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by S. K. Das and M. Kaminsky

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EROSION OF STAINLESS STEEL FIRST WALL BY HELIUM BLISTERING†

S. K. Das and M. Kaminsky
Argonne National Laboratory, Argonne, Illinois 60439

Summary

The blister formation and the erosion rates associated with helium blistering in annealed 304 stainless steel have been investigated for helium projectiles with energies ranging from 100 keV to 1.5 MeV and for irradiation temperatures ranging from room temperature to 550°C. The total dose was varied from 0.1 C/cm² to 1.0 C/cm². The results show that the blister size increases with increasing projectile energy. The degree of blistering, and the erosion rates associated with blister rupture and exfoliation are found to be strongly temperature dependent. Maximum erosion rates were observed at irradiation temperature of around ~450°C. The erosion rates decreased at higher temperature ~550°C. The degree of blistering is found to increase with increasing dose.

Introduction

It has been pointed out[1—3] that energetic helium projectiles from a D-T plasma in a fusion reactor can leak out of the confining field either as charged particles or as neutrals (formed, for example by charge exchange near the plasma boundary), strike the first wall surfaces, and form blisters. It has also been shown[4—9] that helium blistering will be an important erosion process in the first wall of fusion reactors made out of niobium, vanadium and their alloys. The present work on stainless steel is a continuation of our previous work[4—6, 8, 9] on helium blistering in niobium and vanadium. Stainless steel is being considered as the structural material for the first wall of fusion reactors in several reference designs.[10] Although refractory metals such as niobium and vanadium are potential candidates for the first wall, the first generation of prototype fusion reactors may use stainless steel. Recently Bauer and Thomas[11] have reported blister formation and helium re-emission from 316 stainless steel surface during irradiation with 300-keV helium ions. In this paper experimental data on erosion rates associated with helium blistering in annealed 304 stainless steel will be presented for helium in energies of 100 keV, 0.5 MeV and 1.5 MeV and for different irradiation temperatures.

Experimental Techniques

Commercial grade 304 stainless steel sheets containing 0.052% C, 1.69% Mn, 0.017% P, 0.025% S, 0.60% Si, 9.38% Ni, 18.49% Cr and 0.1% Mo was obtained from the House of Stainless, Inc. These sheets were rolled to a thickness of about 0.25 mm. The foils were encapsulated in quartz tubes in vacuum, annealed for 2 hrs. at 1050°C, and then water quenched. They were first mechanically polished and then electropolished in an electrolyte containing 60% glycerine and 40% phosphoric acid at 65°C, at an applied voltage of 9 volts. The targets were then irradiated with $^4He^+$ ions with energies of 100 keV, 0.5 MeV and 1.5 MeV from a 2-MeV Van de Graaff accelerator. During the irradiation the vacuum in the target chamber was maintained at $1-2 \times 10^{-8}$ Torr by ion pumping. The ion flux was 1×10^{14} ions/(cm²-sec) for the 100-keV and 0.5-MeV helium ion irradiations and was 3×10^{13} ions/(cm²-sec) for the 1.5-MeV helium ion irradiations. Other experimental details of target heating and irradiations are the same as those given previously[4—6, 9] for niobium and vanadium irradiations.

Results

The blister formation in annealed 304 stainless steel will be described separately for different irradiation temperature. The irradiation temperature was varied from room temperature to 550°C, which is most likely the upper limit of the operating temperature of a stainless steel first wall in a D-T fusion reactor.

