International Conference on Economic Engineering and Manufacturing Systems
Braşov, 25 – 26 October 2007

STUDY ABOUT COPPER INFILTRATION AT MANUFACTURING OF HARD MATERIALS COMPOSITE

Cristina Ileana PASCU
University of Craiova, Romania

Abstract. This paper presents a study about the porosity after sintering and the final weight percent of copper after infiltration into the parts produced by Powder Metallurgy. The pieces are based on tungsten powder, 78%W - 20% Cu-2%Ni. For this research two kind of tungsten powders with different grain-size distributions have been used. After the homogenisation of mixture 98%W-2%Ni the paraffin content was varied. Thus, were obtained six different mixtures, with 1%, 2% and 3% paraffin contents. The homogeneous mixture W-Ni was pressed with 400 MPa compaction pressure. After sintering the porosity of tungsten skeleton determination was made with the method of the surface integration. After copper liquid infiltration the copper weight percent content for each final piece has been determined. Also, the final mechanical and physical properties of the composites based on tungsten powder have been specified. The microstructure characteristics and manner of copper infiltration into porous tungsten skeleton have been presented, too.

Keywords: porosity, copper infiltration, tungsten, microstructure

1. Introduction

The low thermal coefficient of tungsten and the high thermal conductivity of copper make W-Cu composites attractive for thermal management applications, like for the electric contacts [1]. Processing of W-Cu composites is usually done through copper infiltration of a porous, pre-sintered tungsten compact, or through liquid phase sintering of compacts [2]. The actual technology of pieces from WNiCu for electric contact achieves the density necessary for copper infiltration from paraffin dissolved in alcohol [3, 4, 5]. Study about the influence of paraffin contents on the properties of these materials has been made.

2. Experimental results

For the experimental research, two tungsten-powders with different grain-size distributions have been used. Thus, a kind W-powder with a big grain-size distribution of 50 µm, noted like B and a fine W-powder, noted F, with 6 µm grain-size distribution. In figures 1 and 2 the form of these W-powders is presented.

Copper-powder was obtained through electrolysis, having a grain-size distribution less than 0,060 mm. Before utilisation Cu-powder was deoxygenated through reduction into hydrogen.
Nickel-powder has been obtained from the reducing of the Nickel azotize into hydrogen.

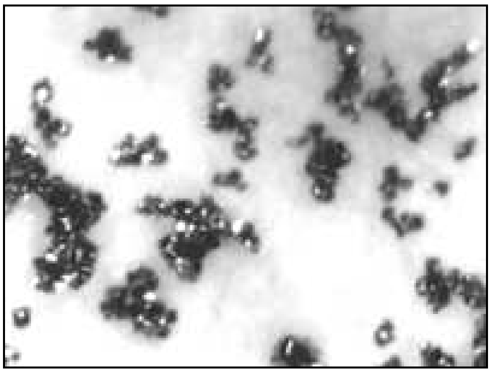
Figure 1. W-powder noted B

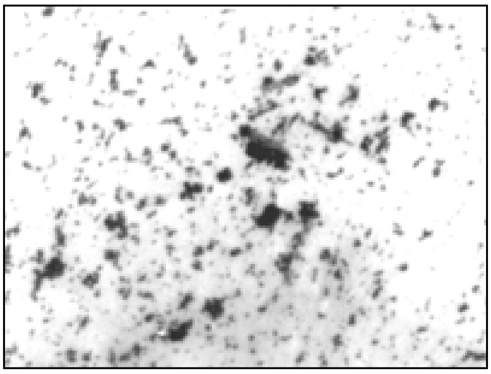
Figure 2. W-powder noted F

After the homogenisation of mixture W-Ni (98% W, 2% Ni) the paraffin content was varied. Thus, were obtained six different mixtures, with 1%, 2% and 3% paraffin contents.

The homogeneous mixture W-Ni was pressed with 400 MPa compaction pressure.

