A Method for Rapid Crystal-Surface Characterization

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Patterns of reflexion of a visible light beam from chemically etched crystal surfaces were used to identify the crystal orientations at the surfaces. Aluminum and copper specimens with (100), (110) and (111) surface orientations were tested. The main advantage of the method appears to be the simplicity and effectiveness of the experimental procedure. The method makes possible the orientational control of crystal specimens to within less than 1°. Disordering and recrystallization effects induced on crystalline surfaces by ion bombardment were revealed by this method.

Introduction

Many different methods are known for the determination of crystalline orientation characteristics in thin films and surfaces (Chopra, 1969). The method described here was developed in order to obtain a simple testing method for crystal-surface orientation. It also provides evidence of changes on the crystal surface induced by ion bombardment.

Experimental method and results

The method utilizes an intense (Osram halogen lamp, 12V, 55W) narrow beam of visible light, with a circular cross section, that falls onto the crystal surface and is reflected to the screen, film or photographic paper, Fig. 1. When the crystal surface is polished, the pattern obtained on the screen is a circular light spot, the projected image of the light-beam collimator. However, when an etched crystal specimen is used, there appears a quite different pattern, a regular multiangle geometrical pattern which is closely related to the crystal symmetry and the orientation of the macroscopic surface of the crystal specimen investigated. The appearance of these geometrically regular patterns of reflected light can be explained by the fact that under the action of chemical etching there is established a surface relief, consisting of numerous tiny crystal faces arranged in a repetitive way. Reflexion of the light beam from these faces gives a light pattern closely related to their regular and repeating distribution.

The method was checked with large single crystals of Al and Cu of basic low-index orientations (100), (110) and (111). The aluminum crystals were etched for a few seconds at room temperature in a solution of 10 ml HNO₃, 30 ml HCl and 80 ml H₂O. The single-crystal Cu specimens were etched by the same solution, but at 60°C. Different temperatures in the interval 20°C to 100°C and different concentrations of the etching solution – concentrated to dilute – did not affect the results.

Fig. 2 shows the pattern obtained by reflexion from the (100) aluminum surface at normal incidence while Fig. 3(a) represents the microstructure of the same etched specimen. Fig. 2 clearly reveals the fourfold symmetry of the (100) surface of the f.c.c. aluminum. An example of the light reflexion pattern from the etched (100) Cu crystal surface, placed normal to the light beam, is given in Fig. 4. In this case the reflexion pattern has also a fourfold symmetry, but it is not so sharply defined as in the case of Al.

Fig. 5 shows the pattern of light reflected from the (110) Al specimen with the microstructure given in...
Fig. 2(b). It clearly reveals the expected twofold symmetry, while Fig. 6, with threefold symmetry, was produced by reflexion from a (111) Al crystal surface, with the microstructure shown in Fig. 3(c).

Rotating the crystal, so that the light beam falls on the (100) crystal surface at a certain angle different from 90°, shifts the reflected pattern from the centre (Fig. 7). By using coordinates of the cross point in the light figure, and knowing the distance from the crystal to the screen, one can determine the angle
between the \( \langle 100 \rangle \) crystal direction and the direction of the light beam.

It was noticed that on the \( \langle 100 \rangle \) patterns certain parts of the 'star' arms are more intense than others, which indicates that some faces in the specimen surface dominate. In Fig. 8, these more intense reflexions appear at \( 20^\circ \) to the light axis, \textit{i.e.}, the responsible 'dominant' faces are developed at \( 10^\circ \) to the light axis.

This method was used to give evidence of the changes induced by energetic, heavy-ion bombardment of the surface. The Al and Cu crystalline specimens of low-index orientiations were bombarded by 40 keV argon ions, with the dose of about \( 10^{30} \) ions cm\(^{-2}\). Light figures obtained before the crystal bombardment were those shown in Figs. 2,4–8 and the figures obtained after bombardment are illustrated in Fig. 9 [Al \( \langle 100 \rangle \)] and Fig. 10 [Cu \( \langle 100 \rangle \)].

The appearance of many crosses on the Al \( \langle 100 \rangle \) pattern (Fig. 9) indicates that ion bombardment gives rise to some kind of recrystallization, \textit{i.e.}, the formation of several individual grains on the surface situated at different angles relative to each other. Therefore, the reflexion crosses obtained from these different grains appear to be in disorder.

The diffuse, structureless light figure obtained from the bombarded \( \langle 100 \rangle \) Cu specimen (Fig. 10) reveals that the crystal surface has apparently become amorphous, which indicates that the Cu specimen was disordered to a high degree.

Post-bombardment etching of Al and Cu specimens removed the surface deformations. The appearance of reflexion patterns from those specimens was similar to that of the patterns from unbombarded crystals.

Reference