Available online at www.sciencedirect.com

jmr&t Journal of Materials Research and Technology

www.jmrt.com.br

Original Article

Stereological analysis of the microstructure of pure iron with random nucleation☆

Daniel Souto de Souza, Weslley Luiz da Silva Assis, Paulo Rangel Rios, Gláucio Soares da Fonseca**

Escola de Engenharia Industrial Metalúrgica de Volta Redonda, Universidade Federal Fluminense (UFF), Volta Redonda, RJ, Brazil

ARTICLE INFO

Article history:
Received 14 October 2013
Accepted 30 August 2014
Available online 13 October 2014

Keywords:
Cellular automata
Recrystallization
Stereology
Pure iron

ABSTRACT

Microstructural characterization of metallic materials is of paramount importance in the qualification and quantification of their desired final properties. Metallic materials have often been characterized by means of optical and electronic microscopy. Results from these techniques are in most of the cases based on 2D analysis. Stereological methods are then employed to obtain 3D information. However, such methods are based on assumptions and approximations of the real material structure. Therefore, it is essential to know the limitations of these methods. A further complication arises when one wishes to compare real materials with computer simulation results and stereological analytical techniques. In this paper, methods normally applied to real microstructure are applied to microstructures simulated by cellular automata (CA) simulation. Experimental results from microstructural characterization of polycrystalline pure iron were used as the starting point for the simulation. Consequently, we could apply analytical formulae of stereology to experimental data from pure iron and to computer generated microstructures from cellular automata simulation. Comparison of analytical formulae, experimental results and computer simulation provided useful insights on limits of applicability and on the meaning of the stereological analysis.

1. Introduction

Recrystallization and grain growth are fundamental issues of Materials Science. Therefore, continuous progress is always required in understanding those phenomena in order to achieve better control and consequently increasingly better final properties of polycrystalline materials. In this regard, detailed knowledge of the 3D microstructure of the polycrystal is paramount. Traditional metallographic techniques invariably carry out measurements on a planar section [1,2]. From those measurements, one cannot obtain all 3D microstructural parameters. For example, intrinsic 3D quantities, such as the volume of the individual grains, the number of

☆ Technical contribution presented at the 66th ABM International Annual Congress, São Paulo, Brazil, July 18th to 22nd, 2011.

* *Corresponding author.*
E-mail addresses: daniel.souto@hotmail.co.uk (D.S. de Souza), glaucio@metal.eeimvr.uff.br (G.S. da Fonseca).

http://dx.doi.org/10.1016/j.jmrt.2014.08.002
2238-7854/

Table 1 – Analytical formulae employed in the calculations carried out in this work.

Approximation method	DeHoff [2]	Voronoi [1]	Sphere	Cube
N_L (mm^{-1})	$= \frac{7}{6}N_A^{1/2}$	$= 1.205N_A^{1/2}$	$= \frac{1}{4}\sqrt{6\pi N_A}$	$= \frac{1}{2}\sqrt{6N_A}$
N_A (mm^{-2})	$= 0.735N_L^2$	$= 0.6887N_L^2$	$= \frac{8}{3\pi}N_L^2$	$= \frac{2}{3}N_L^2$
N_V (mm^{-3})	$= 0.422N_L^3$	$= 0.3247N_L^3$	$= \frac{16}{9\pi}N_L^3$	$= \frac{8}{27}N_L^3$
N_V (mm^{-3})	$= 0.670N_A^{3/2}$	$= 0.5681N_A^{3/2}$	$= \frac{\sqrt{6\pi}}{6}N_A^{3/2}$	$= \frac{2\sqrt{6}}{9}N_A^{3/2}$

grains per unit of volume or the Gaussian curvature per unit cannot be obtained from classical 2D quantitative metallographic techniques.

