

EFFECT OF PARTICLE SHAPE AND SIZE OF COPPER POWDERS ON THE PROPERTIES OF SINTERED PARTS

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ABSTRACT

The particle shape and size of the starting powders represent the most important physical properties, on which the quality of the compacts and final sintered products depends. Two types of powder were analyzed in the paper - electrolytic copper powder with a dendritic particle shape and water-atomized copper powder with an irregular particle shape. The starting powders were sieved through a sieve system with openings of 45 μm , 80 μm , and 120 μm . The characterization of the obtained fractions of both powders was performed by determining the shape and dimensions of the particles using SEM microscopy in combination with ImageJ software, and the apparent density and flow rate were determined using the Hall flowmeter funnel. Pressing of each powder fraction was done using a pressure of 600 MPa. The compacts were further sintered at 1000°C for 2 hours to obtain the final sintered parts. After sintering, their density, hardness, and electrical conductivity were determined and their microstructure was analyzed. The results indicate a great influence of the characteristics of the starting powders on the properties of the final parts obtained by the powder metallurgy route. The particle shape of the powders had a more pronounced influence compared to the particle size.

1. INTRODUCTION

The powder metallurgy (PM) route includes obtaining the final products through compact the starting powders and sintering them under controlled conditions. The main disadvantage of the classical PM process is the impossibility of achieving a full density of final products, due to their porosity, and that is why the mechanical properties of sintered parts are lower [1,2]. Properties of sintered parts depend on the parameters such as characteristics of the starting powders, compaction pressure, sintering temperature and time, etc. If the compaction pressure, sintering temperature, and time are higher, the density is superior, and the porosity is lower. Mechanical and electrical properties are also dependent on the density and porosity, therefore, they increase with an increase in compaction pressure, sintering temperature, and time, but all this increases the process cost [3].

In addition to the process parameters, the particle shape and size have a great influence on all operations during obtaining parts using the PM route and on the quality of the final sintered products. The particle shape has a large effect on the contacts between particles and therefore has a significant influence on their behavior during pressing and sintering. Generally, the compacts obtained from coarse powder particles have a higher density compared with the compacts from fine powders [4].

To obtain the required properties in the final sintered parts, a suitable combination of process parameters is required [5,6]. This paper presents the effects of particle shape and size of the starting copper powders, on the properties of powders, green compacts, and sintered samples. In the case of powders, the evaluated properties were apparent density and flow rate. In green compacts, density was evaluated, and in sintered samples hardness, electrical conductivity, and microstructure of PM parts were discussed. To obtain samples with different average particle sizes, water-atomized and electrolytic copper powders were sieved and separated with sieves with openings of 45 μm , 80 μm , and 120 μm .

2. EXPERIMENTAL PART

Water-atomized copper powder (supplied by Centrochem) with an irregular particle shape and electrolytic copper powder (supplied by Pometon) with a dendritic particle shape were used as starting materials. Commercial powders were sieved to obtain fractions with a narrow powder particle size distribution (in the range of about 40 μm). The powders were sieved on sieves with openings of 45 μm , 80 μm , and 120 μm . In this way, three fractions were obtained: - 45 μm , + 45 μm - 80 μm , and + 80 μm - 120 μm . The powder's designations are given in Table 1. The first letter in the designations indicates whether the powder was obtained by water-atomization (A) or by electrolytic method (E). Other letters indicate the range of particle sizes in that powder: very fine (VF) with particles below 45 μm , fine (F) with particles from 45 μm to 80 μm , and medium (M) with particles from 80 μm to 120 μm . This classification is based on the paper [7].

Table 1. The powder's/sample's designation

| Fractions | Atomized powders | Electrolytic powders |
|-----------|-----------------------------|---------------------------------|
| - 45 | A-VF (Atomized - Very Fine) | E-VF (Electrolytic - Very Fine) |
| + 45-80 | A-F (Atomized - Fine) | E-F (Electrolytic - Fine) |
| + 80-120 | A-M (Atomized - Medium) | E-M (Electrolytic - Medium) |

The characterization of the powders included the determination of the shape and morphology of the particles, particle size distribution of powders, the apparent density, and the flow rate of the powders. The shape and morphology of the particles were determined using SEM "Tescan VEGA 3LMU" microscope. The average particle size and particle size distribution of each powder were obtained based on SEM microphotographs, using the image analysis software ImageJ. The particle size was measured along the longest axis. The apparent density and the flow rate of the powders were examined using the Hall flowmeter funnel. The apparent density was done according to the ISO 3923 standard, and the flow rate was according to the ISO 4490 standard [8,9].

