

LOW INCIDENCE X.RAY GONIOMETRY FOR THIN FILMS TEXTURE ANALYSIS.

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INTRODUCTION.

The Schulz goniometric arrangement (Bragg-Brentano conditions) (1), working with a parafocussing geometry, is generally used for a bulky sample, we do not need to apply intensity correction when the sample rotates around its azimuthal axis φ and its tilting axis ψ .

In the case of thin films (2 - 3), the diffracted intensity is very weak compared to the background, and we have developed a low incidence angle diffraction technique to increase the diffracting volume, and consequently the diffracted intensity. In this case, it is necessary to correct all the information coming from the detector.

I EXPERIMENTAL PROCEDURES.

A texture goniometer built in our laboratory is used. Each rotation movement ω , ψ , φ is driven by a computer. The incident beam wavelength comes from a graphite monochromator, the cross section of the beam is adjusted by the slits of the monochromator and knives slits close to the sample. The diffracted beams are detected by a INEL Curved Position Sensitive Detector, and analyzed by the computer. The azimuth step and the tilt step are respectively 5° and $2,5^\circ$ or 5° and 5° .

II LOW INCIDENCE ANGLE.

In the case of low incidence $\alpha = \theta + \omega$ (where $\omega < 0^\circ$ is the gap between the Bragg-Brentano geometry and the new one).

This new geometric arrangement leads to:

- an enlargement of diffracted beam coming from the increasing of the irradiated area and from the defocussing
- an angular location of the (hkl) lattice planes which depends on the angles (ω , ψ , φ) [4].

The integrated intensity detected is given by the relation

$$I_{\omega, \psi, t} = I_{\infty} \cdot \left[1 - \frac{\operatorname{tg} \omega}{\operatorname{tg} \theta} \right] \cdot \left[1 - \exp \left(\frac{-\mu \cdot t}{\cos \psi} \cdot \left(\frac{1}{\sin (\theta + \omega)} + \frac{1}{\sin (\theta - \omega)} \right) \right) \right] \quad \text{Equ. 1}$$

where t is the thickness of the film, μ the linear absorption coefficient and I_{∞} the integrated intensity which the detector would measure for an infinite thickness of the film in Bragg-Brentano geometry.

So in low incidence geometry whatever the thickness the intensity must be corrected.

Enlargement of the beam

The true incidence angle of the beam on the surface of the sample depends on the ψ tilt angle and on the ω angle.

On the Fig.1 are drawn some 2θ spectrum of a copper 865 Å thick film deposited on glass. Two of them are measured in Bragg-Brentano conditions ($\alpha = \theta$; $\psi = 0^\circ$ and $\alpha = \theta$; $\psi = 60^\circ$), the two others are measured in low incidence geometry ($\alpha = 5^\circ$; $\psi = 0^\circ$ and $\alpha = 5^\circ$; $\psi = 60^\circ$). We can see :

- an important background coming from the substrat
- the enlargement of the (111) line and the coming out of (200) line as the true incidence angle decreases.

On this figure the background can be evaluated and subtracted to obtain the real intensity of the lines.

Verification of the correction laws

We have chosen isotropic copper films to check the validity of the previous relation (1). The experimental results agree with the theoretical values of $I_{\omega, \psi, t} / I_{\infty}$.

The location corrections will be checked on all the presented pole figures. The defocussing, background and enlargement corrections of the diffracted beam are directly taken into account by the use of the Curved Position Sensitive Detector, when we measure the integrated intensity.

III APPLICATIONS.

Several experiments on thin films of different materials are presented to give a general idea of the method and its ability.

First example :

A 2000 Å thick copper film deposited cleaved (100) face of NaCl, heated at 300 °C. Two pole figures were done simultaneously (5). The first one (Fig.2a) made on the copper film and the second one (Fig.2b) made on the NaCl single cristal. The film pole figure which is corrected shows only two orientations of the copper which is twinned. The twinned part represents about 10 % of the orientations. The superposition of the each pole figures which represents each material in the same reference give us the well known orientation relationships between the two phases: (100)NaCl // (100)Cu and [001]NaCl // [001]Cu.

