

Radiation blistering

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The basic physics of radiation blistering and exfoliation is described. Experimental results are reported on the dependence of blistering on the radiation dose, the ion energy, the bombardment temperature, and the target orientation. Data are reported on a synergetic effect on blistering during the simultaneous bombardment of the first wall of a fusion reactor by fast atoms of various isotopes of hydrogen and helium. Methods for reducing blistering are reported. A theory is derived for radiation blistering.

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1. INTRODUCTION

In 1963 Primak¹ and Kaminsky² observed the formation of bubbles at the surfaces of insulators bombarded by H⁺, D⁺, and He⁺ ions. Shortly thereafter, Primak and Luthra³ also observed bubbles at the surfaces of metals (Cu, Ni, and Al) bombarded by H⁺ and He⁺ ions (Fig. 1). The name used for this phenomenon in the Russian-language literature has been borrowed from the English: blistering. As the radiation dose is increased, the bubbles or blisters rupture, and their caps fall off (Fig. 2), and in this manner the blistering results in an erosion of the surface. In certain cases the formation of bubbles is replaced by (or accompanied by) an exfoliation or flaking at the surface (Fig. 3). When exfoliation occurs, the flakes do not have a definite size or shape, in contrast with the blisters, but they are usually larger than the blisters. Furthermore, the erosion in the case of exfoliation is more rapid than that in the case of blistering. As we will see later in this review, the mechanisms for blistering and exfoliation are different, although the two processes are driven by the same event: the penetration of relatively insoluble gases. These two effects are now referred to under different names, blistering and exfoliation, but

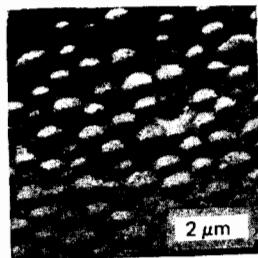


FIG. 1. Blisters at the surface of a vanadium single crystal bombarded by 10-keV helium ions to a dose of $6 \cdot 10^{17} \text{ cm}^{-2}$ (Ref. 67).

the term blistering is frequently used in a more general sense to refer to both blistering proper and exfoliation. We should point out that, in addition to radiation blistering, which occurs during bombardment by fast ions, bubbles can also form at a surface as a result of a treatment of the material in a medium containing gases.^{4,5} The bubbles of nonradiation origin are usually much larger than the radiation-induced bubbles, and their appearance is determined by the treatment method and by the particular material. We will be concerned with only radiation blistering in the present review.

Blistering has been the object of intense research since 1972–73 in connection with the problem of developing a fusion reactor, since the first wall and elements of the divertor of a fusion reactor will be subject to intense bombardment by ions or fast charge-exchange neutrals of deuterium, tritium, and also the helium which is formed in the DT reaction. Blistering is still regarded as one of the most dangerous mechanisms leading to erosion of the first wall of a fusion reactor, since the erosion coefficient of the material during helium blistering and (particularly) exfoliation

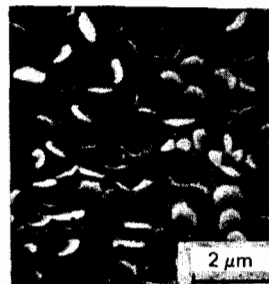


FIG. 2. Blisters with broken caps on the surface of a vanadium single crystal which has been bombarded by 10-keV H⁺ ions to a dose of $6 \cdot 10^{18} \text{ cm}^{-2}$ and then by 10-keV He⁺ ions to a dose of $3 \cdot 10^{18} \text{ cm}^{-2}$ (Ref. 69).

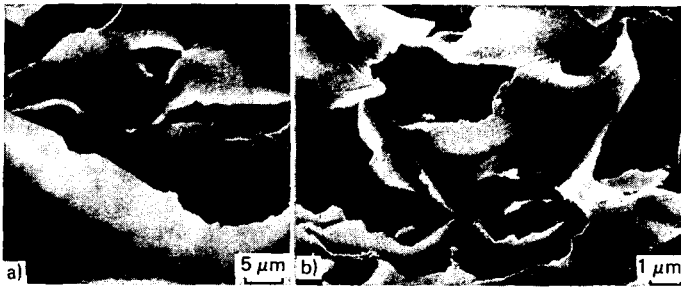


FIG. 3. Exfoliation at the surface of type 0Kh16N15MZB stainless steel at 200°C. a—Dose of 10^{18} cm $^{-2}$; b— $3 \cdot 10^{18}$ cm $^{-2}$.

may be one or two orders of magnitude greater than the coefficient for sputtering of the same material by helium ions and two or three orders of magnitude greater than the hydrogen-ion sputtering coefficient.

A study of blistering is of major interest for both the materials-science problem of fusion reactors and other problems in which materials are bombarded by ions of relatively insoluble gases. The basic characteristics of blistering and exfoliation have now been studied for bombardment by monoenergetic helium ions. Conditions have been found under which the radiation erosion due to helium blistering and exfoliation does not represent an important part of the erosion of the structural elements of a fusion reactor. Radiation blistering is also interesting in itself from the physical standpoint, since the ideas which have emerged from research on this effect have greatly extended our understanding of the interactions of atomic particles with solids.

2. BASIC FEATURES OF BLISTERING AND EXFOLIATION

A certain minimum dose is required for the appearance of blisters or for the beginning of exfoliation in bombardment by ions of relatively insoluble gases. This minimum dose is called the "critical dose for blister formation,"^{6,7} Φ_{cr} . The critical dose increases with increasing ion energy (Fig. 4) and with increasing solubility of the gas. For example, Φ_{cr} is higher for hydrogen than for helium. The critical dose furthermore depends on the particular material, the target temperature, and the crystallographic orientation of the surface, but in each case the dependence is weaker

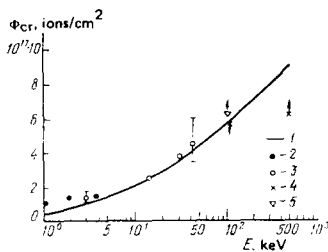


FIG. 4. Dependence of the critical dose for blistering of niobium on the energy of the helium ions. 1—Theoretical curve from Ref. 51; 2–5—experimental data; 2—Ref. 70; 3—Ref. 71; 4—Ref. 6; 5—Ref. 72; (4—niobium which had not been annealed; 2, 3, 5—niobium which had been annealed).

than that on the nature of the ion or the ion energy. The energy distribution of the ions during bombardment by ions with a broad distribution also affects the critical dose.⁸⁻¹⁰

If a surface layer thicker than the ion penetration depth R_p (more precisely, thicker than the cap on the blister) is sputtered off during the bombardment time required to attain Φ_{cr} , the concentration of the gas atoms in the target surface layer will obviously always remain below the critical concentration, and blisters will not form. We thus have a natural condition for blistering:

$$S\Phi_{cr} < R_p N, \quad (1)$$

where S is the sputtering coefficient, and N is the number of target atoms per unit volume. For the light ions H^+ and He^+ , condition (1) holds at all energies, while for heavy ions (argon, krypton, and xenon), which have high sputtering coefficients, condition (1) holds only at sufficiently high energies (100–1000 keV), at which R_p becomes large and S small. Interestingly, when a copper single crystal is bombarded by 20-keV neon ions,¹¹ condition (1) holds only if the ions enter the crystal under channeling conditions; under such conditions, the ion penetration depth R_p is at a maximum, while the sputtering coefficient S is at a minimum.