The powders were further compacted on a hydraulic press "Mohr & Federhaff & Losenhausen" with a pressure of 600 MPa. The compacts were sintered in a furnace "Elektron ELP-08" in an atmosphere of argon at a temperature of 1000 $^{\circ}\text{C}$ for 2 h, to obtain sintered parts. Density, hardness, and electrical conductivity were measured and the microstructure was analyzed using optical microscopy. The hardness was measured using "VEB Leipzig" Vickers hardness tester with a load of 10 kg. The device "Institute dr. Forster Sigmatest 2.063" was used for measuring electrical conductivity. The optical microscope used to observe the microstructures was "Carl Zeiss Jena Epytip 2".

3. RESULTS AND DISCUSSION

3.1 Characterization of powders

Figure 1 presents SEM microphotographs of copper powders with different sizes and shapes depending on the obtaining method. The water-atomized powders (Figs. 1a-c) appear in an irregular shape while the electrolytic powders show a typical dendritic shape (Figs. 1d-f).

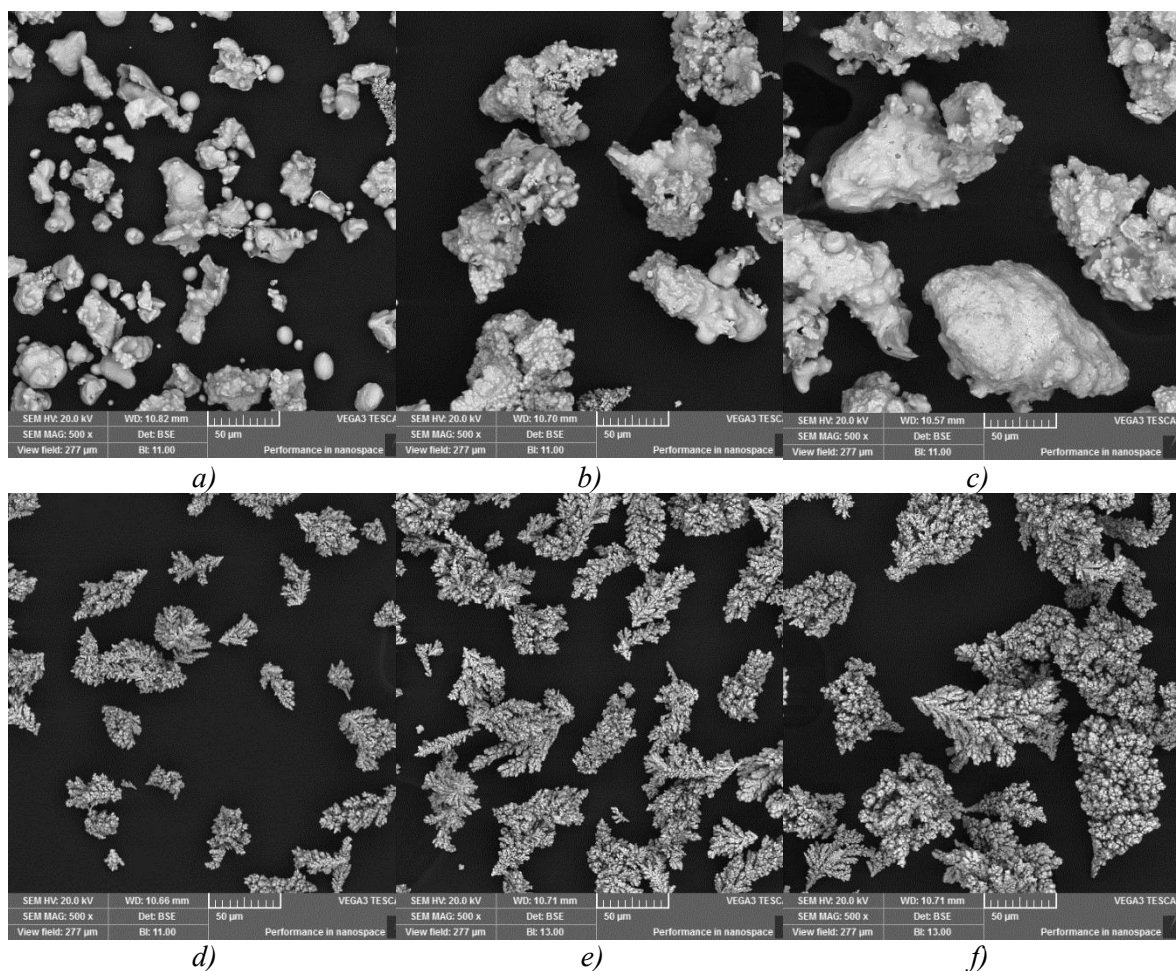
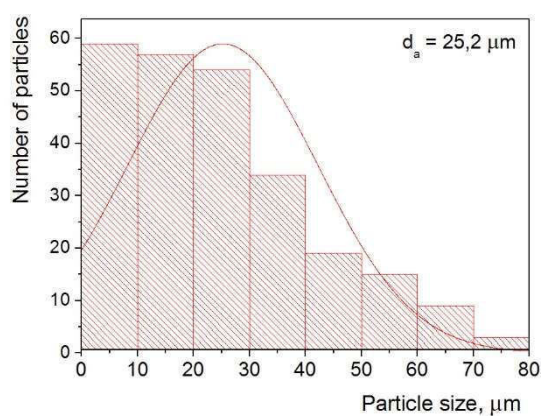
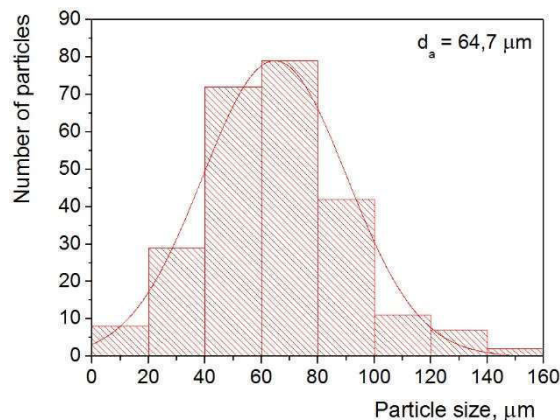


