EAN 2019
57th conference on experimental stress analysis

On a possible use of neutron three axis diffractometer for studies of elastic and plastic deformation of polycrystalline materials

MIKULA Pavol[1,a] RYUKHTIN Vasil[1,b] and ROGANTE Massimo[2,c]

[1]Nuclear Physics Institute ASCR, v.v.i., 25068 Řež, Czech Republic

[2]Rogante Engineering Office, I-62012 Civitanova Marche, Italy

[a]mikula@ujf.cas.cz, [b]ryukhtin@ujf.cas.cz, cmain@roganteengineering.it

Keywords: Neutron diffraction, instrumentation, strain measurements

Abstract

Feasibility of using a high-resolution three axis neutron diffractometer performance for elastic and plastic deformation studies of metallic polycrystalline samples is presented. The method consists of unconventional set up employing bent perfect crystal (BPC) monochromator and analyzer with a polycrystalline sample in between (see Fig. 1). After the realization of focusing conditions in real and momentum space at the neutron wavelength of 0.162 nm, a high angular resolution up to $FWHM(\Delta d/d)=2\text{x}10^{-3}$ was achieved on the standard α-Fe(110) sample (2 mm diameter) which then opened the possibility for the measurements of small lattice parameter changes of samples. The feasibility of the instrument for macro- and micro-strain as well as grain size studies is demonstrated on the polycrystalline samples of low carbon shear deformed steel wires and the NiTi plates subjected to heat treatment.

Introduction

At present, the investigations of residual strains/stresses are usually carried out at the dedicated double axis diffractometers (strain scanners) with a BPC focusing monochromator situated on the first axis, a sample situated on the second axis and with a position sensitive detector (PSD) [1-5]. However, the $\Delta d/d$ resolution of these dedicated scanners derived from the $FWHM$ of the diffraction lines is sufficiently high for small sample gauge volumes (when the width of the irradiated part of the sample is about 2 mm or less) but rarely better than $8\text{x}10^{-3}$ for bulk samples and therefore they are used for the measurements of elastic strain

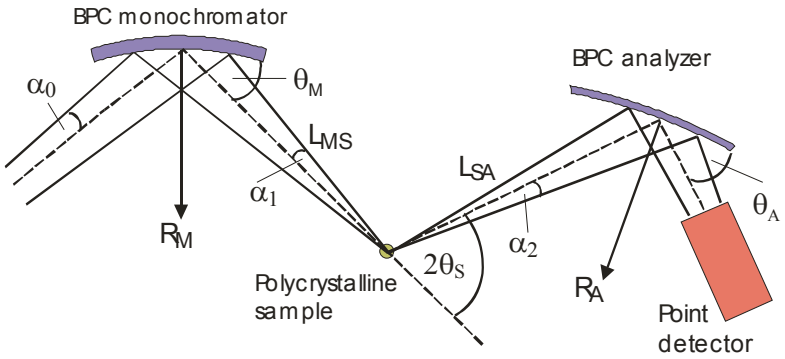

Fig. 1: Three axis diffractometer setting employing BPC monochromator and analyzer as used in the experiment (R_M, R_A - radii of curvature, θ_M, θ_A - Bragg angles).

effects resulting in the changes of lattice constants and angular shifts of the diffraction lines. For the investigation of micro-strain effects resulting in a change of the *FWHM* and shape of diffraction profiles a substantially higher resolution is required which can be achieved just by the proposed 3-axis diffraction set-up.

Experimental details

The set-up shown in Fig. 1 was realized on the 3-axis neutron diffractometer installed at the Řež research reactor LVR-15. Si(111)-monochromator and Si(400)-analyzer (or Si(311)-analyzer) single crystals had the dimensions of 200x40x4 mm^3 and 20x40x1.3 mm^3 (length x width x thickness), respectively. The monochromator Si(111) providing the neutron wavelength of 0.162 nm had a fixed curvature with a radius R_M of about 12 m. The curvature of the analyzer was R_A= 9 m, where the best resolution was found. For a practical demonstration of the feasibility of using the three axis set-up we used two types of polycrystalline samples: First, non-deformed as well as deformed α-Fe(110) wires (low-alloyed steel) of the diameter of about 5 mm with accumulated shear deformation as a result of rolling with the shear of the metal ingot and conventional wire drawing (see Tab. 1 and 2). The fourth column in Tab. 1 shows the reduction degree in drawing deformation. Then, we used 3 Ni(50%)Ti(50%) alloy samples (untreated, treated at 900 ºC for 1.5 hour and treated at 900 ºC for 3 hours). They were in the form of discs of the diameter of 25 mm and the thickness of 5 mm.