The nature of the surface erosion—blistering or exfoliation—is determined primarily by the target temperature during the bombardment.^{12,13} At low temperatures, $T_{bomb} < T_1 \approx 0.1-0.2T_m$ (T_m is the melting point), blisters with a definite diameter form. In the temperature interval $T_1 < T_{bomb} < T_2$, exfoliation occurs. The upper end of this interval is $T_2 \approx 0.4T_m$, and it increases logarithmically with increasing ion energy.¹²⁻¹⁶ In the interval $T_2 < T_{bomb} < T_3$, exfoliation again gives way to blistering; the upper limit of this interval is $T_3 \approx (0.5-0.6)T_m$, and this upper limit also increases logarithmically with increasing ion energy. At $T_{bomb} > T_3$ we find, instead of blisters, a porous surface (Fig. 5), not subject to blistering. Figure 6 shows the dependence of the critical concentration of helium atoms required for the formation of blisters, flakes, or pores on the ratio T_{bomb}/T_m for various metals and alloys.¹²

We see that the critical helium concentration (and also the critical dose Φ_{cr}) is roughly the same for blistering and exfoliation, while the temperature depend-

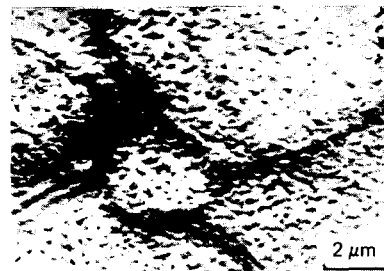


FIG. 5. Formation of a porous surface on type 0Kh16N15MZB stainless steel as a result of bombardment by 40-keV He^+ ions at 600°C ($T_{bomb} > T_3$) (Ref. 13). The bombardment dose is 10^{18} cm $^{-2}$.

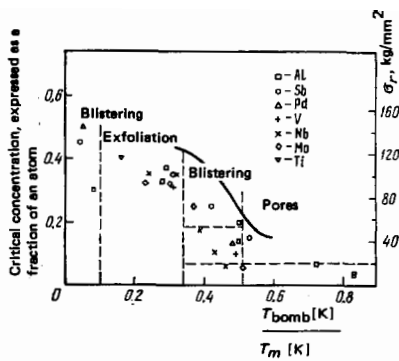


FIG. 6. Temperature dependence of the critical helium concentration required for surface deformation.¹² Solid curve—Temperature dependence of the yield point of steel.

ence of the critical concentration of helium is determined by the temperature dependence of the mechanical properties of the material. Near T_1 and T_2 there are some narrow transition regions in which both blistering and exfoliation occur (see, for example, Fig. 7). Shown for comparison in Fig. 6 is the temperature dependence of the yield point of steel, $\sigma_y(T)$.

Figure 8 shows a plot of the rate of erosion due to helium blistering against the temperature for several metals, according to measurements by the Kaminsky group.⁴ According to Ref. 18, the maximum surface erosion is observed at $T_{\text{bomb}} \approx (0.3-0.4)T_m$. The shift of the erosion peak ($S = 1.75$ atom/ion) toward room temperature in the case of aluminum is a consequence of the low melting point of this metal. We should point out that, according to Ref. 19, multilayer exfoliation of an aluminum surface is observed at a temperature of only 265 K during bombardment by 20-keV He^+ ions. According to that report, five aluminum flakes each 1800 Å thick were observed to peel off at a dose of $1.9 \cdot 10^{18} \text{ cm}^{-2}$. The erosion rate was 1.5 atom/ion.

There is an important difference between the times at which blisters form and at which exfoliation occurs after the critical dose is reached. Thomas and Bauer¹⁷ have observed blistering and exfoliation with a scanning electron microscope, connected directly to the ion accelerator, during the bombardment of a titanium target with 300-keV helium ions. They observed that the blister formation does not occur instantaneously but only as a result of the growth of the bubbles over a time ~ 1 s (after the critical dose Φ_{cr} has been reached), while ex-



FIG. 7. Surface topography of type 0Kh16N15MZB stainless steel after bombardment with 40-keV He^+ ions ($0.3T_m < T_{\text{bomb}} < 0.4T_m$). The bombardment dose is 10^{18} cm^{-2} .

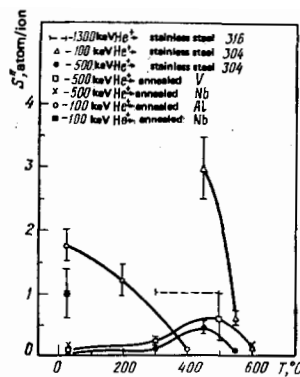


FIG. 8. Temperature dependence of the erosion rate during bombardment of various materials with He^+ ions.⁴²

foliation occurs in ≤ 0.016 s (after Φ_{cr} is reached).

The thickness t of the blister caps and of the pieces which flake off is determined by the ion penetration depth (Fig. 9). At a high ion energy ($E > 40-50$ keV), t is equal to the average ion penetration depth R_p , while at lower ion energies t is several times R_p (Ref. 7). This fact has been explained theoretically, as we will see in the following section.

As we mentioned earlier, the pieces which flake off the surface do not have definite dimensions. Their dimensions are apparently determined by the distances between crystal defects and usually lie in the range 10–100 μm . In contrast with the flakes, the blisters have a quite narrow size distribution. The scatter in the blister diameters d about the average value \bar{d} is usually no greater than \bar{d} . Figure 10 shows some typical blister size distributions.