Then, it was realised by pre-sintered at the 1200 ^{0}C in hydrogen.

After that, when was obtained the tungsten porous skeleton, was measured the porosity in accordance with the method presented in [6, 7].

This method consists in porosity selection on the basis of the contrast and the integrating occupied surfaces with equations.

The porosity value on scanned images with grey and black colour nuances of the microstructures had been evaluated.

Further on, the primary statistical analyze of colours had been achieved.

In figure 3 the result of this primary statistical analyse is presented.

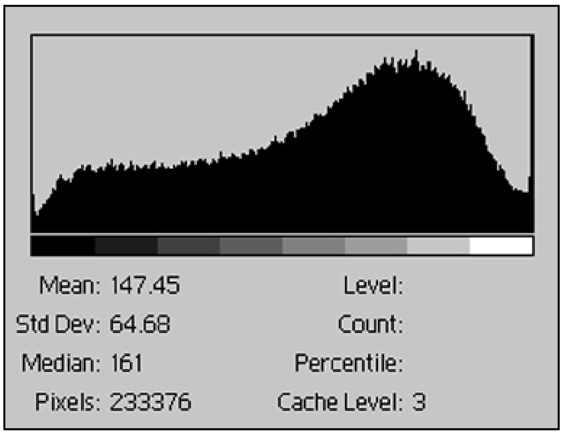

Figure 3. Result of primary statistical analyse

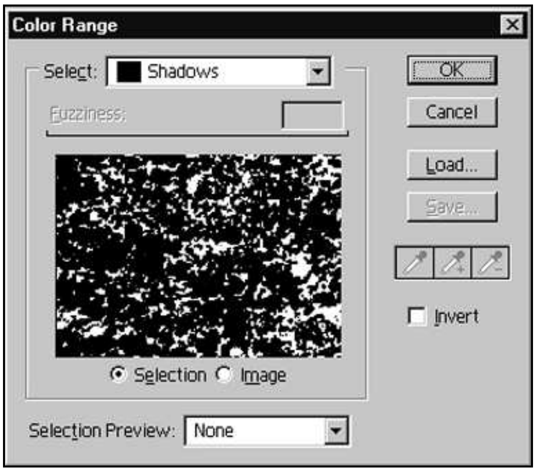

Figure 4. The dialog window

Next step in numerical determination of porosity the pores automat selection on the basis of characteristic colour constituted.

It can be mentioned that with this method of integrating surfaces on the microstructure of the pieces it is considered that the opened pores are black and the interconnected pores are grey.

In figure 4 the dialog window that allows this operation is shown.

After automatically pores selection based on their characteristic colour, figure 5, for selected zones that means the pores, it reverts to frequency analyze stage. Thus, in figure 6 the outcomes of this analyse is presented.

