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QUANTITATIVE ANALYSIS OF DISLOCATION SUBSTRUCTURES

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ABSTRACT

Microstructural defects in materials are usually modelled in terms of point, fibre or surface processes in stochastic geometry. Dislocations are a typical example of fibre structure in metals. In addition to the dislocation density, the arrangement of dislocations, described by the pair correlation function, is an important characteristic of the dislocation substructure which reflects a prior treatment of the material.

For an isotropic structure, the function can be approximated by the planar pair correlation function, obtained from TEM micrographs, i.e. from projections of thin foils of thickness t.

The computer program for PC AT was prepared using a graphic card for the estimation procedure. The circular probes with increasing radius were used to evaluate the pair correlation function. The method was used to investigate the dislocation substructure in Cr-Mo-V ferritic steel before and after irradiating.

In addition to that, the relation of a dislocation substructure to both homogeneously distributed precipitate particles and heterogeneously formed radiation-induced defects in ferritic reactor pressure steels was studied by means of the cross-correlation function. Applying this function to a couple of random fibre process X and a random point process Y of particles and defects, respectively, centroids, provides information on the relation between these processes.

Keywords: Cross-correlation function, dislocation substructure, evaluating software, fibre process.

INTRODUCTION

The microstructure of crystalline materials is a complex of structural elements (phases, defects), including their qualitative (type, nature), quantitative (size, number), crystallographic (lattice) and topographic (shape, morphology) characteristics. The microstructure of crystalline materials is formed by phase transformations and by interactions between crystal defects. When materials are subjected to in-service or in-testing conditions or both, a substantial restructuring of their microstructure can occur.

For example, when a metal is irradiated by fast neutrons in a nuclear reactor, the vacancies and interstitials created in and surviving the collision cascade processes migrate freely through the crystal lattice at the irradiation temperature. They interact with each other, with solute atoms in matrix and also with dislocation substructure and precipitates, resulting in the formation of extended defects: dislocation loops, point defect clusters and new precipitates. These defects concentrate to dislocation substructure at elevated irradiation temperatures and higher neutron fluences. Simultaneously, the recovery of a dislocation substructure can occur; dislocations move and become pinned to existing particles. The radiation-induced microstructural evolution is strongly dependent on irradiation conditions, mainly on the irradiation temperature and the neutron flux and fluence.

Some of the radiation effects mentioned above were observed using transmission electron microscopy of thin foils in specimens of 440-WWER reactor pressure vessel steel irradiated to higher neutron fluences (Kočík and Keilová, 1990).

In this contribution, an attempt was made to corroborate the above mentioned findings using ideas of stochastic geometry. Both, the first order and the second order microstructural parameters were used. The first order microstructural parameters are densities of fibres and points, the second order parameter is the cross-correlation function (cross-CF). The cross-CF is related to a couple of a random fibre process and a random point process, respectively, providing a possibility of identification and evaluation of microstructural processes under the study.

The idea to use cross-CF for simultaneous quantification of two or more random substructures comes from Stoyan and Ohser (1984) and includes some ecological application. Dislocation substructures were also thoroughly investigated by means of second-order stereological methods (Hanisch et al., 1985; Stoyan and Ohser, 1986) as a fibre process. Moreover, the concepts of anisotropic fibre process of dislocation lines was studied by second-order methods in Beneš and Postler (1993).

However, a joint study of dislocations and precipitates and their interactions using stochastic geometry seems to be the first application of this type, especially in connection with the physical property studied, namely irradiation.

When a projection of a thin foil microstructure is evaluated, the resulting plannar cross-correlation function is a good approximation of the spatial cross-CF.

The cross-correlation function provides information about the neutron irradiation-induced changes of a dislocation substructure (fibre process) and on mixed population of small radiation-induced defects (point processes) in relation to the original dislocation substructure and precipitate distribution.

In addition to that, another case was selected as an example of the microstructure where dislocations are the dominant nucleae centres for precipitated particles. The matter is the β_1-phase particles precipitated during slow-cooling and subsequent annealing in an electron beam welded Zr-1Nb alloy plates directly on a dislocation network.

Algorithms and methods used for computing and evaluating described in this article have been implemented to the developed software.

