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ATOMIC STRUCTURE OF W MICROCRYSTALS WITH DIMENSIONS DOWN TO 60 Å

With the advent of the ion projector (^1), broad prospects opened up for studying the real structure of crystals of metals and semiconductors while visually observing surface atoms. It became possible to compare the directly observed structure with those ideas about it that had been developed on the basis of data from X-ray structural, electron-diffraction, and electron-microscopic analyses.

Unfortunately, the low brightness of the image on the screen of the ion projector and the difficulty of using electron-optical converters at present greatly limit the potential possibilities of this new and promising technique. This limitation proves especially severe in the case of the most interesting objects—microcrystals containing only a small number of atoms, with dimensions from several hundred down to several tens of angstroms. It is precisely on such crystallites, free of the usual macroscopic defects, that it is important to study growth and destruction, the formation of various microdefects (vacancies, elementary dislocations, etc.), new phases, adsorption processes, and so on.

According to (^2), the brightness of the image on the screen L is proportional to

$$L \sim CpV^3, \tag{1}$$

where p is the pressure of the "imaging" gas, and V is the voltage applied to the tip. The voltage for the optimum image is determined by the radius of the tip r approximately according to a linear law; therefore,

$$L \sim C'pr^3. \tag{2}$$

Thus, the image brightness falls very sharply as r decreases. Even when very fast objectives and sensitive photoemulsions are available, the exposures become so long that photography of changing images is completely ruled out. In addition, at radii below 100 Å, the weak brightness also makes visual monitoring of the image difficult.

On the other hand, it is precisely in the case of small radii that it is easier to achieve resolution of individual atoms, since under such conditions the latter distort the potential relief relatively more strongly. Thus, work with microcrystals of the smallest dimensions appears extremely interesting.

Figure 1 shows an image of a W crystal with a radius of ~ 200 Å, obtained in helium ions at a voltage of 9.5 kV. The conditions for accommodation of helium on the surface of the tip were not optimal, since the tip was cooled not with liquid hydrogen but with solid nitrogen. Nevertheless, owing to the small value of r, along the edges of the {112} faces and in the vicinity of the emergence of the [111] direction the atomic structure of the microcrystalline tip proved to be well resolved. The (111) faces look gray, without details, and not black, which indicates that the field strength above these faces is higher than optimal and that the images of individual atoms were blurred, forming a continuous gray background. The picture was extremely dim and required an exposure of 45 min.

In this article we wish to show, using W as an example, the existence of one simple possibility for obtaining relatively intense and sharp images of microcrystals of various metals. The very unfavorable ratio for normal tips (2) can be circumvented if one uses protuberances on tips obtained in the process of vacuum breakdown between the tip and the screen. An analogous method has been successfully used in our laboratory to obtain autoemission electron images of microcrystals of certain low-melting metals (3).

A protuberance on a tip, obtained in the process of vacuum breakdown, has a broad base and a small-radius apex, with a very short length. It is an insignificant local disturbance of the hyperboloidal shape of the tip. The brightness of the image of the atomic structure of the protuberance will be greatly increased, since the voltage for it will be considerably higher than that which would have to be applied if an entire tip with a radius of curvature equal to the radius of curvature of the protuberance were used. The brightness of the image will be further increased because of the greater influx of molecules to the protuberance due to local field gradients. In the process of vacuum breakdown at a positive potential of the tip, the protuberances are probably formed by local extrusion in the near-surface layers of the tip, heated as a result of the development of an avalanche of charge carriers. It should be noted that extrusion can easily lead to destruction of the tip because it can itself initiate vacuum breakdown (4). The experiment must be conducted so as to prevent the development of a powerful avalanche of charge carriers leading to melting of the tip and an increase in its radius. This is most simply done by placing in the high-voltage source circuit an ohmic resistance of about 10^9 ohms.

The photograph in Fig. 2 shows an image of a W microcrystal in helium ions. From the clearly observed atomic structure of the crystal it is not difficult to conclude that this crystal is of extremely small dimensions (about 60 Å in diameter). Nevertheless, its image, quite bright for an ion projector, was observed at 37 kV and was photographed at

Fig. 3. Model of the most regular part of the microcrystal of Fig. 2. The shaded spheres correspond to atoms observed in the ion image.

Figure 1: Fig. 3. Model of the most regular part of the microcrystal of Fig. 2. The shaded spheres correspond to atoms observed in the ion image.

Figure 1

Figure 2: Figure 1

Fig. 3. Model of the most regular part of the microcrystal of Fig. 2. The shaded spheres correspond to atoms observed in the ion image.

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Fig. 1. Helium ion image of a tungsten crystal with a radius of about 200 Å. Voltage $V = 9.5$ kV, temperature $T < 63°$K (solid nitrogen under pumping), helium pressure $p = 1 \cdot 10^{-3}$ mm, exposure $\tau = 45$ min.

Fig. 2. Helium ion image of a tungsten microcrystal with a diameter of about 60 Å. $V = 37$ kV, $T = 63°$K, $p = 1.2 \cdot 10^{-3}$ mm, $\tau = 1.5$ min.

exposure time of only 1.5 min. This microcrystal is a small "hillock" on a protrusion formed after a breakdown of the type described in ([4]). The hillock itself arose as a result of extrusion at the stage when the breakdown was dying out, when the surface had not yet had time to cool and the field began to increase. During observation the hillock evaporated intensely under the electric field. Since for pure W the difference between the imaging and evaporating field strengths is tens of MV/cm, the evaporation should be explained by the presence of impurities (the original tip was only weakly heated). Probably, because of the inhomogeneity of the impurity distribution, the rate of field evaporation changed. Photography was carried out at the moment when it was small.

On the microcrystal in Fig. 2 the faces (110), (011), (211), (121) are clearly manifested. The last face consists of only three rows of atoms. The region of the (111) face consists of plane layers (nets) of atoms, the uppermost layer being represented by only three atoms, i.e., by the minimum number sufficient for the formation of a face. In Fig. 2 the distances 4.46 Å are resolved with exceptional clarity. This is the distance between atoms on the plane nets {111} and the distance between rows on (121). The small size of the crystal made it possible to build an exact model of its regular part from steel balls, shown in Fig. 3. On the model all the faces listed above, visible in Fig. 2, and the region of the (111) face are clearly seen. The images of the balls corresponding to the most protruding atoms in this region and revealed in the ion pattern of Fig. 2 are

Figure 2

Figure 3: Figure 2

shaded; the numbers on the balls correspond to the number of the plane net of type (111), starting from the top. In comparing Figs. 2 and 3 it is interesting to note that not all atoms occupying equivalent crystallographic positions are imaged with equal sharpness. Apparently the surface of a real crystal, even one devoid of noticeable defects, may differ from the ideal crystal to which the model assembled from balls corresponds. Undoubtedly, the sharpness of the image of individual atoms depends on the micrograd ients of the field, determined not only by the edge atoms but also by their neighbors in the given row.

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Note: Figure translations are in progress. See original paper for figures.