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RADIATION BLISTERING IN METALS AND ALLOYS

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ABSTRACT

Radiation blistering in solids has been identified as a process leading to damage and erosion of irradiated surfaces. Some of the major parameters governing the blistering process in metals and some metallic alloys are the type of projectile and its energy, total dose, dose rate, target temperature, channeling condition of the projectile, orientation of the irradiated surface plane, and target material and its microstructure. This paper reviews both experimental results and models proposed for blister formation and rupture. The blistering phenomenon is important as an erosion process in applications such as fusion reactor technology (plasma-wall interactions) and accelerator technology (erosion of components and targets). The review includes a description of methods for the reduction of surface erosion caused by blistering.

I. INTRODUCTION

The irradiation of metal surfaces with energetic particles causes a variety of surface phenomena such as physical and chemical sputtering, secondary electron emission, x-ray emission, optical photon emission, release of absorbed and adsorbed gases, backscattering of particles, trapping and re-emission of trapped particles, and radiation damage (For recent reviews see Refs. 1-4 and related articles in the proceedings of this conference). If such energetic particles penetrate a metal lattice they may displace lattice atoms

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from their sites and create vacancies and interstitials. When the incident particles have slowed down sufficiently they may be trapped in the lattice either interstitially or substitutionally.

Depending on the type of both implanted particle and the surrounding lattice atoms and on the impact parameters, the interaction between them may be either physical or chemical or both. For example, during the irradiation of titanium with hydrogen isotopes formation of metal hydrides has been observed^(5,6) leading to a partial trapping of the incident particles. Such chemical trapping processes are discussed elsewhere in these proceedings.⁽⁷⁾ In other cases the interaction between the implanted atoms and the lattice atoms may not lead to strong chemical bonds and compound formation, but is more physical in nature. For example, when the implanted particles are inert gas atoms, they can combine with the vacancies created by lattice displacements and nucleate as gas bubbles.

The formation of gas bubbles has been observed by Barnes et. al.⁽⁸⁻¹⁰⁾ after annealing of copper and aluminum which had been irradiated with 38-MeV α particles to total doses ranging from 7×10^{16} to 1.7×10^{17} α particles cm^{-2} . These gas bubbles were observed in the bulk material. If the gas bubbles form in near-surface regions and the gas pressure is high enough, bubbles may plastically deform the surface layers above them and when the deformation is extreme, the surface layers may rupture. This phenomenon of surface deformation associated with gas bubbles formed due to irradiation has been called radiation blistering.^(11,12) It is of historical interest to note that an early indication of this phenomenon was obtained by Stark and Wendt⁽¹³⁾ in 1912, when irradiating insulating materials such as calcite and calcium fluoride with ~ 10 -keV hydrogen ions. However this work went unnoticed, and in the early sixties Primak^(12,14) and Kaminsky⁽¹¹⁾ first reported experimental evidence for blister formation in insulators irradiated with 100-140-keV protons and helium ions and in metals irradiated with

125-keV deuterons, respectively. Primak et. al.⁽¹⁵⁾ first observed flat-bottomed pits in silicon irradiated with 100-keV protons, using optical interferometry for surface examination. The pits were due to ruptured blisters. Kaminsky⁽¹¹⁾ first reported mass spectrometric observations of gas bursts from ruptured blisters during irradiation of copper with 125 keV deuterons. He also observed settling of surface regions where blisters had exploded (using surface replica techniques in conjunction with transmission electron microscopy). The number of gas bursts correlated well with the number of pits observed on the surface, indicating that the pits were indeed due to the rupture of gas bubbles.

After the early work by Kaminsky and Primak et. al. there was some interest in studying the effect of space radiation on materials because a major component of space radiation consists of low energy protons from cosmic rays and solar winds.⁽¹⁶⁾ During the last five years the interest in the radiation blistering phenomenon has increased greatly due to its importance in the operation of controlled thermonuclear fusion devices and reactors.⁽¹⁷⁻²⁰⁾ In a fusion reactor having D-T plasma, energetic D, T, and He particles (formed by the D-T fusion reaction) can leak out of the confining magnetic field either as charged particles or as neutrals (formed for example by charge exchange near the plasma edge) and strike the surfaces of reactor components and form blisters. A detailed discussion of this is given in the review paper by Kaminsky in these proceedings. More recently, interest in radiation blistering has also developed in connection with other applications such as possible erosion of containers of short-lived transuranic nuclides (e.g. ^{242}Cm or ^{252}Cf) due to exposure to high alpha-radiation fluxes over extended periods of time.⁽²¹⁾

It may be pointed out that the radiation blistering phenomenon should be distinguished from other types of blistering phenomena observed in metals caused by entirely different processes. For example, blistering is quite common in aluminum castings⁽²²⁾ and it arises from exposing molten aluminum to a gaseous environment containing hydrogen. This is due to the relatively high solubility of hydrogen in molten aluminum and the low solubility in the solid aluminum (the ratio of solubility in the liquid phase to that in the solid phase at the freezing point is approximately twenty to one⁽²³⁾) resulting in the precipitation of excess hydrogen from the metal lattice. Blister formation has also been observed in silver annealed first in an environment containing a high partial pressure of oxygen and then in an environment containing high partial pressures of hydrogen.⁽²⁴⁾ In hydrogen embrittlement studies, blisters have been observed in iron electrolytically charged with hydrogen.^(25,26)

In the following we will discuss first some of the general aspects of the experimental techniques used in radiation blistering studies. Then we will describe the experimental results obtained for various target-projectile systems under different irradiation conditions. The description will be limited to metals and alloys. Radiation blistering in nonmetals is discussed in the paper by Thomas et. al. in these proceedings.⁽²⁷⁾ This review does not include data which have been reported after October 1975.

II. GENERAL ASPECTS OF EXPERIMENTAL TECHNIQUES

In order to detect even small changes in the surface topography due to radiation blistering it is often desirable to start with a high degree of optical finish on the surface by mechanical polishing,^(28, 29) electropolishing,⁽³⁰⁾ chemical polishing⁽³¹⁾ or by some combinations of these three processes. Irradiations of the targets are normally done under high or ultrahigh vacuum

conditions to avoid serious surface contamination.^(28, 30, 31) Various types of ion accelerators have been used for irradiation of the targets. Duoplasmatron sources have been used for the low energy range (1-30 keV)⁽²⁸⁾, Cockroft-Walton generators for the medium energy range (25-500 keV)^(12, 14, 15), and Van de Graaff accelerators for high energy (100-2000 keV)^(10, 11, 14, 16) irradiations. The facilities used for the production and manipulation of ion beams for implantation studies have been the subject of many recent reviews^(2, 3) and will not be discussed here.

In certain blistering studies the gas released from irradiated targets has been measured mass spectrometrically by Kaminsky⁽¹¹⁾, Daniels⁽³²⁾, Bauer et. al.,^(29, 33, 34) and Erents and McCracken⁽³⁵⁾. There are also other studies (not connected with radiation blistering studies) on gas released from surfaces during ion irradiation and during high temperature annealing after ion implantation, and reviews can be found elsewhere.^(2, 4)

The surface topography after irradiation can be examined by a variety of techniques. No attempt will be made here to review all the available techniques for examining surface features. In the early work on radiation blistering the irradiated surfaces were examined by optical microscopy^(12, 15), interferometric techniques^(12, 15), and by using surface replicas in transmission electron microscopes^(11, 16). More recently and in most of the studies to be reviewed here, scanning electron microscopy has been used. Transmission electron microscopy (TEM) has been used⁽³⁶⁻³⁹⁾ to study gas bubble and blister formation in near-surface regions. Most examinations of the blistering of irradiated surfaces have been done after irradiation. Blewer and Maurin⁽⁴⁰⁾ used a hot stage in a scanning electron microscope to make in-situ observations of blister formation during heating of thin films of rare earth metals after helium-ion implantation.

Thomas and Bauer⁽⁴¹⁾ have recently constructed a scanning electron microscope facility for observation of surfaces undergoing ion bombardment. However, in such in-situ observations the secondary-electron emission during ion bombardment constrains one to use the backscattered electrons for imaging, and this limits the resolution.

Roth et. al.^(31, 42) have used Rutherford backscattering techniques to study blister formation in monocrystalline niobium surfaces. They derived information about the thickness of the misaligned region (which they equate with the thickness of the blister skin) from dechanneling measurements.

For an understanding of the basic mechanism of blister formation it is important to know the depth profile of the implanted gas. Backscattering techniques have been used⁽⁴³⁻⁴⁸⁾ for depth profiling of low-Z implanted gases in metals, and this technique is reviewed elsewhere in these proceedings.⁽⁴⁸⁾ Nuclear reaction techniques have been used⁽⁴⁹⁻⁵²⁾ to measure the depth profiles of implanted gases in metals, and discussions on this topic can be found in the papers by Overley et. al.⁽⁵³⁾ and Terreault et. al.⁽⁵⁴⁾ in these proceedings. More recently, a very good depth resolution in the 20-30-Å range has been claimed for ^3He implanted in niobium using the $d(^3\text{He}, p)^4\text{He}$ reaction.⁽⁵⁵⁾

III. BLISTERING PARAMETERS

For a description of the various features of radiation blistering the following terms are commonly used: (1) the "blister diameter" and the "distribution of blister diameters" [in cases where the blisters are irregular in shape an "average blister diameter" is given⁽³⁰⁾ which is defined as the diameter of a circle having approximately the same area as the blister] (2) the "blister height" and the "distribution of blister heights"; (3) the number of blisters which are visible per unit irradiated area (often called "blister

density"), [this number depends strongly on the resolution of the instrument used for surface examination (e.g. whether a scanning electron microscope or an optical microscope is used)]; (4) the fraction of the total irradiated area occupied by blisters (often called "degree of blistering"); (5) the "Blister shape"; (6) the "blister skin thickness"; and (7) the amount of blister skin material lost due to exfoliation of blister skin (this quantity has been often expressed as "erosion yield" which is equal to the number of target-atoms lost due to blister skin exfoliation per incident projectile ion). Some authors^(56, 57) make a distinction between "surface bubbles" which refer to more circular surface features and "blisters" which refer to irregular surface features. In the present discussion no such distinction will be made and all types of surface features (irrespective of their shape or size) resulting from surface deformation due to gas bubbles will be referred to as blisters. Many studies have been conducted to determine how one or several of the blistering features listed above depend on various parameters which are related to the target material and to the irradiation conditions chosen. Table 1 lists some of the major parameters which appear to affect radiation blistering. Here it can be seen that some of the parameters are more target related, some are more projectile related, and some are dependent on the target-projectile combinations chosen. Many of the parameters listed in Table 1 are interdependent with the other parameters listed. For example, the yield strength of the target is strongly dependent on the type of target, target temperature, and the microstructure. At this time, the relative importance of many of the parameters listed in Table 1 on the blistering of metals and alloys has not been fully established.

In the following, a description of the major results on the importance of certain parameters on the radiation blistering of metals and alloys will be given. Some recently developed models will also be discussed.

IV. PROJECTILE-TARGET SYSTEMS

Most of the blistering studies on metals and alloys have been conducted with light projectiles such as H, D, and He ions, mainly because of the recent strong interest in the blistering effect in connection with the controlled thermonuclear fusion program.⁽¹⁷⁻²⁰⁾ Many of the target materials that have been investigated in recent years are structural materials proposed for use in controlled thermonuclear fusion devices.

Very little data are available for materials irradiated with heavier ions such as Ar⁺, Xe⁺, etc. A list of radiation blistering studies for various target metals and alloys which have been irradiated with hydrogen-isotope ions (e.g. H⁺, H₂⁺, D⁺) is given in Table A1 in the Appendix. A similar list is given in Table A2 in the Appendix for various target materials which have been irradiated with helium ions. In both Tables A1 and A2 some of the irradiation conditions are also listed. More metals and alloys have been investigated for helium-ion irradiation than for hydrogen-isotope-ion irradiation. One notices that for irradiations with hydrogen-isotope ions, the ion energies range from a low value of 10 keV for certain metals (Al and Nb) to a high value of 3.8 MeV for monocrystalline Sn (100). For helium-ion irradiations the ion energies range from a low value of 1 keV [for Nb and Nb (100)] to a high value of 5.8 MeV (for Pd). Two additional studies are listed in Table A2 where Type 304 stainless steel and platinum were irradiated with α particles from a 50% pure ²⁴²Curium-oxide source with an energy of 6 MeV.⁽²¹⁾ The total dose values reported for both hydrogen-isotope-ion and helium-ion irradiations range from 1.4×10^{16} ions cm⁻² [for He⁺ on Nb (100)] to 7×10^{20} ions cm⁻² (for He⁺ on polycrystalline niobium). The target temperatures range from -196°C (for H⁺ on Al) to 1327°C (for He⁺ on Mo).

