Determination of residual stresses and dislocations density in surface layers using multireflection grazing incidence diffraction

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The geometry based on the grazing incidence X-ray diffraction was applied to measure gradient of residual stresses in surface layers. In this case a non-symmetrical diffraction with small incident angle (α) is used and the penetration dept (τ) of X-ray radiation (limited by absorption) depends mostly on the long path of incident beam (Fig. 1). In this work we have used multireflection grazing incident X-ray diffraction geometry (called MGIXD) [1,2] in which for a given penetration depth τ, the interplanar spacings are determined using: constant wavelength, small and constant incidence angle α and different *hkl* reflections (ψ varies with 2θ angle, i.e. $\psi = \theta - \alpha$; see Fig.1).

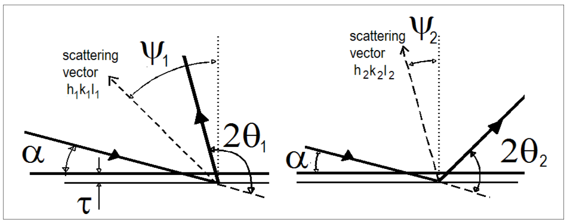

Figure 1. Geometry used in MGIXD method. The incidence angle α is fixed during measurement while the orientation of the scattering vector is characterised by the angle $\psi = \theta - \alpha$.

In our project measurements were performed at G3 beamline at the DORIS III storage ring using Soller collimator and scintillation detector. For three different wavelengths (λ_1=1.2527 Å, λ_2=1.5419 Å and λ_3=1.7512 Å) we have calculated three corresponding incidence angles (α) for which the penetration depth is the same. The important question verifying our methodology is if the same stresses will be determined for such combination of wavelengths and incident angles.

First studied sample was Ti, slightly polished using paper grade 2000. In this case strong asymmetry of diffraction peak suggest that two irradiated regions of the sample have different microstructure, i.e. layer of about 0.5-1 μm which has been severely plastically deformed (region of high density of dislocations) and the base material, under this layer, having much lower density of dislocations (smaller plastic deformation). Indeed the diffraction peaks can be easily separated into two pseudo-Voigt functions having different integral widths and position (Fig. 2a). It should be underlined that in this case the separated peaks represent different regions in the sample and they can be treated independently. The broad peak (representing hard material in the layer) shifts relatively to the narrow one (coming from soft base material), i.e. when ψ angle (Fig. 1) increases the broad peak shifts from the left to the right site of the narrow one, as shown in Fig.2a. It was also found that the relative contribution of the narrow peak increases for deeper penetration depths when larger volume under the layer is irradiated. The residual stresses were determined using measured lattice parameters $< a(\phi,\psi) >_{\{hkl\}}$ vs. $\sin^2\psi$ graphs (Fig. 2b) [1,2]. In-depth profiles of the measured stresses are similar for the three different wavelengths used in experiment (Fig. 3a). High compressive stress of about 500-700 MPa has been found in the layer (irradiated for all wavelengths and geometrical conditions), while in the base material a small tensile stress increases with penetration depth within the range of about 0-120 MPa. The stress determined by classical X-rays (PANanalitical instrument with Cu radiation, $\lambda_{k\alpha 1}$=1.54056 Å and Göbel mirror) is equal to the average from the values measured in the layer and base material, weighted by the intensity of reflected beam. For smaller penetration depth contribution of the layer causes relatively higher value of the measured stress, while for deeper penetration stress value approaches to this measured in the base material. It should be also noted that using synchrotron radiation (λ_1=1.2527 Å) the stress was determined for much deeper regions in comparison with classical X-ray results (in this case stresses in the layer cannot be determined because of very low contribution of the broad peaks).

