

RESEARCH OF GRAIN SHAPE CHANGES DURING SINTERING OF Fe POWDER COMPACTS BY STEREOLOGICAL METHODS¹

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ABSTRACT

The size and number of microstructure constituents, such as grains of phases present or pores, are basic stereological parameters, which can be used to follow the process of material synthesis. Grain and pore shapes need to be analyzed with the purpose of a more complete definition of the synthesis process. In this paper characterization of grain shape of Fe powder compacts was performed as a function of temperature of non-isothermal sintering. A polycrystal Fe powder obtain by spraying in air was used in this analysis. The powder was pressed to a set porosity of 10%. Compacts were non-isothermally sintered in hydrogen flow until temperatures of 300, 600, 760 and 1000°C were attained. The samples were then rapidly cooled in water. Analysis of the form compacts obtained was performed on an automatic image analyser ("QUANTIMET 500MC"-Leica). The following parameters which describe the grain shape were determined: perimeter form factor (f_l), area form factor (f_A), waviness (f_w) and ratio between maximal and minimal diameter ($f_{D/D}$). Based on results obtained from measurements, changes of each selected parameter of grain shape were followed during sintering and their dependence degree was determined.

Keywords: Grain shape, Sintering, Stereological methods

1.INTRODUCTION

Processes occurring during sintering such as densification, grain growth and recrystallization can be observed through both changes of the size and form of microstructure constituents [1,2,3]. Numerous parameters defining the form exist [4]. In this paper changes of the grain shape of Fe compacts during non-

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isothermal sintering were observed. The following grain shape parameters were determined:

- perimeter form factor (f_L); $f_L=1$ for a circle; area form factor (f_A); $f_A=1$ for a circle and waviness (f_W):

$$f_W = \frac{L_{pconvex}}{L_p} \quad \text{and}$$

as well as the ratio between maximal (D_{max}) and minimal (D_{min}) diameter ($f_{D/D}$):

$$f_{D/D} = \frac{D_{max}}{D_{min}}$$

2. MATERIAL AND METHODS

A polycrystal iron powder obtained by spraying in air was used in this investigation. The powder was pressed to a set porosity of 10%. Compacts were non-isothermally sintered in hydrogen flow until the temperatures of 300, 600, 760 and 1000°C were attained. This was followed by rapid cooling in water. Analysis of the grain shape of compacts obtained in this way was performed on an automatic image analyser ("QUANTIMET 500MC"-Leica), using the planimetric method for measuring the grain area and perimeter. The following parameters describing the grain shape were determined: perimeter form factor (f_L), area form factor (f_A), waviness (f_W) and ratio between maximal and minimal diameter ($f_{D/D}$).

3. RESULTS

In Table 1. means values and relative standard errors of measurement of parameters describing the grain shape are given.

Table 1 - Mean values and relative standard errors of measured grain shape parameters

	f_L		f_A		f_W		$f_{D/D}$	
	mean	RSE, %	mean	RSE, %	mean	RSE, %	mean	RSE, %
powder	0,517	0,968	0,777	0,471	1,666	0,801	0,853	0,312
300°C	0,545	0,708	0,788	0,396	1,774	0,708	0,883	0,204
600°C	0,566	0,624	0,800	0,352	1,777	0,781	0,897	0,164
760°C	0,580	0,649	0,810	0,365	1,770	0,848	0,904	0,165
1000°C	0,509	0,932	0,774	0,451	1,729	0,916	0,855	0,321

In Figure 1. curves of cumulative distribution of mesured form factors are shown.

