Nuclear Inst. and Methods in Physics Research, A 877 (2018) 56–64

Contents lists available at ScienceDirect

Nuclear Inst. and Methods in Physics Research, A

journal homepage: www.elsevier.com/locate/nima

Residual stress analysis of energy-dispersive diffraction data using a two-detector setup: Part II — Experimental implementation

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ARTICLE INFO

Keywords:
X-ray residual stress analysis
Energy-dispersive diffraction
Two-detector setup

ABSTRACT

Based on the theoretical concept of a two-detector setup for the energy-dispersive diffraction method (Apel et al., 2017), the experimental implementation of the proposed measurement concepts is demonstrated. The measurement configurations as well as the formalism for data evaluation introduced in the first part of this series are applied to the analysis of in- and out-of-plane near surface residual stress gradients in mechanically surface treated steel. Diffraction experiments carried out with the Bremsstrahlung of a conventional X-ray tube are shown to yield results with a quality comparable to measurements conducted at a synchrotron beamline. Furthermore, it is demonstrated that the simultaneous measurement of the positive and the negative ψ-branch can partly compensate for the much higher counting times due to the lower photon flux of the X-ray tube. The results are compared and assessed with those obtained by means of synchrotron radiation (beamline EDDI@BESSY II) and the layer removal method (LRM).

1. Introduction

Although the first diffraction experiment using a white X-ray beam was performed more than 100 years ago by the pioneers of X-ray diffraction, Laue, Friedrich and Kipping, it took more than half a century before the white beam diffraction technique was applied to polycrystalline materials [1,2]. The reason for this delay is the lack of appropriate solid state detector systems with efficient energy resolution, which were not available in the early days of X-ray diffraction. Thenceforward the potential of the energy-dispersive (ED) diffraction method gained more and more recognition as shown by the great variety of applications in materials research compiled in the bibliography of the first ten years of ED diffraction by Lähteenmäki [3]. The main advantage of ED method compared to angle-dispersive (AD) diffraction consists in the detection of complete diffraction spectra under fixed geometrical conditions. With the increased availability of modern 3rd generation high-brilliance synchrotron facilities, which provide much better conditions for advanced ED diffraction experiments than available in the lab, numerous efforts have been made regarding the development of new and enhanced methods for X-ray stress analysis (XSA), which exploit the properties of white high energy synchrotron radiation [4–10]. However, the increased number of possible applications for the ED method regarding the investigation of materials properties is accompanied by an increased demand of beamtime at the respective synchrotron facilities. Therefore, it is often challenging for laboratory researchers and manufacturers to gain access to high-energy synchrotron X-rays. Although conventional X-ray diffractometers are easier to access, the rather small information depth which can be realised with characteristic X-rays up to about 17 keV (MoKα) in engineering materials limits its applicability in the case of depth resolved residual stress analysis. In [11] it was shown that a diffractometer designed for AD diffraction can be modified with comparatively little effort for residual stress analysis in the ED mode of diffraction allowing to make use of the advantages of the ED method on a laboratory scale. Due to the (in principle) 'unlimited access to beamtime' the significantly lower photon flux of the X-ray tube can be compensated by longer counting times. The two-detector setup introduced in the first part of this series aims for offering new measurement configurations in order to further decrease overall measurement time on conventional diffractometers operated in the ED mode of diffraction. In this paper we discuss the experimental implementation of the new two-detector setup. The feasibility of performing ED-XSA experiments applying the new setup is demonstrated in various investigations of an austenitic steel sample showing an inhomogeneous residual stress state.

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http://dx.doi.org/10.1016/j.nima.2017.09.006
Received 28 April 2017; Received in revised form 11 July 2017; Accepted 1 September 2017
Available online 18 September 2017
0168-9002/

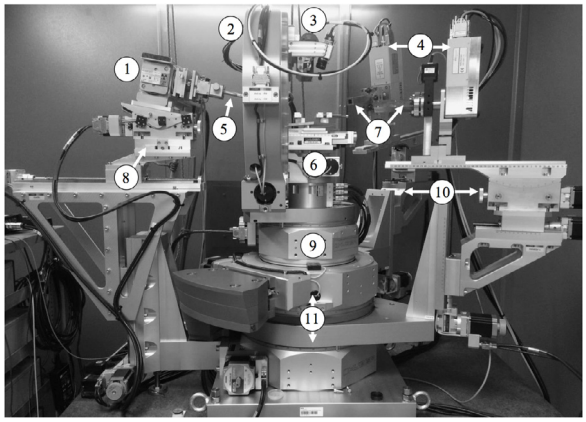

Fig. 1. The energy-dispersive diffractometer LEDDI equipped with the two-detector setup introduced in (Apel et al., 2017) [12]. The numbered components are: 1 –tungsten X-ray tube, 2 –X-cradle, 3 –laser and CCD camera system, 4 –Si(Li)-detectors, 5 –collimator/monocapillary, 6 –sample positioning unit with $x - y - z$ translation stage and Φ-rotation table, 7 –0. 15°soller slits, 8 –positioning unit for the X-ray source, 9 –Ω-table for rotation of the X-cradle, 10 –positioning units for vertical detector alignment, 11 –positioning units for horizontal detector alignment. See text for further details.

