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Computer aided determination of porosity in sintered steels

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ABSTRACT

Purpose: Purpose of this paper was to apply a computer method of open porosity analysis for determination of porosity in sintered Astaloy CrL and CrM powders.

Design/methodology/approach: The powders used in the present papier are pre-alloyed iron-base powders containing low amounts of chromium and molybdenum. The amount of graphite which is mixed with the iron-base powder is 0.6% and lubricant is 0.75%. Green compacts were sintered in a vacuum furnace at 1120ºC for 30 minutes in vacuum and rapidly cooled in nitrogen at four different rates: high cooling (7 ºC/s and 6.5 ºC/s) and medium cooling (1.6ºC/s) low cooling (0.3 ºC/s). Next the samples were tempered in vacuum in the same furnace at 200ºC for 60 minutes and cooled in nitrogen, with the exception of low cooling. Obtained samples were examined by light optical microscopy (LOM) for microstructure observation and scanning electron microscopy (SEM).

Findings: The investigations of porosity in the pre-alloyed steels were performed using ”Image-Pro Plus 4.5” computer program. Leica light microscope (MEFA4) was applied to prepare series of micrographs. The microstructures were observed at 100x magnification and recorded as*jpg files. After calibration of the digital images, the perimeter, area, diameter, roundness, Feret max were calculated.

Practical implications: The program has some limitations (built in mathematical functions), which made difficult to obtain some results automatically.

Originality/value: The obtained results prove that this program is very useful in the statistical analysis of microstructures, because it saves the time during calculations and is well suited for checking sintered materials.

Keywords: Powder metallurgy; Sintering pre-alloyed Astaloy CrL and CrM steels; Computer method of porosity analysis

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

The porosity is define as a solid, that includes in inside a huge number of voids, so some spaces often are filled by gas, liquid or other chemical properties rather than a solid. These internal spaces are often called as “*pores*”. Pores should be uniformly distributed in a continuous way and their sizes should be bigger than the molecular distances and smaller from bigger material voids, called “*cavities*”. A solid is a matrix of porous body, called as a "*skeleton*" or a "*vehicle*"[1]. In the literature closed and open porosity are complete distinguished. The pores joined with themselves and with an external surface of product are determined as open porosity. Open porosity is a continuous skeleton of gas phase which penetrates a constant phase. Closed porosity does not create this skeleton, but it consists of isolated from themselves pores. Complete porosity is a volume sum of open and closed pores. Figure 1 present open and closed porosity [2-4]. Generally it is acceptance of specific properties as presented in Figure 2.

a)

b)

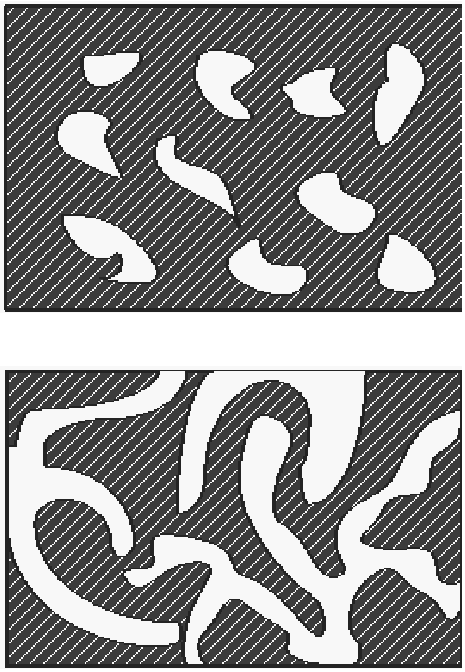


Fig. 1. Diagram of sinter structure with porosity: a) closed; b) open [2]

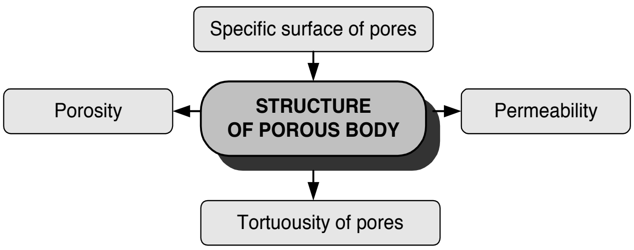


Fig. 2. Description of a porous body structure [1]

Porosity as structure property that can be a positive or negative feature. Materials with good insulating properties in a warm, could be characterized by a huge porosity. Presence of pores allows to modify porosity of materials by saturation of different isolating and refining substances. Porosity is a negative property when, for example, it decreases mechanical properties of sintered material or in the case of material contact with moisture. *Permeability is* define a ability of porous material to pass by pores a liquid and gases under the influence of gradient pressure. *Tortuous* is understood as a kinematic property; define a medium length of liquid molecule track moving from one point medium porosity to second, treated to a length between these points in a straight line. There are two known definitions of specific surface of pores, which differ from each other concerning internal surface: in the first case to a volume, in second to a mass of all porous body [2-4, 7-8].