Irradiation at 550°C

Figures 1(a) and 1(b) show typical examples of the blisters formed when annealed 304 stainless steel surfaces at 550°C were irradiated with 100-keV $^4He^+$ ions for total doses of 0.1 C/cm² and 0.5 C/cm², respectively. The average diameter of the blisters formed for a total dose of 0.1 C/cm² (Fig. 1a) ranges from 5 to 30 μm. Many of the blisters have ruptured and their skins have exfoliated. Examination of many micrographs show that the blister rupture near the grain boundary is quite frequent. As the total dose is increased to 0.5 C/cm² the blister rupture and exfoliation becomes severe (Fig. 1b). In certain regions three exfoliated skin layers could be seen as indicated by the arrow in Fig. 1b. Approximate values of the erosion rates were determined directly from micrographs such as in Figs. 1(a) and 1(b) by measuring the skin thickness and the area from which the skin has fallen off as described earlier.[4—8] The erosion rate estimated in this way for 100-keV $^4He^+$ irradiation at 550°C is ~0.3 atoms per incident helium ion for the total dose of 0.1 C/cm². The erosion rate for a dose of 0.5 C/cm² was relatively difficult to determine because of multiple exfoliations but the value is estimated to be ~0.5 atoms per incident helium ion.

Figures 1(c) and (d) show blisters formed by irradiation of annealed 304 stainless steel surfaces at 550°C with 0.5-MeV $^4He^+$ ions for total doses of 0.1 C/cm² and 0.5 C/cm², respectively. At this higher projectile energy the average blister diameters range from 5 to 60 μm for doses of both 0.1 C/cm² and 0.5 C/cm², and are larger than those for 100-keV irradiations (Figs. 1a and 1b). No exfoliation of the blister skin is observed up to a total dose of 0.5 C/cm². However, for a total dose of 1.0 C/cm² blister skin exfoliation was observed as will be described later. The blister density increased from ~1×10^5 blister/cm² to ~2×10^5 blister/cm² as the total dose was increased from