Table 1: Description of deformation of the samples.

Sample number	ϕ [mm]	Shear def. [%]	Drawing def. [%]
1	5.10	0	0
2	4.28	8	23.2
3	5.35	0	0
4	4.28	16.6	23.2
5	5.57	0	0
6	4.28	23	23.2

Table 2: Chemical composition of low-alloyed structural steel grade 08G2S GOST 1050 element

Element	C	Mn	Si	S	P	Cr	Ni	Cu	N2
wt %	0,08	1,87	0,82	0,020	0,022	0,02	0,02	0,02	0,007

Low-carbon shear deformed steel wires

In this case, a Si(400) bent perfect slab was used as the analyzer. The description of the non-deformed as well as accumulated shear deformed samples is shown in Table 1. Table 2

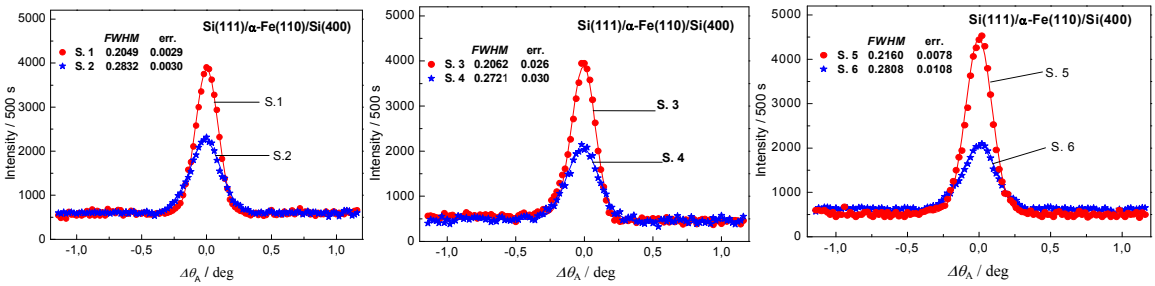

Fig. 2: An example of diffraction profiles related to the samples S.1-S.6 situated in the vertical position as analyzed by BPC Si(400) analyzer (strain measurements of the radial components).

describes the chemical composition of the steel. For the measurements of the radial component of strains the steel samples were situated on the second axis of the diffractometer in the vertical position. The width of the incident beam was 8 mm and therefore, the whole volume of the sample was irradiated (within the whole diameter). The height of the incident beam was 20 mm. The obtained results of the analyzer rocking curves are shown in Fig. 2. From the introduced individual profiles (see Fig. 2), we can detect the following features: the peak intensities and *FWHM*s related to the deformed samples differ very little. This is brought about by the fact that the diameter of the deformed samples were equal and that the shear deformation had a negligible effect. Very close values of *FWHM* point out the fact that the lattice deformation in the radial direction was basically brought about by drawing deformation. The integrated intensity under the peak profiles related to the non-deformed samples S.1, S.3 and S.5 primarily corresponds to the irradiated volume of the sample, which is, of course, maximum for the sample S. 5.
In the next step the steel samples were situated on the second axis of the diffractometer in the horizontal position. The obtained results are shown in Fig. 3. In comparison with the previous

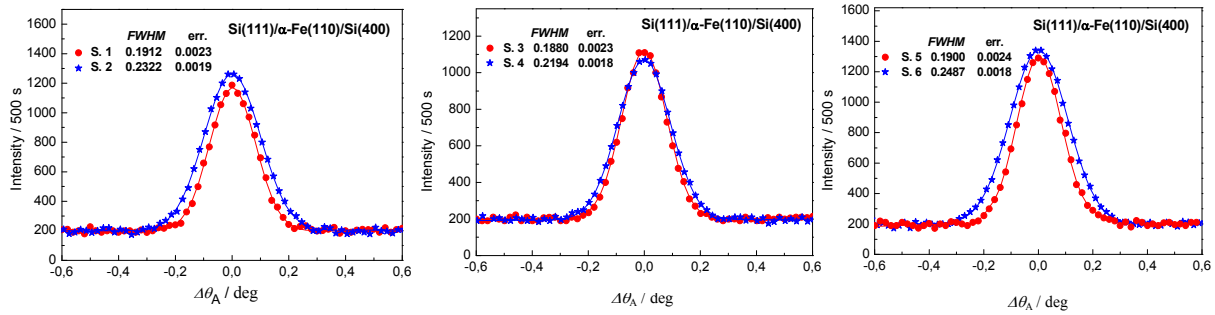

Fig. 3. Diffraction profiles related to the samples S.1-S.6 situated in the horizontal position for measurement of the axial component as analyzed by the bent perfect Si(400) analyzer.

case, for the samples in the horizontal position their irradiated volume is much smaller and correspondingly neutron signal was smaller. In this case, it was found out that the resolution was dependent on the width of the incident beam impinging the sample and therefore, we used the slit width of 5 mm. It can be seen from Fig. 3 that the resolution represented by *FWHM* was practically the same for all non-deformed samples S.1, S.3 and S.5. Small differences in *FWHM* can be seen for plastically deformed samples. It points out the fact that the lattice deformation was basically brought about by drawing deformation and influencing the radial strain component and much less the axial strain component.

