W-Cu COMPOSITE POWDER BY MECHANICAL MILLING

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ABSTRACT: The technological evolution in powder metallurgy was in connection in the last ten years with the apparition and development of new elaboration procedures of metallic powders or composite powder by mechanical alloying. This paper presents the preoccupation of author in production of homogenous mixtures of W-Cu powders system by mechanical milling and alloying. As known W-Cu system is completely insoluble in solid state.The elaboration of these alloys by conventional methods is difficult, resulting products with low mechanical properties, due to their structure unhomogenity.The influence of the tehnological parameters on structurals, physical and mechanical characteristics of the materials thus obtained are also presented.

KEYWORDS: mechanical milling and alloying, powders, characterization

1. INTRODUCTION

Mechanical alloying is a versatile method of some advanced materials producing by the solid state powder processing and has already received numerous industrial applications.

At a qualitative level, the phenomenon occurring during mechanical alloying have been understand and consists, essentially in a continuos process of deformation, fracturing, local heating, solid state welding and re-fracturing of powder particles under the effect of the transferred energy from the milling balls [1]. It could obtain submicron or nanocristallin powders, amorphous phase, intermetallics or other compounds at room temperature.

Mechanical alloying has a wide range of possibly processed materials: alloys (Al-Fe, Al-Ni, W-Cu, W-Ni-Cu, W-Ni-Fe); intermetallics (Si-Cu, Ti-C, Ti-Si, Ti-Br); magnetics (Fe-Si, Fe-Be-N, Ba-Fe-O); hard materials (W-C, V-C, Ti-C, Si-N, Fe-C) and many others [2]. Those tehnique has a very important influence on the resulting material characteristics, firstly on microstructure (very fine structure, high dispersion grade, amorphous phase, etc.) [3].

The present paper shows some experiments concerning to establish the microstructural changes during mechanical milling and alloying for W-Cu composite powders.

2 EXPERIMENTAL CONDITIONS

As raw materials is has been used pure W, Cu powders with the following weight ratio: W:Cu = 85:15.

The characteristics were determined according to International Standards: ISO 3923/1 – Apparent density; ISO 4490 – Flowing rate. The particle size was measured with a Fisher apparatus (powder permeability to water). A mixture of reduced copper and tungsten powders was grinded in a planetary mill.

***Table 1.** – Raw powders characteristics for the starting materials.*

Material	Flowing rate $^{s}/_{50g}$	Apparent density $^{g}/_{cm^3}$	Particle size FSSS (μm)
W	not flowing	3.40 ± 0.01	1.62 ± 0.01
Cu	not flowing	1.16 ± 0,01	5.81 ± 0.01

The tank of planetary is of stainless steel and its dimensions are:

- $d_{int\ tank}$ = 100 mm;
- $h_{int\ tank}$ = 300 mm;

the tank is fixed on the plane tree with diameter

- d_{pl} = 500 mm;

For milling it has been used a planetary mill with the following parameters:

- milling speed = 400 rot/min;
- milling time = 22 hours;
- ball / powder weight ratio = 3 : 1;
- filling grade = 25 %;

Copper powder used for the experiment had an average particle size of 5 μm and a morphology of irregular shape. Tungsten powder was of a spheroidal particle shape with an average size of 1 μm.

Figures 1 and 2 show the scanning electron microscopy (SEM) morphologies of copper and tungsten powder particles before mechanical milling processing.

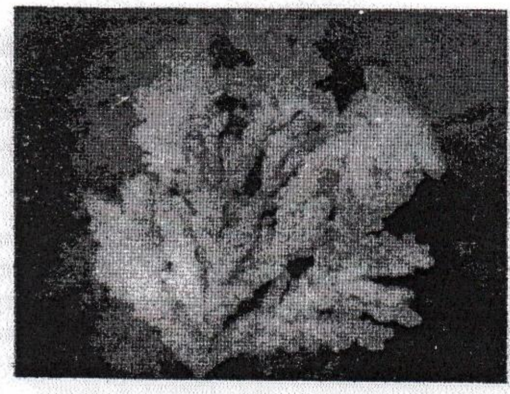


***Figure 1.** – Copper powder (SEM X 1000)*

***Figure 2.** – Tungsten powder (SEM X 3000)*

3. EXPERIMENTAL RESULTS AND DISCUSSION

The mechanically alloyed powder was taken out of the container periodically to follow the progress of alloying (from 5 to 5 hours).

The grinding total time is 22 hours. After 20 hours milling we can observe the sticking of the powder to steel balls and tank. After a longer time the powder started to agglomerate.

