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ESTIMATION OF RESIDUAL STRESS IN COLD ROLLED IRON-DISKS FROM STRAIN MEASUREMENTS ON THE HIGH RESOLUTION. FOURIER DIFFRACTOMETER

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INTRODUCTION

Forming process outcomes can be influenced by residual stress states in the formed material. For example, cold rolled steel disks used for forming small, gas pressure tanks can have inadmissible folds on their borders after the first forming step. Residual stresses might appear in the disks due to several reasons, one being the means of cutting the disk from rolled sheet metal. To have an effective forming process it is important to be able to check residual stress states, as well as the initial material texture by a quick and easy testing method. For this purpose, magnetic [1] and ultrasonic [2] nondestructive testing methods have been developed and utilised. These techniques have certain disadvantages, however. Magneto-elastic measurements do not permit direct testing of residual stresses and provide information on stress states in the near surface region only. The ultrasonic technique gives stress values averaged over the scanned path and the sound-wave beam cross-section. In both cases the rolled texture complicates the determination of stress states in sheet metal. Calibration of these techniques is therefore necessary for a reliable assessment of rolled plates used in forming processes. This calibration can be performed by neutron diffraction, a unique non-destructive reference method [3].

This paper presents and discusses the results of estimating residual stresses in cold rolled iron disks by measurements with the high resolution Fourier diffractometer (HRFD) at the IBR-2 pulsed reactor in Dubna [4]. A ⁶Li -glass scintillation detector is installed at the scattering angle 2θ =+152° on the HRFD (Fig. 1). The neutron intensity on the sample is 10⁷ n/cm²/s and the resolution achieved by the diffractometer is $\Delta d/d$ =10⁻³. This allows the measurement of strain in bulk metallic specimens with sufficiently high precision and within a reasonable measuring time.

To verify expectations for the possibility of strain measurements in steel components with HRFD, a relatively simple program has been started using the back scattering geometry of this spectrometer. Prospects of HRFD development will be discussed in the concluding section of this paper.

EXPERIMENTS

The tested objects were cold rolled steel disks of 2.5 mm thickness and diameter of about 500 mm (steel - C(0.127 %), Si(0.100 %), Mn(0.760 %), P(0.011 %), S(0.008 %), and AlS(0.043 %). The bulk elastic constants were: $E \equiv 180 \text{ kN/mm}^2$, and $\mu \equiv 0.3$. The yield point $R_{p0.2}$ of this material was 276 MPa and the tensile strength R_m was 434 MPa. A first indication of the presence of residual

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stress states in the considered disk is disk unevenness (Fig. 2). The magnetic and ultrasonic measurements carried out at the Fraunhofer-Institute for Nondestructive Testing in Saarbrücken indicated characteristic changes in stress states across the disk. The magnetic testing method had a lateral resolution of about $20 \times 20 \text{ mm}^2$, which was determined by the geometry of the magnetic sensors. The gauge volume for the neutron diffraction measurements was chosen accordingly. With the help of a boron nitride (BN) mask, the cross-section of the neutron beam was reduced to $2 \times 20 \text{ mm}^2$ (Fig. 3).



<u>Fig. 1.</u> Setup of the HRFD high resolution Fourier diffractometer at the IBR-2 reactor: 1 - moderator, 2 - background chopper, 3 - Fourier chopper, 4 - mirror neutron guide, 5 - main detectors at the scattering angles of $2\theta = \pm 90^{\circ}$ (in progress) and $2\theta = \pm 152^{\circ}$ and -152° (in project), 6 - auxiliary, low resolution detectors, 7 - measuring module.

2



Fig. 2. Disk unevenness: off-plane deviations were measured at different angles φ around the disk at 50 mm from the disk border, the z-axis is perpendicular to the disk plane.



Fig. 3. The back scattering geometry for strain measurements in a cold rolled disk, Q - the scattering vector, ϕ - the scattering angle.



Fig. 4. Definition of the global (x,y) and local (x',y') coordinate system. The rolling direction is along the x-axis.

