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STUDY ON THE ACTIVATED SINTERING OF MECHANICALLY PROCESSED TUNGSTEN POWDERS

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Abstract: *There are some tungsten powders that exhibit a poor sinterability. A part of them are produced by chemical recovery of tungsten from metallic waste. The goal of the present work is to improve sinterability of such tungsten powders, applying them a short mechanical process in a planetary ball mill. Experiments to determine the effects of the milling duration on the change of the powder properties (density, specific surface, average particle size, particle shape, micro hardness) have been made. Changes in the sinterability of mechanical processed tungsten powders were followed on cylindrical samples pressed in a die with different compaction pressures (50-125 MPa), and successively sintered in the same conditions (T=1200 ^{0}C for 1 h, hydrogen atmosphere). Apparent density, compressive strength and the ratio of compressive strengths to apparent density after sintering were selected as estimating parameters for sinterability depending on the milling time of tungsten powders.*
Key words: *Tungsten heavy alloy, liquid phase sintering, activated sintering*

1. INTRODUCTION

There are a lot of cases in which W-porous bodies with controlled porosity. As examples on this way they could be given the W-sintered skeletons for copper infiltration to obtain high quality composite materials from the (W-Ni)-Cu system. To satisfy the quality material conditions these W-sintered skeletons should fulfill certain basic requirements as follows:

- to have a porosity corresponding to the copper volume percentage of the material composition;
- the porosity existing throughout the skeleton should be uniform distributed and interconnected one;
- to be as strong as possible to avoid their damaging by handling and infiltration.

In determining of the skeleton properties, the characteristics of the starting W-powders play an important role. So, when it is utilized a fine W-powder, the sintering temperature can be lowered and the strength of the sintered body increases because of a good powder sinterability. But, such a powder is often expensive and can give rise to closed porosity, that is disadvantageous for infiltration. In turn, if it used a coarser W-powder, it could be obtained only a weak porous body imposing the temperature rising for sintering. Instead, a coarser powder is cheaper and can assure more easily an interconnected porosity. It results from the above considerations that it must be done a compromise between the cost and sinterability of W-powder.

In our present work we have trying to enhance the sinterability of a coarser and cheaper W-powder, obtained by chemically recovery of W from the metallic waste at S.C. Sinterom S.A. Cluj-Napoca, Romania, by applying a short mechanical treatment (1-4 minutes) in a ball planetary mill. Experiments to activate sintering by mechanically treatment of powders were made before by Schatt et al. [1], [2], [3], but their experiments were made with longer processing durations (30 minutes). No studies about the influence of milling duration of W-powders on the properties of sintered porous body could be found.

Therefore we have conducted experiments to determine the effects of mechanically processing duration of mentioned W-powder both on the changes of powder characteristics (density, particle size, specific surface, micro

hardness, particle shape) and on some properties of pressed and sintered porous W-bodies.

2. EXPERIMENTAL RESEARCH

The W-powder utilized in experiments produced at S.C. Sinterom S.A Cluj-Napoca, Romania, had the following characteristics:

- Apparent density 4 g/cm^3
- Particle size under 45 μm
- Average particle size d_{50} 27,3 μm
- Specific surface BET 560 cm^2/g
- Fe_2O_3 content 0,05 [w/o]

The W-powder was mechanically treated in a planetary ball mill for 1, 2 respectively 4 minutes. There were constituted four powder batches, denoted with W0-original powder, and with W1, W2 and W4 – mechanically treated powders for 1, 2 respectively 4 minutes. For each batch of powder there were determined the apparent density (ρ_a), average particle size (d_{50}), specific surface (BET), micro hardness (HV with 0,1 N load), and powders were also observed and taken them up photographs by SEM.

From each powder batch was prepared a mixture with 0,5 [w/o] Ni (INCO 134) and 1,5 wt% paraffin, and then were pressed cylindrical specimens with a cross area of about 1 cm^2 and height of about 10 mm, applying them four different compression pressures: 50, 75, 100 and 125 MPa. After a debinding treatment, the specimens were sintered at 120 ^{0}C for 1 h in dry hydrogen (dew point ~-50 ^{0}C), in a tunnel sintering furnace.

