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ION BEAM SPUTTERING AND DEPTH PROFILING: ON THE CHARACTERISTICS OF THE INDUCED ROUGHNESS AND THE MEANS TO CURE IT AT BEST

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ION BEAM SPUTTERING AND DEPTH PROFILING : ON THE CHARACTERISTICS OF THE INDUCED ROUGHNESS AND THE MEANS TO CURE IT AT BEST

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In principle the theoretical ultimate depth resolution power of the SIMS analysis is very high, of the order of one or two atomic layers. However the practical limit is always much poorer. Experimentaly the (normalized) blurring of an initialy perfectly sharp concentration step is frequently found to be of the form $z = (a*z)^b$, a being of the order of one atomic layer and b varying between .5 and 1. This global effect results from the superposition of various phenomena due to the radiation effects of the primary beam :

- surface roughness due either to statistical fluctuations in sputtering or sample microstructural inhomogeneities
- atomic diffusion enhanced by point defects, either in the bulk or on the surface
- atomic delocalization in cascades
- differential pulverization in alloys.

The last three terms have been given now fairly complete models, either from the fundamental point of view of understanding the effects, or the practical one of obtaining the true profile from the blurred one (for a recent review see for example [1] and references therein). On the contrary the first point is still obscure. In particular many efforts have been devoted to the calculation of the evolution of the topography of a given surface under ion sputtering in the case of a very large roughness. On the contrary the understanding of the initiation of a small one on an initialy very smooth surface, a very important part of the in-depth analysis, is still in infancy. The purpose of the present communication is to report the first results of a study devoted to such an understanding. In a second part, we shall propose a new method to correct the experimental profiles from the blurring effect of the sample roughness in typical cases of in-depth analysis.

BEAM-INDUCED ROUGHNING

We have studied the beam induced roughning by observing two polycristalline samples of nickel. Both have been polished by a classical mechanical grinding (-> 5 µm mesh diamond). Then, one of them has been annealed under vacuum, at high temperature by high

frequency heating, in order to eliminate the work-hardening due to the mechanical grinding and to increase the grain size. So, grains of few mm large have been obtained. Specially we have noticed a large grain with twins, very well observable by crystallographic contrast in SEM. The two samples have been sputtered in a SMI 300 CAMECA apparatus in the same operating conditions with Ar^+ or O_2^+ ion bombardment (E_o = 6 keV, δ = 100 $\mu A/cm^2$). Under these conditions the sputtering rate amounts to 0.25 nm/sec. under Ar^+ bombardment and 0.2 nm/sec. under O_2^+ bombardment.

Concerning the sample mechanically polished, the formation of cones appears in the first minutes of the bombardment. The axis of cones is parallel to the direction of ion beam and the structure changes unceasingly during the sputtering as showed by [2]. This classical result does not necessitate a special development in the context of this study. The annealed sample containing twins has been specially studied. During the first thirty minutes of sputtering no particular roughness has been observed by Talystep investigation or SEM images of the crater. On the contrary, after one hour, a very fine "hill and valley" structure (mesh $\simeq$ 0,1 μm) appeared. It is related to the crystallography as it desappeared in a twin as shown on fig. 1 which represents two scanning electron images enregistrated in the crater at the limits of the twin. This observation cancels the possibility of a consequence of a residual work-hardening due to polishing.

These results are in good agreement with the observations made on polycrystalline copper foils [3] or aluminium specimens [4].

Our observations seem to show that this type of roughning does not depend qualitatively on the orientation or the nature of the ion bombardment. Its origin should be related to the bombardment - induced dislocations network [3]-[5].

We are devoting all our energies to understand this phenomenon and to know for example if the resulting roughness is due to a point defects clustering depending on the crystalline orientation or to the orientation of the dislocation loops with regard to the surface [5]. Then, we shall able to define the best suited sample to a good in-depth profiling analysis.

PROFILE CORRECTION

From a practical point of view, once settled the best experimental conditions, one can try to substract the residual blurring due to the roughness, from the measured profile $C_a(\bar{z})$, in order to recover the true one $C_r(z)$. Up to now the most frequently used approach was the deconvolution one [6], in which the various deleterious effects of the primary beam, including the roughning term, were supposed to be described by a "resolution function" $f(\bar{z},z)$, giving for a mean depth $\bar{z}$, the contribution to the secondary flux coming from the depth z. The apparent profile is then given by :

$$A) \qquad C_a(\bar{z}) = \int C_r(z) * f(\bar{z},z)\, dz$$

If the f function doesn't depend explicitely on $\bar{z}$, this equation becomes a convolution equation which can be solved by standard methods. However it is well established that from the four perturbations above mentionned, the last three are not amenable to such a form, although theoretically well founded procedures can be

proposed to handle with [1]-[7], and in the case of roughness the f function depends on $\bar{z}$, and the deconvolution method is no more founded.

Let us describe the induced roughness on an initially well prepared sample by its area density, $\rho_a(\bar{z},u)$ giving the fraction of the total emitting area located at the depth u with respect to the mean depth $\bar{z}$. In this case the actual surface is made of small facets [8], and as shown above, making a very small angle with the mean surface. The apparent concentration $C_a(\bar{z})$ includes the contribution from all depths u from $-h/2$ to $+h/2$. h being the amplitude of the roughness. To a good approximation the area density can then be taken as $\rho_a(\bar{z},u) = 1/h(\bar{z})$, i. e. a constant density. If the actual profile is not to sharply curved, it can be Taylor expanded in u giving :

$$B) \quad C_a(\bar{z}) = C_r(\bar{z}) + h^2/6 * d^2C_r/dz^2 + O(h^4)$$

and later :

$$C) \quad C_r(\bar{z}) = C_a(\bar{z}) - h^2/6 * d^2C_a/dz^2 + O'(h^4)$$

The equation C) is now the basis of our "deblurring" procedure, either using an "a priori" known roughness amplitude h, or fitting it on some well known in-depth concentration curve.

