ANALYSIS OF THE SURFACE POROSITY OF A SINTERED COPPER-NICKEL ALLOY OF DISSOCIATED METALLIC POWDERS

1Ricardo Cadaval Gonçalves, 1Diego Rodrigues da Silva, 1Luciano VolcanogloBiehl, 1Jorge Luis Braz Medeiros, 2José de Souza, 3Elton Gimenez Rossini, 4Alexandre Beluco and 4Alfonso Risso

1Universidade Federal de Rio Grande (FURG) - PPMec - Av. Itália, Km 8 - Campus Carreiros - Rio Grande/RS - 96201900 – Brazil; 2Fundação Liberato (FETLVC) - DPPI - Rua Inconfidentes, 395, Bairro Primavera, Novo Hamburgo/RS - 93340140 – Brazil; 3Universidade Estadual do Rio Grande do Sul (UERGS) - Engenharia - Rua 7 de Setembro, 1156 - Centro Histórico, Porto Alegre - RS 90010191 – Brazil; 4Universidade Federal do Rio Grande do Sul (UFRGS) - IPH - Av. Bento Gonçalves, 9500 - Porto Alegre/RS - 91509900 – Brazil

ABSTRACT

Powder Metallurgy P.M. is a technique that uses the principle of forming metal alloys from the mixture of metal powders to form materials with particular characteristics such as controlled hardness, mechanical strength, and porosity. It is possible to develop this control by varying the parameters, such as temperature and pressure of compaction, reaching the desired levels. This paper evaluated the behavior of surface porosity in the variation of the copper-nickel concentration of dissociated powders, maintaining sintering temperature and compaction pressure constant. To determine the porosity of the copper-nickel alloy, the Scanning Electron Microscope (SEM), ImageJ, and statistical calculations used to generate graphs to represent the results. The results indicated that there was no linear behavior of the surface porosity.

INTRODUCTION

New properties in the metal alloys have been researched and are being achieved with the same materials, but with new compositions, additions of alloying elements, techniques involving mainly the management of energy and time. To optimize profitability and compliance with environmental standards, Powder Metallurgy is at the forefront of most conventional techniques, such as smelting, when considering such criteria [1-2]. There is a trend to explore sintered materials for the manufacture of self-lubricating bearings and products with complex geometry and good quality finishing. Elimination of the need for finishing processes such as machining or polishing. Self-lubricating bearings are a new trend as oil passes through the pores of the bearing material [3-4]. By determining the porosity rate of the material, it is possible to determine the oil flow that will reach the axis.

This paper aimed to realize the sintering of copper-nickel alloy to analyze the influence of the independent variable, chemical composition, of the alloy on the mechanical-metallurgical properties and analyze the crystalline structure microscopically to determine the degree (percentage) of porosity of the piece, using software, influenced by the effect of the independent variable. Powder Metallurgy (PM) consists of the technique of transforming metal powders into parts with excellent strength, without melting, but only using pressure and heat [5-6]. The P.M. technique differs from the other manufacturing processes by the absence of the liquid phase or the occurrence of this stage in only one of the materials involved. Parts with complex geometries fabricated with the least waste of material since the part will not require subsequent finishing processes [7-8]. P.M. is composed of two phases, one of compaction or molding and the other of heating or sintering [9-10]. Nickel is one of the most versatile materials in metallurgy and is an essential alloying element present in many materials such as cast irons,
steels, and non-ferrous alloys. Also outstanding for its excellent corrosion resistance. This element is used for the manufacture of rechargeable batteries, coins, and metallic coatings [11]. Copper is a non-magnetic metal and can be used pure or forming alloys with other metals, which gives it excellent chemical properties. The most important characteristics of copper are its high electrical conductivity, good magnetic permeability and its malleability, which makes this metal the most used in the manufacture of electrical materials [12]. Cu-Ni alloy has good ductility, good mechanical and oxidation resistance, excellent thermal conductivity. Is easily cold-formed and can be processed into sheets, wires, tubes and bars. It can be welded by most welding methods [12]. The alloy Cu-Ni (monel) alloy has a commitment to mechanical strength, ductility and corrosion resistance particularly in corrosive media such as sea water [13]. The most common alloy is 30% copper and has the monel designation. Monel 400 is an alloy more resistant than nickel under reducing conditions and more than copper under oxidizing conditions and is mainly free of the phenomenon of stress corrosion. The equilibrium diagram of the Cu-Ni system as a function of temperature and composition consists of a solid (bottom) line and a liquid line, which divide the diagram into three areas. In the field above the liquid line, all the compositions exist in the liquid state, while below the solid line all existing structures are solid. In the area between the liquid and solid lines, the solid coexists with the liquid [14]. Sintering is the final phase of the process and joins loose particles and decreases porosity through thermal energy. The process gives the piece the ideal mechanical strength because of the strengthening of the contact between the particles, altering the pore geometry and standardizing the microstructure [9]. It generally occurs at a temperature below the melting point of the main constituent element [10]. For sintered steels with high density and high strength specifications it is necessary to reach the final stage, sometimes incompletely, or only until the end of the intermediate stage [15]. The porosity is a characteristic, which is most often undesirable because it makes the material more mechanically fragile, that is, the probability of fractures is relatively greater to the same material that does not have pores in its structure [15]. There are many applications for the use of this feature as metal filters, the manufacture of tools for the drilling of oil deposits or mining that benefit from porosity [16]. Particle size is another factor that has a direct influence on the porosity, together with the irregularity of the article can cause the porosity to vary significantly maintaining the same pressure and temperature (Fig. 1) [17].