Recently, it has become apparent that there is a transition from 2D to 3D microstructural characterization. Specifically, today one has 3D computer simulation, 3D analytical theories and 3D microstructural characterization techniques. This can be seen in the widespread number of papers, conference sessions and even entire conferences dedicated to the topic of 3D Materials Science.

Still, for a long time simple techniques will persist and one may use the 3D developments to improve the simpler 2D techniques. For example, some assumptions may now be tested against a 3D computer simulation. In this way, one may develop useful approximations that may be very helpful for the Engineer to obtain a reasonably accurate result for a fraction of the cost and the time of a full 3D experiment. Following this reasoning, the present work utilizes experimental measures, analytical methods and computer simulations to revisit earlier results and test new approximate expressions that relate 2D measurements to 3D quantities by means of approximate expressions.

2. Methods and materials

2.1. *Experimental methods and materials*

Samples of polycrystalline pure iron were used. The composition of the pure iron in ppm was: C-41, Mn-940, P-15, S-20S Si-160, Al-20, N-80, Ti-10, Cu-30, Cr-100, Mo-20, Nb-10, V-10, B-4, O-165. The sample was cold rolled up to 80% then annealed 550 °C for 3600 s (1 h) in a quartz tube containing Ar. Specimens were cut from this sample taking care to avoid deformation during cutting. The specimens were then ground and polished. A final polishing step consisting of polishing in a colloidal silica solution was employed. After this, the specimens were etched with Nital 3% solution for observation under the optical microscope. Stereological measurements were carried out in a Nikon Eclipse optical microscope with image analysis software NIS-Elements D 3.0. The number of grains per unit of area, N_A, and the number of grains per unit of length of test line, N_L, were measured using standard techniques [3].

2.2. *Computer simulation*

A 3D cellular automata computer simulation was carried out. A cubic mesh with $300 \times 300 \times 300$ cubic cells, henceforward also referred to as the "matrix", was employed. A von Neumann neighborhood was adopted. Simulation details can be found in Ref. [4–9]. The simulation employed a 22,300 nuclei uniform randomly located within the matrix at the start of the transformation. This number of nuclei was chosen according to reasons explained in a previous work [10]. The simulation was carried out in a dual processor Intel Xeon workstation with eight cores in each processor and 48GB of memory. The computer program was written in Fortran 2003 and compiled in an Intel FORTRAN compiler using OPEN MP 3.0 directives for shared memory parallelization. Each simulation run took about 10,000 s. In order to assign units to this simulation a length to the edge of a cubic cell has to be chosen. We chose this length in such a way to make the number of nuclei *per unit of volume* equal to that calculated for pure iron using DeHoff's formula. This was done because DeHoff's result was considered the better approximation in that case. Details are given below and results are in Tables 1 and 2, later in this paper. This choice gave a cubic cell with an edge length equal to about 0.5578 μm.

2.3. *Analytical expressions*

The analytical expressions employed in this study either derived above or obtained from publications are summarized in Table 1.

In addition to N_L, N_A, and N_V obtained by DeHoff [2] and for Voronoi polyhedral [1] we derived expressions for these quantities supposing that the grains could be considered as cubes or spheres.

For spherical polycrystalline grains, using $S = 4\pi R^2$, $V = \frac{4}{3}\pi R^3$ and the caliper, $D = 2R$, gives, S_V, the grain boundary area by unit of volume

$$S_V = \frac{S}{V} = \frac{3}{2R} \quad (1)$$

Deriving expressions for N_V is straightforward

$$N_V = \frac{1}{V} = \frac{3}{4\pi R^3} \quad (2)$$

Table 2 – Measurements of N_L and N_A in pure iron compared with calculations from approximate methods.

Stereological quantities	Pure iron (measured)	DeHoff [2]	Voronoi [1]	Sphere	Cube
N_L (mm^{-1}) from N_A	67.3	67.0	69.2	62.4	70.4
N_A ($\times 10^{-2}$ mm^{-2}) from N_L	33.0	33.3	31.2	38.5	30.2