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Application of multireflection grazing incidence method for stress measurements in polished Al–Mg alloy and CrN coating

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ABSTRACT

Multi-reflection grazing incidence geometry, referred to as MGIXD, characterized by a small and constant incidence angle, was applied to measure low surface stresses in very thin layers of Al–Mg alloy and CrN coating. These two materials were selected in order to deal with the low and high levels of residual stress, respectively. The influence of different mechanical treatments on residual stresses was studied for Al–Mg samples. It was found that both rolling and mechanical polishing influence the distribution and amplitude of residual stress in surface layers. In the case of CrN coating, a very high compressive stress was generated during the deposition process. The stress distributions determined by the MGIXD method is in good agreement with the classic $\sin^2\psi$ technique results for all studied samples. In performing stress measurements for a powder sample, it was found that the application of the Göbel mirror in the incident beam strongly reduces statistical and misalignment errors. Additionally, the root mean square values of the third order lattice strain within diffracting grains were determined.

1. Introduction

Both the magnitude and the spatial distribution of residual stress play key roles in the behaviour of materials subjected either to heat treatment or plastic deformation. The residual stress influences the fatigue response of solids, stress corrosion, and is important for the manufacturing process of all products. Depending on its direction and magnitude, the residual stress superimposed with an external load can be destructive or beneficial to a component. For example, the mechanical strength of a surface layer is improved by machining, which creates a compressive residual stress. The same type of stress increases fracture resistance and minimises spalling of the coating. The opposite type of stress, tensile residual stress, can accelerate the growth of the crack and cause the coating's destruction, when external loads are superimposed.

Diffraction methods are commonly used for determining lattice elastic deformation and distortion (i.e., of macrostrains and microstrains) from the displacement and broadening of the diffraction peak [1–4]. The stress present in the near surface volume can be measured using the standard X-ray $\sin^2\psi$ method. However, this method is not advisable for the analysis of the in-depth stress gradient, because the penetration depth of X-ray radiation varies significantly during measurement. Therefore, the grazing incidence X-ray diffraction geometry (GIXD) was applied to measure residual stresses in thin surface layers [5–12]. In this work one of the version of the GIXD method referred to as the multi-reflection grazing incidence X-ray diffraction (MGIXD, [10–12]) was used in the laboratory diffractometer equipped with the Göbel mirror in the incident beam optics. Using this method, it is possible to perform a non-destructive stress analysis for different (and well defined) volumes below the surface of the sample and the stress can be measured at very shallow depths of a few micrometers. The main advantage of this method is that apart from the residual stress the root mean square of the lattice strain, caused by defects within polycrystalline material, can also be determined using Williamson–Hall method [13,14].

The disadvantage of the MGIXD method was its low accuracy in stress determination, when a classic line focus and a parallel plate collimator in the reflected beam optics were used in the measurement. For example the systematic error of about 50 MPa for Fe powder was reported in [10] (this value corresponds to 20 MPa if the Al elastic constants are used in the analysis). This is why our previous measurements were performed only for samples with high surface stresses [10,11]. Recently, the accuracy was considerably

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improved using the Göbel mirror to collimate an incident beam. A perfectly collimated parallel beam radically decreases the uncertainty of the determined peak position and, consequently, one reaches the accuracy of a few MPa for an Al powder sample. Due to very good precision and high beam intensity, the new experimental setup can be applied to measure low surface stresses in thin layers.

The reproducibility of the experimental setup with the Göbel mirror was already tested repeating measurements for different powder specimens [12]. It was found that the difference between the stresses measured using the MGIXD method was smaller than 15 MPa for the Al powder. This value can be considered as the uncertainty of measurements arising from misalignments of the diffractometer and the sample position. From the measurements on the powder samples and also from theoretical analysis it was found that the important source of systematic error is the shift of 2θ zero value. The misalignment of the diffractometer equal to $\Delta 2\theta = 0.01^\circ$ leads to the fictitious stress of about 10–15 MPa determined for the Al stress-free powder. This error can be minimised by the careful alignment of the diffractometer or the results obtained for the studied sample can be corrected by using the powder diffraction data.

The application of the MGIXD method is especially recommended for determining of the in-depth stress gradients close to the surface, which can significantly change the corrosion resistance of the material [15]. This methodology can be also used to determine the stress distribution in thin films. The latter stress influences the fracture and spalling resistance of the coating.

In the present work we demonstrate the possibilities of the MGIXD method on two examples of surface layers. The stress distribution is determined in a mechanically polished Al–Mg alloy (isotropic elastic properties) and a CrN coating (anisotropic single crystal elastic constants).

2. Experimental principles

2.1. Stress measurements using grazing incidence geometry

The standard method for stress determination (called $\sin^2\psi$) is based on the measurement of interplanar spacing for various directions of the scattering vector [1–3]. These directions are defined by ϕ and ψ angles (Fig. 1a). Using diffraction, the mean interplanar spacing $\langle d(\phi,\psi)\rangle_{\{hkl\}}$, averaged over reflecting crystallites, is measured. As it has been mentioned, the orientation of the scattering vector varies in this method, however the reflecting plane index $\{hkl\}$ is kept constant during strain measurements. Consequently, different penetration depths of X-ray radiation appear for different tilt angles ψ in standard geometry.