The average blister diameter is related to the blister cap thickness by

$$d \propto t^\nu. \quad (2)$$

The exponent ν here is approximately²¹ $3/2$, although smaller values are possible: $\nu = 1.15-1.25$ (Ref. 22) or $\nu = 0.9-1$ (Ref. 23). The value of ν depends on the mechanical properties of the target material. For low-temperature blistering ($T_{\text{bomb}} < T_1$) the blister dimen-

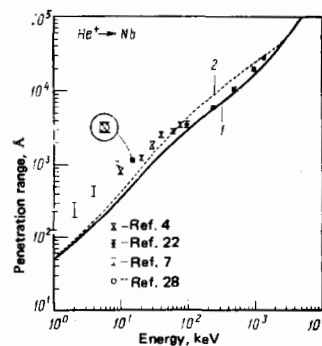


FIG. 9. Dependence of the blister cap thickness t for niobium on the energy of the helium ions.⁴ Shown along with the experimental data from various studies are theoretical results on the average ion penetration range R_p : 1—Calculations by Brice⁵⁶; 2—calculations by the Lindhard-Scharff-Schiott method.²⁰

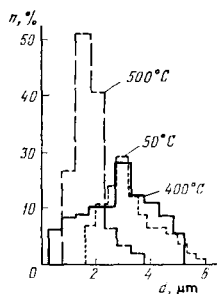


FIG. 10. Blister diameter distributions at various temperatures.

sions are independent of the temperature, while at high temperatures ($T_2 < T_{\text{bomb}} < T_3$) the blister dimensions decrease with increasing temperature (Fig. 10). These facts will be explained in the theoretical section of this review. The surface concentration of blisters (the number of blisters per unit area of the surface) increases with increasing dose—more rapidly, the higher the ion current density. The blisters reach a concentration at which the distance between them is roughly equal to their dimensions, and for this reason the blister concentration is higher at low energies than at high energies. Studies^{24,25} of blistering during oblique incidence of helium ions have revealed a decrease in erosion with increasing angle of incidence. The results found on blistering during oblique incidence of helium ions have certain features in common with the results found on blistering caused by normally incident ions of a lower energy (with a penetration depth equal to the penetration depth of the obliquely incident ions), but there are also some differences in the two cases. The behavior of the cap thickness t for example, can be described well by

$$t \propto \cos \theta,$$

where θ is the ion incidence angle, while the blister dimensions (in the temperature interval $T_{\text{bomb}} < T_1$) can be described approximately by

$$d \propto \cos^{3/2} \theta.$$

The critical dose for blistering Φ_{cr} , on the other hand, remains independent of the angle of incidence up to $\theta \approx 75^\circ$, because the dependence on θ is offset by some of the other factors which determine θ_{cr} ; at large values of θ , Φ_{cr} increases sharply, because of an increase in the ion reflection coefficient (Fig. 11). As the angle of incidence increases, the temperature interval T_1 in

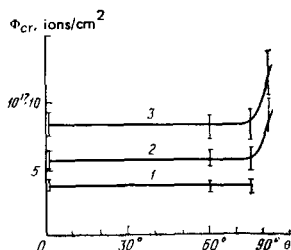


FIG. 11. Dependence of the critical angle for helium blistering of steel on the angle of incidence for various ion energies²⁵; 1—20; 2—40; 3—80 keV.

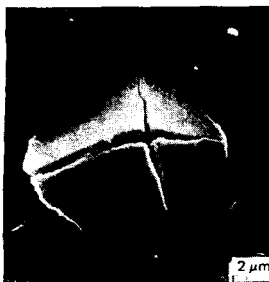


FIG. 12. Blisters with central cap ruptures.

which exfoliation is observed becomes narrower, primarily because of a lowering of T_2 . This effect can be seen most strongly at low ion energies; at $E = 40$ keV, with $\theta = 75^\circ$, for example, there is essentially no exfoliation ($T_1 \approx T_2$).

In contrast with exfoliation, which involves an immediate rupture of a surface layer, the caps or domes on the blisters remain intact at doses up to slightly above Φ_{cr} (Fig. 1). As the dose is increased by a factor in the range of one to two, however, the caps rupture. The caps may rupture either at their periphery (Fig. 2) or at their center (Fig. 12) (Ref. 16). Which type of rupture occurs depends on several factors—the temperature, the current density, etc.—but the plasticity of the material apparently plays the major role. For relatively plastic materials the caps typically rupture at their center, while for more brittle materials a peripheral rupture is more likely. In the case of a peripheral rupture the caps usually drop off, while in the case of a central rupture the dose must be increased even further in order to remove the caps. The removal occurs because the caps roll up, and the surface left after the caps drop off is reminiscent of the surface left by exfoliation. These results occur not only because of the additional penetration of gas into the caps of the blisters but also because of the heating of the cap caused by the ion beam; the cap is in poor thermal contact with the bulk of the sample.

After the blister caps have ruptured, and flakes have dropped off part of the surface, a new generation of blisters and flakes may form. Das and Kaminsky^{26,27} have recorded the exfoliation of more than three layers of niobium ($T_{\text{bomb}} = 400^\circ\text{C}$) each $0.3 \mu\text{m}$ thick and 15 layers of stainless steel ($T_{\text{bomb}} = 450^\circ\text{C}$) each $0.55 \mu\text{m}$ thick (Fig. 13), during bombardment by 100-keV helium

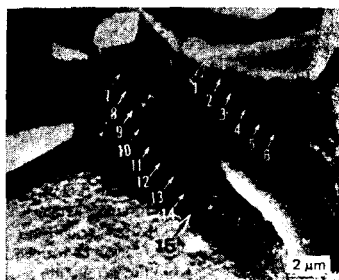


FIG. 13. Exfoliation of 15 layers of niobium bombarded with 100-keV He^+ ions.²⁶

ions with a dose of $1.2 \cdot 10^{20} \text{ cm}^{-2}$. They observed no hint that the exfoliation would come to a halt. Those experiments suggest that the blistering and flaking would repeat endlessly, although Behrisch *et al.*²⁸ have reported that pores appear on a niobium surface when bombarded with a dose of $1.2 \cdot 10^{20} \text{ cm}^{-2}$ by He^+ ions of the same energy, but at a current density of 1 mA/cm^2 . When the pores appear, the surface is no longer subject to blistering. During the bombardment of niobium with He^+ ions at much lower energies²⁹ (5, 10, and 15 keV) only a single generation of blisters was observed, and the blistering ended once a surface layer with a thickness equal to the thickness of the blister cap was sputtered off. Behrisch *et al.*²⁸ studied the dependence of the surface topography on the dose during bombardment by ions at 50 and 100 keV. At the dose of $1.2 \cdot 10^{20} \text{ cm}^{-2}$ they observed the formation of a porous surface, extremely reminiscent of the surface after bombardment at a high temperature ($T_{\text{bomb}} > T_3$). After the surface became porous, it was no longer subject to blistering. The studies which have been made of the dependence of blistering on the ion dose at high doses and at relatively low current densities³⁰ ($30 \mu\text{A/cm}^2$) revealed the following: After the caps detach from the blisters formed by the implantation of He^+ ions at $E = 40 \text{ keV}$, a next generation of blisters forms, but only on that part of the surface which is not covered by remnants of the upper layer; i.e., the new blisters are in a sense nested in the craters left by the blisters of the preceding generation (Fig. 14). The blister diameters become smaller from one generation to the next; the difference is roughly twice the lateral scatter in the ion range, $\sqrt{\Delta R_1^2}$:

$$d_{n-1} - d_n = 2\sqrt{\Delta R_1^2}. \quad (3)$$

This decrease in the blister diameter from one generation to the next has been attributed³⁰ to an edge effect of the ion bombardment near the boundary of a region which is covered by remnants of a layer of preceding generation. Furthermore, there is a minimum possible blister diameter d_{min} (for ions of a given energy).³⁰ It

follows that for each ion energy there is a definite and finite number of blister generations, given by

$$n_{\text{max}} = \frac{d_1 - d_{\text{min}}}{2\sqrt{\Delta R_1^2}}. \quad (4)$$

Once the maximum number of blister generations has formed, the only effect which continues is a sputtering of the surface structure, without the formation of new blisters, according to observations by Gusev *et al.*³⁰ After a layer with a thickness equal to the depth of the blisters of the preceding generation has been sputtered off, a porous surface forms; this porous surface was first observed²⁹ in bombardment by low-energy helium ions, in which case only a single generation of blisters can occur. Behrisch *et al.*²⁸ have calculated the dose required to sputter off a niobium layer with a thickness equal to the blister caps during bombardment with He^+ ions at various energies. Gusev *et al.*³⁰ report $n_{\text{max}} = 3$ for 40-keV helium ions and $n_{\text{max}} = 6$ for 100-keV helium ions. In accordance with the results discussed above, the dose at which blistering stops decreases with increasing angle of incidence,²⁵ θ , because of a decrease in the dimensions of the blisters. The increase in the sputtering coefficient with increasing angle of incidence leads to a decrease in the dose at which a porous surface is formed.

In the case of exfoliation, the craters which remain after the pieces of surface fall off are also "nested" in each other; these craters decrease in size from one generation to the next (Fig. 13). Since the pieces which fall off the surface during exfoliation do not have a definite dimension, however, there is no definite number of possible generations of layers which can be removed. Since the pieces which drop off the surface are large, there may be many exfoliation generations (up to 15 layers), as has been observed in several studies.^{26,28,30,36} The decrease in the size of the pieces from generation to generation, however, shows that again in the case of exfoliation the number of generations of layers which can be removed must be finite [Fig. 14(d)]. Furthermore, there is frequently a transition from exfoliation to blistering after a few generations of layers have been removed.⁷¹ After this transition, the number of possible blister generations is determined by the expression given above.

Blistering and exfoliation are thus transient phenomena. At a sufficiently large dose, an equilibrium porous surface is formed which is not subject to blistering.

To a large extent, the blistering and exfoliation as described above are of a universal nature for different materials during bombardment by helium ions over a broad energy range and with a broad range of current densities. The particular target material, however, and its treatment do affect such characteristics as the critical blistering dose Φ_{cr} and the temperatures (T_1 , T_2 , and T_3) of the blistering-exfoliation-blistering-pores transitions. For annealed materials the critical dose Φ_{cr} is slightly larger than that for cold-rolled materials, while annealed samples exhibit a broader temperature range in which exfoliation is observed, primarily because of a decrease in the transition temperature T_1 (Ref. 17).

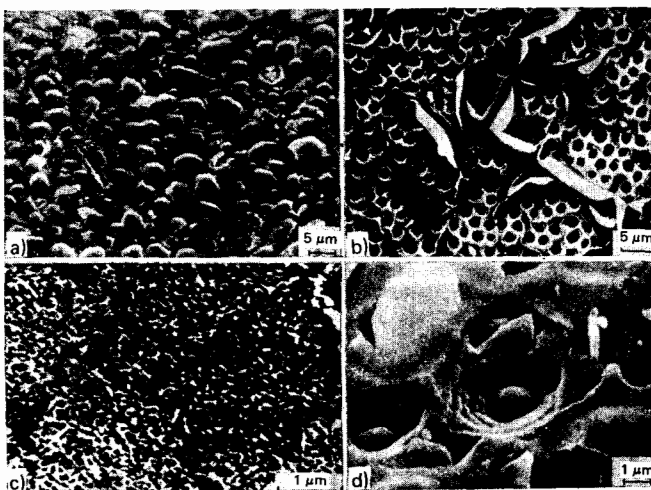


FIG. 14. Change in the surface structure of a chromium-nickel alloy bombarded with 40-keV He^+ ions in various doses $\Phi(\text{cm}^{-2})$: a— $3 \cdot 10^{18}$; b— $6 \cdot 10^{18}$; c— $6 \cdot 10^{19}$ (Ref. 30); d— 6×10^{18} , 100 keV.

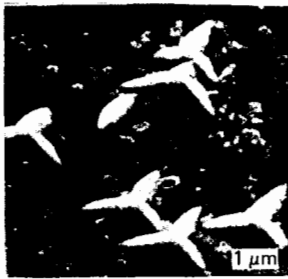


FIG. 15. Blisters on the surface of a niobium single crystal (the $\langle 111 \rangle$ face) produced by bombardment with 500-keV He^+ ions in a dose of $6 \cdot 10^{18} \text{ cm}^{-2}$ (Ref. 34).

The transition temperatures T_1 , T_2 , and T_3 and also the dimensions of the exfoliation area depend on the target material. For example, as the amount of nickel in stainless steel is increased there is an increase in the temperature interval in which exfoliation is observed, and there is also an increase in the fraction of the surface area which experiences exfoliation. In certain cases, we should note, even a small impurity concentration can substantially change the nature of the erosion. For example, a titanium admixture of only 3% in vanadium shifts the temperature of the transition from blistering to exfoliation (T_1) and that from exfoliation to blistering (T_2) by 300°C (Ref. 31). Experiments have shown³² that the blistering of 0Kh16N15M3B steel is reduced by admixtures of manganese, nickel, carbon, and boron ions. The addition of chromium ions, in contrast, increases the surface erosion during blistering.

