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HELIUM BLISTERING OF CERAMIC COATINGS ON HASTELLOY X AND Nb-1% Zr†

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Abstract

The surface damage of insulating ceramic coatings on Hastelloy X and Nb-1% Zr held at room temperature and at 300°C has been studied for both 100 KeV and 250 KeV helium ion irradiation for a dose range from 3.7×10^{18} to 1×10^{19} ions cm^{-2}. Blisters were observed after room temperature irradiation with both 100 KeV and 250 KeV helium ions. However, for irradiation at 300°C no blisters could be observed. The sharp rise in the helium permeation with temperature, observed by others for some glasses and ceramics, is thought to be responsible for this behaviour. These results suggest that for the energy range studied helium blistering has a negligible surface erosion effect on such coatings if they are operated at temperatures above 300°C.

INTRODUCTION

Certain fusion reactor concepts require that the entire first wall or parts of it consist of electrically insulating material. For example, the first wall of the reference theta-pinch reactor (RTPR)[1] is a structure with an insulator on a metal backing. This design is necessary to electrically insulate the first wall metal against the emf which develops the implosion heating. The insulating layer serves to prevent electrical breakdown between the plasma and blanket segments during the short implosion heating stage. It is necessary that the dielectric and mechanical properties of this composite structure of the first wall be maintained during the pulsed mode operation of such a reactor for a reasonable lifetime. In the RTPR-design alumina has been specified as the electrically insulating liner of the first wall, and Nb-1% Zr as the first wall structural metal. In order to minimize the radiation damage in the insulating liner the design incorporated a neutral gas blanket near the wall.

For such theta-pinch type of fusion reactors different types of ceramic insulator coatings are being developed by Atomics International. It was the aim of these studies to determine the behaviour of such coatings under energetic helium impact for different target temperatures.

EXPERIMENTAL TECHNIQUES

Two different ceramic coatings on two different types of substrates, Nb-1% Zr and Hastelloy X, were supplied by D. W. Keefer, Atomic International. The coating on Hastelloy X (coating #13) consisted of a mixture of 54.3% SiO_2, 38.7% BaO, 7.0% Al_2O_3, and the coating on Nb-1% Zr (coating #14) was a mixture of 52.3% SiO_2, 40.5% BaO, 7.2% Al_2O_3. (The concentrations are given in wt%.) Details of preparation of these coatings can be found elsewhere.[2] The coatings on Hastelloy X substrates had thicknesses ranging from 71 to 130 μm and the thickness of coatings on Nb-1% Zr substrates ranged from 62 to 99 μm. The thicknesses of these coatings are about 40 to 65 times larger than the projected range (R_p) of 250 keV $^4He^+$ in Silicon ($R_p \sim 1.53$ μm). The coatings were irradiated with 100 keV or 250 keV $^4He^+$ ions from a 2-MeV Van de Graaff accelerator in high vacuum at a total pressure of $\sim 5 \times 10^{-8}$ Torr. The flux of the incident helium ions was held at 1×10^{14} ions cm^{-2} sec^{-1}. For the determination of the actual dose for the irradiation of these surfaces correction for the secondary electron emission for these surfaces vs. those for metal target surfaces were made. The total dose values given in this paper are correct within only ~ 20%. Other details of the irradiations are similar to those of the irradiations of metal targets described earlier.[3] No optical or chemical polishing of the targets was considered necessary but all the targets were cleaned in ultrasonic baths of trichloroethylene, acetone, distilled water and methanol, prior to irradiation. For irradiation at 300°C the targets were heated directly by ohmic heating and the target temperatures were measured by an infrared pyrometer. The target surfaces were examined prior to and after the irradiations with the aid of a Cambridge stereoscan S4-10 scanning electron microscope. The ceramic coatings were [illegible] 200 Å [illegible] during observation in the scanning electron microscope.