Second example :

The texture of an aluminium coating of 0,6 μm thick deposited on copper is analysed. The Bragg-Brentano pole figure without correction presented in Fig.3 shows a smooth texture of aluminium which can be described by a fiber texture, the [111] fiber axis of which makes 15° with the normal of the sample. After intensity corrections, we obtain pole figure (Fig.4). The ratio of the intensities on the 70° ring to that of the maximum located near the center corresponds now to the ratio for a fiber texture. A low incidence angle measurement of the pole figure presented in Fig.5 gives the same information [except in the blind area which depends on the low incidence technique (4)]. The intensities are about twice as high as in Bragg-Brentano pole figure even with a smaller counting time (3.75 seconds instead of 5.00 seconds).

Third example :

Two samples of 1 μm thick PVD + CVD tungsten deposits on a silicon single cristal are presented. As the tungsten absorption coefficient is high we have chosen the Bragg-Brentano geometry. Only half a pole figure is drawn by sample (Fig.6).

On the left part, we can observe a first fiber texture which [100] axis is normal to the surface; it is represented by the ring located at 45° on the pole figure. A second fiber texture with [001] normale to the surface, weaker than the first one, the pole of which (110) is located at the center. The ring located at 60° belonging to this fiber texture is not observable.

On the right part, we have drawn the pole figure, coming from the same kind of sample which elaboration conditions are different. In this case only the second fiber texture exist.

We have studied the first tungsten deposit with the low incidence technique so we are able to advantage the information coming from the surface. The comparison of these intensities with the Bragg-Brentano ones can give us the texture variation across the thickness of the film. On the Fig.7 the diffracted intensities of the (200) lattice planes across a radius of the pole figure are drawn. The peak (200) located at 45° belongs to the [110] fiber texture. Its intensity increases as the incidence angle increases too. This indicates that the second texture component is weaker at the surface than inside the film.

Fourth example.

We present a pole figure (Fig.8) a $1\mu\text{m}$ thick multilayers Al/Fe deposited on a silicon crystal. This film is constituted by successive layers of Al (175 \AA) and Fe (32 \AA). The pole figure shows a perfect fiber axis of the two components.

CONCLUSION

We have shown that it is possible to obtain the texture of a thin film by low incidence X-RAY texture goniometry. The intensity and the location of the information corrections have been checked and correspond to the theory.

It is possible to obtain the texture of thin film with a thickness under 1 000 \AA . The use of the position sensitive detector is helpful to take the defocussing phenomenon and the background values into account.

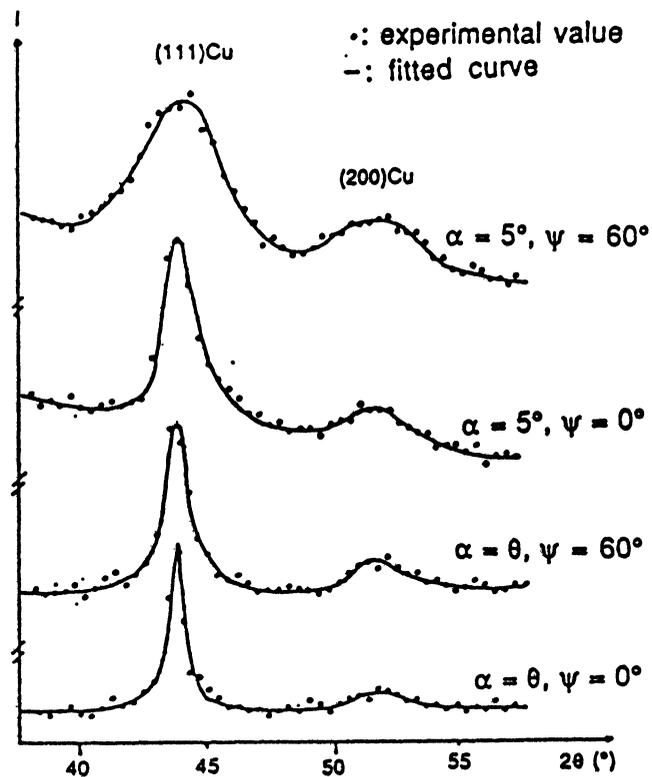
The low incidence technique allows us to get some information on the texture evolution across the thickness of the film.