Solubility and diffusivity of the implanted gas in metals are two of the important parameters affecting the blistering process (Table 1). In general, hydrogen isotopes have higher solubility and diffusivity in many metals than inert gases such as helium, and thus differences in blistering behavior for irradiation with hydrogen-isotope ions and helium ions are to be expected. An example⁽⁵⁸⁾ of the difference in the blistering behavior of niobium for helium-ion and deuteron irradiation is shown in Figures 1 (a) and 1 (b). The blisters formed during irradiation at 700°C with 250-keV $^4\text{He}^+$ ions (Fig. 1a) after a total dose of 6.2×10^{18} ions cm^{-2} are of two sizes. The larger size blisters have an average diameter of 5-8 μm , while the smaller size blisters have an average diameter of $\sim 0.5\mu\text{m}$. The blisters formed during 250-keV D^+ ion irradiation at the same temperature of 700°C after an even higher dose of 1.3×10^{19} ions cm^{-2} are also of two types, but are much smaller in size (Fig. 1b) than the helium blisters. The larger size deuterium blisters are more elongated, and their average length ranges from 1 to 3 μm , while the smaller size blisters have an average diameter of $\sim 0.15\mu\text{m}$. The observation of different blister shapes in Figs. 1 (a) and 1 (b) can be related to the orientation of the grains with respect to the ion beam, as will be discussed later in Sec. XI. Most of the deuterium blisters are unruptured (Fig. 1b), whereas some of the large helium blisters have ruptured. The observation that blister size for deuteron irradiation is smaller than for helium-ion irradiation under nearly comparable irradiation conditions (note that the dose for deuteron irradiation was higher) has been related^(58, 59) to the fact that the gas buildup is greatly reduced for deuterium in niobium, since the deuterium permeability (determined by the solubility and diffusivity) is many orders of magnitude larger than that of helium. For example, the diffusion coefficient of deuterium in niobium⁽⁶⁰⁾ is $D_D = 1.3 \times 10^{-4} \text{ cm}^2 \text{ sec}^{-1}$ at 800°C, while that for helium in

niobium⁽⁶¹⁾ ranges from 10^{-19} to 10^{-14} $\text{cm}^2 \text{sec}^{-1}$ between the temperatures of 600°C and 1200°C.

A difference in blistering behavior has also been observed by Verbeek and Eckstein⁽⁶²⁾ in Molybdenum for D^+ and He^+ ion irradiations. Figures 2 (a) and 2 (b) show some of their results on blistering in molybdenum after irradiation at room temperature with 15-keV D^+ ions to a dose of 2×10^{18} ions cm^{-2} and with 15-keV He^+ ions to a dose of 2.5×10^{18} ions cm^{-2} . It can be seen that the number of blisters per unit area (blister density) is higher for helium-ion irradiation (Fig. 2a) than for deuteron irradiation (Fig. 2b). They also observed more exfoliation of the blisters for helium-ion irradiation (see inset in Fig. 2a) than for deuteron irradiation (see inset in Fig. 2b). However, one should note that the total dose for helium-ion irradiation was slightly higher than for deuteron irradiation.

Thomas and Bauer^(63, 64) studied proton and helium blistering in Type 316 stainless steel. For irradiation with 150-keV protons to a dose of 1×10^{19} ions cm^{-2} at -93°C, they observed blisters with an average diameter of 5 μm . For proton irradiation at temperatures of -78°C, 70°C, 270°C and 350°C no blisters were observed even after irradiation to total doses ranging from 1.3×10^{19} to 2×10^{19} ions cm^{-2} . For 300-keV helium ion irradiation to a lower dose of 4×10^{18} ions cm^{-2} large blisters with severe exfoliation of the blister skin was observed in the temperature range -170° to 500°C.

No blisters were observed⁽⁶³⁾ for vanadium irradiated at 115°C with 150-keV protons to a dose of 1.5×10^{19} ions cm^{-2} and at -105°C to a dose of 2.5×10^{19} ions cm^{-2} . In contrast, blisters have been observed in vanadium during helium ion irradiation for much lower dose ranges (2×10^{18} - 6.2×10^{18} ions cm^{-2}) over a wide energy range of 100-1000 keV^(65, 66) and a wide temperature range.^(36, 37, 58, 63, 67, 68)

Primak and Luthra⁽¹²⁾ did not observe any blisters on nickel which had been irradiated with protons for total doses up to approximately 4×10^{18} ions cm^{-2} , but they observed blisters after irradiation with He^+ ions up to the same total dose values in the energy range 40 - 140 keV. More recently, blisters have been observed in nickel irradiated with 400-keV D^+ ions to total doses above 8×10^{18} ions cm^{-2} at a low temperature of -153°C .⁽⁶⁹⁾ For this low-temperature deuteron irradiation the average blister diameter was found to be of the same order of magnitude as the blister diameter observed for 500-keV $^4\text{He}^+$ ion irradiation of Ni (110) surface to a dose of 6.2×10^{18} ions cm^{-2} at room temperatures⁽⁷⁰⁾ as can be seen in Figs. 3 (a) and 3 (b). One should note that in these irradiations one large blister appears to cover most of the irradiated area.

Several of the examples cited above illustrate the difference in the blistering behavior of a given target material to the implantation of hydrogen-isotope ions and helium ions under otherwise nearly identical irradiation conditions. We will now discuss some of the major parameters affecting the radiation blistering process in the following sections.

V. PROJECTILE ENERGY:

The depth at which the ions are implanted in a solid depends on the projectile energy. In fact both the depth profile of the implanted ions and the energy deposited into damage are functions of the projectile energy and affect the gas bubble and subsequent blister formation significantly.

Several of the features which describe the blistering phenomena such as blister diameter, blister density, blister skin thickness, and critical dose for blister appearance have been observed to increase with increasing

projectile energy in a number of target projectile systems. Figures 4 (a) to 4 (d) illustrate this effect for vanadium irradiated at room temperature with helium ions in the energy range 100 - 1000 keV. For irradiation with 100-keV $^4\text{He}^+$ ions to a dose of 3.1×10^{18} ions cm^{-2} the blisters are barely resolvable in the micrograph shown in Fig. 4 (a), but at higher magnifications (see inset in Fig. 4a) blisters with average diameters ranging from 1 to 8 μm were observed. Irradiation with 250-keV $^4\text{He}^+$ ions to the same dose showed slightly larger blisters with diameters ranging from 3 to 40 μm (Fig. 4b). For 500-keV irradiation to a larger dose of 6.2×10^{18} ions cm^{-2} the average blister diameters range from 15 to 350 μm (Fig. 4c). On further increasing the projectile energy to 1000 keV only one large blister occupied the entire bombarded area (Fig. 4d), and this blister had already ruptured and a second blister had formed.

Similar results have been obtained on niobium irradiated at room temperature with helium ions for an even larger energy range from 2 keV to 1.5 MeV. Figure 5 is a plot of the available data^(28, 30, 31, 35, 58, 71) on average blister diameters as a function of helium-ion energy. The bars indicate the smallest and the largest average blister diameter observed at a given energy. In general a range of blister diameters is observed in a given irradiated area, and Fig. 6 shows an example⁽³⁰⁾ of the distribution of blister diameters for polycrystalline niobium irradiated at room temperature with 500-keV $^4\text{He}^+$ ions to a dose of 6.2×10^{18} ions cm^{-2} . In this particular example it can be seen that the average blister diameters range from 9 μm to as large as 500 μm and largest fraction of blisters have an average diameter of $\sim 12 \mu\text{m}$. For the cases where such blister diameter distributions are known, the average blister diameter corresponding to the peak in the distribution has been indicated in Fig. 5. Even though there is a large range of average blister

diameters observed at a particular projectile energy it can be seen in Fig. 5 that the average blister diameters increase with increasing projectile energy for niobium irradiated at room temperature with helium ions. At energies above 500 keV very large blisters covering nearly the entire bombarded area have been observed as indicated by the arrows in Fig. 5. An increase in blister diameters in molybdenum irradiated with helium ions was observed by Erents and McCracken⁽³⁵⁾ with increasing ion energy from 7 keV to 80 keV (Fig. 5). For nickel irradiated at -153°C with deuterons in the energy range 150 keV to 400 keV a similar dependence was observed by Möller et. al.⁽⁶⁹⁾

The increase in the average blister diameter with increase in projectile energy may be understood^(58, 71) qualitatively as follows. One can express the gas pressure p inside a blister as⁽³⁰⁾

$$p = 4\sigma_y ht / (r^2 + h^2) \quad (1)$$

where σ_y , is the yield strength of the material; t , is the blister skin thickness; r , is the radius of the blister; and h , is the height of the blister. This equation was originally derived by Hill⁽⁷²⁾ for spherical bulging by plastic deformation due to the gas pressure applied to one side of a circular metal diaphragm firmly clamped around its perimeter. It has been observed that the blister skin thickness t is a strong function of ion energy E and increases with increasing projectile energy as will be discussed below. If one assumes that the blister height h and pressure p are only weak functions of E and σ_y is not dependent on E , a higher projectile energy (and correspondingly higher skin thickness t) will result in a larger blister radius r (see Equation 1). The value of σ_y in an irradiated sample may differ from the value for an unirradiated material due to radiation damage and may be a weak function of the projectile energy.

The blister-skin thickness in all metals studied has been observed to increase with increasing projectile energy over a wide range of energies. Figure 7 shows an example for vanadium irradiated at room temperature with helium ions of energies ranging from 100 keV to 1000 keV.⁽⁶⁶⁾ Figures 7 (a) - (d) illustrate the increase of blister skin thickness for increasing helium-ion energies of 100 keV, 250 keV, 500 keV, and 1000 keV. For helium ion irradiation of niobium the blister skin thicknesses have been measured^(30,31,59,73-75) for a much larger energy range (1 keV - 1500 keV) and the measured skin thickness for different projectile energies have been compared with corresponding calculated projected ranges of $^4\text{He}^+$ ions incident on niobium. Figure 8 shows a plot of projected ranges of helium ions in niobium for different energies calculated according to the method formulated by Schiøtt.⁽⁷⁶⁾ The dotted curve represents calculations using Thomas-Fermi nuclear stopping cross sections together with the electronic stopping cross sections of Lindhard et. al.⁽⁷⁷⁾, which are proportional to projectile velocities. This latter assumption of velocity-proportional electronic stopping is strictly true for velocity v smaller than $v_0 Z_p^{2/3}$ (here $v_0 = e^2/h$, Z_p = atomic number of projectile), which corresponds to helium-ion energies less than 250 keV. The solid curve was calculated using Thomas-Fermi nuclear stopping cross sections together with Brice's⁽⁷⁸⁾ expression for electronic stopping, which uses the semi-empirical values of stopping power from Ziegler and Chu.⁽⁷⁹⁾ The available data on measured blister skin-thickness values for niobium irradiated with helium ions of different energies are plotted in Fig. 8. It can be seen that the measured skin thickness values for the energy range from 250 keV to 1500 keV⁽⁷³⁾ agree within 15% with the calculated projected range values according to Brice. For the energy range 60-80 keV⁽⁷⁵⁾ the agreement is better with the calculated projected-range values according to Schiøtt. For He^+ ion energies below 40 keV

the blister skin thickness values^(31, 74, 75) are higher than the projected range values, the difference increasing with decreasing ion energy. For 15-keV He⁺ ion the value obtained by Behrisch et. al.⁽⁷⁴⁾ agrees closely with the one obtained by St-Jacques et. al.⁽⁷⁵⁾ It should be noted that blister skin thickness values of Roth et. al.'s⁽³¹⁾ for the low energy range were indirectly obtained from Rutherford backscattering measurements, whereas values of Kaminsky et. al.⁽⁷³⁾ and of St-Jacques et. al.⁽⁷⁵⁾ were directly measured from the scanning electron micrographs of blister skins.

In the unfolding of Rutherford backscattering data for a determination of blister skin thickness, however, uncertainties in the values for differential energy losses are reflected in corresponding uncertainties in the thickness values. Figure 9 shows a typical example of energy distributions of backscattered protons from which the estimates of skin thickness were made by Roth et. al.^(32, 42) for blisters resulting from 4-keV helium ion irradiation of Nb (100) surface in random direction. The backscattered spectra were taken in <100> / <111> double alignment with 150-keV protons before and after bombardment at room temperature with different doses of 4-keV He⁺ ions. A random backscattered spectrum is also shown in Fig. 9. The backscattered energy distributions have been transformed into a depth distribution as shown in Fig. 9 by assuming a differential energy loss. From Fig. 9 one can see that for a dose of 1.4×10^{17} ions cm⁻² a surface layer of about 500 Å thick becomes misaligned. This thickness of the misaligned region has been interpreted by Roth et. al.⁽³¹⁾ to be equal to the thickness of the blister skin.

The fact that the observed blister-skin thickness for niobium irradiated with low-energy helium ions (1-15 keV) was higher than the theoretical projected range was initially attributed^(31, 42) to channeling of a large fraction of incident helium ions along close-packed lattice directions or planes.

Subsequently the same authors⁽⁷⁴⁾ explained this difference between the blister skin thickness and the calculated projected range as being due to the blister skin separation at the end of the range. An alternative explanation suggested by other authors⁽⁷⁵⁾ is that the observed difference may in part be due to the more uniform distribution of helium bubbles for a large portion of the implant depth leading to volumetric changes (swelling due to helium bubbles) of that region for sufficiently high doses as will be discussed later in Sec. XIII.