Apparent density, relative density, and porosity both of pressed and sintered specimens were determined. In addition, on the sintered specimens was measured the compressive strength (σ_{comp}) on an universal testing machine type ZD10. On the basis of compressive strengths and apparent density, evaluation coefficients (ρ_{comp}/ρ_a) [4], were calculated.

3. RESULTS AND DISCUSSION

Table 1 is summarizing the determined characteristics of the four batches of W-started powders, and in figure 1, a-d, is showing the shape of powder particles observed and photographed at SEM.

Table 1

Characteristics of originally and mechanically treated W-powders.

Powder	Mechanical process duration t_m, [min]	Apparent density ρ_a, [g/cm^3]	Average particle size d_{50} [μm]	Specific surface BET [cm^2/g]	Micro hardness [daN/mm^2]
W0	0	4.00	27.3	560	838.0
W1	1	7.72	13.8	680	913.2
W2	2	7.90	12.0	750	933.0
W4	4	7.94	11.0	860	975.3

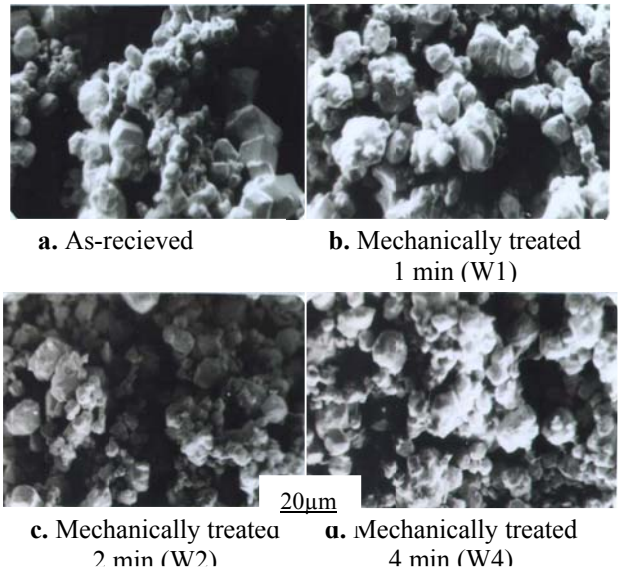
The influence of compaction pressure on the relative density of the compacts made of original W powder and mechanically treated ones is illustrated in figure 2 and 3, while in figure 4 and 5 is represented the relative density of the same compacts vs. milling duration of the started W-powders

Analyzing the data from table 1 in corroboration with evolution of powder morphology presented in figures 1a to 1d, permits us to ascertain that already after 1 minute of mechanical treatment the powder aggregates present in as-received W-powder, were near completely broken up. This determined a sharp change of all characteristics involved in the table 1.

Further mechanical treatment (2 and 4 minutes) had a much smaller effect on the characteristics in discussion, with the exception of specific surface, which increased continuously. That is perhaps because of shape modifying of powder particles by flattening and fragmentation, maintaining in the same time approximatively the same particle size distribution.

The compaction behaviour of the four powder mixtures prepared from untreated and mechanically treated W-powders are reflected in figures 2 and 4. From the two figures it can be seen that both the compaction pressure and the milling duration determine an increasing of density, but more important is the effect of milling duration.

Both compaction pressure and mechanical treatment produce in W-particles a strain hardening, but in different ways.



a. As-recieved **b.** Mechanically treated 1 min (W1)

c. Mechanically treated 2 min (W2) **d.** Mechanically treated 4 min (W4)

Fig.1. Morphology of W-powders

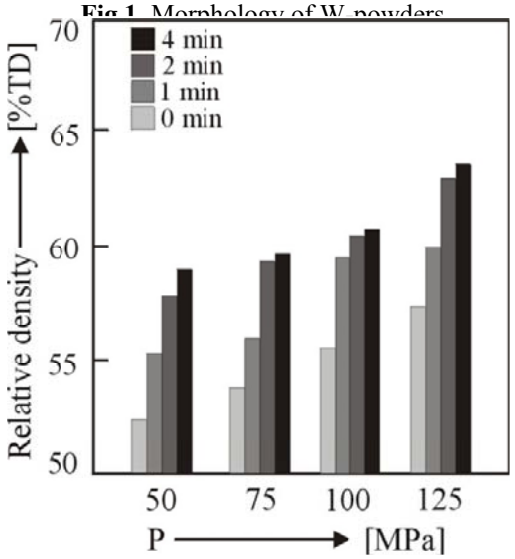


Fig.2. Relative green density vs. compaction pressure of W-specimens.

