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Researches regarding the elaboration of W-Cu nanocomposites powders

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The paper aims to present the experimental work concerning the elaboration of some W-Cu nanocomposites powders by mechanical alloying (MA) technique. The MA process was carried out in a vario planetary ball mill with different condition: shock mode (the main disk and the planets have reverse direction) friction mode (the main disk and the planets have the same direction). Three types of samples with different concentrations as fallowing: 85 W-15 Cu, 80 W-20 Cu, 75 W-25 Cu were studied. The mixtures obtained after 8 hours of MA were studied by scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDAX) and particle size distribution.

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Keywords: Mechanical alloying, Nanocomposite powders, Particle size distribution

1. Introduction

Alloys based on W-Cu are often used in electronic and electrical industry as their good mechanical properties conferred by tungsten and electrical and thermal properties conferred by copper [1].

Due to large differences between the physical and electrical properties of tungsten and copper is difficult to elaborate these alloys by conventional technologies such as casting.

W/Cu composites are generally produced by infiltration, liquid phase sintering and hot pressing [2,3]. To improve the homogeneity and uniformity of W/Cu components, conventional ball milling [4], oxide co-reduction [5], mechano-chemical methods [6], and mechanical alloying (MA) [7-13] have been examined. The latter process is particularly attractive since it possesses the advantages of the synthesis of nanostructured materials with high purity at large quantities. Additionally, the components can be fabricated at lower temperatures without the need to addition of sintering activators [14]. Finally, better properties compared to conventional processed materials are obtainable. In the mechanical alloying of the W–Cu system dissolution of a small amount of Cu into W takes place. This is evident from the shift in the tungsten peak of W–Cu as-milled system [15].

The paper presents experimental results on the effect of two type of MA process on the morphology and particle size distribution of different concentrations of W-Cu.

2. Experimental work

At the industry level, to develop electrical contacts based on W powders it was used the composition which have code 2 (W80%-20%Cu).

From this point of view it was studied W-Cu system with different concentrations such as that presented in Table 1.

Table 1. Chemical composition of the powders mixtures used for the research.

Sample code	W [%]	Cu [%]
1.	85	15
2.	80	20
3.	75	25

Tungsten respectively copper were commercial powders obtained from SC ELECTROPUTERE S.A. Craiova and their properties are presented in Table 2.

Table 2. Characteristics of initial powders.

Metal	Density at 20 °C [g/cm³]	Atomic volume [cm³/atg]	Thermal conductivity [cal/cm°CS]	Fusion point [°C]	Brinell hardness [daN/mm²]
W	19,25	9,53	0,48	3410	380
Cu	8.92	10,28	1	960	35

Tungsten powder was mechanically milled for 20 hours to obtain a finer particle size. After this process was achieved a particle size in the range of [73.2, 88.3] nm.

The MA was carried out in a vario planetary ball mill Pulverisette 4 with the following parameters:

- grinding bowls volume: 250 ml;
- grinding bowls material: stainless steel;
- grinding balls diameter: Φ=10 mm;
- grinding balls number: 50;
- grinding balls material: stainless steel;
- material/balls ratio: 1/5 (40g powder and 200 g balls);
- alloying medium: air;
- alloying times: 8 hours.

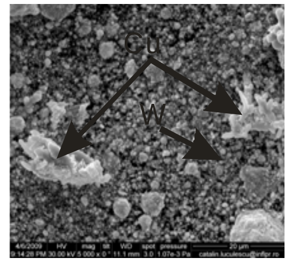
Two types of MA were used:

- shock mode, direction of rotation of the planets will be opposite to that of the main disk;
- friction mode planets and main disk have the same direction of rotation.

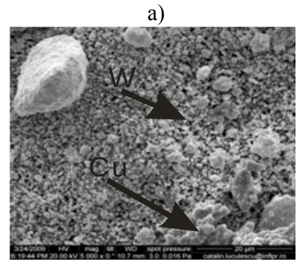
3. Results and discussion

Initial mixtures were analyzed by SEM microscopy and they are presented in Fig. 1.

