Applied Surface Science 466 (2019) 151–164

Contents lists available at ScienceDirect

Applied Surface Science

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

Full Length Article

Investigation of complex residual stress states in the near-surface region: Evaluation of the complete stress tensor by X-ray diffraction pattern decomposition

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ARTICLE INFO

Keywords:
Pattern decomposition
Instrument parameterization
Asymmetric X-ray diffraction
Near-surface residual stress states
Complete stress tensor evaluation
Plastic deformation

ABSTRACT

The presented work introduces an alternative to the classical $\sin^2\Psi$-method for the analysis of complex stress states by X-ray diffraction, with particular focus on the near-surface region. A procedure, based on the multiple *hkl*-method and implemented with a generalized least square minimization routine according to the Pawley pattern decomposition, is applied for evaluating the complete residual stress tensor in molybdenum sinter-parts after severe plastic deformation. Further, the requirements on the X-ray diffraction setup and the physically meaningful description of the instrumental aberrations are discussed. Finally, the successful implementation is demonstrated.

1. Introduction

In 2009 Balder [1] galvanized the experts with the statement "Why we should give up the $\sin^2\Psi$-method", discussed the weak points and limitations of applicability and concluded, that many different methods have been developed to overcome some of these difficulties, but without much success.

The present investigation contributes further aspects to the determination of complex residual stress states. A pattern decomposition procedure, developed for the evaluation of biaxial stress states by multi-reflection grazing incidence diffraction [2,3], is extended to the evaluation of the complete stress tensor considering the effects of elastic anisotropy. Most of the classical stress determination techniques using X-ray diffraction assume that the stress normal to the surface is zero. Triaxial stress states have been described in many experimental studies [4,5]. The classical stress analysis theory was extended by Dölle and Hauk [6,7] to include the calculation of triaxial stress states within the classical technique and upgraded by Winholtz and Cohen [8]. This work investigates, whether a pattern decomposition procedure can resolve complex residual stress states, with particular focus on the near-surface region.

2. Experimental details

2.1. The model system molybdenum

Starting from a high purity (≥99.97%) molybdenum powder, an un-textured, stress-free and well-crystallized bulk material was produced using a powder manufacturing route [9]. After screening and homogenization, the molybdenum powder was pressed into rods and plates of various geometries and dimensions. This took place using either a hydraulic press with steel dies, or by isostatic pressing, in which a rubber bag was filled with powder, for consolidating into a compact by water pressure acting on all sides. Sintering then took place, usually in hydrogen furnaces at temperatures in the range 1800–2200 °C (2073–2473 °K). The resulting molybdenum sinter parts exhibit a residual microporosity of approximately 5% [10] (see Fig. 1).

After characterization of the unstressed state, residual stresses have been introduced by a mechanical treatment of the sintered bulk material. For all investigations a disc shaped sample geometry has been used (diameter 25 mm, thickness 3 mm). One sample has been ground repeatedly in 6 steps, by alternating the direction by 90° after each single execution step (wet grinding, abrasive media: diamond, granularity 4 µm, full automated procedure controlled by the infeed of the grinding wheel, coolant: ST-B (Günther Karl GmbH, Germany)). Within all X-ray diffraction experiments the first direction has been chosen to be $\Phi = 0°$and the second as $\Phi = 90°$. A second sample has been plastically deformed in uniaxial compression until a maximum stress of 800 MPa, and on a third sample, a high pressure torsion experiment [11,12] (pressure 4 GPa; 156° rotation; temperature 200 °C) was performed to introduce additional significant shear stress components.

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https://doi.org/10.1016/j.apsusc.2018.10.010
Received 26 June 2018; Received in revised form 28 August 2018; Accepted 1 October 2018
Available online 03 October 2018
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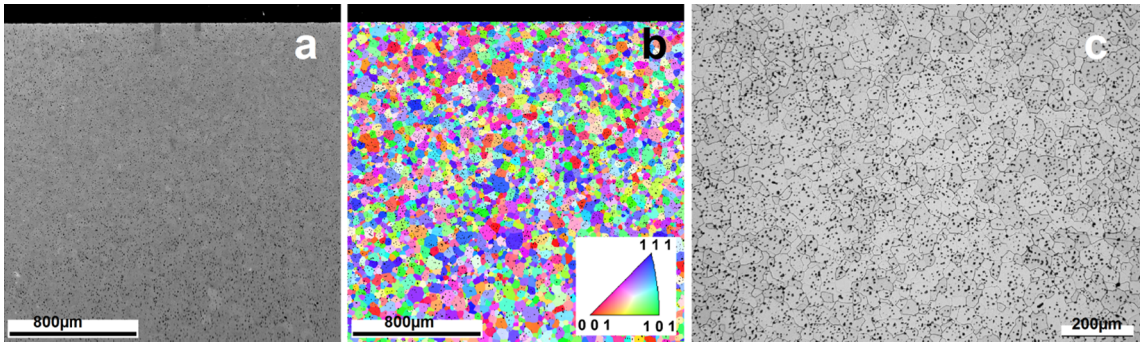

Fig. 1. Cross-section of the sintered molybdenum bulk material: scanning electron microscope image (secondary electron contrast) (a), the corresponding IPF coloring map of the EBSD scan (b) (area: 2 × 2 mm, step size: 1.2 µm), light optical micrograph after grain boundary etching (c).