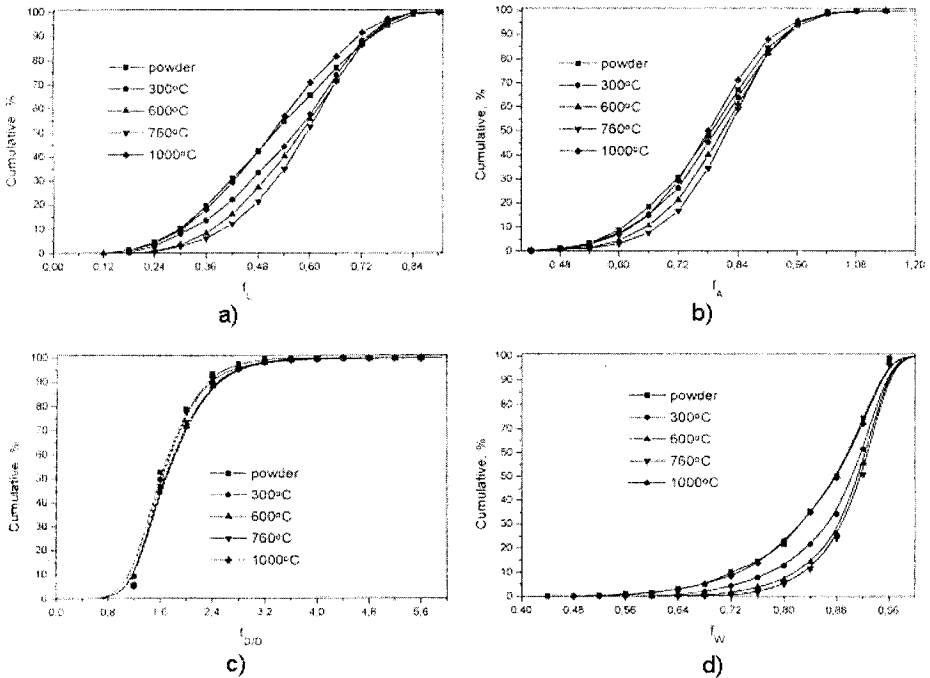


Figure 1. Cumulative distribution curves a) perimeter form factor, b) area form factor, c) waviness and d) the ratio between maximal (D_{max}) and minimal (D_{min}) diameter.

Figure 2. shows the dependence of the grain area on its perimeter is shown (scatter diagram)

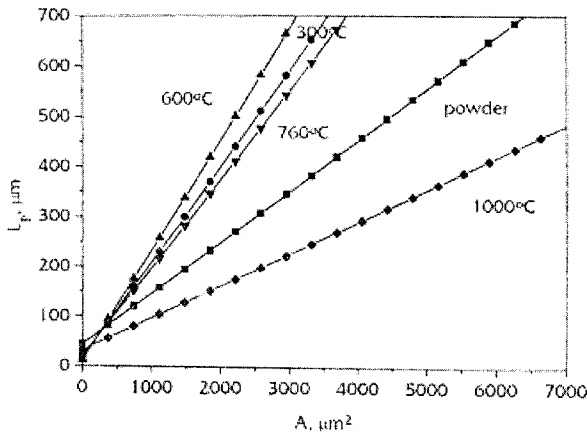


Figure 2 - Scatter diagram of $A-L_p$

4. DISCUSSION

The presented results (Table 1. and Figure 1.) indicate that the measured grain shape parameters change according to the same laws as a function of the sintering temperature, but with different, growing degrees of sensitivity, starting with $f_{D/D}$, f_A , f_W , and ending with f_L . Such laws of grain shape changes with temperature, can be explained by processes occurring during sintering, such as densification, recrystallization (above 600°C) and grain growth. All measured parameters display the structural heredity property, i.e. the grain shape of starting powder corresponds to the same after sintering at 1000°C [1].

It is obvious that the perimeter form factor registers the finest differences of the dependence of grain shape on the sintering temperature. However, the scatter diagram of the dependence of area on perimeter, Figure 2. gives the best insight into changes of the grain shape in the case of small differences.

All measured parameters display the structural heredity property, i.e. the grain shape of the initial powder corresponds to the same after sintering at 1000°C.

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