STEREOLOGICAL CHARACTERISATION OF PRE~ AND SINTERING PHASES IN PIM MADE W-Cu-Ni ELECTRICAL CONTACTS

Daniela Tarata*, S. Ghita*, M. Tarata**

* Faculty of Mechanics, University of Craiova, Romania

** Medical Informatics, University of Medicine and Pharmacy, Craiova, Romania

Abstract: *The work deals with the characterization of presintering and sintering phases in powder injection molding (PIM) made W-Cu-Ni electrical contacts. Usually, such electrical contacts are made by pressing. The major drawback of this process is that its applicability is limited to simple shapes. Through powder injection molding the design restrictions are eliminated and the mechanical and electrical properties of the components thus obtained are closed to the pressing technology. The analysis method described in the paper allows collecting data to objectively describe the granulometric repartition of the components in pseudo alloys, and indirectly, their properties.*

1. INTRODUCTION

PIM is a cost effective production route for relatively small, complex and precision metal components. This process has become competitive with investment casting, cold isostatic compaction, slip casting, discrete machining and conventional powder metallurgy. Due to its advantages: material flexibility, precision surface finish, high properties, low cost production and shape complexity, the applications of the PIM process are being expanded in a variety of market areas such as automobiles, business machines, medical/dental apparatus and electrical appliances. We focussed our attention to the electrical contacts, which are usually produced by pressing (Tarata, 1994, 1999). Recently, due to the specific advantages of the PIM process, it was applied to forming W-Ni-Cu electrical contacts. The actual work briefly presents the technology and results, focussing to the characterization of presintering and sintering phases in powder injection molding (PIM) made W-Cu-Ni electrical contacts.

2. METHOD

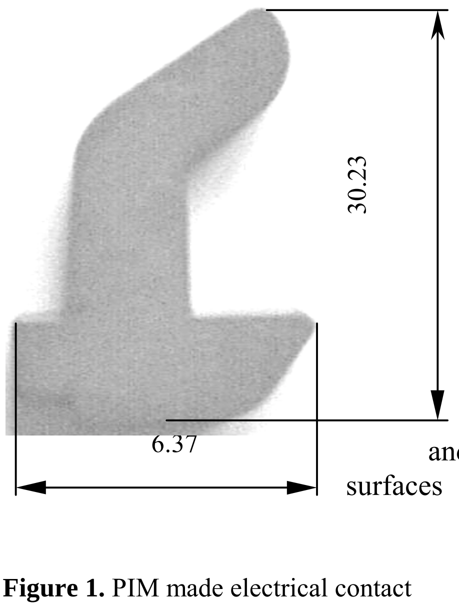


Figure 1. PIM made electrical contact

To obtain W-Cu pseudo alloys (Taubenblat,1980), the process starts with a mix of W powder and a small quantity of Ni, bee wax and alcohol. The mix is homogenized and has to have a viscosity suitable for the injection. After the injection, the composition is submitted to slow, controlled thermal treatment aiming to eliminate the wax, and thus, a porous skeleton of W results. This phase is called presintering (PRE~). Within this skeleton Cu is infiltrated eventually, by diffusion at 1250 ^{o}C in a reducing H_2 environment. This phase is called infiltration with sintering. Figure 1 shows an example of electrical contact made via the technology above described. The samples were prepared for the measurement by polishing the inspection surfaces with abrasive paper, and then with alumina solution and chemically attacked with ammonium Copper chloride. The surfaces were inspected (SEM analysis) with Philips XL30 FEG microscope (Netherlands). Examples are shown in figures 2,3,4 left.

The photograms were processed with LUCIA V 3.5 (Laboratory Imaging Ltd., Czech Republic) system for image processing and analysis.
The following morphological and stereological parameters were determined:

- average area of selected objects (A_a [μm^2]) and rate frequency of objects section (statistical distributions);
- area fraction of object section A / A_t (geometrical distribution) - A_t area of the measurement frame;
- Perimeter p ([µm]);
- the equivalent diameter *Eqd* ([µm]):