RESULTS AND DISCUSSION

Figure 1 shows scanning electron micrographs of blisters formed on coatings 13 (Figures 1a and 1b) and 14 (Figures 1c and 1d) during room temperature irradiation with 100 keV and 250 keV helium ions for a total dose of 3.7 x 10[illegible] ions cm^{-2}. The appearance of dome shaped blisters in both ceramic coatings suggests that the coatings can be plastically deformed readily. This is quite different from the pits observed in other ceramic materials such as SiC after helium ion irradiation.[3] The ceramic coatings used in the present studies have an amorphous structure with very little crystallinity. For both coatings the majority of the blisters have diameters ranging from 0.6 to 3 μm for 100-keV He^+ ion irradiation, and ranging from 3 to 5 μm for 250-keV He^+ ion irradiation. In several regions clusters having about 2-6 interconnected blisters have formed. The blister density (i.e. the number of blisters per unit area) for both coatings [illegible] the [illegible] $1.9 \pm 0.3 \times 10^6$ blisters cm^{-2} for both helium ion energies used and for the total dose of 3.7×10^{18} ions cm^{-2}. These results differ from those given in the summary[4] of this paper, since a correction to the total dose value due to secondary electron emission had not been applied [illegible]. Since the compositions of these two coatings [illegible] different it is not surprising that the blister [illegible] [illegible] 3.7×10^{18} ions cm^{-2} the blisters have not exfoliated to a measurable degree [illegible].

[illegible] 2 shows blisters formed at higher doses. The micrograph [illegible] [illegible] blisters [illegible] for room temperature [illegible] [illegible] spherical [illegible] have increased in [illegible] (see Figure [illegible]). The blister diameters [illegible] 10^6 blisters cm^{-2}, a value which is about a factor of 3 higher than the one for the lower [illegible]

[illegible] shows blisters [illegible] irradiated at room temperature with 250-keV He^+ ions for a total dose of [illegible] $\times 10^{19}$ ions cm^{-2}. The blisters appear to be more dome shaped and have increased in size as compared to the low dose case (Figure 1d). The blister diameters range now from 4 to 5 µm. The blister density for this higher [illegible] is [illegible] 1.0 ± 0.2 × 10[illegible] blisters cm^{-2}, a value which is about a factor of [illegible] [illegible] than the one for the lower d[illegible].

To measure the blister skin thickness [illegible] [illegible] [illegible] procedure did not result in any large scale exfoliation. However, in several blisters holes were observed as shown in Figure [illegible] for 100 keV [illegible] [illegible] ions irradiation. The blister skin thicknesses estimated from such [illegible] [illegible] 0.7 [illegible] [illegible]

[illegible]

insulator surfaces were coated with gold as mentioned earlier). The projected [illegible]

[illegible]

target the projected range values would be reduced.

[illegible] irradiation at room temperature (Figure [illegible] [illegible] [illegible] are seen in Figure [illegible]. Examination of the irradiated [illegible] irradiated area (Figure 4b) show no significant [illegible] do not [illegible] in helium [illegible]

permeation rate through Corning glass no. 7740 increases by more than two [illegible] to 300°C. Of course the permeation rates depend strongly on the glass com- [illegible]

SUMMARY

[illegible]

The blister [illegible] with increasing ion energy and [illegible] well.

The irradiation temperature has a strong effect on blister formation. [illegible] could be observed [illegible] of the type [illegible] effect at these temperatures.

2. T. G. Parker, Jr., editor "Development of Glass Electrical Insulator for

3. M. [illegible] and S. K. [illegible], "Erosion of Silicon Carbide Surfaces Under

8. V. O. Altemose, J. Appl. Phys. "Helium diffusion through Glass" 32, 1309

FIGURE CAPTIONS

Fig. 1. Scanning electron micrographs (SEMs) of ceramic coatings after irradiation at room temperature with $^{4}He^{+}$-ions to a total dose of [illegible] ions/cm^{-2}: [illegible] #13 (see text for composition) [illegible] $^{4}He^{+}$, (b) coating #13 [illegible] (c) [illegible] (see [illegible] for composition) [illegible] $^{4}He^{+}$, [illegible] #14 irradiated with 250-keV $^{4}He^{+}$.

Fig. 2. SEMs of coating #14 on Nb-1% Zr irradiated at room temperature (a) with 100-keV $^{4}He^{+}$-ions to a dose of 1 x 10^{19} ions cm^{-2}, (b) [illegible] $^{4}He^{+}$ [illegible] 10^{19} [illegible] $^{-2}$.

