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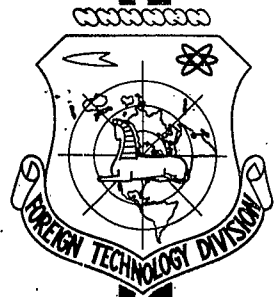
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TRANSLATION

ANGULAR FEATURES OF THE DISINTEGRATION OF METALS BY ION BEAMS

By

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FOREIGN TECHNOLOGY DIVISION

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ANGULAR FEATURES OF THE DISINTEGRATION OF METALS BY ION BEAMS

V. A. Molchanov and V. G. Tel'kovskiy

Several years ago a study of the angular features of the disintegration of metals by ion beams was begun under the auspices of the Department of Atomic Physics at Moscow University. This problem, as L. A. Artsimovich has emphasized, is becoming more timely and urgent every year owing to the increase in work on radiation processes in solids. The technical aspects are also important: A knowledge of the angular features of metal disintegration is necessary for designing thermonuclear reactors (since the cooling rate of a high-temperature plasma depends to a considerable degree on the presence of even a small amount of heavy element impurities in the plasma), as well as ion jet engines for interplanetary rockets [1]. For this reason it was decided to give special attention to the study of the disintegration of metals when the angle of incidence of the ions onto the target is close to the glancing angle.

1. Apparatus for Obtaining Ion Beams.

The growth of the work necessitated the creation of an apparatus for obtaining monoenergetic ion beams. The vagueness which

has existed until recently in the understanding of the mechanism of cathode sputtering was explained to a considerable degree by the absence of reliable experimental data and the contradictory nature of the results obtained by different researchers [2, 3]. Very many experiments were being conducted in gas discharges, i.e., under conditions extremely remote from those necessary for obtaining dependable results [4].

A large mass spectrometer apparatus was constructed having double focusing of the ion beam in a sectorial magnetic field, low angular ion beam convergence, and low residual gas pressure near the focal spot of the apparatus in the chamber where the targets are bombarded by ions [5].

2. Sputtering Coefficient of Polycrystalline Surfaces.

The results of measurements of the sputtering coefficient differ appreciably in even the most carefully conducted works. For example, the sputtering coefficient of copper by argon ions (energy ~25 kev) according to data obtained by Yonts et al. is 9 atoms/ion [6], while according to data published by Rol and his colleagues it is almost 25% less [7]. A special study was undertaken to ascertain the reasons for so large a discrepancy. It was found that the sputtering coefficient increased with an increase in the length of the irradiation time [8]. Such a dependence had been discovered earlier by many authors (cf. for example Massey and Barhope [2]). However, the majority of them explained it by the removal of adsorbed molecules of air and contaminants from the surface of the sample [9]. The other explanation in the literature is that of reverse diffusion of the sputtered material onto the surface of the sample [10]. Both explanations appear to be untenable in application to our experiments. The

pressure near the target was $2 \cdot 10^{-7}$ mm Hg, and at such pressures reverse diffusion does not play a significant role [11]. The surface contaminations may be primary, arising during the polishing and long storage of the sample, and secondary, arising when the sample is withdrawn from the vacuum for weighing, and occurring because of the oil film appearing on the surface of the target during the preliminary evacuation of the apparatus. We avoided primary contaminants by scouring the sample chemically and roasting it in a vacuum oven. Secondary contaminations remain; apparently, however, they cause no observable effect. Were this not so, we would have obtained identical results each time for the same dose of irradiation regardless of the degree of previous erosion of the surface. Actually, different results were obtained for the same dose of irradiation depending on whether we irradiated a polished surface or a surface already disintegrated by previous irradiations (cf. Fig. 1 in a previously published paper [8]). The independence of the sputtering coefficient of a single crystal on the duration of the irradiation and the fact that the sputtering coefficient of a surface specially contaminated with oil is the same under the conditions of our experiments, within the limits of the accuracy of the measurements, as that of an uncontaminated surface, also support the assumption that the degree of surface disintegration is significant. The experimental setup permitted the condition of the surface of the sample to be controlled with respect to secondary emission current. Usually this current varies during the first 5-10 minutes of irradiation, after which it becomes constant. It apparently makes sense that the sputtering coefficient of "smooth" and "eroded" polycrystalline surfaces should differ. For commercial copper this difference is 25%, i.e., it exactly explains the discrepancy in the results of the earlier papers.