Pores arrangement in a material, their shape and size depend on many factors. Indirect influence of a porosity of final product exert pressure on process of producing powders. The main factors include sinter porosities as factors presented in Figure 3. Obtaining non-porous products, manufactured by powder metallurgy, could make a possible extend a range of used agglomerates. There exist some methods which allow to obtain products with high density, close to theoretical [1, 4-6, 13-15].

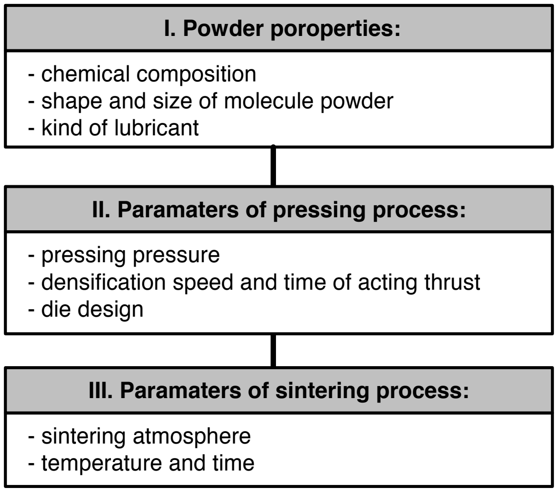


Fig. 3. Factors infulence on sinters porosity [1]

1.1. The main aim and range of examinations

The aim of this work was to perform an analysis with using computer program that helps to determine porosity occurring in sintered steels. Moreover, a program with aid of different statistical parameters allow to classify shapes of pores an their quantity [9-15].

2. Experimental procedure

2.1. Samples preparation

Two iron powders were used in the investigations: Höganäs atomised iron powder grade Astaloy CrL (Fig. 4a), Astaloy CrM (Fig. 4b) and lamellar graphite powder grade GR12 (Fig. 4c).

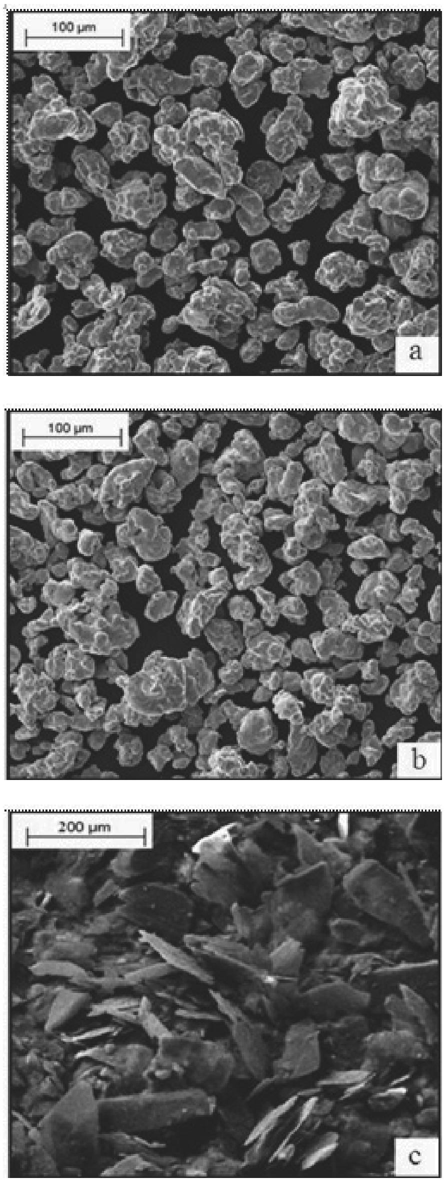


Fig. 4. Micrographs of base powders: a) Astaloy CrL pre-alloyed powder. b) Astaloy CrM pre-alloyed powder. c) GR12 graphite powder (SEM)

2.2. Powders mixture preparation

In order to investigate the influence of high carbon content and cooling rates on the microstructure and properties of sintered steels from initial sponge powders Astaloy CrL and Astaloy CrM, and graphite CR12 four mixtures were prepared. Table 1 contain some information about the chemical composition of the base iron powders. Next the base powders were mixed with graphite to obtain the desired strength of the materials and with lubricant to reduce the friction force between the powder and tool walls, and also between the particles.

Iron base powder was mixed with 0.6% of graphite. Lubricant "Acrawax" was used in the quantity of 0.75% Two mixtures (cooling rates: 7 and 1.6ºC/s) were mixed using a turbula mixer (20 min. 80 rpm), the others in a double cone laboratory mixer (20 min. 50 rpm).

Table. 1.
Chemical composition of iron-base powders

Base iron	Mo[%]	Cr[%]	C[%]	O_2-tot[%]
Astaloy CrL	0.20	1.50	<0.01	0.16
Astaloy CrM	0.50	3.00	<0.01	0.21

2.3. Pressing

From prepared powder mixtures the three sets of samples were prepared using a uniaxial pressing die (die with free motion):

- dog bones, according to standard ISO: PN-EN 10002-1, for tensile test.
- rectangulars, dimensions about 5 x 10 x 55 mm for Charpy test, hardness and cross-section observations three bending test;
- disks, diameter about 40 mm, for the wear test.

