THE INFLUENCE OF SURFACE ROUGHNESS ON THE REFRACTION OF X-RAYS AND ITS EFFECT ON BRAGG PEAK POSITIONS

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ABSTRACT

The influence of surface roughness on the refraction effect for X-rays at grazing incidence was investigated. A set of brass samples with different surface roughnesses was used to measure the Bragg peak positions of \{111\} lattice plane in asymmetric $\Omega$-mode with a constant incidence angle $\alpha$. In a set of measurements with different incidence angles $\alpha$ the shift in Bragg peak positions was measured. The observed peak shift is due to the refraction effect at the sample surface and also due to residual stresses. Additional measurements in symmetrical $\theta/2\theta$-mode ($\Psi$-geometry) made it possible to separate both effects. The refraction effect at the sample surface was calculated and compared with theoretical values derived from Snell’s law of refraction. For a smooth surface theoretical and measured data are in very good agreement. But with increasing surface roughness the refraction effect decreases.

INTRODUCTION

The determination of the residual stress state in a component is of increasing importance for its evaluation. Several measuring methods for the assessment of two-axial stress states parallel to the surface are available [1,2]. In the last years methods for the determination of stress gradients close to the surface have been developed, one of those being the piecewise-polynomial-method [3]. This method is based on the fact that X-rays are weakened exponentially when passing the material and the measured Bragg peak positions are an average over the irradiated sample volume weighted by the beam intensity following

$$2\theta_{\psi,\phi} = \frac{\int_0^z 2\theta_{\psi,\phi}(z) \cdot e^{-z/z^*} dz}{\int_0^z e^{-z/z^*} dz}$$

(1)

$z$: depth normal to surface; $z^*$: X-ray penetration depth normal to surface

For solving the backtransformation of eq.1 it is necessary to measure Bragg peak positions in a wide range of directions $\Psi,\phi$ and to change the effective penetration depth of the X-rays by changing the incidence angle $\alpha$ [4]. If the stress gradient is steep, i.e. the stress state changes significantly in only a few $\mu$m and the penetration depth of the X-rays at common incidence angles is larger than the thickness of this surface layer, the incidence angle of the X-rays can be
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reduced to obtain relevant information about this surface layer. For this kind of evaluation it is necessary to measure the Bragg peak positions with an accuracy of 1/100° or even less. Therefore refraction of X-rays at the sample surface has to be taken into account when such small incidence angles have to be used as it has an influence on Bragg peak positions and, hence, on the evaluation of the stress state [5]. The used refraction correction for Bragg peak positions is based on Snell's law of refraction

\[ n_1 \cdot \cos(\alpha_1) = n_2 \cdot \cos(\alpha_2) \]  

(2)

It assumes that the surface is perfectly smooth. The influence of surface roughness on the refraction correction of the measured Bragg peak positions is not considered in the calculations. Only solutions for a refraction correction at smooth surfaces exist so far. A first model for an explanation of how surface roughness could influence the refraction effect is given by Ely et al. [6]. In this paper we present a method how to measure the refraction effect at surfaces of different roughnesses. Measurements with a set of samples of different surface roughnesses will show the influence of surface roughness on the refraction effect.

MATERIALS AND METHODS

A set of samples of Cu-Zn28 (α-brass), which have different surface roughnesses resulting from a gradated grinding and polishing treatment [Tab.1], was used for the measurements. In the polishing process the samples had been ground with different abrasive papers and colloidal SiO₂ suspensions to stepwise reduce surface roughness. At each stage one sample was taken out of the polishing process. Even though almost no difference is found by roughness measurements between samples D2 and D1, metallographic inspection yields definite differences of the surface topography of the polished sample D2 and the fine polished sample D1. The roughness was measured with a confocal, white light microscope according to DIN 4768. The samples were 11mm in diameter, and 5mm thick.