2. Experimental

2.1. The energy-dispersive two-detector diffraction setup

The diffractometer LEDDI (Laboratory Energy Dispersive DIffraction) was planned and developed in a co-operation between the two working groups in Kassel and Berlin and Huber Diffraktionstechnik GmbH & Co. KG (see Fig. 1). It is operated in the energy-dispersive mode of diffraction using the Bremsstrahlung generated by a long fine focus tungsten X-ray tube (voltage 60 keV). For data acquisition two Peltier-cooled energy-dispersive Si(Li)-detectors from Baltic Scientific Instruments (BSI) are used. The intrinsic detector resolution as specified by BSI is about 170 eV at 5.9 keV and 420 eV at 59.6 keV. The primary beam size can be defined either by collimators of different diameter or by a 300 μm monocapillary. Both detectors are equipped with 0. 15°soller slits in order to minimise the equatorial divergence of the diffracted beam.

Since the detector movement in the horizontal plane leads to an inclination of the diffraction plane spanned by the primary and the diffracted beam and the scattering vector, the soller slits are rotatable in order to adjust their plates exactly perpendicular to the diffraction plane. In this way the optimum diffraction conditions are defined by the maximum peak intensity and minimum of the integral breadth of (see Fig. 2). The alignment of the source and both detectors regarding the sample occurs through a combination of rotary and translatory movements. The 6-axes sample positioning system consists of a closed X-cradle with an integrated x-y-z-translation stage and a Φ-rotation table below the cradle which fulfils two functions. On the one hand, considering a symmetrical diffraction geometry in the vertical plane realised with detector D1, it allows for a direct sample rotation around the scattering vector. On the other hand, taking additionally into account the inclined diffraction geometry realised with the second detector D2, the rotation around the Φ- axis allows to adjust the cradle in such a way that the X-circle coincides with the plane spanned by the two scattering vectors. A laser and CCD camera system are pivoted opposite the sample thus allowing for its exact adjustment in any orientation. The diffractometer and the experiment are controlled using the software package SPEC [13] from Certified Scientific Software. Data pre- and post-processing is done using a software package developed by the authors.

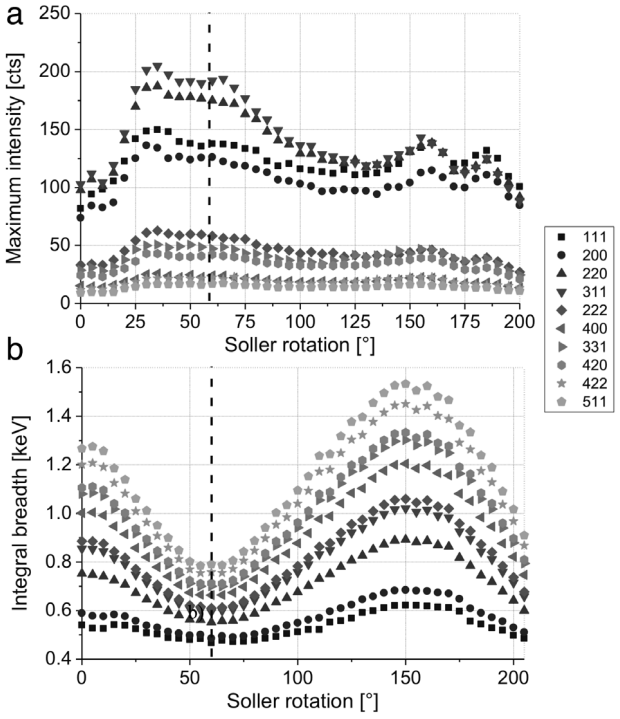

Fig. 2. (a) Maximum intensity and (b) integral breadth from the diffraction lines of a gold powder spectrum as a function of the rotation angle of the soller slits. The data were recorded by detector D2 using measurement configuration 2. The dashed line marks the angle of $\chi_0 = 60°$ between the scattering vectors (see Section 3.3 and Fig. 5 in the first part of this series). It can be seen that this position coincides with a maximum of intensity and a minimum of the integral breadth. Consequently, in measurement configuration 1, where the angle between the two scattering vectors is $\chi_0 = 45°$, the soller slits have to be rotated by 45°.

Table 1
Chemical analysis (in mass fraction %) of the steel investigated.

C	Si	Mn	P	S	Cr	Fe
1.025	0.325	0.318	0.014	0.005	0.048	Balance

2.2. Sample pre-characterisation by conventional XSA methods

The material investigated here is a unidirectionally ground steel C100 (German grade 1.1274, for the composition and grinding parameters, see Tables 1 and 2, respectively). The material is characterised by a uniform, fine-lamellar pearlitic microstructure achieved by the following heat treatment process:

(i) Annealing in pre-heated retort furnace under argon atmosphere at 850 °C.
(ii) Temperature variation three times between 700 °C and 850 °C to reach fine-granular microstructure.
(iii) Cooling to 500 °C with a hold time of 15 s in salt bath furnace.
(iv) Subsequently, cooling to room temperature under ambient atmosphere.

The square specimen of 25 mm edge length and 20 mm height was pre-characterised using synchrotron radiation at the EDDI@BESSY II beamline and by means of the layer removal method. Regarding the layer removal method, layers of 1 –2 μm thickness were stepwise removed from the surface followed by subsequent XSA in the near surface region of the sample applying CrKα radiation ($\lambda_{\alpha_{1+2}} = 2.2911$Å) at the 211 ferrite lattice planes ($2\theta \approx 156°$). At a depth of about 15 μm layer removal step width was 5 μm. The applied ψ-range was $-60° < \psi < 60°$ with 15 values. The incident beam was formed by a 2 mm polycapillary, while the diffracted beam was limited by a 0.