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# ORIENTATION DETERMINATIONS OF CRYSTALS USING EJECTION PATTERNS RESULTING FROM ION BOMBARDMENT

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# Orientation Determinations of Crystals Using Ejection Patterns Resulting from Ion Bombardment*

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(Received 3 February 1964)

Data on six common metals are presented to indicate the general usefulness of ion bombardment for orientation determinations of fcc and bcc crystals. The accuracy is 3° or better and is attained by the use of hemispherical collectors and a simple optical projection device. No film processing is involved and a complete determination, including all operations, usually takes less than 20 min.

## INTRODUCTION

RESEARCH, to an ever increasing extent, is concerned with investigations of directional lattice properties and properties of specific surfaces so that single crystals of known orientation are frequently required. The orientation is usually determined by Laue x-ray patterns or by an x-ray spectrometer technique. This paper presents another technique applicable, at least, to face-centered and body-centered cubic structures, and fragmentary data indicate that it may be useful for other structures as well. The technique is based on the directional ejection of atoms from crystals while being subjected to ion bombardment under vacuum. Wehner,[1] who discovered the phenomenon, noted that the ejection was largely restricted to a few principal crystallographic directions, and suggested that it might be useful in roughly determining crystal orientation.

Nelson,[2] using 2-keV argon ions in transmission at normal incidence and flat collectors, determined the preferred orientation of thin foils of five fcc metals prepared both by evaporation on salt substrates and by rolling and annealing.

In an investigation in which aluminum (fcc) crystals were bombarded with 8-keV argon ions along chosen lattice directions and at various angles to surfaces prepared close to (100), (110), and (111), Cunningham, Gow, and Ng-Yelim,[3] using hemispherical collectors and an optical device, demonstrated a technique which showed that the predominant ejections invariably centered about the [110] directions with an accuracy sufficiently high to make orientation determinations practical.

Since that time an investigation of the method *per se* has been undertaken. This paper, based on data from several fcc and bcc metals, demonstrates the usefulness of this technique for the orientation determination of single crystals and crystals in a coarse-grained sample by a microtechnique.

## EXPERIMENTAL TECHNIQUE AND RESULTS

Samples should preferably be electropolished to remove the cold worked layer. The oxide layer which forms on some metals, such as aluminum, may be removed in the early stages of bombardment if mass-spectrometer grade argon is used and if the apparatus is sufficiently tight to prevent oxygen contamination, e.g., $\approx 10^{-5}$ Torr.

To obtain useful patterns, operating conditions must be chosen so as to prevent buildup of severe damage in the surface layers. This may be accomplished by bombarding with ions of sufficiently low energy for the metal concerned or by annealing out the damage during bombardment.[4] However, published data indicate that many metals have given satisfactory patterns when bombarded over a wide range of ion energies; e.g.: Au at 0.1–0.8 keV,[5] 2 keV,[2] 40 keV,[6] and 43.5 keV[7]; Al at 8 keV[3] and 50 keV[8]; and Ag at 0.15 keV,[1] 2 keV,[2] and 8 keV by ourselves. Keeping in mind the necessity for rapid orientation determinations for a large number of common metals, the authors chose a 100-$\mu$A beam of 8-keV argon ions. In the interests of sharp patterns the beam was confined to about 2 $\text{mm}^2$, giving a current density of about 5 $\text{mA/cm}^2$. Under these conditions satisfactory patterns were obtained from single crystals of silver in 15 sec, copper in 2 min, aluminum, tungsten, iron, and beta-brass in 5 min or less. No patterns were obtained from the reactive metals tantalum, niobium, and vanadium. Anderson,[9] however, reported patterns from these metals using energies below 0.8 keV. The authors were limited to experiments in the range 4 to 12 keV by an ion source of the glow discharge type.

Figure 1 is an assemblage of ejection data from the fcc metals silver, copper, and aluminum, and from the bcc metals iron, tungsten and beta-brass, each plotted on standard (100), (110), and (111) projections. The fcc metals gave identical results and hence the data from each of these metals are plotted without distinction in each of the top row projections. The bcc metals gave

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[1] G. K. Wehner, J. Appl. Phys. 26, 1056 (1955).

[2] R. S. Nelson, Brit. J. Appl. Phys. 11, 475 (1960).

[3] R. L. Cunningham, K. V. Gow, and Joyce Ng-Yelim, J. Appl. Phys. 34, 984 (1963).

[4] G. S. Anderson, G. K. Wehner, and H. J. Olin, J. Appl. Phys. 34, 3492 (1963).

[5] G. S. Anderson, J. Appl. Phys. 33, 2017 (1962).

[6] M. W. Thompson, AERE Report M-1262.

[7] R. S. Nelson, Phil. Mag. 7 (8th ser), 515 (1962).

[8] R. S. Nelson and M. W. Thompson, Phil. Mag. 7 (8th ser), 1425 (1962).

[9] G. S. Anderson, J. Appl. Phys. 34, 659 (1963).

