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# Enhancement of energy dispersive residual stress analysis by consideration of detector electronic effects

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## Abstract

The effects of the germanium detector electronics on diffraction line patterns is investigated. It is shown that not only the detector resolution and the throughput but also the energy stability depend on both the specific detector settings and the dead time. For a moderate resolution versus throughput setting a correction function is proposed and applied to the near-surface residual stress analysis of three samples with considerably different stress states. It is demonstrated that without the correction function ghost stresses up to hundreds of MPa in the near-surface region are obtained. The correction procedure is verified by conventional X-ray measurements. In conclusion, the authors strongly suggest quantifying the electronic shifts of any individual detector systems prior to the analysis of residual stresses.

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## 1. Introduction

Regarding the mode of data acquisition, diffraction experiments can be classified into angle-dispersive (AD) and energy-dispersive (ED) methods.

In AD diffraction, the X-rays are emitted as characteristic emission lines of X-ray tubes or from synchrotron sources using double crystal monochromators which absorb all but the energy of interest. The diffraction information is recorded by zero-, one- or two-dimensional detectors. Zero-dimensional detectors, e.g. scintillation counters, require the angular scan of the diffraction angle $2\theta$. By means of one- and two-dimensional (position sensitive) detectors, parts of the diffraction spectrum and of the Debye–Scherrer ring, respectively, are recorded simultaneously.

In ED diffraction, the bremsstrahlung generated in X-ray tubes or the white beam as originally obtained from synchrotron photon sources is used. The diffracted spectrum is recorded by means of solid state semiconducting detectors. In X-ray residual stress/strain analysis (XSA), ED diffraction provides significant advantages compared to AD diffraction. Complete spectra are obtained at a constant diffraction angle within a comparatively short time. As the diffracted beam is collimated by a relatively simple double slit system, scanning experiments in transmission [1–3] enable high spatial resolution with considerably lower effort than in AD diffraction where spiral or conical slits are used in combination with two-dimensional detectors [4,5].

In reflection geometry, the multitude of diffraction lines recorded simultaneously in ED diffraction, supply additional depth information [6,7]. Each spectrum recorded at a given inclination angle also contains information on phases and texture.

However, the data analysis is complicated. A multitude of correction factors have to be taken into account such as source specification, geometrical considerations and

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sample properties. The most sensitive parameter in the evaluation of the line positions $E(hkl)$ is the stability of detector electronics. In order to detect strains within a range of $\epsilon = 10^{-4}$, the determination of the diffraction line positions in a precision of $\Delta E/E = 10^{-4}$ is required, i.e. 2–10 eV for diffraction lines positioned from 10 to 100 keV, respectively. At sufficient counting times, such a precision is easily reached by fitting the individual profiles and calculating the mass of centre. It should be stressed that the detector resolution determines the separability of neighbouring peaks but does not necessarily influence the precision in the determination of the line position. However, in XSA the diffraction intensity varies significantly with different inclination angles or when scanning samples of different thicknesses and composition. Thus, a further demand on the detector system is the energy stability at different count rates, i.e. detector dead times.

In the energy range from tens to hundreds of keV, the detectors used are low energy germanium (LEGe). The analog signal generated by the photons in the semiconductor is converted into a digital signal before storage in a multi channel analyzer (MCA). The data are processed either before digitalization by conventional electronics or after by recently introduced digital signal processors (DSP).

Unfortunately, Ge detector systems are almost only specified in the MeV range as they are designed for high-energy-γ-spectroscopy in nuclear physics applications. Here, the focus is on detector efficiency, resolution and throughput [8,9]. Little can be found in literature on the limited energy stability [10,11]. Few investigations exist concerning the demands of XSA performed in the keV range. Some research has been performed on the energy calibration in order to find a fast and precise way for the channel-to-energy conversion at constant detector settings and dead times [12,13]. The relation was found to be described by a polynomial of 3rd order in a precision of approximately 10 eV which is sufficient in XSA since small relative energy position shifts (but not absolute energy offsets) are of interest.

Early works on the analytic fitting of energy dispersive diffraction lines, however, show that the profile obtained is the sum of different electronic effects which depend on the detector settings and count rate [14–16]. If the signal is digitally processed, the profile also depends on internal algorithms (filters), the details and characteristics of which are inaccessible to the user.

Hence, great concerns exist on the influence of the detector system settings and the influence of the dead time on the line positions $E(hkl)$. As these factors affect the residual stress measurement more than all other known correction factors, the goals of the paper are (a) the investigation of the influence of detector settings and the dead time on the diffraction line positions, (b) the introduction of a calibration function and (c) the demonstration of the achieved improvements on the RSA.

## 2. Experimental setup

All experiments were carried out at the Berlin synchrotron facility BESSY at the high energy white beamline EDDI (beamline for energy dispersive diffraction) being operated by the Hahn-Meitner-Institute.

The source is a 7 T multipole wiggler providing energies up to ≈130 keV with reasonable intensity for diffraction experiments. All major components are shown in Fig. 1. The beam is collimated by a mask at 19 m and a pair of slits S1/S2 at 27 m and 30 m from the source, respectively, before it impinges the sample which is mounted on a five-axis diffractometer allowing a sample rotation in $\omega$, $\varphi$ and $\psi$ as well as an $x$-, $y$- and $z$-translation. At a fixed scattering angle $2\theta$ the diffracted beam travels through a double slit system S3/S4. Close behind S4, the diffracted beam is monitored by a LEGe detector. The experimental hutch is fully air conditioned keeping the temperature

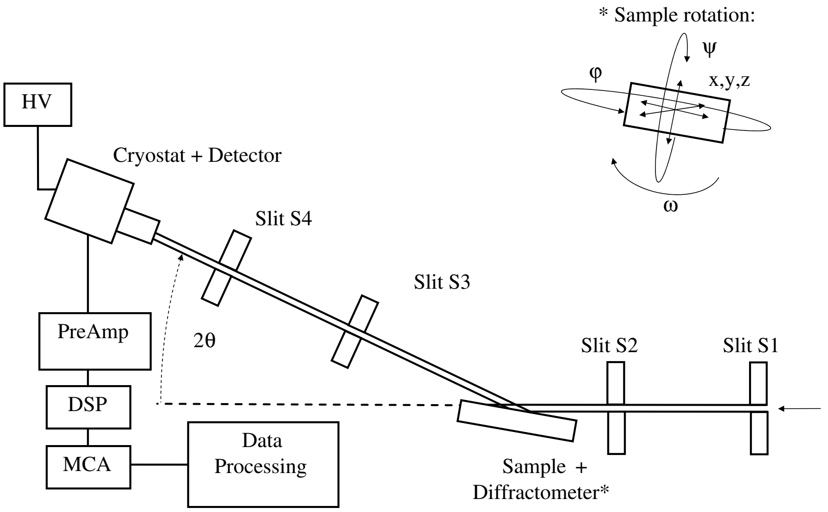

Fig. 1. Setup of the energy dispersive diffraction beamline EDDI at BESSY.