

1.12.23. OPTIMIZED STRAIN/STRESS DIFFRACTOMETER EQUIPPED WITH FOCUSING BENT PERFECT CRYSTAL MONOCHROMATOR WOULD PERMIT SOME KINETIC PROCESSES IN POLYCRYSTALLINE MATERIALS

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New variant of neutron diffractometer for nondestructive analysis of strain/stress in polycrystalline materials has been installed at the thermal neutron beam ST-1 of HANARO reactor and introduced in full operation. After construction of the shielding for cold neutron source in the HANARO reactor hall, the space for the previous strain/stress diffractometer performance has been significantly reduced and the maximum possible monoch-

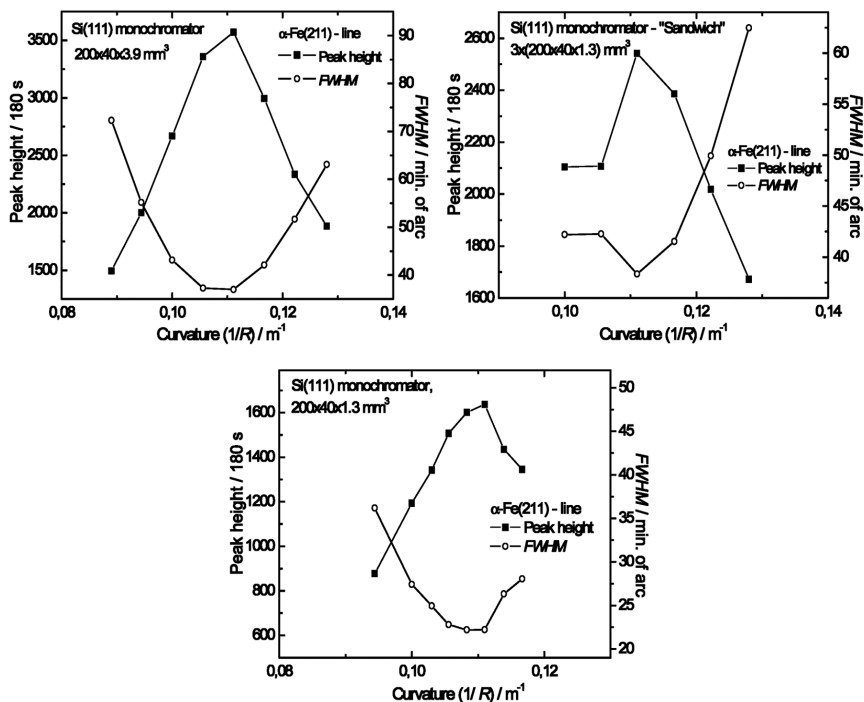


Fig. 1. The resolution and luminosity characteristics of the strain/stress diffractometer performance for Si(111) crystal slabs of different thickness of 3.9 mm, 3 × 1.3 mm (sandwich) and 1.3 mm