The nature of the erosion is also affected by the crystallographic orientation. In the bombardment of single crystals, as mentioned earlier, the results depend on whether channeling conditions are satisfied. If channeling conditions are satisfied, the ions penetrate to a greater depth, the thickness of the blister caps increases, and (in accordance with the theoretical interpretation; see the next section) there is a preferential blistering³³ instead of exfoliation. Furthermore, because of the anisotropy of the properties, the blisters on single crystals assume a particular shape for each particular face^{11,34,35} (Fig. 15). The nature of the subsequent erosion of different faces may also be quite different in the case of unoriented bombardment. In experiments³⁶ on vanadium bombarded by 40-keV He^+

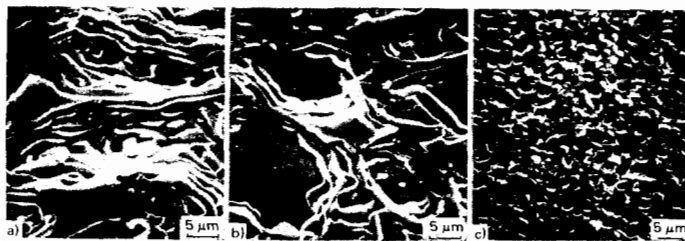


FIG. 16. Surface microstructure of a vanadium single crystal after bombardment with 40-keV He^+ ions in a dose of $3 \cdot 10^{18} \text{ cm}^{-2}$ ($T_{\text{bomb}} = 200^\circ\text{C}$) (Ref. 36). a— $\langle 110 \rangle$ face; b— $\langle 100 \rangle$ face; c— $\langle 111 \rangle$ face.

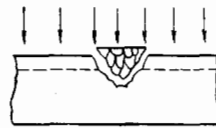


FIG. 17. Failure of a brittle material.³⁷

ions with a dose of $\Phi = 3 \cdot 10^{18} \text{ cm}^{-2}$, for example, a multilayer exfoliation of ten layers was observed on the $\langle 110 \rangle$ face [Fig. 16(a)], and the erosion rate was $S = 10$ atom/ion, while on the $\langle 111 \rangle$ face under the same bombardment conditions three generations of blistering were observed [Fig. 16(c)], and the erosion rate was $S = 0.2$ atom/ion.

Exfoliation has been observed on one face of polycrystalline niobium bombarded by helium ions with a broad energy spectrum with a dose of $\Phi = 10^{19} \text{ cm}^{-2}$ (Ref. 8), while blistering was observed on another face—in the same experiment.

For brittle materials, such as certain types of graphite, the nature of the erosion during the penetration of ions of relatively insoluble gases may be quite different from the blistering and exfoliation typical of metals. In the case of brittle materials, the resulting pressure causes a brittle failure at the surface³⁷ (Fig. 17). Figure 18 shows the surface topography of various types of graphite bombarded with He^+ ions. The nature of the surface erosion of especially pure graphite indicates that in the case of brittle materials the surface layer does not break off along a crack running parallel to the surface; instead, irregular pieces of material break off, and pits and cracks form on the surface. In the case of pyrolytic graphite, which has an anisotropic structure, the damage at the surface is also anisotropic³⁷ [Fig. 18(b)]. Bombardment with 100-keV He^+ ions

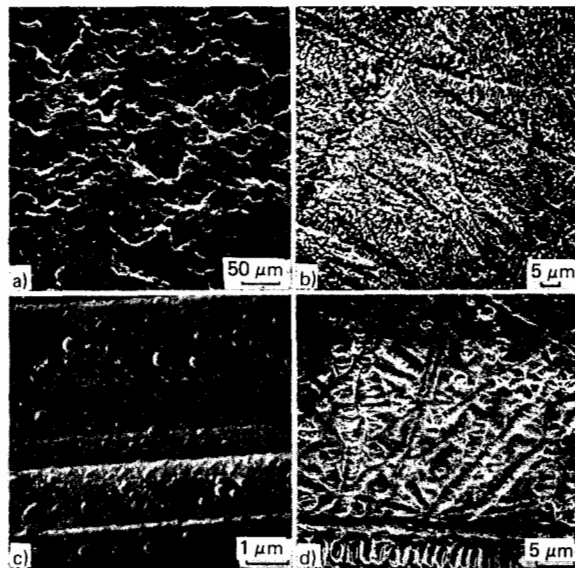


FIG. 18. Surface erosion of various types of graphite bombarded with 20-keV He^+ ions (dose of 10^{18} cm^{-2}). a—especially pure graphite; b—pyrolytic graphite³⁷; c—WCA graphite cloth; d—glassy graphite.

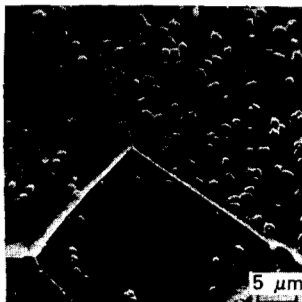


FIG. 19. Surface microstructure of TaC after bombardment with 40-keV He⁺ ions at 500°C (dose of $3 \cdot 10^{18}$ cm⁻²).

with a dose of $10^{18} - 5 \cdot 10^{18}$ cm⁻² has resulted in the exfoliation of a previously polished surface of pyrolytic graphite.³⁸ For certain brittle materials, on the other hand—tantalum carbide (Fig. 19) and niobium carbide, for example—the blistering caused by helium ions is of the same nature as on metal surfaces.³⁹

At the customary current densities, $j = 1 - 10^3$ μA/cm², the dependence of the blistering on the current density is comparatively weak⁴: With increasing j there is a slight decrease in the critical dose for blistering, while there are increases in the size of the blisters, their concentration, and the temperature interval in which exfoliation is observed. This effect, however, has sometimes been attributed⁴⁰ to an additional heating of the surface by the ion current.

In describing blistering and exfoliation up to this point we have been dealing primarily with helium blistering. Because of the high solubility of hydrogen and its rapid diffusion in metals in comparison with helium, blistering is much less apparent in bombardment with hydrogen ions. The critical blistering dose Φ_{cr} for hydrogen blistering is much higher than that for helium blistering, usually lying in the range $10^{18} - 10^{19}$ ions/cm² (cf. Fig. 4, which shows values of Φ_{cr} for helium blistering). Hydrogen blistering is observed primarily for metals which dissolve hydrogen poorly (Al, Mo, Ni, and stainless steel, among others^{37,40-41}). Metals which dissolve hydrogen well generally do not exhibit hydrogen blistering. Hydrogen blistering has been observed in vanadium.^{37,43} Exfoliation does not result from hydrogen bombardment; the only result is the formation of blisters. In most cases, the hydrogen blisters do not break open⁴⁴ (Fig. 20).



FIG. 20. Hydrogen blistering on an aluminum surface after implantation of 20-keV He⁺ ions (dose $D = 10^{18}$ cm⁻²) (Ref. 36).