Figure 1. SEM microphotographs of copper powders (a) A-VF; (b) A-F; (c) A-M; (d) E-VF; (e) E-F; (f) E-M

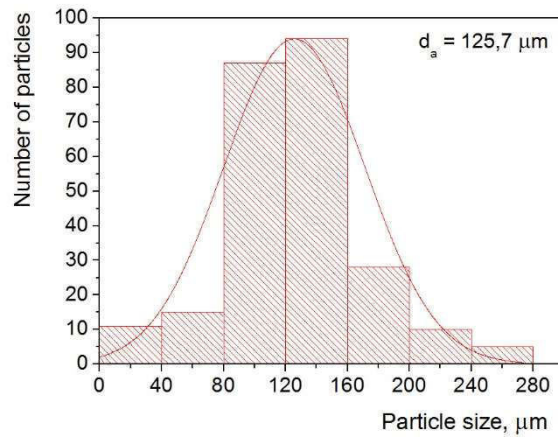
Figure 2 shows the average particle size and particle size distribution of atomized copper powders A-VF, A-F, and A-M. The A-VF powder has the finest average particle size as compared to others, which is 25.2 μm . Powder A-F is coarser, with an average particle size of 64.7 μm . The powder A-M is the coarsest with an average particle size of 125.7 μm .



a)



b)



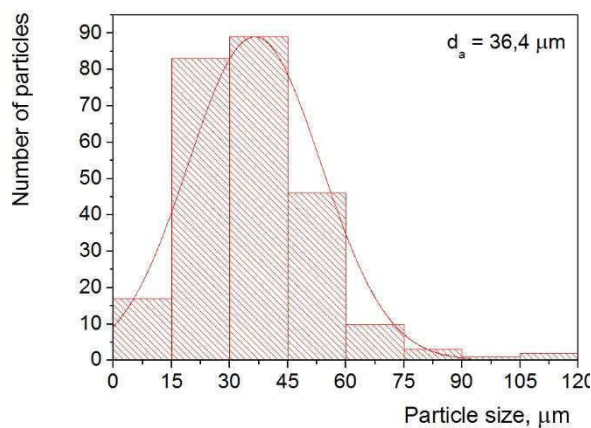
c)

Figure 2. The average particle size (d_a) and particle size distribution of atomized copper powders (a) A-VF; (b) A-F; (c) A-M