The samples were pressed using 200 kN hydraulic press with pressure of 500 MPa and 600 MPa.

2.4. Debinding

Debinding process was performed in fully nitrogen atmosphere for 8 hours in a separate plain furnace. before the sintering process. Green compacts were kept in the furnace at the 550ºC for one hour to remove the possible largest fraction of lubricant.

2.5. Sintering

Prepared green compacts were then sinter-hardened with the following parameters:

- green compacts were sintered at 1120ºC for 30 minutes in a vacuum furnace with argon backfilling ($5.0 \cdot 10^{-1}$ mbar);
- sintered compacts were rapidly cooled in nitrogen at four different rates: high cooling (with a rate 7ºC/s and 6.5ºC/s), and medium cooling (with a rate 1.6ºC/s) and slow cooling (with a rate 0.3ºC/s and no gas added);
- sintered compacts were tempered in a vacuum in the same furnace (at 200ºC for 60 minutes) and cooled in a nitrogen.

After, the samples were grinded on abrasive papers of grain 120-2000 µm/mm^2 and polished with diamond paste. Etched microsections were observed on the metallographic microscope Leica MEF4A with a total magnification carried out 5x, distance scaling: 1.160 µm in the Image-Pro Plus computer program.

2.6. Software description

The" Image-Pro Plus" software is a useful tool for processing and analysis of graphic files in Ms Windows environment. It is usually used for statistical analysis of different kind of pictures, recorded as graphical files (files in a form such as: *tif, *jpw, *seq, *jpg, *flf, *tga, *avi, *bmp, *pct, *cut, *pcx, *eps). It gives a possibility to a measure surface areas, distances, perimeter, diameter, and densities of elements on the picture. Input data for the program can be graphical files from camera installed on microscope. This program can calculate different statistical parameters, only a few were analysed in the experiments such as:

- Area: Reports the area of each object (minus any holes). The area comprised of pixels having intensity values within the selected range is reported unless the fill holes option has been enabled. If fill holes is enabled. all pixels within the object perimeter are included in the area measurement
- Diameter (max): Reports the length of te longest line joining two outline points and passing through the centroid.
- Diameter (mean): Reports the average length of the diameters measured at two degree intervals joining two outline points and passing through the centroid.
- Diameter (min): Reports the length of the shortest line joining two outline points and passing through the centroid.
- Perimeter: New measurement to report the length of the outline of each object. When holes are outlined, the perimeters of the holes are added to the perimeter of the object.
- Perimeter2: Old measurement from version 3.0. Faster but less accurate than current perimeter measurement. Reports the length of the outline of each object. When holes are outlined. the perimeters of the holes are added to the perimeter of the object.
- Feret (max): Reports the longest caliper (feret) length.
- Roundness: Reports the roundness of each object, as determined by the following formula:
  (perimeter2)/(4*p*area). Circular objects will have a roundness = 1; other shapes will have a roundness > 1.

The algorithm used in present study determine some parameters of pores is as follows:

- Reading in a picture (Fig. 5).
- Program calibration based of characteristic length (marker length) (Fig. 6 a, b).
- Parameters recess, which will be measured (grain length, grain size, etc.) (Fig. 7).
- Manual or automatic description of elements, that must be measured (Fig.8.)
- Points out of some elements.
- Mask forming (change of a colour picture to binary picture) (Fig. 9).
- Counting pixels getting in composition binary picture.
- Calculation of given parameter based on the amount of pixels (Fig. 10). From data presented in a sheet we can create a simple diagram or accomplish other calculations.

Late, the results must be recorded as a Excel data file and then user can calculate their statistic. User should remember also about a lower limit of noises ("noise" is a certain level of data, which leads to increase some measuring errors), which should be rejected by him. Data given into a calculation sheet of program can be illustrated by a histogram, that is created using the Histogram command in the Measure/Analysis menu. Histogram measures and illustrates in graph form, the intensity characteristics of an image.

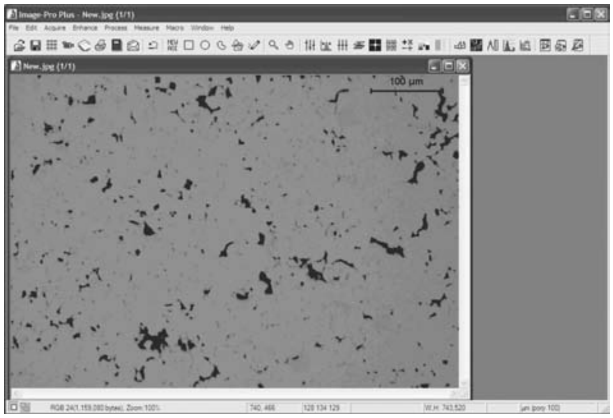
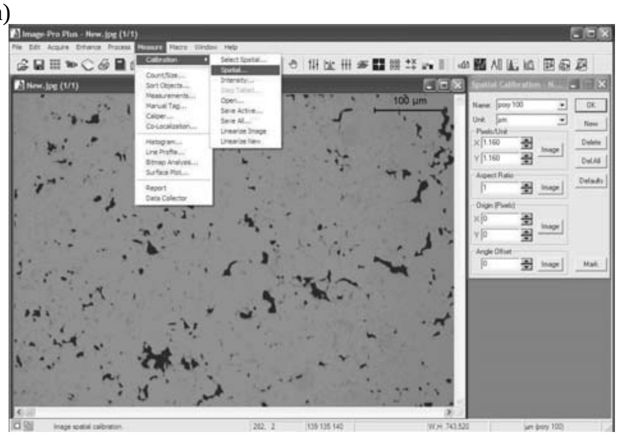