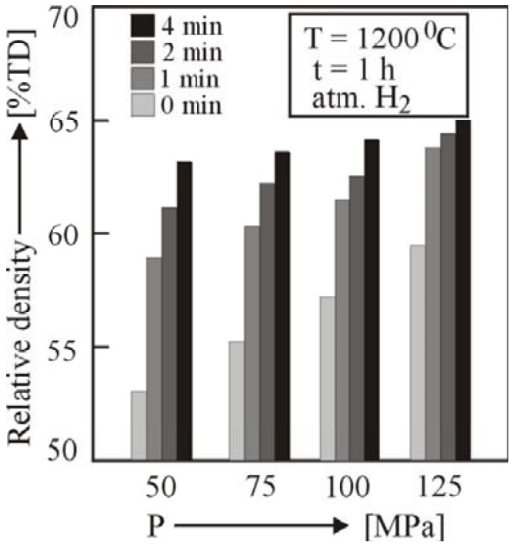


Fig.3. Relative sintered density vs. compaction pressure of W-specimens.

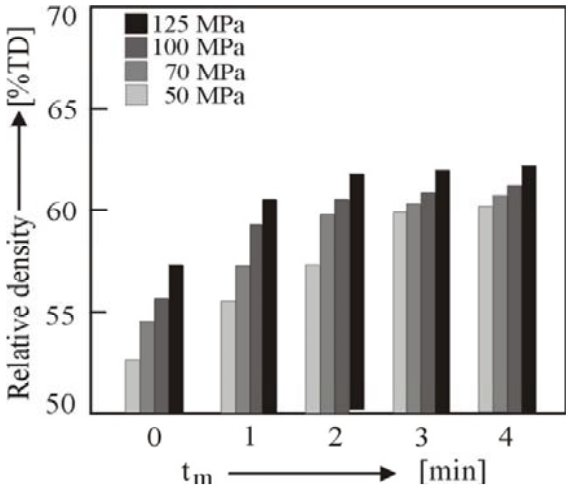


Fig.4. Relative green density vs. mechanical treatment duration of W-powders.

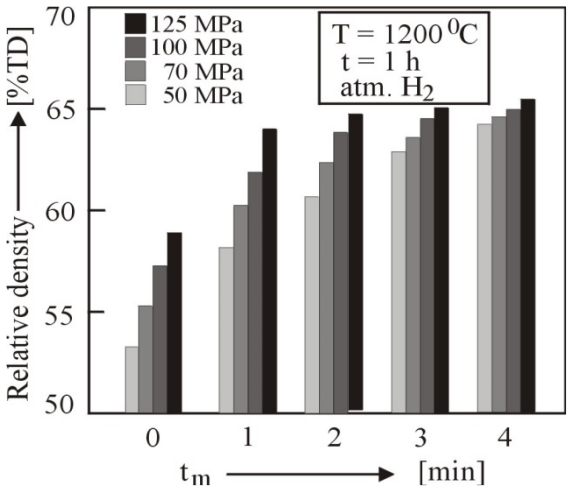


Fig.5. Relative sintered density vs. mechanical treatment duration of W-powders.