From the SEM images it can be observed that the copper powder have particle bigger than tungsten powder. Also it can be observed dendrite form of granules of copper powder, specific form of electrolysis process in which the powder was developed.



a)



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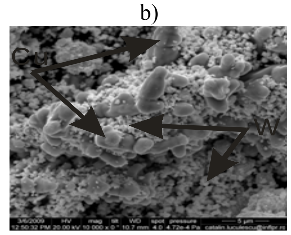
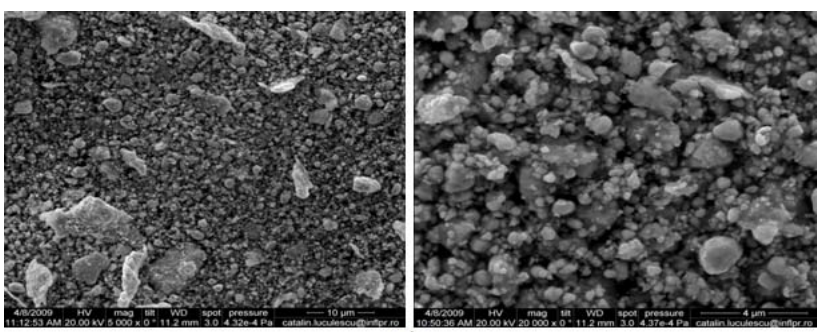


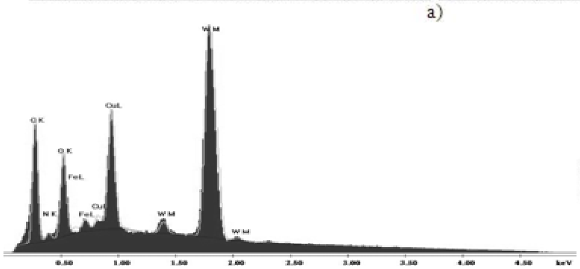
c)

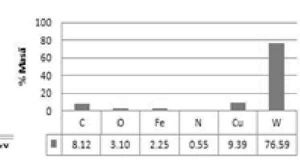
Fig. 1. SEM images of the initial mixtures: a) 85 W-1 5Cu; b) 80 W-20 Cu; c) 75 W-25 Cu.

In the below pictures are presented the SEM, EDAX and particle size distribution for each mixture.



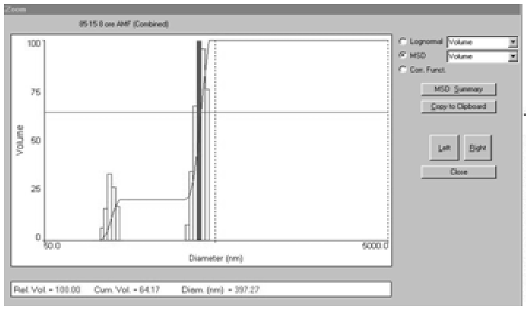
a)





b)

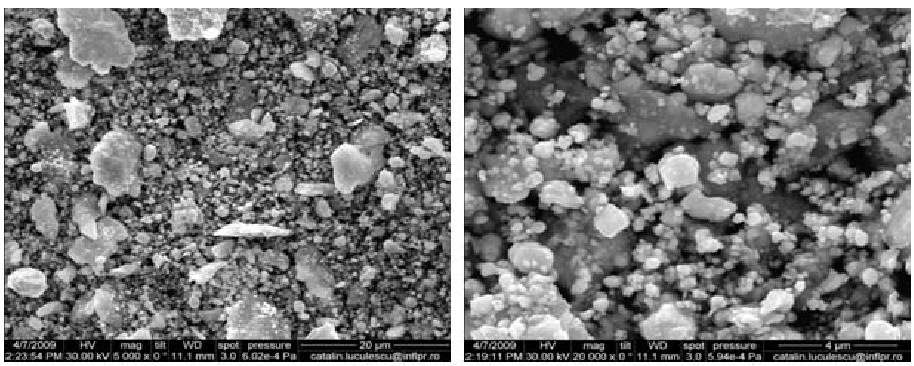
Fig. 2. a) SEM images; b) EDAX of 85W-15Cu after 8 hours of MA friction.



d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)
97.8	0	0	173.1	0	21	306.4	0	21
103.0	0	0	182.3	0	21	322.8	0	21
108.5	6	1	192.0	0	21	340.0	8	22
114.2	16	5	202.2	0	21	358.1	34	29
120.3	33	11	213.0	0	21	377.2	67	43
126.7	27	17	224.4	0	21	397.3	100	64
133.5	17	21	236.3	0	21	418.4	96	84
140.6	0	21	248.9	0	21	440.8	76	100
148.1	0	21	262.2	0	21	464.2	0	100
156.0	0	21	276.2	0	21	489.0	0	100
164.3	0	21	290.9	0	21	515.1	0	100

Fig. 3. Particle size distribution of 85W-15Cu mixture after 8 hours of MA friction.