[6, 7].

At small sputtering coefficients it makes sense to shape the surface with heavy ions and only after this to sputter it with light ions. Some of Dushkov's results [12] were obtained on a previously shaped surface.

The question of the mechanism of the shaping of a sputtered surface has been studied [13]. Apparently, the results obtained do not yet permit a unique conclusion to be drawn about this process.

3. Angular Features of Sputtering in Polycrystals.

The radiation mechanism was first applied to polycrystals [14, 15]. It was of interest therefore to compare the theoretical conclusions theories with experiment. In addition, the features of sputtering of polycrystalline samples, particularly when the angle of incidence of the ions onto the target is close to the glancing angle, are of technical interest. Radiation theories contain a number of assumptions and therefore not all of their conclusions carry the same assurance. In the opinion of the authors of one of the theoretical papers [15], the most reliable conclusion from the theory proposed by them is the dependence of the sputtering coefficient on the angle of incidence of the ions onto the target. This dependence was subjected to verification. To determine the sputtering coefficient it is necessary to know the value of the ion current, the time of irradiation, and the target weight loss due to the irradiation. For correct measurement of the current it was necessary to use the circuit in Fig. 1. Diaphragm D serves to core out the portion of the beam with maximum current density. Lens L prevents the passage of electrons both from the beam into the chamber and from the chamber

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into the beam. Sample O is fastened to a turning mechanism which at the same time serves to draw off heat. The target temperature is measured with the aid of thermocouple T. The sum of the ion current and secondary emission current is measured simultaneously with the ion current. The chamber walls are cooled with liquid nitrogen to improve the vacuum.

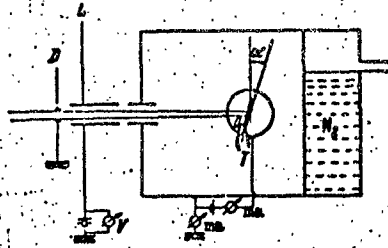


Fig. 1.

It has been established [8 and 12] that in accordance with Glodmann's theory [15] the sputtering coefficient increases with an increase in the angle of incidence of the ions onto the target α in inverse proportion to the cosine of the angle of incidence, the numerical values of the coefficients for normal incidence of the ions being practically coincident with the results obtained by Yonts [6]. A deviation from the cosine law is noted at large angles of incidence. This deviation cannot be explained merely by the decrease in the transfer of energy from the ions to the target due to the reflection of high energy particles. The deviation from the cosine law for ions of the same mass begins the earlier (at lesser angles of incidence) the lower the energy of the ions, while for ions of the same energy it ensues the earlier the greater the mass. Apparently, these deviations cannot be explained by the effect of the microcontour

of the sample either, since measurements on helium have shown no decrease in the sputtering coefficient even at angles of incidence of 84° .

4. Anisotropy of the Sputtering Coefficient of Single Crystals.

Study of the sputtering of monocrystalline samples presents much pure scientific interest [16] and is essential for an understanding of the dynamics of the shaping of surfaces obtained during the irradiation of polycrystalline samples by ion beams. The results of an experiment which have been previously published [17, 18] and presented in Fig. 2. Curve 1 corresponds to rotation of the crystal about a diagonal of a cube face, while curve 2 corresponds to rotation about a cube edge. It is apparent that the dependence of the sputtering coefficient on the angle of incidence of the ions is not monotonic in nature. On the average the sputtering coefficient increases with the angle of incidence in inverse proportion to the cosine of this angle. However, when the direction of the ion beam approaches the directions of the principal crystallographic axes of the targets, the sputtering coefficient decreases sharply. This decrease occurs at the angles 0° ($\{100\}$ axis), 35° ($\{112\}$ axis), and 55° ($\{111\}$ axis). The decrease in the sputtering coefficient on curve 2 corresponds to the angles 0° ($\{100\}$ axis) and 45° ($\{110\}$ axis).