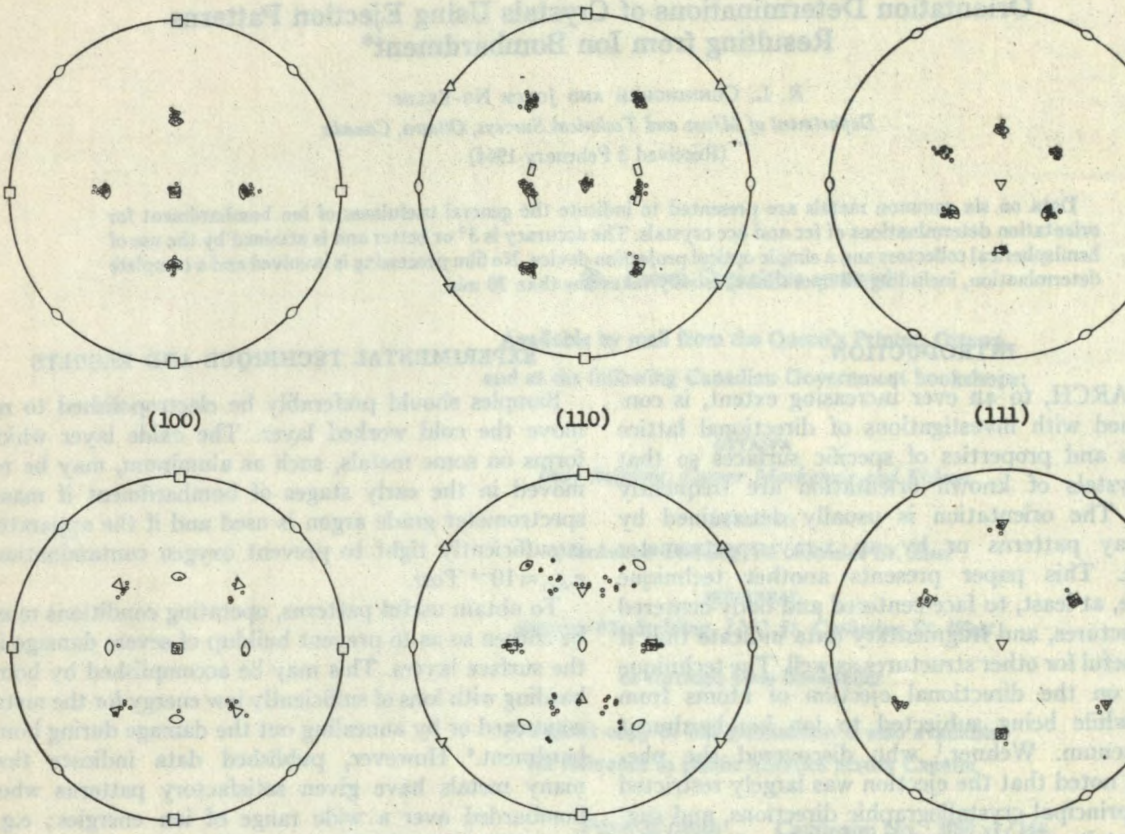

FIG. 1. *Top row*, each stereographic plot is a compilation of ejection data obtained from fcc metals silver, copper, and aluminum. *Bottom row*, each stereographic plot is a compilation of ejection data obtained from bcc metals iron, beta-brass, and tungsten. (□, the oval, △, and the rectangle refer to [100], [110], [111], and [116], respectively).

identical results for [111] and [100] ejection for crystals having surfaces close to both (100) and (111). However, considerable scatter occurred in the region about [111] for crystals with surfaces close to (110) as seen in Fig. 1, bottom row, center. The position of these [111] satellites seemed to be characteristic of each of the three metals. Iron showed the greatest divergence and its [111] satellites are indicated by black dots. These [111] satellites are not used for orientation determinations and will be discussed more fully below.

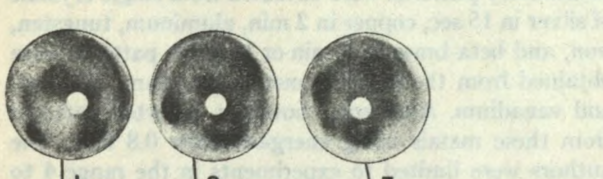

FIG. 2. Polycrystalline silver sample with oriented patterns from the grains indicated. Input beam enters through hole at top of each hemisphere.

## APPLICATION OF THE TECHNIQUE TO A POLYCRYSTALLINE SAMPLE

A coarse-grained silver sample is shown in Fig. 2, along with the oriented patterns from the grains indicated. Since the patterns are on hemispherical collectors, they necessarily suffer from distortion in presentation on paper and also show loss of contrast due to a difficult lighting problem.

The resolution depends on the divergence of the beam. If a grain is smaller than the cross section of the beam the surrounding grains may be masked with a thin polycrystalline foil.

