MORPHOLOGICAL AND STRUCTURAL ASPECTS USING ELECTRONIC MICROSCOPY AND IMAGE ANALYSIS OF IRON POWDERS OBTAINED BY WATER ATOMIZATION PROCESS

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Abstract: *Quality of metal powders depends on manufacturing process and heat treating parameters. Among the physical, mechanical and structural characteristics of powders, the average particle size, size distribution, particle shape and microstructure of powders are defining characteristics to obtain powders with superior performance. The objective of the present paper is to analyze, using electronic microscopy and image analysis , the particle shape and size of iron powder obtained by water atomization of molten iron. We separately computer analyzed three types of powder, and then we realized the graphical representation of particle size distribution of powders in function of different atomizing temperature. It was established correlation between water atomization process parameters and physical, technological and structural characteristics of iron powders.*

Keywords: *electron probe X ray microanalyzer, image analysis, iron powders, water atomization process*

1. INTRODUCTION

In generally, any material can be made into a powder by one or more of manufacturing methods (mechanical processing of solid material, atomization of molten metals, chemical reactions and decompositions, electrolytic deposition), but choosing one of the above mentioned methods depending on desired properties, the structure of the final product and the costs of the powder manufacturing process. Among of these methods, the most widely used methods are chemical reactions, decomposition and atomization of molten metals.

By atomization process (disintegration of the molten metal using high pressure water or inert gas, etc) we can obtain a wide variety of powders, such as powders of iron, aluminum, tin, cadmium, zinc, lead, copper or their alloys [1]. One of the advantages of atomization method consists in high purity, uniformity and high reproducibility of structure and chemical composition of resulted powders [2]. The disadvantages of the method are the particle shapes which are close to the spherical one and other disadvantage is that they have high degree of oxidation, with negative implications on compaction and sintering processes. By water atomization process we can obtain powders with dimension of particles generally corresponds between 20-400 micrometers, dimension requires in powder metallurgy processes.

Through variations of atomization temperature (the temperature of molten iron stream), water pressure and atomization's time (sampling period) we influenced, mainly, the following characteristics of iron powder: oxygen content, apparent density, proportion of fine and large particles and shape and size of iron powders.

Using electron probe X ray micro analyzer and image analysis of samples with the aid of software provided by IPA institute from Bucharest have could analyze and quantify the

shape of iron particles and particle size distribution of three types of powders at of different atomizing temperatures.

2. STUDI ES OF PHENOMENON OCCURRING DURING AT OMIZATION AND TH E TECH NOLOGICAL PARAMETE RS THAT INFLUENCE THE PARTICLES MORPHOLOGY

Researches conducted until now have shown that water spray mechanism consists in five stages [2]: Stage I - to initiate the formation of smaller jet turbulent liquid surface; at this stage there is interaction between water and liquid metal valve; Stage II - jet fragmentation and formation of ligament-type fragments in areas of turbulence; Stage III - primary disintegration (atomization), which occurs breaking of ligaments in drops and tearing thus will form irregularly shaped particles (when surface tension is high and low cooling rate) and regular (when surface tension is small and high cooling rate). In stage IV the secondary atomization of the melt flow occurs when molten primary particles are deformed and fragmented into very fine particles. The stage V consists in secondary disintegration (atomization), where particle agglomeration and coalescence of them in larger porous or compact particles, followed by solidification of atomized particles and forms very fine particles took place.

The required characteristics of the powders depending on the atomization process parameters. Thus, by modifying the processing parameters we can control the shape and particle size distribution of powders. The most important atomization process parameters are the characteristics of molten metal (chemical composition, temperature of molten metal stream, design and configuration of the jets, nozzle diameter), the atomizing fluid characteristics (pressure and volume of the water, thickness of the stream of metal, the atomization angle, α, according with Fig. 1 and duration of atomization [3].

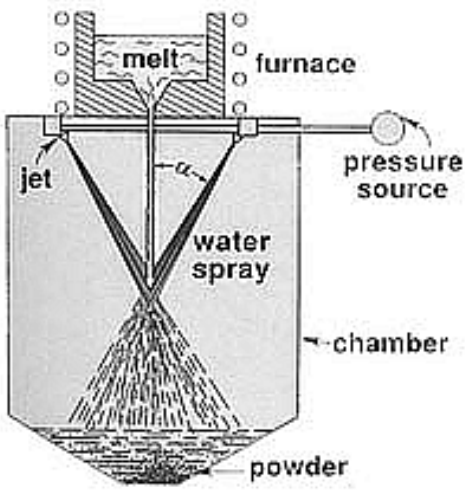


Fig. 1. Water Atomization Process [3]

An appropriate distribution size can be obtained by increasing the pressure of atomizing fluid and by decreasing the nozzle diameter. The condition to ensure a higher proportion of fine particles are a) low viscosity of the melted metal; b) low surface tension of liquid metal flow; c) low speed flow of molten metal, d) high pressure and speed of the water spray, e) reducing the length of metal flow and water jet and finally, f) optimum atomization angle, α [1-3].