<table>
<thead>
<tr>
<th>Sample</th>
<th>D1</th>
<th>D2</th>
<th>D3</th>
<th>D4</th>
<th>D5</th>
<th>D7</th>
<th>D8</th>
<th>D9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra in grinding direction in [µm]</td>
<td>0.055</td>
<td>0.048</td>
<td>0.098</td>
<td>0.083</td>
<td>0.346</td>
<td>0.854</td>
<td>1.02</td>
<td>1.26</td>
</tr>
<tr>
<td>Ra in perpendicular direction in [µm]</td>
<td>0.050</td>
<td>0.052</td>
<td>0.146</td>
<td>0.163</td>
<td>0.442</td>
<td>0.952</td>
<td>1.93</td>
<td>2.92</td>
</tr>
</tbody>
</table>

Tab.1 Average roughness Ra of sample surface measured in grinding direction and perpendicular.

The measurements were made with synchrotron radiation at HASYLAB, Germany and LURE, France. The primary beam was parallel, and a soller slit was used in front of the detector. The wavelength of the X-rays was \( \lambda_1 = 0.154 \) nm and \( \lambda_2 = 0.180 \) nm. For these wavelengths the refraction index is \( n_1 = 0.9999764 \) for \( \lambda_1 \) and \( n_2 = 0.9999670 \) for \( \lambda_2 \). According to eq.3 the calculation of the refraction index were made with data of the Lawrence Berkeley National Laboratory (USA) database and with data of the National Institute of Standards and Technology (USA) FFAST-database, which led to the same values.
The samples D1, D2, D4 and D8 were measured with $\lambda_1 = 0.154$ nm at LURE (sample set A), a second sample set B with samples D1, D3, D5, D7 and D9 with $\lambda_2 = 0.180$ nm at HASYLAB. For all samples the Bragg peak of the \{111\} lattice plane was measured in an asymmetric setup, known as $\Omega$-geometry, with incidence angles of the primary beam between 0.4° and 21° for wavelength $\lambda_1 = 0.154$ nm and 25° for $\lambda_2 = 0.180$ nm respectively.

Comparing the measured peak shifts with theoretical peak shifts due to the refraction effect also the peak shift caused by residual stresses has to be taken into account. To separate both effects the peak positions of the \{111\} lattice plane have also been measured in a symmetric $\theta$-setup, known as $\Psi$-geometry, at the same $\phi$- and $\Psi$-angles as occur in the asymmetric setup (Fig.1). In the symmetric setup the incidence angles are larger than 19° for $\lambda_1$ and 22° for $\lambda_2$, respectively, and so the refraction effect can be neglected in the symmetric setup. Therefore the appearing peak shifts are only caused by the residual stress. So it is possible to separate the influence of residual stress and the effect of refraction on peak positions in the asymmetric setup. For the calculation of the peak shift due to refraction the results of the $\alpha = 21°$ ($\lambda_1$), $\alpha = 25°$ ($\lambda_2$) measurements were set as reference point, which is not the stress free $2\theta_0$ value.

To evaluate the Bragg peak positions Pearson-VII functions were fitted to the measured data. The surface normal was adjusted to be parallel to the $\phi$-axes of the goniometer within 0.05°.

As in principle it is not possible to rule out the effect of steep residual stress gradients on Bragg peak positions with this kind of experiment, and because this can result in similar peak shifts as the refraction effect, the measurements were done with two different wavelengths. As the attenuation length depends on the X-rays wavelength the possible peak shifts due to residual

\[
n = 1 - \frac{r_0}{2\pi} \sum_j a_j f_j^2
\]

$r_0$: classical electron radius; $a_j$: atom number density element $j$; $f_j$: atomic form factor element $j$. 