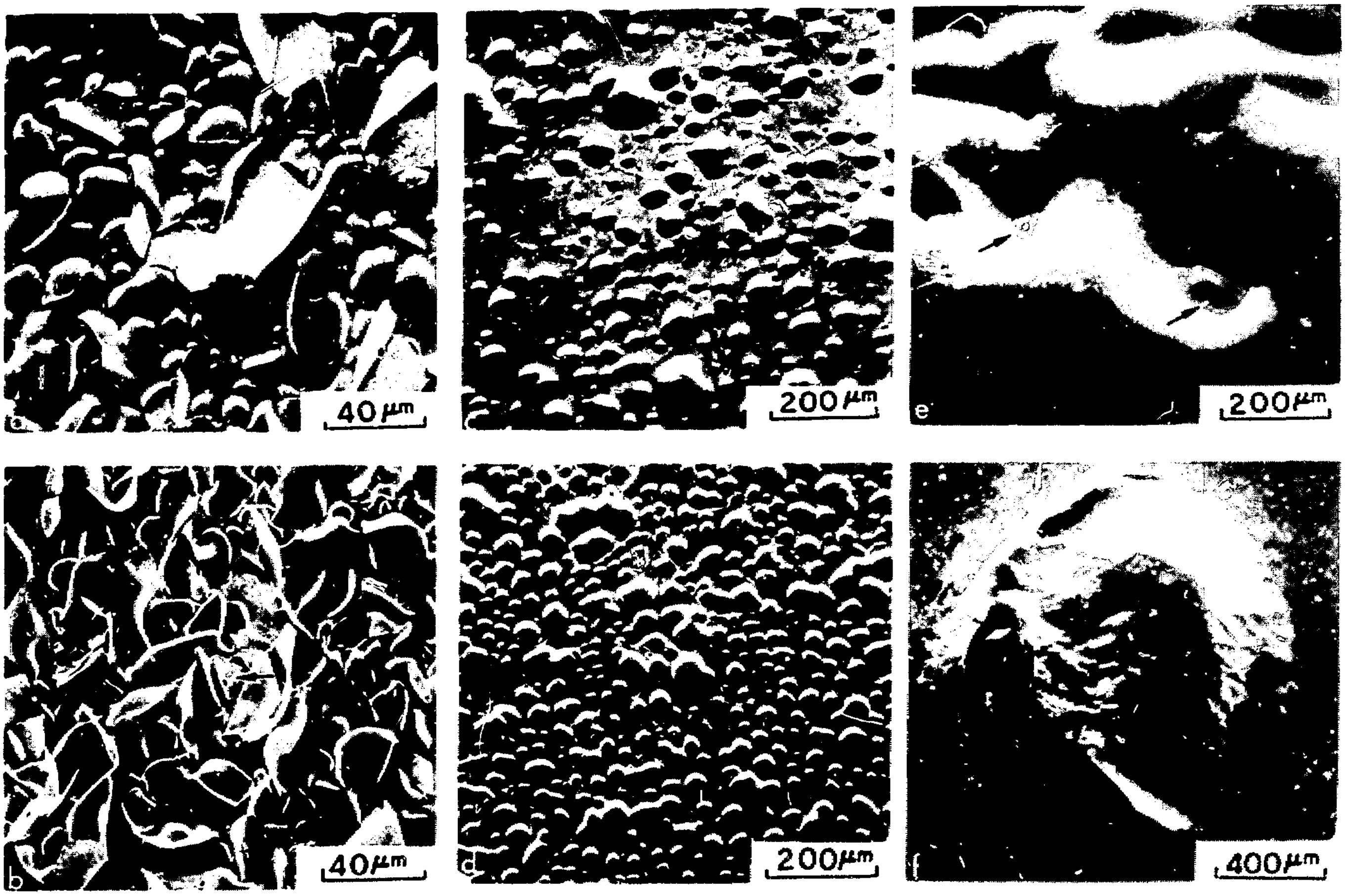

Fig. 1. Scanning electron micrographs (SEMs) of surfaces of annealed 304 stainless steel after irradiation at 550°C, with 100-keV $^4He^+$ ions for total doses of (a) 0.1 C/cm^2 and (b) 0.5 C/cm^2, with 0.5-MeV $^4He^+$ ions for total doses of (c) 0.1 C/cm^2 and (d) 0.5 C/cm^2, and with 1.5-MeV $^4He^+$ ions for total doses of (e) 0.1 C/cm^2 and (f) 0.5 C/cm^2.

0.1 C/cm^2 to 0.5 C/cm^2. The fraction of total irradiated area that is occupied by blisters is ~33% for a total dose of 0.1 C/cm^2 and ~42% for a total dose of 0.5 C/cm^2.

As the projectile energy was increased to 1.5 MeV the blister size increased (Figs. 1e and 1f) in comparison to the 100-keV (Figs. 1a and 1b) and 0.5-MeV (Figs. 1c and 1d) helium ion irradiation runs. This increase in blister size can be understood on the basis of the blistering model suggested in Ref. 5. For a total dose of 0.1 C/cm^2 very large interconnecting blisters formed (Fig. 1e). It is interesting to note that these large interconnecting blisters seem to have left certain grains unaltered (marked by arrows). This could be due to the fact that these grains are oriented in such a way that the helium projectiles have been implanted at a larger depth as compared to the neighboring grains. It is also possible that the helium release from these grains may have been higher than the neighboring grains. For a total dose of 0.5 C/cm^2 almost the entire irradiated area was occupied by one large blister (Fig. 1f). The blister skin has ruptured at several places but there is no large exfoliation of the skin as was observed for 100-keV irradiations (Figs. 1a and 1b).

Irradiation at 450°C

Figures 2(a) and 2(b) show typical examples of blisters formed on annealed 304 stainless steel by irradiation at 450°C with 100-keV $^4He^+$ ions to total doses of 0.1 C/cm^2 and 0.5 C/cm^2, respectively. For a total dose of 0.1 C/cm^2 (Fig. 2a) most of the blisters have ruptured and their skins have exfoliated, exposing the inner surface. At this irradiation temperature of 450°C the skin exfoliation has increased as compared to that at 550°C (Fig. 1a) for the same total dose of 0.1 C/cm^2. As the total dose is increased to 0.5 C/cm^2 the blister rupture and exfoliation becomes more severe (Fig. 2b). At least three exfoliated skin layers can be seen in the area marked by the arrow in Fig. 2(b) as compared to only one in Fig. 2(a). Large portions of the exfoliated skins have completely fallen off at this dose of 0.5 C/cm^2. Approximate values of the erosion rates determined directly from the micrographs such as in Figs. 2(a) and 2(b) gave values of ~1 ± 0.3 and 3 ± 0.5 atoms per incident helium ion for the total doses of

