Peak broadening and peak shift pole figures investigations by STRESS-SPEC diffractometer at FRM II

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Abstract This paper studied for the first time peak intensity, peak position and FHWM pole figures with one time measurement at the neutron diffractometer STRESS-SPEC via in-situ tensile deformation on austenitic steel. Fibre distribution with its evolution from central tensile direction to normal direction of these three kinds of pole figures was obtained. Variation of peak position and FWHM can be correlated to the reorientation of the texture component.

1. Introduction

Diffraction (X-ray or neutron) analysis on polycrystalline metals is a non-destructive method which gives real information on materials, such as crystal size, their deformation, preferred orientation and structural defects [1]. Preferred orientation or crystallographic texture analysis through measuring the relative intensities of certain Bragg peaks by diffraction methods has been well established after the pioneered work of Bung [2]. With this method intensity variation corrected with background or absorption and defocusing (using X-ray) of the considered lattice plane is measured with respect to the sample orientation, intensity pole figure can therefore be built.

It is known that coherent X-ray or neutron diffractions of poly crystals result in plentiful peak profile information. The real crystals are not idea such as there always exits defects, micro strains, grain boundaries, stacking faults, etc. [3]. Generally, the diffraction peaks get broader when the grains in a material become smaller, especially for nano-crystals. It was also noted that contribution of dislocations to peak broadening was much more apparent than small grains when they were larger than 1 μm. In addition, strains that vary from grain to grain or within a grain (micro strains) due to local environment can also produce peak broadening. Macro strain due to an external load leads to the peak shift. X-ray diffraction peak profile for grain size and micro strain analysis based on Williamson-Hall and Rietveld methods has been widely investigated using powder diffraction data [4, 5]. However, researches on peak broadening and peak shift pole figures have been rarely done because of the requirement of high flux and high resolution instruments having a fast detector readout system. Especially, under loading condition the peak broadening pole figure caused by dislocations and peak

shift pole figure caused by macro strains have not been performed so far. Peak shift pole figure can describe the deformation anisotropy. The relationship between micro strain and texture is still unclear.

Using neutron diffraction bulk samples rather than surfaces can be measured. Therefore, coarse-grained materials can be characterized easily; averaging over texture inhomogeneities of mechanical test samples can open the direct correlation between texture and material's properties. Moreover, environmental cells (heating, cooling and deformation, etc.) are available and the angular resolution in orientation space is better than for an X-ray goniometer because no defocusing occurs. The materials science neutron diffractometer STRESS-SPEC at FRM II was developed as a multi-purpose diffractometer for strain and crystallographic texture [6, 7]. This diffractometer is located at a thermal beam port and comprises a highly flexible monochromator arrangement, utilizing three different monochromators: Ge, bent Si and pyrolytic graphite (PG) which give continuous opening angle that allows a wavelength selection between 1 Å and 2.4 Å. In addition, STRESS-SPEC offers the users various sample environments such as tensile/compression, torsion, cyclic loading, temperature controlling, etc.

Current study was carried out to study the evolution of peak intensity, peak broadening and peak position pole figures with increasing loads using the neutron diffractometer STRESS-SPEC, which is the first time to perform such kind of measurements on this diffractometer. Microstructure and strain anisotropies, correlations among these three kinds of pole figures will be discussed.

2. Experimental

The in-situ tensile/compression device was designed under the cooperation with SPODI group at FRM II, as shown in Figure 1 (left). It achieves a chi 0~90º tilting and a phi 0~360º rotation which can cover a complete pole figure under loading by combining reflection and transmission methods. It can also be mounted and aligned easily on the STRESS-SPEC sample table.

Austenitic steel was used to prepare the tensile sample with its dimension shown in Figure 1 (right). Wavelength of 1. 73 Å produced by Ge monochromator was selected. Primary slit which controlled the coming beam size to Ø10 mm with the second slit open were utilized. An area detector of 30×30 cm^2 which can cover the (111) and (200) reflections simultaneously was installed. Pole figure measuring points were selected under the yield point at 0 MPa, 100 MPa, 200 MPa and 300 MPa, respectively. At each point the standard equal angular scan method for complete pole figure measurement was realized, of which χ tilts from 0º to 90º and φ rotates from 0º to 360º at each χ. Due to a large area detector and its distance to 850 mm only 6 times χ tilting can cover 90º. At STRESS-SPEC a continuous scanning routine of φ rotation with each 5° was implemented, i.e., the detector collects all the diffraction patterns when φ rotates 5° continuously in 1 min. Total time of 7.5h was needed at one load for a complete pole figure measurement.

