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THERMAL ETCHING FEATURES IN POLYCRYSTALLINE COPPER AND THE HELP OF LIGHT OXIDATION IN THEIR OBSERVATION

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Abstract: *Thermal etching figures in deformed by rolling and then annealed polycrystalline copper are presented and explained. Using light oxidation as a decoration technique, slip bands and twins were observed and crystallographic informations taken out of them are also given.*

1. INTRODUCTION

Evaporation is essentially the reverse of crystal growth. Both procedures produce steped features, in opposite directions, on the surface. These come usualy from imperfections within the crystal, such as dislocations, grain boundaries or twins, and can be revealed by means of etching. There are different methods for etching (chemical, electrolytic or thermal). Etch-pit figures have been widely used to get crystallographic informations as well as to study dislocation densities, the general direction of dislocation lines and motion of dislocations[1,2]. Mecking and Bulian[9] used etch pits to investigate the basic mechanism of strain hardening in copper single crystal. Decoration techniques are sometimes neccessary for making these steps visible. Betghe and co-workers[4,5], using a decoration method based on evaporation of very thin layer of gold onto the surface investigated, showed a dissociation of a $\alpha[100]$ dislocation into two $\alpha/2\langle 110\rangle$ partials.

It was long ago when thermal etching was introduced as a technique for studying growth of crystals. As early as 1912, W Rosehain and D. Ewan[10] noticed the characteristic striations produced by thermal etching of silver. Recently, Doeser et al.[7] studied, by thermal etching, the influence of the structure immediately below the surface on etch figures.

In a previous paper[3] the presence of thermal etch figures in heavily deformed and then annealed polycrystalline copper was noticed. This paper presents the results of the study of this phenomenon. These were observed by optical microscope and some of them were made visible by the help of preferential oxidation.

2. EXPERIMENTAL PROCEDURE

Polycrystalline copper of 99.999% purity was heavily deformed by rolling up to 90% reduction in thickness of the original sheet, then annealed for 48h at 1000° C in a vertical furnace through which high purity argon was flowing, and finally quenched in iced water. With slow quenching rates, a thin layer of cuprous oxide was formed on the surface. Such specimens were mostly used in this work. The specimens were examined under an optical microscope either straight after quenching or after their surfaces have been cleaned by sodium cyanide which disolves the oxide film formed.

3. RESULTS AND DISCUSSION

When metal sections are heated, their initially smooth surfaces become roughened, forming the so-called thermal etching figures. When the normal to the specimen surface is close to a crystallographic direction with small indices, the shape of such figures is symmetrical.

The results of this work have been divided into three parts: General observations, slip bands and twins. Photographs were taken using a Reichert Zetopan metallurgical microscope.

a) General observations.

Figure 1 shows different examples of etch figures. Fig. 1a is a dark field image showing the existence of steps on the surface These appears as white lines in the photograph.

In Fig. 1b there are etch figures in a generally oriented crystal surface. Their shapes are irregular and this can be attributed to the combination of different sources (some are marked by S) from which evaporation of copper atoms preferentially occurs. These sources might be