The measurements of blister skin thicknesses on vanadium (Fig. 7) irradiated at room temperature with helium ions in the energy range 100-1000 keV also show a good correlation with the calculated projected ranges.⁽⁶⁶⁾ In contrast, for aluminum irradiated at room temperature with helium ions for the same energy range 100-1000 keV the same authors obtained blister skin thickness values that were slightly lower than the calculated projected ranges.⁽⁶⁵⁾

It is of interest to determine whether the blister skins separate from the bulk closer to the region of maximum lattice damage or the region where the concentration of the implanted ions is at a maximum. Recent studies by Kaminsky et. al.^(66, 73) and Das et. al.⁽⁶⁵⁾ have provided some insights here. Figure 10 is a comparison of blister skin thickness values for aluminum, vanadium and niobium with the corresponding damage energy distributions and projected range distributions for the energy range above 100 keV.⁽⁶⁵⁾ The data points marked (a)-(d) in Figs. 10A, 10B and 10C are blister skin thickness values for aluminum, vanadium and niobium, respectively. The solid curves in Figs. 10 (a)-10 (c) are calculated damage-energy distributions and the dashed curves are projected-range distributions for the helium-ion energies indicated. The distributions in Fig. 10 were obtained using the tabulated projected-range,

straggling and damage-energy values for helium ions according to Brice.⁽⁸⁰⁾ These tabulated values for damage-energy distributions were calculated using an intermediate method developed by Brice⁽⁸¹⁾ which includes the spatial distribution of the energy transferred by primary recoil atoms. The total error in the calculation of the projected-range distribution, and the damage-energy distribution is estimated to be 12%.^(65, 66, 73) The results shown in Fig. 10 (A) show that the average skin-thickness values for aluminum correlate more closely with the maxima in the damage-energy distributions than with the maxima in the projected-range distributions. On the other hand, for vanadium (Fig. 10B) and niobium (Fig. 10C) the average blister skin thickness values correlate more closely with the maxima in the projected-range distributions than with the maxima in the damage-energy distributions. In this connection, one should consider the possibility that for a ductile metal like aluminum the blister skin thickness could be lower due to the uniform elongation of the skin during plastic deformation and possible necking during rupture. However, measurements of blister skin thickness in aluminum made near the base of a blister and near a blister top by Das et. al.⁽⁶⁵⁾ showed no significant differences in the thickness values within the experimental error.

More recently, blister skin thicknesses have been measured in nickel irradiated at -153°C with D^{+} ions in the energy range 200 keV to 400 keV by Möller et. al.⁽⁶⁹⁾ They compared the blister skin thickness values with experimental projected ranges, which were obtained from depth profile measurements⁽⁵²⁾ and calculated maxima in the damage energy distributions, which were computed with a Monte-Carlo program (taking into account primary events only). The blister skin thickness values were closer to the maxima in the damage energy distribution than the maxima in the projected range distribution, a result similar to those described above for aluminum.⁽⁶⁵⁾

VI. CRITICAL DOSE FOR BLISTER FORMATION

In considering the effect of dose on blister formation a distinction should be made between the critical dose C_b needed for the formation of gas bubbles in the bulk of the material during irradiation, and the critical dose C_{bl} for the appearance of blisters on the surface of an irradiated material. The determination of both of these parameters depends strongly on the sensitivity and the resolution of the detection technique used. For example, the more commonly-used transmission and scanning electron microscopic techniques (used for detection of gas bubbles and blisters on the surface respectively) often employ commercially-available instruments with resolutions ranging from $5\text{-}10\text{ \AA}$ and $50\text{-}100\text{ \AA}$, respectively. Other techniques such as Mössbauer effect⁽⁸²⁾ and positron annihilation techniques^(83, 84) have been used to study vacancy clusters in metals and may be used for detection of gas bubbles. It is to be expected that the critical dose for gas bubble formation will depend on several parameters such as the concentration of the gas in the implant region, the initial and the radiation-induced defect concentrations, the type of lattice impurities and their concentration, target temperature, and the permeability of the implanted gas. While systematic studies of the critical dose for bubble formation are lacking, some information about the existence of gas bubbles in different metals after irradiation with noble gas ions is available. For example, helium bubbles have been observed in many metals such as aluminum⁽⁸⁵⁻⁸⁷⁾, vanadium⁽⁸⁸⁻⁹¹⁾, stainless steel⁽⁹²⁻⁹⁴⁾, copper^(8-10, 95, 96), niobium^(36, 37, 67, 97), molybdenum^(38, 39, 98), silver⁽⁹⁹⁾, and gold^(96, 100, 101) after helium ion irradiation for total doses ranging from $\sim 1 \times 10^{14}$ ions cm^{-2} to $\sim 6 \times 10^{18}$ ions cm^{-2} (the helium concentrations ranging from ~ 10 ppm⁽⁹⁴⁾ to several atomic percent⁽³⁷⁾). In a few studies^(38, 98) the bubbles were observed after room temperature irradiation while in other studies^(8-10, 36, 37, 67, 85-101)

either the irradiations were done at higher temperatures or the targets were annealed at high temperatures to observe bubbles. In vanadium irradiated with 240-keV He^+ ions, C_b for bubbles with diameters $\geq 40 \text{ \AA}$ was observed using TEM to be $1 \times 10^{17} \text{ ions cm}^{-2}$ for 500°C irradiation, and it decreased markedly to a value of $7 \times 10^{14} \text{ ions cm}^{-2}$ for a higher target temperature of 600°C.⁽⁹¹⁾ Evans et. al.⁽⁹⁸⁾ could not resolve gas bubbles (the resolution in their observation was $\sim 15\text{-}20 \text{ \AA}$) in molybdenum implanted at room temperature with 36-keV He^+ ions to a dose of $1 \times 10^{16} \text{ ions cm}^{-2}$. However, at a dose of $3 \times 10^{17} \text{ ions cm}^{-2}$ bubbles became readily visible (in TEM) which aligned themselves on a b.c.c. superlattice exactly analogous to the void lattice.⁽¹⁰²⁾ For irradiation with low energy (5 keV) Ar^+ ions, bubbles of $\sim 20 \text{ \AA}$ in diameter have been observed in gold after irradiation to a dose as low as $1.5 \times 10^{15} \text{ ions cm}^{-2}$.⁽¹⁰³⁾ Gas bubbles can contribute to volume swelling, and for niobium irradiated at 800°C with 300-keV He^+ ions to a dose of $5 \times 10^{17} \text{ ions cm}^{-2}$ a fractional volume increase $\Delta V/V$ of $\sim 1\%$ has been measured.⁽¹⁶⁷⁾

The critical dose for blister appearance C_{bl} depends also on several parameters, such as the concentration of the implanted gas in the implant region, the projectile energy, permeability of the implanted gas in the solid, target temperature, initial defect concentration, and yield strength of the material. Systematic studies of the dependence of C_{bl} is very limited.

Even though gas bubbles have been observed⁽⁹¹⁾ in metals at doses as low as $7 \times 10^{14} \text{ ions cm}^{-2}$ for 240-keV He^+ on V at 600°C as mentioned above, the value for the critical dose for blister appearance has been observed to be approximately two orders of magnitude higher. Only a few data are available on the dependence of

C_{bl} on the projectile energy. For example for helium ion irradiation of niobium and molybdenum, Figure 11 shows a plot of available data^(30, 31, 35, 50, 104) on critical dose for blister appearance as a function of helium-ion energy. One notices a general trend, that with increasing projectile energy the C_{bl} values increase for the type of metals and the energy ranges studied. The range of C_{bl} values shown in Fig. 11 ($C_{bl} > 1 \times 10^{17}$ ions cm^{-2}) is generally larger than the dose ranges mentioned above for which gas bubbles have been observed. A similar trend of increasing C_{bl} -values with increasing projectile energy has also been observed in nickel irradiated at -153°C with D^+ ions for the energy range 200 keV - 400 keV.⁽⁶⁹⁾ This trend appears plausible since with increasing projectile energy the blister skin thickness increases. The critical pressure, p_{cr} , in a gas bubble needed to deform the surface can be written as,^(16, 29, 30)

$$p_{cr} = \frac{4\sigma_y t^2}{3 r^2} \quad (2)$$

where the symbols have the same meaning as before in Equation 1. This expression is slightly different from the one given in Equation 1 and will be derived later in Sec. XIII. For a bubble with a given radius r and for a given yield strength of the material σ_y , a larger skin thickness t resulting from an increase in projectile energy requires a larger p_{cr} , and this can be obtained at larger doses. However, there are other complicating microstructural effects that may render this simple relationship between critical dose and projectile energy invalid.

For example, the critical dose for blister appearance in niobium at room temperature has been found to be dependent on the initial dislocation density.⁽³⁰⁾

It can be seen in Fig. 11 that the C_{bl} -value for 50-keV He^+ -ion irradiation of annealed niobium at room temperature is $\sim 2 \times 10^{18}$ ions cm^{-2} , while for cold worked niobium only an upper limit value of 6.2×10^{17} ions cm^{-2} can be given. This lowering of C_{bl} has been suggested⁽³⁰⁾ to be due to easier nucleation of

helium bubbles on the more numerous dislocations in cold-worked samples.

For 20-keV helium-ion irradiation of molybdenum at room temperature, Erents and McCracken observed $C_{bl} \sim 5 \times 10^{17}$ ions cm^{-2} , a value which is close to the interpolated one for annealed niobium for comparable irradiation conditions. For gases like hydrogen which have high permeability in metals, C_{bl} is generally higher than for inert gases like helium. For example, for cold worked polycrystalline niobium irradiated at room temperature with 500-keV D^+ no blisters were observed⁽⁵⁹⁾ after a dose of 6.2×10^{18} ions cm^{-2} , whereas for helium-ion irradiation under identical conditions C_{bl} was less than 6.2×10^{17} ions cm^{-2} (Fig. 11).

A dependence of C_{bl} on irradiation temperature has been observed. For example, for 500-keV He^+ ion irradiation of niobium C_{bl} at room temperature it is $\sim 2 \times 10^{18}$ ions cm^{-2} and it decreases to $< 6.3 \times 10^{16}$ ions cm^{-2} for irradiation at 900°C.⁽⁵⁹⁾ However, for 4-keV He^+ -ion irradiation, the decrease in C_{bl} with increasing irradiation temperature is not so large (see Fig. 12). This trend can be understood since with an increase in temperature the yield strength, σ_y , of most metals decreases, and thus for constant values of r and t in Equation 1, the value for p_{cr} will decrease correspondingly.

VII. EFFECT OF TOTAL DOSE

In this section we will consider the effect of the total dose values which are above the critical dose C_{bl} on the blistering phenomenon. Once the C_{bl} values have been exceeded the dose values will affect such features as blister diameter, blister density and the exfoliation of the blister skin.

The ranges of average blister diameters depend strongly on the projectile energy (as described in Sec. V), but less strongly on the total dose.

For example, for cold worked niobium irradiated at room temperature with 500-keV He^+ ions, the average diameters of a majority of the blisters range from 10 to 30 μm for both total doses of 6.2×10^{17} ions cm^{-2} ($\sim 64\%$ of total number of blisters) and 6.2×10^{18} ions cm^{-2} ($\sim 75\%$ of total number of blisters).⁽³⁰⁾ For annealed polycrystalline vanadium irradiated at 900°C with 500-keV He^+ ions, it has been observed⁽¹⁰⁵⁾ that as the dose is increased by one order of magnitude from 6.2×10^{17} ions cm^{-2} to 6.2×10^{18} ions cm^{-2} , the range of the values of average blister diameters does not change appreciably but the mean value of the average blister diameters shows a small increase from 6.2 to 7.4 μm . Similar results have been obtained⁽¹⁰⁵⁾ for niobium irradiated at 900°C with 500-keV He^+ ions, where the range of average blister diameters does not change appreciably for doses of 6.2×10^{17} ions cm^{-2} and 6.2×10^{18} ions cm^{-2} . For 125-keV D^+ irradiation of Cu (100) surface the pits formed by blister rupture does not increase in size appreciably as the total dose is increased (above 1.9×10^{19} ions cm^{-2}).^(17, 18)

The blister density (i.e. the number of blisters per unit irradiated area) has been observed to increase with total dose. For palladium implanted at with 300-keV He^+ ions Thomas and Bauer⁽²⁹⁾ observed an increase in blister density by a factor of 5 by increasing the total dose from 1×10^{18} ions cm^{-2} to 2×10^{18} ions cm^{-2} , but the values for the blister diameters did not change. For molybdenum irradiated at room temperature with 36-keV He^+ ions, Erents and McCracken⁽³⁵⁾ observed an increase in blister density by about a factor of 4 when the total dose was increased by a factor of two from 6×10^{17} ions cm^{-2} to 1.2×10^{18} ions cm^{-2} . For higher temperature irradiation (at 900°C of niobium with 500-keV He^+ ions, Das and Kaminsky⁽¹⁰⁵⁾ observed an increase in the blister density from $\sim 6 \times 10^4$ blisters cm^{-2} to $\sim 1.5 \times 10^5$ blisters cm^{-2} with an increase in total dose from 6.2×10^{17}

ions cm^{-2} to 6.2×10^{18} ions cm^{-2} .