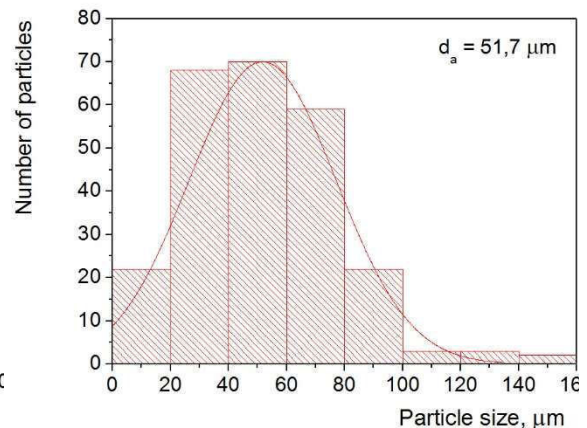
Figure 3 shows the average particle size and particle size distribution of electrolytic copper powders E-VF, E-F, and E-M. The E-VF powder has the finest average particle size, which is 36.4 μm . Powder E-F is coarser, with an average particle size of 51.7 μm . The coarsest electrolytic copper is E-M powder with an average particle size of 84.2 μm .

Comparing the same fractions of atomized and electrolytic powder, it can be noticed that VF atomized powder is finer than electrolytic one, while F and M atomized powders are coarser than F and M electrolytic copper powders, respectively.

Figure 4 shows the apparent density and the flow rate of loose powders. The apparent density of the metal powder is influenced by the theoretical density of the metal, the granulometric composition of the powder, particle shape, surface and roughness of the individual particles, particle packing, oxidation degree, and porosity. Generally, the apparent density increases with an increase in particle size (this behavior was not shown in this paper, but vice versa), if the particle shape becomes less spherical and more irregular and with an increase in surface roughness [10].



a)



b)

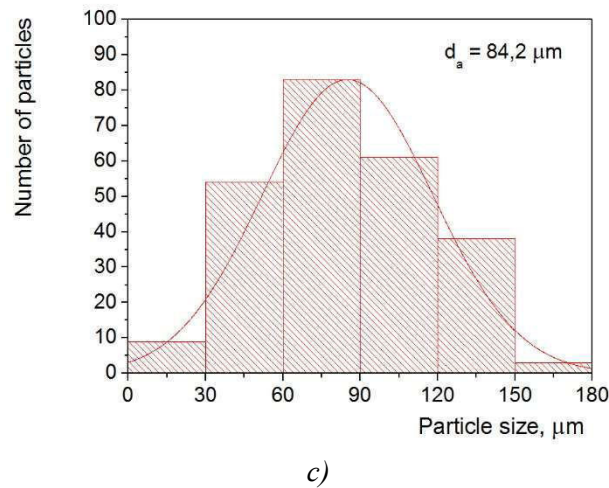


Figure 3. The average particle size (d_a) and particle size distribution of electrolytic copper powders (a) E-VF; (b) E-F; (c) E-M

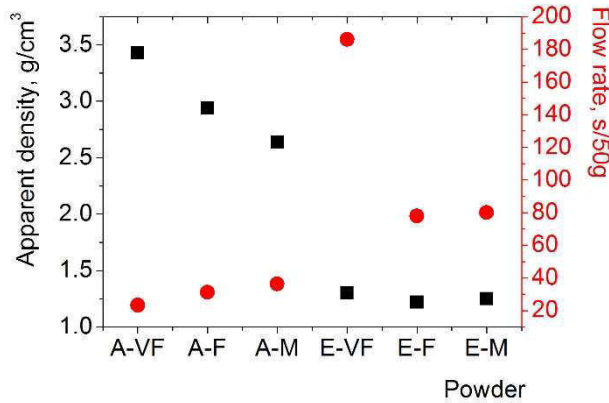


Figure 4. The apparent density and the flow rate of loose powders

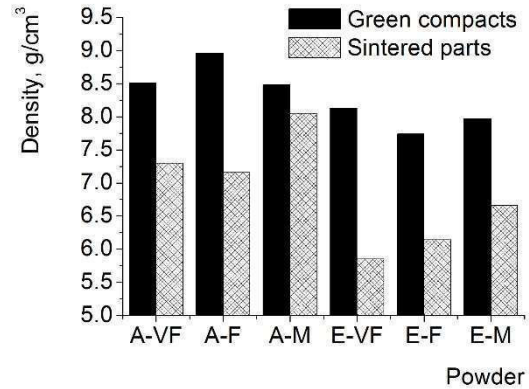


Figure 5. The densities of green compacts and sintered parts

It is generally observed that atomized copper powders have higher apparent densities and flow rates compared to electrolytic powders. This is related to the shape of the particles. In the case of dendritic electrolytic copper powder, the presence of bulges and dents in the particle surface, as well as the increase in the specific surface area of the particles increase the friction between particles, this makes it difficult to move the particles relative to each other and causes poor packing of the dendrite particles. Because of that, atomized powders have a higher apparent density and flow rate than dendritic powders [11,12].

