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FINAL REPORT ON THE START PROGRAMME

Calculation of dislocation density for high-strength steel using neutron diffraction

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Abstract

This work is devoted to the calculation and comparison of dislocation density values obtained by the Williamson-Hall (WH) and Halder-Wagner (HW) methods from neutron diffraction data. The dislocation density estimation is important for understanding the physical and mechanical properties of metals - strength and ductility. The calculation was carried out using two methods. The Williamson-Hall (WH) and Halder-Wagner (HW) methods were used to determine the size of crystallites and micro strain by broadening the profile of diffraction lines obtained after analyzing neutron diffraction data. The application of the methods is demonstrated on the example of the dislocation density changes in wear-resistant steel B1700 in the quenched state and after tempering within the temperature range 150÷400 °C. According to the results of the study it is established that the value of dislocation density decreases with increasing tempering temperature. The comparison of dislocation density values obtained by different methods is given.

Key words: neutron diffraction, dislocation density, Williamson-Hall method, Halder-Wagner method, crystallite size, micro strain.

1. Introduction

The analysis of neutron diffraction data is a powerful tool for determining the following structural and microstructural characteristics such as: lattice parameter, coherent scattering region (CSR) size, lattice strain, mass fractions (multiphase samples), macro-stresses, dislocation density, etc.

In this paper we calculate the dislocation density by means of determining the lattice microdistortions and coherent scattering region (CSR) in wear-resistant steel B1700 in the quenched state and after tempering at different temperatures $(T = 150, 250, 400\,^{\circ}C)$ from neutron diffraction data obtained on a diffractometer installed at the pulse IBR-2 Reactor (pulse fast reactor) in JINR (Joint Institute for Nuclear Research), Dubna.

The purpose of this work: calculation and comparison of dislocation density values obtained by Williamson-Hall (WH) and Halder-Wagner (HW) methods for determination of crystallite sizes and micro strain by broadening of diffraction line profiles obtained as a result of neutron diffraction data analysis for wear-resistant steel mark B1700 in the quenched state and after tempering in the temperature range 150÷400 °C.

To achieve the purpose, the following **objectives** need to be addressed:

1. Calculate the dislocation density on the example of wear-resistant steel grade B1700 in the quenched state and after tempering at temperatures of 150, 250 and 400 °C by the WH and HW method from the neutron diffraction results obtained by imaging the sample in all directions;
2. Demonstrate the distribution of dislocation density values on the sphere according to the neutron diffraction results obtained by imaging the sample in all directions on the example of wear-resistant steel grade B1700 in the quenched state and after tempering in the temperature range 150÷400 °C;
3. To compare the values of dislocation densities obtained by two methods on the sphere, on the total spectrum and average values of distribution on the example of wear-resistant steel grade B1700 in the quenched state and after tempering in the temperature range 150÷400 °C.

The relevance of this work lies in the fact that the dislocation density largely determines the most important mechanical characteristics of metals - strength and plasticity. In addition, neutron diffraction allows us to demonstrate the distribution of dislocation density values on the sphere when imaging the sample in all directions.

In real metals there are always deviations from the ideal order in the arrangement in the crystal structure. According to the geometrical factor, structural defects can be subdivided into point, linear and surface defects.

Linear defects are called dislocations. In turn, the appearance of dislocations is caused by the presence in some parts of the crystal of 'extra' atomic half-planes, called extraplanes. Extra planes are formed during crystal growth or as a result of plastic deformation. Figure 1 schematically shows the formation of an edge dislocation.