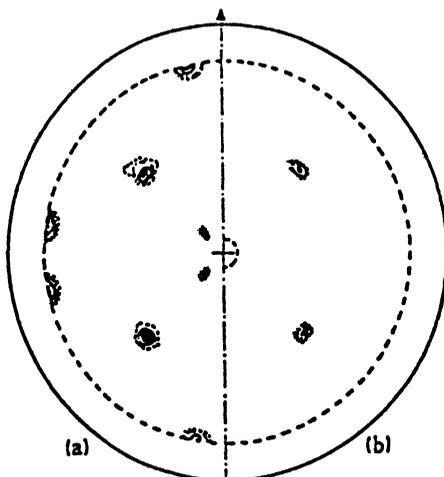
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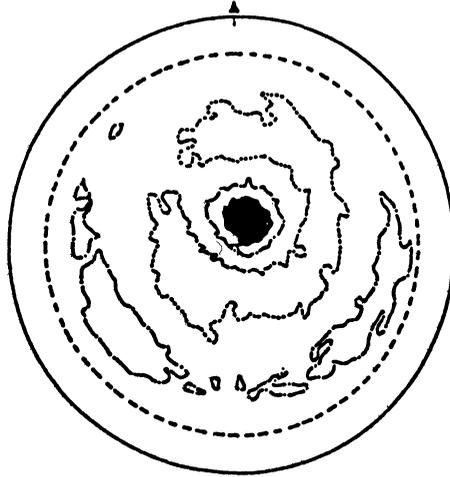
Fig.1: Widening of the diffracted peaks (111) and (200) of a copper film for some values of α and ψ .





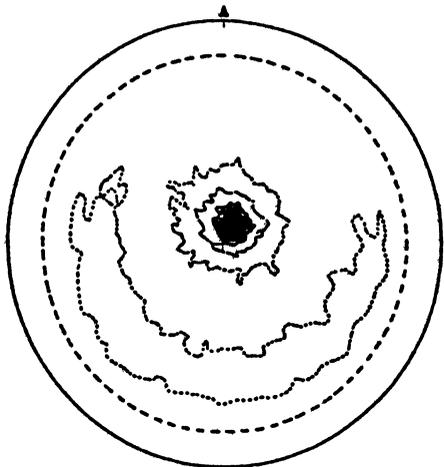
--- 80 mini : 0	--- 100 mini : 16
--- 300 maxi : 10712	--- 500 maxi : 3725
--- 1500 Time : 20s.	--- 2000 Time : 20s.

Fig.2a:(100)pole figure:
copper film deposited on NaCl
Fig.2b:(100)pole figure:
NaCl seen across the copper



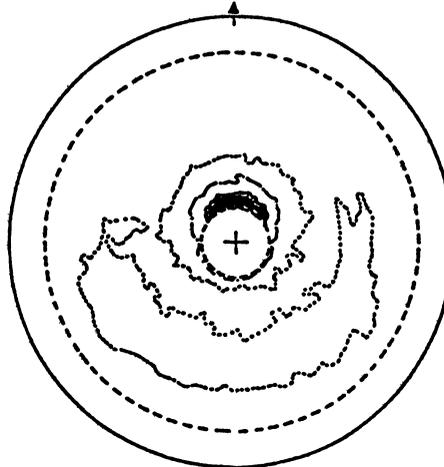
--- 253 mini : 193
--- 346 maxi : 592
--- 439 Time : 5s.

Fig.3:(111)pole figure
aluminium film without
intensity correction
(Bragg Brentano arrangement)



--- 60 mini : 0
--- 153 maxi : 398
--- 245 Time : 5s.