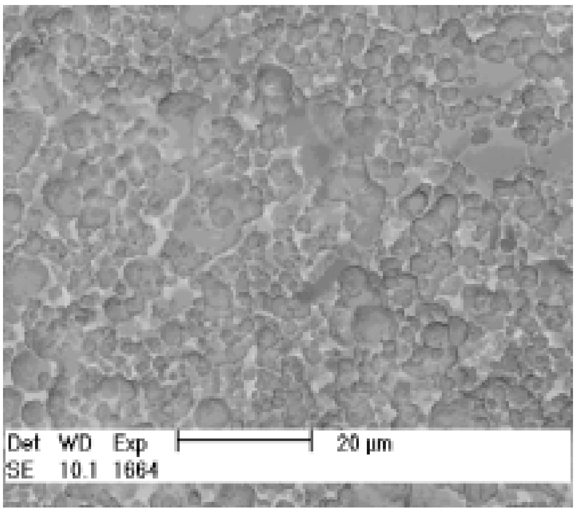
$$Eqd = \sqrt{\frac{4 * Area}{\pi}} \qquad (1)$$

- circularity: $$c = \frac{4 * \pi * A}{p^2} \qquad (2)$$

Measurements were performed both on the surface and on sections after the two important technological phases: prsintering and sintering.

3.RESULTS

To estimate the porosity in the presintering phase (Figure 2) the image was processed via the following steps: Complement Color; Erode (Serra, 1982); Sharpen (Banks, 1990); Contrast; Threshold; Smooth; Clean; Invert ;detection and measurement. After erosion, the inner layer of the objects is subtracted. If an object or narrow cape is smaller than thickness of the layer they disappear from the image. The effect is a better separation of the objects. Clean is also called geodesic opening. First it erodes the image, so small objects disappear. Then remaining eroded objects are reconstructed to their original size and shape. Advantage of this algorithm is that very small objects disappear but the rest of the image is not affected. The image processing is complicated for this phase, because of the wax content, which makes the pores difficult to detect.
For the sintering phase, two steps were performed:
a. the detection and measurement of the holes (Figure 3), with the following steps: Threshold(); Clean;
b. the detection and measurement of the WNi grains (Figure 4), with the following steps: Complement Color; Erode Color; Threshold; Fill Holes; detection and measurement. Fill Holes is a procedure to fill the holes inside the image. For example this is useful if you can detect only boundaries of the objects because objects have rich inner structure with intensities typical for background and boundaries.



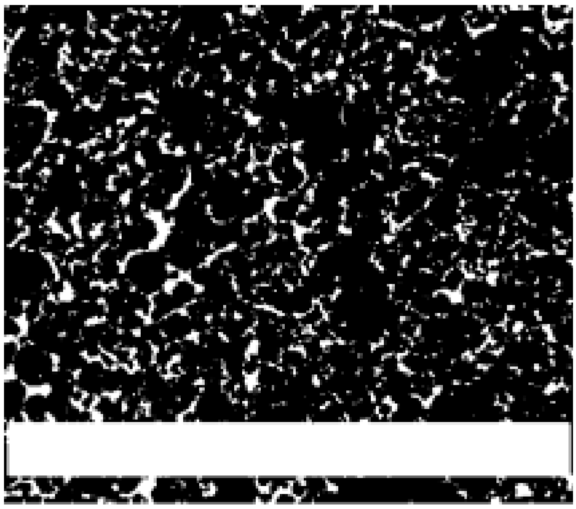


Figure 2. The presintering phase before (left), and after the image processing (right). White areas are pores.