The shape of particles depends upon the degree of cooling and solidification of liquid droplet. If the cooling rate is high, particles will solidify faster and will have an irregular shape. If the cooling rate is slow or if it was more overheated liquid metal, surface tension forces will restore the spherical particle as soon as a drop of liquid leaves the high turbulence.

Therefore, to obtain a irregular shaped form particles we must to decrease the nodulizing's time (by increasing the range of solidification), to decrease the surface tension by addition of surface active elements (as B, Mg, P, S, Ti, Zn, Zr, etc.) and to control the melt temperature, so that the melt solidification to take place inside the atomization zone with high turbulence. Another aspect followed during the water atomization process is that the degree of oxidation of molten particles to be minimal. For this reason, melt temperature and water pressure must be as low as possible and the cooling speed of the particles formed to be higher.

3. EXPERIMENTAL MATERIALS AND METHOD

The three types of powder S069, S070 and S071 was elaborated by water atomization of molten iron in the following condition. The atomization parameters were: the temperature of molten iron stream is 1680-1720^{o}C; the nozzle diameter is 13-15 mm; the atomization angle, $\alpha$is in the range 8.5-11^{o}; water pressure: 40-90 bar; water flow: 40-70 l/min; and time of atomization: 5-40 min. The technological parameters after atomization were: drying temperature was about 330^{o}; holding time for drying 90 min; heat treatment temperature: 950-1050^{o}C; holding time: 60-90 min; the flow of NH_3 was 110-150 m^3/h; and thickness layer of powders in trays was 9-18 mm.

Samples obtained by water atomization, dried and heat treated were characterized from physical, chemical, technological and structural point of view. By the chemical macro-analysis (performed with similar methods used to analyze castings` metals and alloys) were determined the chemical composition and especially the oxygen content of investigated powders (according with SR EN 24491-4 standard). We know that a higher quantity (higher than 1.2 %) of oxygen content decrease the compressibility of the powder and conduct to obtain the lower mechanical characteristics of the final product. The apparent density of powders is one of the most critical and important characteristics of powders, because it determines the actual volume occupied by mass of powder and determines the magnitude of the applied force for compact and densification the loose powder [2-4].The apparent density was made according with SR EN 23923-1:1998 standard.

The structural analysis was investigated by X ray microanalyzer and with Optic Quantitative Microscopy with computer assistance. Analyses were carried out using a JEOL JXA5A electron probe X ray microanalyzer. Samples were deposited on a glass support upon which sat a carbon double-sided tape (on one side the tape was stuck on the support device and the other side of the tape were pasted powder samples) [5].

Quantitative analysis of powder have been suspended in alcohol and put for analysis on a glass` slide for microscopic analysis at "Neophot 2" –type optical microscope, which on was installed a video camera. The captured image analysis was done with specific software. The main objective of X-ray microanalyses and optical microscopic quantitative analysis was to determine the shape and size of particles. Determination of particle shape was performed on powder samples with size less than 100 μm.

Qualitative estimation of the powder grains shape is expressed by the shape factor "f" which represents the statistic average ratio between maximum diameter (D_{max}) and minimum diameter (D_{min}) of grains. For spherical shape, the shape factor is equal to 1 and for a elongated form of granules that "f" have values between 2 and 4. For needle shape "f" is more than 8. The average diameter of particles for each analyzed fields was determined using the relationship: $D_m = (D_{max} + D_{min}) / 2$.

4. RESULTS AND DISCUSSION

There were investigated three types of iron's powders (S069, S070 and S071) from chemical and technological point of view. The obtained values of chemical compositions are given in Table 1 and the values of oxygen content and apparent density depending on atomization's time are given in Table 2.

