A Study of Cones Developed by Ion-Bombardment of Gold

I. H. WILSON*, M. W. KIDD
School of Mathematical and Physical Sciences, The University of Sussex, Brighton, UK

Argon and xenon bombardment of gold surfaces at 5 to 20 keV resulted in considerable erosion and the formation of cones. A high cone density was found to be characteristic of amorphous (mechanically polished) surfaces whilst a low cone density with height contrast of the individual grains was discovered on polycrystalline (etched) surfaces. The cone angles were found to vary only slowly with energy, indicating an ion/atom potential that varied much more rapidly with separation than predicted by using a screened coulomb potential.

The cones were found to have hexagonal facets and in the case of a polycrystalline specimen, two families of cones were found. These phenomena are attributed to single crystal effects. Similar bombardment of single crystal specimens resulted in a much lower sputtering rate and no cones were generated, probably as a result of channelling of the incident ion beam.

1. Introduction

Microscopic study of surface features produced by ion-bombardment of metals has revealed interesting features in the form of hillocks, grooves and cones [1, 2]. This has been explained [2] in terms of the variation of sputtering rate with angle of ion incidence discovered by several workers [3, 4].

In this work the cones formed by ion-bombardment of gold surfaces were studied to see if the variation of cone-angle with bombarding ion energy could be determined, as a test of the theory of Stewart and Thompson [2], and to see what could be determined concerning the sputtering mechanism from an examination of the surface damage.

2. Apparatus

All specimens were 4N purity gold supplied with a spark-machined finish. These were mechanically polished, finishing with 1 μm alumina powder (12 h vibratory polish). Some samples were etched electrolytically in concentrated hydrochloric acid.

Specimens were placed at the primary focus of an accelerator capable of producing ions at 2.5 to 25 keV of any species with a beam current of up to 200 μA mm^{-2} (for argon at 20 keV). Argon and xenon ions were used for this experiment.

The specimens were mounted in a goniometer aligned with the surface normal to the beam, and masked by an earthed tantalum sheet with an aperture 1 mm in diameter. Current to the specimen was monitored by a Keithley electrometer. Temperature was checked by means of a thermocouple in the molybdenum mount and never rose above 50°C during a run. A cold finger was placed so that the specimen could be cooled to liquid nitrogen temperatures by attaching a strap of copper braid. The background pressure in the system was in the region of 4×10^{-5} torr with the ion source running.

Surface damage was examined using a Cambridge Instruments scanning electron microscope. Specimens were mounted on a stud with a plane surface at 40° to the axis to enable tilting towards and away from the collector for determination of cone angles. This was done by arranging the tilt axis to lie in the surface of the specimen. The cone angle could then be determined by measuring the angle of rotation necessary to move the apex along a diameter from apparent coincidence with one side of the base to the other. This method was found to be

*Now Department of Electronics, University of Surrey, Guildford.

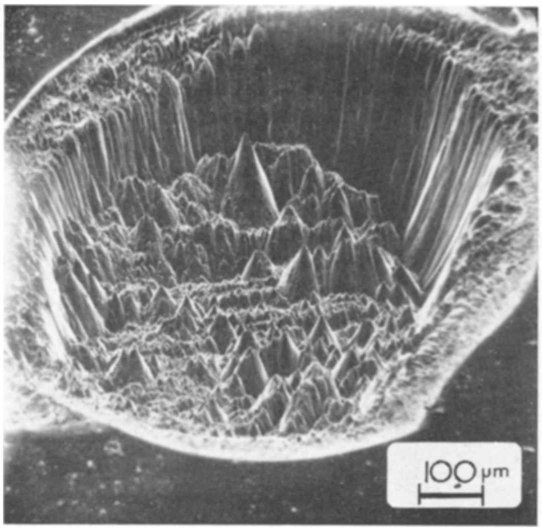


Figure 1 10 keV argon bombardment of mechanically polished polycrystalline gold (dose 6 × 10^{17} ions mm^{-2}).

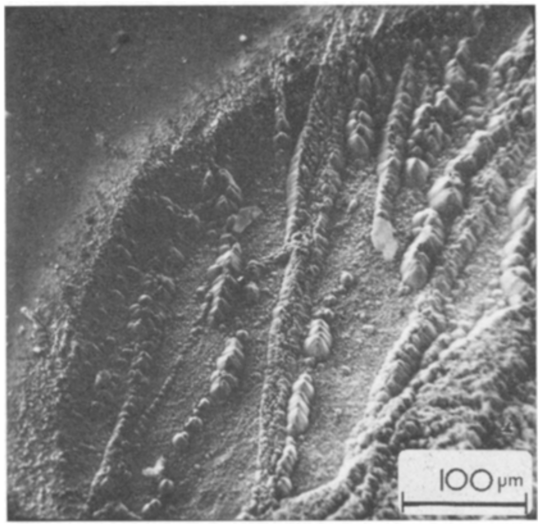


Figure 3 20 keV argon bombardment of mechanically polished polycrystalline gold (dose 5 × 10^{17} ions mm^{-2}).

insensitive to errors in alignment and a cone angle could be determined to $\pm$ 0.5°.