Fig. 5. Turn on a program and reading a picture in

a)



b)

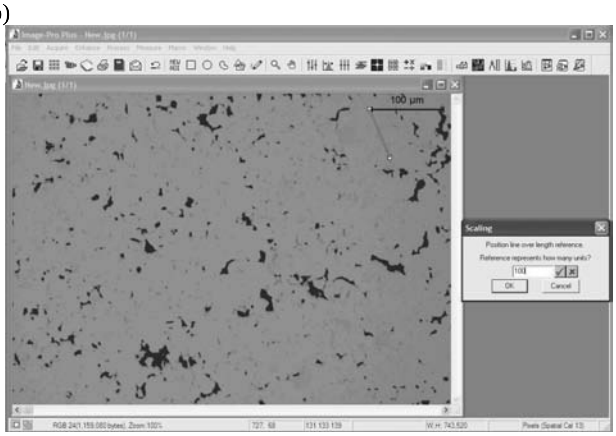


Fig. 6. Scaling a picture a) setting data, b) in the right corner, is shown a dimensional line, which we put marker, after we enter numeral into a window; after pressing a button "ok", the picture will be scaled; the next a stage will be research

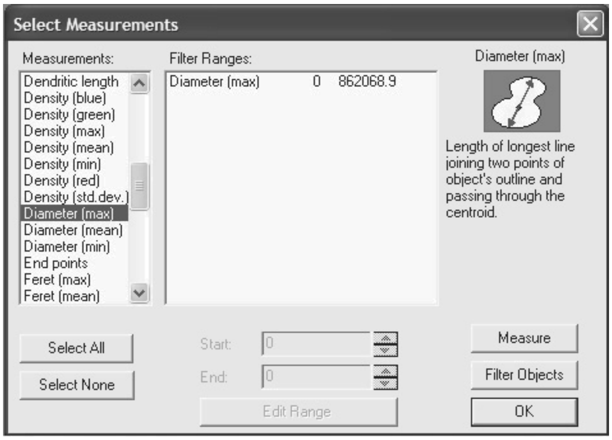


Fig. 7. Some of the parameters calculated in a program

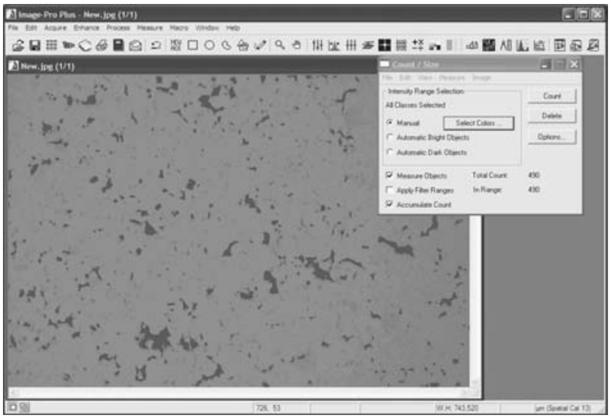


Fig. 8. Some parameters calculated in a program

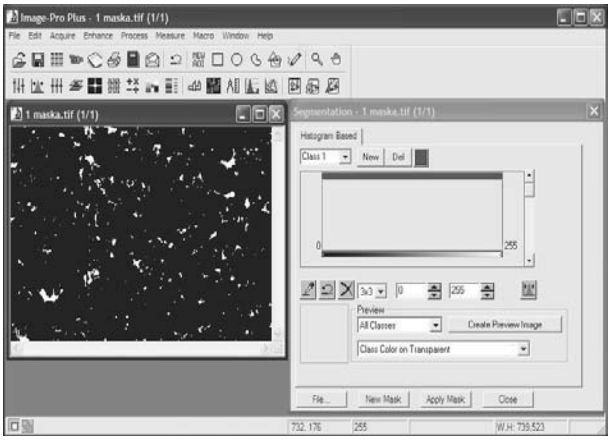


Fig. 9. Apply mask and close. A command "count" calculates specified parameter earlier given

Observation of non etched samples was finished with performing structure photos. There were chosen 5 best structure photos for each variant. Photos of each sample were taken in the centre of polished cross-section. The structure analysis was done using a Image-Pro Plus 4.5 program. Some statistical parameters were determined, but one of the biggest problem with analysis of photo is correct preparation of microsection to the microscopic examinations. In a case of the plastic materials, banks of pores often are distorted, what causes their underrating into a photo.

