SOME ASPECTS CONCERNING MANUFACTURING OF W-Ni COMPOSITE POWDER BY MECHANICAL ALLOYING

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Lucrarea prezintă rezultatele cercetărilor experimentale în realizarea unor amestecuri omogene de pulberi din sistemul W-Ni obținute prin aliere mecanică. Elaborarea acestor aliaje prin metode conventionale, este dificila, deoarece rezulta produse cu proprietati mecanice scazute si structura neomogena. Pulberile elementare $W_{93}Ni_7$ au fost aliate mecanic într-o moara planetara, fără atmosferă protectoare sau lubrifiant. Timpul total de măcinare a fost 110 ore. Pe parcursul celor 110 ore de măcinare au fost prelevate probe, din 20 în 20 de ore, pentru evaluare calitativa si cantitativa a pulberilor aliate mecanic prin difractie de raze X. Se prezintă de asemenea influența parametrilor tehnologici asupra caracteristicilor fizico – mecanice şi structurale ale materialelor experimentate.

This paper presents the preoccupation of author in production of homogenous mixtures of W-Ni powders system by mechanical alloying.The elaboration of these alloys by conventional methods is difficult, resulting products with low mechanical properties, due to their structure unhomogenity. The milling of W and Ni powders respectively to less than 4 μm particle size, for mechanical alloying, was carried out in a vertical axis planetary mill, with no protective atmosphere or lubricant. The milling time was 110 hours. Powder samples were taken every 20 hours during the 110 hour milling time for qualitative and quantitative evaluation by X – ray diffraction. The influence of the technological parameters on structurals, physical and mechanical characteristics of the materials thus obtained are also presented.

Keywords: mechanical alloying; composite powders; mechanical characteristics.

Introduction

Mechanical alloying is a versatile method for manufacturing of some advanced materials that implies solid state powder processing. This method has already received numerous industrial applications.

At qualitative level, the phenomenon occurring during mechanical alloying have been understood and consists, essentially, in a continuos process of deformation, fracturing, local heating, solid state welding and re-fracturing of

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powder particles under the effect of the transferred energy from the milling balls. [1]

By this route one can obtain submicron or nanocristalline powders, amorphous phases, intermetallics or other compounds at room temperature.

This paper discusses some experiments regarding microstructural changes during mechanical alloying of W-Ni composite powders.

1. Experimental conditions

Raw materials were pure W, Ni powders with the following weight ratio: W:Ni = 93:7. A mixture of nickel and tungsten powders was ground 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
Ni	not flowing	2.46 ± 0,01	4.62 ± 0.01

Milling was realised in a planetary mill with the following parameters: • milling speed = 700 rot/min; • milling time = 110 hours; • ball / powder weight ratio = 43: 1; • filling grade = 25 %.

Nickel powder used for the experiment had an average particle size of 4μm and was round shaped. Tungsten powder was spheroidal shaped with an average size of 1 μm.

Figs. 1(a,b) and 2(a,b) show the scanning electron microscopy (SEM) morphologies of nickel and tungsten powder particles before mechanical alloying processing.