NiTi shape memory alloy samples

In this case we tested the feasibility of study of structure changes as a result of a thermomechanical external load of NiTi alloy samples. It is well known that the outstanding

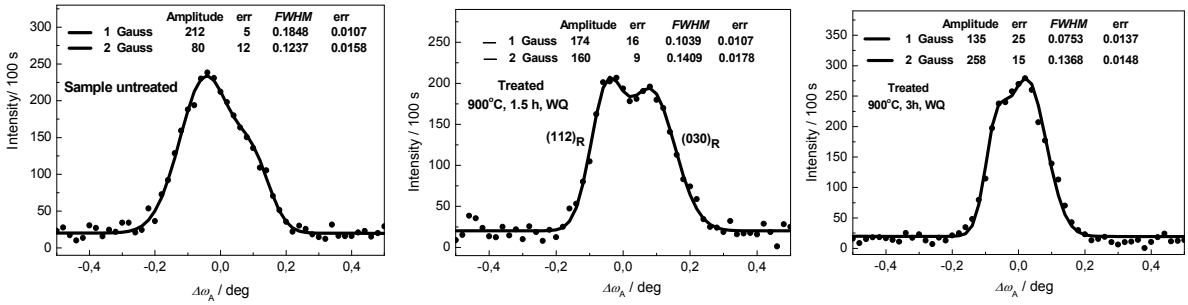

Fig. 4: An example of diffraction profiles related to the NiTi samples situated in the vertical position as analyzed by BPC Si(311) analyzer. The curves are fitted by two Gaussians.

properties of NiTi super elastic shape memory alloys which are accompanied with high mechanical properties make them suitable for a variety of applications. NiTi alloys can also contain such other chemical elements as e.g. Co, Fe, V, etc. NiTi alloys have in principle two distinct phases: austenite phase having high symmetric B2 structure that exists at high temperatures and martensite phase having low symmetric orthorhombic (or monoclinic) $B19^0$ structure that exists at low temperatures. When cooling the austenite phase then right before the transformation to martensite phase in the vicinity of 20 °C under certain circumstances an intermediate R-phase with rhombohedral crystal structure can be formed [6,7] Fig. 4 shows an example of the analyzer rocking curves obtained on the mentioned samples. The measurements were carried out at the ambient temperature of 18.5 °C (the temperature in the reactor hall). It can be seen from Fig. 4 that the temperature treatment had an influence on the quality of the NiTi alloy and that two close reflections of the R-phase could be clearly identified, namely, for the treatment at 900 °C for 1.5 hour. Then, by warming this sample to 45 °C, R-phase was transformed to the austenite B2 one as shown in Fig. 5.

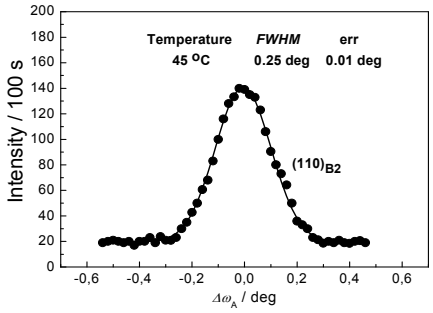

Fig. 5: Austenite phase diffraction profile obtained at 45 °C.

Summary

After similar preliminary instrumentation studies [8] another 3-axis neutron diffractometer setting employing BPC monochromator and analyzer with the studied polycrystalline samples between them was tested for practical application. It has been proved that the diffractometer setting provides a sufficiently high $\Delta d/d$ resolution (d is the lattice plane distance) permitting macro- and micro-strain studies as well as the grain size distribution after applying shape analysis of neutron diffraction peaks [9,10]. The neutron diffraction results obtained on the samples of low-alloyed quality structural steel and NiTi shape memory alloys document the feasibility of this unconventional 3 axis set-up for studies of structure properties of polycrystalline samples in the plastic deformation region. It can be seen from Tab. 3 that though the *FWHM*-effects resulting from the applied deformation on the samples are very small and cannot be studied by the conventional strain scanner, thanks to the high resolution of the present experimental set-up, they are clearly measurable. In particular, such results can be used as an additional support to complement the information achieved by using the other characterization methodologies. As to NiTi samples it should be pointed out that major characteristic peaks of different phases (martensite, austenite, R-phase) are within only a few $\Delta\theta_A$ degrees in the vicinity of $2\theta_A$ scattering angle [7], where the best resolution of the neutron diffraction set-up was adjusted.