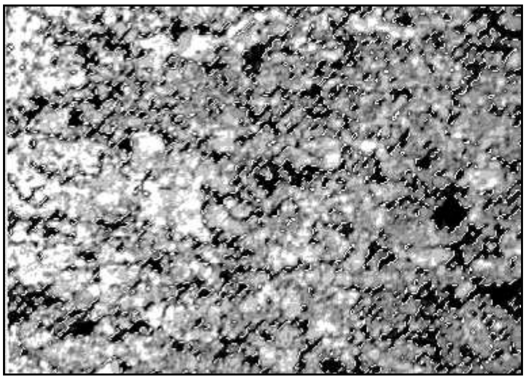

Figure 5. Automatically selection of pores

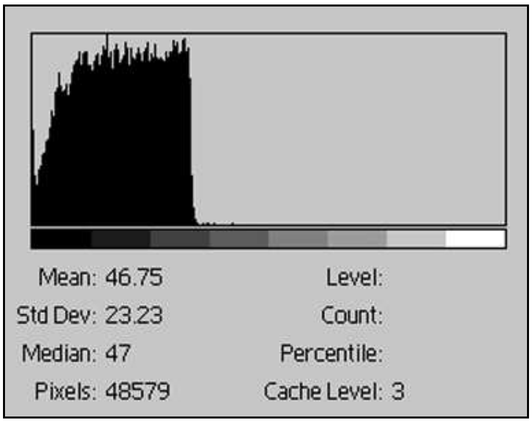

Figure 6. The result of statistical analyze for selected area

In window shown in figure 6, one of the statistical results displayed is number of selected points. By reporting on the initial point's number, presented in figure 3, the percentage relative surface of pores is obtained by primary statistical analyze. This result is a directly measure of pores volume percentage.

For analysed case:

$$\text{Porosity} = \frac{48579}{233376} \times 100 = 20{,}81\% \qquad (1)$$

The opened and interconnected porosity is achieved with relationship [3]:

$$P_d = \frac{\delta_l (G_2 - G_1)}{\delta_{par} (G_2 - G_3)} \times 100, \ [\%], \qquad (2)$$

where: $\delta_{par} = 0.778$ is paraffin density[g/cm^3].

Thus, the value of close porosity results:

$$P_i = P_t - P_d, \qquad [\%], \qquad (3)$$

where P_t is effective porosity and P_d is open and interconnected porosity.

Thus, it have been made three measurements, in different part places, and then had been calculated the measurements' mean.

The porosity values dependent on the paraffin content are presented in table 1.

Table 1. The porosity of the porous compacts

W-powder	Paraffin content %	Calculated porosity %
W-powder noted B	1	19.67
	2	24.85
	3	29.61
W-powder noted F	1	16.13
	2	21.34
	3	23.53

For each porous compact the added copper quantity was the same as that for the ordinary technological itinerary [8].

Thus, in the next figures is presented the microstructure of the porous compacts after pre-sintered.