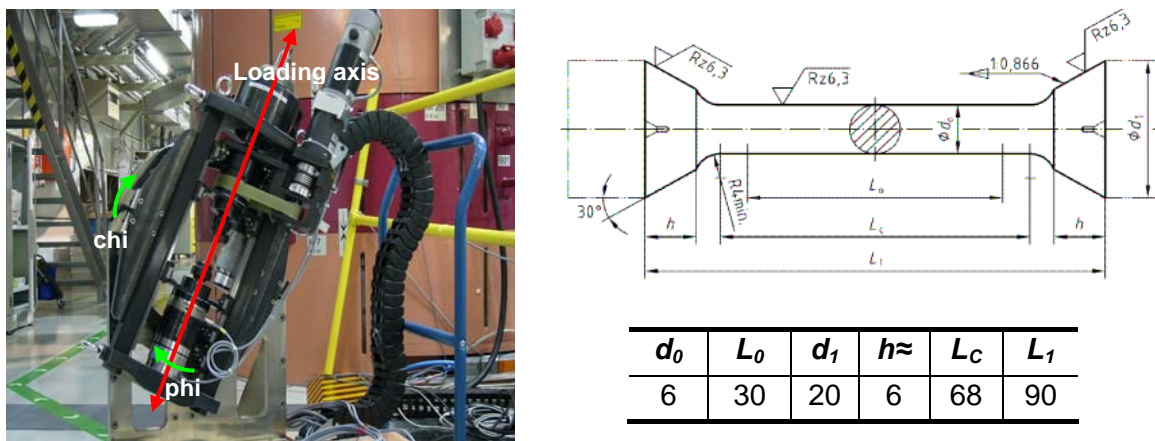| d_0	L_0	d_1	$h\approx$	L_C	L_1
6	30	20	6	68	90

Figure 1 View of the tensile machine (left); and right is the dimension of austenite sample (in mm).

A software package StressTexureCalculator (SteCa) developed mainly by C. Randau at STRESS-SPEC group was used for data treating of current measured data [8]. SteCa allows to extract pole figure data for intensity pole figures (crystallographic texture), for peak position pole figures (macro strain) or for peak broadening pole figures (micro strain) and to construct diffraction patterns (phase analysis) as well as strain profile patterns as function of x, y, z (strain mapping). Fundamentals and functions of SteCa have been described in detail from the reference paper [8]. Gaussian fitting was used to calculate the peak intensity, peak position and FWHM using linear background of an *hkl* reflection. The output ASC II data each containing intensity, peak position and FWHM variation with equal angular 5°×5° grid can be plotted using PfPlot.

3. Results and discussions

Figure 2 shows the variation of (200) peak position at start position of each different load. Clear is that the central peak position was shifted with increasing load. This is due to the lattice planes perpendicular to the loading direction are compressed.
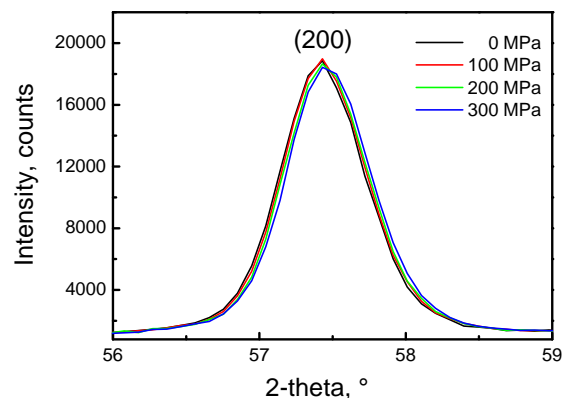

Figure 2 Variation of (200) peak with different loads.

Figure 3 shows the (111), (200) and calculated ODFs under load of 0 MPa and 300 MPa, respectively. The tensile direction is the pole figure centre. There exits a strong brass {110} <112>, a cube {001} <100> and a weak rotated goss {110} <110> components at the initial material. Looking on the pole figures at load=300 MPa they indicate that there exist a rotation from pole figure central tensile direction to the edge. But the maximum intensity was remained.

Figure 4 shows the peak position pole figures of (111) and (200) at two loads, respectively. At loads=0 MPa both (111) and (200) peaks show a position variation about 0.02°. When the load was increased to 300 MPa the (111) peak position distributes from χ=40° of 49.01° to χ=90° of 49.05°, and (200) peak from χ=60° of 57.43° to χ=90° of 57.49°. High load leads to the tensile effect on the lattice planes at central part and a compression effect on the planes at sample's edge. The 2θ angle was shifted about 0.06° for (200) peak under load 300 MPa which was still under yield point of this austenitic steel.