After 8 hours of MA by friction method the 85W-15Cu alloy presents a particle size reduction comparative with initial mixture, which is in the range size [340; 440.8] nm. Also it can be observed the presence of finer particles with size range [108.5; 133.5] nm but in a smaller volume. The particle size distribution was carried out using a Brookhaven 90 Plus apparatus.



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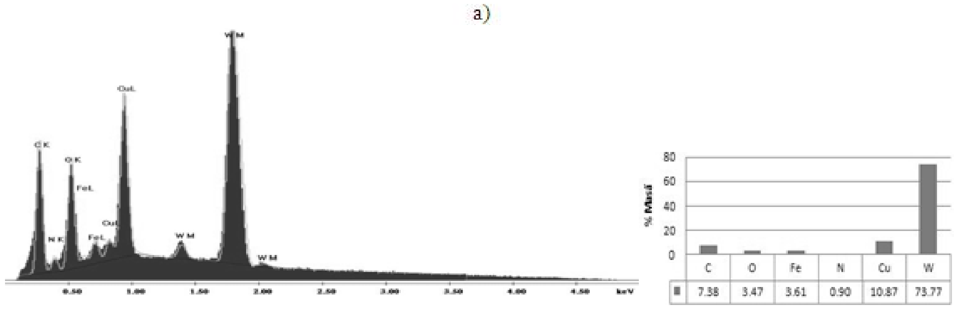
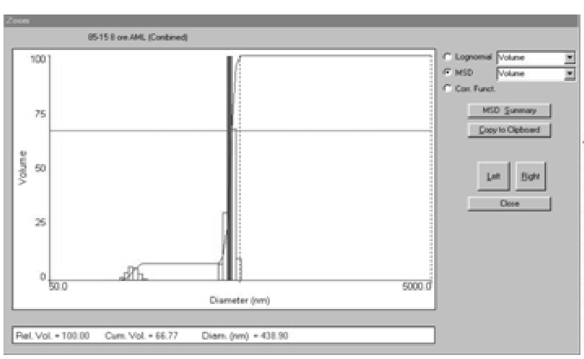


b)

Fig. 4. a) SEM images; b) EDAX of 85W-15Cu after 8 hours of MA shock.



d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)
102.1	0	0	185.0	0	8	335.0	0	8
107.8	0	0	195.2	0	8	353.6	0	8
113.8	0	0	206.1	0	8	373.3	0	8
120.1	1	0	217.5	0	8	394.0	7	11
126.8	3	2	229.6	0	8	415.8	30	24
133.8	6	4	242.3	0	8	438.9	100	67
141.2	5	6	255.8	0	8	463.3	68	96
149.0	3	8	270.0	0	8	489.0	10	100
157.3	0	8	284.9	0	8	516.1	0	100
166.0	0	8	300.7	0	8	544.7	0	100
175.3	0	8	317.4	0	8	574.9	0	100

Fig. 5. Particle size distribution of 85W-15Cu mixture after 8 hours of MA shock.

Particle size distribution of the 85W-15Cu mixture obtained by MA shock method falls in the range of [394; 489] nm, most particles being around 438.9 nm. From the analysis of the SEM images respectively of the curves and data on particle size distribution of nanocomposite powders based on W and Cu (85W-15Cu) after 8 hours of mechanical alloying found that:

- the form of particles is irregular;
- by the MA friction method are obtained particles with finest distribution comparative with those obtained by MA shock method.