3. Observations

Samples were bombarded with 5, 10 and 20 keV argon and 20 keV xenon. The result of heavy bombardment (6 × 10^{17} ions mm^{-2}) of an amorphous (mechanically polished polycrystal) surface is illustrated in fig. 1. A deep pit was formed with a jumble of cones at the bottom. The striations on the walls of the pit are similar to features observed by Cunningham *et al* [1] when bombarding at 70° to the surface normal. These can now be seen to be a development of the cones formed by normal bombardment.

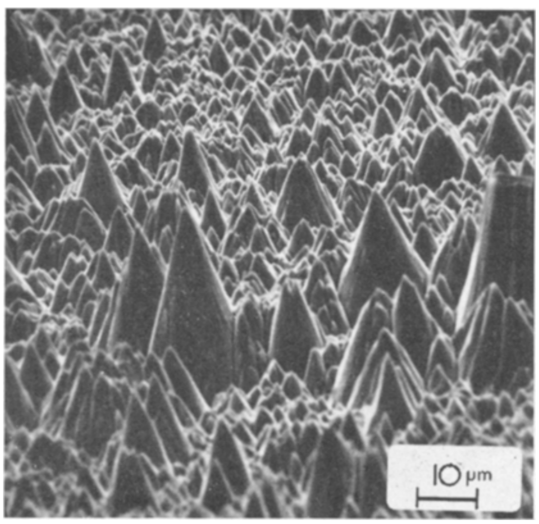
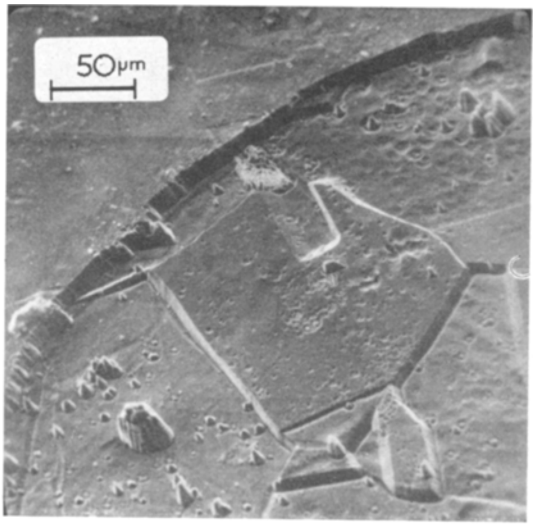


Figure 2 5 keV argon bombardment of mechanically polished polycrystalline gold (dose 7 × 10^{15} ions mm^{-2}).

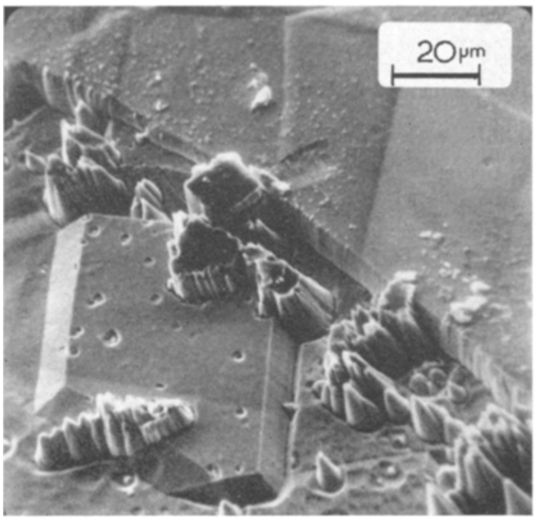
The high density of cones was found to be characteristic of mechanically polished surfaces; a typical example is shown in fig. 2. Possible nuclei for the formation of cones are (*a*) debris on the surface; (*b*) inclusion of grinding compound, (*c*) inclusions at grain boundaries. Occasionally the cones lie in strings with a low density of cones in the intervening spaces. This is shown in fig. 3, and these are possibly generated by large scratches or grain boundaries lying below the region of mechanical damage.

When the surface is polycrystalline (etched) the different sputtering rates of individual grains results in height contrast, as illustrated in fig. 4a. The cones are mainly generated by surface debris, as shown in fig. 4b. Cones tend to be isolated with a groove at the base. The groove is probably caused by high energy sputtered particles enhancing the effective flux at the base of the cone [5]; there were also a large number of empty pits where presumably the cone had sputtered away completely. Grains sputtered away in such a way that a bevelled edge was formed, presumably at an optimum angle for sputtering and evidence of the flux enhancement was seen in the grooves formed at the bottom of the slope.

