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Texture Determination in Multiphase Materials with Lamellar Structure

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Abstract

The X-ray anisotropic absorption of lamellar multiphase materials will influence the accuracy of texture determination. A simple method is presented in this work, which enables an approximation to the true texture in the materials to be obtained using conventional texture goniometer. By this method the measurement error can be reduced to a certain extent.

1. Introduction

In multiphase materials, X-ray absorption takes place in all phases present in the materials, depending on the shape, size and arrangement of the phases. This effect is particulary strong in lamellar structures such as eutectiic alloys. In this case the reflected intensity may depend on the angle γ, a rotation about the diffraction vector $\vec{s}$, additionally to the orientation of $\vec{s}$ with respect to the sample, specified by the pole figure angles $\alpha\beta$(1,2,3,4).

$$I_{hkl}(\alpha\beta\gamma) = I_0 \; V^i \; R^i_{hkl} \; P^i_{hkl}(\alpha\beta) \; A^i_{hkl}(\alpha\beta\gamma) \tag{1}$$

where I_0 is the intensity of the incident beam, V^i is the volume fraction of the reflecting phase i, R^i_{hkl} is the reflectivity of the Bragg-reflection (hkl), $P^i_{hkl}(\alpha\beta)$ is the (hkl)-pole figure of the phase i and A^i $(\alpha\beta\gamma)$ is the absorption factor of the material, which depends on the three orientation angles $\alpha\,\beta\,\gamma$.

This effect must be taken into account in X-ray pole figure measurements, which are thus three-dimensional $(\alpha\beta\gamma)$ instead of the conventional two-dimensional pole figures $(\alpha\beta)$. The measurement of three-dimensional pole figures may be accomplished with a conventional texture goniometer with three rotation axes $(\omega\chi\varphi)$, This only requires a geometrical coordinate transformation.

$$(\alpha \;\; \beta \;\; \gamma) \Longrightarrow (\omega \;\; \chi \;\; \varphi) \tag{2}$$

But there are some technical problems, such as the selfshadowing effect of equipment and sample as well as the severe defocalization caused by deviation of the sample surface from the focussing sphere. Hence the measurable angular range and the accuracy of the measured intensity will be considerablly limited. It was thus necessary to find a simple and reliable method, which allows us, as exactely as possible, to determine the textures of lamellar multiphase materials.

We consider the case of large but thin lamellae and sufficiently different absorption coefficients. The absorption factor can be approximated in the form

$$A^i = \frac{1}{\bar{\mu}} + \left[\frac{1}{\mu^i} - \frac{1}{\bar{\mu}} \right] \delta_{n,t} \tag{3}$$

where μ^i is the absorption coefficient of the reflecting phase, $\bar{\mu}$ is the averaged absorption coefficient weighted according to the respective volume fractions of the phases in the sample, n is the normal direction of the lamellae and t is the normal direction of the diffraction plane. This means that the absorption factor takes on its maximum value

$$A^i = \frac{1}{\mu^i} \tag{4}$$

in the case of n // t, and it is minimum

$$A^i = \frac{1}{\bar{\mu}} \tag{5}$$

for n $\not\parallel$ t, Thereby it is assumed, that the reflecting phase has a lower absorption coefficient, $\mu^i<\bar{\mu}$. If, on the other hand, $\mu^i>\bar{\mu}$ then Eq.4 corresponds to the minimum and Eq.5 to the maximum value of A^i.

There is,in practice,a continuous transition from one to the other limiting value. Numerical calculation of anisotropic absorption factors for lamellar two-phase materials shows that the transition has different slope, half maximum width and limiting value depending on the thickness of the lamellae and the absorption coefficients of the phases/5/. A specific simple case of lamellar two phase structure, where all lamellae are parallel to each other and perpendicular to the sample surface and the diffraction vector perpendicular also to the surface ($\alpha = 0°, \beta = 0°$), has been considered. The calculated absorption factor as a function of the angle γ is given in Fig. 1. Thereby it is assumed, that the thickness of the lamellae is smaller than or equal to the reciprocal of the absorption coefficients of the phases in the materials, and that the difference of the lamellar thicknesses does not exceed an order of magnitude. The shape of the curves in Fig.1 is typical for many practically important cases, one sees that the absorption factor is near to $1/\bar{\mu}$ in a wide angular range except for $\gamma = 0°$, where n // t. Hence, an approximation to the true (α,β)-pole figure can be obtained by taking the maximum or minimum value as a function of γ separately corresponding to the two cases, $\mu^i < \bar{\mu}$ or $\mu^i > \bar{\mu}$. On this basis, a method of pole figure measurement in lamellar multiphase materials was developed, which uses intensity measurement of few γ-values, for example three γ-values, for each pole figure point $(\alpha\beta)$.

