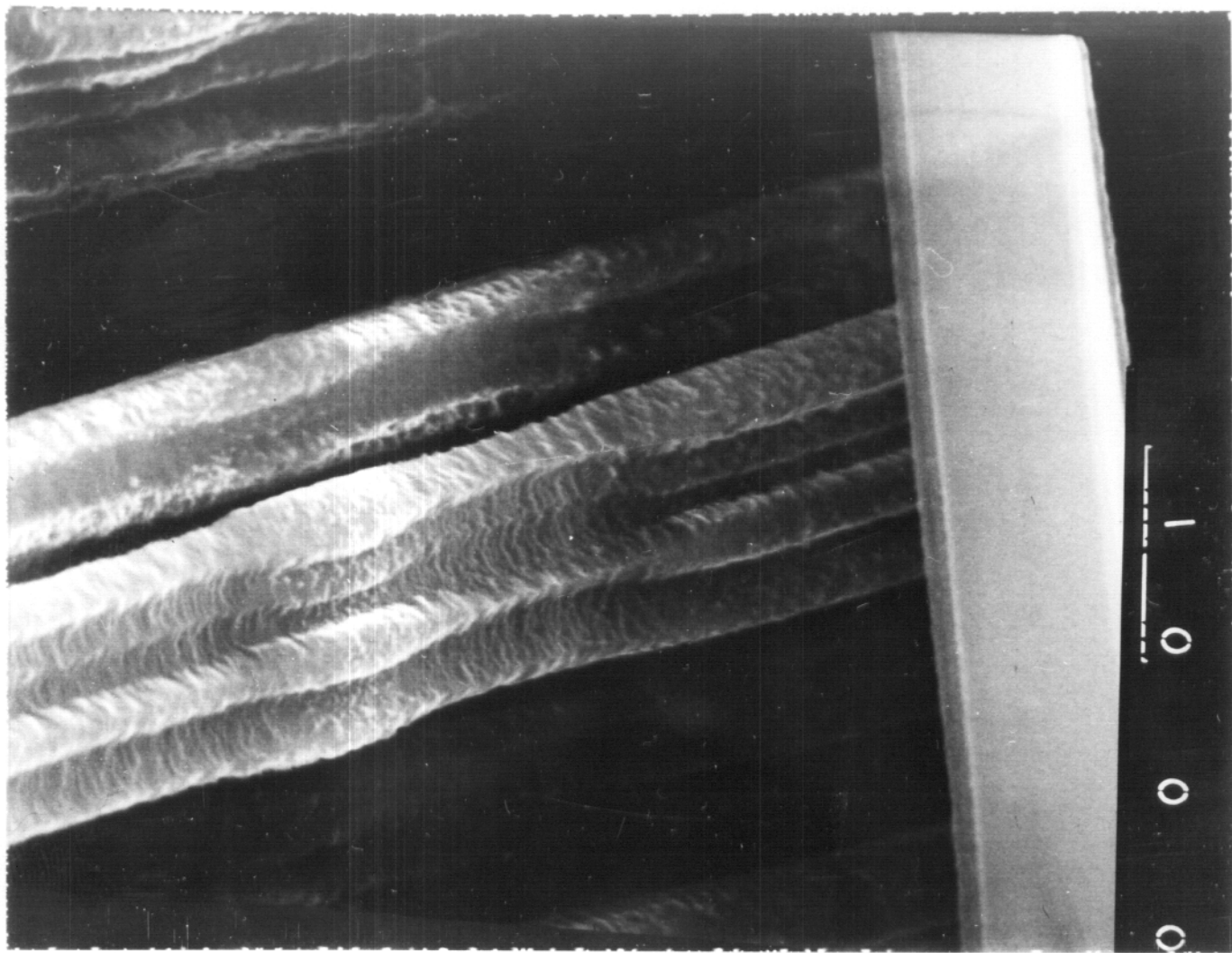
Figure 3 - WCA graphite cloth exposed to 10^{20} cm^{-2} hydrogen atoms and ions. This scanning electron micrograph is taken at a 45° angle, and the fiber diameter bulges where it was shadowed from the ion source flux.

Figure 4 - WCA graphite cloth exposed to 10^{20} cm^{-2} helium atoms and ions. This scanning electron micrograph shows ridges or blisters perpendicular to the fiber axis. There are about 5 ridges per micron of fiber length.





1 MICRON



5 MICRONS