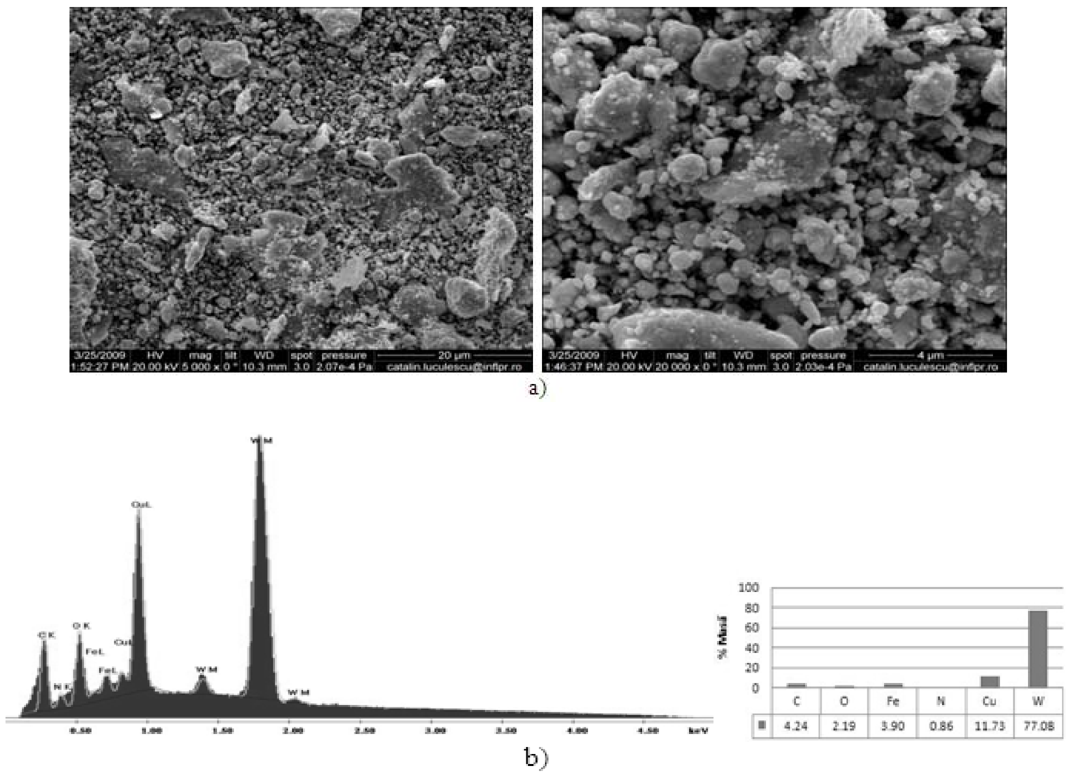
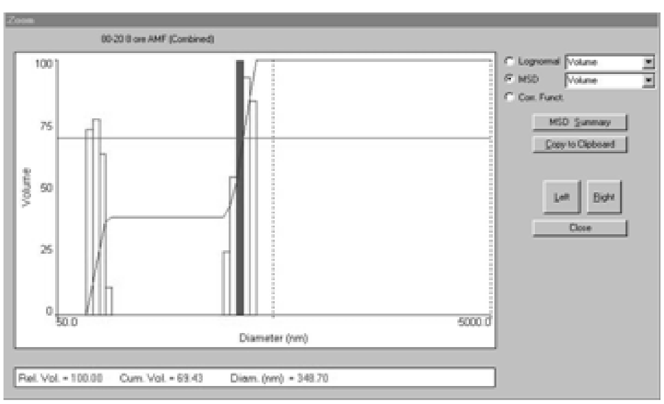


Fig. 6. a) SEM images; b) EDAX of 80W-20Cu after 8 hours of MA friction.



d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)
53.5	0	0	114.8	0	39	246.4	0	39
57.3	0	0	123.1	0	39	264.1	0	39
61.5	0	0	131.9	0	39	283.1	0	39
65.9	0	0	141.4	0	39	303.5	25	43
70.6	73	13	151.6	0	39	325.3	54	52
75.7	77	26	162.5	0	39	348.7	100	69
81.1	63	37	174.2	0	39	373.8	93	86
87.0	11	39	186.7	0	39	400.6	84	100
93.2	0	39	200.1	0	39	429.4	0	100
99.9	0	39	214.5	0	39	460.3	0	100
107.1	0	39	229.9	0	39	493.4	0	100

Fig. 7. Particle size distribution of 80W-20Cu mixture after 8 hours of MA friction.

The mixture 80 W-20 Cu obtained after 8 hours of MA by friction method has particles with size in the size range [303.5; 400.6] nm. Also there is a large volume of particles with size below 90 nm.