The increase in blister density with increasing total dose is observed only when there are numerous small blisters over the irradiated area. However, when there are only a few large blisters covering most of the irradiated area (as observed for high projectile energies, for example see Figs. 4c and 4d), one observes an increase in blister skin exfoliation with increase in total dose. Figures 13 (a), 13 (b) and 13 (c) illustrate this effect for Type 304 stainless steel irradiated at 450°C with 500-keV He^+ ions to a total doses of 6.2×10^{17} , 3.1×10^{18} and 6.2×10^{18} ions cm^{-2} , respectively.⁽⁵⁸⁾ For a dose of 6.2×10^{17} ions cm^{-2} a large portion of the irradiated area is occupied by a single blister with an average diameter of $\sim 700 \mu\text{m}$ (Fig. 13a) which has ruptured. At a higher dose of 3.1×10^{18} ions cm^{-2} three exfoliated skin layers are observed (Fig. 13b) as compared with one for a dose of 6.3×10^{17} ions cm^{-2} (Fig. 13a). For an even higher dose of 6.2×10^{18} ions cm^{-2} the number of exfoliated skin layers increases to five in some regions (Fig. 13c). One can estimate the erosion yields (see Sec. III) for these cases by measuring the area from which the blister skin has fallen off and the blister skin thickness. The estimated erosion yields for the three doses 6.2×10^{17} , 3.1×10^{18} and 6.2×10^{18} ions cm^{-2} were 0.1 ± 0.05 , 0.45 ± 0.1 and 0.8 ± 0.2 atoms per incident helium ion, an almost linear increase in erosion rate with total dose.⁽⁵⁸⁾

For low energy ($< 15 \text{ keV}$) irradiations the successive exfoliation of blister skin with increasing total dose is not observed. Martel et. al.⁽²⁸⁾ observed blisters to appear in niobium irradiated at room temperature with helium ions for energies of 5 keV, 10 keV and 15 keV for doses or dose ranges of 3.1×10^{18} ions cm^{-2} , $6.2 \times 10^{17} - 3.1 \times 10^{18}$ ions cm^{-2} and $6.2 \times 10^{17} - 9.4 \times 10^{18}$ ions cm^{-2} for the respective energies. However, when the dose

values were increased to $\sim 6.2 \times 10^{18}$ ions cm^{-2} , $\sim 6.2 \times 10^{18}$ ions cm^{-2} and 1.9×10^{19} ions cm^{-2} for 5-, 10- and 15-keV helium ion irradiations, respectively, no blisters were observed. Figures 14 (a) and (b) show surfaces of niobium irradiated with 15-keV He^+ ions to total doses of 3.1×10^{18} ions cm^{-2} and 6.2×10^{19} ions cm^{-2} , respectively. Blisters are seen for the lower dose of 3.1×10^{18} ions cm^{-2} (Fig. 14a) but are not observed at the higher dose of 6.2×10^{18} ions cm^{-2} (Fig. 14b). The upper dose limit beyond which no blisters are observed has been termed "cut-off dose" and was found to be of the same order of magnitude as that was necessary to sputter off a thickness equivalent to the thickness of the blister skin. This behavior has been recently confirmed by Behrisch et. al. ^(20, 106) for 9-keV He^+ -ion bombardment of niobium at room temperature. They bombarded niobium surfaces with total doses up to 7×10^{20} ions cm^{-2} and observed formation of ridges and grooves on the surface at this highest dose after the blisters (observed initially at lower doses) had disappeared. Similar results have also been obtained ⁽²⁰⁾ for Type 304 stainless steel irradiated with 5-keV hydrogen ions, where blisters were initially observed for a dose of 4.9×10^{18} ions cm^{-2} but no blisters were observed at a dose of 4.9×10^{19} ions cm^{-2} . The cut-off dose for blister disappearance has recently been observed to decrease with increasing irradiation temperature for helium-ion irradiation of niobium. ⁽¹⁰⁷⁾ It has been suggested ⁽²⁸⁾ that after a surface layer equivalent to the blister-skin thickness has been sputtered off (e.g. for 10-keV He^+ irradiation of niobium a dose of 5.1×10^{18} ions cm^{-2} is needed, assuming a sputtering yield of 0.085 niobium atoms per helium ion and blister skin thickness of 800 Å) the resulting surface roughness and the damaged surface layers may increase the effective sputtering yield, and thus physical sputtering may prevent the formation of blisters. In addition, it has been suggested that the diffusion of the implanted gas through the highly-damaged surface layers may contribute ⁽¹⁰⁶⁾ to the observation of no blisters at high doses.

VIII. DOSE RATE:

The rate of gas build-up near the implant depth and the subsequent blister formation for a given irradiation temperature depends on the dose rate (i.e. the incident ion flux) and the rate of gas release from the surface. Depending on the balance of gas trapping and gas release, there may or may not be an effect of dose rate on blister formation. The blister density and the critical dose for blister appearance has been observed to be affected by dose rate in some cases.⁽¹⁰⁵⁾

For vanadium irradiated at 900°C with 500-keV helium ions, Das and Kaminsky⁽¹⁰⁵⁾ observed an increase in blister density with increasing dose rate. Figures 15 (a), 15 (b) and 15 (c) illustrate this for dose rates of 1×10^{13} , 1×10^{14} and 1×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$, respectively. The total dose in all three cases was the same (6.2×10^{17} ions cm^{-2}). There are two types of blisters observed in these micrographs (Figs. 15 a-c), one type is very small in size, and the other is larger in size. The average diameters of blisters of both types do not seem to depend significantly on the beam flux. For the small size blisters the value for the average blister diameter ranges from $\sim 0.1 \mu\text{m}$ to $\sim 0.3 \mu\text{m}$, and for the large size blisters the average diameter ranges from $\sim 3 \mu\text{m}$ to $\sim 15 \mu\text{m}$ for all three dose rates. It is observed that the blister density for the small size blisters is about the same $[(5 \pm 2) \times 10^6 \text{ blisters cm}^{-2}]$ for the three fluxes. For the larger size blisters, however, a dependence of the blister density on the flux is observed. The values increases from $(1.0 \pm 0.5) \times 10^4$, to $(3 \pm 1) \times 10^4$ and to $(7 \pm 2) \times 10^4$ blisters cm^{-2} as the flux increases from 1×10^{13} , to 1×10^{14} and to 1×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$, respectively. For niobium irradiated at 900°C with 500-keV He^+ ions to a dose of 6.2×10^{17} ions cm^{-2} , a similar increase in blister density has been observed⁽¹⁰⁵⁾ for the larger size blisters. Again, two types

of blisters were observed, one type having smaller diameters ($0.3 - 1.0 \mu\text{m}$) and the other type having larger diameters ($3 - 15 \mu\text{m}$). The blister density for the large blisters increased from $(3 \pm 2) \times 10^4$ blisters cm^{-2} to $(6 \pm 3) \times 10^4$ blisters cm^{-2} as the flux was increased from 1×10^{13} to 1×10^{14} ions $\text{cm}^{-2} \text{sec}^{-1}$, but a further increase in flux to 1×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$ did not seem to change this value within the quoted error limits. However, for niobium irradiated at 700°C with 250-keV D^+ ions, Das and Kaminsky⁽¹⁰⁵⁾ observed an increase in blister size with increasing dose rate. Some of the blisters for these irradiations had "crow-foot" shape (as will be discussed later in Sec. XI), and the length of each of the three prongs of a particular blister increased by approximately a factor of three as the dose rate was increased from 1×10^{14} ions $\text{cm}^{-2} \text{sec}^{-1}$ to 1×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$ at constant total dose.

The dependence of the critical dose for blister appearance, C_{b1} , on dose rate has been investigated for 15-keV H^+ irradiation of molybdenum at room temperature by Verbeek and Eckstein.⁽⁶²⁾ Figure 16 (a) shows a plot of C_{b1} as a function of the dose rate. It can be seen that C_{b1} decreases with increasing dose rate, ranging from 2.9×10^{14} ions $\text{cm}^{-2} \text{sec}^{-1}$ (this is equivalent to the current density of 0.46×10^{-4} A. cm^{-2} plotted in Fig. 16a) to 2.4×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$. This can be understood if one considers that during the irradiation the loss of hydrogen from the implant depth by diffusion competes with the incoming flux. Thus, C_{b1} may be reached at a lower dose for a higher incident flux than for a lower incident flux. Recently, Möller et. al.⁽⁶⁹⁾ studied the effect of dose rate for 300-keV and 350-keV deuteron irradiation of nickel at -153°C . Their results show [see Fig. 16 (b)] that with increasing dose rate ranging from 3.7×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$ ($\approx 6 \times 10^{-4}$ A. cm^{-2}) to 1×10^{16} ions $\text{cm}^{-2} \text{sec}^{-1}$ ($\approx 16.9 \times 10^{-4}$ A. cm^{-2}) that C_{b1} increases. It may be noticed in Figure 16b that at low dose rates the critical dose increases linearly and shows

a tendency to level off at higher dose rates. These opposite trends, observed for the low-energy proton irradiation of molybdenum (Fig. 16a) and for high-energy deuteron irradiation of nickel (Fig. 16b), cannot be readily understood at this time. According to Möller et. al.⁽⁶⁹⁾ if one assumes that an increase in dose rate will increase the bubble density and the volume of each bubble increases with increasing total dose, then for a given total dose the trapped gas will be distributed into a fewer number of gas bubbles in the case of low dose rate than in the case of high dose rate.

IX. TARGET TEMPERATURE

The target temperature is one of the most important parameters affecting the radiation blistering process. For example, the average blister diameter, blister density and the exfoliation of blister skin (which determines the erosion yield) have been observed to depend on target temperature. An example^(37, 58, 68) of the effect of irradiation temperature on these parameters is illustrated in Fig. 17 for vanadium irradiated with 500-keV He^+ ions to a total dose of 6.3×10^{18} ions cm^{-2} . For irradiation at room temperature, blisters with average diameters ranging from 15 μm to 350 μm can be seen in Fig. 17a. There is exfoliation of blister skins in some areas. As the irradiation temperature is increased from room temperature to 300°C (Fig. 17b) the average blister diameter increases, and most of the irradiated area is occupied initially by one large blister which subsequently exfoliates and a second blister is formed and starts to exfoliate. A third blister has also been formed in the area which was exposed by the exfoliation of the first two blister skin layers (Fig. 17b). Irradiation at 500°C (Fig. 17c) shows an even stronger increase in blister exfoliation since six exfoliated skin layers are observed in certain regions. On further increasing the irradiation temperature to 600°C one still

observes a large blister covering most of the irradiated area, but now the number of exfoliated blister skin layers decreases to three (Fig. 17d).

Increasing the irradiation temperature even higher to 800°C causes the average blister diameters to decrease significantly to only 10 - 20 μm , and no large-scale exfoliation is observed (Fig. 17e). Similar results have been obtained by Bauer and Thomas^(36, 63, 67) for a slightly-lower energy (300 keV) helium-ion irradiation of vanadium to a lower dose of $2 - 4 \times 10^{18}$ ions cm^{-2} . For irradiation at 400°C to a dose of 4×10^{18} ions cm^{-2} , they observed severe exfoliation of blister skins, but only a few small blisters with no severe exfoliation were observed for irradiation at 800°C to a dose of 2×10^{18} ions cm^{-2} . They increased the irradiation temperature to 1200°C and observed no blisters after a dose of 2×10^{18} ions cm^{-2} , but the surface had small holes with crystallographically-oriented edges (see Fig. 18). The origin of these holes has been suggested to be due to the intersection of large bubbles with the surface. Thus, the blister diameter and exfoliation of blister skin in vanadium is maximum at an intermediate irradiation temperature (300°C - 600°C). A similar temperature-dependence of blister diameter and blister exfoliation has been observed for a number of other metals and alloys irradiated with helium ions as will be discussed below.

For niobium irradiated with 300-keV helium ions to a total dose of 4×10^{18} ions cm^{-2} , severe exfoliation has been observed^(36, 67) at 400°C and 600°C. On increasing the irradiation temperature to 1000°C and 1200°C, holes were observed on the surface.^(67, 108) For annealed polycrystalline niobium irradiated with 500-keV He^+ ions to a dose of 6.2×10^{18} ions cm^{-2} , blister skin exfoliation was observed⁽¹⁰⁹⁾ at room temperature and 600°C but at a temperature of 900°C only small blisters were observed (average diameters of $\sim 15 \mu\text{m}$) with no exfoliation of blister skin.

Irradiation of stainless steel Type 304^(58, 110) and Type 316^(63, 111) with 500-keV and 300-keV helium ions, respectively, showed a similar temperature-dependence of blister skin exfoliation. Similarly, for molybdenum^(36, 63) irradiated with 300 keV He^+ ions severe exfoliation of blister skin was observed at 400°C and 600°C after a dose of 4×10^{12} ions cm^{-2} , but at 800°C only small blisters were observed (average diameters of 20 - 30 μm) with no severe exfoliation after a dose of 1×10^{18} ions cm^{-2} . At even a higher irradiation temperature of 1200°C some blisters and some holes were observed on the surface after a total dose of 2×10^{18} ions cm^{-2} . A similar behavior has also been observed^(35, 39) in molybdenum irradiated with low energy (36 keV) helium ions, where severe exfoliation was observed at an intermediate irradiation temperature of 527°C but exfoliation was less severe at room temperature and at 827°C. At high irradiation temperature of 1327°C the irradiated surface was covered with "pin holes". These pin holes were similar to those shown in Fig. 18 but did not have crystallographically-oriented edges.

Since the exfoliation of blister skin is strongly dependent on target temperature, it is apparent that the erosion yields will also show a strong dependence on target temperature, if the other irradiation conditions remain the same. The erosion yields have been estimated for a number of metals and alloys irradiated with helium ions as a function of temperature and Fig. 19 shows a plot of some of the available data.^(58, 63, 109, 112) It can be seen in Fig. 19 that the temperature-dependence of erosion yield shows a maximum for Type 304 stainless steel and vanadium. For Type 304 stainless steel irradiated with 500-keV He^+ ions, the erosion yield goes through a maximum in the temperature range of about 350-550°C. For polycrystalline vanadium irradiated with 500-keV He^+ ions, the maximum erosion yield appears to be somewhere between 300°C and 500°C. The temperature at which the erosion yield is a maximum depends on the type of

target material. For annealed aluminum irradiated with 100-keV He^+ ions, it appears that the maximum in the erosion yield is at a much lower temperature as compared to stainless steel and vanadium (possibly below room temperature).

The erosion yield has been observed to be dependent on projectile energy for some materials. Figure 19 also shows the few available data for the erosion yields for niobium irradiated with 100-keV and 500-keV He^+ ions. It may be noted that the erosion yield for niobium at room temperature is about a factor of five higher for 100 keV helium-ion irradiation than for 500-keV irradiation. Similarly, for Type 304 stainless steel one observes that as the helium projectile energy is reduced from 500 to 100 keV, the erosion yield increases by approximately a factor of seven for the same dose of 3.1×10^{18} ions cm^{-2} at a temperature of $\sim 450^\circ\text{C}$. The observation in niobium and stainless steel that the erosion yield is higher for the low ion energy than for the high one for the temperature and dose range studied, does not seem to hold for all target materials. Some recent results⁽¹¹²⁾ on annealed polycrystalline aluminum irradiated at room temperature with 100-keV, 250-keV and 500-keV helium ions show erosion yields of $\sim 1.75 \pm 0.25$, 1.44 ± 0.5 , 1.67 ± 0.8 , respectively. The reason for this difference in behavior in aluminum as compared to niobium or stainless steel is not clearly understood at present.