2. Experimental Technique

2.1 Geometrical Coordinate Transformation

Eq.2 can be expressed in the following form /6/

$$\cos\omega = \frac{\cos\alpha}{\sqrt{1-\sin^2\alpha\cos^2\gamma}} \quad (6)$$

$$\sin\chi = \sin\alpha \cdot \cos\gamma \quad (7)$$

$$\sin\varphi = \frac{\cos\alpha\sin\beta\cos\gamma-\cos\beta\sin\gamma}{\sqrt{1-\sin^2\alpha\cos^2\gamma}} \quad (8)$$

$$\cos\varphi = \frac{\cos\alpha\cos\beta\cos\gamma-\sin\beta\sin\gamma}{\sqrt{1-\sin^2\alpha\cos^2\gamma}} \quad (9)$$

In the general case the values of the angles $\alpha\beta\gamma$ are limited to the range

$$\begin{aligned} &0^\circ \leqslant \alpha < 90^\circ \\ &0^\circ \leqslant \beta \leqslant 360^\circ \\ &0^\circ \leqslant \gamma \leqslant 180^\circ \text{ (experimental symmetry)} \end{aligned} \quad (10)$$

The values of the correspondent Euler's angles are in the range

$$\begin{aligned} 0^\circ &\leqslant \omega < 90^\circ \\ -90^\circ &< \chi < 90^\circ \\ 0^\circ &\leqslant \varphi \leqslant 360^\circ \end{aligned} \quad (11)$$

2.2 Consideration of Defocalization

At $\alpha \neq 0^\circ$, the sample surface deviates from the focussing sphere. A stronger defocalization effect may take place depending on the angle γ (Fig.2). In order to reduce the influence of defocalization on the measured results, we can select an as wide as possible receiving slit. As is seen from Fig.2a, the width d of receiving slit is determined by the radius r of the round specimen, the Bragg angle θ, the smallest angle σ between the X-ray beam and the sample surface and the goniometer radius R

$$d = 2\{ r\sin(2\theta-\sigma) - [R-r\cos(2\theta-\sigma)]\,\mathrm{tg}\Delta\theta \} \quad (12)$$

where

$$\cos\Delta\theta = \frac{R + r\cos\sigma}{\sqrt{R^2+ r^2+ 2Rr\cos\sigma}} \quad (13)$$

During measurement the integral illuminated area always covers the total surface of the specimen, which was made up into a round thin slice, to avoid the influence of inhomogenous texture or structure. Hence, the specimen radius can be considered as the size of the illuminated area. A too wide receiving slit may, however, lead to overlapping with the reflections from other crystal planes. As is illustrated in Fig.2b, the maximum and minimum Bragg-angle corresponding to a given receiving slit width can be calculated according to the following expressions

$$\theta_{max} = \theta + [\Delta\theta_r^1 - \Delta\theta_i^1]$$

$$= \theta + [\arcsin[\frac{r\sin(2\theta-\sigma)+0.5d}{R+r\cos(2\theta-\sigma)}] - \mathrm{arctg}[\frac{r\sin\sigma}{R-r\cos\sigma}]] \quad (14)$$

and

$$\theta_{min} = \theta - [\Delta\theta_r^2 + \Delta\theta_i^2]$$

$$= \theta - [\arcsin[\frac{r\sin(2\theta-\sigma)+0.5d}{R-r\cos(2\theta-\sigma)}] - \arccos[\frac{R+r\cos\sigma}{\sqrt{R^2+r^2+2Rr\cos\sigma}}]] \quad (15)$$

3. Experimental Results

In order to check the influence of lamellar structure on the texture determination, a directionally solidified lamellar Pb-Sn sample was used, the longitudinal section of which is shown in Fig.3. Texture measurement was carried out with the automatic texture goniometer ATMA-C using a sample of 10 mm diameter cut parallel to the growth direction. The reflected intensity was measured as a function of $(\alpha\,\beta\,\gamma)$ using a new controlling program based on the coordinate transformation eqs. 6-9. The measurements were taken with Co-Kαradiation. With the dimensions of the goniometer and the receiving slit a maximum divergence $\Delta\theta = 3°$ was obtained according to Eqs. 14-15. Tab.1 shows the θ-values of some neighbouring diffraction peaks of the composite. Hence, the $(220)_{Pb}$ reflexion could be measured free of superposition with those conditions.