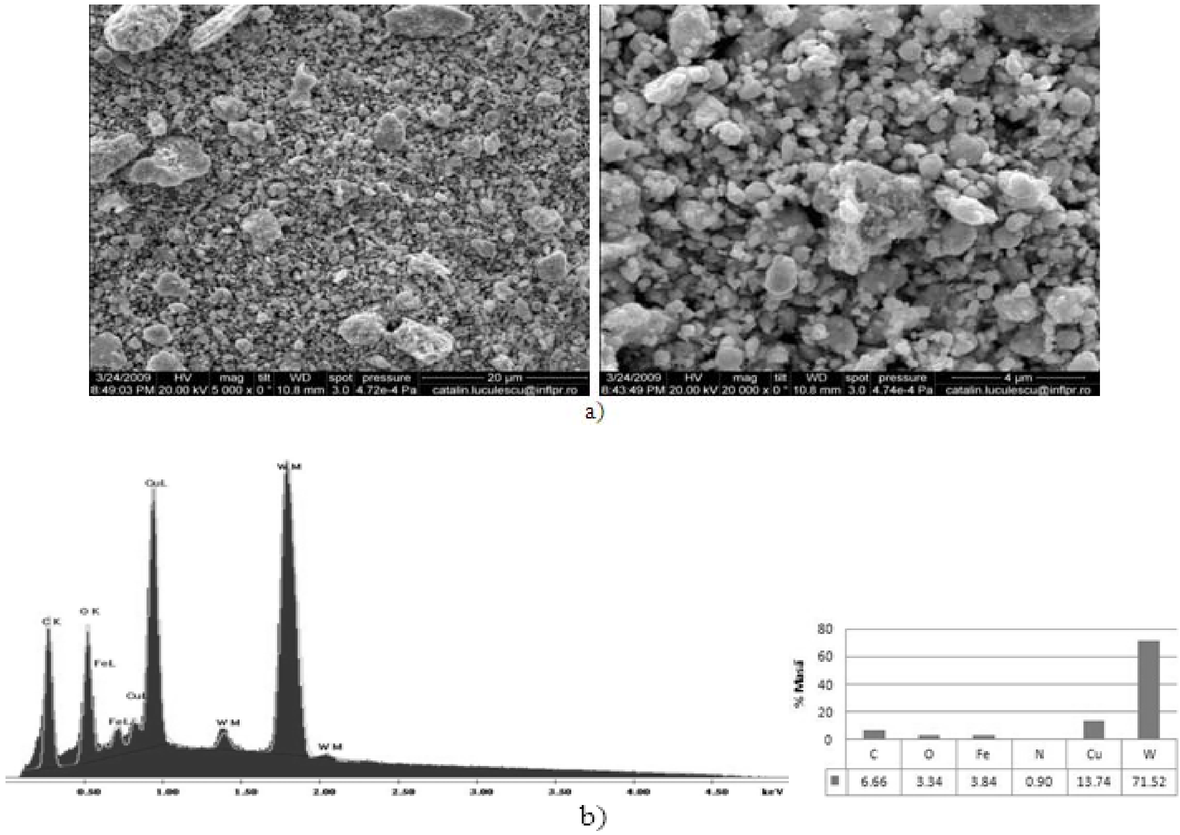
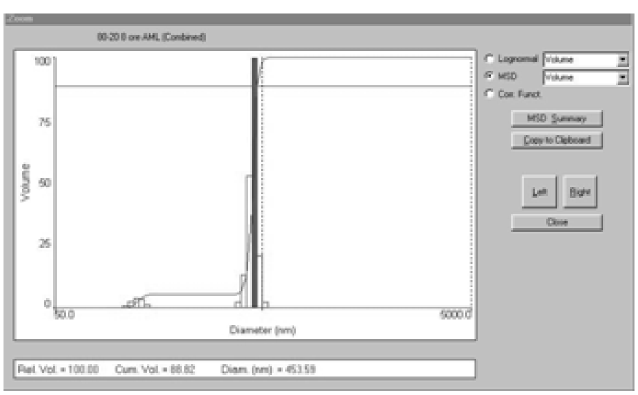


Fig. 8. a) SEM images; b) EDAX of 80W-20Cu after 8 hours of MA shock.



d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)
91.6	0	0	175.8	0	5	337.3	0	5
97.2	0	0	186.5	0	5	357.9	0	5
103.1	0	0	197.9	0	5	379.7	2	6
109.4	0	0	210.0	0	5	402.9	13	13
116.1	2	1	222.8	0	5	427.5	53	39
123.2	4	3	236.4	0	5	453.6	100	89
130.7	3	5	250.8	0	5	481.3	21	99
138.7	1	5	266.1	0	5	510.6	2	100
147.2	0	5	282.4	0	5	541.8	0	100
156.2	0	5	299.6	0	5	574.9	0	100
165.7	0	5	317.9	0	5	610.0	0	100

Fig. 9. Particle size distribution of 80W-20Cu mixture after 8 hours of MA shock.