The multi-reflection grazing incidence geometry (MGIXD) is a non destructive diffraction method, which can be used to determine lattice elastic distortion in the near surface layers of a material. The characteristic feature of this method is a small and constant incidence angle [10–12]. Consequently, the penetration depth of X-ray radiation is well defined and does not change during the experiment. Measurements are performed for different sets of $\{hkl\}$ planes using the appropriate values of $2\theta_{\{hkl\}}$ scattering angles. The incidence angle α is fixed during measurement, while the orientation of the scattering vector is characterized by the angle $\psi_{\{hkl\}}$. The $\psi_{\{hkl\}}$ angle depends on hkl reflection ($2\theta_{\{hkl\}}$ angle) and on a constant incidence angle α (Fig. 1b):

$$\psi_{\{hkl\}} = \theta_{\{hkl\}} - \alpha \tag{1}$$

Consequently, possible values of $\psi_{\{hkl\}}$ angles are limited to the number of hkl reflections used in the experiment.

In the case of the MGIXD method the measurements of interplanar spacings $\langle d(\phi,\psi)\rangle_{\{hkl\}}$ are performed in the near surface volume, which is limited by radiation absorption. To define this volume, the path of the X-ray beam through the sample must be considered (Fig. 1b). The measured average interplanar spacings $\langle d(\phi,\psi)\rangle_{\{hkl\}}$ are equal to:

$$< d(\phi,\psi)>_{\{hkl\}} = \frac{\int_0^t d(\phi,\psi,z)_{\{hkl\}} \exp[-\mu l(z)]dz}{\int_0^t \exp[-\mu l(z)]dz} \quad \text{and}$$

$$l(z) = z\left(\frac{1}{\sin\alpha} + \frac{1}{\sin(2\theta_{\{hkl\}} - \alpha)}\right) \tag{2}$$

where μ is the linear coefficient of absorption and the above formula can be used if $\alpha \gg \alpha_{cr}$ (α_{cr} is the critical angle for the total external reflection [2]), z is a depth below the surface and the average is calculated over the volume of all reflecting grains in the beam path $l(z) = a(z) + b(z)$ (see Fig. 1b), i.e. from the surface ($z = 0$) to the thickness of the coating ($z = t$). If the stresses are measured in a monolithic material or in a thick coating (comparing to penetration of X-ray radiation) we assume that $t \to \infty$.

Eq. (2) is usually expressed in the equivalent form:

$$< d(\phi,\psi)>_{\{hkl\}} = \frac{\int_0^t d(\phi,\psi,z)_{\{hkl\}} \exp(-z/\tau)dz}{\int_0^t \exp(-z/\tau)dz} \quad \text{and}$$

$$\tau = \left(\frac{\mu}{\sin\alpha} + \frac{\mu}{\sin(2\theta_{\{hkl\}} - \alpha)}\right)^{-1} \tag{3}$$

where "penetration depth" τ is defined as the distance from the sample surface, for which $\left(1 - 1/e\right) = 0.63$ part of total intensity of the incident beam is absorbed.

The above average corresponds to the so called "information" or "effective" depth $\bar{z}$, which can be understood as the mean value of z-depth weighted by an attenuation factor [8]:

$$\bar{z} = \frac{\int_0^t z\exp(-z/\tau)dz}{\int_0^t \exp(-z/\tau)dz} = \begin{cases} \tau - \dfrac{t\exp(-t/\tau)}{1-\exp(-t/\tau)} & \text{for limited } t \\ \tau & \text{for } t \to \infty \end{cases} \tag{4}$$

When small α angles are used (i.e., for long incident beam paths, i.e. $a(z) \gg b(z)$ in Fig. 1b) the penetration depth can be approximated by the expression $\tau = \sin\alpha/\mu$ (see Eq. (3)). Consequently both the penetration depth τ (Eq. (3)) and the information depth $\bar{z}$ (Eq. (4)) do not depend on 2θ and related ψ (Eq. (1)) angles. The information depths $\bar{z}$ vs. $\sin^2\psi$ calculated for the MGIXD and the standard method (Ω mode [1,2]) are shown in Fig. 2, where the materials studied in this work are considered. It is visible that only in the case of the MGIXD method, the information depth is almost constant for a fixed small α angle and for a large range of ψ angle. Moreover, it is shown that stress can be measured for different layers under the sample surface by setting different α angle values (Fig. 2).

In the MGIXD method, the $\langle d(\phi,\psi)\rangle_{\{hkl\}}$ interplanar spacings are measured in the directions defined by ϕ and ψ angles for different hkl reflections. Hence, the orientations of the scattering vector with respect to the sample are strictly determined by Eq. (1), and the