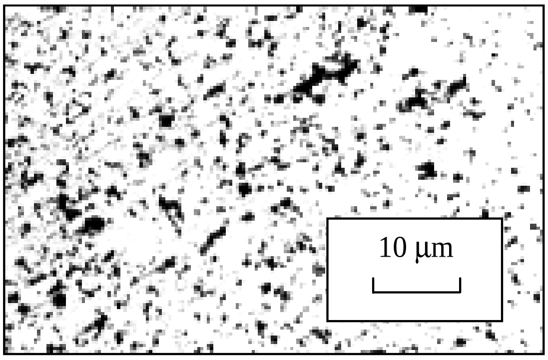

Figure 7. Microstructure of part produced by W-powder, noted B, with 1% paraffin

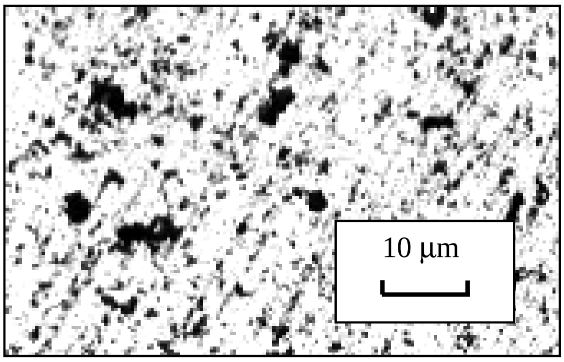

Figure 8. Microstructure of part produced by W-powder, noted B, with 2% paraffin

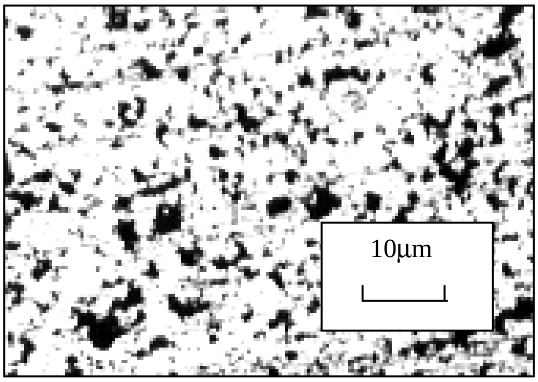

Figure 9. Microstructure of part produced by W-powder, noted B, with 3% paraffin

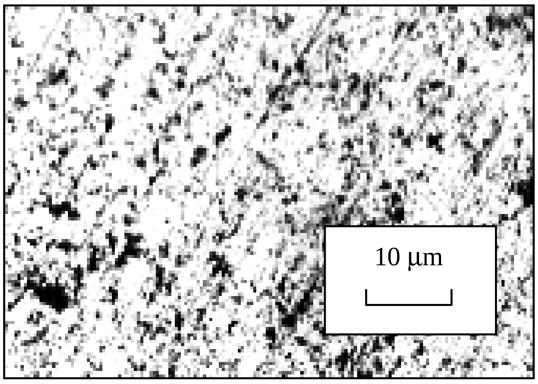

Figure 10. Microstructure of part produced by W-powder, noted F, with 1% paraffin

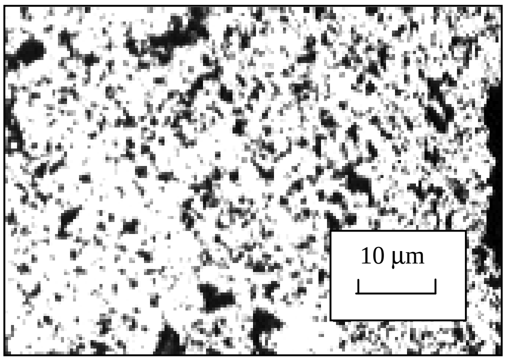

Figure 11. Microstructure of part produced by W-powder, noted F, with 2% paraffin

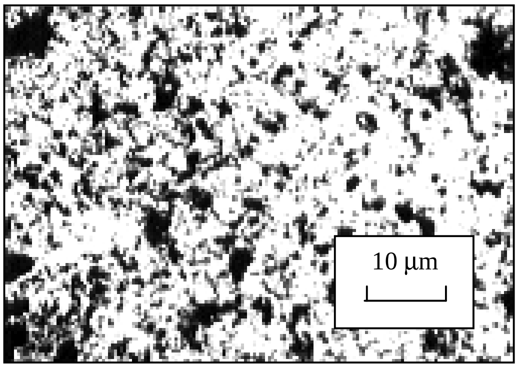

Figure 12. Microstructure of part produced by W-powder, noted F, with 3% paraffin

In the table 3 is presented the copper quantity remained after infiltration.

From values shown in table 2 it can be noticed that for porous compacts with 1% paraffin non-infiltrated residual copper had been observed, but it's a normal situation because the porosity of these parts was less than 20% and the copper added quantity before infiltration had been calculated for a porosity of 20%. Also, it can be observed that the residual copper quantity is more obvious on the parts surface based on coarse-grained W-powder, noted with B than the compacts achieved with fine W-powder, noted F.

Table 2. The characteristics of weight of sintered-compacts after copper infiltration

Kind of W-powder	Paraffin content %	Cu-powder added, g	Compact's weight after the infiltration,	The quantity of the remained
B	1%	41	204	10.6
	2%	41	197	-
	3%	39	196	-
F	1%	39	198	6.7
	2%	38	190	0.4
	3%	38	192	-

After copper liquid infiltration the values of density and hardness for final pieces obtained have been determined.

For the part obtained by coarse-grained W-powder with 2% paraffin a small quantity of residual copper after infiltration had been observed but, the part porosity was 19.8-20% after sintering therefore is normal situation.

In table 3 the values of dimensional, physical and mechanical final properties of the final pieces are presented.

From the results present in table 3 can be noticed that density and hardness after copper infiltration increase with increasing of copper infiltrated quantity into porous tungsten skeleton [9, 10].