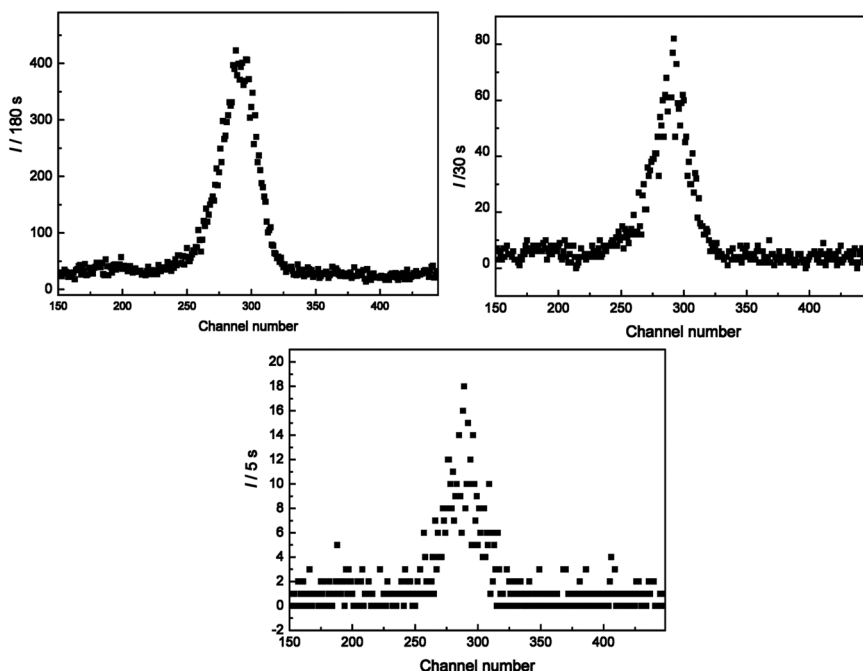


Fig. 2. Diffraction profiles of the sample of α -Fe(112) pin of 1 mm diameter and 40 mm height for different measurement times

romator take-off angle, $2\theta_M$, available for strain/stress measurements of bulky samples has decreased from 60° to $\sim 30^\circ$. The diffraction geometry with such a small monochromator take-off angle is far from conventionally used values and namely, it is not used in powder diffraction requiring a high resolution. It follows from the formula $(\Delta\lambda/\lambda)_M = \Delta\theta \cot \theta_M$ that even though a larger neutron wavelength spread $\Delta\lambda_M$ is impinging on the polycrystalline sample, the angular resolution on the powder diffraction lines considerably deteriorates which is not acceptable. It is namely at larger scattering angles ($2\theta_S \sim 90^\circ$), typical for strain/stress measurements. However, by using Bragg diffraction optics with a focusing bent perfect crystal monochromator, the problem of resolution can be satisfactorily overcome. Moreover, one can profit from the larger neutron wavelength spread $\Delta\lambda_M$ resulting in very good luminosity of the diffractometer.

After optimization of the diffractometer design, finally, $2\theta_M$ up to 45° has become possible. However, for $2\theta_M \leq 45^\circ$ only the Si (111) reflecting planes at $2\theta_M \sim 30^\circ$ provide neutrons with the wavelength of $\lambda = 1.5 - 1.7 \text{ \AA}$, suitable for strain measurements in the most industrial materials. By using an α -Fe(112) pin of 2 mm diameter and 40 mm height which was mounted at the sample position and imitated the measurement gauge volume, bent Si(111) crystals with different cutting angles (different diffraction geometries) were tested and optimized for $2\theta_M = 30^\circ$. Experiments showed that Si(111) crystal

in symmetric diffraction geometry would be the best choice. This conclusion was also confirmed by Monte Carlo simulations. However, the angular resolution of the instrument is dependent on the thickness of the bent monochromator crystal. Therefore, several bent crystal slabs of different thickness (3.9 mm, 3×1.3 mm (sandwich) and 1.3 mm) were tested. Figure 1 displays the resolution and luminosity characteristics obtained during the optimization of the crystal curvature for three monochromator alternatives. Thanks to the employment of the Bragg diffraction optics minimum resolution takes place at the maximum height of the diffraction profile. When comparing the characteristics related to the thicknesses 3.9 mm and 1.3 mm, it can be seen that the resolution *FWHM* (measured as $\Delta(2\theta)$ at the position sensitive detector) changed from 37' to 23' and the peak height decreased only by a factor of 2, respectively. Considering that the accuracy of the peak position is proportional to the intensity and inversely proportional to the square of the peak width, the characteristics of the instrument with Si(111) crystals in symmetric diffraction geometry are comparable with the diffractometer performance before the reinstallation. However, very good luminosity of the new instrument performance reveals also a possibility of studies of some kinetic processes related to macro- and/or micro-strain/stress distribution. Namely, so called *in-situ* experiments with samples subjected to external termo-mechanical load can be attractive. It can be seen from fig. 2 that even at the measurement time of 5 s the peak position can be determined with a relative error of about 10^{-4} which is sufficient in most cases of residual strain/stress measurements.

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