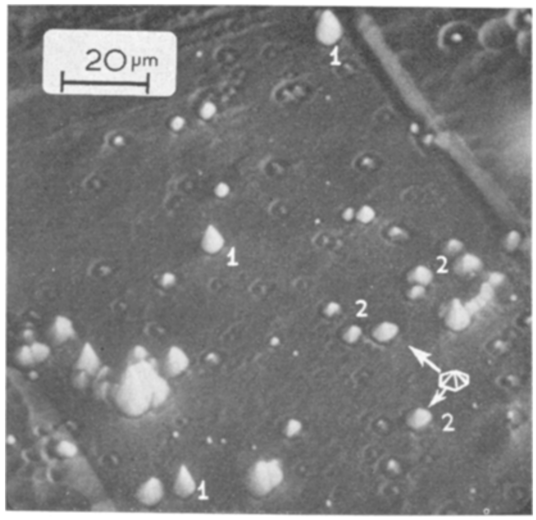
The measured values of the cone angle θ_c and the equivalent angles of incidence of the ion



(a)



(b)



(c)

Figure 4 20 keV xenon bombardment of etched polycrystalline gold (dose 5×10^{15} ions mm^{-2}). (a) General view; (b) illustrating generation of cones at surface debris (c) illustrating the presence of cones of two angles (marked 1 and 2) and the phenomenon of faceting (the orientation of the facets shown diagrammatically).

beam on the cone surface θ_I, (see section 4) are given in table I.

Two families of cones were discovered after 20 keV xenon bombardment of the etched surface, one corresponding to the only type observed on a mechanically polished surface subject to 20 keV xenon bombardment.

Careful study revealed that those cones which could be studied in isolation had facets with hexagonal symmetry. The faceting of the cones and the two populations of cones described above are shown in fig. 4c. Cooling to liquid nitrogen temperatures during bombardment had no effect on the formation of cones and faceting still occurred. The effect of faceting on the cone angle was too small to be measured.

Gold single crystals with (100), (110) and (211) surfaces were bombarded with 10 keV argon. Surfaces were either spark-eroded, mechanically polished or etched. In all cases

TABLE I Cone angles measured for polycrystalline gold

Bombarding Ion		Surface Finish	Cone angle (θ_c)	θ_I $\left(= \frac{\pi - \theta_c}{2}\right)$
Type	Energy (keV)			
Ar	5	Amorphous (Mechanical Polish)	$36.5° \pm 0.5°$	71.8°
Ar	10	,,	$33.0° \pm 0.5°$	73.5°
Ar	20	,,	$27.5° \pm 0.3°$	76.3°
Xe	20	,,	$59.0° \pm 0.3°$	60.5°
Xe	20	Polycrystalline (Etched)	$\begin{cases} 59° \pm 1° \\ 40° \pm 1° \end{cases}$	60.5° 70°

there was a very low sputtering rate resulting in little erosion and no cones were formed. The main surface features observable were a series of ridges and pits (possibly faceted), the ridges seeming to be correlated with spark-machining marks.

4. Discussion

The sputtering ratio $S(\theta)$ for angles of incidence near the normal can be shown to be proportional to $\sec\theta$ for polycrystalline materials. For $\theta > 50°$, $S(\theta)$ rises less sharply, going through a maximum $\hat{\theta}$ and falling to 0 at $\theta = 90°$ [2–7].

It has been postulated [2] that the cones are formed in such a way that the angle θ_I between the ion beam and the normal to the cone surface, is $\hat{\theta}$, thus achieving the maximum sputtering rate. Therefore the cone angle $\theta_c = \pi - 2\hat{\theta}$. At angles above $\hat{\theta}$ the ion cannot penetrate the potential barrier of the surface atoms. If a screened coulomb potential is used it can be predicted [2, 8] that:

$$\theta_c \propto E^{-\frac{1}{4}}[Z_1Z_2/(Z_1^{2/3} + Z_2^{2/3})^{\frac{1}{2}}]^{\frac{1}{4}}$$

where E is the energy and Z_1 is the atomic number of the bombarding ion, Z_2 is the atomic number of the target atom.

Using the results in table I, the cone angle $\theta_c \propto E^{-0.15}$ between 5 and 10 keV and $\theta_c \propto E^{-0.25}$ between 10 and 20 keV for argon bombardment.

If we assume that $\theta_I = \hat{\theta}$, this low dependence of cone angle on energy would indicate that the ion/atom potential varies much more rapidly with separation than the screened coulomb potential used in deriving the equation for θ_c. This is in agreement with the results of Cheney and Pitkin [3] who measured $S(\theta)$ v. θ for xenon on polycrystalline copper. Although $\hat{\theta}$ cannot be determined accurately from their results, there is only a variation of $\sim 5°$ in going from 1.5 to 30 keV.