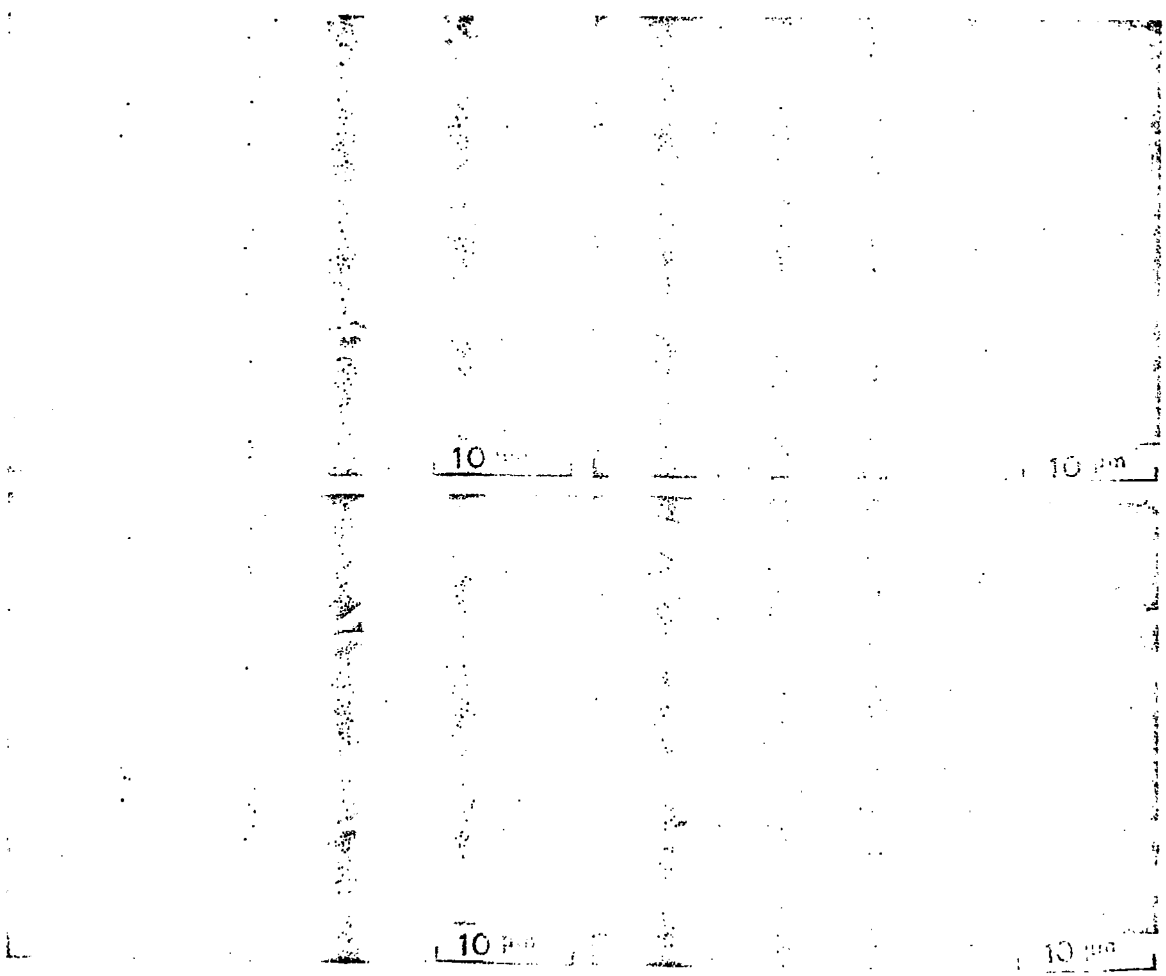
[illegible] coating #14 on Nb-1% Zr [illegible] (b) [illegible] 250-keV $^{4}He^{+}$ ions to a total dose of 1 x 10^{19} ions/cm^{-2}.

[illegible] (a) [illegible] Zr [illegible] irradiated at 500°C with 100-keV $^{4}He^{+}$ ions to a total dose of 3.7 x 10^{18} ions cm^{-2}, [illegible] (c) coating #15 on Hastelloy X [illegible] .7 [illegible] 10^{18} [illegible] coating [illegible]

INSULATOR CERAMIC COATINGS IRRADIATED AT ROOM TEMP. TO A DOSE OF 0.5 C/cm^2

COATING no. 13 (54.3 % SiO_2, 38.7% BaO, 7.0% Al_2O_3) ON HASTELLOY X

COATING no. 14 (5[illegible] % SiO_2, 40.5% BaO, 7.2% [illegible]O_3) ON Nb-1% Zr



WITH 100 keV He^+

WITH 250 keV He^+

CERAMIC COATING no.14 (52.3% SiO_2, 40.5% BaO, 7.2% Al_2O_3)
ON Nb-1% Zr IRRADIATED AT ROOM TEMP.