The angular width of the dip near the $\{100\}$ axis does not depend on the mass and energy of the ions within the limits of accuracy of the experiment ($\pm 2^\circ$). It was of interest to ascertain to what extent the inaccuracy of the slice affected the anisotropy of the sputtering coefficient. For this purpose the angular dependence was studied in the case when the plane of the slice made at 20° angle with the

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($\{110\}$ plane). Comparing curves 1 and 3, we can note that despite the considerable deviation of the slice plane from the crystallographic plane, the curves differ little. On curve 3 the minimum of the sputtering coefficient is achieved when the direction of the ion beam coincides with the direction $[100]$, instead of with the direction of the normal to the plane of the slice. This suggests the idea that the unsputtered grains present on a sputtered surface were retained because they proved to be oriented with one of their principal axes in the direction of the incident ion beam. To check this assumption it was necessary to perform an X-ray diffraction study of the grains on a polycrystalline surface which had been subjected to irradiation by an ion beam. This was carried out by I. A. Shakh-Malikova at our request. It was discovered that some grains were oriented with the $\{110\}$ axis in the direction of the incident ion beam; the orientation of the other grains could not be determined uniquely on account of the small number of diffraction spots. However the diffraction pattern does not contradict orientations of these grains with the $\{110\}$ or $\{100\}$ axis in the direction of incidence of the ion beam. Thus the assumption mentioned is qualitatively supported by experimental results.

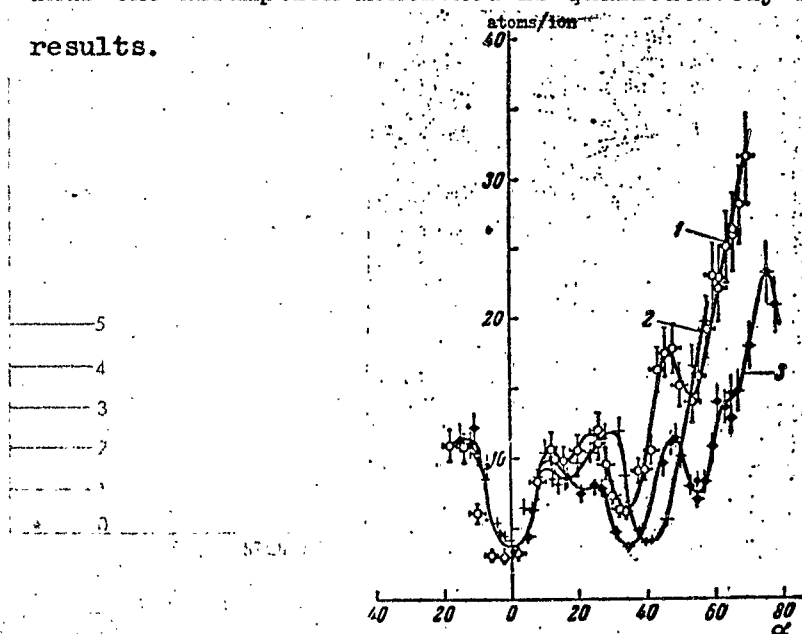


Fig. 2.

6. Angular Distribution of Sputtered Particles

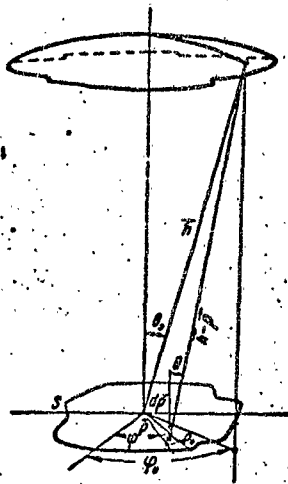


Fig. 3.

The occurrence of directional exit of the particles during the irradiation of a single crystal by an ion beam is a convincing argument in favor of the "chain" mechanism of cathode sputtering [19, 20]. An experimental study of the appropriate angular features is of considerable interest at the present time. Usually [7, 21] the angular distribution is judged on the basis of the distribution of target material deposited on the collector. Let S (Fig. 3) be the irradiated surface; $h(h, \theta_0, \varphi_0)$, the vector drawn from the center of the target to some point on the collector; $\vec{p}(s)$, the radius vector of the surface element dS ; $j(\vec{p})$, the ion current density; and Φ , the function for the angular distribution of the sputtered particles. Then the amount of substance deposited at some point on the collector is proportional to the quantity

$$c(h) = \int_S \frac{\Phi \left(\frac{h - \vec{p}}{|h - \vec{p}|} \right) \cdot j(\vec{p}) \cdot \left(dS, \frac{h - \vec{p}}{|h - \vec{p}|} \right)}{|h - \vec{p}|^2}$$