Table 1. The chemical composition of the investigated iron powders

Type of powders	Chemical composition, %						
Standard / Specification / Sample no.	C	S	P	Si	Mn	O_2	Fe
	Max. 0.12	Max. 0.02	0.018	Max. 0.05	Max. 0.20	Max. 1.20	
S069	0.021	0.018	0.011	0.017	0.105	1.09	Balance
S070	0.123	0.013	0.006	0.014	0.135	0.96	Balance
S071	0.071	0.008	0.002	0.019	0.064	0.92	Balance

Table 2. The values of oxygen content and apparent density of iron powders depending on atomization's time

Atomization's Time, min / Sample no.	The oxygen content, %			The apparent density, g/cm^3		
	S069	S070	S071	S069	S070	S071
5	0.928	1.09	0.989	3.367	3.296	3.286
10	0.937	1.124	1.123	3.414	3.389	3.341
15	0.965	1.136	1.130	3.492	3.428	3.384
20	0.98	1.148	1.189	3.52	3.504	3.486
25	1.03	1.159	1.212	3.576	3.526	3.526
30	1.196	1.173	-	3.612	3.616	-

As it can be seen in Tables 1 and 2, it is noted that, during atomization, the oxygen content is within the prescribed standards, at max. 1.2% (except the sample no. S071 obtained at 25 minutes). It can be seen from Table 2 that the oxygen content and also, the apparent density continuously increase with the increasing atomization's time.

Samples obtained at the highest atomization temperature of 1720^o C were noted with "1" (for e.g. S070-1) at 1714^{o}C, were noted with "2" and so on until the last (the sixth) at temperature of 1687^o C denoted by "6".

The medium diameter obtained as a result of image analysis with the aid of Optic Quantitative Microscopy with computer assistance is shown in Fig. 2. We presented the values of minimum, maximum and medium diameter of sample S069 obtained at 1720^{o}C (Fig. 2a) and respectively at 1687^{o}C (Fig. 2b).

After analyzed all samples S069, S070 and S071 obtained at all six atomization temperature (1720-1687^{o}C), the values of medium diameter depending on atomization temperature were graphically presented in Figs. 3, 5 and 7. The size of particles of samples S070 are presented in Fig. 4 and respectively Fig. 5 (a and b).The form factor and structural analysis investigated by X ray microanalyzer are shown in Figs. 8 and 9.

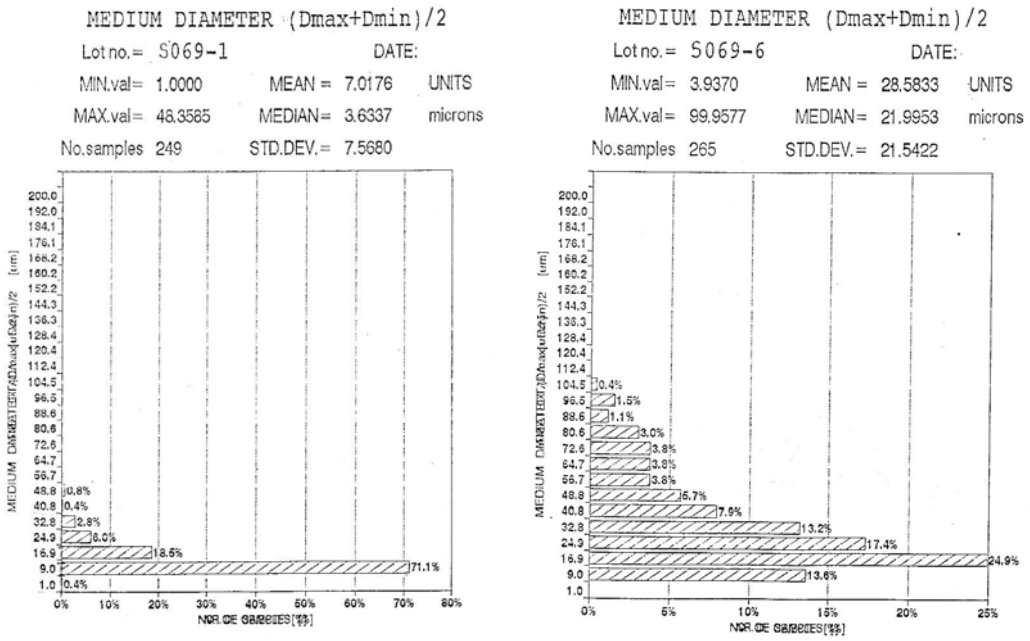


Fig. 2. The medium diameter obtained as a result of image analysis with the aid of Optic Quantitative Microscopy with computer assistance for samples: a) S069-1; and b) S069-6

As it can be seen in Fig. 2a is found that 0.4% of particles are very fine particles with average diameter dm = 1 to 10 μm, the largest proportion, about 98.8% were fine particles with an average diameter of 10 to 40 micrometers from which about 71.7% are particles with dm = 9-16.9 μm and only 0.5% are fine powders having an average diameter dm = 40-150 μm. Figure 2b) shows that the majority, around 77% owning a fine of dm = 9-40.8 μm, plus a percentage of 23.1% of fine particulate medium degrade with dm = 40.8-150 μm, 0.4% of which are particles with average diameter greater than 100 microns (dm = 104.5 μm).