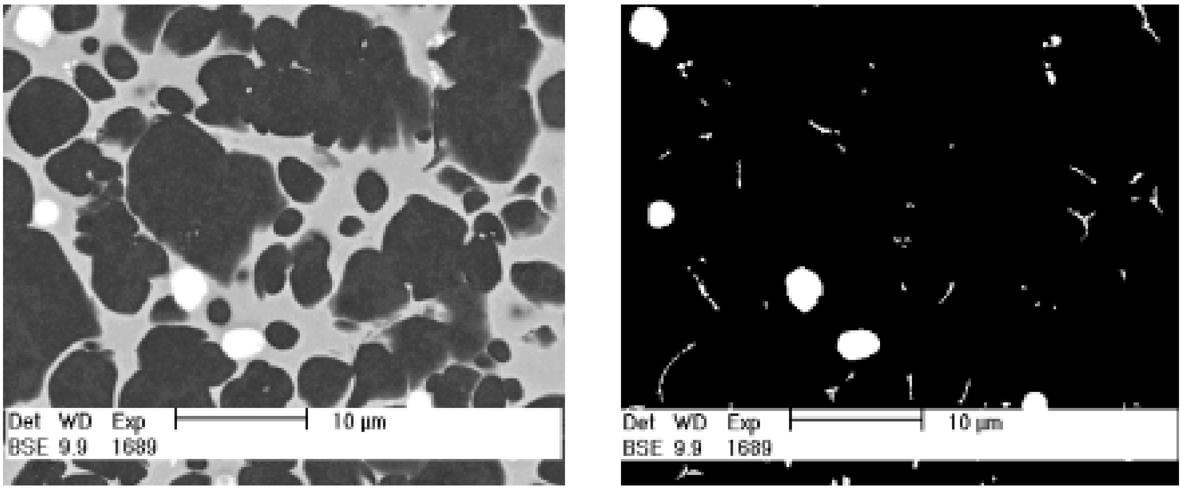


Figure 3. The sintering phase before (left), and after the image processing (right). White areas are holes.

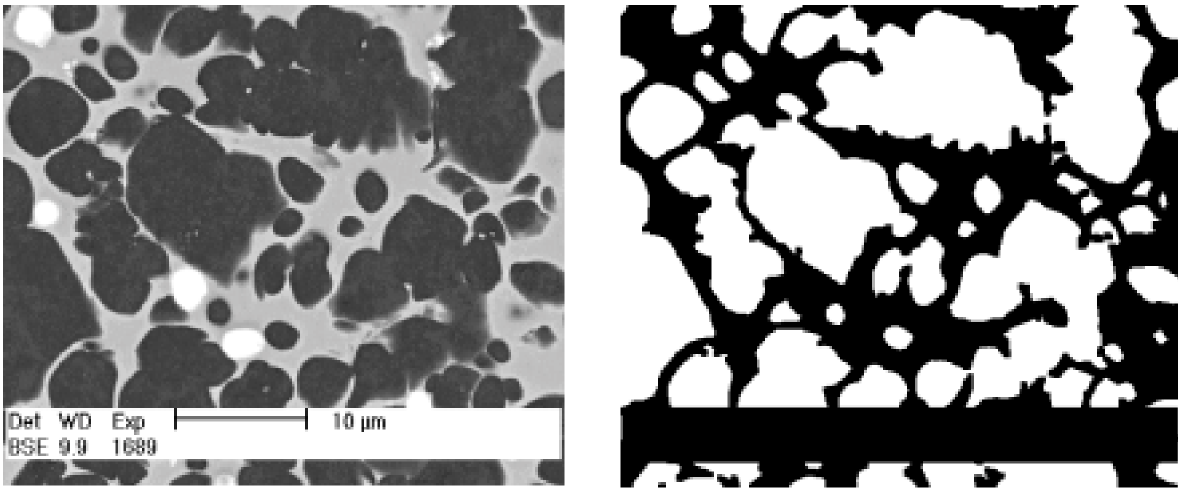


Figure 4. The sintering phase before (left), and after the image processing (right). White areas (right) are WNi grains. The holes disappeared after the processing.

Then after Fill Holes boundaries are transformed to closed areas. The grain boundary detection is a rather complicated problem (Kuc, 1999, Wojnar, 1999). In our case it is simpler, due to the larger spaces in between.

Table 1 details the parameters as measured on two frames in each phase, for the porosity, and grain evaluation.

Table 1. Typical results of the measurements in the two phases

Phase	A_m [µm^2]	A / A_t [%]	P [µm]	Eqd [µm]	c
Porosity Presintering	0.62+-1.15	13.2	2.926+-3.52	.69+-.56	.8+-.23
	0.62+-1.22	14.2	2.84+-3.5	0.67+-0.57	0.81+-0.23
Porosity Sintering	0.77+-1.3	1.2	3.1+-2.14	0.8+-0.58	0.72+-0.24
	0.64+-1.43	2.6	3.02+-2.7	0.65+-0.62	0.58+-0.27
Grains in solid WNi solution	16.28+-36.6	48	15.25+-22.83	3.12+-3.31	0.7+-0.23
	17.12+-47.7	46	14.17+-23.01	2.95+-3.61	0.71+-0.23

The measurements performed after the sintering, both on the surface and on the transversal section, show a similar homogeneity of the sample. The parameters after the sintering show normal values for the relative [%] quantity of the WNi solid solution. The equivalent diameter of the WNi solid solution grains stay between 0.1 and 20 μm, which explains their non-uniform repartition in the structure.