The observed change in erosion yield with temperature has been related^(37, 58) to the strong temperature dependence of the yield strength of the material. For example, the yield strength of annealed Type 304 stainless steel at 450°C is half of its value at room temperature.⁽¹¹³⁾ Similarly the tensile strength of vanadium at 900°C is less than a third of its value at room temperature.⁽¹¹⁴⁾ Thus, for a particular dose, the collected gas in a bubble can deform the surface skin more readily at high temperature (e.g. at 450°C in stainless steel) than at room temperature. In addition,

the kinetic pressure of the gas in the bubble will increase with temperature and may also enhance this process. However, at very high temperatures (e.g. at 900°C in vanadium) helium may be released through the surface either by atomic diffusion or by migration of small bubbles. Therefore, the amount of helium trapped in the lattice will be affected by the helium-release rate and the bubble-nucleation rate and will in turn determine the size and density of helium bubbles and the subsequent blister size. Thus, the degree of blistering and exfoliation of blister skin is maximized if the temperature is high enough so that the surface can be deformed easily but low enough that the helium release from the surface is still very small.

X. TEMPERATURE DEPENDENCE OF GAS RE-EMISSION:

The temperature dependence of blister rupture and exfoliation is closely related to the temperature dependence of the re-emission of trapped gas. Gas re-emission measurements, associated with blister rupture, have been made by a number of authors for different target-projectile systems. For monocrystalline copper irradiated at room temperature with 125 keV D^+ ions, gas bursts were observed by Kaminsky⁽¹¹⁾ due to blister rupture, as described earlier in Sec. 1. These early observations showed that the gas bursts were indeed due to blister rupture, since the number of gas bursts correlated very well with the number of pits observed on the surface. Extensive measurements of helium gas re-emission from Nb, V, Mo, stainless steel and Pd during irradiation have been made by Bauer et. al.^(29, 33, 36, 64) for different target temperatures and different total doses. Figure 20 shows an example of helium re-emission from vanadium as a function of helium fluence for three different irradiation temperatures.⁽⁶⁴⁾ For 400°C irradiation it can be seen that an abrupt change in re-emission occurs after a certain dose is attained. The sudden onset of re-emission, with a peak

value well in excess of 100% of the incoming particle flux, has been observed to occur at nearly the same dose as that at which blister rupture is observed optically. The gas build-up prior to the sharp increase in the gas release explains the observed re-emission rates in excess of 100% of the incoming flux. After the burst the re-emission rate returns to a relatively low value. With continued irradiation helium again accumulates resulting in the periodic re-emission of "bursts", as seen in Fig. 20 for 400°C. The number of re-emission bursts observed have a one-to-one correspondence with the number of the large size exfoliated blister skin layers on the surface. At higher irradiation temperature (e.g. at 800°C) no large periodic bursts are observed, consistent with the observed changes in the surface topography. At a high temperature of 1200°C the re-emission rate increases to $\sim 100\%$ almost immediately for small dose values, and appears to remain constant thereafter. At this high irradiation temperature small pores were observed on the surface as shown earlier in Fig. 18.

The first burst of gas in the re-emission measurements is related to the exfoliation or rupture of the first blister skins. Figure 21 is a plot of the dose for the onset of first re-emission burst for Nb, V, Mo and Type 316 Stainless Steel irradiated with 300 keV He^+ ions [taken from the helium re-emission curves of Bauer et. al. (36, 64)] as a function of irradiation temperature. One notices that the onset dose for the first re-emission burst decreases with increasing target temperature. This can be expected since the critical dose for blister appearance also shows a decrease with increasing target temperature.

Hydrogen re-emission from some metals during irradiation with H^+ ions shows a different behavior than helium re-emission for comparable irradiation conditions. The hydrogen re-emission has been observed to rise

smoothly to an equilibrium value in a large number of cases. For example, during 150-keV H^+ implantation in Type 316 stainless steel at $-93^\circ C$, $-75^\circ C$ and $60^\circ C$, Bauer and Thomas⁽⁶⁴⁾ observed that the hydrogen re-emission rose smoothly as the dose was increased and attained an equilibrium value after a certain dose. The dose at which the equilibrium value was attained was lower at the higher temperature of $60^\circ C$ than at $-93^\circ C$. It may be mentioned that only for irradiation at $-93^\circ C$ were blisters observed on the surface.⁽⁶³⁾ For 150-keV H^+ implantation in molybdenum a similar smooth rise in the re-emission was observed for implantation at $0^\circ C$ and in the range $25 - 100^\circ C$ but an abrupt rise in re-emission was observed for $-115^\circ C$ irradiation (somewhat similar to the helium re-emission behavior). No periodic re-emission bursts (as seen for He^+ irradiation of many metals) have been reported for hydrogen irradiation of metals. This is consistent with the observation that no severe exfoliation of multiple blister skin layers occurs in most metals, because of their high solubility and diffusivity for hydrogen.

XI. CHANNELING CONDITION OF THE PROJECTILE:

In a monocrystalline solid a penetrating projectile can be guided by the regular lattice arrangement, e.g. through the spaces between the planes (planar channeling) or along channels formed by parallel rows of atoms (axial channeling). The channeling of projectiles affects the implant depth of the projectiles and the radiation induced defect concentration (For a recent review of the various aspects of channeling phenomenon see Ref. 115). The blister formation on the surfaces of monocrystalline targets will be expected to depend on the channeling condition of the projectile. For monocrystalline Nb (111) surfaces irradiated at room temperature with 500-keV He^+ ions, Das and Kaminsky⁽⁷¹⁾ observed blisters with larger average diameters for axially-channeled ions than

for unchanneled ions as shown in Figs. 22 (a) and 22 (b), respectively. This effect is somewhat similar to the effect of projectile energy on blister diameter (discussed in Section V), because the channeled ions are implanted at a greater depth from the surface than the unchanneled ions. Similar observations were also made by Verbeek and Eckstein⁽⁶²⁾ in Mo (100) surface irradiated with 150-keV He^+ ions. The average blister diameter was 6.0 μm for the channeled case, as compared to 4.8 μm for the unchanneled case. They also found the blister skin thickness to be larger for the channeled case ($\sim 0.4 \mu\text{m}$) than for the unchanneled case ($\sim 0.3 \mu\text{m}$).

Channeling of the projectiles has also been observed to affect the blister density significantly.⁽¹¹⁶⁾ Figure 23 shows an example of blisters formed on Nb (111) surface irradiated at 900°C with 500-keV He^+ ions for ions channeled along $\{111\}$ axis (Fig. 23a) and for nonchanneled ions (Fig. 23b) for the same total dose of 6.2×10^{18} ions cm^{-2} . These blisters have a three fold symmetry and have been called "crow-foot" blisters. Each of the three prongs of the blister has two distinct facets, which intersect along the lines whose projections on the (111) surface are in the $[\bar{1}2\bar{1}]$, $[\bar{1}\bar{1}2]$, and $[2\bar{1}\bar{1}]$ directions. It may be noted that no crow-foot blisters are observed whose prongs are pointed along the other equivalent $[1\bar{2}1]$, $[11\bar{2}]$, and $[\bar{2}11]$ directions. It can be seen that the blister density is lower by approximately two orders of magnitude for the channeled helium projectiles (Fig. 23a) as compared to the unchanneled ones (Fig. 23b). Such a behavior is not unexpected since the channeling of projectiles helps to reduce the radiation damage in near surface regions (corresponding to the initial part of the trajectory of the channeled projectiles), and thereby reduces the number of radiation-induced nucleation sites for bubble formation. The lower blister density for the channeled case helps to retain the well defined crystallographic character of the blisters (Fig. 23a).

XII. CRYSTALLOGRAPHIC ORIENTATION OF THE IRRADIATED SURFACE:

The effect of the crystallographic orientation of the irradiated surface on blister formation has not been investigated in as much detail as some of the other parameters described above. The density, diameter and shape of the blisters depend on the crystallographic orientation of the surface with respect to the incident ion beam. Milacek and Daniels⁽¹¹⁷⁾ studied orientation dependence of blister density in polycrystalline aluminum crystals with large grains (1 - 1.5 mm) bombarded with protons in the 10 - 100 keV range. The orientations of the individual grains were determined by the standard Laue backreflection technique but using a microfocus x-ray beam. They observed the lowest blister density in grains with orientation near {111}, and this was followed next by grains near {100} orientation. The grains near {110} and higher indices orientations had the maximum density of blisters. They attributed this to the orientation dependence of the range of ions; the greater the range of ions the greater is the amount of retained gas and consequent blistering. Figure 24 shows an example of orientation dependence of blister diameter for polycrystalline niobium irradiated at room temperature with 15-keV He^+ ions. The variation of the blister diameter among the different grains can be readily seen. This may be due to the fact that even in polycrystalline material certain grains may be favorably oriented so that the ions may be channeled to some extent.

For irradiation of monocrystalline copper with 125-keV D^+ , Kaminsky⁽¹⁷⁾ observed the pits due to ruptured blisters to be more square, more rectangular, and more triangular for (100), (110) and (111) surfaces, respectively. Some recent results by Kaminsky and Das⁽¹¹⁸⁾ on monocrystalline niobium surfaces irradiated at 900°C with 500-keV $^4\text{He}^+$ ions show different blister shapes for

different orientation of the irradiated surface. The blisters formed on the Nb (111) surface had a "crow-foot" shape with three prongs as described earlier in Fig. 23. The blisters formed on the (110) plane had two major prongs, each with two facets. The two facets of one prong intersect along a line whose projection on the (110) plane is close to the $[1\bar{1}\bar{1}]$ direction and the two facets of the other prong intersect along a line whose projection is close to the $[1\bar{1}3]$ direction. The blister alignment with respect to each other had a two fold symmetry on the (110) plane. The blisters formed on the (001) surface plane had one major prong with two facets intersecting along a line whose projection on the (001) surface was in the $[110]$ direction. In addition, there were two lobes at one end of the blister. The orientation of the blisters with respect to each other had a four fold symmetry. These observations on blister shape in niobium have been related⁽¹¹⁸⁾ to the intersection of certain active slip planes with the surface plane of the monocrystal during blister formation.

Surface features similar to the "crow-foot" blisters described above in Fig. 23 have been reported by Clausing et. al.⁽¹¹⁹⁾ for a polycrystalline niobium sample oxidized at 850°C for 12 hours in oxygen at 5×10^{-5} Torr. These features were identified⁽¹²⁰⁾ as oxides formed due to internal oxidation and eruption of oxide particles through the surface of niobium samples. More recently Bauer and Thomas^(121, 122) observed similar surface features in polycrystalline niobium samples implanted with 150-keV H^+ at temperatures from 700°C to 1000°C and at doses up to 3×10^{19} ions cm^{-2} . They observed that the appearance of these surface features was also dependent on the procedure for polishing the surface. For electropolished samples, they observed these features both in the implanted area and in the unimplanted area; whereas for mechanically-polished surfaces these features were observed only in the irradiated area. They initially identified these features to be due to oxides and/or carbides of

Nb. This is in contrast to the observation by Das and Kaminsky⁽¹²³⁾, who did not observe any crow-foot blisters during helium implantation in unirradiated areas of electropolished niobium samples. Moreover, they observed⁽¹²³⁾ holes on the surface after a quick etching of the blistered surface and the thickness of the blister skin measured from the holes was close to the projected range of the ions. More recently Thomas and Bauer⁽¹²⁴⁾ have identified the surface features observed during their 150-keV H^+ implantation to be niobium carbide precipitate, Nb_2C , and not due to oxides. These different observations imply that surface features can arise by different processes even though they may display similar crystallographic features.

It may be of interest to note that monocrystalline Cu (111) surfaces show triangular shaped oxide microcrystallites after oxidation at elevated temperatures without being exposed to any irradiation.⁽¹²⁵⁾ Similar features have also been observed on (111) copper surfaces after sputtering with He^+ ions.⁽¹²⁶⁾ Similar triangular pits were observed by Kaminsky⁽¹¹⁾ due to rupture of deuterium blisters on the (111) surface of copper.

XIII. MODELS FOR BLISTER FORMATION:

One of the necessary conditions for blister formation in metals and alloys is the agglomeration of implanted gas atoms to form gas bubbles in near surface regions. Furthermore, for irradiation temperatures above liquid nitrogen temperature, a part of the lattice damage caused by the incident projectiles anneals out by recombination of interstitials and vacancies (Frenkel - pair recombination) due to the high mobility of self interstitials. Implanted inert-gas atoms such as helium are mobile at room temperature by interstitial diffusion (e.g. helium in W^(127, 128) and Ni⁽¹²⁹⁾) unless they are trapped. If both the vacancy and implanted-gas concentrations

are sufficiently high, trapping in the form of helium - vacancy complexes can occur. Experimental studies by Kornelsen et. al.^(128,129) and Picraux and Vook⁽¹³⁰⁾ show that more than one helium atom can be trapped at a vacancy to form such helium - vacancy complexes. Theoretical calculations by Wilson et. al.⁽¹³¹⁻¹³³⁾ for the binding energies of the helium atoms trapped at the vacancies in tungsten show reasonable agreement with the experimental binding energies for various postulated helium - vacancy complexes. These helium - vacancy complexes can be considered as embryos for nucleation of helium bubbles in metals. There is little theoretical work on the homogeneous nucleation of helium bubbles from these helium - vacancy complexes. Less theoretical work has been done on hydrogen implantation in metals from the diffusion and trapping point of view, than on helium in metals. As mentioned earlier in Sec. I, for hydrogen there is an added complication due to its chemical trapping in many metals. In the following discussion of models for blister formation, we will emphasize the formation of helium blisters.

