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In Situ Neutron Diffraction Studies of Substructure Evolution in Metals under Uniaxial Loading

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Neutron diffraction technique was used for the investigation of internal strains in metals under an external mechanical loading. Besides conventional mechanical response of tested specimens, further microstructural parameters in dependence on the external loading could be extracted from a position, width and shape of individual diffraction profiles by using an appropriate method of the profile analysis.

KEYWORDS: in situ neutron diffraction, mechanical test, internal stresses / strains, metals

§1. Introduction

Neutron diffraction methods of determination of internal strains and stresses in structural materials have found a lot of applications in material sciences and engineering.[1)] Due to a high penetration ability of thermal neutrons, this probe is preferentially used for mapping residual stresses in bulky materials, e.g. engineering components after different technological treatment and/or phase specific stresses in multiphase materials and composites. Recently, this method has been used also in situ during mechanical testing of materials.[2,3)] In comparison with conventional diffractometer used, the high-resolution neutron diffraction technique can provide additional new information on material microstructure behaviour in deformation process.[4)] The analysis of the neutron diffraction profiles gathered along the $\sigma - \varepsilon$ curve yields accurate bulk information on the structural changes which can be associated with the deformation process. Usually, the precise angular position of the profile maximum provides information on an average elastic strain in the sampled specimen volume whereas the width and shape of the diffraction profile can be related to the evolution of the plastic deformation. Multiphase materials and composites are particularly interesting subjects for such kind of experiments because the microstructural characteristics could often be determined for each individual component yielding thus a scheme of their interaction in deformation process.

§2. Principles of the Method

The evaluation of internal strains and stresses in crystalline materials from diffraction measurements is based on the precise measurement of the deviations of d_{hkl} lattice spacing from stress free state in particularly oriented crystal grains. Lattice strain ε_{hkl} is calculated from the displacement of the profile center $\Delta\theta_{hkl}$ as

$$\varepsilon_{hkl} = (d_{hkl} - d^0_{hkl})/d^0_{hkl} = -cot\theta_{hkl}\Delta\theta_{hkl}, \qquad (2.1)$$

where θ_{hkl} is the Bragg angle, d_{hkl} is the measured interplanar spacing and d^0_{hkl} is the stress-free interplanar spacing. In the case of conventional neutron stress/strain instruments, the elastic lattice strains are usually the only evaluated parameters. If a diffractometer with a sufficiently high instrumental resolution ($\Delta d/d \leq 2.5 \times 10^{-3}$) is used the further microstructural parameters can be evaluated from broadening and shape changes of the diffraction profiles. The proper method of the profile analysis would be used to separate two main contributions affecting generally the width and shape of diffraction profiles, microstrain and grain size (or size of mosaic blocks, more correctly), respectively. The new procedure based on a transformed model fitting has been proposed to solve this task.[5)] Modeling is performed in the reciprocal space and the convolution of the model with the instrumental resolution curve is fitted to the profiles recorded in the diffraction experiment. In the case of conventional metals, the modeling employs some elements of the integral breadth technique to include both the influence of microstrain and size of coherently diffracting blocks.[6)] The model of Gaussian distribution of lattice spacing has been successfully used to treat microstrain contribution in plastically deformed metals. This extracted parameter can be further used for an estimate of dislocation density.[7)] The shape memory alloys /SMA/ exhibit a different mechanism of plastic deformation when the martensitic transformation is induced and controlled in a reversible way by the applied stress and/or temperature. Due to a strong anisotropy, the SMA samples often yield significantly asymmetric diffraction profiles and the previous concept of simple Gaussian distribution fails. In this case, the volume distribution of the d_{hkl}-lattice spacing is used as an output of the fitting procedure.[13)] These d-spacing profiles vary significantly with the applied stress and could be compared with predictions of micromechanical models.

§3. Instrumentation

Two high-resolution neutron diffractometers dedicated to strain measurements are available at the medium-power reactor LVR-15 in NPI Řež. The instruments are equipped with curved Si and Ge monochromators and with linear high-resolution position-sensitive detectors for fast recording of diffraction profiles. The elastically bent perfect crystal monochromators work as focusing elements enabling us to adjust an optimum high $\Delta d/d$-resolution ($\approx 2 \times 10^{-3}$). The strain scanners are equipped with a deformation rig permitting both tensile and compressive tests up to maximum loading of ±20kN. A relatively small and compact rig equipped with a stepping motor was designed to be easily mounted to both neutron diffractometers. The loading tests are performed step by step and neutron diffraction spectra are recorded during the temporary stops of the deformation machine. The setup is also equipped with the specimen heating system (up to 150° C). The geometrical arrangement is shown in Fig. 1.