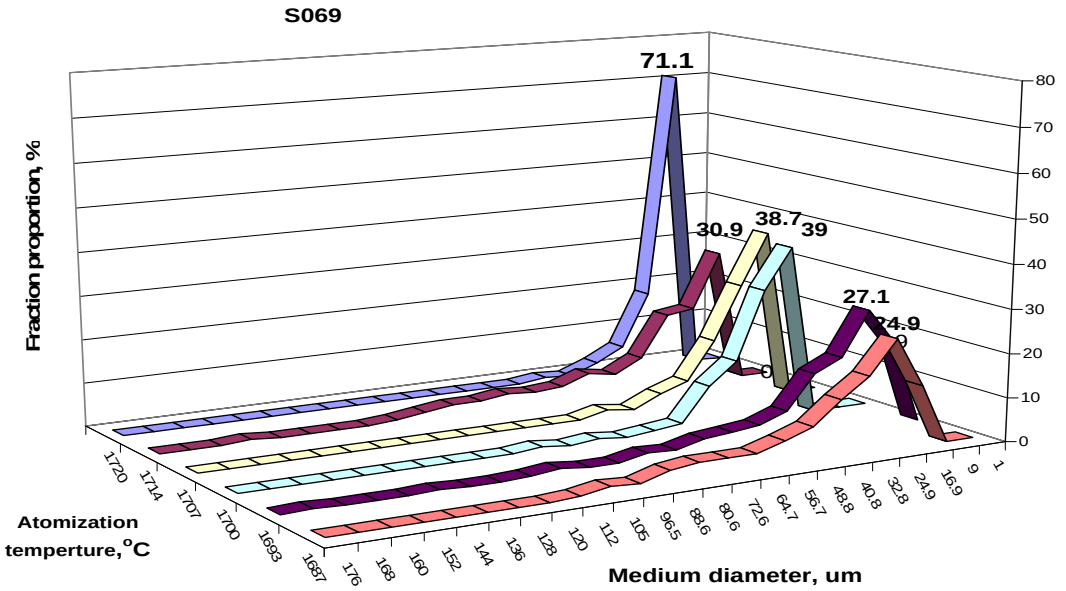
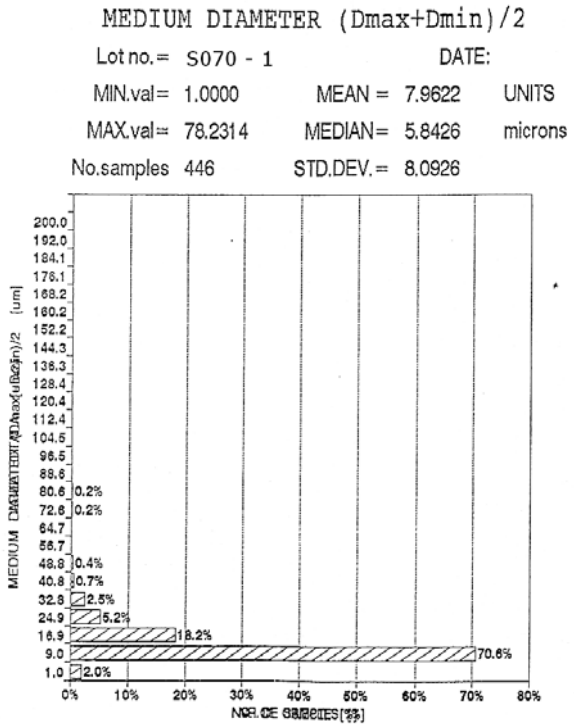


Fig. 3. The particle size distribution (fraction proportion of medium size) of samples S069 depending on atomization temperature

For sample S069 note that with decreasing of atomization temperature decreases the fractions proportion of fine powders (Fig. 3).