Another important aspect for the control of porosity is the compression pressure, as the pressure increases, the voids decrease proportionally, this is because the particles are forced to occupy spaces that previously were empty.

**MATERIALS AND METHODS**

The alloy is composed of Cu-Ni in the form of powder, obtained through the atomization process and have a granulometry respectively of 11% of the element with 200 Mesh and 89% of the element with more than 325 Mesh. For the preparation of the test samples, the mass concentration varied from 90% Ni and 10% Cu to 90% Cu and 10% Ni in 10%. The samples have a composition granulometry of 11% with 200 and 89% of the element with more than 325 Mesh. For the samples preparation, the mass concentration composed of 90% Ni and 10% Cu. The temperature remains at 1000 °C, which is of the order of magnitude lower than the melting point of Cu-Ni. The time at which the compressed to thermal energy was 30 minutes. From the images of the SEM analyzed with that of EDS (Energy Despertive X-ray Spectroscopy) the surface porosity was measured with the aid of image manipulation software, ImageJ, which distinguished by the difference of color shades, the concentration of material and where it has not characterized the porosity. After applying the filter "Analyze Particles" tool used to measure the percentage of each element.

**RESULTS AND DISCUSSION**

Random points were chosen to perform the analyses, to exemplify this procedure, the sample S1 with 90% Cu and 10% Ni concentration (Fig. 2).

![Fig. 2. Micrograph of sample S1 with magnification 2000x](image)

Point 1 that deals with an image indicating an enlargement of the point (Fig. 2) as shown in the EDS graph of the chosen point.

**Table 1. Percentage of mass of elements found at point 1 of sample S1**

| Element | C-K | O-K | Al-K | Si-K | Ni-K | Cu-K |
|---------|-----|-----|------|------|------|------|
| S1-(2000x1) | 1.04 | 0.00 | 0.05 | 0.05 | 7.59 | 91.26 |

The software resembled the actual image to make the points count to measure the porosity (Fig. 3).
With 80% copper and 20% nickel. The software and after statistical analysis showed the mean of 52.3% surface porosity and a standard deviation of 18.1%. Sample 2 resulted in an average 52.3% porosity, high when compared to the results of research [17] that has a porosity variation of sintered pure titanium in the order of 35 to 40%. It also remained above the range of porosity of the multicomponent white cast iron spray coating that is 0.9 to 8.7% [19]. It is possible to count the points to measure the surface porosity (Fig. 4). With seventeen images of the S2 sample, with 90% Cu and 10% Ni, after statistical analysis, the software showed to an average of 19.8% surface porosity and a standard deviation of 3.6%. The sample S3, which had an average porosity of 19.8%, can be considered low since the porosity variation of pure titanium is between 35 and 40%. The range of porosity of the spray-dried multicomponent white cast iron coating that is 0.9 to 8.7% [19].