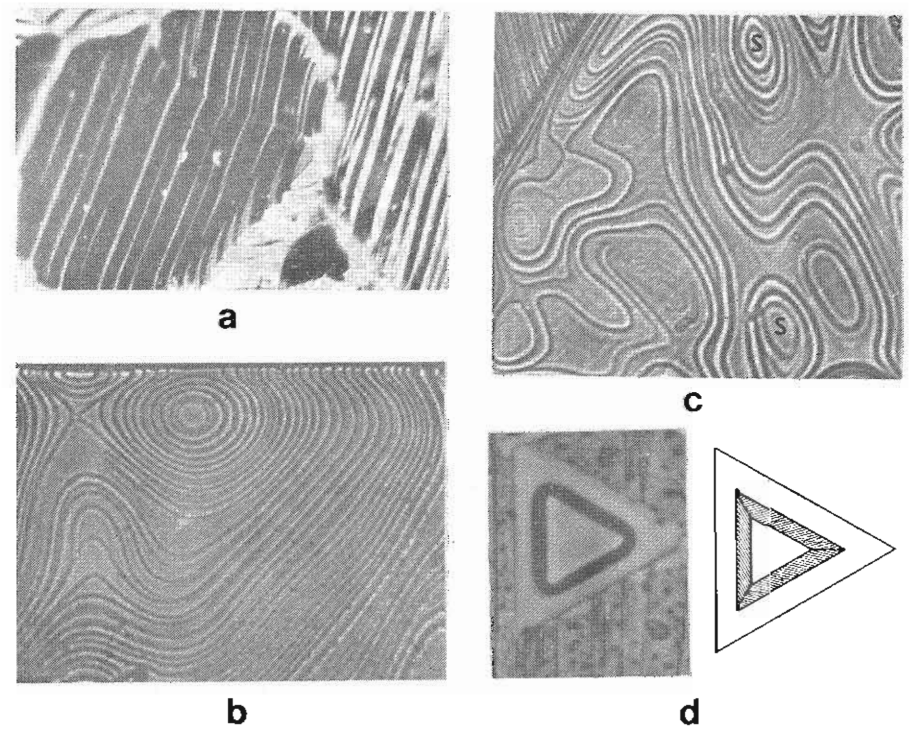

Fig. 1. a) A dark field phatograph showing the existence of steps on the copper surface (X 2600).

b) Thermal etching figures an an arbitrary crystal surface (X 2600).

c) Circular thermal etch figure (X 4300).

d) A crystallographie etch pit (X 3500).

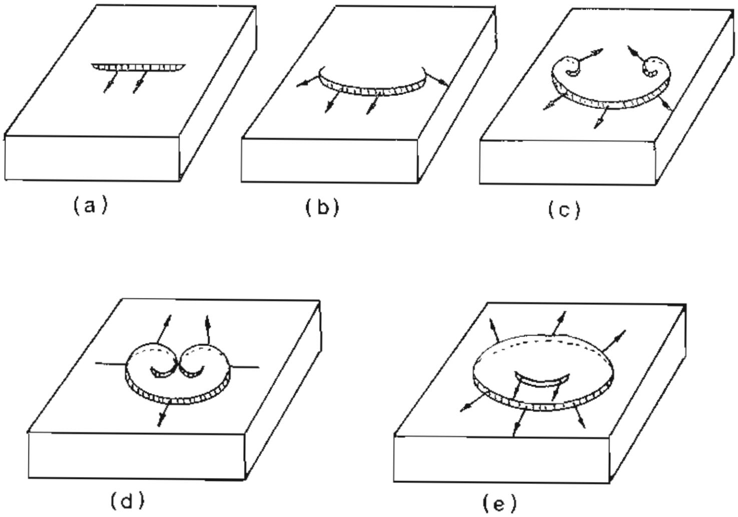

Fig. 2. Successive stages, possibly explaining the formation of the figure in 1c.

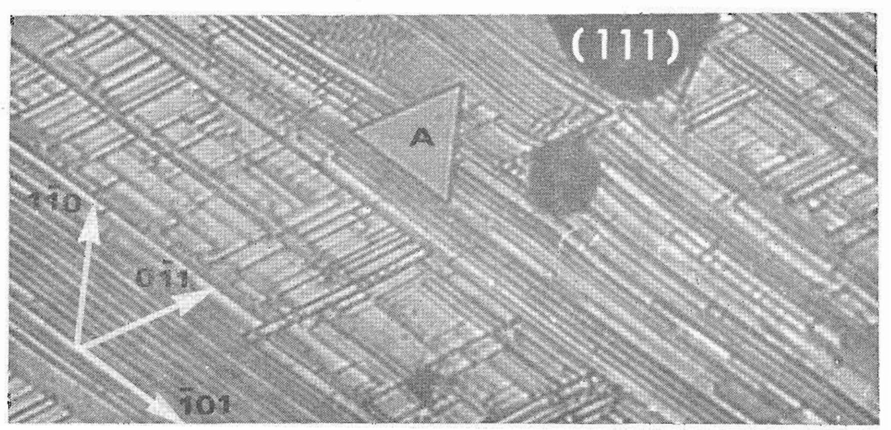

Fig. 3. Slip bands on a (111) surface. Oxide film covers the surface, making the observation easy (X 2300).

connected with emerging points of dislocations from the bulk.

Formation of thermal etch spirals have been previously reported[3]. Fig. 1c shows a spiral-like growth which could be explained as a result of interaction between spirals. This is possible if we think of two dislocations with the same but opposite in sign Burgers vector.

Fig. 2 shows successive stages (in the growth process) of a model for such generation. When the two spirals come close to each other (stage d), a closed front is formed and the two sources start producing a new step (stage e). It is also worth noticing the presence of small oxide particles, mainly sited along the steps produced by thermal etching.