The highest apparent density and flow rate are observed for A-VF powder (3.43 g/cm^3 and 23.32 s/50g , respectively). The lowest apparent density was achieved in E-F powder (only 1.22 g/cm^3), while E-VF powder showed the lowest flow rate (186 s/50g).

3.2 Characterization of sintered parts

Figure 5 shows the results of measuring the densities of green compacts and sintered parts from different types of powders. It can be concluded that the density of sintered parts decreased compared to the density of green compacts. This is unusual behavior. The expansion of the parts most likely occurred due to the reaction of residual gases in compacts with the atmosphere in the sintering furnace, i.e. with residual oxygen inside the furnace.

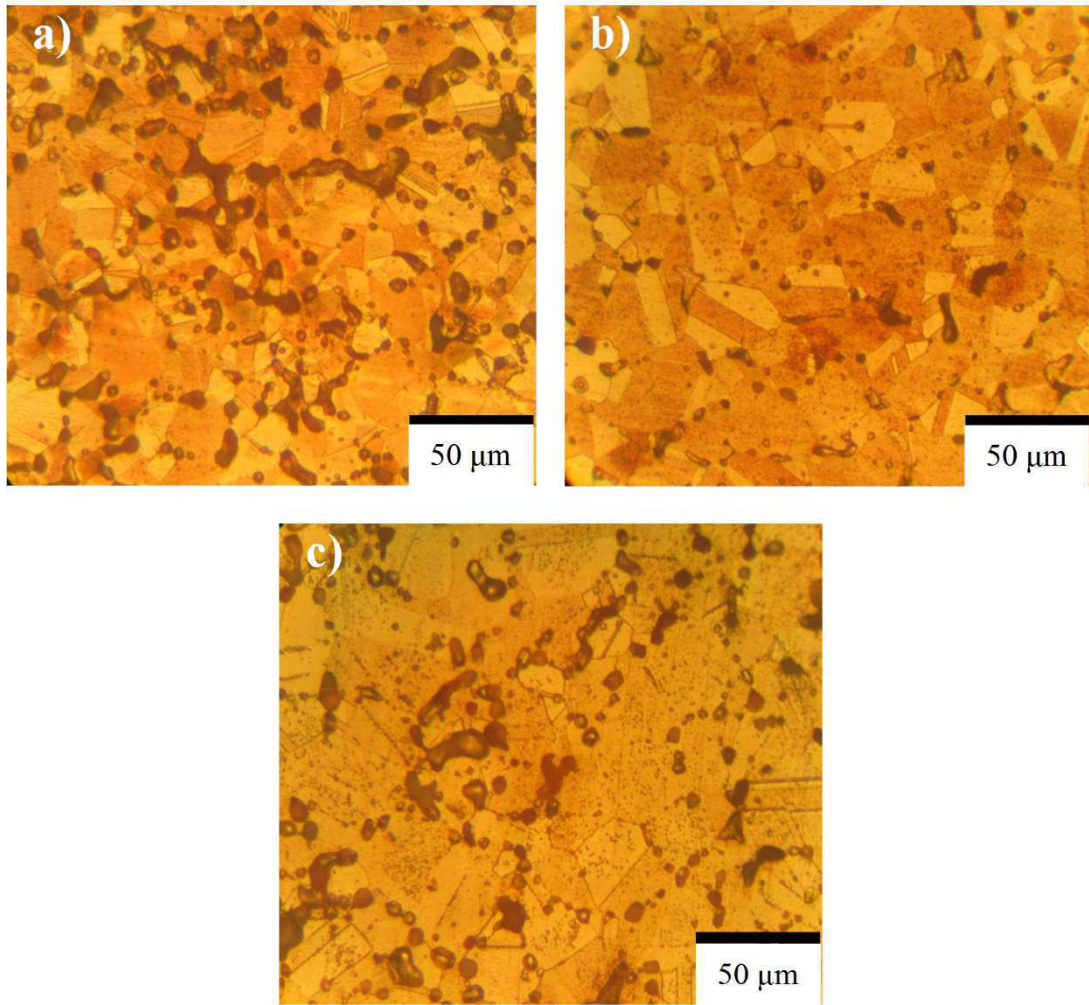


Figure 6. Optical micrographs of sintered copper obtained from atomized powder (a) A-VF; (b) A-F; (c) A-M

