*Textures and Microstructures*, 1991, Vols 14–18, pp. 175–180
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# CORRECTION OF POLE FIGURES MEASURED ON SMALL DIAMETER METALLIC WIRES

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## INTRODUCTION

In order to improve the mechanical properties of metallic wires and to achieve better understanding of their microstructure, it is necessary, among other characteristics, to determine their crystallographic texture and to know how it changes through the manufacturing process. The most commonly used technique to determine the texture of a specimen is to measure intensity pole figures by X-ray diffraction, however, in the case of small diameter wires, this technique cannot be directly applied. Various methods, semi-empirical or experimental, has been proposed (1)(2)(3)(4) but they either lack rigour in their assumptions or are performed on comparatively large diameters (> 3mm). Two papers published recently (5)(6), are dealing with the subject on much more rigorous basis. Both of them start with the expression of the intensity $I(\Phi,\Psi)$ diffracted by a sample in a given direction $\Phi\Psi$ (see figure 2). The sample is an array of wires placed side by side for intensity reasons.

$$I(\Phi,\Psi) = \int_{V(\Phi,\Psi)} K(\Phi,\Psi,\mathbf{r}).\exp(-\mu.x(\mathbf{r},\Phi,\Psi)).dV(\mathbf{r}) \qquad (1)$$

$I(\Phi,\Psi)$ representing the variation of the intensity due to texture of the sample and other physical phenomena such as defocusing and background noise. Since the purpose of the present paper is to study absorption corrections, $I(\Phi,\Psi)$ will be assumed to be corrected by other means and carry on only texture and geometry information. Although the actual texture of drawn wires is not homogeneous (texture gradients), we shall only treat the simplified case where $K(\Phi,\Psi,\mathbf{r})$ does not depend on $\mathbf{r}$. Equation (1) then becomes $I(\Phi,\Psi) = K(\Phi,\Psi).A(\Phi,\Psi)$ with

$$A(\Phi,\Psi) = \int_{V(\Phi,\Psi)} \exp(-\mu.x(\mathbf{r},\Phi,\Psi)).dV(\mathbf{r}) \qquad (2)$$

$A(\Phi,\Psi)$ is the intensity diffracted by an isotropic sample and is also the correction coefficient that allows to determine the texture information $K(\Phi,\Psi)$ knowing the total diffracted intensity $I(\Phi,\Psi)$. $\mu$ being the absorption coefficient of the studied material relatively to the wavelength of the radiation used for analysis, $dV(\mathbf{r})$ being an elementary portion of volume located by vector $\mathbf{r}$ and $x(\mathbf{r},\phi,\psi)$ the path length of incident and diffracted rays inside the specimen.

## INTENSITY CORRECTION FOR A FLAT SPECIMEN

Though this case is well documented, it is important for a better understanding of some problems in the case of wires. For an isotropic flat specimen, the diffracted intensity is given by equation (2). To calculate the volume element dV, two cases may be distinguished:

a) The X-ray beam section is constant, i.e. when the angle between the normal to the specimen surface and the incident beam increases, the irradiated surface also increases. In this case, the volume element is equal to $dV = S(z).dz = S_0/\cos\alpha_i$, $S_0$ being the section of the incident beam. Provided that the sample thickness is considered infinite, it can be shown by integration that the diffracted intensity does not depend on the angles $\Phi$ and $\Psi$ and, therefore, there is no need to make intensity corrections. It is the most frequent case for practical pole figures measurement.

b) The irradiated surface is constant, i.e. a screen has been put on the specimen, leaving only a limited part of the beam giving rise to diffracted intensity. In this case, the volume element is equal to $dV = S_0.dz$, $S_0$ being the section of the uncovered area. Then, even if the sample thickness is considered infinite, the diffracted intensity is no longer constant but proportional to $\sin\Theta.\cos\Psi$. It is therefore necessary to make intensity corrections.