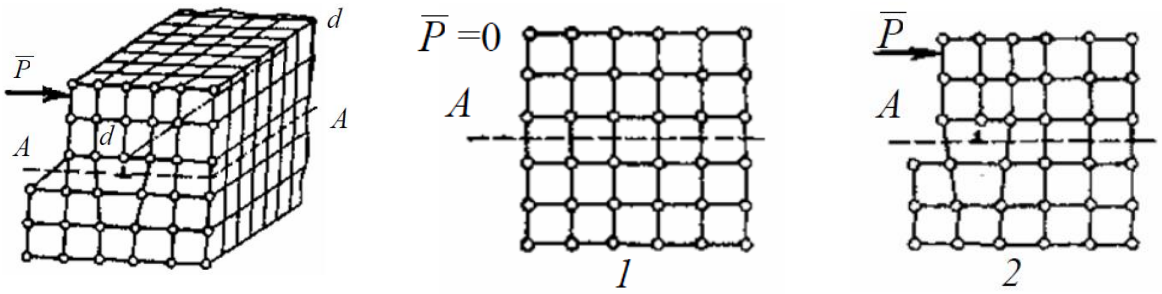

Fig 1. Edge dislocation generation

It can be seen that the applied force caused a partial shift of the upper part of the crystal relative to the lower part (AA - sliding plane) - an extra plane was formed. The line along and near which there is a violation of the correct and periodic arrangement of the atomic planes of the crystal is called a dislocation line. The dislocation structure of a material is characterized by the dislocation density, which in a crystal is defined as the average number of dislocation lines crossing an area of 1m^2 inside the body, or as the total length l of dislocation lines in a volume V of 1m^3:

$$\rho = \sum l/V \qquad (1)$$

Dislocation density largely determines the strength and ductility of metals.

With the help of neutron diffraction, it became possible to study microstructures by broadening of the diffraction peak profile. The broadening of the profile can be influenced by both micro strain of the crystal lattice and the size of the coherent scattering region. The diffraction peak profile for a sample with small crystallite size and lattice deformation will be a combination of profiles caused by these factors. It follows that an important task is to separate the profile into its components.

The profile broadening due to the size of the coherent scattering region obeys the $1/\cos\theta$ function, while the profile broadening due to lattice micro strains has a $\tan\theta$ dependence [1]. These dependencies allow us to separate these two effects in diffraction data over a wide range of 2θ. Two different methods for determining the dislocation density from neutron diffraction data will be discussed below.

2. Materials and methods of research

The chemical composition of the investigated high-strength medium carbon steel grade B1700 is presented in Table 1 [2]:

Table 1: Chemical composition of the investigated steel, %.

C	Si	Mn	Cr + Mo	Ni + Cu	Ti + V + Nb	Al
0.45	0.36	1.13	1.66	1.3	0.11	0.04

The research using neutrons was carried out on a diffractometer installed at the IBR-2 Reactor (pulsed fast reactor) at JINR (Joint Institute for Nuclear Research), Dubna [3]. The neutron texture diffractometer SCAT [4 - 6] was used to reveal the phase composition. Three samples were investigated (tempering temperatures: 150, 250, 400 °C).

Table 2. Main characteristics of SCAT

Distance retarder - sample	104 m
Neutron flux on the sample	~ $5 \cdot 10^5$ neutrons / cm^2 / s
Wavelength range	max – 7 Å (14.6 Å when using a beam interrupter)
Interplanar distance range	max. – 5.1 Å (10.2 Å when using a beam interrupter)
Resolution, $\Delta d/d$	3.1 – 6.2 10^{-3}

To solve the problem of separating the contributions to the diffraction peak broadening of crystallite size and lattice micro strains, two methods have been developed: the Williamson-Hall method [7] and the Halder-Wagner method [8,9]. With these methods, the coherent scattering areas (D) and micro-distortion areas (ε) can be easily determined from the integral widths (β), which can be defined as the peak area divided by the peak intensity. Diffraction data obtained by neutron or X-ray diffraction are good enough to obtain reliable values of (D) and (ε).

2.1. Williamson - Hall method

Crystallites smaller than about 1 μm contribute to the profile broadening. The integral width (in radians), β_D, due to the influence of small crystallites, is related to D using the Scherrer equation [10],

$$\beta_D = \frac{K\lambda}{D\cos\theta}, \tag{2}$$

where λ is the wavelength of radiation, K is the Scherrer constant (for the sample considered in this work it can be taken as 1), θ is the diffraction angle. In this work, for convenience, the spectrum obtained from each detector was converted into the dependence of intensity on the angle θ (in radians).