Finally, Fig. 1d shows a «crystallographic» etch-pit and its schematic diagram. It is a regular flat bottomed triangle and it can only be explained if the specimen surface is a {111}. Then, the sides of the triangle point directions of <110> type.

b) Slip bands.

Figure 3 shows surface steps which were attributed to the slip. Fig. 4 presents the successive steps for the nucleation of such steps. As stress continues, more dislocations are formed within the crystal and move to the crystal surface. When they arrive there, they produce a step of height proportional to the number of dislocations which have gone out and not to the total number of dislocations nucleated. The line produced on the surface has now kink sites and it is logical to suppose that evaporation will take place easier from such points of the crystal surface, so these could remain during anealing (and might increase their height by the movement of some of the remaining dislocations)*. If new slip lines are formed within a few hundred Angstroms distance from the original one, we observe clustering of slip lines into bands. Oxide particles will then growth preferentially along these lines, during a slow quenching, making them visible.

It is known that slip planes in f.c.c. materials, like copper, are the {111} planes. Therefore, slip bands belong to such planes. In Fig. 3 we can see two sets of slip-bands appearing on the surface with an angle of 60° between them. The plane which can fulfill all these is a {111},

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* This is not always the case. When a plastically deformed metal is heated to a temperatnre at which self diffusion can occur, new strain-free grains nucleate amongst the deformed grains and grow so as to consume them. This is called recrystallization.

say the (111). Therefore, the indices of the slip lines should be a combination of the $[1\bar{1}0]$, $[\bar{1}01]$, and $[0\bar{1}1]$. This conclusion is supported by the presence of the triangle feature A, which is a rather characteristic pit for octahedral faces, as we pointed previously.

c) Twins.

It is known that a number of metals when deformed undergo sudden localized shear processes called twinning, which involve a small but well-defined volume within the crystal. It was two decades ago when confirmation of twin formation in f.c.c. metals as a result of deformation has been obtained by precision x-ray methods, electron microscopy and electron diffraction. Copper crystals deformed at 4°K were found[6] to form twins. Silver, gold and nickel also twin[8,11].

Crystals M (matrix-crystal) and T (twin-crystal) in Fig. 5a are an observed typical example of twinning. The angle between the slip bands and the intersection of the twin plane with the crystal surface is 52°. Because slip bands should have a <110> direction, the intersection is a <112> direction (angle between the two directions 54.7°). In f.c.c. metals the twin plane is invariably the close packed {111}, which is also the slip plane, while the twinning direction is <112>. If we choose the (111) as the twin plane and the $[11\bar{2}]$ as the twinning direction, then the twinning axis is the [111], the direction of the slip bands are the $[\bar{1}\bar{1}0]$ for the matrix and the [110] for the twin crystal and the crystal surface is the (110). Slip is taken place in $(\bar{1}1\bar{1})$ planes. If the crystal surface is not exactly oriented, the angle of the slip bands can varies to some degrees

Fig. 5b is a micrograph, taken with a JEM 100U electron microscope, showing twins within the bulk material. This comes out from the characteristic fringe pattern. Finally, Fig. 5c shows the increase of the visibility of the features observed by the help of thin oxide layer. The region of the specimen in the photograph has not completely cleaned by sodium cyanide. It is clear that it would be very difficult for twins and slip lines to be observed without the oxide layer.

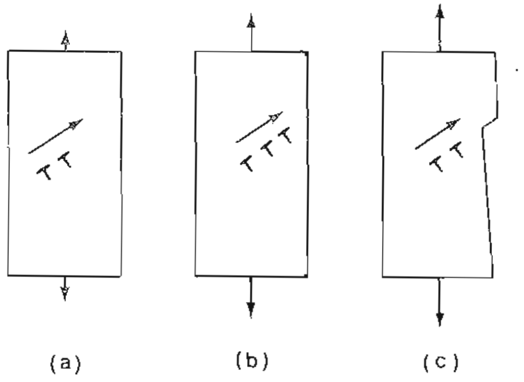

Fig. 4. Successive stages for slip-line formation on a surface by dislocation slip, according to the theory of dislocations.