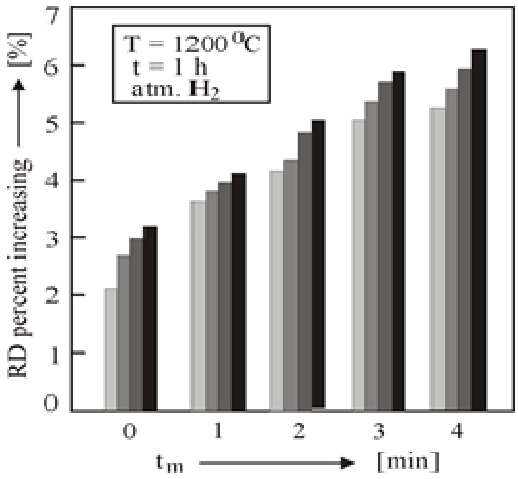


Fig.6. Percent increasing of relative density (%TD) from pressed to sintered state vs. duration of W-powder mechanical treatment.

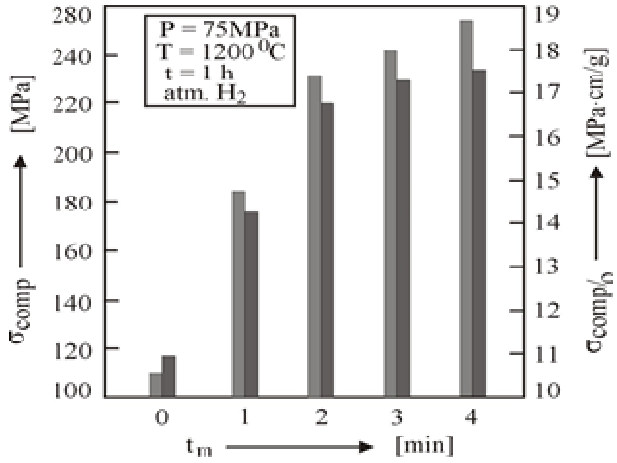


Fig.7. Compressive strengths and evaluation coefficient of W-sintered compacts vs. duration of W-powder mechanical treatment.

The pressure produces the hardening mainly at the contacts between the powder particles, while mechanically treatment harden the whole particle under the blows of milling balls and between the balls and the container wall. The cumulative effect of the two above mentioned actions is illustrated in figures 3, 5 and 6.

A carefully analysis of these three figures lead us to the conclusion that more effective on the activation on sintering is the influence of milling duration of the started W-powder. Another evidence of the sintering activation by mechanical treatment is given in figure 7, through the increasing of compressive strength and evaluation coefficient of sintered compacts with milling duration.

3. CONCLUSIONS

• The results obtained in the present study have proved that W-powders with a poor sinterability can be activated by means of a short mechanical treatment effectuated in a planetary ball mill. The activation can be attributed to the Ni activations: this should be considered.

• A mechanical processing for only 1 minute determines a significant sintering activation of W-powder. The effect of activation is diminished by further increase of treatment duration.

• The mechanical treatment of W-started powder determines, also, an important rising of neck strengths, a matter of a great interest in producing of porous tungsten bodies to be infiltrated with lower melting point metals, like copper or silver.

4. REFERENCES

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Studii privind sinterizarea activată a pulberilor de wolfram procesate mecanic

Rezumat: Anumite pulberi de wolfram prezintă o sinterizabilitatea scăzută. O parte din aceste pulberi de wolfram sunt obținute prin metode chimice din deşeuri metalice. Scopul acestei lucrări este de a îmbunătăți sinterizabilitatea acestor tipuri de pulberi prin aplicarea unor tratatamente mecanice scurte în mori planetare. S-au efectuat o serie de teste pentru a determina efectul duratei tratamentului mecanic asupra modificării proprietăților pulberilor respective: densitatea, suprafața specifică, dimensiunea medie a particulelor de pulbere, forma particulelor, microduritatea. Sinterizabilitatea pulberilor de wolfram procesate mecanic a fost studiată pe probe cilindrice obținute la diferite presiuni de compactizare (50-125 MPa) şi sinterizate succesiv în condiții identice (T=1200°C pentru 1 h, în atmosferă de hidrogen). Densitatea aparentă, rezistența la compresiune şi raportul dintre rezistența la compresiune după sinterizare şi densitatea aparentă au fost considerate ca şi parametri de evaluare a sinterizabilității funcție de durata de măcinare a pulberilor de wolfram.

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