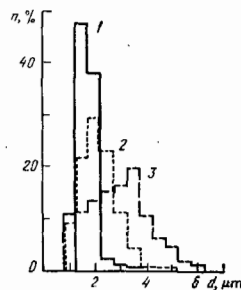


FIG. 21. Diameter distribution of the blisters for various bombardment sequences under the conditions $\Phi^{D^+} = 6 \cdot 10^{18}$ cm⁻², $E^{D^+} = 20$ keV, $\Phi^{He^+} = 10^{18}$ cm⁻², $E^{He^+} = 40$ keV. 1—Sequential bombardment, first by D⁺ ions and then by He⁺ ions; 2—simultaneous bombardment with D⁺ and He⁺ ions; 3—bombardment with helium ions.

Since the materials in fusion reactors will be subjected to simultaneous bombardment by helium ions and ions of deuterium and tritium, a study of blistering during simultaneous bombardment with D⁺ and He⁺ ions is particularly worthwhile. Even the first experiments⁴⁵ on the simultaneous bombardment of nickel with D⁺ and He⁺ ions revealed that the critical blistering dose was lower than that for purely helium blistering. More-detailed studies⁴⁶ of blistering during simultaneous and successive bombardment of stainless steel with D⁺ and He⁺ ions [the D⁺ dose was $6 \cdot 10^{18}$ cm⁻², and the ion energy was $E = 20$ keV; the He⁺ dose was 10^{18} cm⁻², and the energy was $E = 40$ keV (for this choice of energies, the depth profiles of the D and He are essentially the same)] have shown that helium plays the major role in the development of the blistering, while deuterium causes only some secondary effects (despite the fact that the deuterium dose is six times as great), because of the poorer solubility of helium. The penetration of deuterium does, on the other hand, substantially change the erosion pattern. At the temperatures characteristic of blistering ($T_2 < T_{bomb} < T_3$) the penetration of deuterium (Fig. 21) reduces the size of the blisters; if deuterium and helium penetrate into the target simultaneously, the number of blisters per unit surface area is ~3.5 times as high as that in the case of helium blistering. If deuterium enters the target first, the blister concentration increases only inconsequentially. At the temperatures characteristic of exfoliation ($T_1 < T_{bomb} < T_2$) the simultaneous penetration of D⁺ and He⁺ leads to a sharp increase in the erosion (Fig. 22): Three layers are lost in the course of the exfoliation, in contrast with the single layer in the case of helium bombardment. During successive bombardment with D⁺ and He⁺ ions, in contrast, the exfoliation is replaced by two generations of blisters. During successive bombardment in the opposite order, with He⁺ and then D⁺ ions, a single layer is lost through exfoliation, and then two generations of blisters form. These experiments clearly demonstrate the differences in the blistering during simultaneous and successive bombardment, and they show how complicated the processes are, how sensitive the processes are to the bombardment conditions, and how difficult it is to predict the

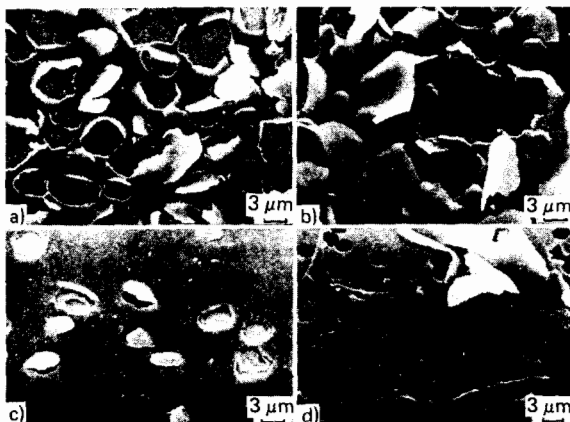


FIG. 22. Surface microstructure of type 0Kh16N15M2B stainless steel after bombardment. a—With He^+ ions; b—simultaneously with D^+ and He^+ ions; c—first by D^+ ions and then by He^+ ions; d—first by He^+ ions and then by D^+ ions.⁴⁶ $T_{\text{bomb}} = 200^\circ\text{C}$, $\Phi_{\text{D}} = 6 \cdot 10^{18}$, $E_{\text{D}} = 20$ keV, $\Phi(\text{He}^+) = 10^{18}$, $E(\text{He}^+) = 40$ keV.

effects under bombardment conditions which have not yet been studied.

3. THEORY

The theoretical work on blistering and exfoliation has focused primarily on the mechanism for the buckling of the target surface layer after a crack has formed below it (Fig. 23). Two mechanisms have been proposed for this buckling.

The first⁴⁷⁻⁴⁹ attributes the buckling to the gas pressure in the cavity of the crack. According to Evans,⁴⁸ for the gas pressure actually to cause plastic deformation (this is the type of deformation which is observed experimentally) of the blister cap, something of the order of 10% of the gas which is introduced into the target during bombardment at the critical dose must be evolved.

According to the second mechanism,^{40,50} the gas pressure serves as only a small nucleating force and may be low, while the buckling results primarily from an internal lateral stress in the surface layer, i.e., in the blister cap. This stress is caused by the intrusion of relatively insoluble gases. Risch *et al.*⁴⁰ and Eer Nisse and Picraux⁵⁰ work from the fact from the theory of plates that the displacement of the center of a circular plate is

$$w = \frac{w_0}{1-\alpha}, \quad \alpha = \frac{Sd^2}{kN}, \quad (5)$$

where w_0 is the displacement in the absence of an inter-

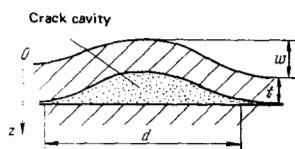


FIG. 23. Buckling of a surface layer of the target.

nal lateral stress (with $S = 0$), d is the plate diameter, $N = Et^3/12(1-\nu^2)$ is the stiffness of the plate, t is the thickness of the plate, E is the Young's modulus, ν is the Poisson ratio, and k is a coefficient which depends on the boundary conditions ($k = 5.6$ for an edge resting on a support and $k = 19.6$ for a pinned edge). The integrated stress is

$$S = \int_0^t \sigma_{rr}(z) dz, \quad (6)$$

where $\sigma_{rr}(z)$ is the radial (lateral) stress at depth z . It can be seen from (5) that the plate becomes unstable even at a small value of w_0 , and there is a transition to plastic deformation when the denominator in (5) reaches zero. It follows that at a constant value of S the diameter of the blister and its thickness are related by

$$d \propto t^{3/2}, \quad (7)$$

and this result has been observed experimentally,²¹ as we mentioned earlier. On this basis it has been concluded that the lateral-stress mechanism is responsible for the formation of blisters with a definite diameter.