The effect of lattice strains, ε, on the profile broadening can be obtained by differentiating Bragg's law,

$$n\lambda = 2d\sin\theta, \tag{3}$$

where $n = 1$, d is the interplanar distance. After differentiation with respect to θ (in radians):

$$\Delta\theta = -\frac{\Delta d}{d}\tan\theta = -\varepsilon\tan\theta. \tag{4}$$

where the broadening of the profile is due to micro strains, $\varepsilon = \Delta d/d$ (d is the interplanar distance), proportional to $\tan\theta$. Stokes and Wilson showed that the integral broadening β_ε, arising due to lattice microstrain, is related to the micro strain value as follows [11],

$$\beta_\varepsilon = 4\varepsilon\tan\theta. \tag{5}$$

Williamson and Hall [7] introduced a simple approximation that the integral width, β, due to both small crystallite sizes and micro strains, is simply the sum of the two components β_D and β_ε:

$$\beta = \beta_D + \beta_\varepsilon. \tag{6}$$

After substituting equations (2) and (5) into (6), we obtain relation (7):

$$\beta = 4\varepsilon \tan\theta + \frac{K\lambda}{D\cos\theta}. \quad (7)$$

Let us reduce equation (7) to the form (8):

$$\frac{\beta\cos\theta}{\lambda} = 4\varepsilon\frac{\sin\theta}{\lambda} + \frac{K}{D}. \quad (8)$$

Equation (9) is considered as a straight line of the form $y = ax + b$. The graph whose abscissa is $x = \sin\theta$, and ordinate is $y = \beta\cos\theta$, is called the Williamson and Hall (WH) graph, Williamson and Hall [7] proposed this method in 1953. Hall was the first to report the idea in 1949 [12]. The tangent of the angle of slope of a straight line is 4ε, and its intersection with the ordinate axis is $K\lambda/D$. K is the Sherrer constant, assumed to be equal to 1 [13].

2.2. Halder - Wagner method

To determine D and ε, Halder and Wagner [8, 9] proposed an alternative equation that contains the integral width of the diffraction peak, β^*, and the lattice plane spacing, d^*, for the corresponding cell:

$$\left(\frac{\beta^*}{d^*}\right)^2 = \frac{K}{D}\frac{\beta^*}{\left(d^*\right)^2} + (2\varepsilon)^2 \quad (9)$$

In equation (9):

$$\beta^* = \frac{\beta\cos\theta}{\lambda}, \quad (10)$$

$$d^* = \frac{2\sin\theta}{\lambda}. \quad (11)$$

Equation (9) can be written taking into account (10) and (11) in the following form:

$$\left(\frac{\beta}{\tan\theta}\right)^2 = \frac{K\lambda}{D}\frac{\beta}{\tan\theta\sin\theta} + 16\varepsilon^2. \quad (12)$$

Equation (12) has the form of a straight line, $y = ax + b$, similar to equation (8). In the Halder-Wagner (HW) plot, the ordinate takes the values $y = \left(\beta/\tan\theta\right)^2$ is postponed with respect to the abscissa $x = \beta/\left(\tan\theta\sin\theta\right)$. Then, the slope and the

point of intersection with the axis of the resulting line take the values of $K\lambda/D$ and $16\varepsilon^2$ respectively. As described in the previous section, the value of $K=1$ is considered to be fair in determining the value of CSR [13].

2.3. Calculation of dislocation density

The formula proposed by Murugesan [14] is used to calculate the dislocation density. The dislocation density ρ can be decomposed into two components:

$$\rho = \sqrt{(\rho_D \cdot \rho_\varepsilon)}, \tag{17}$$

where ρ_D and ρ_ε are the components that arise due to crystallite size and micro strains, respectively:

$$\rho_D = \frac{3}{D^2}; \tag{18}$$

$$\rho_\varepsilon = \frac{k\varepsilon^2}{b^2} = \frac{k\varepsilon^2\sqrt{2}}{a^2}. \tag{19}$$

$k=14.4$ is the material constant, b is the Burgers vector (assumed to be 0.25 nm), a is the lattice parameter.

3. Results

Three diffraction lines (110), (220), and (211) were chosen to calculate the values of coherent scattering regions, micro strains, and their corresponding dislocation densities. The choice of these lines is conditioned by the values of relative errors for the corresponding points.