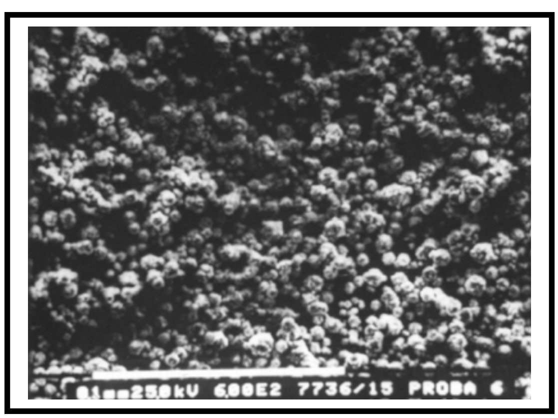

Fig. 1(a) – Morphologic aspect of Ni powder 2840 X

Fig.1(b) – View of Ni powder 6000X

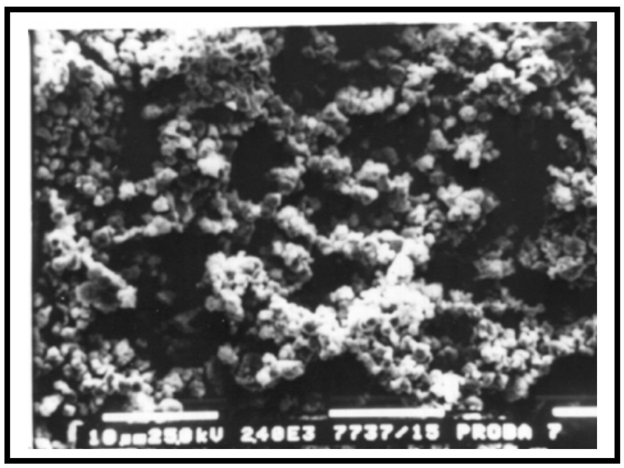

Fig.2(a) – Morphologic aspect of W powder 2480X

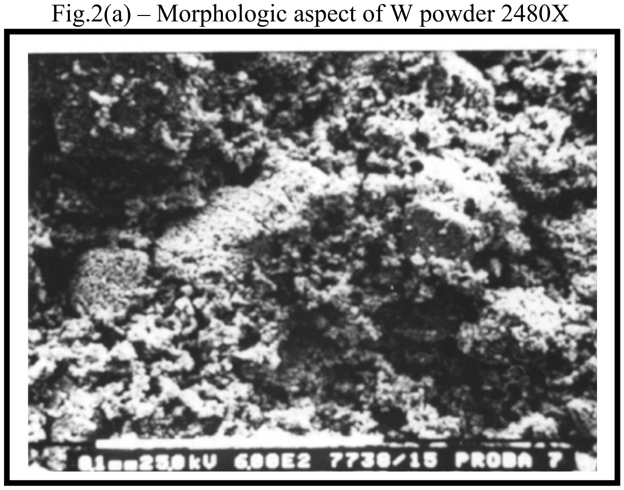

Fig.2(b) – View of W powder 6000X

2. Experimental results and discussions

The mechanically alloyed powder was sampled periodically from the container in order to study alloying progress (with a 20 hours cycle). The grinding total time was 110 hours. After 30 hours milling it could be noticed the sticking of the powder to steel balls and tank walls. After a longer processing 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-Ni	not flowing	4.08 ± 0.01	0.10 ± 0.01

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

Microstructural analysis of mechanically alloyed powder materials was performed with a Philips 515 type electron scanning microscope, fitted out with an scattering energy analysis system (EDX). Fig. 3 emphasises the aspect of W-Ni composite powders and Fig. 4 presents secondary electron images of the mechanically alloyed W-Ni composite powders.

Fig. 3 – Mechanically alloyed W-Ni composite powders after 110 hours milling time 4780X

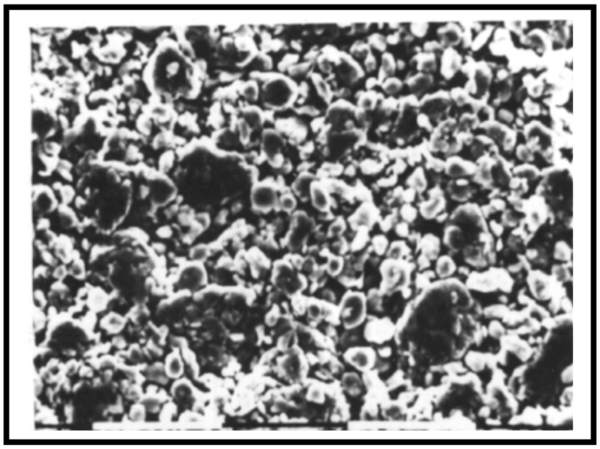

Fig. 4 – Secondary electron images of the mechanically alloyed W-Ni composite powders 2480X

In these figures it could be noticed the following: W and Ni particles are clearly defined and, also, composite particles; many particles are W-Ni composites particles, and particle shape become irregular; also, there is an uniform repartitions of the components with a very fine granulation.

Then powder mixtures were binded with paraffin (2%), sieved in a semidry state with a sieve of 0,1 mm mesh size and dried, obtainig finally a mixture “ready to be pressed”.