Fig. 2. SEMs of surfaces of annealed 304 stainless steel after irradiation at 450°C, with 100-keV $^4He^+$ ions for total doses of (a) 0.1 C/cm^2 and (b) 0.5 C/cm^2, with 0.5-MeV $^4He^+$ ions for total doses of (c) 0.1 C/cm^2 and (d) 0.5 C/cm^2, with 1.5-MeV $^4He^+$ for total doses of (e) 0.1 C/cm^2 and (f) 0.5 C/cm^2.

0.1 C/cm^2 and 0.5 C/cm^2, respectively. It may be noted that these values are much higher than the erosion rates obtained for irradiation at 550°C with 100-keV $^4He^+$ ions for similar doses.

As the projectile energy is increased to 0.5 MeV, the blister size increases (Figs. 2c and 2d) for the same irradiation temperature of 450°C. For a total dose of 0.1 C/cm^2 the average blister diameter is ~700 µm (Fig. 2c) and a large portion of the irradiated area is occupied by this single blister. A portion of the blister skin has ruptured and fallen off. At the higher dose of 0.5 C/cm^2 large blisters with exfoliated skins were also observed and Fig. 2(d) shows a portion of the exfoliated skin. Here there are three exfoliated skin layers (Fig. 2d) as compared with one for a dose of 0.1 C/cm^2 (Fig. 2c). The erosion rates estimated for 0.5-MeV $^4He^+$ irradiations at 450°C from micrographs such as in Figs. 2(c) and 2(d) gave values of ~0.1 ± 0.05 and 0.45 ± 0.1 atoms per incident helium ion for total doses of 0.1 C/cm^2 and 0.5 C/cm^2, respectively. It may be noted that these erosion rates are lower than those obtained for 100-keV $^4He^+$ irradiations for the same doses and temperature.

On further increasing the projectile energy to 1.5 MeV no blisters were observed (Fig. 2e) for a total dose of 0.1 C/cm^2 but a large blister covering most of the irradiated area (Fig. 2f) was observed for a total dose of 0.5 C/cm^2. At 450°C the threshold dose for blister formation for 1.5-MeV $^4He^+$ ions appears to be greater than 0.1 C/cm^2. The blister formed at a dose of 0.5 C/cm^2 has ruptured (Fig. 2f) at several places in the skin but no large-scale skin exfoliation is observed as seen for 100-keV and 0.5-MeV $^4He^+$ ion irradiations (Figs. 2b and 2d).

Irradiation at 300°C and Room Temperature

Figures 3(a) and 3(b) show typical examples of blisters formed in annealed 304 stainless steel after irradiation with 0.5-MeV $^4He^+$ ions to a total dose of 1.0 C/cm^2 at target temperatures of room temperature and 300°C, respectively. After room temperature irradiation most of the irradiated area was occupied by one large blister (Fig. 3a). The blister skin has ruptured at several places as marked by the arrows. The blisters formed by irradiation at room temperature with 0.5-MeV $^4He^+$ ions but for a lower dose of 0.5 C/cm^2 were very