	θ
$(211)_{Sn}$	26.34° (< θ_{min})
$(220)_{Pb}$	30.76°
$(301)_{Sn}$	32.65° (> θ_{max})

Tab.1 The Bragg angle of the $(220)_{Pb}$ diffraction and its neighbouring diffraction angles

Some of the results are shown in Fig.4b. It is seen that the reflected intensity depends strongly on the angle γ, i.e. the rotation about the diffraction vector. For comparision Fig.4a shows the corresponding results for a random eutectic sample.

4. Conclusion

In lamellar multiphase materials the reflected X-ray intensity depends not only on the usual pole figure angles α, β describing the orientation of the diffraction vector $\vec{s}$, but also on the rotation angle γ about $\vec{s}$. This is caused by the anisotropic structure.

The effect of anisotropic absorption must be taken into account in texture measurement of lamellar multiphase materials.

The 3-γ method presented in this work can be used to correct the texture determination in these materials and reduce the measurement error to a certain extent.

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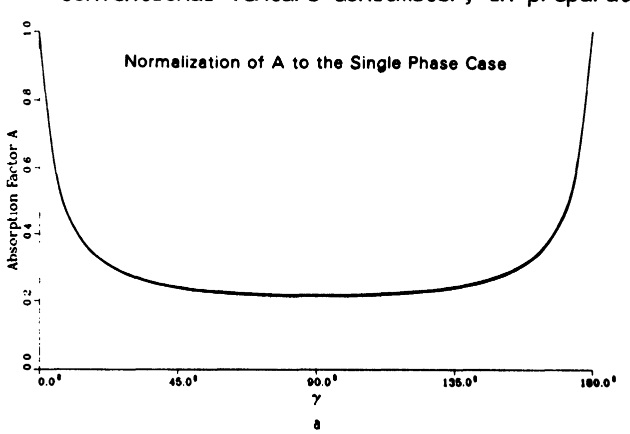

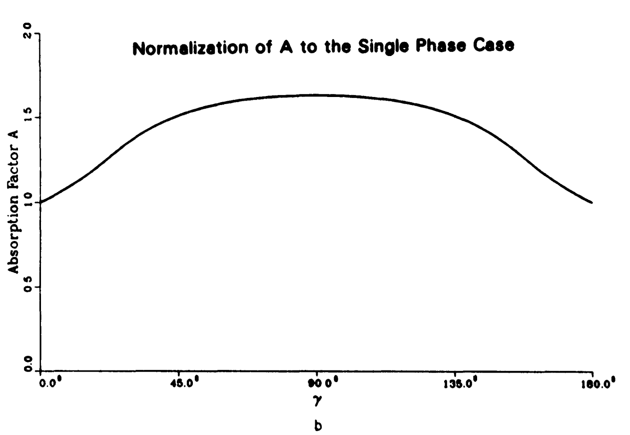

Fig.1

Absorption factor for lamellar structure with the lamellar thickness of 1 µm when $\alpha, \beta = 0°$, $\theta = 45°$
a) $\mu^1 = 0.1/\mu m$, $\mu^2 = 1.0/\mu m$
b) $\mu^1 = 1.0/\mu m$, $\mu^2 = 0.1/\mu m$

