Materials Characterization 134 (2017) 387–397

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Characterizing dislocation configurations and their evolution during creep of a new 12% Cr steel

Surya Deo Yadav[a,*], Moustafa El-Tahawy[b,c], Szilvia Kalácska[b], Mária Dománková[d], David Canelo Yubero[e], Cecilia Poletti[e]

[a] Mechanical Metallurgy Division, Indira Gandhi Centre for Atomic Research, India
[b] Department of Materials Physics, Eötvös Loránd University, Pazmany Péter s. 1/A, H-1117 Budapest, Hungary
[c] Department of Physics, Faculty of Science, Tanta University, 31527 Tanta, Egypt
[d] Institute of Materials Science of MTF STU, Trnava, Slovakia
[e] Institute of Materials Science and Welding, Graz University of Technology, Graz, Austria

ARTICLE INFO

Keywords:
Creep
Dislocations
Subgrains
XRD
EBSD
TEM

ABSTRACT

In this research, different types of dislocations are characterized and quantified in a newly developed 12% Cr steel in as-received as well as in crept conditions. X-ray diffraction (XRD) patterns are analyzed using three different models. The dislocation densities are determined employing the Convolution Multiple Whole Profile (CMWP) fitting, the analysis of the asymptotic part of the X-ray line profile (Groma's method), and the Rietveld refinement coupling Popa's and Williamson-Smallman approaches. CMWP method is used to measure the internal dislocation density inside the subgrains consisting of mobile and dipole dislocations, and the results show good agreement with Transmission Electron Microscopy (TEM) measurements. Groma's method is used to evaluate the total dislocation density, with results comparable to those obtained from the combination of Electron Backscatter Diffraction (EBSD) and TEM. Rietveld method gives the internal dislocation density or mobile dislocation density depending on the model chosen. The experimentally determined results such as creep strain, dislocation densities and subgrain size are compared with theoretical predictions and they are found in good agreement.

1. Introduction

The dependency on thermal power plants will still remain for many years to fulfil the growing power demand in many part of the world [1]. Thermal power plants require large amount of materials with good high temperature properties for constructing different components. 9–12% Cr steels are used to fabricate many critical components of these power plants [1–3]. These steels with martensitic/ferritic structure are appropriate candidates for super heater tubes, boiler tubes, large forgings and accelerator-driven system (ADS), due to their low cost and good high temperature properties [1–10]. They combine creep strength and oxidation resistance up to a certain extent [8]. The increment in steam temperature and pressure are directly related to the increment of the efficiency. This aspect is essential not only from the economic, but also from the ecological point of view [1,11–13]. The power plant components should have desired properties to resist this increased temperature and pressure for long term without breakage.

In tempered condition, also frequently designated as as-received condition, the microstructure of the martensitic/ferritc steels comprises of different interfaces such as: prior austenitic grain boundaries (PAGBs), block boundaries, packet boundaries, lath boundaries and subgrain boundaries. These boundaries are decorated with $M_{23}C_6$ carbides while MX type carbonitrides are distributed throughout the matrix [4,7,10,14–16]. The substructure is considered as formed by different dislocations types. According to the configurations and stress fields of the dislocations, they are classified into mobile, dipole and boundary dislocations [17–19]. The mobile and dipole dislocations residing inside the subgrain interior are also referred as statistically stored dislocations (SSDs) and boundary dislocations as geometrically necessary dislocations (GNDs).

For the further development of new alloys those are resistant to creep at severe conditions, it is necessary to understand both, the response of the material under high temperatures loading and the governing creep mechanisms. The development process can be more efficient if it is coupled with physical based modelling [20]. Physical models of metallurgical processes such as creep deformation, rolling, forging and extrusion, require experimental data based on microstructural features. These features can be different types of dislocation

* Corresponding author.
E-mail addresses: suryadeo4514@gmail.com, surya1587@igcar.gov.in (S.D. Yadav).

https://doi.org/10.1016/j.matchar.2017.11.017
Received 21 May 2017; Received in revised form 3 November 2017; Accepted 5 November 2017
Available online 06 November 2017
1044-5803/

densities such as: mobile (ρ_m), dipole (ρ_{dip}) and boundary (ρ_b), and subgrain size (R_{sub}). In this way, for example, creep strain rate is considered as a function of different variables, as discussed in [19]:

$$\dot{\varepsilon} = f(\rho_m, \rho_{dip}, \rho_b, R_{sub}, \sigma, T, r_m, N_v) \quad (1)$$

where σ is the applied stress, T is the temperature, r_m is the mean radius of precipitates and N_v the number density of precipitates. Therefore, the numerical values of the internal variables are needed in order to develop, validate and calibrate the models [17–27].

In this regard, the precise characterization and quantification of different dislocations inside the substructure is necessary. In the last ten years significant amount of research has been done and the results of the measurement of dislocation densities were published applying different techniques, such as XRD, EBSD and TEM [17,28–36]. On the other hand, comments on the particular dislocation configuration measured in this type of steels are very rare [17,28]. Thus, it is necessary to find out the applicability of XRD models in combination with TEM and EBSD for the measurement of specific dislocation density.

In present research work we discuss the three different XRD models to evaluate the dislocation densities. The obtained dislocation densities from XRD models were compared with those obtained using complementary TEM and EBSD methods. Finally, experimental results such as dislocation densities, subgrain size and creep strain are compared with theoretical predictions based on a hybrid creep model [19].