Sintered parts obtained from atomized copper powders have significantly higher density values compared to parts obtained from electrolytic copper powders. In the case of parts obtained from electrolytic copper powder, a clear tendency is observed that the density of the parts increases with an increase in the particle size of the starting powder. The same results were obtained by Chang and Wu [4]. This tendency is not expressed in parts obtained from atomized copper powder.

Figure 6 shows the microstructures of sintered parts obtained from atomized powders with different particle sizes. Polygonal grains with annealing twins are observed. The tendency of grains and microstructure consolidation is observed with an increase in the particle size of the starting powder from which the parts were obtained. Spherical pores are present at the grain boundaries and within the crystal grains themselves. The porosity is indicated, especially for the samples obtained from powders A-VF and A-M.

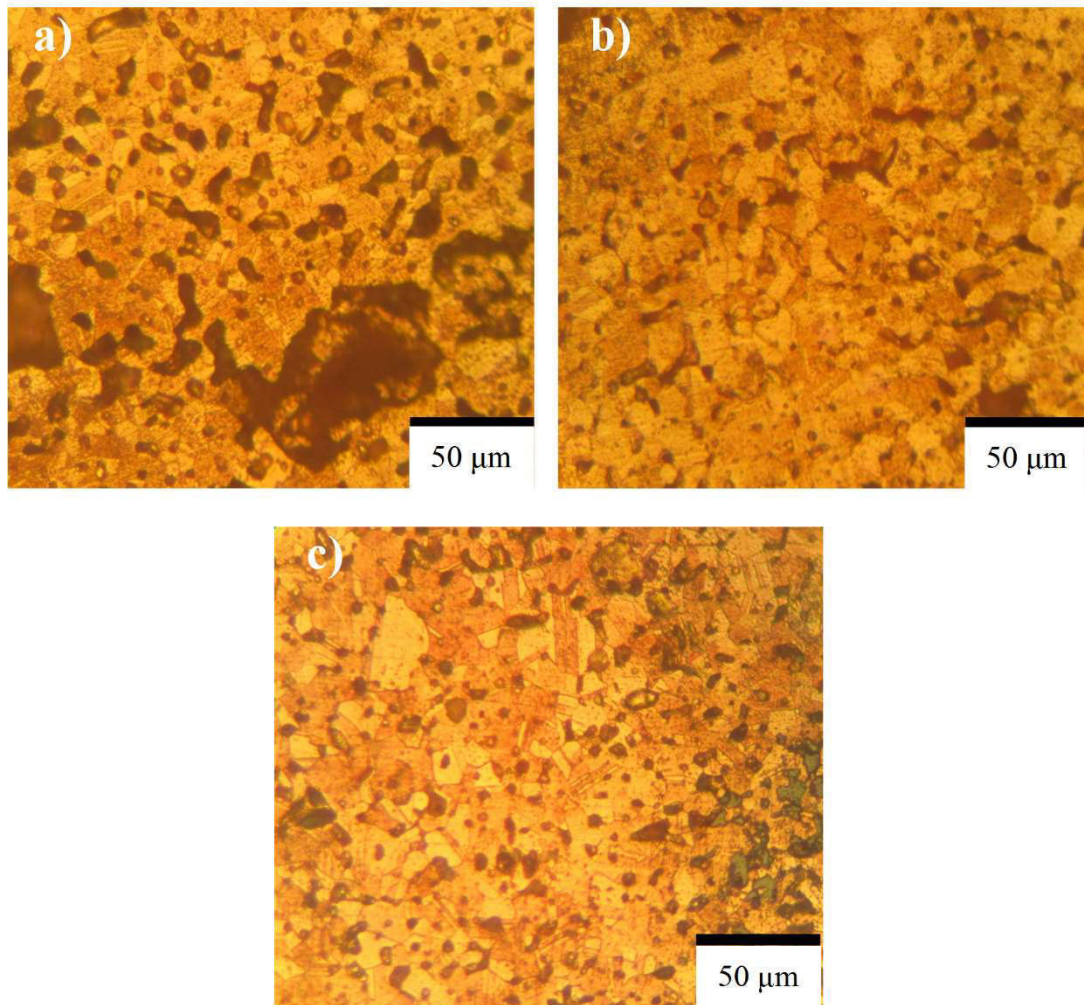


Figure 7. Optical micrographs of sintered copper obtained from electrolytic powder (a) A-VF; (b) A-F; (c) A-M