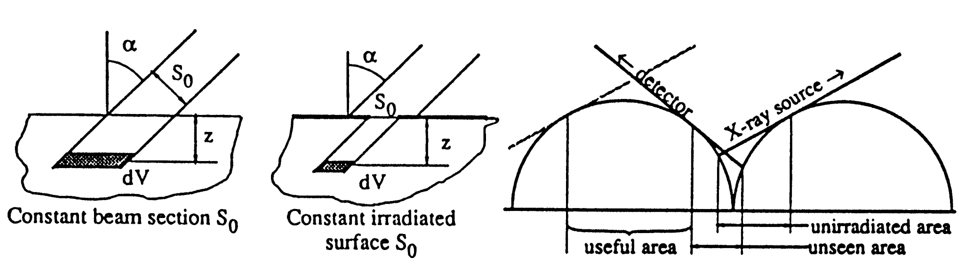


Figure 1: irradiated volume (for a flat sample) and shadowing effect

## INTENSITY CORRECTION FOR CYLINDRICAL SPECIMENS.

In the case of cylindrical specimen, direct calculation of integral (2) has to be performed over the irradiated volume of the specimen. The calculation of $x(\mathbf{r},\phi,\psi)$ is mainly a matter of geometrical relations, however, for the irradiated volume element $dV(\mathbf{r})$ it is not so easy. If the array of wires is considered as infinite in the transverse direction, due to the periodicity, the calculation can be performed on one wire only. In the longitudinal direction, the length of the volume element can be considered as constant (it may not always be the case as we shall see below). The lines for which the path length of incident and diffracted beam is constant are parallel to the wire axis.

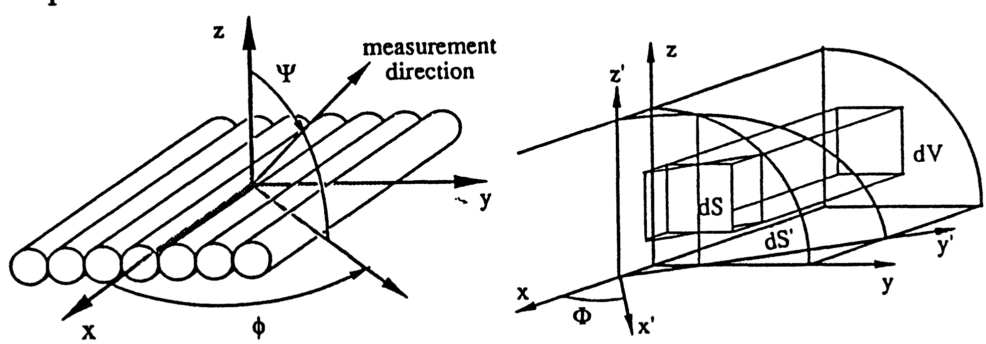


Figure 2: definition of angles and of irradiated volume element.

In papers [5] and [6] authors have chosen to perform integration in the diffracting plane. In this case, the volume element can be written $dV(\mathbf{r}) = l.dS'(\mathbf{r})/(\cos\Psi\cos\Phi)$, $dS'(\mathbf{r})$ being the surface element in the diffracting plane. In the present paper, we have chosen to perform integration over the orthogonal section of the wire. In this case, the volume element is $dV(\mathbf{r}) = l.dS(\mathbf{r})$ with $dS(\mathbf{r}) = dy.dz = r.d\rho.dr$. This way enables one to avoid numerical overflow for $\Phi = 90°$.

Four models can then be used to determine the intensity diffracted by an array of isotropic wires.

a) <u>The infinite absorption model</u>: Setting $\mu=\infty$, equation (2) becomes

$$I(\Phi,\Psi) = \int_{V(\Phi,\Psi)} dV(\mathbf{r})$$

The intensity is then equal to the irradiated volume whose thickness is infinitely thin, i.e. the intensity is proportional to the irradiated surface (the surface of the wire limited by the shadowing effect).