In the absence of a lateral stress ($S = 0$) the gas which enters the crack cavity creates at the boundary of the crack a lateral moment which tends to bend the upper layer. The magnitude of this moment is⁴⁰

$$M_r = \sqrt{n_g RTN}, \quad (8)$$

where R is the universal gas constant, T is the temperature, N is the stiffness of the upper layer, and n_g is the number of gas atoms which evolve into the cavity per unit surface area. As can be seen from (8), the moment M_r does not depend on the blister size d ; in other words, the detachment of the surface layer, once begun, will continue to its conclusion without stopping. The situation is similar to exfoliation involving the detachment of large pieces of the surface. Risch *et al.*⁴⁰ have accordingly suggested that exfoliation results from the breakoff of a surface layer caused by gas pressure in a crack cavity.

These studies did not, however, take up the process by which the crack forms and which would have to precede the buckling of the blister caps and the exfoliation. Furthermore, it is not clear under which conditions the lateral-stress mechanism will be predominant and under which the predominant mechanism will be the removal of a surface caused by gas pressure in a crack cavity.

The formation of a crack as a result of a failure of the material between small gas bubbles (~ 10 – 100 Å) in which there is a high pressure was studied first by Evans⁴⁸ and then, in more detail, by Martynenko.^{51,52} Martynenko⁵² also found the conditions under which each of the two mechanisms for the buckling and detachment of the upper layer would be predominant.

It was suggested in Refs. 51 and 52 that gas atoms¹⁾ and the vacancies produced during ion bombardment of

¹⁾ In hydrogen blistering, the hydrogen atoms which enter bubbles form H_2 molecules. When hydrogen dissolves from a bubble into the metal matrix, a dissociation energy must be expended in addition to the energy required to dissolve the atomic hydrogen.

the target would coalesce to form bubbles 10–100 Å in size. Such bubbles have in fact been observed⁵³ in electron-microscope examination of material bombarded with a dose slightly lower than the critical dose for blistering. The depth distribution of such bubbles was in fact found somewhat later.⁵⁴

Because of the poor solubility of the gas, the pressure in the bubble can reach values $p \approx 0.1E \approx 0.1 \text{ eV}/\text{Å}^3$ (E is the Young's modulus). According to calculations based on the macroscopic model of Ref. 52 for comparatively large bubbles, there may be three or four helium atoms per vacancy in a bubble. Computer calculations⁵ for small bubbles (≤ 4 vacancies) have yielded up to six helium atoms per vacancy. Such pressures with a bubble are withstood by deformation stresses around the bubble, and they create a stress in the bombarded layer. A simplified version⁵¹ of the distribution of the stress σ over the depth z is

$$\sigma(z) = H \left(C_{\text{He}}(z) - C_v(z) \frac{\Omega}{\Omega_{\text{He}}} \right), \quad (9)$$

where H is the dissolution energy of the gas atoms (we will assume He for definiteness), $C_{\text{He}}(z)$ and $C_v(z)$ are the concentrations of the implanted helium atoms and the vacancies which they produce which enter the bubbles, and $\Omega/\Omega_{\text{He}}$ is a parameter, the ratio of the vacancy volume Ω to the volume of the helium atom, Ω_{He} (Fig. 24). A more accurate expression for $\sigma(z)$, containing no adjustable parameters, was derived later,⁵² but we will not write out that expression here because of its length and also because Eq. (9) gives a clearer picture of what is happening.

A transformation from the microscopic picture of gas bubbles in an implanted layer to the macroscopic effects of blistering and exfoliation can be made by adopting the following natural assumptions: We assume that the failure of the material (in regions between bubbles) and the formation of a crack occur at that depth $z = t$ at which $\sigma(z)$ is at its maximum and at that dose at which $\sigma_{\text{max}} = \sigma(t)$ becomes equal to the strength of the material, σ_F . On this basis we can find the critical dose Φ_{cr} for blistering and exfoliation (see Fig. 4, which compares the theoretical calculations with experimental results). The position of the $\sigma(z)$ maximum determines the depth of the crack or the thickness of the blister caps, t . Since the distribution $C_v(z)$ of the vacancies produced by the ions is shifted toward the surface with respect to the distribution $C_{\text{He}}(z)$ of the im-

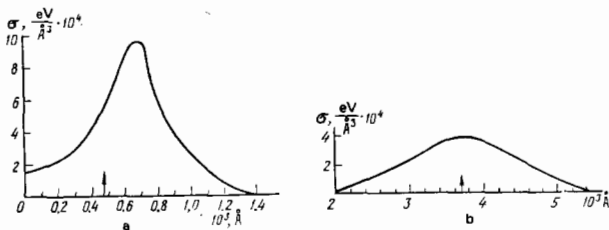


FIG. 24. Stress profiles $\sigma(z)$ in niobium calculated in Ref. 52 for bombardment with He⁺ ions at two energies: a—10 keV; b—80 keV. The arrows show the maxima of the distributions of the implanted helium ions. It is assumed that the blister cap thickness is equal to the position of the $\sigma(z)$ maximum.

planted ions,⁵⁶ the maximum of the $\sigma(z)$ distribution lies at a greater depth than the $C_{\text{He}}(z)$ maximum. In other words, at small depths ($z < t$) there is a large number of bubbles, but the pressure in them is lower (the ratio C_v/C_{He} is higher) than in the bubbles at greater depths ($z > t$). The cap thickness t therefore turns out to be greater than the average ion penetration depth R_p . As shown in Ref. 52, this effect is seen more strongly at low ion energies.

Knowing the depth distribution of the stress, $\sigma(z)$, we can calculate the integrated stress S and also the blister diameter from Eq. (5); in the same manner, we can find the conditions under which the blister caps may buckle as a result of the internal-stress mechanism.

To evaluate the role played by the gas pressure in the crack cavity we must determine just how much gas is evolved from the material into the cavity. This has been done in Ref. 52, under the assumption that the gas is evolved into the crack from a layer adjacent to the crack in which the stress exceeds the yield point when the critical dose Φ_{cr} is reached.

The numerical estimates of Ref. 52 show that it is possible in principle for a surface layer to buckle as a result of both the gas pressure in the crack cavity and the lateral stress which arises in this surface layer. The accuracy of these calculations, however, was such that it was not possible to determine which mechanism would be predominant, i.e., to determine when blisters of a definite size would form and when exfoliation would occur. The theory is far better at predicting the functional relationships. For example, it follows from estimates of the lateral stress S and of the relative amount of gas which is evolved into a crack cavity that exfoliation is more likely at high ion energies, while blistering is more likely at low energies. Furthermore, since S increases only slightly with increasing t , we see why the blister diameter, given by

$$d = \sqrt{\frac{kN(t)}{S}} = \sqrt{\frac{kEt^3}{12(1-\nu^2)S}}, \quad (10)$$

according to (5), is proportional to t^ν , where ν is slightly smaller than 3/2.