(cf. Chandrasekar's article [22]). If Φ and j are known, calculation

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of the density of the deposit on the collector presents no difficulties. However, in the experiment a quantity proportional to σ is measured, and Φ must be found with respect to it. When the geometry of the experiment is arbitrary, there apparently exists only one way of finding Φ - measuring the ion current density on the target, assigning some form for the distribution Φ' , calculating σ' and comparing this quantity with the measured σ . If σ' coincides with σ then we must investigate the uniqueness of the obtained solution, or, in other words, we must investigate how sensitive the distribution σ' is to the shape of the curve with the aid of which we approximate the sought-after distribution of the sputtered particles. If this investigation is not made, we can verify only that the deposit distribution obtained on the collector does not contradict the law for the angular distribution of the sputtered particles Φ' . A geometry was used in the experiment which permitted considerably more reliable information to be obtained about the sought-after angular distribution. It follows from the expression for σ that when $|\vec{\rho}| \ll |h|$ it converts to

$$\sigma(h) = \frac{\Phi\left(\frac{h}{h}\right)}{|h|^2} \cdot \frac{h}{|h|} \int_S j(\vec{\rho}) \vec{dS}$$

or, in coordinates,

$$\sigma(h, \vartheta_0, \varphi_0) = \frac{\cos \vartheta_0}{|h|^2} \Phi(\vartheta_0, \varphi_0) \int_S j(\rho, \varphi) \rho d\rho d\varphi.$$

When a spherical collector with center at the middle of the irradiated sample is used, the density distribution of the deposit of the collector will be proportional to the function for the angular distribution of the sputtered particles. The experimental setup is shown in Fig. 1 of a previous paper [23]. The crystal is fastened with a special holder which is kept at the desired temperature. The collector

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is either an X-ray film backing fastened to a cylindrical screen, or a glass plate. The use of cylindrical and plane screens instead of spherical lessened the accuracy of the measurements somewhat, but it was vindicated from the point of view of convenience in performing the experiment and analyzing the results. Measurements reported in the literature [18, 23] have shown that the deposit density distribution in spots close to the $\{110\}$ axis are described very well by a Gaussian curve with a half-width of 20° ; the distribution of the deposit in the central $\{100\}$ spot cannot be described by one Gaussian curve (close to the center of the spot the dependence of the deposit density on the escape angle of the particles is considerably stronger than it is far from the axis). Apparently, this is due to the fact that another smoother distribution (background) is superimposed on the deposit distribution in the central spot. Irradiation of single crystals by ions of different masses and energies has shown that the distribution of particles close to the axes does not depend significantly on the mass and energy of the ions. In Fig. 4 a photomicrograph (2) of the deposit in the $\{110\}$ spot is presented. Also given there is curve (1), which approximates this distribution (with a half-width of 18°).