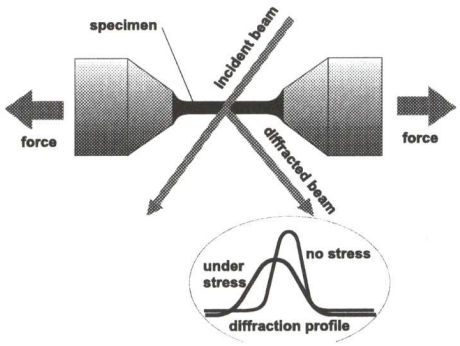

Fig. 1. Arrangement of the in situ neutron diffraction strain measurement.

§4. Examples

4.1 Duplex austenitic-ferritic stainless steel

The α/γ duplex steels usually exhibit large residual stresses due to different thermal expansion or constriction behavior and/or different plastic deformability between α and γ phases. Because of the different thermal expansion coefficients of the constituent phases, the tensile and the compressive thermal residual stresses are introduced in α and γ phase, respectively, after solution treatment. These residual stresses in the dependence on phase composition were studied in our previous experiments,[7,8] while the in situ strain measurements are described in ref.9. Two reference single-phase materials and three α/γ dual phase alloys of various volume fractions were studied, however, in this review we will present just some results from the alloy containing 32.6% of α phase only. In diffraction experiment, two reflections (110) and (111) of α and γ phases, respectively, were recorded during the tensile deformation loading (Fig. 2). The parameters of the lattice strain (Fig. 2), microstrain and dislocation density (Fig. 3) were evaluated for each phase as a function of external loading. Typical behaviour of lattice strains can be seen from Fig. 2. In the elastic region of the deformation curve, the lattice strain response is linear.

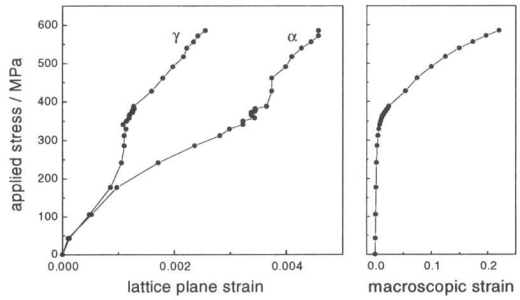

Fig. 2. Lattice plane strains of both phases and macroscopic strain as a function of the applied stress. The thermal residual lattice strains were reset to zero.

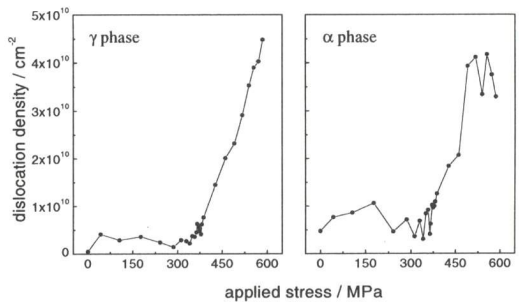

Fig. 3. Changes in dislocation densities obtained by neutron diffraction as a function of applied stress.

Fig. 2 also shows how the softer γ phase is preferentially plastically deformed in comparison with the harder α phase beyond the yield point. In principle, using a proper microstructural model the lattice strains from each phase could be used for an estimate of elastic stress partitioning.[10] However, the present measurement related to one reflection and one strain component of each phase is not sufficient to provide an unambiguous solution. On the other hand, the effect of plastic strain partitioning during tensile deformation can be estimated under a simplifying assumption that microstrain distribution in crystal lattice is much more isotropic in comparison with elastic lattice strains. In this case, the curves of macroscopic strain as a function of dislocation density ρ obtained in the single-phase material test are used as calibration curves to convert the $\rho_{\alpha\gamma}$ of the relevant constituent phase to the averaged plastic strain in the dual phase alloys.[9] The result of this procedure is displayed in Fig. 4.

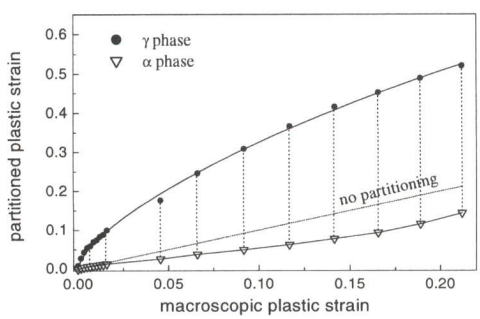

Fig. 4. The estimate of partitioned plastic strains for α and γ phase.