To determine the strain tensor, ε , from neutron diffraction spectra, the scattering vector, **Q**, has to be oriented in different directions (Fig. 4). Because stresses are averaged over the interior of the scattering volume, the internal stress components belonging to the normal direction to the disk plane should vanish. As a result, the determination of the main components of the local strain and stress states is reduced to a quasi two-dimensional problem. Extensive investigations were not possible due to the limited measuring time and, therefore, only four directions of the scattering vector **Q** were chosen the radial direction ($\varphi'=0^\circ$) of the disk, the tangential

component ($\phi'=90^{\circ}$), and the $\phi'=\pm 45^{\circ}$ directions in the (x',y') local coordinate system built by the radial and tangential components. At several points the normal component of the strain perpendicular to the disk-plane was also measured. A survey of the changes in the internal strain and residual stresses was obtained for eight observation points on a track lying around the disk at a distance of ~55 mm from the border of the disk.

The lattice spacing in an unstrained state, d_0^{hkl} , was derived from the reverse time-of-flight (RTOF) spectrum for an annealed powder sample made from the disk material (Fig. 5). Hereafter, Miller indices of crystal lattices are given in square brackets, i.e. [hkl]. The lattice strain is determined as a relative shift of the Bragg reflexes $\Delta^{hkl} = (d^{hkl}-d_0^{hkl})/d_0^{hkl}$, where d_0^{hkl} is the measured lattice spacing.





RESULTS

By analysing the positions of available Bragg reflexes the corresponding crystallite lattice plane spacing, dhkl, was determined by the centre of gravity method, which takes into consideration the upper 50 % of the peak heights. The results are presented in Figs. 6-8.

Fangential component

× [310] ● [222]

200

φ, [°]

scattering vector, Q, along the tangential

⊕ [321] ☆ [330]/[411]

300

400

the unevenness of the disk
+ [110] △ [211] × [31
 \bigcirc [200] □ [220] ● [22

100

vector of the disk.

1



Fig. 6. The lattice strain, $\Delta^{hkl}(\varphi)$, for the scattering vector, O, along the radial vector of the disk.



Fig. 8. The lattice strain, $\Delta^{hkl}(\varphi)$, for the scattering vector, Q, parallel to the $\pm 45^{\circ}$ direction in the local coordinate system (x', y').

4

A striking feature of all $\Delta^{hkl}(\phi)$ is their correlation with disk unevenness. The obtained strain variations for different angles, φ , as well as for different local orientations (angle φ ') of the scattering vector are rather strong. In addition, $\Delta^{hkl}(\phi)$ shows a pronounced anisotropic effect.

In Fig. 9 the internal strain as a function of the anisotropy factor is presented. The irregular behaviour of $\Delta^{hkl}(\Gamma,\phi)$, points to a strong influence of plastic anisotropy and large stresses due to grain interaction (microstresses - see [5], [6]), a linear dependence would have been caused by elastic anisotropy. Microstrains, which are related to the line widths of Bragg reflexes, do not show any significant dependence on the angle φ or on Γ . Only stochastic fluctuations seem to be present (Fig. 10).



Fig. 9. The dependence of the radial and tangential components of the internal strain on the anisotropy factor $\Gamma = (h^2 l^2 + h^2 k^2 + k^2 l^2)/(h^2 + k^2 + l^2)^2$

Here, a remark on strain measurement errors should be made. The first source of errors is disk positioning, for which a relatively simple facility was used. The position of the observation point on the disk relative to the neutron beam can be reproduced with an accuracy of up to 0.5 mm. To estimate scattering peak shifts within this accuracy, a measurement was performed with the disk shifted by 2 mm in a direction perpendicular to the disk plane. As a result, a relative change in the scattering peak positions of about 2.4-10-3 was obtained, and the positioning error was determined to be 6.10-4. It should be emphasised that repeated measurements for one and the same observation point where disks were placed in turn have yielded peak shifts of the same order of magnitude as the estimated error. The second problem in the precise determination of lattice spacings is connected with the large gauge volume. The difference in neutron flight paths from the upper and lower border of the gauge volume results in a theoretical peak shift of about 5 10⁻³. This mainly affects the broad

line shapes in Fig. 10. Measurements with the annealed powder contained in tubes 5 mm in diameter and 5 cm in height yielded smaller line widths. Finally, statistical errors connected with the RTOF technique should be considered. As the resolution of the diffractometer is 10^{-3} , these errors are not crucial. Further improvement is expected as soon as the 90° scattering geometry with collimators and a neutron scanner can be used.