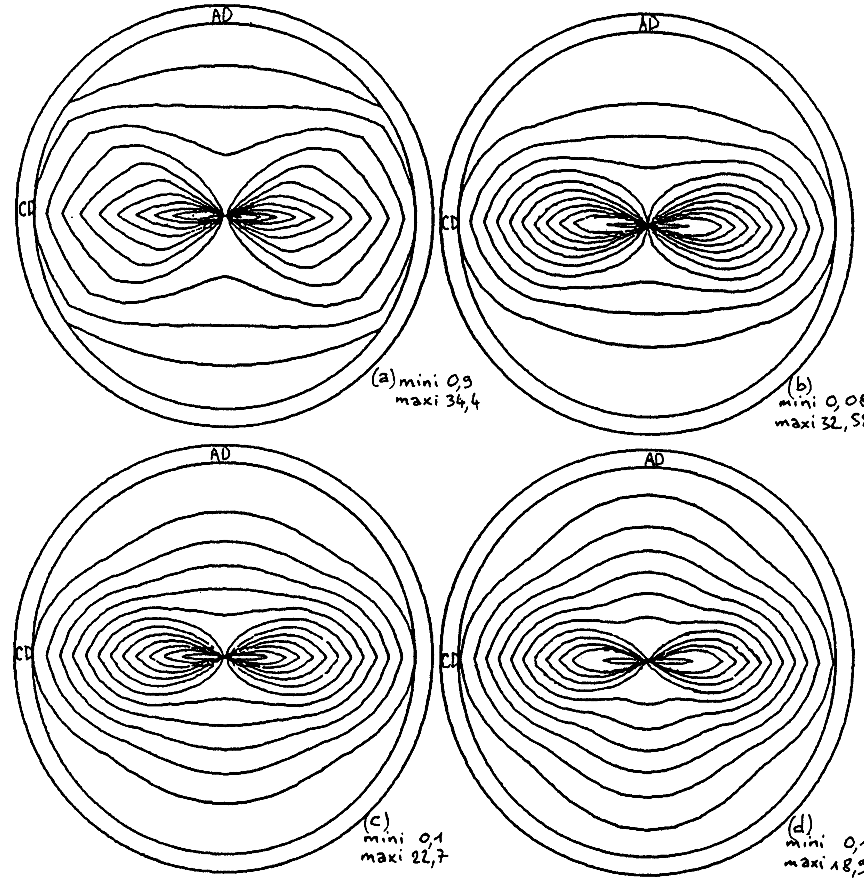


Figure 3: Calculated pole figures with the four models for isotropic steel wires of diameter 0.23 mm. {110} plane with CoKα radiation. 12 levels with constant steps between maxi and mini.

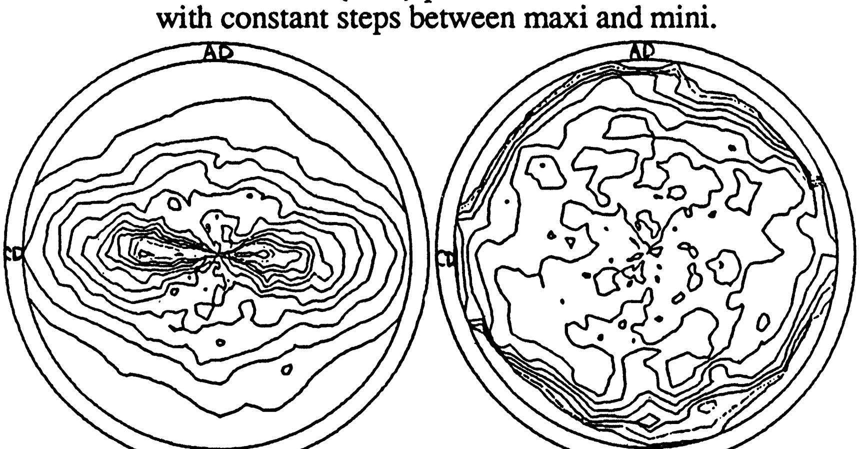


Figure 4: experimental pole figures on heat treated 0.23mm diameter wires on {110} with CoKα radiation. Before and after geometrical corrections. 12 levels with constant steps. AD:axial direction, CD:circum.direction.