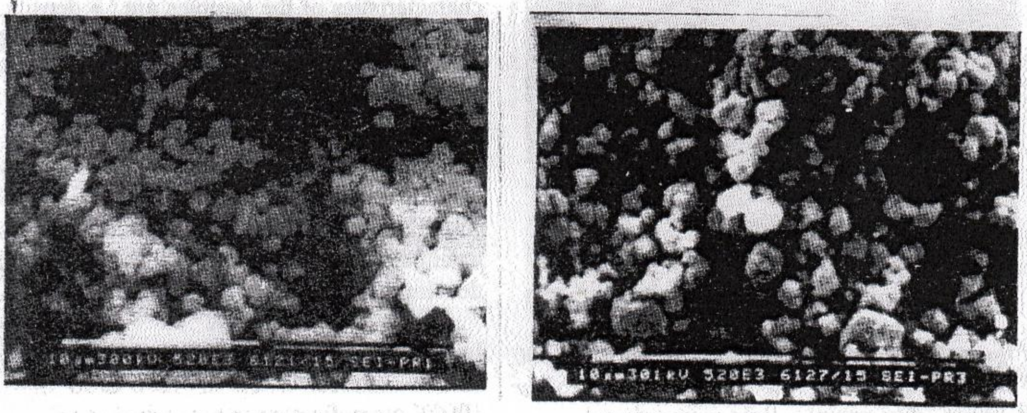
Table 2. – Obtained powders characteristics.

Material	Flowing rate $^{s}/_{50g}$	Apparent density $^{g}/_{cm^3}$	Particle size FSSS (μm)
W-Cu	not flowing	3.985 ± 0.01	1.102 ± 0.01

From table 2 could be observed that increasing milling time lead to increasing apparent density and decreasing particle size; increasing of apparent density could be explained by particle shape changes during milling, so, by increasing milling time, the sharp edge of the particles becomes rounded. It means that particles packing amount increase and, also mechanical interlocking are reduced. [4]

The speciments microstructural analysis of mechanically alloyed powder materials was performed at a Philips 515 type electron beam scanning microscope fitted out with an scattering energy analysis system (EDX).

In the figures 3 a,b are presented the electron microscope images obtained for the mechanically alloyed powders W-Cu.



a b

Figure 3. *– Mechanically alloyed W-Cu composite powder, at 22 hours milling time, X 5200*

In these figures it could be observed as follows: W and Cu particles are individualized and composites also; mostly of particles are W-Cu composites; particle shapes become irregular; an uniform repartition of the components with a very fine granulation.

There are presented both secondary electron images for emphasising the porosity, the form and the distribution mode of the componds (Figure 4) and secondary electron image with the tungsten and copper distibution mode (Figure 5 and 6).

Using the homogenized mixes a number of 5 samples of 30 x 12 x 4 mm were made by specific pressing with 400 MPa.

The sintering experiments comprised the following stages:

Sintering temperatures: •$T=1250^0C$;
Maintaining time at the sintering temperature: •t= 60 min;
Sintering atmosphere: • vacuum;
The whole treatment time : •8 h.

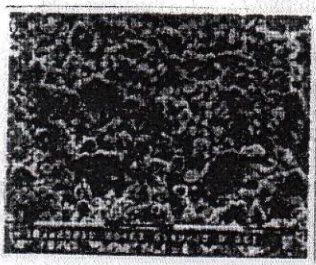


Figure 4.– *Secondary electron images of the mechanically alloyed W-Cu composite powder, X 2840*

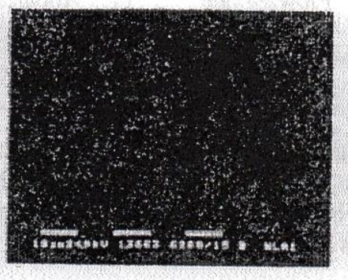


Figure 5. – *X ray image with the tungsten distribution mode, X 2840*

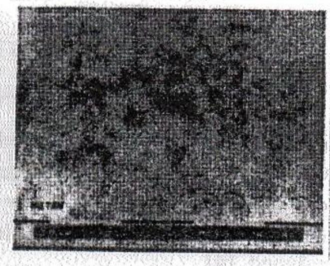


Figure 6. – *X ray image with the copper distribution mode, X 2840*

Figure 7.– *Microstructure sample W-Cu, X1000; (presence of phases: Cu- matrix – white colour; W phase – fine grains – light gray colour)*

The produced sintered material was characterised by the physical – mechanical and structural properties all along the stage. The characteristies of the samples are : • density: $\gamma = 12{,}6\ g/cm^3$; • Brinell hardness: 108 HB. The sintered samples were prepared for metallographic examination in order to determine microstructure determination (according to ISO 4499). The test sample were examined by optic microscope to a magnitude X1000, etched state.The microstructures examination were performed by the gradual development of phases by Murakami etching.

In the figure 7 are presented the microstructure of the sintered W-Cu at 1250^0C/60 min at 1000X magnification in the etched state.

4. CONCLUSIONS

The experiments lead to the following conclusions:

1. During the milling process, powder particles are trapped between the milling balls and the plastically deformed, thus rupturing the layers of surface contaminants on individual particles and exposing a clean metal surface;

2.As processing continues, the alloyed particles further break-up and reweld to give a more uniform structure of stabilizing particle size.

3. By the mechanical milling tehnique was obtained powder composed of W-Cu with a very fine granulation, a uniform repartition ;

4. The more homogeneous of the materials provided by the mechanical alloying process appears to have had a significant effect on the densification of the particular powders, perhaps by allowing both of the liquid-phase-driven sintering mechanisms to proced more effectively.

5. As a result of the process the tungsten particle size within the mechanicaly alloyed composite particle is reduced, and the tungsten itself is cold welded to what becomes the liquid phases. Any oxide film originally on the powders is broken. On sintering this will give a larger wetted area of finely divided tungsten matrix.

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