WITH 100keV He^+ WITH 250keV He^+

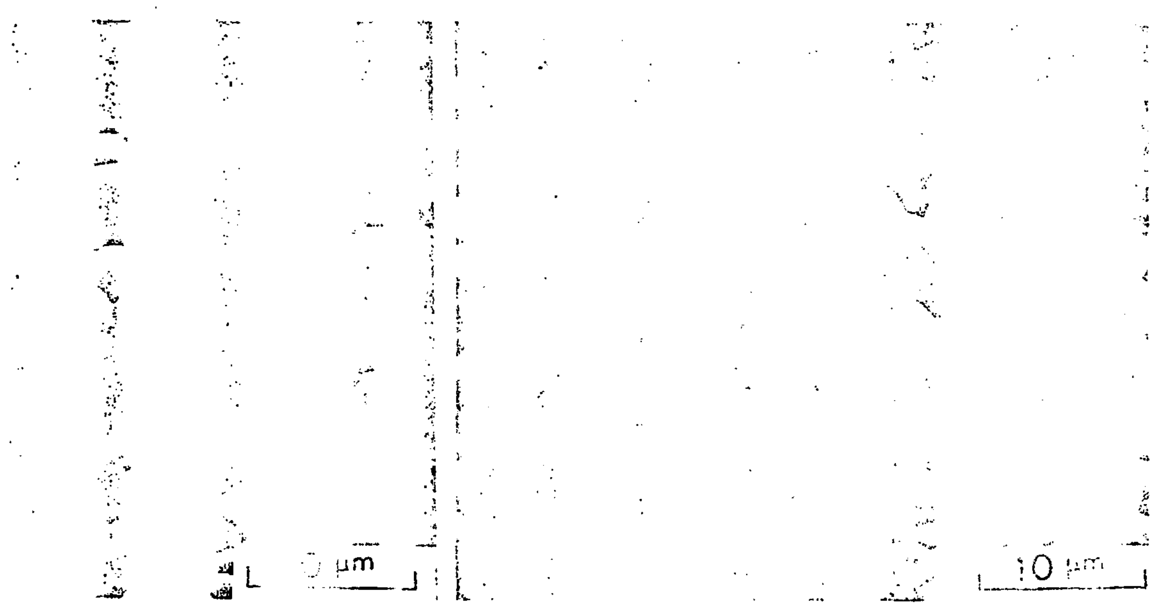


FIGURE 2

INSULATOR CERAMIC COATING no. 14 (52.3% SiO_2, 40.5% BaO, 7.2% Al_2O_3)

ON Nb-1% Zr IRRADIATED AT ROOM TEMP.