In next figures are presented the values of final density and hardness for composites based on tungsten powder.

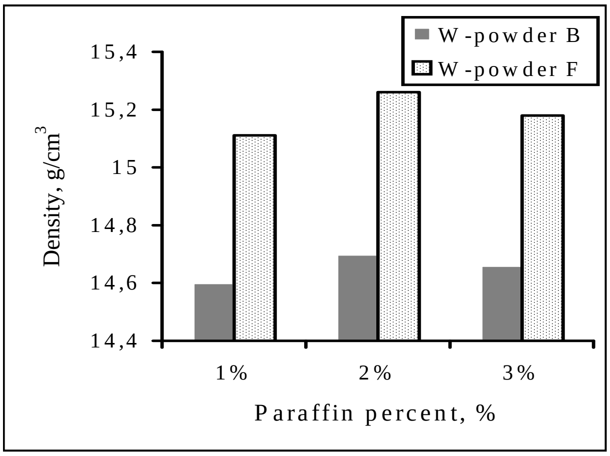

Figure 13. Final density obtained after copper infiltration

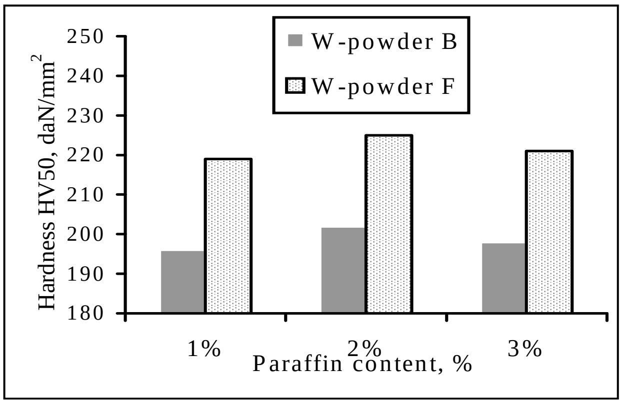

Figure 14. Final hardness obtained after copper infiltration

Table 3. Final characteristics for infiltrated pseudoallys

Kind of W-powder	Paraffin content, %	Weight g	Diameter mm	Height mm	Density g/cm^3	Hardness HV50 daN/mm^2
B	1	179,9	29,9	17,5	14,64	196
	2	184,5	29,8	18	14,696	202
	3	187,5	29,7	18,45	14,669	198
F	1	187,5	29,3	18,4	15,113	219
	2	192,5	29,3	18,7	15,267	225
	3	191,25	29,1	18,85	15,174	221

From diagrams 12 and 13 can be noticed that the values of final density and hardness obtained for 2% paraffin content for both composites achieved with coarse-graining W-powder noted B and fine tungsten powder noted F.

The smallest values of density and hardness have been obtained for pieces with 1% paraffin content because the low quantity of infiltrated copper, the porosity of parts before infiltration being smaller than the other situations.

For composites with 3% paraffin content the final density and hardness values have been intermediary.

3. Micro structural aspects

In the next figures the pieces microstructures after copper infiltration are shown.

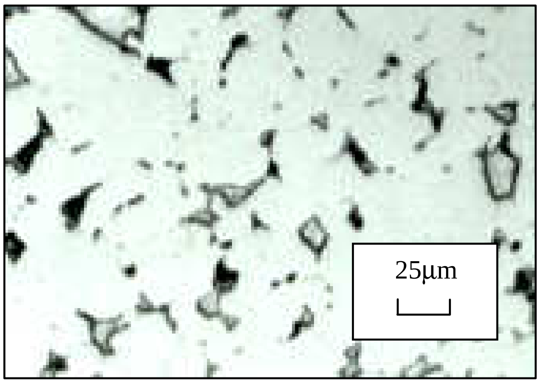

Figure 14. The microstructure of the pieces produced by W-powder, noted B, with 1% paraffin