From the data of the particle size distribution and SEM images of the 80 W-20 Cu mixture obtained after 8 hours of MA it is observed that the mixture obtained after 8 hours of MA friction presents particles finest than the other one.

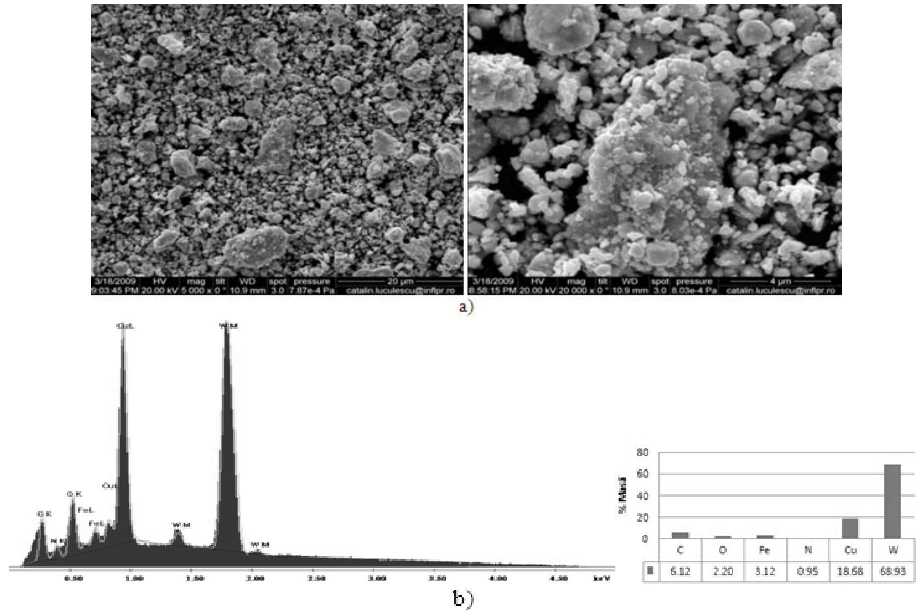
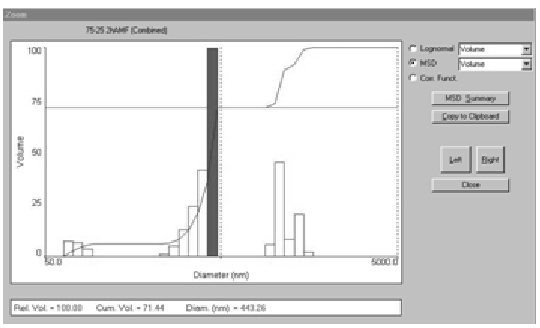


Fig. 10. a) SEM images; b) EDAX of 75W-25Cu after 8 hours of MA friction.



d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)
46.4	0	0	184.3	0	6	732.0	0	71
52.6	0	0	208.9	0	6	829.8	0	71
59.6	0	0	236.8	1	6	940.7	5	73
67.6	7	3	268.4	5	8	1066.3	45	89
76.6	6	5	304.3	12	13	1208.8	8	92
86.8	3	6	344.9	24	21	1370.3	20	99
98.4	0	6	391.0	41	36	1553.4	2	100
111.6	0	6	443.3	100	71	1760.9	0	100
126.5	0	6	502.5	0	71	1996.2	0	100
143.4	0	6	569.6	0	71	2262.9	0	100
162.5	0	6	645.7	0	71	2565.2	0	100

Fig. 11. Particle size distribution of 75W-25Cu mixture after 8 hours of MA friction.