Another important parameter in determining the nature of the surface erosion is the target temperature.^{12,13,18} As the target temperature is raised, the yield point σ_T increases, and the $\sigma(z)$ profile broadens because of the diffusion of gas atoms and vacancies. The results are a decrease in the integrated stress S and an increase in the relative amount of the gas which is evolved into the crack cavity, n_a , with increasing temperature. At a certain temperature $T = T_1$, the lateral-stress mechanism therefore gives way to the gas-pressure mechanism, and there is a transition from blistering to exfoliation. A further increase in the temperature leads to an even greater broadening of the $\sigma(z)$ profile and to an enlargement of the plastic-deformation region. When this region becomes comparable to the cap thickness t , there is an abrupt decrease in the stiffness N of the surface layer.

According to Ref. 57, for an incompressible material with $\nu = 1/2$ the stiffness of a plate of thickness t in

which the elastic deformation extends to a depth θ , while in the layer of thickness $t - \theta$ the deformation is plastic, is

$$N = \frac{E}{6} \theta \left(t^2 - \frac{\theta^2}{3} \right). \quad (11)$$

In the limit $\theta \rightarrow 0$ we find $N \rightarrow 0$, and according to (5) there is a decrease in α in this limit, with the result that the lateral-stress mechanism becomes predominant. In other words, a plate with a lower stiffness is deformed by a smaller lateral stress. At a certain temperature $T = T_2$, therefore, we see another transition, from exfoliation back to blistering. At temperatures $T \geq T_2$ the broadening of the $\sigma(z)$ profile results from the diffusion of helium atoms and vacancies, and the width of this profile becomes proportional to $\sqrt{D} = \sqrt{D_0} \exp(-U/2T)$, where D is the diffusion coefficient, T is the temperature, and U is the activation energy for the diffusion. We are thus led to a logarithmic dependence of the temperature T_2 on the cap thickness t or on the ion energy E , in agreement with experiment.¹²⁻¹⁶ In the region $T_2 < T < T_3$ the blister size falls off with increasing temperature according to (10), because of a further decrease in the stiffness N .

At the temperature $T = T_3$, at which the stiffness reaches zero, the blistering stops. This event occurs when the profile $\sigma(z)$ is stepped and begins at the surface. A crack running parallel to the surface does not form here. The distribution of gas bubbles is also stepped and also begins at the surface, so that as the gas bubbles coalesce channels form which extend to the surface and through which gas can be evolved from the target. The surface acquires a porous, spongy structure. The temperature of the transition from blistering to the porous structure also increases logarithmically with increasing energy.

We wish to point out that a similar porous structure forms at low temperatures, at which the distributions $C_{He}(z)$ and $C_v(z)$ are stepped (cf. Figs. 5 and 13c). As we mentioned in the preceding section, this event occurs at high bombardment doses, at which a surface layer with a thickness greater than the thickness of the blister caps is sputtered off. Stepped $C_{He}(z)$ and $C_v(z)$ distributions may also occur during bombardment by ions with a broad energy spectrum.⁸⁻¹⁰

4. METHODS FOR REDUCING THE EROSION OF MATERIALS DURING BLISTERING AND EXFOLIATION

We have seen that blistering and exfoliation are transient phenomena, but at the temperatures characteristic of exfoliation ($T_1 < T_{\text{bomb}} < T_2$) the erosion may continue at doses $> 10^{20} - 10^{21}$ ions/cm², seriously degrading the operation of a fusion reactor. Furthermore, if the surface is renewed during the operation (for example, through the deposition of sputtering products from other parts of the surface⁵⁸ or through a deposition carried out deliberately to compensate for the erosion), the blistering and the exfoliation may continue endlessly. We must therefore seek methods for reducing the erosion which occurs during blistering and exfoliation.

One possibility is to choose an appropriate target working temperature. From the blistering standpoint, we would like to operate either at high temperatures, at which blistering does not occur at all ($T_{\text{bomb}} > T_3$) or at which small blisters form ($T_2 < T_{\text{bomb}} < T_3$), or at low temperatures, at which exfoliation does not occur, and all the blisters have a definite diameter.

Another method for reducing blistering will occur automatically: bombardment with ions having a broad energy distribution. Again in this case the defects and implanted ions have approximately stepped distributions beginning at the surface, or these distributions decay with distance from the surface into the interior. As has been shown theoretically and experimentally,^{9, 28, 29, 59} such distributions lead to the formation of a porous surface which is not subject to blistering.

Kaminsky⁶⁰ has suggested cold rolling as a method for reducing erosion during blistering. Although the critical dose for blistering is reduced by cold rolling, the blisters break open, and their caps drop off, at higher doses than for annealed samples. Furthermore, the temperature interval in which exfoliation is observed is narrower for cold-rolled samples. On the whole, cold rolling reduces the erosion rate.

Blistering can be suppressed substantially or completely by special preparation of the material. If the target is a porous material, prepared by sintering a powder, for example, and if the distance between pores is smaller than the minimum blister diameter d_{min} (Sec. 3), the conditions become favorable for evolution of gas at the pores, and even if microscopic cracks do form in the material no blistering can occur, and the gas is evolved along a crack into a pore. In fact, blistering is not observed for samples of sintered aluminum and beryllium powders.^{61, 62} We might also note that porous materials have a lower sputtering coefficient.^{63, 64} However, the use of porous materials may be restricted by difficulties in evacuation and by the degradation of the thermal conductivity. Furthermore, porous materials will not be effective for the renewal of a surface by deposition.

From the physical standpoint, the most interesting method for suppressing blistering is to use special additives in the material. If, for example, the additives act as traps for helium atoms, and if the energy of a helium atom at a trap is much lower than in an interstitial position, the net result is a sort of quasisolubility of helium, which reduces the stress in the layer with the implanted helium ions and which suppresses blistering. If, on the other hand, the additives act as traps for vacancies and occupy them, thereby reducing the number of vacancies in helium bubbles, the pressure in the corresponding layer rises, and blistering intensifies. Experiments³² have in fact demonstrated a decrease in the blistering of type 0Kh16N15M3B steel when doped with manganese, nickel, carbon, and boron atoms. Doping of the same steel with chromium ions, in contrast, increases the erosion due to blistering. This result shows that doping can substantially reduce, and in some cases completely suppress, blistering in alloys.⁶⁵

The intense research on blistering and exfoliation in recent years has shown that the erosion of the first wall in fusion reactors because of these radiation effects will be less than the erosion caused by sputtering,^{66,67} because the fluxes of helium atoms are much lower than those of deuterium and tritium atoms and also because of the unavoidable suppression of blistering and exfoliation.

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