Once the gas bubbles have nucleated, depending on the various parameters mentioned earlier, they can grow by absorbing gas atoms and/or vacancies or by coalescence. Experimentally, large diameter blisters (as described earlier) have been observed even at low temperatures where vacancies are not mobile. Qualitative models for the formation of blisters from the small gas bubbles (e.g. helium bubbles in molybdenum with diameters of 20-40 Å^o) have been suggested by a number of authors.^(29, 30, 38, 39, 56, 57, 98, 134-136) These fall into three major categories: the first category will be called the bubble coalescence model; the second category is based on the concepts of percolation theory and will be called the percolation model; the third

category considers mainly the stresses in the implanted layer and will be called the stress model.

Models based on coalescence of bubbles have been suggested independently by Blewer and Maurin, (56, 57) Das and Kaminsky, (30,134) McCracken⁽³⁹⁾ and Evans et. al. (38, 98) These models mostly consider the formation of helium blisters in metals. Since the diffusivity of helium in most metals at sufficiently low temperatures is very low, it is very unlikely that the small (20-40 Å diameter) bubbles will grow by diffusion of helium atoms into them. Also the mobility of helium bubbles in metals at low temperatures is very low (e.g. see estimates by Martin^(137, 138) for helium bubbles in niobium). However, growth of bubbles by absorption of vacancies is possible when the vacancies become mobile. As the total dose of helium ions increases, the density of these small bubbles increases. Very high densities of small helium bubbles have been observed in many metals irradiated to high doses ($> 1 \times 10^{17}$ ions cm⁻²). (29, 36, 98) Figure 25 shows an example in a transmission electron micrograph of molybdenum irradiated at room temperature with 18-keV He⁺ ions to a dose of 5×10^{17} ions cm⁻². (38,98) An outstanding feature of this micrograph is that it shows blisters (as can be seen from the bend contour pattern resulting from the dome-shaped blister skin) together with the small helium bubbles. Evans et. al.⁽⁹⁸⁾ observed high density of small bubbles both in the blister skin and in the areas in between the blisters.

At the critical dose for blister appearance the bubble density is high enough that the coalescence of small bubbles occurs. Figures 26 (a) - (e) show schematically various stages leading to blister formation. The transition from the stages shown in Fig. 26 (c) to Fig. 26 (e) occur very rapidly with only a relatively small increase in the helium-ion dose as the in-situ observations

indicate.⁽¹³⁹⁾ Evans⁽³⁸⁾ has suggested that the coalescence of bubbles becomes very rapid when the volume swelling $(\frac{\Delta V}{V})$ due to the gas bubbles in the implant region reaches a critical value. Thus the critical dose for blister appearance C_{bl} is governed by a critical value of volume swelling $(\frac{\Delta V}{V})_c$ due to the gas bubbles. At low temperatures, the initial coalescence of two bubbles will occur essentially at constant gas volume, thus leaving the total volume swelling in the implant region unchanged. The coalescence, once started becomes a run-away process and an unstable high pressure cavity easily (Fig. 26d) can form which leads to the plastic deformation of the surface layer. This dose at which the critical volume condition is reached can be considered as the critical dose for blister appearance, C_{bl} . Using this critical volume condition Evans et. al.⁽⁹⁸⁾ calculated the C_{bl} values for niobium and molybdenum irradiated with He^+ ions of different energies (30-300 keV) by assuming certain $(\frac{\Delta V}{V})_c$ values and certain initial helium bubble radii. A value of $(\frac{\Delta V}{V})_c \approx 50\%$ gave better agreement with the available experimental C_{bl} values.^(31, 35, 50)

Now let us consider the pressure in the unstable cavity that will lead to eventual plastic deformation of the top surface. Before coalescence the gas pressure p in a small bubble can be given by the well-known relation

$$p = \frac{2\gamma}{a} \quad (3)$$

where γ is the surface energy and a is the bubble radius. If we consider a bubble of radius $\sim 20 \text{ \AA}$ in niobium the pressure will be $2.1 \times 10^{10} \text{ dynes cm}^{-2}$ (20,725 atm), taking γ for niobium to be 2100 ergs cm^{-2} .⁽¹⁴⁰⁾ Now if two bubbles coalesce and the new bubble is assumed to be spherical with a volume that is sum of the volume of each bubble the new equilibrium gas pressure should be lower by a factor of $2^{1/3}$, but since the number of gas atoms has

not changed there will be an excess internal pressure. Thus as the coalescence progresses the excess internal pressure will increase rapidly. Once the excess pressure within the coalesced bubble (the unstable cavity in Fig. 26 d) exceeds a critical value it can deform the material above it to form visible blisters (Fig. 26 e). An expression for this critical pressure, p_{cr} , needed in the cavity to deform the surface layer can be derived by considering the stress needed for the onset of buckling in a circular plate that is fixed along its periphery and has a uniform pressure applied to it. For a uniform pressure p , a plate radius r , and a plate thickness t , the maximum stress (σ_r) can be written as^(141, 142)

$$\sigma_{r \max} = \frac{3pr^2}{4t^2} \quad (4)$$

The critical pressure, p_{cr} , in the gas filled cavity (Fig. 26 d) needed to deform the skin will be simply that value which makes the stress, σ_r , exceed the yield strength, σ_y , of the material. Thus,

$$p_{cr} = \frac{4\sigma_y t^2}{3r^2} \quad \text{as has been given earlier in Equation (2)}$$

This equation has been used by a number of authors^(16, 29, 30) for estimating gas pressure needed to form the blisters. For example, if we consider the blisters formed in niobium at room temperature during irradiation with 100-keV He^+ ions, the blister skin thickness t is 0.36 μm (as given earlier in Fig. 8). For a gas filled cavity with a radius of 0.1 μm , the critical pressure needed for blister formation will be $8.46 \times 10^9 \text{ dynes cm}^{-2}$ which is larger than the pressure ($\approx 4.2 \times 10^8 \text{ dynes cm}^{-2}$) in a spherical bubble of radius 0.1 μm estimated from Equation (3). Thus a cavity with a radius of 0.1 μm will not form a blister unless further coalescence occurs. Here we have assumed a spherical bubble but the coalescence is limited in the direction of the incident beam by the distribution in the implanted ions, and the cavity has a shape somewhat

like the one shown in Fig. 26 d. During the coalescence process the non-equilibrium pressure in the cavity cannot be estimated readily. It is possible that during the rapid coalescence process the cavity grows to larger diameters than needed to exceed the critical pressure. The coalescence can start at several regions in the irradiated area and in certain cases form a cavity extending over the entire irradiated region. This gives rise to a large blister covering most of the irradiated area as observed for high projectile energies^(58, 69, 71) [e.g. above 1.0 MeV in Nb and V]. One can view the rapid coalescence process as a crack growing near the tip of the cavity. Thus the extent of coalescence will also be limited to some extent by the bulk microstructure of the materials. It will be shown in the next section that for identical irradiation conditions the coalescence extends to much larger diameters for the annealed targets than for the cold-worked targets. In those cases where the coalescence extends very rapidly to give cavity diameters much larger than needed for plastic deformation, the excess pressure can build up to much higher values, and the blister skin can readily rupture and exfoliate. This exfoliation process has been sometimes called "flaking" and has been distinguished from blistering (see for example Ref. 111). This process occurs for higher implantation energies (> 100 keV) in many annealed metals in certain temperature ranges. Elevated temperatures help in the rapid coalescence and in increasing gas kinetic pressure, both of which lead to increased exfoliation. The temperature range in which this occurs has been discussed earlier in Sec. IX.

At high target temperatures (e.g. niobium at $800 - 900^{\circ}\text{C}$), helium bubbles have been observed to be faceted^(36, 37), and the average diameters range from 250 to 1500 Å which is larger than those observed at room temperature. In this temperature range the growth of helium bubbles to

such large sizes can occur by migration and coalescence of small bubbles as shown schematically in Figs. 26 (f)-(h). Some post-irradiation annealing studies on niobium implanted with α particles to a dose of 5×10^{20} α particles cm^{-2} indicate that during annealing at 950°C for 1 hour the growth of bubbles in the size range ($38 \text{ \AA} - 80 \text{ \AA}$ in diameter) occurs solely by migration and coalescence rather than by diffusion of helium between individual bubbles.⁽¹⁴³⁾ It is possible that the bubbles can migrate and coalesce to large enough diameters so that the internal pressure exceeds the critical value for blister formation (Fig. 26 h). However, it is also possible that near the critical dose for blister appearance a process similar to the rapid coalescence process (Fig. 26 d) at low temperature may occur. The diameter to which coalescence will continue will be lower than for the low temperature case because the critical pressure needed for deforming the top layer will be lower at high temperature due to the lower yield strength (σ_y) of the metal. Also the blister-skin exfoliation is reduced at high temperature, as was discussed earlier (see Sec. IX).

Another model of helium clustering leading to blister formation has been considered by Thomas and Bauer⁽²⁹⁾ and Wilson et. al.⁽¹³⁶⁾ using percolation theory.⁽¹⁴⁴⁾ Wilson et. al.⁽¹⁵³⁾ assume that the diffusion coefficient of helium atoms is concentration dependent and rapidly increases when the helium atoms become "connected" to each other. The percolation theory predicts the concentration required for the onset of an infinitely-connected region of helium atoms to occur. For bcc metals at an atom fraction of 0.243 and for fcc metals at an atom fraction of 0.199, helium atoms become "connected" to each other and hence mobile along this infinitely connected chain.⁽¹³⁶⁾ Now when the gas pressure in this interconnected helium layer exceeds the critical pressure needed for surface deformation (Eq. 2) blisters can form.

According to this model the critical dose for blister appearance is the dose at which the concentration of helium reaches the value for the onset of percolation (i.e. an atomic fraction of 0.243 for bcc and 0.199 for fcc metals). Wilson et. al.⁽¹³⁶⁾ calculated the critical dose for blister appearance C_{bl} as a function of diffusion coefficient of isolated helium atoms for 300-keV He^+ ion irradiation of Nb and Pd for a given ion flux. From the experimental values⁽³⁶⁾ of C_{bl} for 300-keV He^+ irradiation of Nb at 400°C ($\approx 1.2 \times 10^{18}$ ions cm^{-2}), they obtained an effective diffusion coefficient of helium in niobium to be $6.1 \times 10^{-13} \text{ cm}^2 \text{ sec}^{-1}$. It must be noted that this value is orders of magnitude higher than the values quoted by Blow⁽⁶¹⁾ for helium in niobium in the temperature range 600°C to 1200°C, and the difference may be due in part to radiation-enhanced diffusion of helium. Using such calculated effective diffusion coefficients for 15-keV H^+ irradiation of molybdenum and 300-keV He^+ irradiation of niobium and palladium, Wilson et. al.⁽¹³⁶⁾ showed that the critical dose for blister appearance C_{bl} should decrease with increasing flux, as observed by Verbeek and Eckstein⁽⁶²⁾ (Fig. 16a). However, one cannot readily explain the opposite trend observed by Möller et. al.⁽⁶⁹⁾ for 300-keV D^+ irradiation of nickel at low temperatures (Fig. 16b). This model does not take into consideration the yield strength of the metal which is strongly dependent on target temperature, and the target microstructure influences the critical dose for blister formation. The fact that the majority of the implanted helium is in the form of bubbles at high doses does not enter into this model directly.

The mechanism of blister formation discussed so far considers the blister formation to be due to plastic deformation of the surface layer resulting from high gas pressure in the bubbles. More recently Behrisch et. al.⁽⁷⁴⁾ and Roth⁽¹³⁵⁾ have suggested that for low-energy helium-ion irradiation of niobium

(< 15 keV) the blister formation may be due not to the high gas pressure but to the stresses induced in the implanted layer. The high concentration of bubbles give rise to large lateral stresses which are responsible for the deformation of the surface layer and the separation of the surface layer from the bulk is thought to occur near the end of the range.⁽⁷⁴⁾ So the thickness of the blister skin is equal to the width of the implanted layer which according to the author⁽¹³⁵⁾ is responsible for the observation that the blister skin thickness for niobium irradiated with low helium ion energies is larger than the calculated projected ranges. This interpretation is somewhat doubtful because it is not clear why the stress distribution in the implanted layer should be at a maximum at the end of the range of ions and not at the peak in the projected range distributions. As discussed earlier in Sec. V the higher blister skin thickness for low-energy implantations may in part be due to the swelling of the skin caused by a more uniform distribution of helium bubbles over a large portion of the implant depth.

Among the three basic models described above, the coalescence model attempts to give a qualitative explanation of many of the experimental observations, but none of the models considers all the parameters (e.g. diffusion of atomic helium under irradiation, migration of helium bubbles, the mechanical aspects such as gas pressure needed for onset of plastic deformation, yield strength of material, microstructural factors influencing the size and distribution of helium bubbles) sufficiently to explain the various observations.