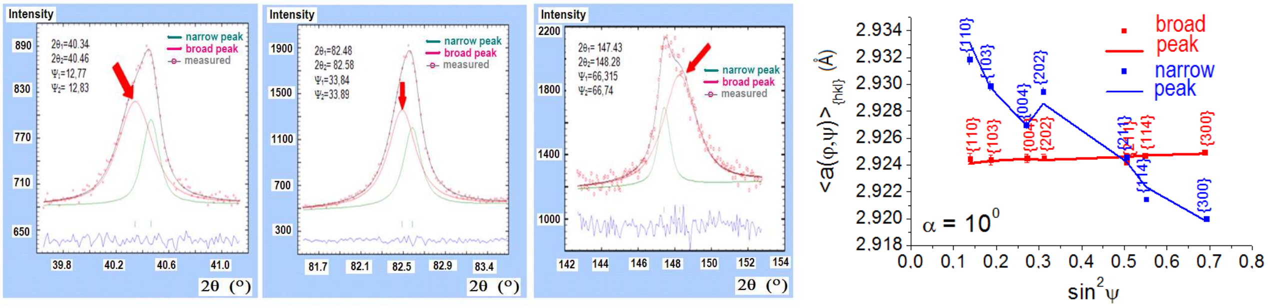

Figure 2. Separation of diffraction peak into two components (a) for Ti sample and $<a(\phi,\psi)>_{\{hkl\}}$ vs. $\sin^2\psi$ graphs obtained from the positions of narrow and broad peaks (b).

The second studied sample was Al 2017 alloy mechanically polished using paper grade 2000. The measured diffraction peaks were slightly asymmetrical due to stress gradient but their separation into different components was not possible. It was found that for three different wavelengths the same in-depth profiles of stresses were obtained. What is more the results perfectly agree with those obtained previously using classical X-ray diffraction ($\lambda_{k\alpha 1}$=1.54056Å). Synchrotron radiation ($\lambda_{k\alpha 1}$=1.2527) allows us to measure the stress profile for deeper regions in comparison with classical X-rays ($\lambda_{k\alpha 1}$=1.54056Å).

Finally the root mean square strains $\sqrt{<\varepsilon^2>}$ corresponding to density of dislocations were calculated using Williamson-Hall method independently for two separated peaks for Ti sample. As expected higher value of $\sqrt{<\varepsilon^2>} = 0.2\% - 0.3\%$ was obtained from the severely deformed layer in comparison with the base material ($\sqrt{<\varepsilon^2>} = 0.05\% - 0.1\%$). Almost the same value of $\sqrt{<\varepsilon^2>} = 0.12\% - 0.17\%$ was found for different depths in the Al sample.

It can be concluded that all aims of the experiment were gained and the MGIDX method was successfully verified.

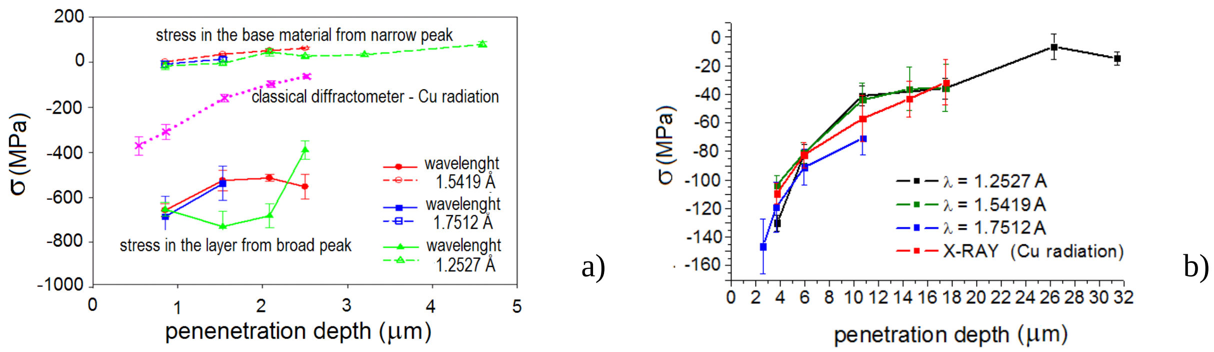

Fig. 2. In-depth profiles of stresses in mechanically polished Ti (a) and Al (b) samples.

References

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