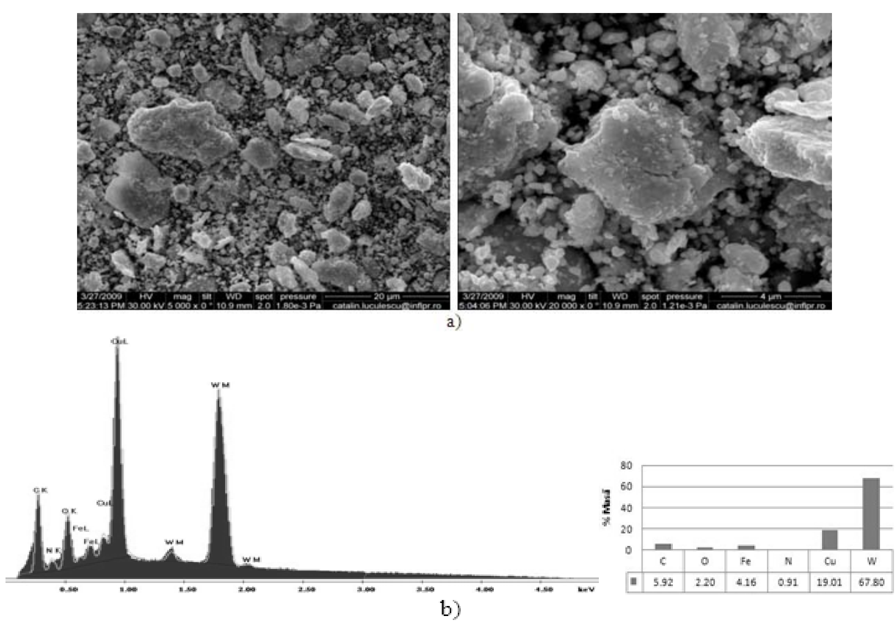
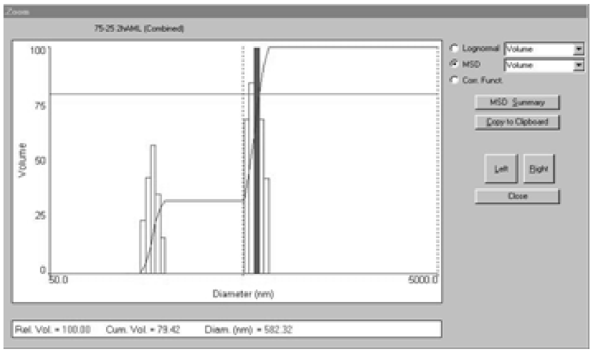


Fig. 12. a) SEM images; b) EDAX of 75W-25Cu after 8 hours of MA shock.



d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)	d(nm)	G(d)	C(d)
127.3	0	0	242.2	0	32	460.9	0	32
134.9	0	0	256.8	0	32	488.6	0	32
143.1	0	0	272.2	0	32	518.0	68	45
151.7	23	4	288.6	0	32	549.2	84	61
160.8	42	12	306.0	0	32	582.3	100	79
170.5	57	23	324.5	0	32	617.4	68	92
180.8	35	29	344.0	0	32	654.6	42	100
191.7	16	32	364.7	0	32	694.0	0	100
203.2	0	32	386.7	0	32	735.8	0	100
215.5	0	32	410.0	0	32	780.1	0	100
228.4	0	32	434.7	0	32	827.1	0	100

Fig. 13. Particle size distribution of 75W-25Cu mixture after 8 hours of MA shock.

After 8 hours of MA, the particle size of the mixture 75W-25Cu decreases to values between [268.4, 448.3] nm for those obtained by MA friction respectively [518, 654.6] nm for those obtained by MA shock.

4. Conclusions

According to the experimental results regarding the elaboration by MA process of the W-Cu nanocomposite powders can be drawn the following conclusions:

- Regarding the MA process the best results were obtained for the MA friction comparative with the MA shock. Thus, the maximum fractions of the 3 categories of powders after MA process are in the following size intervals:
  - For the mixture 85 W-15 Cu: [340; 418.4] nm for MA friction respectively [415.8; 463.3] nm for MA shock;
  - For the mixture 80W-20Cu: [303.5; 400.6] nm for MA friction respectively [402.9; 481.3] nm for MA shock;
  - For the mixture 75W-25Cu: [304.3; 443.3] nm for MA friction respectively [518; 654.6] nm for MA shock;
- The finest particles size are obtained for the mixture 80W-20Cu mechanically alloyed for 8 hours;
- From the SEM analysis it can be seen an agglomeration of particles, which indicates that particle size is very fine. All the 3 mixtures obtained after 8 hours of MA by friction and shock method present irregular grain shape;
- The grain surface of the nanocomposite powders is roughness;

In the EDAX images it is observed the presence of other elements such as C, Fe due to the

- contamination of powder with the material of grinding bowl and balls.

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