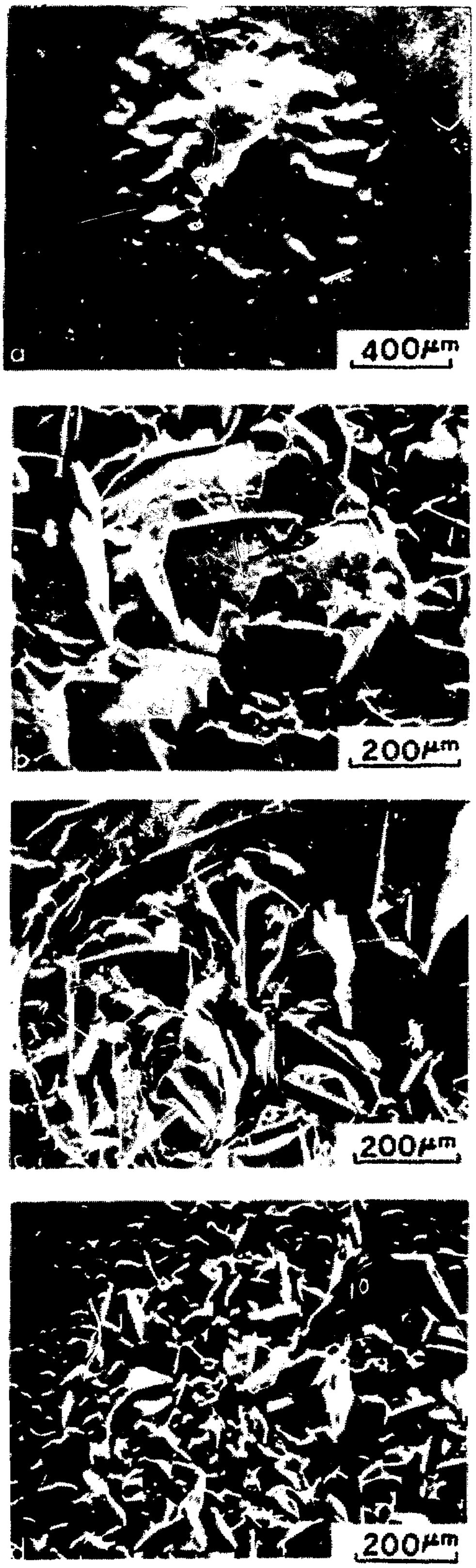

Fig. 3. SEMs of surfaces of annealed 304 stainless steel after irradiation with 0.5-MeV $^4He^+$ for a total dose of 1.0 C/cm^2 at temperatures of (a) room temp. (b) 300°C (c) 450°C (d) 550°C.

similar to those in Fig. 3a. For a still lower dose of 0.1 C/cm^2 no blisters were observed for room temperature irradiation. At the higher irradiation temperature of 300°C and for the same total dose of 1.0 C/cm^2 and a projectile energy of 0.5-MeV, the blister rupture and exfoliation (Fig. 3b) became more severe than the one shown in Fig. 3(a). Two exfoliated skin layers can be readily seen in Fig. 3(b) and a large portion of the blister skin has fallen off. For a total dose of 0.1 C/cm^2 no blisters were observed for 300°C irradiation and for a higher dose of 0.5 C/cm^2 the blisters showed only one exfoliated skin layer. The erosion rates estimated for irradiation temperatures of 300°C are ~0.1 ± 0.02 and ~0.2 ± 0.05 atoms per incident helium ion for total doses of 0.5 C/cm^2 and 1.0 C/cm^2, respectively.

For comparison the blisters formed for a total dose of 1.0 C/cm^2 after irradiation at 450°C and 550°C are shown in Figs. 3(c) and 3(d), respectively. The blister rupture and exfoliation of the skin is considerably higher for 450°C irradiation (Fig. 3c) than for 300°C (Fig. 3b) because there are five exfoliated skin layers for the first case (Fig. 3c) and approximately two in the second case (Fig. 3b). On further increasing the irradiation temperature to 550°C (Fig. 3d) the blister size and the exfoliation of skin is decreased. One observes only one or in certain surface regions two exfoliated skin layers. The erosion rates estimated for these two temperatures of 450°C and 550°C for a total dose of 1.0 C/cm^2 gave values of ~0.8 ± 0.1 and ~0.18 ± 0.07 atoms per incident helium ion, respectively. If one compares the results obtained at 450°C and 550°C for a dose of 1.0 C/cm^2 (Figs. 3c and 3d) with those shown earlier for the lower doses of 0.1 C/cm^2 (Figs. 1c and 2c) and 0.5 C/cm^2 (Figs. 1d and 2d) it is apparent that the blister skin exfoliation increases with an increase in total dose.