DISCUSSION

Actually, these results do not permit a quantitative analysis of stress [5]. Further investigations, i.e., measurements of the $\sin^2\psi$ -dependence of $\Delta^{hkl}(\psi,\phi)$ are necessary. To acquire experience in analysing the measured strain data, the following evaluation procedure was performed. The least squares method was used to calculate components of the strain tensor:

$\epsilon^{hkl} = (\epsilon^{hkl}(x', x'), \epsilon^{hkl}(y', y'), \epsilon^{hkl}(x', y'))^T$

where T means the transposed form. For this purpose, the measured $\Delta_i^{hkl}(\phi)$ are collected to form a 4-dimensional vector:

 $\Delta^{hkl} = (\Delta_1^{hkl}(\phi), \Delta_2^{hkl}(\phi), \Delta_3^{hkl}(\phi), \Delta_4^{hkl}(\phi))^T,$

where i denotes the different directions of Q in the (x',y') coordinate system: $i=1 - \phi'=45^{\circ}$, $i=2 - \phi'=-45^{\circ}$, $i=3 - \phi'=0^{\circ}$, $i=4 - \phi'=90^{\circ}$. Then, the strain tensor ϵ^{hkl} satisfies the equation:

$$\Delta^{hkl} = A \star \epsilon^{hkl}$$

The line of the A matrix is defined as:

 $A_i = (m_i^2, n_i^2, n_i m_i),$

6

where $m_i = \cos(\varphi_i)$ and $n_i = \cos(90^\circ - \varphi_i)$ are the direction cosines of Q. According to the least squares fit procedure, ϵ^{hkl} can be obtained by the formula:

 $\epsilon^{hkl}=(A^T\star A)^{-1}\star A^T\star \Delta^{hkl}$ The relative fitting error can be estimated by the following formula:

$\delta \epsilon_r^{hkl} = [(A * (A^T * A)^{-1} * A^T - I) * \Delta^{hkl}] / |\Delta^{hkl}|.$

The principal strain, $\varepsilon^{D,hkl}$, and the principal strain axis can be determined by diagonalizing the strain tensor. Then, under the assumption of an elastic model, the components of the principal stress tensor are related as:

$$\sigma_{dd}^{D} = 2 G^{hkl} \epsilon_{dd}^{D,hkl} + \lambda^{hkl} \sum_{d=1}^{3} \epsilon_{dd}^{D,hkl},$$

where $G^{hkl}=E^{hkl}/2(1+\mu^{hkl})$ and $\lambda^{hkl}=\mu^{hkl}E^{hkl}/(1-2\mu^{hkl})(1+\mu^{hkl})$. The third component $\varepsilon_{33}^{D,hkl}$, which has not been considered so far, can be derived from the condition that $\sigma_{33}^{D}=0$. Unfortunately, the X-ray elastic constants are not known for the steel used to make disks. Because the investigated material is nearly isotropic in our case, the values of G^{hkl} and λ^{hkl} were estimated on the basis of those for bulk material which were determined by the manufacturer as G=70 GPa and λ =104 GPa.

As shown in Fig. 9 the disk material does not belong to a pure elastic region. Hence, in a strict sense, the above formula cannot give a correct answer about actual residual stresses in the disk, but σ_{dd}^{D} can be estimated by this formula if the effective elastic constants are used. The calculations were performed for the data sets obtained from several [hkl]-reflexes at the observation points $\varphi=0^{\circ}$, 90°, 180°. Table 1 contains the results of a least squares fit for the [222]-reflex, i.e. the measured strain, Δ , the strain tensors ε , the principal strains ε^{D} , the relative error $\delta\varepsilon_{r}$ of the fit, and the principal stresses σ_{dd}^{D} along the principal strain axes rotated through the angle $\delta\varphi'$ in the local system (x',y'). The ε_{33}^{D} and $\varepsilon_{33}^{D,exp}$ quantities are the theoretical ($\sigma_{33}^{D}=0$) and measured normal strain components, respectively.

The obtained results look quite reasonable, though the fitting errors are relatively large. The situation similar to that illustrated in Table 1 also takes place for other [hkl]-reflexes. It would be interesting to investigate whether the errors of the fit can be reduced by increasing the number of observation points. Repeat measurements with better statistics are planned.