4.2 CuAlMnZn shape memory alloy

The in situ neutron diffraction method was used to study the stress induced martensitic transformation /SIMT/ in the Cu-based shape memory alloy during tensile pseudoelastic deformation cycle.[11–13] Two diffraction profiles (scattering vector parallel to the tensile axis) were recorded simultaneously along the $\sigma-\varepsilon$ curve - 220 reflection from the β_1 cubic austenite phase and common 0018_{18R} (002_{2H}) reflections from the β'_1 (γ'_1) martensite phase, respectively. During the uploading, the martensite phase is stress induced from the austenite yielding large inelastic strain that disappears again on reverse unloading together with the martensite phase. The profile analysis provides quantitative bulk information on the evolution of the volume fractions of the transforming phases (integral intensities), elastic lattice strains in the interacting phases (profile positions)[11,12] and, particularly interesting d-lattice spacing distributions evolving in the austenite and martensite phases.[13] Because of large elastic anisotropy of CuAlMnZn and transformation anisotropy of the $\beta_1 \rightarrow \beta'_1$ martensitic transition, load sharing among individual austenite grains varies during the tensile test due to a redistribution of stresses in them. As a result, recorded diffraction profiles (see Fig. 5) reveal complex shape changes characterized by widening already in the elastic region and a strong asymmetry above 250 MPa when SIMT starts.

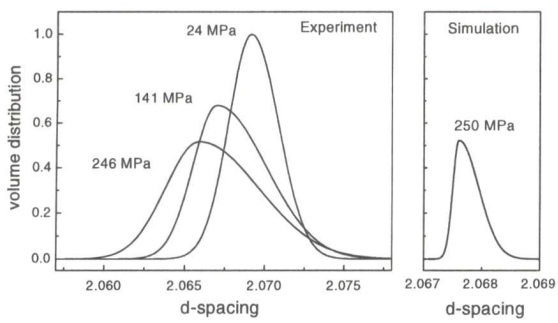

Fig. 5. The evolution of the lattice spacing distribution extracted from 220 austenite reflection with external loading. For comparison, see the result of simulation procedure.

Such effects are very far from those observed in other metallic materials. The selfconsistent micromechanics model (based on Crystallographic model of SMA[14]) of polycrystal transformation taking into account the SIMT deformation mechanism and elastic anisotropy has been used to calculate statistical distribution of d-lattice spacings and austenite volume fractions in individual austenite hkl reflections and interpret thus the diffraction results. Mainly, the model captures quite well different behaviours of lattice plane responses (lattice strains, intensity variations) of individual crystal lattice planes.[11,12] However, it also provides rough qualitative explanation of the complex profile shape evolution (see the simulated asymmetric profile at the transformation start stress in Fig. 5). The peak asymmetry reflects the load partitioning in the specimen when the SIMT starts.[11,12] However, the width of the simulated d-distributions is much narrower than observed ones. There are at least two reasons for this: i) simplistic consideration of the δ-function at d^0_{220}=2.069 Å in the model as a starting d-distribution of a system, and ii) neglecting the d-distribution widening due to type II microstresses associated with advancing transformation. The model is being further elaborated in this respect.

§5. Discussion

The described method provides a variety of microstructural data, however, their interpretation is not usually a straightforward. In connection with a microstructural model, it might become a powerful tool in material sciences, bringing additional information on the deformation processes and contribute to the understanding of the deformation mechanism involved. Interesting applications are multiphase materials and composites.[15] In the particular case of SMA's, the constituent phases which are mutually transformed by external mechanical and/or thermal loading, recent in-situ neutron diffraction studies[11–13,16] brought interesting results concerning load partitioning mechanism in CuAlZnMn[11] and transformation texture evolution in TiNi.[16] The in situ method can be very useful even in conventional stress/strain mapping experiments. Generally, in plastic region, the lattice strain response is nonlinear (see Fig. 2). This effect could cause significant errors in correct evaluation of residual stresses when the concept of linear response would be used.[2] The examination of the in situ lattice responses of metallic materials of technological interest can yield the calibration curves (ε_{hkl},σ) which may be used for more correct calculations of internal stresses in engineering components.

Acknowledgements

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