For the statistical analysis, a total of twenty manipulated images of the sample 1 were used, with 90% of Cu and 10% of Ni. In the software and statistical analysis resulted in an average of 50.2% surface porosity and a standard deviation of 22.2%. The sample S1 had an average of 50.2%, being above the porosity range of pure titanium that remained between 35 and 40%. It also remained above the porosity range of the multicomponent white cast iron spray coating that is 0.9 to 8.7% [19]. It possibly counts the points to measure the apparent porosity (Fig 4).

The software resembled the actual image to make the points count to measure the surface porosity (Fig. 6). With twenty manipulated images of the S4 sample, with 20% Cu and 80% Ni, the software pointed to an average of 28.4% surface porosity and a standard deviation of 7.6%. With a total of seventeen manipulated images of the S3 sample, with 50% copper and 50% nickel, the software pointed to an average of 19.8% surface porosity and a standard deviation of 3.6%. Comparing to the results of existing research [17] the S4 sample, which presented a mean of 28.4%, being below 35%, which is the minimum level of pure porosity variation of 35% and 40% of pure titanium. Exceeding the range of porosity of the multicomponent white cast iron coating made by spraying is 0.9 to 8.7% obtained by [19]. The software resembled the actual image to make the points count to measure the surface porosity (Fig. 7).

With a total of nineteen manipulated images of the S2 sample, with 80% copper and 20% nickel. The software and after
With a total of eighteen manipulated images of the sample S6, with 10% Cu and 90% Ni, in the software and after the statistical analysis an average of 22.7% surface porosity and a standard deviation of 4.7%. The multicomponent white cast iron spray coating has the porosity ranging from 0.9 to 8.7% [19]. Under the average porosity found in sample S4, which presented a porosity of 22.7%, but about existing studies [17] with a porosity of 35 to 40% for pure titanium. The software was used to count the points to measure the surface porosity (Fig. 8). With a total of twenty manipulated images of the sample S7, with 70% of Cu and 30% of Ni, in the software and after the statistical analysis an average of 58.2% surface porosity and a standard deviation of 10.7%.

**Fig. 8. Image made by SEM from a random sample point 7 with ImageJ filter in B&W**

The EDS technique is one of the features of the SEM and allows the compositional analysis of the materials, being possible to quantify the elements present in percentage or weight [18]. The sample S1 had an average of 50.2%, being above the porosity range of pure titanium that remained between 35 and 40%. It also remained above the porosity range of the multicomponent white cast iron spray coating that is 0.9 to 8.7% porosity. Comparing to the results of existing studies [17] the sample S7 presented an average of 58.2% porosity. This value is above the range of pure titanium porosity that remained between 35 and 40% and remained above the white cast iron coating that is 0.9 to 8.7% porosity [19]. With analysis of each sample, the graph (Fig. 9) presents the surface porosity, characterized by the percentage of the average values of each sample, with the percentage of Cu in the Cu-Ni alloy. After the determination of the surface porosity, there is a possibility, through three-dimensional techniques, to find a 3D model for the porosity [20].

**Fig. 9. Graph of the porosity averages of all samples with trend line**

**Conclusion**

The research showed out that after the sintering process:
- The confirmation of formation of the Cu-Ni alloy, since at all points, chosen at random, there was the presence of the alloy.
- After the image collection and manipulation of the same through software, it was possible to perform a statistical analysis that proved a high porosity of all the compositions of the samples analyzed.
- The high percentage of porosity found in all analyzed samples is due to the low compaction pressure used.
- There is a tendency to increase the porosity of the alloy with increasing percentage of Cu about Ni.

**REFERENCES**

Askeland, D. R.; Fulay, P. P.; and Wright, W. J. The Science and Engineering of Materials. Sixth Edition. Stamford, USA, 2006.

Atwater M. A. Reconsidering functional powder metallurgy with intraparticle porosity Metal Powder Report, 2019 DOI: 10.1016/j.mprr.2019.01.004

Averá, A. C.; Biehl, L. V.; Medeiros J. L. B.; and Souza, J. de. Comparative Study of the Diffusibility of a Nickel Al alloy composed by Ni-Cr-Fe. International Journal of Engineering Research & Technology (IJERT). v. 5 n. 12, 2016.

Braga, N. de A.; Ferreira N. G. and Cairo, C. A. A. Obtenção de titânio metálico com porosidade controlada por metalurgia do pó. Química Nova, v. 30, n. 2, 450-457, 2007.