Figure 7 presents the microstructures of sintered samples obtained from electrolytic powders with different particle sizes. For the same starting powders (the same fractions), a finer-grained microstructure was observed in parts obtained from electrolytic powders compared to parts obtained from atomized powders. The microstructure is dominated by pores, which are large and different in shape. All sintered parts are more porous compared to samples obtained from atomized copper powders. The porosity is the most pronounced for the sample obtained from the electrolytic E-VF powder with the finest particle size, which was also confirmed by the results of the density measurement.

Figure 8 shows the hardness and electrical conductivity of sintered parts obtained from powders of different sizes and shapes. Sintered parts obtained from atomized powders have higher hardness and electrical conductivity values compared to parts obtained from electrolytic copper powders. This is understandable, considering the higher density values of the parts obtained from atomized copper. The sample from A-M powder has the highest hardness value, which is 34.57 HV10. The sample from A-F powder has the highest electrical conductivity, which is 38.15 MS/m. The lowest values of hardness and electrical conductivity were obtained for the parts obtained from the electrolytic copper powder E-VF with the finest particles, which also showed the lowest density value.

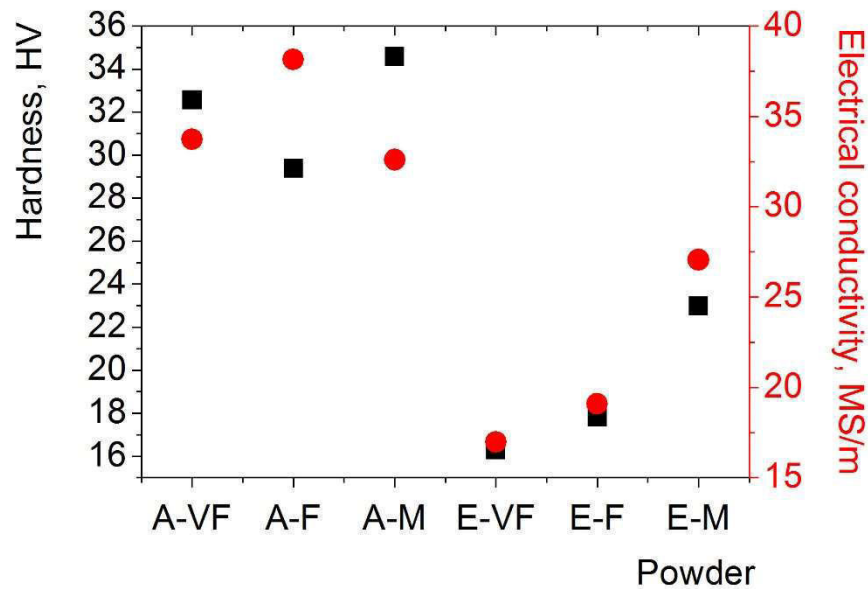


Figure 8. Hardness and electrical conductivity values of the sintered parts

4. CONCLUSIONS

The influence of the copper particle shape and size on some properties of powders, compacts, and PM parts was analyzed. The following conclusions can be drawn:

- Atomized powders showed higher values of apparent density and flow rate compared to electrolytic powders. Increasing the particle size of atomized powder showed a decrease in apparent density and flow rate (atypical behavior). However, increasing the particle size of electrolytic powder showed an increase in flow rate (typical behavior).
- The density of the sintered parts decreased compared to the density of the compacts. Sintered parts obtained from atomized powders have significantly higher density values compared to parts obtained from electrolytic powders.
- The finer microstructure was achieved in sintered parts obtained from electrolytic powders but with much more pronounced pores compared to the parts obtained from atomized powders.
- Sintered parts obtained from atomized powders have higher hardness and electrical conductivity values compared to parts obtained from electrolytic powders.

5. ACKNOWLEDGEMENT

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