MATERIALS AND METHODS

Transmission electron microscopy (TEM) provides for ferromagnetic information on number density, size and distribution of radiation-induced defects with diameter above the resolution limit of 2-3 nm. The weld metal microstructures of the Cr-Mo-V ferritic steel, employed for the pressure vessel of the WWER-440 light-water reactor before irradiation and after neutron irradiation to a fluence of $4\times10^{23}\,m^{-2}$ in a neutron flux $1.7\times10^{17}\,m^{-2}s^{-1}$ ($E>1\,MeV$) are in Figs 1a and 1b, respectively. Figure 1c depicts the microstructure of the Zr-1Nb weld alloy.

The densities of dislocations ρ_V and of precipitated particles N_V (including radiation-induced defects in the case of irradiated materials) were estimated by means of the well-known stereological formulae. The dislocation density equals to

$$\rho_V = \frac{4L_A}{\pi t} \quad , \tag{1}$$

where L_A is the intensity of the projected length and t the foil thickness. N_V was estimated by counting the particles within the projected foil. The measured values ρ_V and N_V are given in the Table 1 for the conventional foil thickness of 150 nm.

Image processing provides information concerning point and fibre processes. The mathematical theory by Stoyan and Ohser (1984) is used to compute the cross-CF.

Let Φ be a stationary point process, Ψ be a stationary fibre process. Further assume $\Phi(W)$ is the number of points inside window W and $\Psi(W)$ is length of fibres inside window W. Then the intensity

$$L_A = \frac{E\Psi(W)}{A(W)} \tag{2}$$

where A(W) is the area of a window W. For an isotropic process and W=b(0,r), i.e. circle with radius r centered in the origin, the second moment function K_{12} is given by

$$L_A K_{12}(r) = E\{\Psi[b(0,r)] \,|\, 0 \in \Phi\} \tag{3}$$

where r is circular probe radius. Then the cross-correlation function equals to

$$p_{12}(r) = \frac{1}{2\pi r}\frac{dK_{12}(r)}{dr} \tag{4}$$

Fig. 1a
Cr-Mo-V ferritic steel ,
non -irradiated

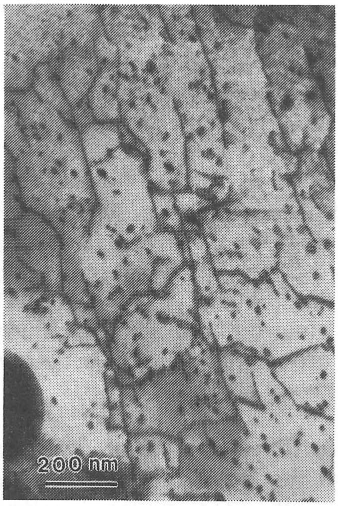

Fig. 1b
Cr-Mo-V ferritic steel ,
irradiated

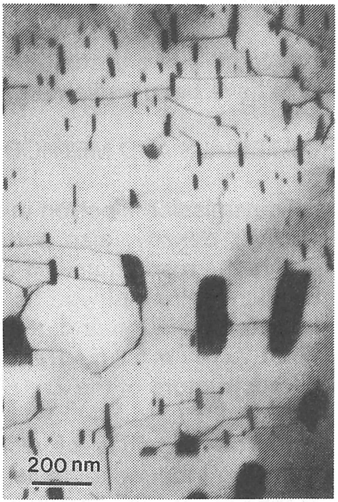

Fig. 1c
Zr-1Nb weld alloy

RESULTS

The material structures of dislocations and a population of precipitates and defects (representing the fibre and point processes, respectively) shown in Figs 1a, b, c were converted to bitmaps of 500x400 pixels. The objects representing points were converted to one-pixel-points located at centre of this object. Objects representing fibres were converted to 1 pixel wide broken lines.

Table 1 - Estimated values of densities ρ_V, N_V and the slope S of cross-CF.