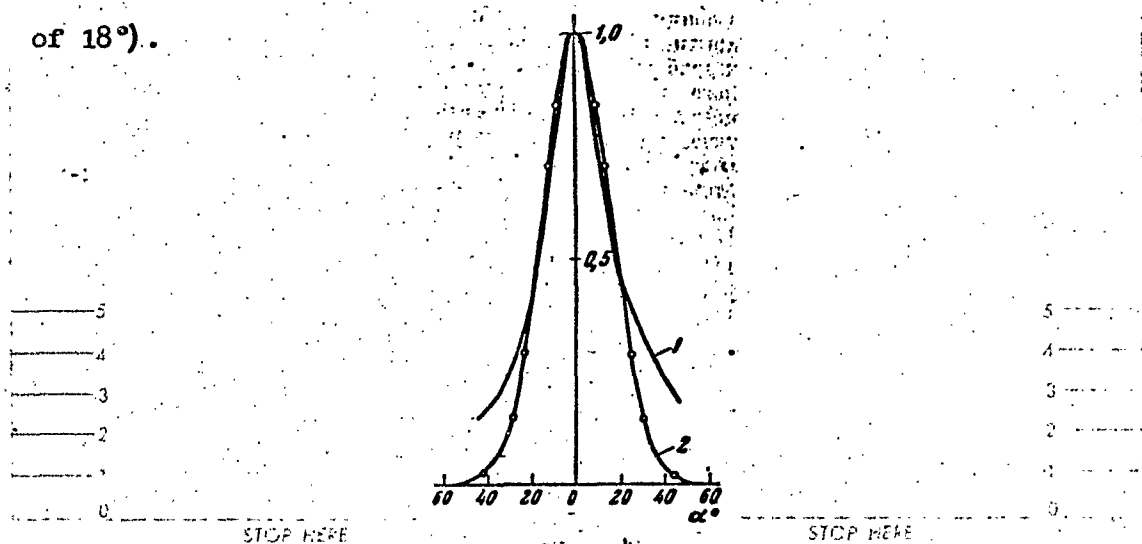
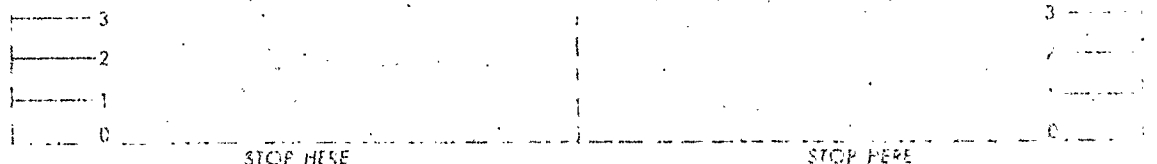


Fig. 4.

The values given for the half-widths are probably somewhat overstated. First, we did not allow for the finite nature of the dimensions of the irradiated surface (the probable overstatement of the half-width is 10%). Also the half-width may have become overstated by our neglecting to allow for the background; we were unable to estimate the size of this error, but apparently it is not especially large. There exists yet another reason because of which the measured distributions may have proved to be widened — the breakdown in crystal structure due to the irradiation itself. In Ogilvie's paper [24] it was shown that these breakdowns prove to be considerable under some conditions. The dependence of the width of the angular distribution of the sputtered particles on the temperature of the target was measured. It is to be expected that if radiation breakdowns in the structures of a single crystal are significant, then when the temperature of a target of a number of interstitial ions is increased the width of the Wenner spots will decrease. Previous measurements [25] have shown that for irradiation doses of 2-3 ma·hr/cm² the radiation breakdowns in the structure of a single crystal do not lead to a noticeable widening of the angular distribution of the sputtered particles, provided the temperature of the target is not made so low that the exit of the interstitial ions is impeded. Thus the half-width of the angular distribution is not more than 20° for particles sputtered in the {110} direction of copper and nickel single crystals, i.e., of the same order as the angular width of the "dips" in the curve of the dependence of the sputtering coefficient on the angle of incidence of the ion beam onto the target (see Fig. 2).



7. Angular Features of Sputtering in Single Crystals and Radiation Theories

In recent years important progress has been made in our comprehension of the mechanism of the disintegration of metals by heavy particles and particularly in understanding the radiation mechanism of the disintegration of single crystals. The discovery of the collision-focusing effect [26-28] and its application to the problem of sputtering in single crystals [19, 20] has made the mechanism of cathode sputtering qualitatively understandable, though a quantitative theory permitting calculation of the sputtering coefficients, and the angular and energy distributions of the sputtered particles has not yet been founded. Apparently we should consider as established the fact that displaced lattice atoms the energy of which is below the focusing limit of the corresponding atomic chains play no appreciable role in the generation of sputtered atoms. The results presented above regarding the independence of the angular distribution of the sputtered particles on the energy and mass of the ions serves as an indirect corroboration of this assertion. In fact, according to Vineyard [28], the role of an incident ion reduces merely to creating a certain number of displaced atoms and vacancies; all other processes including the exit of particles, from a single crystal are the result of their motions within the lattices.

After the appearance of Nelson and Thompson's paper [20], it is hardly possible to have any doubt as to the validity of the radiation mechanism of crystal disintegration. However, at present no theory exists which permits of even a qualitative explanation of the strong anisotropy of the sputtering coefficient noticed in single crystals.

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