2. Theoretical Background of X-ray Line Profile Analysis

The measured X-ray diffraction pattern $P(2\theta)$ can be represented as the Fourier series in reciprocal space, as a function of the angular factor K, the number of unit cells N in the sample and a structure factor F [37]:

$$P(2\theta) = \frac{KNF^2}{\sin^2\theta} \sum_{n=-\infty}^{n=+\infty} (A_n \cos(2\pi nh) + B_n \sin(2\pi nh)) \quad (2)$$

where $h = 1/d$, d being the distance between the lattice planes, and n is harmonic number. The real part of the Fourier co-efficient A_n is the result of two effects: the lattice strain A_n^S and the size effect A_n^D:

$$A_n = A_n^S A_n^D \quad (3)$$

The X-ray diffraction patterns contain the enormous amount of information related to the microstructure of a material at a given condition. Different models have been used to extract the microstructural information such as lattice strain, crystallite size and dislocation density analyzing the X-ray line profile. As our focus is the measurement of different dislocation densities, the most commonly used methods by metallurgical scientists are Groma's method [38], CMWP fitting [39] and Williamson-Smallman approach [40–44]. The basic concepts behind these three methods are discussed briefly in the following subchapter.

2.1. Groma's Method

Groma et al. [38] have shown that the intensity distribution $I(q)$ of the tail of XRD pattern follows the function:

$$I(q) = \Lambda\langle\rho\rangle \frac{1}{|q|^3} - f\left(\frac{q}{|q|^5}\right)... \quad (4)$$

The k^{th}order moment of the intensity distribution $I(q)$ is expressed as:

$$m_k = \int_{-\infty}^{+\infty} q^k I(q)dq \Big/ \int_{-\infty}^{+\infty} I(q)dq \quad (5)$$

The moment can also be evaluated from the Fourier transformation of the intensity distribution $A(n)$ as:

$$m_k = (i)^k \frac{1}{A(0)} \frac{d^k}{dn^k} A(n)\Big|_{n=0} \quad (6)$$

From Eq. (5) restricted moment or k^{th}order variance, $v_k(q')$ can be evaluated applying the finite integration limits as:

$$v_k(q') = \int_{-q'}^{+q'} q^k I(q)dq \Big/ \int_{-\infty}^{+\infty} I(q)dq \quad (7)$$

From Eqs. (4)–(7) the second order restricted moment $v_2(q)$ and fourth order restricted moment $v_4(q)$ are derived as:

$$v_2(q) = \Lambda\langle\rho\rangle \ln\left(\frac{q}{q_0}\right) \quad (8)$$

$$v_4(q) = \Lambda\langle\rho\rangle q'^2 + 12\Lambda^2\langle\rho^2\rangle \ln\left(\frac{q}{q_2}\right)\ln\left(\frac{q}{q_3}\right) \quad (9)$$

Eqs. (8) and (9) can be used to evaluate the total dislocation density inside the material, by determining the parameter Λ that defines the contrast effect of dislocations. The model was applied for the evaluation of total dislocation density in many different alloys [45–54].

2.2. Convolution Multiple Whole Profile Fitting (CMWP)

According to this method, it is assumed that the broadening of a given diffraction peak is caused by the strain fields of the dislocations. The full width at half maximum (FWHM) of diffraction patterns is expressed as [39]:

$$\Delta K \cong \left(\frac{0.9}{D}\right) + \left(\frac{\pi M^2 b^2}{2}\right)\rho_{int}^{1/2}(K^2\overline{C}) + O(K^4\overline{C}) \quad (10)$$

where $K = 2\sin\theta_B/\lambda$ and $\Delta K = 2\cos\theta_B(\Delta\theta)/\lambda$. Being D a size parameter, ρ_{int} is the average internal dislocation density, b is the Burger's vector, C is the contrast factor, M a constant relying on outer cut-off radius of dislocations, θ_B is the Bragg angle, O represents the higher order terms in $K^4\overline{C}^2$ and λ is the wavelength of X-ray. According to the Warren-Averbach approach, the Fourier transformation of the XRD patterns follows Eq. (11) [39]:

$$\ln A(L) \cong \ln A_L^S - 2\pi^2 L^2 g^2 \langle e_{g,L}^2\rangle \quad (11)$$

where A_L^S is the size Fourier coefficient, L is the Fourier parameter, and g the diffraction vector. In terms of Wilkens function $f(\eta)$, the mean square lattice strain causing the peak broadening is given as:

$$\langle e_{g,L}^2\rangle = (b/2\pi)^2\pi\rho_{int}\overline{C}f(\eta) \quad (12)$$

where $\overline{C}$ is the average dislocation contrast factor and can be expressed as:

$$\overline{C} = \overline{C}_{h00}\left\{1 - q\left[\frac{h^2k^2 + k^2l^2 + l^2h^2}{(h^2 + h^2 + h^2)^2}\right]\right\} \quad (13)$$

The internal dislocation density ρ_{int} was evaluated from Eq. (12) in different alloys [55–62].

2.3. Williamson-Smallman (W-S) Approach

According to this model, the mobile dislocation density is expressed by Eq. (14) [42,44]:

$$\rho_m = \frac{ke_{RMS}^2}{Fb^2} \quad (14)$$

where k is a constant that depends on the lattice, e_{RMS} is the root-mean-square microstrain and F accounts for the interaction of dislocations. Assuming that materials have block shape crystallites with effective size D_{eff}, the density of dislocations contributing to the crystallites boundaries is expressed as:

$$\rho_D = \frac{3n}{D_{eff}^2} \quad (15)$$

where, n accounts for the number of dislocations on each block face.