Moreover, dimensionless factors fe, fs were determined from obtained measurement data, which are used to estimate a spheroidzing step [5]:

$$fe = D_{min}/D_{max} \quad (1)$$

$$fs = 4 \cdot \pi \cdot A/P^2 \quad (2)$$

where: D_{min}- minimal pore' diameter, D_{max}- maximal pore' diameter, A- pore' area, P- pore' perimeter

However, in a case of hard and brittle materials, owing to chipping banks of pores during polishing, is overestimated size of pores visible in a structure. A significant difficulty is a fact that using only methods of image analysis, we are not able to unequivocally evaluate, if given structure with pores, were optimally prepared for analysis.

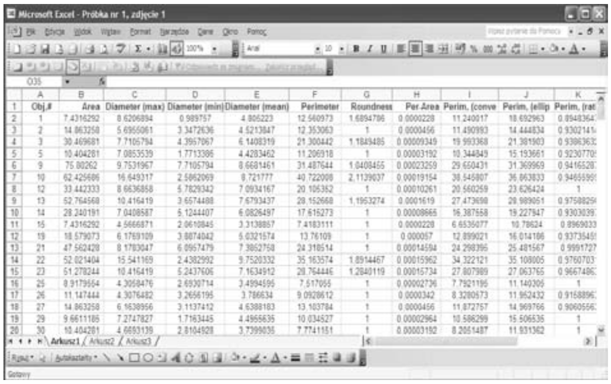


Fig. 10. The spreadsheet with recorded data. The program saves automatically calculated values in a calculation spreadsheet, after choosing an option "add to excel"

Sintered steels do not have homogenous structures (for instance, agglomerates of austenitic steels, where pores are connected with themselves and are not regular can point out less beneficial morphology of porosity rather than agglomerates of ferritic steels, where are not regular and fine pores), but results obtained concern flat metallographic microsections, which are not representative for the overall sample density. As each sample was cut in the same way, the results obtained for particular samples are comparable.

3. Results and discussion

As a result of porosity measurements it was found that analysed pores are characterized by size which does not exceed

100 µm^2, so further research were focused on this group of population. Figure 11 presents the setting-up results of medium size pores measurements depending on cooling rate applied.

The average value of pores' size is in a range from 44 to 58.5 µm^2. The maximum values of pores' size were obtained for Astaloy CrL 600MPa steels cooled slowly cooling. Particular differences values of pores' size are shown between 6.5 and 7ºC/s rates, which could have been caused by mixing mixtures into two different mixers.

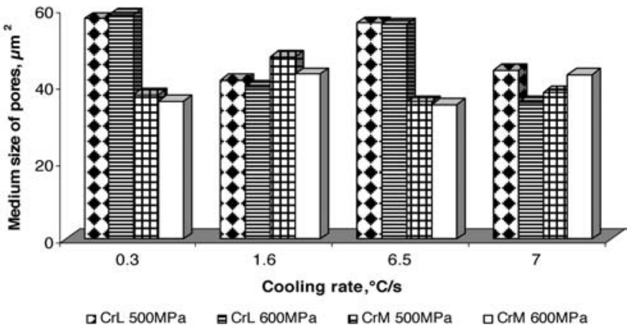


Fig. 11. Size of pores, average values

Density in particular porosity and its morphology decide mechanical properties and corrosion resistance of the Astaloy CrL and CrM steels. The shape factors: fe (Fig. 12) and fs (Fig. 13), were determined medium size of pores (mentioned earlier) and numbers for each size class to examine the extent of spheroidzing pores. The shape factor is equal 1 when it represents a circular pore in a plane analysis and along with decrease the extent dimensionless shape, expressed by factor - fs and extension expressed by factor – fe, increase.

The analysis shape factor revealed that the smallest average perimeter was found for samples fabricated from Astaloy CrM powder, cooled with rapid cooling 6.5ºC/s, whereas the biggest average perimeter was observed for samples fabricated from Astaloy CrL powder, cooled with rapid cooling 1.6ºC/s with the same compacting pressure of 600 MPa. Sintering in the same temperature causes increase of average perimeter from a range 0.1 to 0.5 and decrease from a range 0.6 to 1 for each applied cooling rate. The obtained data asymmetrical Gaussian distribution. Both increase and decrease of fe factor testify the smaller spheroidzing and bigger irregularity of pores.

The analysis of the shape factor (fs) shows that samples fabricated from Astaloy CrL powder (500 MPa pressure) reveal the smallest porosity, independently from the applied cooling rates. The highest size of pores in a range from 55 to 65% characterized samples obtained from Astaloy CrL powder with a pressure of 600 MPa.

In Table 2 and for example Figure 14 average values of parameters describe a porosity sintered materials, determined on the program. The average values of pores area depending on different cooling rates were presented in Figure 14. The mean values of pores' diameter depend on different cooling rates and are presented in Figure 15.