Fig.1 Experimental setup. Comparing $\Omega$–geometry and $\Psi$–geometry. The diffraction cone is shown as a circle only. Direction of measurement $\Psi,\phi$ (central axes of the diffraction cone, normal to lattice) is unchanged between $\Omega$–geometry and $\Psi$–geometry. The intersections of diffractometer plane (white) and diffraction cone are incoming primary beam and outgoing diffracted beam. Incidence angle $\alpha$ is smaller in $\Omega$-geometry than in $\Psi$-geometry.
stress gradients would change as well. A correspondence of refraction effect and possible peak shifts due to residual stress gradients for both wavelengths is unlikely. The preparation of the samples was made slowly and carefully to reduce residual stresses and to do not induce steep residual stress gradients.

RESULTS

In figure 2 the theoretical peak shift for a perfectly smooth surface calculated with Snell’s law (Eq. 2) is compared with the measured peak shifts of sample set A. For the fine polished sample D1 the difference between measurement and theory is smaller than 0.01° down to incidence angles of 0.7°. Close to the critical angle of total reflection $\alpha_{\text{tot}} = 0.3936°$ the difference is about 0.1°. The polished sample D2 shows a somewhat smaller Bragg peak shift than sample D1. With increasing surface roughness the effect of refraction is obviously decreasing. At sample D8 maximum peak shift due to refraction measured is 0.03° at an incidence angle of 0.4° and therefore the difference between theory and measurement is 0.3° as the theoretical peak shift for a perfectly smooth surface at an incidence angle of 0.4° is 0.33°.

![Fig. 2 Refraction effect at the sample surface dependent on the incidence angle $\alpha$ compared with theoretical values for X-rays with a wavelength $\lambda_1 = 0.154$ nm. Samples with different roughnesses were measured perpendicular to the grinding direction.](image)

In figure 3 the results of the measurements of sample set B are compared with the theoretical peak shifts. For the fine polished sample D1 the difference between measurement and theory is again smaller than 0.01° down to incidence angles of 0.8°. Close to the critical angle of total reflection $\alpha_{\text{tot}} = 0.466°$ the difference is about 0.06°. With increasing surface roughness the effect of refraction is obviously decreasing.
Fig. 3 Refraction effect at the sample surface dependent on the incidence angle $\alpha$ compared with theoretical values for X-rays with a wavelength $\lambda_1 = 0.180$ nm. Samples with different roughnesses were measured perpendicular to the grinding direction.

The very good correspondence of the peak shifts measured with the polished sample D1 and the theoretical values calculated with Snell’s law for both wavelengths make it unlikely that the reason for the peak shift is due to residual stress gradients and not due to refraction of the X-rays at the sample surface.

The observed differences between theory and measurement close to the critical angle of total reflection for the fine polished sample D1 are probably due to small errors in sample alignment, zero-angle displacement of the instrument, which is smaller than 0.01°, as well as surface roughness and primary beam divergence.

Additional stress state measurements with the $\sin^2\Psi$-method at the {311} lattice plane in symmetric setup with $\Psi$-angles in a range of 0° up to ±70° yield data between -90 MPa and -200 MPa.

**CONCLUSION**

The investigation shows that for the used X-rays with wavelengths of $\lambda_1 = 0.154$ nm and $\lambda_2 = 0.180$ nm, and therefore a refraction index of $n_1 = 0.9999764$ for $\lambda_1$ and $n_2 = 0.9999670$ for $\lambda_2$ for brass samples, Snell’s law of refraction describes the refraction effect with good accuracy at least down to incidence angles of 0.8° for smooth surfaces. The effect of refraction at the sample surface and therefore the shift in Bragg peak positions depends strongly on surface roughness and it is decreasing with increasing roughness of the surface. The maximum difference in Bragg
peak positions due to the influence of surface roughness occurs close to the critical angle of total reflection and is about 0.25°. This shows clearly that the influence of surface roughness on the refraction effect has to be taken into account when corrections of measured Bragg peak positions are made for the use with stress state determinations.

PROSPECTS

So far it is not possible to calculate the refraction effect of rough surfaces. In future work a model to calculate the refraction correction depending on suitable parameters describing the surface has to be developed. Investigations on special surface topographies have already started.

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REFERENCES