Note that only 67.1% are fractions of fine powders with medium diameters ranging between 9 and 40.8 μm, about 32.2% have a degree of fine of powders with average diameter being dm = 48.8-144.3 μm and 0.9% are coarse powders with dm = 152.2-210 μm.

Fig. 4. The medium diameter obtained as a result of image analysis for samples: S070-1;

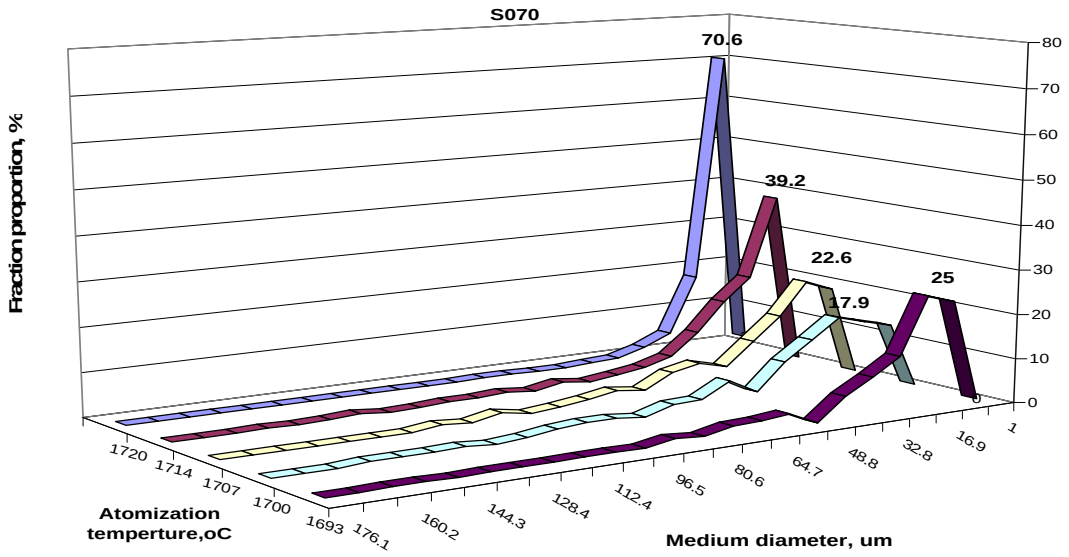
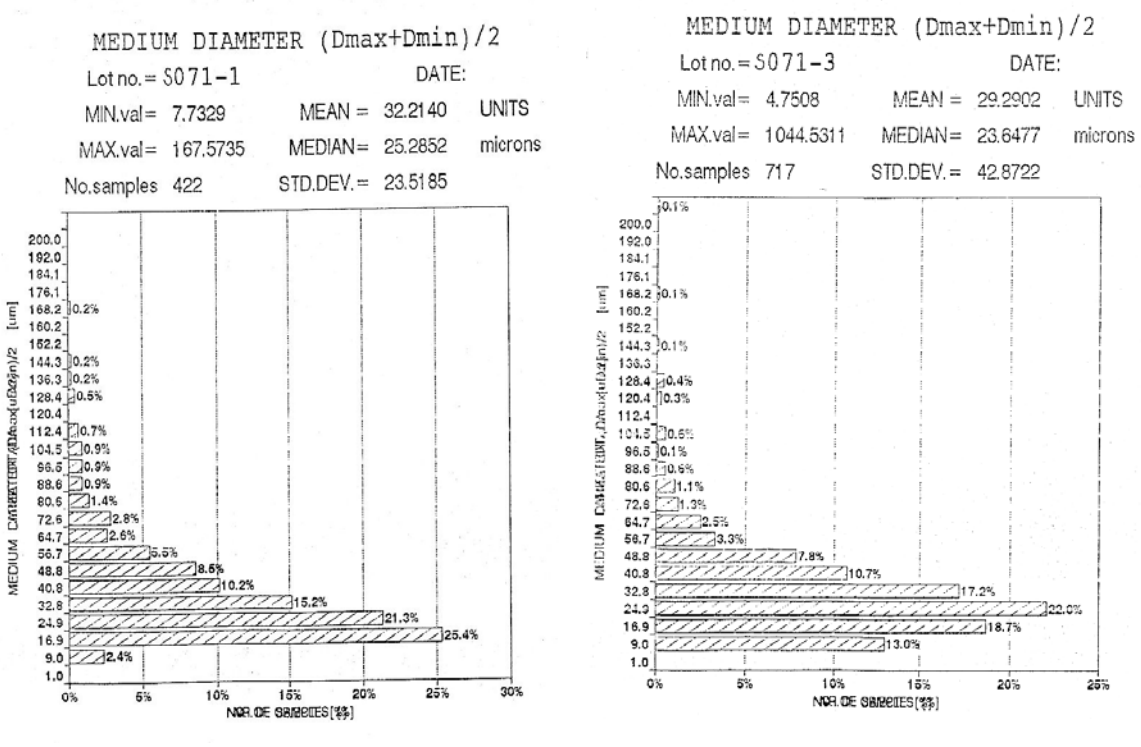