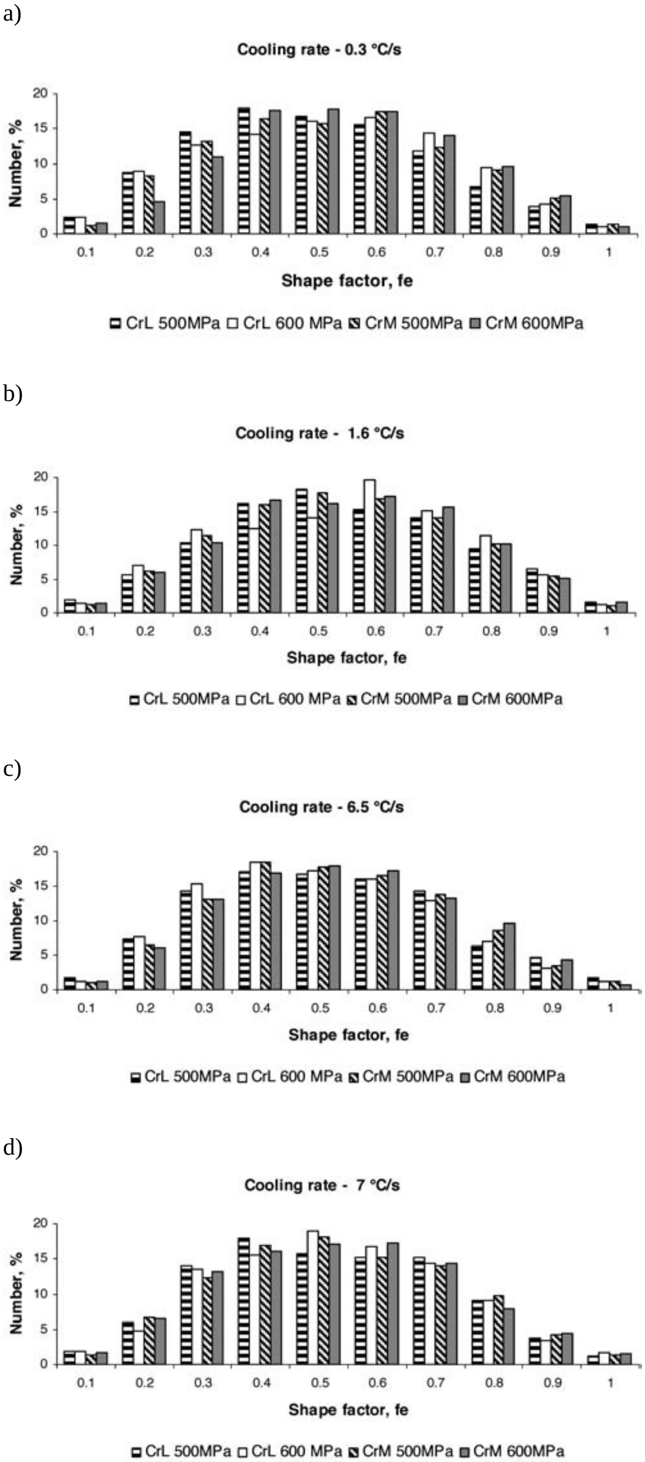


Fig. 12. Influence of four cooling rates: a) slow cooling (0.3ºC/s), b) medium cooling (1.6ºC/s), c) rapid cooling (6.5ºC/s), d) rapid cooling (7ºC/s) on distribution of the dimensionless shape factor- fe