The figure 2 shows the result of a numerical simulation of this process in the case of standard diffusion profile, corresponding to an error function with $\sqrt{Dt} = .5$ (the solid line of the figure). The "true" profile as been blurred according to a constant roughness of h = 1 (dotted line), or to an evolving one with $h = h + \sqrt{a*\bar{z}}$ the value of a being .01, (dash-dotted line). These values correspond to very large roughnesses, with respect to the Dt value. In a second step these "experimental" curves have been "deblurred" according to equation C). In both cases the resulting profiles are undistinguishable from the true curve (maximum error of less than 10^{-3}) despite of the large roughness simulated in this calculation.

CONCLUSION

On samples, the surface quality of which is suitable to in-depth profiling studies, we have observed a beam-induced roughning related to the elimination of point defects. The precise mechanism remains to be explained in more details. Further studies are now in progress.

On the other hand we have proposed a new procedure which prooved to be very efficient in removing from experimental profiles the residual perturbations due to the sample roughness even if evolving during the analysis.

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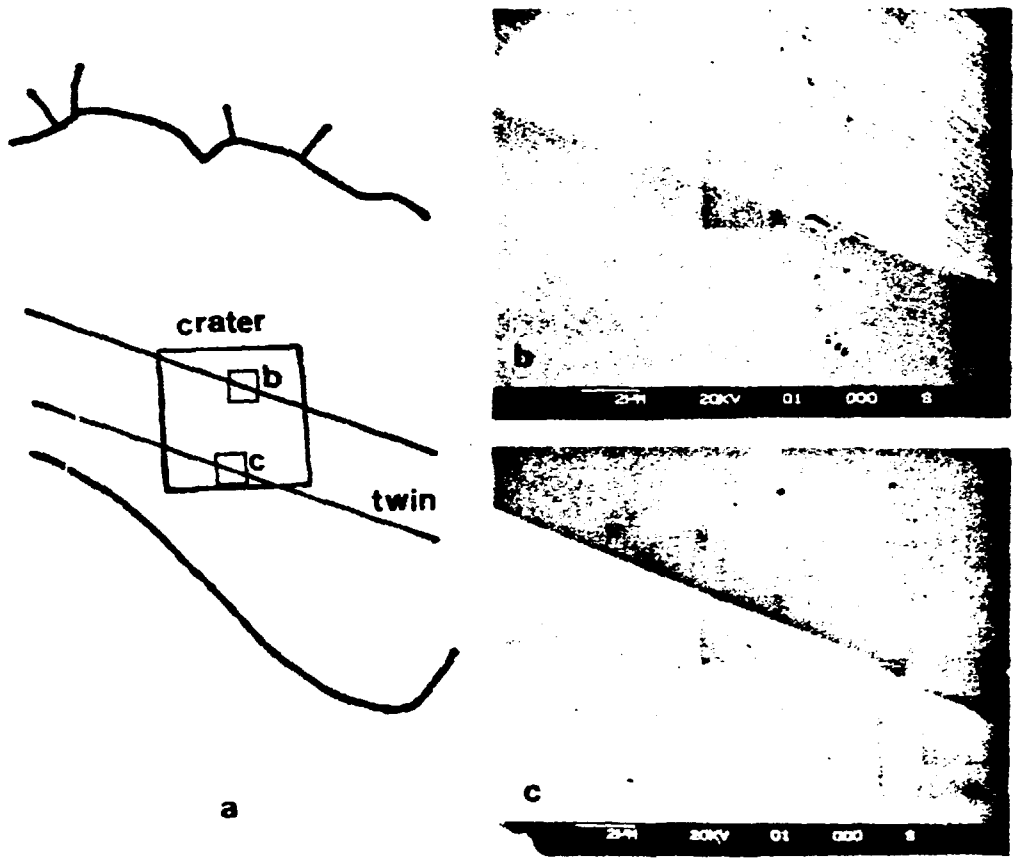

Fig. 1 : Beam-induced roughning observed on a Ni crystal containing twins
a) schematic diagram showing grain, twin, crater and images
b) and c) secondary electron images enregistred at the limits of a twin (roughness is the same outside the twin and there is no relief inside)

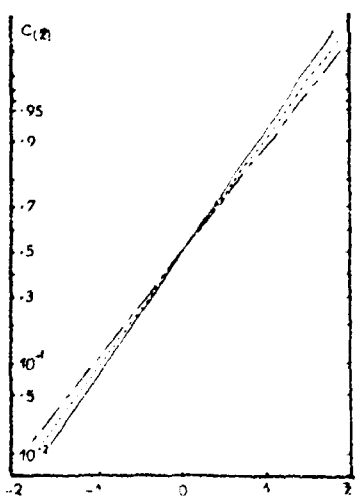

Fig. 2 : Roughness effects on a diffusion profile. The full curve represents a diffusion profile in the semi infinite geometry ($Cr(\bar{z}) = erf(\bar{z})$ for $\sqrt{Dt} = .5$). The dotted and dash-dotted curves represent the apparent concentrations in the presence of a stationnary roughness, h = 1, or of a variable one. The "deblurred" profile is virtually undistinguishable from the full curve.