The pressing operation was perfomed bilaterally on a circular cross section steel die and had the following characteristics: ϕ_{ext}=47 mm, ϕ_{int}=14,3 mm; S=($\phi_{ext}{}^2$-$\phi_{int}{}^2$) =15, 73 cm^2, at a specific pressure, and were debinded in the Siemens Plania furnance, in hydrogene atmosphere, using as packing agent – alumina (calcined previousty at 1450 ^{0}C, for5 h, in order to provide heat resistance endurance).

The parameters of debinding presintering process were: • H_2 atmosphere; • heating rate up to 500^{0}C: 0,8 ^{0}C/min;• heating rate between 500-850^{0}C:3,5^{0}C/min; • presintering temperature: 850^{0}C; • exposure time to presintering temperature: 30 min; • total duration of the debinding – presintering cycle: 8h 30 min.

The sintering operation was realized in an vacuum induction furnace, with intermediate frequency currents, of Balzers type. The sintering operation parameters were: • sintering temperature: 1150^{0}C; • exposure time to sintering temperature 60 minute; • total duration of the cycle: 8 hours.

The sintered manufactured material was characterised regarding physical – mechanical and structural properties variations along all stages. In table no 3 are emphasized the physical and mechanical characteristics of the sintered samples from mechanically alloyed powder mixtures.

Sintered samples were prepared for metallographic examination in order to:

- determine the presence,occurence and repartition of pores;
- establish the microstructure.

Table 3

Physical and mechanical characteristics of sintered markers

Material type	**Density (g/cm^3)**	**Brinell Hardness HB**	**Electric resistivity (Ω m 10^{-8})**	**Stretch resistance (N/mm^2)**
Sintered markers 1150^0C	15.48	115	5.2	758

The samples for analyse were realised by stepped etching of the phases with Murakami etch solution. In Figs. 5-6 it is shown the microstructure of W-Ni sintered material at 1150^0 C/1h, as etched, at 1000X and 2000X respectively.

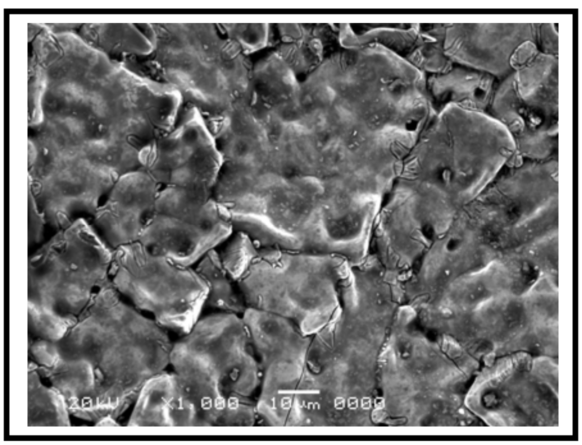

Fig.5 – SEM micrograph of W–Ni sintered material

Fig.6 – SEM micrograph of W-Ni sintered material

It could be noticed the homogenous and uniform structure of the W-Ni sintered material as well as the lack of porosity as result of the particular density of material, close to of the theoretical density.

Conclusions

Experiments led to the following conclusions:

1. The above mentioned aspects proved the possibility to manufacture W-Ni submicronic powders by mechanical alloying.The process occurs in solid state, by intensive diffusional phenomena, in which the lattice disordering and cold hardening produced by mechanical alloying play a decisive role.
2. During the milling process, powder particles are trapped between milling balls and are plastically deformed, than the layers of surface contaminants on individual particles are fractured and a clean metal surface results.
3. As processing go on, the alloyed particles break-up and reweld to lead to a more uniform structure with stabilized particle size.
4. W-Ni powder with a very fine granulation and a uniform repartition have been obtained by mechanical alloying.
5. These more homogeneous materials appear to have a significant effect on the densification during compacting and sintering, perhaps as result of the sintering mechanisms that proceed more efficiently.

6. Mechanical characteristics have greater values for 90% W alloy, confirming the beneficial influence of a higher content of W-Ni solid solution matrix, that implies a more reduced contiguity of the material. These values are generally situated abovethe values required by some of military uses[3], namely: hardness ≥ 100 HV; tensile strength ≥ 700 N/mm^2; toughness ≥ 6,5 J/mm^2.

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