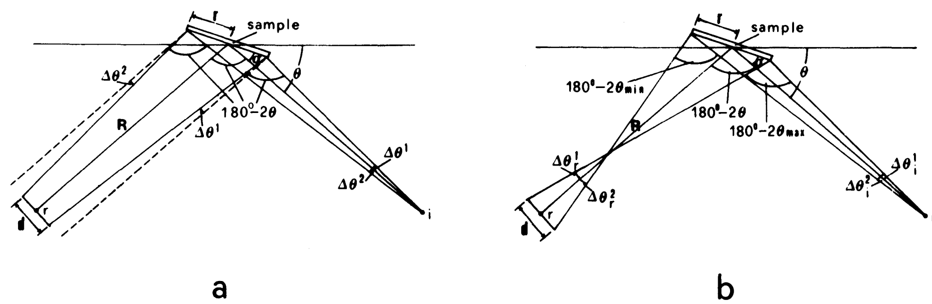

Fig.2 Diffraction geometry when the sample deviates from the focussing sphere

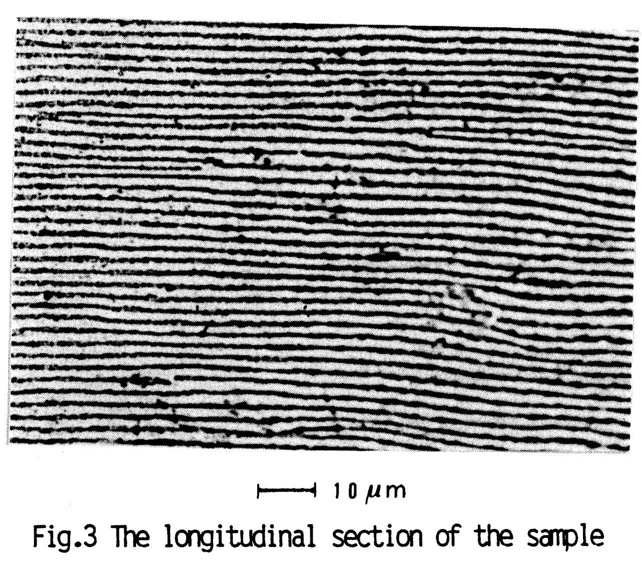

Fig.3 The longitudinal section of the sample

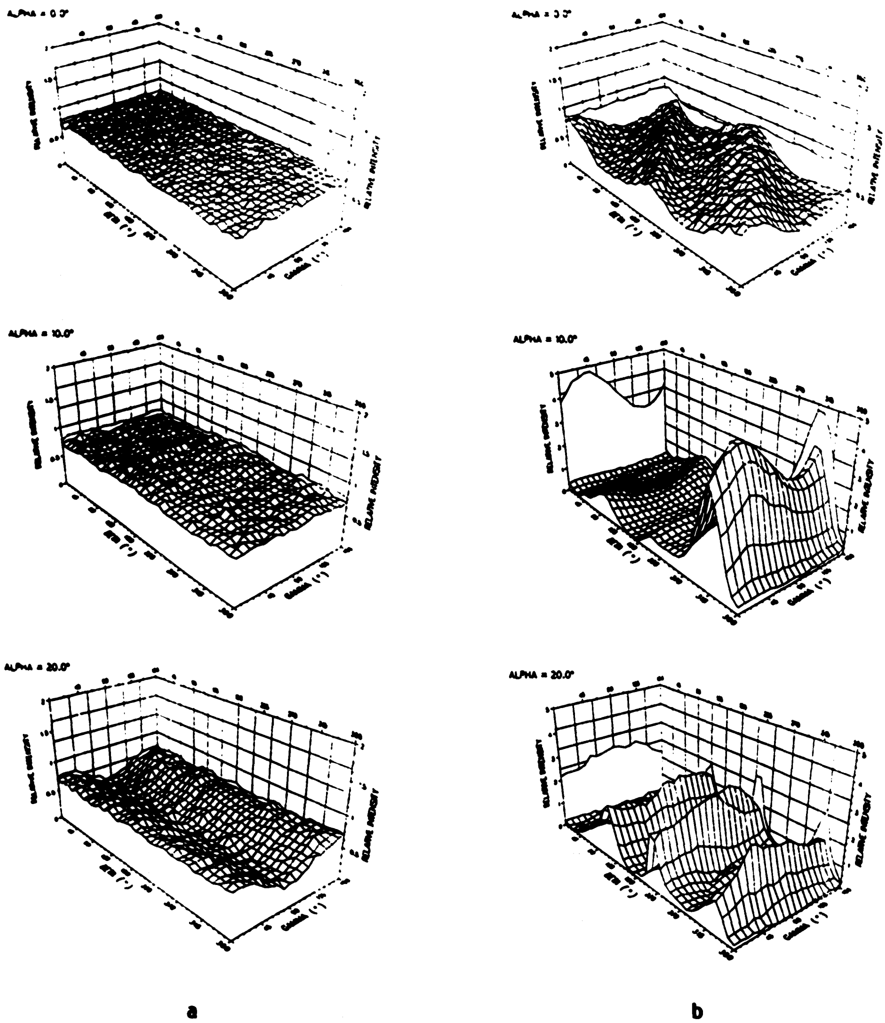

Fig.4 Measured results of three-dimensional pole figures
a) for random sample
b) for directionally solidified sample