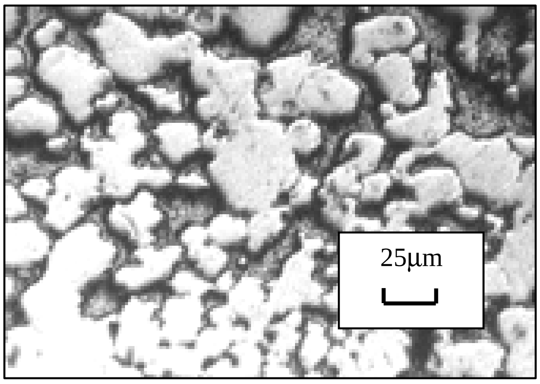

Figure 15. The microstructure of the pieces produced by W-powder, noted B, with 2% paraffin

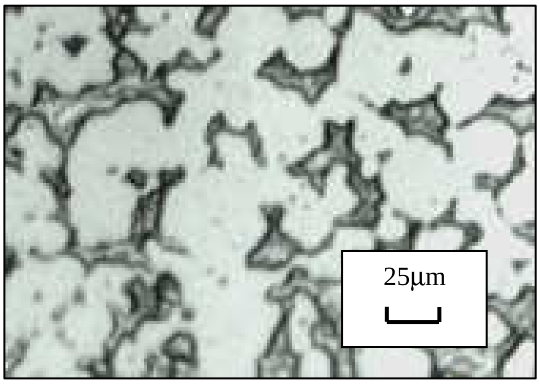

Figure 16. The microstructure of the pieces produced by W-powder, noted B, with 3% paraffin

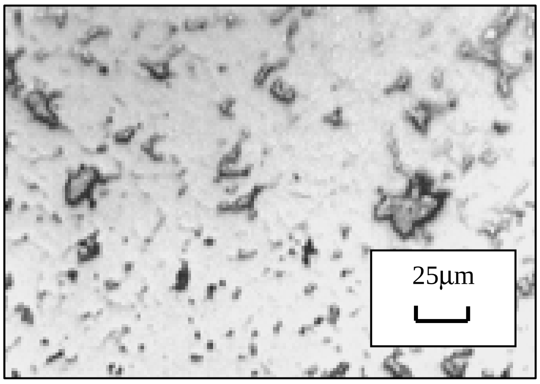

Figure 17. The microstructure of the pieces produced by W-powder, noted F, with 1% paraffin

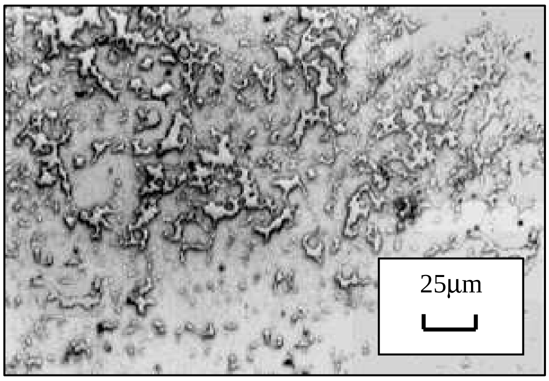

Figure 18. The microstructure of the pieces produced by W-powder, noted F, with 2% paraffin

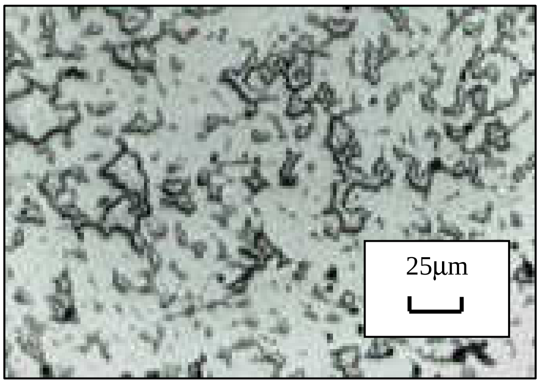

Figure 19. The microstructure of the pieces produced by W-powder, noted F, with 3% paraffin

From the microstructure point of view, for the pieces formed with 1% paraffin content was observed that the copper diffusion was superficial and exist a residual porosity, especially for composites achieved with coarse-grained W-powder, noted B, figure 14, than the same pieces achieved based on fine W-powder grain-size distribution, noted F, figure 17.