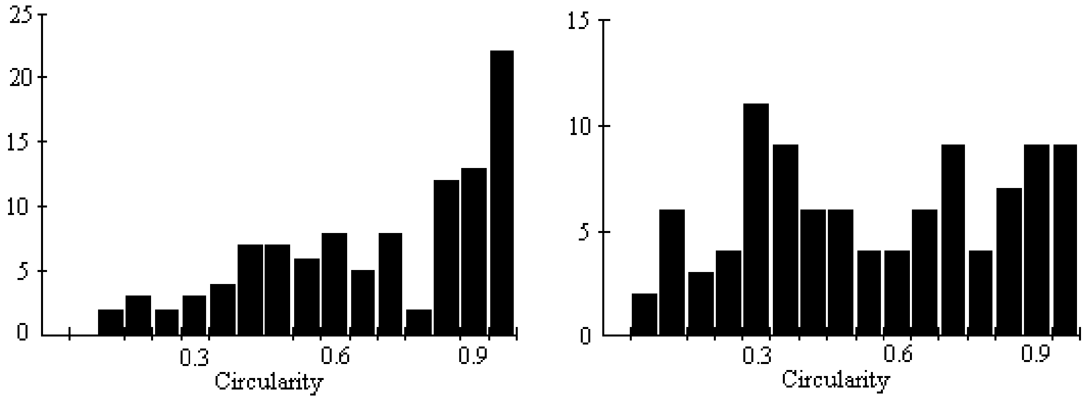


Figure 5. The histograms of the pores circularity, after the sintering.

We estimated the structure porosity (Van Vlack, 1989) via the area fraction. An important decrease can be seen after the sintering due to the Copper infiltration. Figure 5 shows the histogram of the pores circularity after the sintering, showing two groups, one of more circular pores and a second group of more irregular shape pores.

4. DISCUSSION AND CONCLUSIONS

The analysis method above detailed allows collecting data to objectively describe the granulometric repartition of the components in pseudo alloys, and indirectly, their properties.
Evident differences are seen in the presintering and sintering phases related to the repartition of the constituents and porosity.
The infiltrated Cu contents increases with the amount of wax.
The density of the pseudo alloys decreases with increasing granulation and wax amount, respectively.
The method easily estimates the porosity in the two phases. The actual material shows an insufficient homogenization after the insertion of the wax.

5.REFERENCES

Banks S. *Signal processing, Image Processing and Pattern Recognition*, Prentice Hall, London, 1990.
Kuc D, Hetmanczyk M., Cwajna J. Grain Size and Shape of Austenitic Steels after Hot Temperature Deformation, in *Proceedings of the International Conference on Stereology, Spatial Statistics and Stochastic Geometry*, Prague, 21-24 June, pp. 165-170, 1999.
Wojnar L. Grain Boundary Detection – Multiple Solutions of a Single Problem, in *Proceedings of the International Conference on Stereology, Spatial Statistics and Stochastic Geometry*, Prague, 21-24 June, pp. 279-286, 1999.
Serra J. *Image Analysis and Mathematical Morphology*, Academic Press, London, 1982.
Tarata Daniela. A study on the technological factors which affect the quality of the electrical contacts of the electrical contacts made from WCuNi alloys, in *Proceedings of the World Congress on Powder Metallurgy*, Paris, 6 –9 June, pp.2017- 2020, 1994.
Tarata Daniela. Materiale refractare sinterizate si materiale cu conductivitate electrica prestabilita, sinterizate, in Sontea S. et al. METALURGIA PULBERILOR, Ed. Universitaria, Craiova, ISBN 973-9271-80-7, 1999.
Taubenblat P. Copper Base Powder Metallurgy, Metal Powder Industries Federation, Princeton, New Jersey, 1980, pp. 111-123.
Van Vlack L.H. *Elements of materials science and engineering*, Addison-Wesley Publishing Co., Reading Mass., 1989.