Discussion

The results presented here for 304 stainless steel show that increase in projectile energy in the range from 100-keV to 1.5 MeV increases the blister size for irradiation at 450°C and 550°C (Figs. 1 and 2). These results on stainless steel are in qualitative agreement with our earlier results on monocrystalline and polycrystalline niobium.[5,9] With increasing projectile energy the range of the projectiles increases and so most of the helium ions are implanted at a greater depth from the surface. This favors the formation of large blisters as has been discussed earlier.[5,9]

It has been observed earlier for niobium and vanadium[5,6] that the critical dose for blister formation is strongly dependent on irradiation temperature and projectile energy. For example for annealed niobium irradiated at room temperature, no blisters were observed[4] for a total dose of 0.1 C/cm^2, but blisters readily formed at 900°C [5,6] for the same dose. The present results on stainless steel reveal a qualitatively similar temperature dependence. For example, for 0.5-MeV helium projectiles, no blisters were observed for a dose of 0.1 C/cm^2 for irradiations at room temperature and 300°C, but blisters readily formed at 450°C and 550°C for the same total dose. For 1.5-MeV helium projectiles the "critical dose" for blister appearance is larger than 0.1 C/cm^2 even for an irradiation temperature of 450°C (see Fig. 2c). It should be noted that the determination of critical dose for blister appearance depends on the instrument resolution and in the present experiments the resolution of the scanning electron microscope used was ~200 Å. The dependences of critical dose for blister appearance on irradiation temperature and projectile energy are closely related to the temperature dependence of the yield strength of the material and to the increase in blister skin thickness with increasing projectile energy.

One important parameter for the design of the first wall of a fusion reactor is the rate at which the wall is eroded by blister formation and rupture. The erosion rates that were obtained for various irradiation temperatures for 0.5-MeV $^4He^+$ projectiles have been plotted in Fig. 4. The erosion rates are shown as number of atoms from the stainless steel target that were lost per incident helium ion so that these values can be compared directly with the sputtering yield values. It can be seen in Fig. 4 that the erosion rates for total doses of both 0.5 C/cm^2 and 1.0 C/cm^2 have a peak near 450°C ± 20°C. Some recent results by Bauer and Thomas[11] on 316 stainless steel bombarded with 300-keV $^4He^+$ ions show large-scale exfoliation of the blister skin at temperatures ranging from 300 to 500°C, which is qualitatively in agreement with our observations on 304 stainless steel for irradiation with $^4He^+$ ion energies of 100 and 500 keV. Our previous results on vanadium[6, 12] showed a qualitatively similar temperature dependence on blistering, but the actual temperature range for maximum blister rupture was shifted to higher temperatures. This is in part due to the

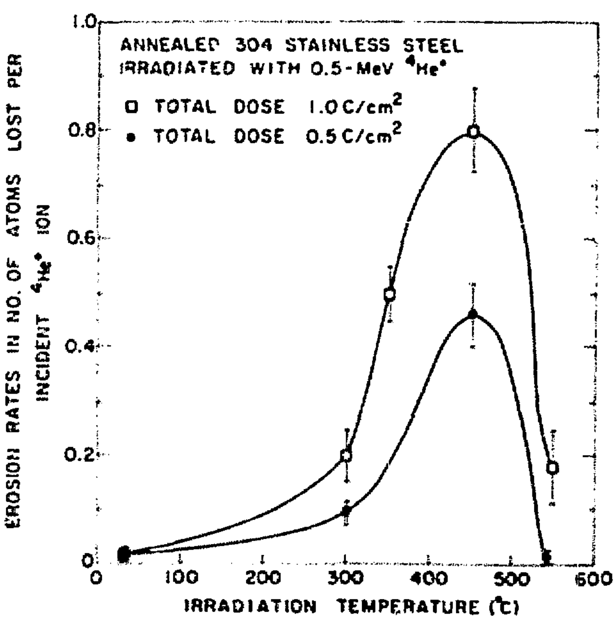

Fig. 4. Erosion rates for annealed 304 stainless steel for different irradiation temperatures.

fact[6] that the yield strength of a material is strongly temperature dependent. For example, for 304 stainless steel the yield strength at 450°C is about half of the room temperature yield strength. This in turn allows the blister skin to be deformed more readily. The increased kinetic gas pressure in the bubble may also aid in this process. At very high temperatures (e.g. niobium at 900°C) significant amounts of helium may be released and a partial annealing of the lattice defects may occur and the degree of blistering is reduced.