One also has to consider the influence of surface erosion due to sputtering during irradiation. The depth distribution of the implanted gas atoms will change by this type of surface erosion particularly for the light ions with energies where the sputtering yield is near maximum.⁽¹⁴⁵⁾

It can be shown that the critical dose for blister appearance C_{bl} has to be lower than a value given by $\frac{t \rho N}{S A}$ in order that the blisters may form, where, t is the blister skin thickness, ρ is the density of the material N is Avogadro's number, S is the sputtering yield and A is the atomic weight of the metal. In cases where the blister skin thickness is unknown, a crude estimation of C_{bl} can be obtained by taking t equal to R_p , the projected range of ions in the metal. However, as discussed earlier, t can be 2-3 times larger than R_p particularly at low projectile energies. For cases where the sputtering yields are very high and depth of penetration R_p is shallow (for example for heavier ions such as Ar, Kr, Xe at low energies) blisters may not form at all. Using the C_{bl} value given by the percolation theory, Roth⁽¹³⁵⁾ showed that for niobium implanted with Ar, blisters may form at energies above 100 keV, whereas for light ions like helium, blisters can form at energies down to 1 keV. Blisters have indeed been observed in niobium during He^+ irradiation for energies down to 1 keV.⁽³¹⁾ However when the metal has a high atomic number, the sputtering yield for heavy ions may not be so high, and blisters may form. More recently, blisters have been observed in uranium irradiated with 25-keV Ar^+ ions. It may be also noted that blisters have been observed in En 40 B steel under irradiation with 200-keV N_2^+ ions to a total dose of 4.0×10^{17} ions cm^{-2} .⁽¹⁴⁶⁾

XIV. REDUCTION OF SURFACE DAMAGE CAUSED BY RADIATION BLISTERING:

From the discussions so far it is clear that considerable surface damage can be caused to metals by radiation blistering, particularly during irradiation with light inert gas ions such as helium. Recently some attention has been paid to investigating methods for the reduction of surface erosion due to radiation blistering. In this section we will discuss some of these methods.

- a) High Target Temperature: From the earlier discussions on the effect of target temperature (Sec. IX) it is obvious that one possible way to reduce radiation blistering is to maintain the target surface at high enough temperature so that the implanted gas is released without forming large bubbles. For many metals a great reduction in surface erosion due to radiation blistering is observed (Fig. 19) at temperatures above $0.4 - 0.5 T_m$ where T_m is the melting point in K. If the irradiation temperature is sufficiently high, helium re-emission during irradiation can reach almost 100% of the incoming flux, and helium agglomeration into bubbles can be prevented. For example, for vanadium and niobium irradiated at 1200°C with 300-keV He^+ ions almost 100% re-emission has been observed.⁽⁶⁷⁾ Irradiation under these conditions gives rise to a porous surface structure consisting of micron size holes with a spacing of several microns as shown earlier for vanadium in Fig. 18. Recently Bauer and Thomas⁽¹⁰⁸⁾ observed that the porous structure in niobium obtained by 300-keV He^+ implantation at 1200°C is rather stable. This type of surface prevented further blister formation during 300-keV He^+ irradiation at $400^\circ\text{C} - 600^\circ\text{C}$ for a dose of $\sim 4 \times 10^{18} \text{ ions cm}^{-2}$ where severe exfoliation of blister skin is normally observed.⁽¹⁰⁸⁾ However, in many applications it may not be always possible to maintain the surface at sufficiently high temperatures. For example, in a controlled thermonuclear fusion reactor the operating temperatures of various components exposed to D, T and He projectiles may be limited by other design criteria. In accelerator technology, maintaining components (exposed to energetic projectiles) at high temperatures may be a problem. A more desirable solution would be to choose materials with microstructure which minimizes the formation of blisters. Only a few microstructural parameters have been investigated so far, and some of them will be discussed below.

- b) Cold Working: Cold working has been observed to reduce surface erosion due to blistering in certain cases.^(30, 37) Figure 27 shows an example⁽³⁷⁾ for polycrystalline vanadium samples irradiated with 500-keV $^4\text{He}^+$ ions to a total dose of 6.2×10^{18} ions cm^{-2} . For the annealed sample irradiated at room temperature (Fig. 27a) the blister diameters range from 15 to 350 μm , and the blister skin has ruptured and fallen off at many places. In the cold-worked sample (Fig. 27b) irradiated under identical conditions most of the blisters have diameters in the range of 5 - 100 μm , and no large scale exfoliation is observed. For irradiation of the same type of samples at 900°C the blistering is similar for the annealed (Fig. 27c) and the cold-worked cases (Fig. 27d). Similar results have been observed for cold-worked and annealed niobium⁽³⁰⁾ samples. It should be pointed out that for irradiation at 900°C some annealing of vanadium and niobium can occur.

Thus, it appears that cold working tends to reduce blister size and blister exfoliation at room temperature, but the effect is not so pronounced above the recrystallization temperature. Several factors may be responsible for the reduction in blistering in cold-worked samples as compared to the annealed ones. The lower yield strength of the annealed material as compared to the cold-worked one will increase blister rupture and exfoliation in the annealed sample. Moreover, the increased dislocation density and the large number of subgrains in cold-worked materials can give rise to a much finer dispersion of helium bubbles and also prevent coalescence to larger diameters.

- c) Grain Size and Dispersion of Second Phase: There exist no systematic studies of the effect of grain size and dispersion of second phase particles on the reduction of blistering. Some recent preliminary studies

on sintered aluminum powder (SAP) containing a nominal 10.5 wt% Al_2O_3 (SAP 895) show a reduction in erosion due to blistering as compared to annealed aluminum under identical irradiation conditions. (148, 149)

Figure 28 illustrates this for SAP 895 as compared to annealed aluminum for 100-keV He^+ ion irradiation to a dose of 6.2×10^{18} ions cm^{-2} at room temperature and at 400°C. Figure 28(a) shows an enlarged view of a portion of an aluminum surface irradiated at room temperature. One can see that four exfoliated layers have been removed. Figure 28(b) shows a portion of an aluminum surface which had been irradiated at 400°C under otherwise identical conditions. The exfoliation of blister skin is reduced at 400°C as compared to room temperature and there is only one exfoliated layer in several areas. Figure 28(c) and (d) show typical examples of blisters formed on SAP 895 irradiated at room temperature and at 400°C, respectively. In contrast to the aluminum, where extensive exfoliation is observed, only a few blisters were ruptured in the case of SAP 895. The erosion rates estimated for the room temperature case from the ruptured and lost skins for aluminum and SAP are 1.75 ± 0.25 and 0.001 atoms per helium ion, respectively. (148)

The results for annealed aluminum held at 400°C give a value of 0.12 ± 0.05 atoms per helium ion, whereas for the SAP 895 sample no exfoliation of the blisters could be observed.

The drastic reduction in erosion yield in sintered aluminum powder as compared to annealed aluminum was attributed⁽¹⁴⁹⁾ to the dispersion of trapped helium at the large Al- Al_2O_3 interfaces at the large grain boundaries in SAP. Formation of helium bubbles at the Al_2O_3 and aluminum interface in aluminum alloys containing dispersed Al_2O_3 particles has been observed. The average grain size in SAP 895 was

$\sim 0.5 \mu\text{m}$ as compared to $\sim 300 \mu\text{m}$ for annealed aluminum. This large grain boundary area in SAP gives a much finer dispersion of helium bubbles than in aluminum and may prevent helium-bubble coalescence to very large diameters. Furthermore, the fact that the yield strength of SAP 895, for example, is much higher ($\sim 35,600 \text{ psi}$) than for annealed aluminum ($\sim 1,700 \text{ psi}$) will help to reduce the blister rupture and exfoliation rate in SAP from that observed in aluminum.

Somewhat similar results have been recently observed for vacuum-cast and sintered beryllium irradiated with 100-keV He^+ ions.⁽¹⁵¹⁾ For vacuum-cast beryllium irradiated at room temperature to a dose of $6.2 \times 10^{18} \text{ ions cm}^{-2}$, blisters with average diameters ranging from $5 \mu\text{m}$ to $35 \mu\text{m}$ with some exfoliation were observed, whereas for sintered beryllium the blisters were smaller (average diameter of $5 - 15 \mu\text{m}$), and there was no exfoliation. For irradiation at 600°C with 100-keV He^+ ions to a dose of $3.1 \times 10^{18} \text{ ions cm}^{-2}$, the vacuum-cast beryllium showed considerable blister exfoliation (erosion yield $\sim 0.3 \pm 0.1 \text{ Be atoms per incident helium ion}$), whereas the sintered beryllium showed greatly-reduced blister exfoliation even for a higher dose of $6.2 \times 10^{18} \text{ ions cm}^{-2}$ (erosion yield $\sim 0.02 \pm 0.01 \text{ Be atoms per incident helium ion}$).

FUTURE DIRECTIONS

Even though a great deal of information has been generated on radiation blistering in metals complete understanding of this phenomenon has not yet been obtained. A unified quantitative theory for this phenomenon is lacking. It appears that considerable amount of additional experimental information is still needed on a large number of parameters (e.g. critical dose for bubble and blister appearance on targets with different microstructures, grain

sizes, yield strengths and target temperatures) before such a theory can be developed.

Since radiation blistering has been identified as an important erosion process for surfaces exposed to plasmas, it is important to study this process for the radiation environments expected in controlled thermonuclear fusion reactors. While, at present, most of the data have been obtained for monoenergetic ion irradiations in a fusion reactor, one expects to have ions with a broad energy distribution impinging on the exposed surfaces. Therefore, in order to assess the severity of surface erosion by radiation blistering in thermonuclear devices studies are needed with ions having a broad energy spectrum.

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FIGURE CAPTIONS

- Fig. 1. Scanning electron micrographs (SEMs) of annealed polycrystalline niobium surfaces irradiated at 700°C:(a) with 500-keV $^4\text{He}^+$ ions to a total dose of 6.2×10^{18} ions cm^{-2} ; (b) with 250-keV D^+ ions to a total dose of 1.25×10^{19} ions cm^{-2} (Ref. 58).
- Fig. 2. Optical micrographs of annealed polycrystalline molybdenum surfaces irradiated at room temperature:(a) with 15-keV He^+ ions to a dose of 2.5×10^{18} ions cm^{-2} ; (b) with 15-keV D^+ ions to a dose of 2×10^{18} ions cm^{-2} . The insets in Figs. 2 (a) and 2 (b) are SEMs showing enlarged views of some of the blisters (Ref. 62).
- Fig. 3. SEMs of (a) monocrystalline Ni (110) surface irradiated at room temperature with 500-keV $^4\text{He}^+$ ions to a dose of 6.2×10^{18} ions. cm^{-2} ; (b) polycrystalline nickel surface irradiated at -153°C with 400-keV D^+ ions to a dose of 1.25×10^{19} ions cm^{-2} (Ref. 69). The blisters in Fig. 3 (b) were observed at room temperature after irradiation at -153°C.
- Fig. 4. SEMs of annealed polycrystalline vanadium surfaces irradiated at room temperature:(a) with 100-keV $^4\text{He}^+$ ions to a dose of 3.1×10^{18} ions cm^{-2} (the inset shows an enlarged view of some of the blisters); (b) with 250-keV $^4\text{He}^+$ ions to a dose of 3.1×10^{18} ions cm^{-2} ; (c) with 500-keV $^4\text{He}^+$ ions to a dose of 6.2×10^{18} ions cm^{-2} ; (d) with 1000-keV to a dose of 6.2×10^{18} ions cm^{-2} .
- Fig. 5. Blister diameter as a function of projectile energy for niobium and molybdenum irradiated at room temperature with helium ions.
- Fig. 6. Histogram of the blister diameter distribution in cold-worked polycrystalline niobium irradiated at room temperature with 500-keV He^+ ions to a dose of 6.2×10^{18} ions cm^{-2} (Ref. 30).

FIGURE CAPTIONS (Continued)

Fig. 7. SEMs of blister skins in polycrystalline vanadium irradiated at room temperature with $^4\text{He}^+$ ions with energies of: (a) 100 keV, (b) 250 keV, (c) 500 keV and (d) 1000 keV; the total dose in (a) and (b) was 3.1×10^{18} ions cm^{-2} and in (c) and (d) was 6.2×10^{18} ions cm^{-2} .

Fig. 8. Projected ranges of $^4\text{He}^+$ ions in niobium as a function of projectile energy. The dotted curve marked "LSS" was calculated using Thomas-Fermi nuclear stopping and the velocity-proportional electronic stopping cross sections of Lindhard, et. al. solid curve marked "Brice" was calculated using Thomas-Fermi nuclear stopping and Brice's semiempirical expression for electronic stopping. The data points with error bars are measured blister skin thicknesses for niobium irradiated at room temperature with He^+ ions, as measured by various authors.

Fig. 9. $\langle 100 \rangle / \langle 111 \rangle$ double aligned backscattered energy distribution of 150 keV protons from a niobium single crystal after irradiation at room temperature with 4-keV He^+ ions in a random direction (incident at 12° off the surface normal) for different doses as indicated (Ref. 31).

Fig. 10. Plots of projected-range probability distributions (as shown by the dashed curves) as a function of depth into (a) Al target, (b) V target and (c) Nb target for different energies of the incident $^4\text{He}^+$ ions. The solid curves are plots of energy deposited into damage as a function of depth into the targets for $^4\text{He}^+$ ions with various projectile energies. The data points with error bars are measured blister-skin thicknesses for helium-ion energies of (a) 100 keV, (b) 250 keV, (c) 500 keV, (d) 1000 keV, and (e) 1500 keV (Ref. 65).