Using the above equation and the results for 20 keV argon bombardment, the cone angle predicted for xenon bombardment at the same energy is 45°. As this falls between the angles measured on the etched specimen (40 and 59°) the mass dependence predicted by the equation appears to be qualitatively correct.

The cones formed on mechanically polished specimens were observed to grow with bombarding dose. This can only be allowed for in the theory of Stewart and Thompson [2] if $S(0) > S(\theta_I)$, which would contradict their model of cone formation. The situation is obviously more complicated than visualised at first. Work on computer simulation of sputtered surfaces [6] predicts that cones are not formed where $\theta = \hat{\theta}$ but where $S(\theta) = S(0)$, i.e. on the rapidly falling part of the $S(\theta)$ v. θ curve where $\theta \sim 85°$. While this could explain the observed growth of cones, our measured range of values of θ_I appears to agree with the expected value of $\hat{\theta}$. Any detailed interpretation would depend sensitively on the variation of $S(\theta)$ with θ and energy. There is at present a sad lack of information of this type.

The presence of two types of cone on the etched specimen is possibly due to single-crystal effects such as channelling [6–11]. As a result, $S(\theta)$ would not vary smoothly with θ but would exhibit more than one peak. Only the 59° cones are present on the mechanically polished specimen subject to the same type of bombardment but with 100 times the dose. The 40° cones, although nearer to the form predicted from the argon results, are only observable on lightly sputtered surfaces with low cone density. This would imply that the 40° cones shrink during bombardment whilst the 59° cones grow.

The hexagonal faceting of the cones could be attributed to equilibrium shapes being taken up as a result of enhanced surface-diffusion during ion bombardment. The lack of any change in surface morphology on cooling to liquid nitrogen temperatures during bombardment would argue against this. Another possibility is that the facets correspond to low index planes with a high sputtering ratio, giving a crystalline "habit" to the cones thus formed.

It should be noted that the ejection patterns resulting from sputtering of single crystal fcc metals have been explained [12] in terms of surface collision chains intersecting the facets discovered on eroded surfaces by diffraction techniques [13–15]. Although it is visualised that this occurs on a much finer scale than the faceting found on cones in this work, it may be that a careful study of relatively macroscopic faceting could throw light on the formation of ejection patterns.

The low sputtering rate discovered for the single crystals can be explained by the mechanism of channelling. Onderdelinden [10, 16] demonstrates that if the incident beam is aligned within a critical angle on certain low index directions it can be divided into

(*a*) an aligned beam which penetrates deeply into the crystal.

(*b*) a random beam which causes sputtering at a rate proportional to the energy dissipated in the surface.

Thus $S(\theta)$ can be very low in the transparent direction. Cones, if formed at all, would shrink rapidly during bombardment owing to the large difference in sputtering rates between normal incidence and θ_I.

Acknowledgement

This work was the MSc project for M. W. Kidd at the School of Mathematical and Physical Sciences, the University of Sussex. The authors would like to acknowledge the helpful advice of Professor M. W. Thompson and the members of the atomic collisions in solids group.

References

1. R. L. CUNNINGHAM *et al*, *J. Appl. Phys.* **31** (1960) 839.
2. A. D. G. STEWART and M. W. THOMPSON, *J. Mater. Sci.* **4** (1969) 56.
3. K. B. CHENEY and E. T. PITKIN, *J. Appl. Phys.* **36** (1965) 3542.
4. I. N. EVDEKIMOV and V. A. MOLCHANOV, Int. Conf. on atomic collisions and penetration studies, Chalk River (1967) paper G3.
5. A. R. BAYLY and P. D. TOWNSEND, Atomic Collisions in Solids group meeting, Polytechnic, South Bank (2 June 1971).
6. G. K. WEHNER, *J. Appl. Phys.* **30** (1959) 1762.
7. J. FLUIT, Thesis, Leiden University (1962).
8. J. LINDHARD, *Matt. Fys. Medd.* **34** (1965) 14.
9. C. CAVALENI, G. CARTER, and J. S. COLLIGON, Atomic Collisions in Solids group meeting; Polytechnic, South Bank (2 June 1971).
10. D. ONDERDELINDEN, *Canad. J. Phys.* **46** (1967) 739.
11. O. ALMEN and G. BRUCE, *Nucl. Instn. of Meth.* **11** (1961) 257, 279.
12. R. L. CUNNINGHAM and JOYCE NG-YELIM, *J. Appl. Phys.* **40** (1969) 2904.
13. *Idem, ibid* **35** (1964) 2185.
14. P. HAYMANN, *J. Chem. Phys.* **57** (1960) 572.
15. G. K. WEHNER, *Adv. Electron Phys.* **7** (1955) 239.
16. D. ONDERDELINDEN, Thesis, Leiden University (1968).

Received 14 July and accepted 22 July 1971.