## EJECTION PATTERNS

### Face-Centered Cubic

The directions of most intense ejection are [110]. These are the directions of closest packing in fcc crystals and this, together with the fact that equivalent directions are stronger ejectors when in a forward position[1,3] relative to the direction of impact of the incident beam, strongly suggests a momentum transfer process. Silsbee[10] suggested a mechanism based on a sequence of collisions in a row of atoms under certain geometrical conditions, by which a large proportion of the momentum available would be accurately focused into these closest packed directions. The [100] directions also eject, relatively weakly, and Nelson and Thompson[11] have proposed a focusing mechanism which takes into account the electrostatic focusing effect from rings of neighboring atoms. The only other ejections detected, [116], are close to [114][3,5,11,12] and this has been explained by Koedam[11] and Anderson[5] as being due to atoms sitting in twin positions on (111) surfaces either originally present or formed during bombardment. However, even with a prepared (111) surface where (111) planes must constitute most of the area, the intensity of the [116] ejection is always considerably less than that of the [110]. Hence, in the case of fcc metals, [110] ejection is the most reliable for purposes of orientation determinations.

### Body-Centered Cubic

The directions that have been recorded are [100], [111], [111] satellites, and [110]. The [111] direction being the most closely packed is, no doubt, the direction along which momentum is most efficiently transferred within the crystal. The [111] satellite spots refer to those ejections recorded by Anderson,[9] Nelson[13] and ourselves in the neighborhood of [111]. These may be attributed[9,13] to last-impact deflections from [111] due to atoms in non-normal positions on surfaces. Nelson[13] postulates relaxation around vacancies created in the surface by ejection, while Anderson[9] postulates that these atoms in non-normal positions are found on (110) surfaces as prepared, or as developed during the bombardment. As evidence, in the case of (100) surfaces where [111] ejection normally occurs, [111] satellites have been recorded for molybdenum,[9] but only after the sample became deeply etched through bombardment, presumably by the formation of (110) microsurfaces. Indeed in the extreme case of a prepared (110) surface, Anderson reports that these satellites can be recorded without evidence of [111] ejection. Hence, the [111] ejection, being very dependent on the (110) content of the surface, may well be second in intensity to [100] or even absent. From the small number of data available, it appears that there is a considerable variation in angular deviation and direction of the satellites from [111]. Thus they cannot be used with confidence for orientation determinations. On the other hand, published data and our own indicate that [100] ejection is always present from (100),[9] (110),[9,13] and (111)[9,13] surfaces. Hence, for bcc metals, [100] ejection is the most reliable for orientation determinations.

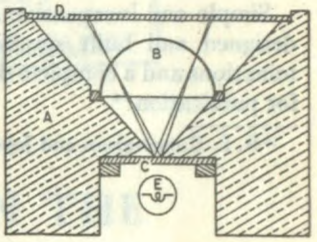

FIG. 3. Optical device for producing stereographic projections directly from hemispheres. A—cylindrical body; B—punctured hemisphere; C and D—ground glass disks; E—light source.

### ACCURACY OF THE METHOD

The accuracy obtained is largely due to the use of hemispherical collectors with the bombarded surface at the center of curvature. This permits the accurate estimation of the center of gravity of the ejection spots. The collectors may be conveniently cut from ping-pong balls or blown from glass. Ping-pong balls have the advantage that they may be pierced with a needle at the center of each ejection spot before being placed in the simple optical device shown in Fig. 3 for the direct production of stereographic projections. By means of a Wulff net all the plotted directions are shifted until the best fit with a standard (100), (110), or (111) stereographic projection is obtained and the orientation may then be described in relation to one of these.

The accuracy of the method was estimated by comparison with x-ray determinations. The values are shown below in Table I.

A comparison of the values in Table I indicates good agreement with x-ray determinations. Since a number of ejection spots are involved in a single determination, any errors in judging the centers of gravity of the spots tend to cancel out. Reproducibility is 2° or better.

TABLE I. Comparsion of x-ray and ion bombardment data for crystal orientation determinations.

| Sample | Surface normal (by x-rays) | Surface normal (by ion bombardment) |
|---|---|---|
| Silver #1[a] | 22° to [111] | 22° to [111] |
| Silver #2 | 18° to [100] | 20° to [100] |
| Silver #3 | 12° to [111] | 13° to [111] |
| Silver #4 | 10° to [111] | 12° to [111] |
| Silver #5 | 8° to [110] | 4° to [110] |
| Silver #6 | 2° to [100] | 3° to [100] |
| Silver #7 | 7° to [110] | 4° to [110] |
| Aluminum | 3° to [100] | 4° to [100] |
| Iron | 8° to [100] | 10° to [100] |
| Tungsten | 3° to [100] | 3° to [100] |
| Tungsten | 2° to [111] | 2° to [111] |

[a] Samples #1 to 7 refer to numbered grains in the polycrystalline silver sample shown in Fig. 2.

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[12] M. Koedam, Philips Res. Repts. 16, 266 (1961).

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Simple and inexpensive apparatus has recently been designed and built specifically for orientation determinations and a complete description is being submitted for publication.[14]

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[14] R. L. Cunningham and Joyce Ng-Yelim (to be published).