b) The infinite absorption model, taking into account the spreading of the beam over the surface of the wire. The intensity irradiating a constant surface element dS decreases with the inclination of the beam relatively to the surface normal. It can be shown that this model is equivalent to the following method: each surface element is considered as flat and with a constant irradiated surface. The correction for flat specimen is applied for each element (with its own incidence) and summed up over the irradiated surface.

c) The finite absorption of a wire surrounded by infinitely absorbing neighbours model. Integral (2) is calculated over the volume limited by the shadowing effect.

d) The finite absorption model. Integral (2) is calculated over the whole volume of one wire and the path length is taken through several wires if needed. Due to the symmetry of the problem, only a quarter of a pole figure needs to be calculated, thus, only neighbours located on one side of the wire must be considered

Figure (3) shows an example of calculation performed with those four models. It can be seen that there are significant changes both in shape and in magnitude between those figures, however model (c) and (d) are rather close. Other calculations have shown that their differences are negligible for over 0.5mm with CoK$\alpha$ radiation.

## LENGTH OF IRRADIATED VOLUME ELEMENT.

The volume element to be considered in integral (2) is equal to $dV(\Phi,\Psi,\mathbf{r}) = l(\Phi,\Psi,\mathbf{r}).dS(\Phi,\Psi,\mathbf{r})$. The expression of $dS(\Phi,\Psi,\mathbf{r})$ has been detailed above. However, $l(\Phi,\Psi,\mathbf{r})$ varies with the incidence of the beam according to the phenomenon described for a flat sample. It depends on wether the surface of the wire array is limited in its longitudinal direction by a screen or not. If it is limited, then $l(\Phi,\Psi,\mathbf{r})$ can be considered as constant, otherwise it varies for each volume element:

$$l=\frac{l_0}{\cos\alpha(\Phi,\Psi,\Theta,\rho)} \text{ with } \cos\alpha(\Phi,\Psi,\Theta,\rho)=\frac{a_3(\Phi,\Psi,\Theta,\rho)}{\sqrt{a_1^2(\Phi,\Psi,\Theta,\rho)+a_3^2(\Phi,\Psi,\Theta,\rho)}}$$

with $a_1 = -\sin\Phi\cos\Theta+\sin\Psi\cos\Phi\sin\Theta$

and $a_3=\cos\Phi\cos\Theta\sin\rho+\sin\Psi\sin\Phi\sin\Theta\sin\rho+\sin\Theta\cos\Psi\cos\rho$

Those corrections lead to little change in the general shape and amplitude of pole figure of correction factors.

## EXPERIMENTAL RESULTS

In order to check the reliability of the method, drawn pearlitic steel wires have been heat treated to remove the crystallographic texture. They were then placed side by side and glued on a flat surface to form an array of wires on which pole figures measurement have been performed with

CoKα radiation on {110}, {220} and {200} planes; background correction was performed for each value of Ψ and Φ (background noise level is also affected by geometrical effects) and defocusing effects were corrected with an iron powder sample. There is a good agreement between the models and the experimental pole figures (see figure 3 and 4), however, for $\Psi > 75°$, the experimental pole figure is overcorrected. With those models, the differences in shape of {110} and {220} pole figures can thus be explained. Several phenomena could explain variations between calculations and experiments:

- the removal of texture by heat treatment was certainly not perfect.
- After heat treatment, the shape and curvature of the wires were not as regular as before and it was therefore difficult to place them side by side regularly.
- The defocusing effects may not be the same for an array of thin wires and a flat sample.

## CONCLUSION

The present study has shown that, in the case of texture measurement of thin metallic wires (diameter < 3mm), it is necessary to take into account the geometry of the specimen with specific absorption corrections. The infinite absorption models are not accurate enough to be of practical use, but up to a penetration depth equal to 2% of the wire diameter (covering a wide domain with CrKα and CoKα radiations), it is enough to consider a finite absorption model on a wire with infinitely absorbing neighbours. The numerical corrections proved to be in good agreement with experimental measurements made on isotropic wires.

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