Chawla, N.; Williams, J. J.; Deng, X., McClimon, C.; Hunter, L. and Lau S. H. Three-dimensional (3D) characterization and modeling of porosity in powder metallurgy (P/M) steels. Int J Powder Metall, v. 45, p. 19-27, 2009.

Feinleib, J.; Scouler, W. J.; and Hanus, J. Optical Studies and Band Structure of Cu–Ni Alloys. Journal of Applied Physics. v. 40, n. 3. 2003.

Germán, R. M. Coarsening in Sintering: Grain Shape Distribution, Grain Size Distribution, and Grain Growth Kinetics in Solid-Pore Systems. Critical Reviews in Solid State and Material Sciences 35(4):263-305, 2010.

Maranho, O. Aspersão térmica de ferro fundido branco multicomponente. PhD Thesis. Universidade de São Paulo, Brazil, 2006.

Neckel, I. T. Crescimento e morfologia de liga Cox Fe100x eletrodepositadas sobre Si (111) tipo – n. Master Thesis. Universidade Federal do Paraná, Curitiba, 2009.

Oliveira, L. A. de. Estudo da sinterização do aço inox 316l e liga Cox Fe100x reforçado com 3% carbeto de tântalo – tac. Master Thesis. Universidade Federal do Rio Grande do Norte, Natal, 2008.

Rodrigues, H.; Margarido, F.; and Nogueira, C. A. The Effects of Sorting on the Aluminium Recycling Process in a Rising Demand Market. Proceedings of the 2nd World Congress on Mechanical, Chemical, and Material Engineering (MCM'16) Budapest, 2016, pp 22 - 23.

Selvakumar, N. Particulate Processing (Powder Metallurgy). Reference Module in Materials Science and Materials Engineering. 2017. DOI: 10.1016/B978-0-12-803581-8.03349-X

Souza, J. de.; Oliveira-Motta, C. A.; and Schaeffer, L. Utilización de Ceniza Volante Aleada al Material Compuesto Hierro-Cobre-Grafito mediante un Proceso de
Pulvimetalurgia. Información Tecnológica. v. 25(5), 21-26, 2014.

Souza, J. de.; Oliveira-Motta, C. A.; Machado, T. G.; Giacomin, A.; and Arabi, H. M. A. Analysis of Metallic Waste from Laser Cutting for Utilization in Parts Manufactured by Conventional Powder Metallurgy. International Journal of Research in Engineering and Science (IJRES). v.4 p.01-05. n. 2016.

Torres, C. dos S. Estudo da moagem de alta energia e sinterização de metal duro Wc-Ni. Programa de Pós-Graduação em Engenharia de Minas, Metalúrgica e de Materiais. (Master thesis). Universidade Federal do Rio Grande do Sul. Porto Alegre, 2009.

Wang, K.; Xia, M.; Xiao, T.; Lei, T. and n Yan, W. Metallurgically prepared NiCu alloys as cathode materials for hydrogen evolution reaction. Materials Chemistry and Physics, v. 186, 15 2017, Pp 61-66 DOI: 10.1016/j.matchemphys.2016.10.029

Webler, B. A. A. Study of the Processes During High Temperature Oxidation that Control Surface hot Shortness in Cooper-Containing Low Carbon Steels. PHD Thesis. Carnegie Institut of Technology. (2008).

Ybarra, L. A. C. Efeitos das características dos pós-industriais de tungstênio e carboneto de tungstênio na microestrutura e dureza de metal duro para ferramentas de perfuração de rochas. Revista Eletrônica de Materiais e Processos, 2010.

Yu, L.; Jiang, Y.; He, Y.; Liu, X. and Zhang, H. Fabrication of porous nickel–copper alloy with controlled micro-sized pore structure through the Kirkendall effectMaterials Chemistry and Physics v 163, 1 2015, pp 355-361 DOI: 10.1016/j.matchemphys.2015.07.050

Zhou, K.; Chen, W. G.; Wang, J. J.; Yan, G. J. and Fu, Y. Q. W-Cu composites reinforced by copper coated graphene prepared using infiltration sintering and spark plasma sintering: A comparative study. International Journal of Refractory Metals and Hard Materials. v. 82, 2019, pp 91-99 DOI: 10.1016/j.ijrmhm.2019.03.026