WITH 100 keV He^+ — WITH 250 keV He^+

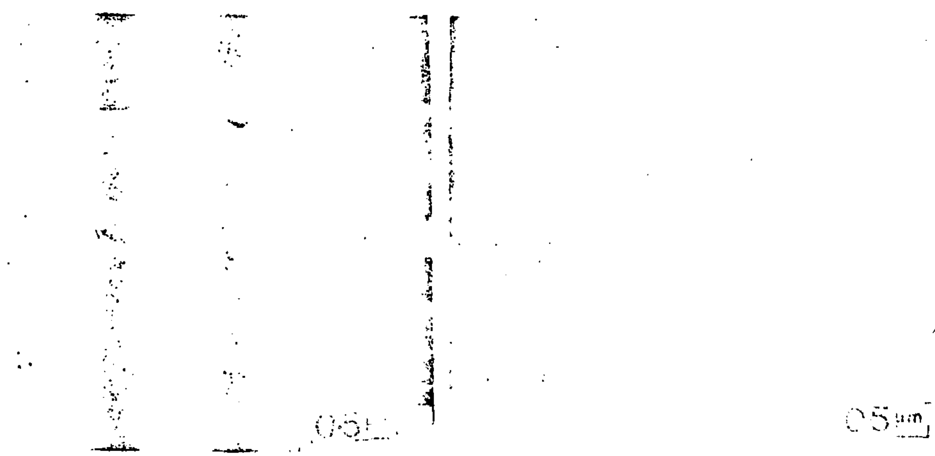


FIGURE 3

INSULATOR CERAMIC COATINGS IRRADIATED AT 300°C WITH 100 keV He^+

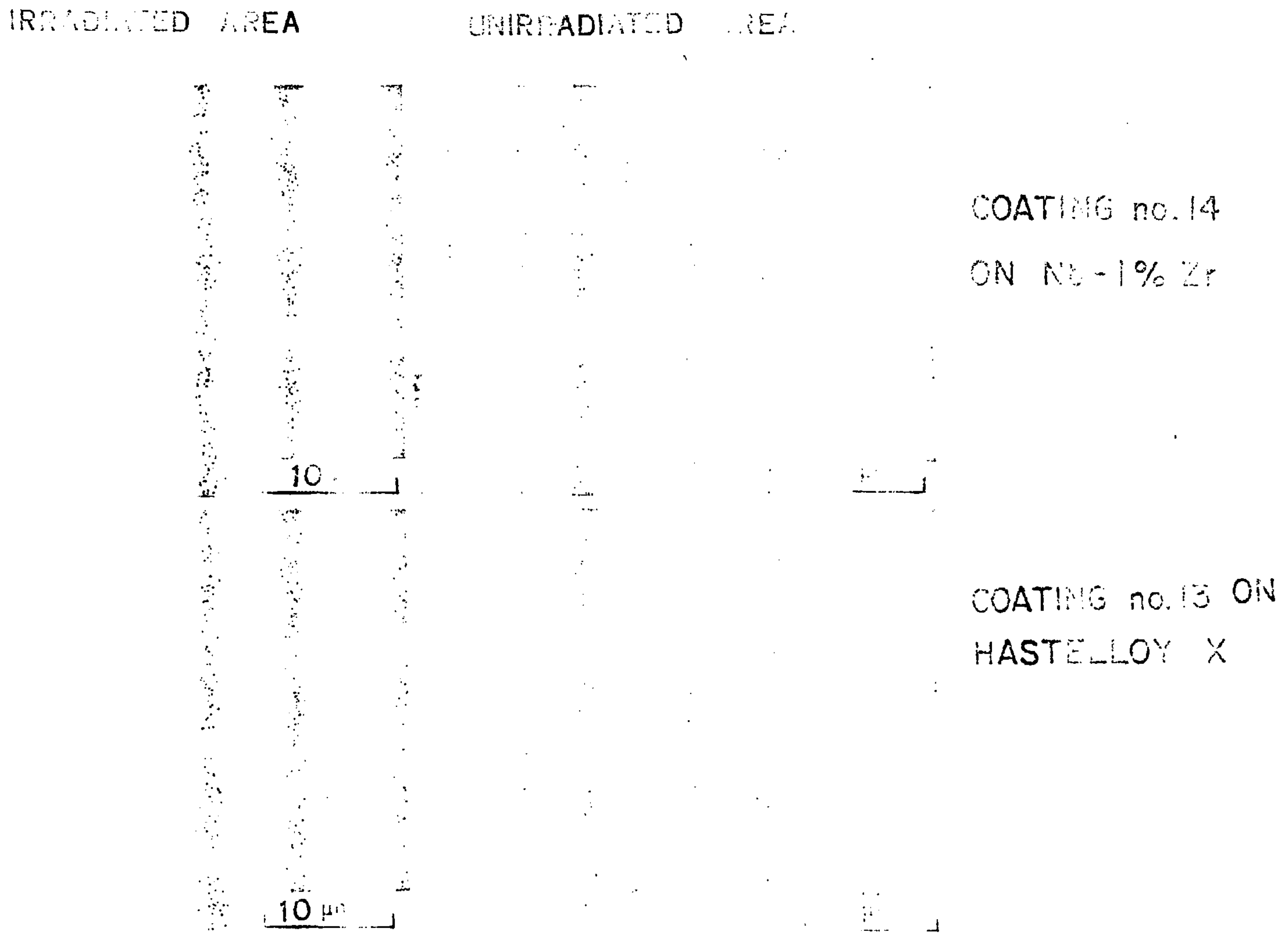


FIGURE 4