Fig.4:The same as fig.3
with intensity correction



--- 106 mini : 0
--- 271 maxi : 707
--- 436 Time : 3,75s.

Fig.5:Low incidence angle
(5°)(111) pole figure of the
same film with intensity and
location corrections.

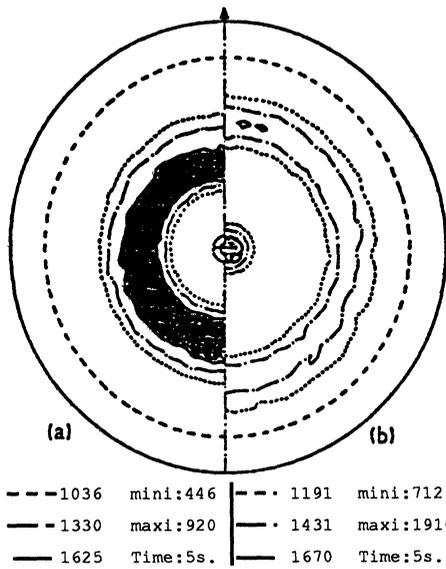


Fig.6: (110) pole figures of two different tungsten films.

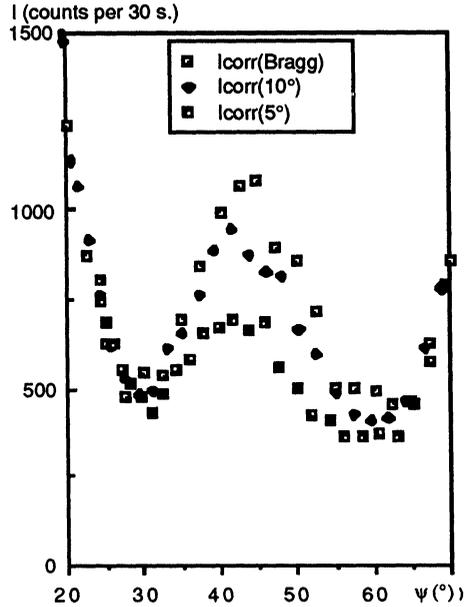


Fig.7: Cross section of (200) pole figure of a tungsten film.

Plane (200)_{Sample}: alifer/SI
 plan 200 ALou 110fe

Omega= -3.32 Ldthe= 54.00 Hdthe= 58.00 Ldk= 53.00/ 54.00 Rdk= 58.00/ 59.00

Nbr: 835
 CR: 1
 ST: 1
 NR: 1
 RE: 0

Measur: 13-SEP-90
 Plot.: 13-SEP-90

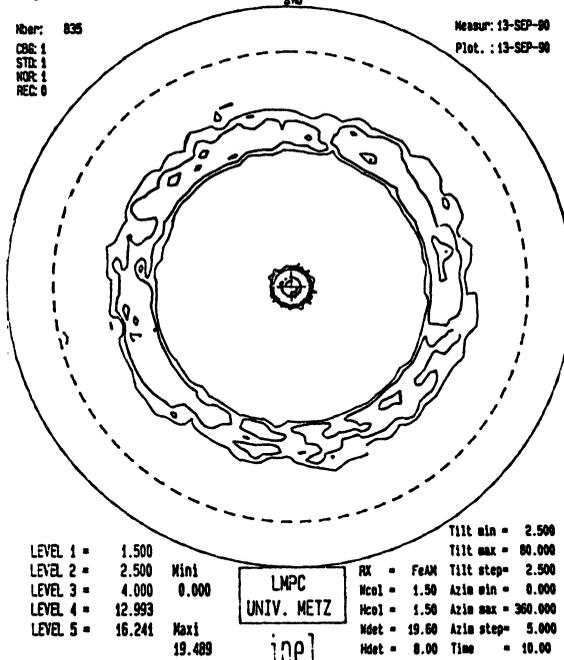


Fig 8: Al(200) and Fe(110) multilayers pole figure.