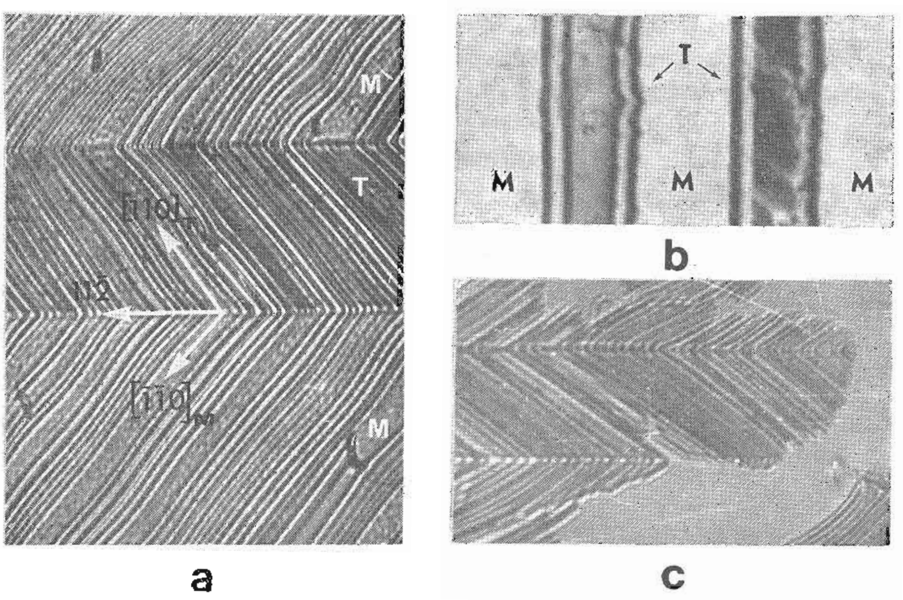

Fig. 5. a) Twinning and slip bands in a surface covered by oxide. M is the matrix and T the twin crystal (X 2300).

b) An electron micrograph showing twinning within the bulk material (X 100.000).

c) Surface of a specimen cleaned by sodium cyanide. From some regions the oxide layer has been removed. In these regions slip and twinning are not visible (X 2300).

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ΠΕΡΙΛΗΨΗ

ΣΧΗΜΑΤΑ ΑΠΟ ΘΕΡΜΙΚΗ ΔΙΑΒΡΩΣΗ ΣΕ ΠΟΛΥΚΡΥΣΤΑΛΛΙΚΟ ΧΑΛΚΟ ΚΑΙ Η ΒΟΗΘΕΙΑ ΕΛΑΦΡΙΑΣ ΟΞΕΙΔΩΣΗΣ ΣΤΗΝ ΠΑΡΑΤΗΡΗΣΗ ΤΟΥΣ

Ὑπὸ
Ι. Γ. ΑΝΤΩΝΟΠΟΥΛΟΥ

Ὅταν ἕνα ὑλικὸ θερμαίνεται, ἀπὸ τὴν ἐπιφάνειά του φεύγουν ἄτομα. Τὸ φαινόμενο ὀνομάζεται θερμικὴ διάβρωση καὶ τὰ σχήματα ποὺ δημιουργοῦνται σχήματα θερμικῆς διάβρωσης. Αὐτὰ ἦταν τὸ ἀντικείμενο αὐτῆς τῆς ἐργασίας. Ὡς ὑλικὸ χρησιμοποιήθηκε πολυκρυσταλλικὸς χαλκός, ὁ ὁποῖος εἶχε ὑποστεῖ ἀρχικὰ ἔλαση, στὴ συνέχεια ἀνόπτηση καὶ τελικὰ ταχεία ψύξη. Παρατηρήθηκαν καὶ ἐξηγήθηκαν διάφορα σχήματα προερχόμενα ἀπὸ θερμικὴ διάβρωση. Χρησιμοποιώντας μικρὴ ταχύτητα ψύξης σὲ μερικὰ δείγματα πετύχαμε ἐλαφριὰ ἐπιφανειακὴ ὀξείδωση. Αὐτὸ ἦταν πάρα πολὺ χρήσιμο γιὰ τὴν παρατήρηση ὁρισμένων ἐπιφανειακῶν σχημάτων, τὰ ὁποῖα δὲ θὰ ἦταν ἴσως δυνατὸ νὰ παρατηρηθοῦν ἀλλιῶς. Ἀπὸ τὰ τελευταῖα αὐτὰ σχήματα προῆλθαν πληροφορίες σχετικὰ μὲ τὴν κρυσταλλογραφία τῶν κρυσταλλιτῶν ποὺ ἐξετάστηκαν.