Fig. 5. The particle size distribution (fraction proportion of medium size) of samples S070 depending on atomization temperature



a) **b)**

Fig. 6. The medium diameter obtained as a result of image analysis for samples: a) S071-1; b) S071-3

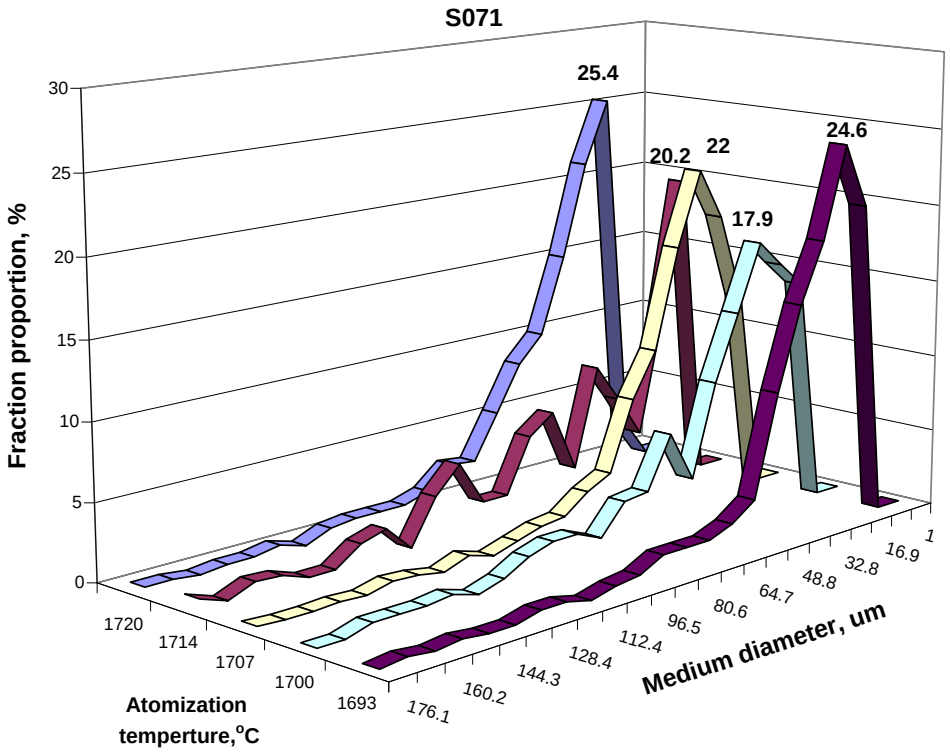


Fig. 7. The particle size distribution (fraction proportion of medium size) of samples S071 depending on atomization temperature

