Radiation Blistering in Niobium


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FUSION TECHNOLOGY INSTITUTE
UNIVERSITY OF WISCONSIN
MADISON, WISCONSIN
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Fusion Technology Institute
University of Wisconsin
1500 Engineering Drive
Madison, WI 53706

http://fti.neep.wisc.edu

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*University of Wisconsin-Milwaukee
During the operation of a thermonuclear fusion reactor, the vacuum wall material must operate under stress at high temperatures (700°-1000°C) under the influence of bombarding neutrons, neutral atoms, ions and photons. In order to determine surface damage and contamination of the plasma, it will be necessary to determine sputtering ratios and possible radiation blistering.1

Sputtering yields have been measured for H⁺, D⁺, and He⁺ over the incident ion energy range of 2 keV to 80 keV.2 It has been observed that these ions incident on metals can form gas bubbles in the solid. If the solubility and diffusion of hydrogen isotopes and helium in the wall material is small, the surface will be quickly over saturated by the trapped gases. This leads to the formation of bubbles which grow after further bombardment. These bubbles can migrate and eventually burst in or near such energetically favored regions as the surface, grain boundaries, or dislocation lines.3 The result is to release bursts of gas and to pit the surface. Results for copper irradiated at room temperature indicate pits one micron in diameter.4

Whether this is a problem for refractory metals at 700°-1000°C has not been known heretofore. Study of helium release has been conducted by Bauer et al.5 in which radiation blisters were observed in materials previously implanted with helium. It was estimated that 25% of the helium was released
in the 450°-750°C range. Complete helium release was not predicted until the temperature is raised above 1000°C.

In this work radiation blistering has been observed in niobium bombarded with 250 keV D⁺ ions at sample temperatures in the range 600°-700°C. The irradiated zone (0.03cm²) was subject to an ion flux of 3×10¹⁴ ions/cm²·sec, to a total fluence of 1×10¹⁹ ions/cm². The target sample was thermally and electrically insulated from a holder which was enclosed by a furnace which was heated by electron bombardment. Temperature was measured to ±5°C by two thermocouples attached to the sample backing. Current to the target was read from the same backing plate.

A scanning electron microscope was used to examine the samples after irradiation. Photomicrographs revealed raised hillocks or domes at 10,000X magnification. At 1000X randomly oriented pits were observed which covered the irradiated zone at an average density of 3×10⁷/cm². At higher magnification the domes appear to be unburst blisters which average 0.72 microns in diameter. Comparison was made with control samples: (a) niobium which had been annealed and electro-polished as prepared for bombardment, but not subjected to temperature rise or irradiation; (b) areas of the samples which were irradiated but were taken from zones masked off from the incident ion beam. One sample which was inserted in the vacuum system during the preliminary de-gassing procedure was heated to 750°C in a poor vacuum (1×10⁻⁵ torr)
showed thermally etched pits exposing the [111] plane in the niobium sample. The vacuum pressure during irradiation was typically $1-2 \times 10^{-7}$ torr.

It is clear that the vacuum wall should be atomically clean at the beginning. It should be operated at a temperature above 700°C, and should be made of a material with a high diffusivity and solubility for hydrogen isotopes and helium. This hypothesis is now being tested in an experiment with a furnace constructed of molybdenum, which will enable heating of the sample to 1000°C and study of the effect on the surface of niobium, vanadium and tantalum subjected to irradiation of D$^+$ and He$^+$ ions.