For pieces formed with 2% paraffin content the copper distribution around tungsten grains is uniform, and for composites based on coarse-grained W-powder, noted B, a lagoonar copper diffusion is remarked, figure 15.

From microstructures presented in figure 16 and 19 for pieces with 3% paraffin content, an opened porosity is remarked because of deficient quantity of copper infiltrated, especially for composites based on fine W-powder, noted F, figure 19.

4. Conclusions

The electric contacts formed of the powder with fine grain size distribution are superior than the powder with big grain-size distribution [11].

The porosity is more obvious for tungsten powder with the biggest grain-size distribution

The best behaviour has the finest tungsten powder. For this W-powder, the values of density and hardness are closer than for the W-powder with big grain size distribution.

The content of the copper diffused determines the increasing of the thermal conductibility that provides a better reliability in operation.

In point of the microscopic, the particles with the fine grain are oriented through the compaction around the powder with the big grain and the copper diffusion is made intercrystalline or in lagoon, in function of the pressing force and intergrain distances.

References

1. Sontea, S., Mangra, M., Pascu, I.: *Powder Metallurgy*. Universitaria Publishing, ISBN 973-9271-80-7, Craiova, Romania, 1999 (in Romanian)
2. German, R.M.: *Sintering Theory and Practice*. Wiley-Interscience, Inc. Publ. 605 Third Avenue, New York, NY 10158-0012, U.S.A., 1997
3. German R.M.: *Powder Metallurgy Science*. Library of Congress Cataloguing-in-Publication, New York, U.S.A., 1994
4. Lassner, E., Schubert, W.D.: *Tungsten: Properties, Chemistry, Technology of the Element, Alloys and Chemical Compounds*. Kluwer Academic/Plenum Publishers, New York, U.S.A., 1999
5. Kim, J.C., Ryu, S.S., Moon, I.H.: *Nanostructural Characteristics and Sintering Behavior of W-Cu Composite*. Journal of Advanced .Materials, vol. 31, no. 10, 1999, p. 37-44
6. Pascu, I.: *Materials for Electrical Contacts*. Universitaria Publishing, ISBN 973-8043-195-4, Craiova,Romania, 2002 (in Romanian)
7. Yoon, E.S., Lee, J.S., Oh, S.T., Kim, B.K.: *Microstructure and Sintering Behavior of W-Cu Nanocomposite Powder Produced by Thermochemical Process.* Internationl Journal Refractory Metall Hard Materials, vol. 20, 2002, p. 201-206
8. Schwenke, G.K.: *Thermodynamics of the Hydrogen-Carbon-Tungsten System, As Applied to the Manufacture of Tungsten and Tungsten Carbide.* Proceedings Fifteenth International Plansee Seminar, P. Rodhammer and H. Wildner (eds.), vol. 2, Plansee Holding, Reutte, Germany 2001, p. 647-661
9. Eliasson, A., Ekbom, L., Fredriksson, H.: *Liquid Penetration and Particle Separation during the Initial Stage of Liquid Phase Sintering.* Met. Trans. A., Manuscript no: E-TP-06-63-A, February, 2006
10. Upadhyaya, A.: *German*, R.M.: *Densification and Dilation of Sintered W-Cu Alloys.* APMI, International Journal of Powder Metallurgy, vol. 34, 1998, p. 43-55
11. Pascu, I.: *Contribution about development of manufacturing technologies for electrical contacts based on Wolfram*. PhD thesis, University of Craiova, Romania, 2000 (in Romanian)