Table 3: Distribution of dislocation density values on the sphere for different temperatures, m^{-2}

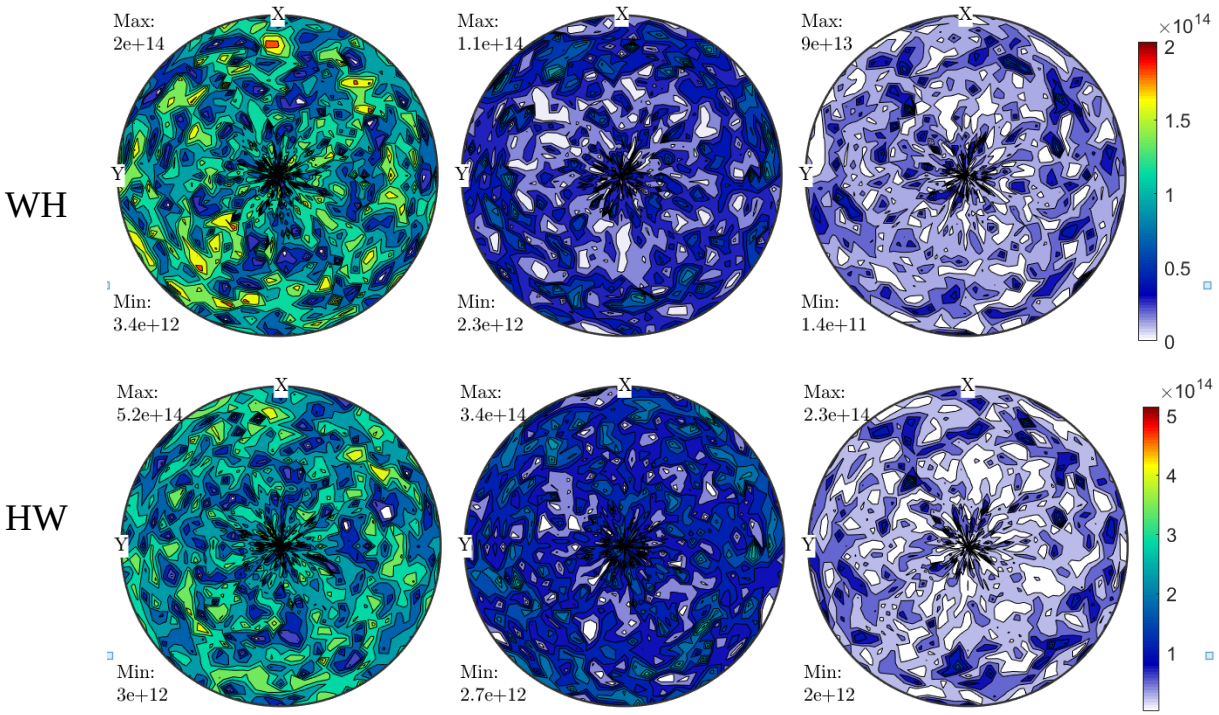

Table 3 shows the distribution of dislocation density value on the sphere when the sample is imaged in all directions by WH and HW methods at different tempering temperatures after quenching of B1700 steel: 150, 250 and 400 °C. The values of minimum and maximum are marked to the left of the sphere . It can be seen that the maximum and minimum values decrease with increasing temperature with each method. In addition, the maxima characterizing these states calculated by the HW method are higher than their corresponding values calculated by the WH method, while the minima are smaller. In other words, the Halder-Wagner method gives a greater spread of values than the Williamson-Hall method.

Table 4 shows the dislocation density values: the minima and maxima of the distribution on the sphere, the average value over the sphere, and the values over the total spectrum.