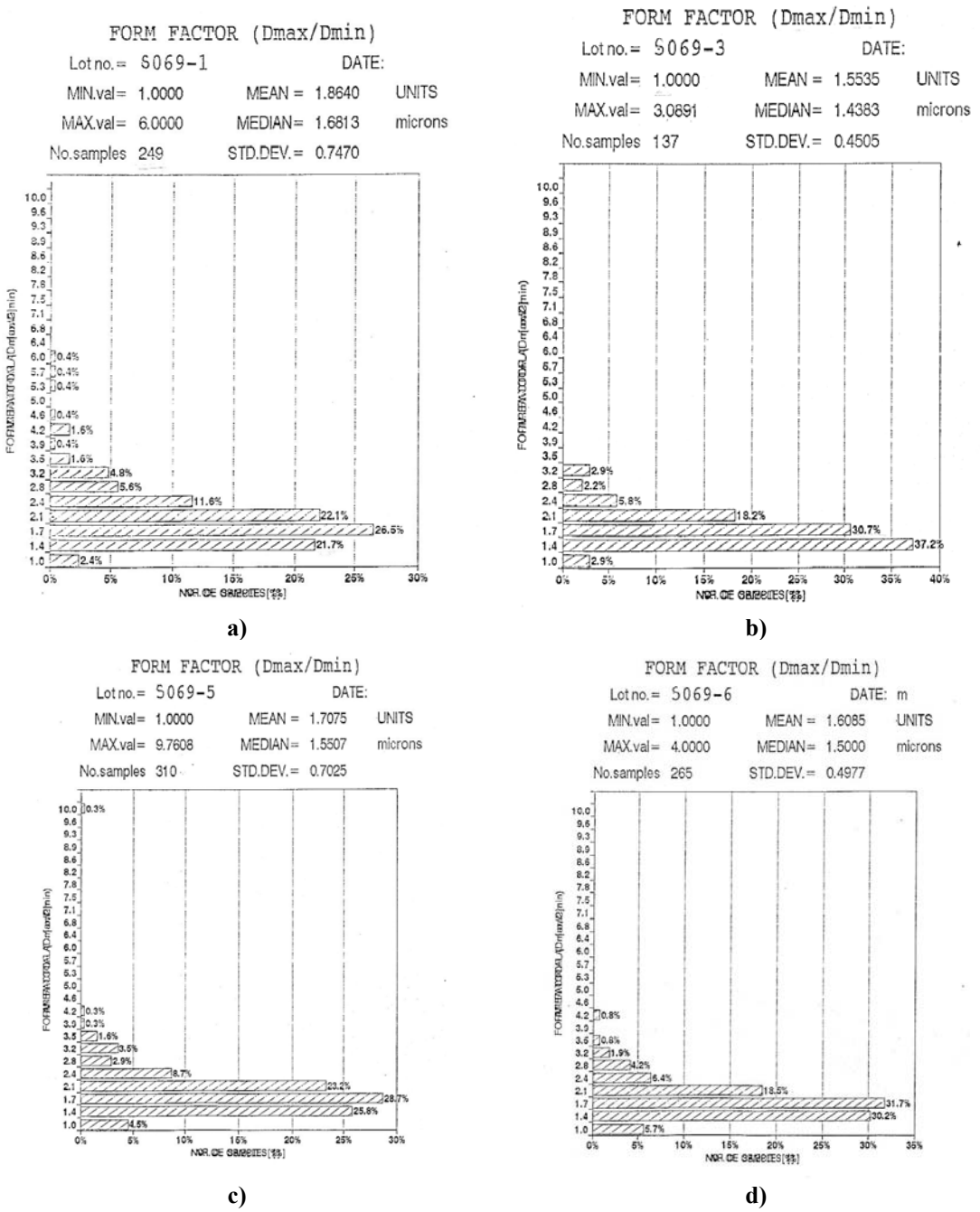
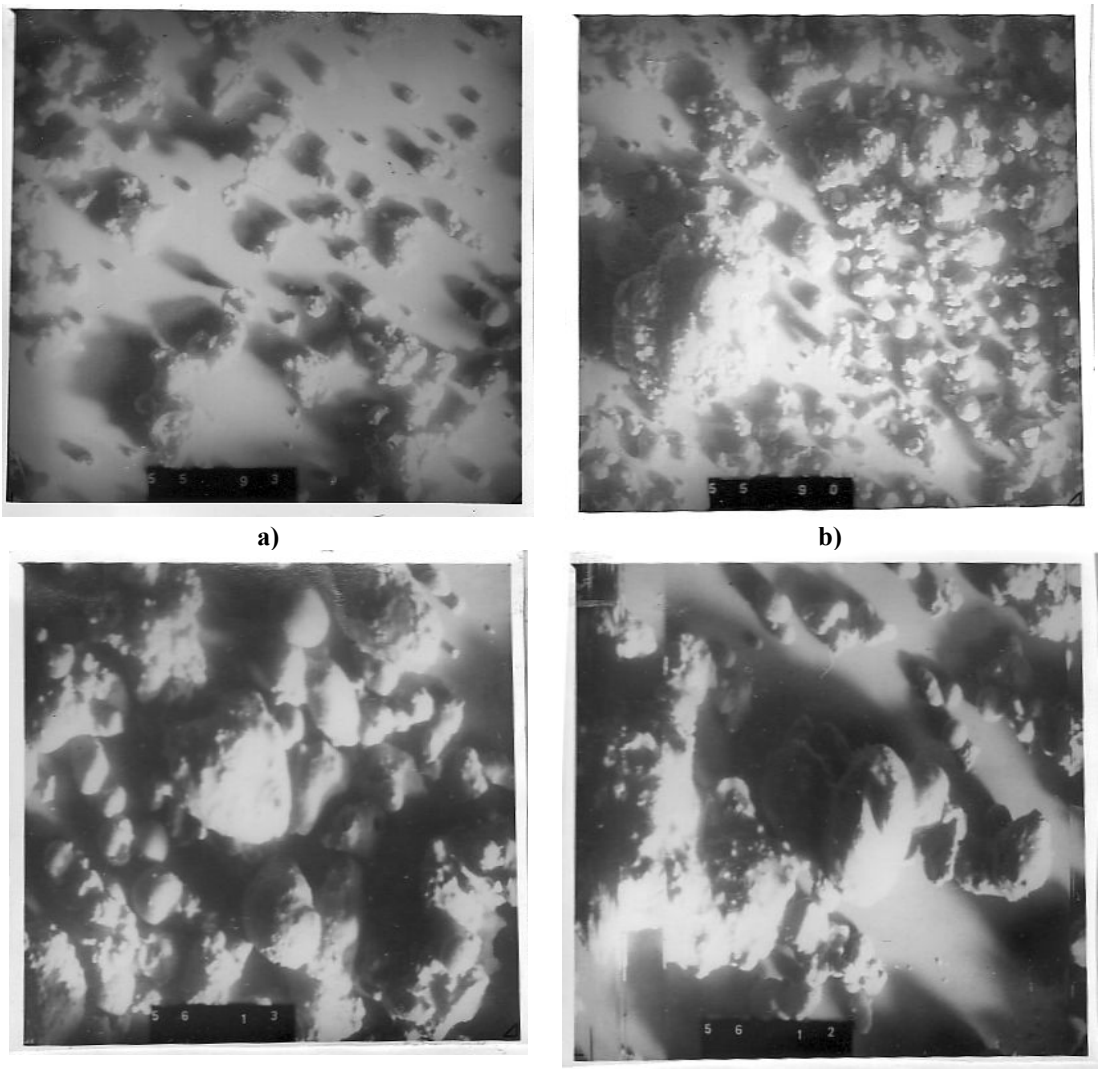