The calculated stress values are also rather large. Stresses along the rolling direction are noticeably larger than at the yield point $R_{p0.2}$, but the tensile stress is still smaller than the tensile strength R_m . In our opinion, large fluctuations of lattice strains for different lattice planes (see Fig. 9) nevertheless point to the fact that the calculated stress values not only reflect the existence of macro-

scopic residual stresses (1st kind residual stress), but are also caused by residual stress of the 2nd kind [6].

			Table 1	
[222]-reflex of Fe	φ=0°	φ=90°	φ= 18 0°	
Δ [10-4]	(-16.6, -13.4, -15.5, 10.1)	(-6.2, 8.0, -2.0, 22.3)	(-16.7, -3.2, -15.5, 13.6)	
ε [10-4]	(-21.6, 4.0, -1.6)	(-6.6, 17.7, -7.1)	(-20.0, 9.2, -6.8)	
$(\epsilon_{11}^{D}, \epsilon_{22}^{D}) [10^{-4}]$	(-21.8, 4.1)	(-8.5, 19.6)	(-21.5, 10.6)	
δε, [10-4]	0.44	0.38	0.34	
$(\sigma_{11}^{D}, \sigma_{22}^{D})$ [MPa]	(-410, -48)	(-53, 340)	(-365, 84)	
δφ' [°]	-4	-15	-12	
$\epsilon^{\rm D}_{33}/\epsilon^{\rm D,exp}_{33}$	7.5 / 2.3	-6.8/-4.7	4.8 / not measured	

Rolled disk textures have never been investigated in detail before. Now, first results on diffraction pole figures obtained for a test specimen of disk material are available. Only weak textures were found (the maximal is 1.53 times larger than the random value, equal to about 0.86). The effect of texture can also be estimated from the RTOF spectra using the Rietveld refinement. For ideal isotropic powder samples, systematic deviations of the measured intensities of the Bragg peaks from the Rietveld fit results give information about the texture (Fig. 11). The inclusion of textures in the Rietveld refinement by using a suitable texture model [7] is also planned. Then, in addition, Rietveld refinement can be applied to average strain data over different diffraction planes [hkl]. The averaged lattice strain can be calculated as $\Delta = (a - a_0)/a_0$, where **a** is the fitted lattice constant.



Fig. 11. The influence of the preferred orientation on the peak intensities.

Finally, a first comparison of the neutron diffraction data with the results obtained by the ultrasonic and magnetic methods can be made. In Fig. 12 the difference between the radial and tangential stress components calculated by the formula $\Delta \sigma^{R-T} = \sigma^R - \sigma^T = 2G(\Delta_R^{211} - \Delta_T^{211})$ is shown together with the corresponding change in the velocity of SH-waves Δv^{R-T} (the frequency is 800 kHz, the transmitter-to-receiver distance is 30 mm), and the Barkhausen noise amplitude, ΔM_{max}^{R-T} , measured in ac fields (50 Hz, 10 A/cm) over the frequency range of 10 - 30 kHz. The coercive field strength. HCM. for the mentioned frequency region does not yield a significant φ -dependence, which is evidence of a weak texture influence in the considered disk.



Fig. 12. Comparison of the angular dependence of the residual stress, σ , determined from the [211]-reflex contained in the neutron diffraction data with the results on the ultrasonic noise amplitude, M_{max}. For all cases the difference between the radial and tangential comz, is also shown.

Despite the fluctuations, a correlation of the estimated stress values with $\Delta \mathbf{v}^{R-T}$ and $\Delta \mathbf{M}^{R-T}$ can be detected. Furthermore, angular dependencies of these quantities are connected in an obvious way with the disk unevenness, z. Hence, both residual stresses and disk unevenness influence the result of the forming process, i.e., folds should appear at the disk positions for which significant changes in these quantities are observed. We do not know yet what the forming procedure will re-⁴⁰⁰ sult in, but non-regular behaviour can be expected for the angles $\phi = 180^{\circ}$ and 270° .

The existing deviations in the angular dependence of the three parameters $\Delta \sigma^{R-T}$, Δv^{R-T} and velocity, v, of SH-waves and the Barkhausen ΔM_{max}^{R-T} , could have several origins. First of all is inhomogenous microstructure. The averaging proponents is drawn. The unevenness of the disk, cedure over different measuring volumes might also be responsible for that. For instance, the in-

spection depth of the magnetic method is on the order of 0.3 - 0.5 mm only, and the magnetic results cannot be in correspondence with the bulk properties for any case.