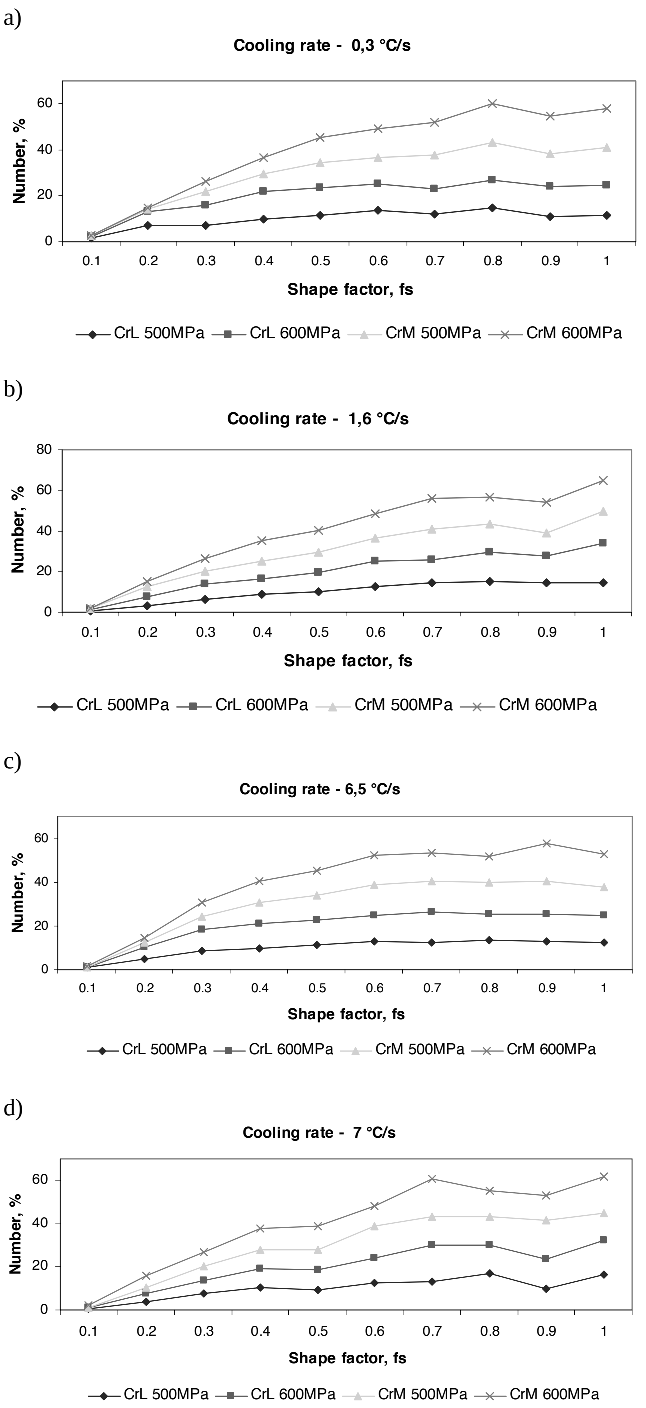


Fig. 13. Influence of four cooling rates: a) slow cooling (0.3ºC/s), b) medium cooling (1.6ºC/s), c) rapid cooling (6.5ºC/s), d) rapid cooling (7ºC/s) on distribution of the dimensionless shape factor - fs

Figure 16 and Table 2 present the average value of pores perimeter obtained by program. Fig. 16 shows the average values of pores roundness depending on different cooling rates (Fig. 17), where values were obtained by manual calculations according formula (2), on the program works with assumption that circular objects will have roundness = 1 or roundness > 1. The average values of pores' Feret max depending on different cooling rates are presented in Fig. 18.

Table 2.
The average value of "Perimeter" obtained by " Image-Pro Plus" (selected examples)

No	Powder Astaloy	Compacting pressure [MPa]	Cooling rate [ºC/s]	Perimeter [µm]	Standard deviation
1	CRL	500	7	28.23	35.46
2		600		26.53	29.69
3	CRM	500		25.27	26.80
4		600		28.72	36.34
5	CRL	500	1.6	26.52	31.51
6		600		25.40	28.76
7	CRM	500		30.34	38.55
8		600		25.40	28.76
9	CRL	500	6.5	34.33	45.97
10		600		33.88	48.12
11	CRM	500		25.54	24.97
12		600		24.01	24.26
13	CRL	500	0.3	35.61	48.56
14		600		33.93	42.48
15	CRM	500		19.73	20.68
16		600		22.94	21.63

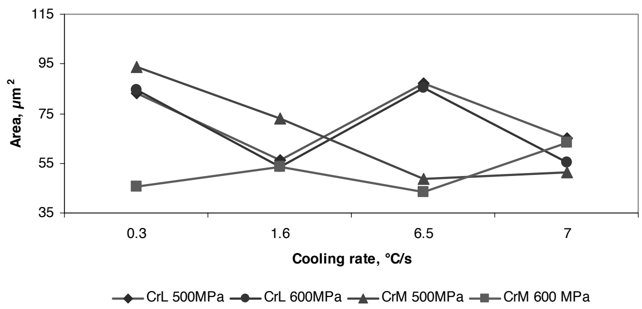


Fig. 14. The average values of pores' area in sintered steel based on pre-alloyed Astaloy CrL and Astaloy CrM powders

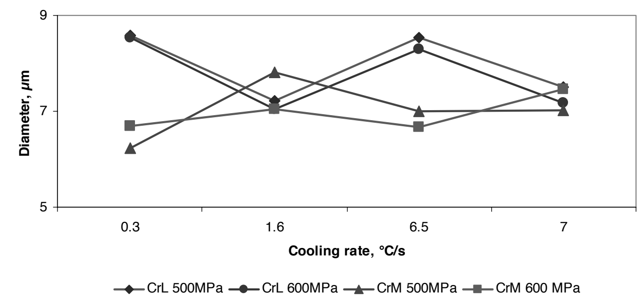


Fig. 15. The average values of pores' diameter existed in sintered steel based on pre-alloyed Astaloy CrL and Astaloy CrM powders

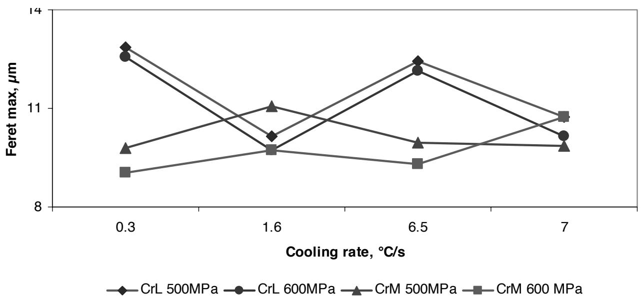


Fig. 16. The average values of pores' Feret max existed in sintered steel based on pre-alloyed Astaloy CrL and Astaloy CrM powders