Fig. 8. The form factor obtained as a result of image analysis for samples: a) S069-1; b) S069-3; c) S069-5; d) S069-6

The factor form of the Fig. 8a reveals that only 2.4% of analyzed particles are spherical, 3.2% of needle-shaped particles and the largest percentage, 94.3% presenting it to elongated particles. In Fig. 8b it observes that approximately 2.9% of the particles are spherical particles, the highest percentage, 97% is the elongated particles. There wasn't evidenced these acicular particles in the field analyzed. But in Fig. 8c) are revealed that sample S069-5 contains about 4.5% spherical granules and a majority share of 95% elongated particles, and there was a rate of 0.3% needle shaped particles. Finally, at the lowest temperature, sample S069 contains 5.7% of spherical particles and a majority share of 94.5% elongated particles. The samples S070 and S071 are a similar shape distribution (the similar percentage of spherical and irregular shapes).



a) **b)**

c) **d)**

Fig. 9. The structural analysis, investigated by electron probe X ray micro analyzer for samples: a) S069-1; b) S069-3; c) S069-5; d) S069-6

5. CONCLUSIONS

During an atomization cycle, due decreasing of molten iron stream temperature, the particle size fractions of powder obtained are reducing with the proportion of fine particles, from 71.7% to 70.6%. This variation is due to the influence of fluid pressure agent (water) that alters the morphology and size distribution of powders obtained. If atomization is done using low pressures (p = 40-50 bar) of water agent of atomization (for samples S069), we obtained fractions, where fine particulates are the majority. Maximum proportion (71.1%) of fine particles is registered for sample S069 and maximum proportion for S070 sample are 70.6%. The shape factor "f" (1.4-4.2) confirmed the irregular shape of powder particles analyzed. It was noted that fine particles predominate average diameter ranging from 9-40.8 micrometers, the smallest proportion of large (coarse) particles with Dm> 150 μm.

Morphological appearance of iron powder show that the powder from Fig. 9a has the appearance of stage III primary disintegration and shows both regular and irregular particles; Fig. 9b has the appearance of secondary disintegration corresponding stage IV, when particles fragment into fine particles. And in Figs. 9c and 9d correspond to stage V of agglomeration (Fig. 9c) and coalescence of particles (Fig. 9d).

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