8

OUTLOOK

At the present stage of the investigation it is not possible to draw any final conclusions yet. Strain measurements and accompanying ultrasonic as well as magnetic testing of more disks are planned. These results will be reported in a forthcoming paper. Now, we are working to better identify the effect of texture on the measured ultrasonic and magnetic quantities. For this purpose improvement of the Rietveld refinement for neutron diffraction data is foreseen. Then a position resolved texture map could be drawn by the RTOF-technique. This map would be used as a starting point for the interpretation of ultrasonic and magnetic measurements. Consequences of the forming process, however, can be discussed only after the behaviour of the formed disks is known.

The disadvantages of strain measurements with a back scattering detector will be largely reduced as soon as the two detectors at the angles of $2\theta = \pm 90^{\circ}$ are installed on the HRFD. This will be done in the frame of the NIDA project - <u>N</u>eutron <u>I</u>nvestigation of <u>D</u>eformation for <u>A</u>pplication [8]. Each detector will have twenty ⁶Li -glasses and two moveable multi-slit collimators in front it, which together with a system of diaphragms in the primary neutron beam will create a gauge volume of about $2 \times 2 \times 2$ or $0.7 \times 2 \times 2$ mm³ in the investigated sample.

At present a prototypes of detector consisting of five ⁶Li-glasses and multi-slit collimator are installed at the diffractometer. Adequate sample positioning will be accomplished by the linear neutron scanner now being manufactured (three orthogonal movement modes and one rotational mode around the vertical axis). Later, a tensor scanner with a sufficient number of modes to measure the total strain tensor will be installed.

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Аксенов В.Л. и др. Оценка остаточных напряжений в холоднокатанных стальных дисках из измеренных деформаций на фурье-дифрактометре высокого разрешения

Представлены результаты оценки остаточных напряжений в холоднокатанных стальных дисках по измерениям на фурье-дифрактометре высокого разрешения. Измерения были сделаны для калибровки магнитных и ультразвуковых экспериментов, проведенных в Институте неразрушающих методов контроля (Саарбрюкен, Германия). Объектом изучения были холоднокатанные стальные диски толщиной 2,5 мм и диаметром около 500 мм, которые используются для изготовления сосудов высокого давления. Дифракционные эксперименты проводились при угле рассеяния $2\theta = +152^\circ$ с разрешением $\Delta d/d = 1,5 \cdot 10^{-3}$. Измеряемый объем был выбран в соответствии с разрешением магнитных измерений 20×20 мм². В ближайшем будущем планируются эксперименты по дифракции нейтронов на холоднокатанных стальных дисках при углах рассеяния $2\theta = \pm90^\circ$. Также предполагается ввести алгоритм анализа текстуры в процедуру подгонки по методу Ритвельда для более точного вычисления остаточных напряжений в холоднокатанных материалах.

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Aksenov V.L. et al. Estimation of Residual Stress in Cold Rolled Iron-Disks from Strain Measurements on the High Resolution Fourier Diffractometer

The results of estimating residual stresses in cold rolled iron disks by measurements with the high resolution Fourier diffractometer (HRFD) at the IBR-2 pulsed reactor are presented. These measurements were made for calibration of magnetic and ultrasonic measurements carried out at the Fraunhofer-Institute for Nondestructive Testing In Saarbrucken (Germany). The tested objects were cold rolled steel disks of 2.5 mm thickness and diameter of about 500 mm used for forming small, gas pressure tanks. Neutron diffraction experiments were carried out at the scattering angle $2\theta = +152^{\circ}$ with resolution $\Delta d/d = 1.5 \cdot 10^{-3}$. The gauge volume was chosen according to the magnetic measurements lateral resolution $20 \times 20 \text{ mm}^2$. In nearest future the neutron diffraction measurements with cold rolled iron disks at the scattering angle $2\theta = \pm90^{\circ}$ are planned. Also the texture analysis will be included in the Rietveld refinement procedure for more correct calculation of residual stress fields in the cold rolled materials.

The investigation has been performed at the Frank Laboratory of Neutron Physics, JINR.

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