The values of erosion rates due to helium blistering in the present experiments are about two orders of magnitude higher than the sputtering yields under similar irradiation conditions. For example, calculation of sputtering yield for iron for irradiation with 0.5-MeV $^4He^+$ ions, using theory of Goldman and Simon[14] gives a value of ~0.002 iron atoms per incident helium ion. The sputtering yields for stainless steel irradiated in the temperature range 300–550°C with 0.5-MeV $^4He^+$ ions are expected to be within a factor of two the same as the value quoted above.

In order to estimate the annual thickness loss of stainless steel first wall due to helium blistering, the flux and energy of the helium projectiles to the first wall has to be known. If one assumes 0.5-MeV $^4He^+$ ions, and a flux of 1×10^{13} projectiles/(cm^2-sec) the erosion rates at 450°C will be ~0.02 mm per year. For 100-keV $^4He^+$ ions the erosion rates can be ~0.09 mm per year corresponding to the erosion rate of ~3 atoms per helium ion for 450°C irradiation (see Fig. 2b). It may be noted that these erosion rate values for 100-keV helium-ion irradiations are substantially larger than the erosion rates obtained for 0.5-MeV helium ion irradiations.

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References

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[1]M. Kaminsky, IEEE Trans. Nucl. Sci., 18, 208 (1971).

[2]M. Kaminsky, in Proceedings of the International Working Sessions on Fusion Reactor Technology, Oak Ridge National Laboratory, Oak Ridge, Tennessee, 1971, U.S. Atomic Energy Commission Report No. CONF-719624 p. 86 (1971).

[3]M. Kaminsky, in Proceedings of the 7th Symposium on Fusion Technology, 24-27 October 1972, Grenoble, France, (Commission of the European Communities, Luxembourg, 1972), p. 41.

[4]S. K. Das and M. Kaminsky, J. Appl. Phys., 44, 25 (1973).

[5]S. K. Das and M. Kaminsky, J. Appl. Phys., 44, 2520 (1973).

[6]S. K. Das and M. Kaminsky, in Defects and Defect Clusters in B. C. C. Metals and their Alloys, Nuclear Metallurgy Vol. 18, edited by R. J. Arsenault, Nat'l. Bureau of Standards, Gaithersburg, Maryland, p. 240 (1973).

[7]W. Bauer and G. J. Thomas, ibid, p. 255.

[8]S. K. Das and M. Kaminsky, in Proceedings of the Texas Symposium on Technology of Controlled Thermonuclear Fusion Experiments and the Engineering Aspects of Fusion Reactors, 20—22 November 1972, Austin, Texas, to be published by National Technical Information Service, Springfield, Virginia.
[9]M. Kaminsky and S. K. Das, Rad. Effects 18, 245 (1973).
[10]Fusion Power: An Assessment of Ultimate Potential, USAEC Report No. WASH-1239, Division of Controlled Thermonuclear Research, U.S. Atomic Energy Commission, Washington, D.C., p. A18 (1973).
[11]W. Bauer and G.J. Thomas, J. Nucl. Mat. 47, 241 (1973).
[12]M. Kaminsky and S. K. Das, Trans. Am. Nucl. Soc. 17, 135 (1973).
[13]K. F. Smith in Reactor Handbook, vol. 1, Interscience Publishers, New York, p. 563 (1960).
[14]D. Goldman and A. Simon, Phys. Rev. 111, 383 (1958).