FIGURE CAPTIONS (Continued)

- Fig. 11. Critical dose for blister appearance C_{bl} as a function of projectile energy for niobium and molybdenum irradiated at room temperature with $^4\text{He}^+$ ions.
- Fig. 12. Critical dose for blister appearance as a function of target temperature for niobium irradiated by helium ions.
- Fig. 13. SEMs of surfaces of annealed Type 304 stainless steel after irradiation at 450°C with 500-keV $^4\text{He}^+$ ions for total doses of: (a) 6.2×10^{17} ions cm^{-2} ; (b) 3.1×10^{18} ions cm^{-2} ; (c) 6.2×10^{18} ions cm^{-2} . (Ref. 58).
- Fig. 14. SEMs of polycrystalline niobium irradiated at room temperature with 15-keV $^4\text{He}^+$ ions to total doses of: (a) 3.1×10^{18} ions cm^{-2} ; (b) 6.2×10^{19} ions cm^{-2} (Ref. 28). The target surface in Fig. 14 (a) was annealed and electropolished, whereas in Fig. 14 (b) it was cold-worked and mechanically polished prior to irradiation.
- Fig. 15. SEMs of surfaces of annealed polycrystalline vanadium after irradiation at 900°C with 500-keV $^4\text{He}^+$ ions for a total dose of 6.2×10^{17} ions cm^{-2} at fluxes of: (a) 1×10^{13} ions $\text{cm}^{-2} \text{sec}^{-1}$; (b) 1×10^{14} ions $\text{cm}^{-2} \text{sec}^{-1}$; (c) 1×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$ (Ref. 105).
- Fig. 16. The dependence of critical dose for blister appearance C_{bl} on the incident ion flux for: (a) 15-keV H^+ irradiation of molybdenum at room temperature (Ref. 62); (b) 300- and 350-keV D^+ irradiation of nickel at -153°C (Ref. 69).
- Fig. 17. SEMs of annealed polycrystalline vanadium irradiated with 500-keV $^4\text{He}^+$ ions to a total dose of 6.2×10^{18} ions cm^{-2} at: (a) room temperature; (b) 300°C; (c) 500°C; (d) 600°C; (e) 900°C (Refs. 58, 68).

FIGURE CAPTIONS (Continued)

- Fig. 18. SEM of annealed polycrystalline vanadium irradiated at 1200° C with 300-keV $^4\text{He}^+$ ions to a dose of 2×10^{18} ions cm^{-2} (Ref. 63).
- Fig. 19. Erosion rates for different metals and alloys as a function of irradiation temperature for different projectile energies and total doses: (i) Type 316 Stainless Steel with 300-keV $^4\text{He}^+$ for 4×10^{18} ions cm^{-2} ; ---, (ii) Type 304 Stainless Steel with 100-keV $^4\text{He}^+$ for 3.1×10^{18} ions cm^{-2} ; Δ , (iii) Type 304 Stainless Steel with 500-keV $^4\text{He}^+$ for 3.1×10^{18} ions cm^{-2} ; $\cdot$, (iv) annealed vanadium with 500-keV $^4\text{He}^+$ for 6.2×10^{18} ions cm^{-2} ; $\square$, (v) annealed niobium with 500-keV $^4\text{He}^+$ for 6.2×10^{18} ions cm^{-2} ; X, (vi) annealed aluminum with 100-keV $^4\text{He}^+$ for 6.2×10^{18} ions cm^{-2} ; O, (vii) annealed niobium with 100-keV $^4\text{He}^+$ for 3.1×10^{18} ions cm^{-2} ; $\blacksquare$.
- Fig. 20. Helium re-emission from vanadium during 300-keV He^+ implantation as a function of dose at three different implantation temperatures (Ref. 64).
- Fig. 21. Dose for the onset of first re-emission burst for Nb, Mo, V and Type 316 Stainless Steel as a function of irradiation temperature during implantation with 300-keV He^+ ions. These doses were taken from the helium re-emission measurements by Jauer, et. al. (Ref. 36, 64).
- Fig. 22. SEMs of (111) surface planes of niobium monocrystal after room temperature irradiation to total dose of 6.2×10^{18} ions cm^{-2} of 500 keV $^4\text{He}^+$ ions: (a) incident at $\sim 5^\circ$ with respect to surface normal (unchanneled ions); (b) channeled axially in the [111] direction (Ref. 71).
- Fig. 23. SEMs of (111) monocrystalline niobium surfaces after irradiation at 900°C to total dose of 6.2×10^{18} ions cm^{-2} of 500-keV He^+ ions: (a) well channeled

FIGURE CAPTIONS (Continued)

- Fig. 23. along the [111] axis; (b) incident at $\sim 5^\circ$ off the surface normal (unchanneled) (Ref. 116).
- Fig. 24. SEM of annealed polycrystalline niobium surface after irradiation at room temperature with 15-keV He^+ ions to a dose of 3.1×10^{18} ions cm^{-2} showing different blister sizes in different grains.
- Fig. 25. Transmission electron micrograph of molybdenum irradiated at 25°C with 18-keV He^+ to a dose of 5×10^{17} ions cm^{-2} showing dome-shaped blisters [after Evans, see e.g. Ref. 38, 98].
- Fig. 26. Schematic diagrams of mechanisms of blister formation in metals at: (a) - (e) low temperatures; (f) - (h) high temperatures.
- Fig. 27. SEMs of polycrystalline vanadium surfaces after irradiation with 500-keV He^+ ions to a total dose of 6.2×10^{18} ions cm^{-2} :
(a) an annealed sample at room temperature; (b) a 40% cold-worked sample at room temperature; (c) an annealed sample at 900°C ; (d) a 40% cold-worked sample at 900°C (Ref. 37).
- Fig. 28. SEMs of surfaces of: (a) annealed aluminum irradiated at room temperature; (b) annealed aluminum irradiated at 400°C ; (c) SAP 895 irradiated at room temperature; (d) SAP 895 irradiated at 400°C with 100-keV $^4\text{He}^+$ ions to a total dose of 6.2×10^{18} ions cm^{-2} .

Table 1. MAJOR PARAMETERS THAT CAN AFFECT RADIATION BLISTERING

A. Target-Related Parameters

1. Type of target metal or alloy
2. Target temperature
3. Target microstructure:
 - a) grain size
 - b) initial defect density (e.g. cold worked vs. annealed structures)
 - c) effects of precipitates: size distribution, volume fraction and type of precipitates
4. Yield strength and rupture strength of target material
5. Crystallographic orientation of irradiated surface
6. Target surface finish

B. Projectile-Related Parameters

1. Type of projectile
2. Projectile energy
3. Total dose (fluence)
4. Dose rate (flux)
5. Channeling condition of projectile
6. Angle of incidence of projectile

C. Parameters Affected by Target-Projectile Combinations

1. Diffusivity and solubility of projectile in metals and alloys
2. Critical dose for blister appearance.

Appendix

Table A 1. List of targets for which radiation blistering has been investigated for hydrogen-isotope irradiation with the irradiation conditions.

Projectile Type	Target Metal or Alloy†	Irradiation Conditions			References
		Projectile Energy or Energy Range (keV)	Dose or Dose Range (ions cm ⁻²)	Irradiation Temperature (°C)	
H ₃ ⁺ H ⁺	Be	100	2x10 ¹⁹	Room Temp.	152
	Al	10-200	5x10 ¹⁶ -3.5x10 ¹⁷	-196 to 200	16,117
	Ti	20	5x10 ¹⁸ -5x10 ¹⁹	45 to 150	153
	V	150	1.5x10 ¹⁹ -2.5x10 ¹⁹	-105 to 115	63,64
	Type 302 St. Steel	20	1x10 ¹⁹ -2x10 ²⁰	75 to 150	153
	Type 316 St. Steel	20, 150	1x10 ¹⁹ -2x10 ¹⁹	-93 to 350	153,63,64
	Type 304 St. Steel	5	4.9x10 ¹⁸ -9.9x10 ¹⁹	Room Temp.	20
	Cu	70-140	1.5x10 ¹⁸ -8x10 ¹⁹	Room Temp.	12,152,154
	Nb (100)	10	2x10 ¹⁹	Room Temp.	31
	Mo	15, 150	2x10 ¹⁸ -6x10 ¹⁸	-115 to 100	62,63,64
H ₂ ⁺ H ⁺	Sn (100)	2200, 3800	6.3-6.8x10 ¹⁷	Room Temp.	155
	Au	50, 100	not known	Room Temp.	117
	Be	15	1x10 ¹⁸	Room Temp.	62
	Cu (100)	125	1.8x10 ¹⁹	Room Temp.	11
	Cu (110)	125, 800	-	Room Temp.	156
	Ni	150 - 400	6x10 ¹⁸ -1.3x10 ¹⁹	-153	69
	St. Steel Type 4301	15	5x10 ¹⁸ -6x10 ¹⁸	27 and 343	62
	Nb	250, 300	1x10 ¹⁹ -1.9x10 ¹⁹	550-700	58,59,105,157,158
	Mo	15	8x10 ¹⁸	Room Temp.	62
	Mo (100)	150		Room Temp.	62
D ⁺	Be	15	1x10 ¹⁸	Room Temp.	62
	Cu (100)	125	1.8x10 ¹⁹	Room Temp.	11
	Cu (110)	125, 800	-	Room Temp.	156
	Ni	150 - 400	6x10 ¹⁸ -1.3x10 ¹⁹	-153	69
	St. Steel Type 4301	15	5x10 ¹⁸ -6x10 ¹⁸	27 and 343	62
	Nb	250, 300	1x10 ¹⁹ -1.9x10 ¹⁹	550-700	58,59,105,157,158
	Mo	15	8x10 ¹⁸	Room Temp.	62
	Mo (100)	150		Room Temp.	62

†All targets are polycrystalline materials unless otherwise indicated.

Table A 2. List of Targets for which radiation blistering has been investigated for helium ion irradiation together with irradiation conditions.

Target Metal or Alloy	Irradiation Conditions			References
	Projectile Energy or Energy Range (keV)	Dose or Dose Range (ions cm ⁻²)	Irradiation Temperature (°C)	
Be and Sintered Be Powder	100	3.1-6.2x10 ¹⁸	Room Temp. to 600	151
Al and Sintered Al Powder	100-1000	3.1-6.2x10 ¹⁸	Room Temp. to 400	148, 65, 149
V	100-1000	~ 1x10 ¹⁷ -6.2x10 ¹⁸	Room Temp. to 1200	36, 37, 58, 63-67, 105, 109, 135
V (111)	500	6.2x10 ¹⁷ -6.2x10 ¹⁸	650 to 750	37
V-20% Ti	500	6.2x10 ¹⁸	Room Temp. to 900	109
Type 4301 Stainless Steel	15	3x10 ¹⁸	27	62
Type 304 Stainless Steel	100-1500	6.2x10 ¹⁷ -6.2x10 ¹⁸	Room Temp. to 550	19, 68, 110
	6000 α-particles from ²⁴² Cm oxide)	3x10 ¹³	Room Temp.	21
Type 316 Stainless Steel	300	4x10 ¹³	-170 to 700	63, 64, 111
Ni	36	-	Room Temp.	35
	40-140	up to 4x10 ¹⁸	-	12
Ni (110)	500	6.2x10 ¹⁸	500 to 950	70
Cu	140	up to 4x10 ¹⁸	-	12, 35
Zr	-	-	-	35
Nb	1-15	3.1x10 ¹⁷ -7x10 ²⁰	Room Temp. to 700	29, 28, 106, 107, 135
	100-1500	6.2x10 ¹⁶ -6.2x10 ¹³	-170 to 1200	30, 33, 36, 37, 58, 59, 67, 73, 116, 105, 108
Nb (100)	1-15	1.4x10 ¹⁶ -4x10 ¹⁷	-110 to 1000	31, 42, 50, 74, 106, 135, 159
Nb (100), (110), (111)	500-150	6.2x10 ¹⁷ -6.2x10 ¹⁸	Room Temp. to 900	59, 71, 116, 118
Mo	7-80	1x10 ¹⁶ -1x10 ¹⁸	Room Temp. to 1327	35, 38, 39, 98
	15	2.5x10 ¹³	Room Temp.	62
	300	~ 1x10 ¹⁷ -4x10 ¹⁸	400 to 1200	36, 63, 64
Mo (100)	150	-	Room Temp.	62
Pd	300	7.5x10 ¹⁷ -2x10 ¹³	-180 to 200	29, 160
	4800-5700	~ 1x10 ¹⁷	Room Temp.	34, 161
Sn (100)	1500-3800	6.2x10 ¹⁷ -1.8x10 ¹⁸	Room Temp.	155
Er	160	5x10 ¹⁶ -1.5x10 ¹⁸	Room Temp.	40, 56, 57
Er D _{1.4} Er D _{1.8}	160	3x10 ¹⁷	Room Temp.	56
W	-	-	-	35
Re	20	9x10 ¹⁶ -3x10 ¹⁸	27	162
Pt	36	-	Room Temp.	35
	6000 α-particles from ²⁴² Cm oxide)	-	-	-
		5x10 ¹⁷	Room Temp.	21
Au	100-1500	3.1x10 ¹⁵ -9.4x10 ¹³	Room Temp.	163

All target metals and alloys are polycrystalline unless otherwise indicated.

FIGURE 1

FIGURE 2

FIGURE 3

FIGURE 4

FIGURE 5

FIGURE 6

FIGURE 7

FIGURE 8

FIGURE 9

FIGURE 10

FIGURE 11

FIGURE 12

FIGURE 13

FIGURE 14

FIGURE 15

FIGURE 16

FIGURE 17

FIGURE 18

FIGURE 19

FIGURE 20

FIGURE 21

UNCHANNELED

CHANNELED

FIG. 22

FIGURE 23

FIGURE 24

FIGURE 25

BLISTER FORMATION IN METALS

FIGURE 26

ANNEALED

COLD WORKED

AT ROOM
TEMPERATURE

AT 900°C

FIGURE 27

ALUMINUM

SAP 895

AT ROOM
TEMP.

AT 400°C

FIGURE 28