Table 3: Summary of the *FWHM*s as calculated in ($\Delta d/d$)-scale

Sample number	S.1	S.2	S.3	S.4	S.5	S.6
FWHM($\Delta d/d$) - Radial [10^{-3}]	4.09	5.66	4.12	5.44	4.32	5.61
FWHM($\Delta d/d$) - Axial [10^{-3}]	3.82	4.64	3.76	4.38	3.80	4.97

Finally, it should be pointed out that contrary to the conventional double axis strain scanner, this three axis setting provides a high resolution for bulk samples e.g. of the diameter of 5-10 mm whilst for a conventional scanner such diameters themselves introduce, to the resolution, an uncertainty in *FWHM* of the diffraction profile at least of $1x10^{-2}$ rad. Thanks to the high resolution property of the 3 axis set-up, it can be, of course, used also for the measurements of

residual elastic deformation macro-stresses, however, less efficiently in comparison with the conventional two axis neutron diffraction measurement [1-5] which has not so high resolution requirements.

Acknowledgement

Measurements were carried out at the CANAM infrastructure of the NPI CAS Řež supported through MŠMT project No. LM2015056. The presented results were also supported in the frame of LM2015074 infrastructural MŠMT project "Experimental nuclear reactors LVR-15 and LR-0". Bragg diffraction optics investigations are in the Czech Republic supported by the ESS project LM2010011: "Contribution to Partnership in Large Research Infrastructure of Pan European Importance" and in Italy by the COST Action CA16122. We thank Ms. B. Michalcová from NPI ASCR for a significant help in the measurements and basic elaboration of the data.

References

[1] I.C. Noyan, J.B. Cohen, Residual stress: Measurement by diffraction and interpretation, 1st edn; 1987, New York, Springer-Verlag.

[2] M.T. Hutchings, and A.D. Krawitz, (Eds.), 1992, Measurement of residual and applied stress using neutron diffraction, NATO ASI Series, Applied Sciences, Vol. 26, Kluwer Academic Publisher, 1992.

[3] P. Mikula, M. Vrána, P. Lukáš, J. Šaroun and V. Wagner, High-resolution neutron powder diffractometry on samples of small dimensions, Materials Sc. Forum, 228-231 (1996) 269-274.

[4] P. Mikula, M. Vrána, P. Lukáš, J. Šaroun, P. Strunz, H.J. Ullrich, V. Wagner, Neutron diffractometer exploiting Bragg diffraction optics - A high resolution strain scanner. In Proc. of ICRS-5, June 16-18, 1997, Linköping, T. Ericsson, M. Odén and A. Andersson (Eds.), 2 (1997) 721-725.

[5] B.S. Seong, V. Em, P. Mikula, J. Šaroun, M.H. Kang, Unconventional performance of a highly luminous strain/stress scanner for high resolution studies, Materials Sc. Forum, 681 (2011) 426-430.

[6] T. Yoneyama, S. Miyazaky, Shape memory alloys for biomedical applications, Woodhead Publ., Cambridge 2009.

[7] Z. Lekston, M. Zubko, X-ray diffraction studies of the reversible phase transformation in NiTi shape memory alloy, Acta Physica Polonica A, 130 (2016) 1059-1062.

[8] P. Mikula, M. Vrána, J. Šaroun, J. Stammers, V. Em, Dispersive double bent crystal monochromators Si(111)+Si(311) and Si(111)+Si(400) with a strongly asymmetric diffraction geometry of the analyzer for powder diffractometry, Powder diffraction, (2019), in print.

[9] R. Delhez, T.H. de Keijser, E.J. Mittemeijer, 1982, Determination of crystallite size and lattice distortions through X-ray diffraction line profile analysis, Fresenius' Zeitschrift für analytische Chemie, 312, 1 (1982) 1-16.

[10] V. Davydov, P. Lukáš, P. Strunz, R. Kužel, Single-line diffraction profile analysis method used for evaluation of microstructural parameters in the plain ferritic steel upon tensile straining, Materials Sc. Forum, 571-572 (2008) 181-188.