Table 1
Summary of the different diffractometer configurations:

Hardware setup	Bragg-Brentano	Parallel beam	χ-inclination $\sin^2\Psi$
Detector	Lynx Eye XE-T	Lynx Eye XE-T	Lynx Eye XE-T
Detector type	psd (opening 2.94°)	psd (opening 2.94°)	psd (opening 2.94°)
Detector mode	1D	0D	1D
Goniometer	*θ-θ*	*θ-θ*	*θ-θ*
Goniometer-radius [mm]	280	280	280
Sample stage	Rotation stage	Rotation stage	Half-circle Euler cradle
Radiation	*Cu* ($\lambda_{K\alpha}$ = 1.5406 Å)	*Cu* ($\lambda_{K\alpha}$ = 1.5406 Å)	*Cu* ($\lambda_{K\alpha}$ = 1.5406 Å)
X-ray tube	KFL Cu 2 K	KFL Cu 2 K	KFL Cu 2 K
X-ray tube focus	Line	Line	Point
Primary optics	–	Göbel mirror	Polycapillary
Primary collimator	–	–	1 mm collimator
Primary Soller [°]	Axial 2.5	Axial 2.5	–
Secondary Soller [°]	Axial 2.5	–	–
Antiscatter slit [mm]	0.6	1.0	–
Receiving slit [mm]	–	2.0	–
Secondary collimator [°]	–	0.1	–
Filter	Ni	–	–

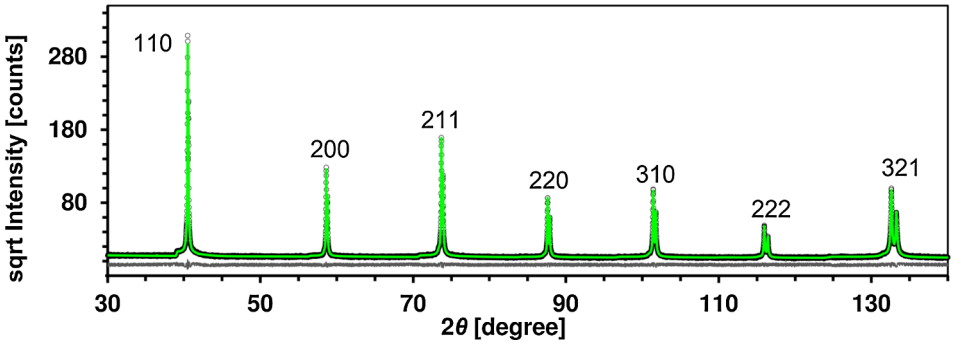

Fig. 2. Pawley decomposition of the molybdenum-powder diffraction pattern (○) measured in Bragg-Brentano geometry, decomposition with full parameter approach (—), difference (scaling factor ×2) (—), minimized lattice constant a = 3.1476 Å, GOF = 1.23.

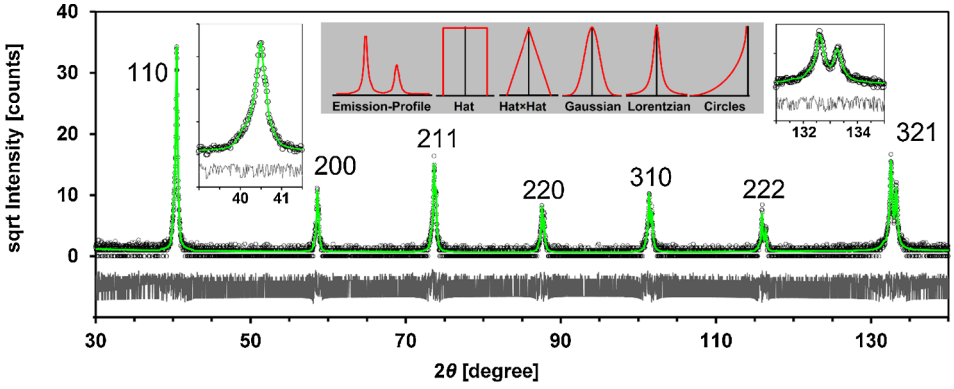

Fig. 3. Pawley decomposition of the molybdenum powder with "measured instrument function", resulting from the convolution of the inserted functions, diffraction pattern (○) measured with the parallel-beam setup in asymmetric diffraction geometry at an incidence angle α = 2°, decomposition (—), difference (scaling factor ×2) (—), minimized lattice constant a = 3.1476 Å, GOF = 1.11.