Table 4: Dislocation density values on the sphere, average on the sphere and on the total spectrum, 10^{14} м$^{-2}$

	On the sphere				Average value on the sphere		On the total spectrum	
	min		max					
	WH	HW	WH	HW	WH	HW	WH	HW
150 °C	0.03	0.03	2.03	5.15	1.03	2.54	1.44	2.75
250 °C	0.02	0.03	1.14	3.36	0.34	1.20	0.89	1.85
400 °C	0.001	0.02	0.90	2.28	0.19	0.45	0.45	0.77

For clarity, the data from Table 4 are plotted in Figures 2a and 2b where ρ_{min} and ρ_{max} are the maximum and minimum values of the dislocation density on the sphere for each temperature, respectively, $\langle\rho\rangle$ is the average value on the sphere for each state, and ρ_{sum} is the dislocation density calculated from the total spectrum.

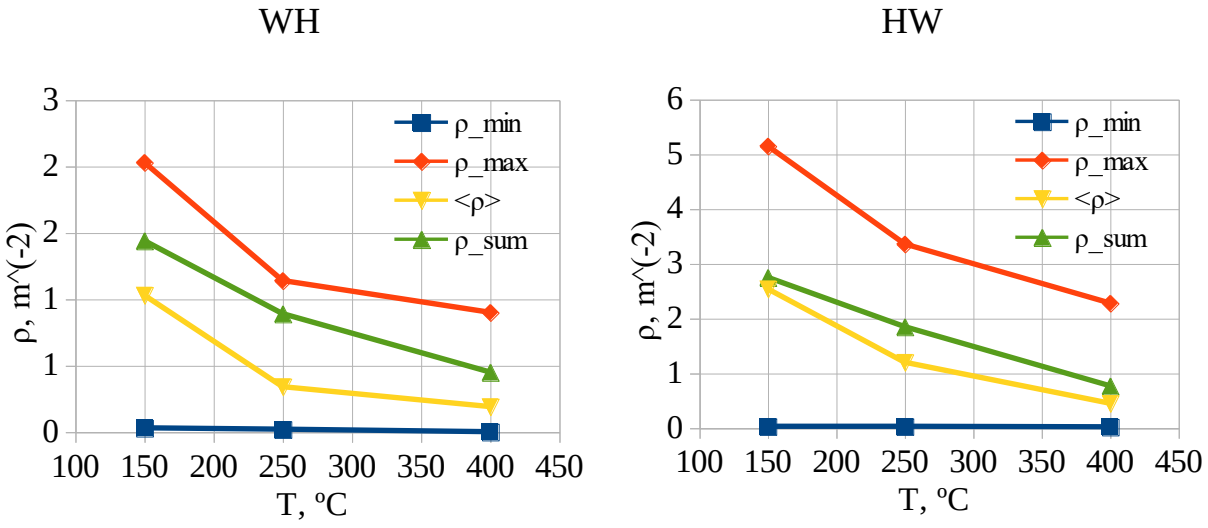

Fig. 2. Dependence of dislocation density on tempering temperature: a - Williamson-Hall method; b - Halder-Wagner method. All dislocation density values are given in 10^{14} m^{-2}

Figures 2a and 2b show:

1. in all cases the value of dislocation density calculated by the HW method is higher than the value obtained by the WH method, the difference

between them decreases with increasing temperature and the dislocation density decreases;

2. in the two methods, the values of dislocation densities calculated from the total spectrum are located in the region of values bounded by the lines ρ_{min} and ρ_{max} ;
3. values obtained by the HW method for the sphere mean and total spectrum have better convergence compared to the WH method.

4. Conclusions

In this paper we have calculated dislocation density by means of determining lattice micro strains and coherent scattering region (CSR) in wear-resistant steel B1700 in the quenched state and after tempering at different $(T = 150, 250, 400\,^{\circ}C)$ temperatures using neutron diffraction data. The dislocation density was calculated using the Williamson-Hall (WH) and Halder-Wagner (HW) methods to determine crystallite sizes and micro strains by broadening of diffraction line profiles. In paragraph 3 of this paper, the results are described and a comparison of the values obtained by the different methods is given.

The WH and HW methods are the fastest and most convenient for determining the type of broadening of the diffraction peak profile and, consequently, allow the estimation of dislocation density values that allow the strength and ductility of metals to be assessed.

It is established that the value of dislocation density decreases with increasing tempering temperature. The comparison of dislocation density values obtained by different methods shows that the two methods give close results.

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