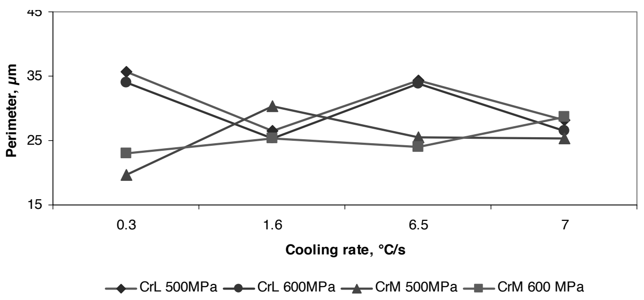


Fig. 17. The average values of pores' perimeter existed in sintered steel based on pre-alloyed Astaloy CrL and Astaloy CrM powders

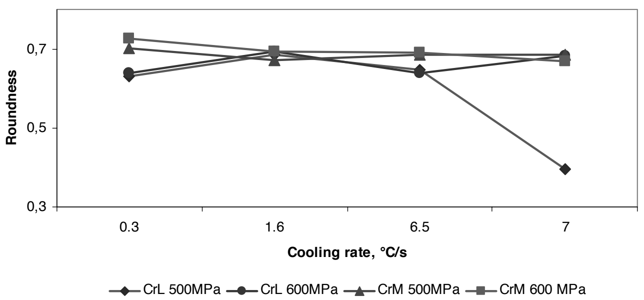


Fig. 18. The mean values of pores' roundness existed in sintered steel based on pre-alloyed Astaloy CrL and Astaloy CrM powders

The values of pores' area are in ranges for fabricated samples: CrL 500MPa – 56-87 µm, Astaloy CrL 600MPa – 53-85 µm, Astaloy CrM500 MPa – 48-93 µm, Astaloy CrM600 MPa – 43-63 µm with different cooling rates (Fig. 14). The highest average value of the occupied area equal to 93 µm was obtained for a cooling rate (0.3ºC/s) for Astaloy CrM powder (pressure 500 MPa), the lowest average value of occupied area equal to 43 µm was obtained for the same powder but higher pressure (600 MPa) and higher cooling rate 6.5ºC/s. Similar results were obtained especially for one powder Astaloy CrL independently from the applied pressure and cooling rate.

The similar range of results of the average values of pores' diameter and Feret max (length of the longest section, which connect two most distant points belonging to the investigated object) was obtained by Astaloy CrL powder for two pressures (Figs. 15 and 16). The maximum diameter was observed for Astaloy CrL 500 MPa, and the minimum was observed for Astaloy CrM500MPa with 0.3ºC/s cooling rate.

The average values of pores perimeter are with the following ranges: Astaloy CrL 500 MPa – 26-35 µm, Astaloy CrL 600 MPa – 25-33 µm, Astaloy CrM500 MPa – 19-30 µm, Astaloy CrM600 MPa – 22-28 µm for different cooling rates (Fig. 17). The highest average value equal to 35 µm was obtained with the same cooling rate (0.3ºC/s) for Astaloy CrL powder (pressure 500 MPa), the lowest average value equal to 19 µm was obtained for Atsaloy CrM powder (pressure 500 MPa).

As mentioned before, the shape factor equals to 1 for ideal circle, based on statistical data it was foun that average values of pores were in the following ranges located in ranges: Astaloy CrL 500 MPa – 0.4 0.6 µm, Astaloy CrL 600 MPa – 0.63-0.69 µm, Astaloy CrM500 MPa – 0.6-0.7 µm, Astaloy CrM600 MPa – 0.6-0.7µm with different cooling rates (Fig. 18). The most irregular shapes of pores were observed especially for Astaloy CrL 500 MPa powder with rapid cooling (7ºC/s).

4. Summary

The investigations performed in present work allow to formulate the following conclusions:

- The conditions of preparing microsections for examination play a considerable role in analyzing pores shape and quantity pores, because during polishing some pores can be ground.
- Samples fabricated from the pre-alloyed Astaloy CrM steel were characterized by smaller values of the average pores diameter of pores rather than samples fabricated from pre-alloyed Astaloy CrL steel, as well with all cooling rates, independently from the applied compaction pressure.
- Use of the same temperature, but different cooling rates cause increase of Feret max parameter for samples fabricated from the pre-alloyed Astaloy CrL and CrM steels.
- High values of pores' roundness (fs factor >1) for samples cooled with different cooling rates can indicate to smoothing pores walls as a result of evaporation.
- The Image-Pro Plus program helps user to choose and calculate different statistical parameters (for instance: it classifies shapes of pores) in a very short time.
- The program has some built-in functions, which can limit user work, for instance, program can only show shapes will have a roundness (fs factor) >=1. For instance, the program calculates minimal and maximal pore' diameter, but if a user want to obtain fe factor, then user will have to calculate it manually with using some calculation sheet.

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