Looking on the (111) and (200) FHWM pole figures of load=0 MPa and load=300 MPa, as shown in Figure 5, both illustrate a similar as a fibre distribution along central tensile direction which demonstrate a precise sample alignment of the instrument setup. There exits a gradient decrease of FWHM in (200) from centre of 0.79° to edge of 0.73° at load= 0 MPa. This indicates that there could exit relatively high density of dislocations in the as received material; and with much higher density in the centre than at the edge. It has been reported that dislocation contributes mainly to the peak broadening when its size is not in the nano region [9]. FWHM at the central part was increased to 0.81° of (200) peak under load= 300 MPa, as shown in the figure, apparent is that much more peak broadening occurred from centre to edge of the pole figure than that at load=0 MPa.
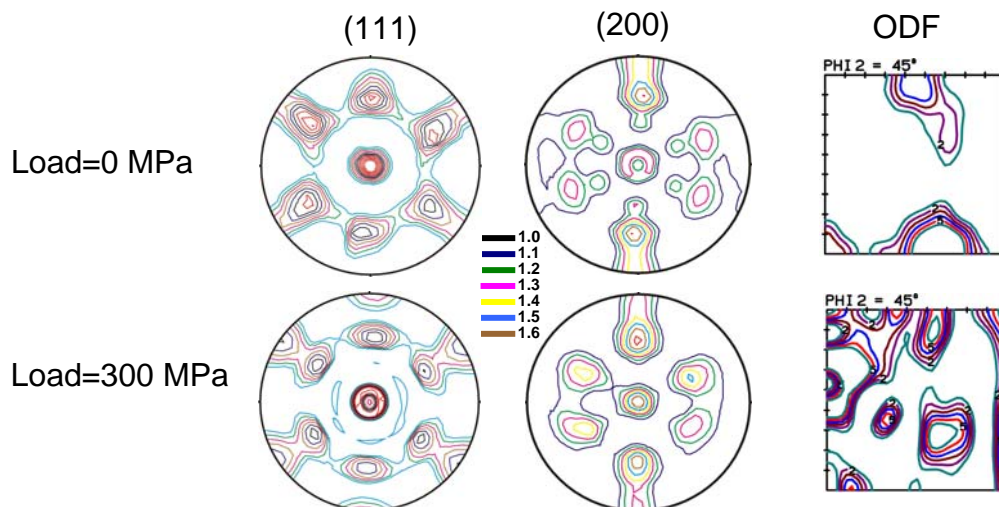
Figure 3 (111) and (200) peak intensity pole figures and calculated ODF at Phi2=45° for loads 0 MPa and 300 MPa, respectively.
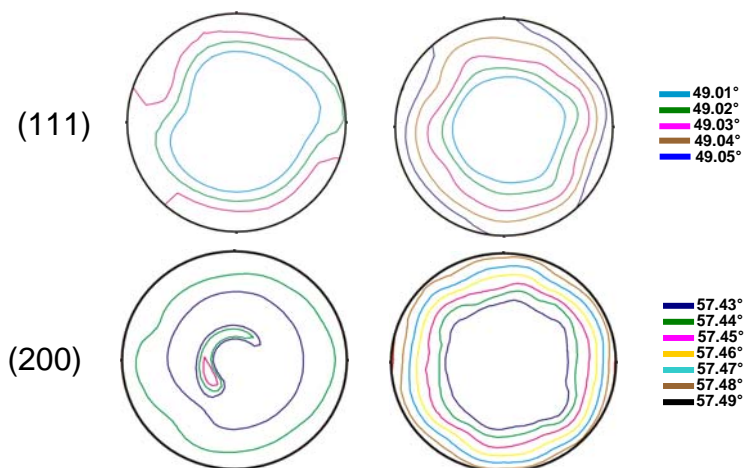

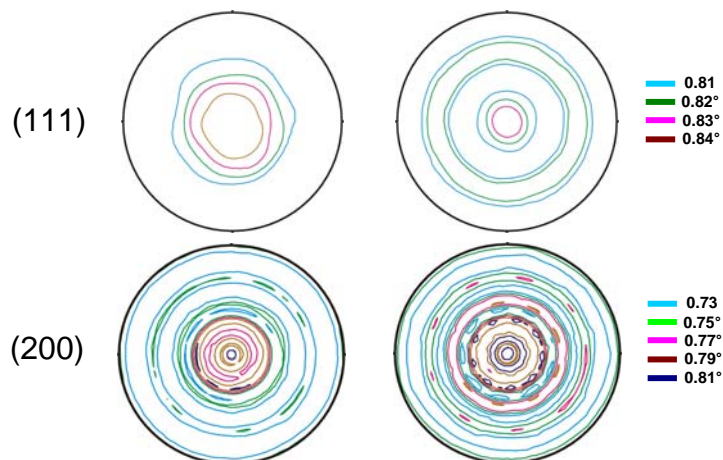Figure 4 (111) and (200) peak position pole figures at 0 MPa (left) and 300 MPa (right).

Figure 5 (111) and (200) FWHM pole figures at 0 MPa (left) and 300 MPa (right).

Two main points should be emphasized when comparing intensity, peak position and FWHM pole figures. Firstly, all the pole figures show a fibre distribution around the tensile direction. Reorientation of some grains at 300 MPa was noticed in intensity pole figure. The variations of peak position and FWHM after tensile are not so large since current load is still under yield strength. Different irradiated gauge volume at each χ tilt position might have some effects on the peak profile. However, tensile deformation of the rod sample, a relatively large coming beam and long counting time can decrease this effect.

Secondly, distribution of contour level in peak position and FWHM pole figures which are presented with α and β angles are similar as that of peak intensity in the (111) and (200) pole figures. In theory, there should be no plastic deformation under 300 MPa. This should be due to the modification of Young's modulus resulted resulted from the preferred orientation distributions.

4. Summary

From this investigation it indicated that the strain and texture distribution can be obtained simultaneously provides a good alignment of the neutron diffractometer. Gradient distribution of peak position and FWHM can be related to the preferred orientation distribution of crystals. This new development will greatly enlarge and improve the application of STRESS-SPEC in the filed of materials science.

Acknowledgements

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