MATERIAL / DENSITY	$\rho_V[\mu m^{-2}]$	$N_V[\mu m^{-3}]$	$S[\mu m^{-2}]$
Cr-Mo-V steel, non-irradiated	74	1 300	0.0015
Cr-Mo-V steel, irradiated	160	1 700	-0.0595
Zr-1Nb, non-irradiated	40	220	-0.1595

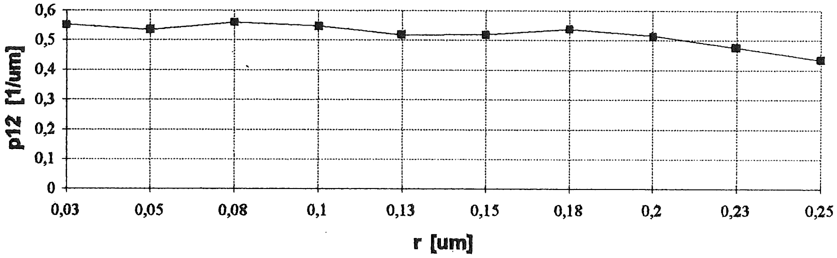

Fig. 2a : Cr-Mo-V ferritic steel in non-irradiated state

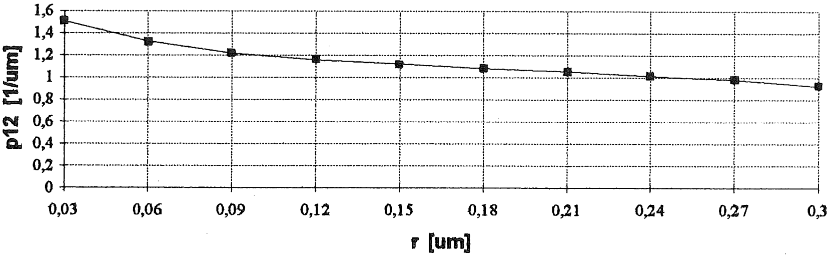

Fig. 2b : Cr-Mo-V ferritic steel in irradiated state

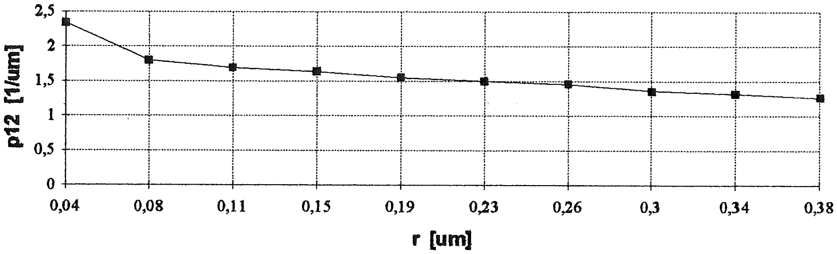

Fig. 2c : Zr-1Nb alloy in non-irradiated state

The cross-CF has been evaluated for 10 probes with radii from 10 to 100 pixels. The first three points of the graph were fitted by a straight line using the least squares method thus obtaining an estimate of the slope S of the cross-CF in the origin.

The diagram in Fig. 2a represents the Cr-Mo-V ferritic steel before irradiation. In this state points (precipitates) are mostly independent on the fibres (dislocation lines). The approximate zero slope of the computed cross-CF coincides with this fact.

The diagram in Fig. 2b represents the Cr-Mo-V ferritic steel after irradiation. The number of both, points and fibres is larger; points start to accumulate around fibres. Correspondingly, the absolute value of S is much larger than in the non-irradiated state.

The diagram 2c represents Zr-1Nb alloy. In this model case, nearly all points are situated directly on fibres. The expected strong dependence is confirmed by the largest absolute value of S.

DISCUSSION AND CONCLUSIONS

Results depicted in Figs 2a, b and c have shown, that the planar cross-CF provides an useful tool to estimate the changes in the dislocation substructure and in the mixed population of objects formed during the neutron irradiation (seen in thin foils by TEM), and to characterize the relations between them.

The change of the cross-CF shape found in the irradiated specimen of Cr-Mo-V ferritic steel in comparison to the non-irradiated state clearly demonstrates the effect of a neutron irradiation on the microstructure. As the radiation-induced defects are small and hardly discernible under the used irradiation conditions (high neutron flux - Kočík and Keilová, 1990), the revealed attachment of dislocations to particles is mainly due to radiation-induced dislocation movement. In addition to the small amount of defects arise on dislocation lines due to the long-range diffusion of primary created point defects is seen.

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