

SINTERING OF TUNGSTEN FOR POROUS COMPONENTS

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Abstract - Tungsten (melting point is 3422^o C) is traditionally sintered at temperatures in excess of 2000^o C under hydrogen atmosphere using high temperature furnaces. The process is extremely expensive and it is difficult to control porosity which will be useful for many of the applications of porous tungsten. Thus the present study has focused on sintering of tungsten for porous components at low temperatures by the addition of activators. Here milled W powder was added in different compositions as an activator (10hrs-milled in high energy ball mill) to increase the sintering ability. The activated powder showed more densification because of the reduced particle size and higher specific surface energy and the strain energy. The sintering time and temperature were varied in order to study the sintering behavior and mechanical properties of the metal samples. The density was increased with increase of sintering temperature and soaking time. But the hardness and transverse rupture strength were reduced due to grain growth at higher soaking time (2 hr).

Key Words: Porous tungsten, Powder metallurgy, Activated solid state sintering, Metallographic study, Hardness, Transverse rupture strength or bending strength.

1. INTRODUCTION

Tungsten (W) has one of the highest melting points among all metals and therefore it is mostly used for high temperature applications. In its porous form, it can be used as dispenser cathodes used in high power lamps, filaments in light bulbs, rocket nozzles in space craft applications, etc. Since melting and casting of tungsten is extremely difficult, highly energy consuming and hence powder metallurgy (PM) has been preferred. Conventionally, tungsten powder, obtained from the reduction of tungsten oxide or Ammonium Para Tungstate (APT), is pressed then sintered at temperatures exceeding 2000^o C [11]. To reduce the sintering temperature, milled powder was added as an activator to increase the sintering ability of the W powder[1]. The main objective of the present study is to understand the effects of high-energy milling on the sinter ability of tungsten and also to make a comparative assessment between as-received (coarser) and activated (finer) tungsten powder in terms of their response to sintered density, microstructure. And the hardness, transverse ruptures were also observed.

1.1. Experimental procedure

Tungsten powder which was supplied by AVIS Machine Pvt. Ltd, Surat was used for the present experiments. Particle size of the powder was 1-3 μ m. This powder was milled for 10 hrs in high energy ball mill to reduce the particle size.

Material	Tungsten powder
Milling time	10 hr
Milling medium	Air
Ball to powder ratio	10:1
Milling speed	300 rpm
Grinding medium	WC balls

The milled powder was added to the as received powder in different compositions of 0%, 10%, 20%, 35%, and 50%. Each composition was added with 1.5% camphor which was dissolved in acetone for the uniform mixing of camphor. The powder was compacted at 100 MPa, 200MPa pressures with 0-25T Palletizing lab press (uni-axial hydraulic press), Kimaya engineers to get cylindrical samples. And TRS compacted samples [17] (which are used to measure transverse rupture strength) were made with the die of dimensions 31.75mm X 12.7mm X 6.5mm. Zinc stearate powder with acetone was applied as die wall lubricant. The compacted samples were sintered in horizontal MoSi₂ heated tubular furnace, OKAY Electric furnace, Model 7017 at 1470^oC and 1550^oC. Sintering was done at soaking time of 1hour and 2hours also. Density of sintered compacts which are impregnated with xylene [4] was measured by Archimedes water displacement principle.

Samples for metallography were also prepared. epoxy impregnation was used to fill the open porosity for the prevention of pore closure during polishing [9]. The polishing was done with 220 to 1000 SiC emery paper and final polishing was carried out with diamond spray of particle size 0.5 μ m. Micrographs were taken in both etched and unetched conditions by Optical Microscope. Etching was done with "Murkami Reagent" (prepared with

10 g of NaOH or KOH and 10 g of K₃Fe(CN)₈ in 100ml of water). Hardness was measured with Vickers hardness tester, Model HSV-20 at a load and dwell time of 5 kg and 15 s, respectively. Transverse rupture strength of the specimens measured with the test method of ASTM designation B528- 83a [17]&[4]. Capillary rise of the sintered compacts having different compositions was measured by keeping the sample in a patridish containing 1mm of water and a little amount of penetrant which was used in dye penetrant test (finding the cracks or flaws in metal).

2. RESULTS AND DISCUSSIONS

2.1 Milling:

XRD pattern of milling powder was shown in fig.1. Peak broadening and the crystalline size of 25 nm explained significant reduction in particle size. And the crystalline size was calculated by Scherer formula i.e. $(0.9\lambda / B \cos\theta)$. Where B is half width at full maximum.

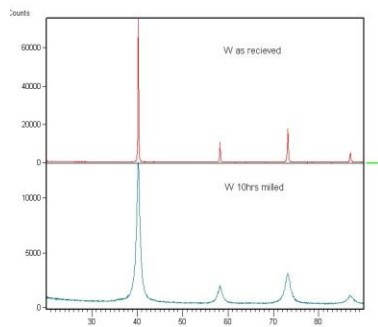


Fig.1 XRD pattern of milling powder

2.2 Density variations with different temperatures and soaking times of sintering:

Density values for different temperatures and different soaking times were given in table.2 and 3 respectively. Density was increased with temperature and soaking time. Increasing the sintering temperature greatly increases the rate and magnitude of any changes occurring during sintering, due to increased diffusion [13]. The density increment in case of pure tungsten (without milled powder) was 1 % but it was 5% in the samples which were added 50% milling powder. According to G.Prabhu et al., the improved densification in the activated powder was essentially due to the following reasons: (a) reactivity of the powder was high due to its reduced particle size and there by increased surface area. High-energy milling had resulted in creation of a new surface and thereby a higher energy state of the powder.

Load (Tones) pressure (Mpa)	density, g/cc		% theoretical density	
	At 1470° C	At 1550° C	At 1470° C	At 1550° C
16T / 325.907	10.3	10.9	53.57	56.48
10T / 203.692	10.3	10.57	53.55	54.78
5T / 101.846	9.5	9.89	49.24	51.25

Table.2 Density values for different temperatures

% MM added	density, g/cc			
	1 hr soaking		2 hr soaking	
	green(% TD)	Sintered (%TD)	green(% TD)	Sintered (%TD)
0	10.36(53.6%)	10.4(53.8%)	10.42 (54%)	10.7 (55%)
10	10.4(54.1%)	10.52(54.4%)	10.55(54.6%)	10.8(56%)
20	10.6(55.1%)	10.7(55.5%)	10.63 (55%)	11.187(57.96%)
35	10.6(55%)	10.8(56.2%)	10.8 (55.8%)	11.189(57.97%)
50	10.7(56%)	11.4 (59%)	10.79(55.9%)	11.8(61%)

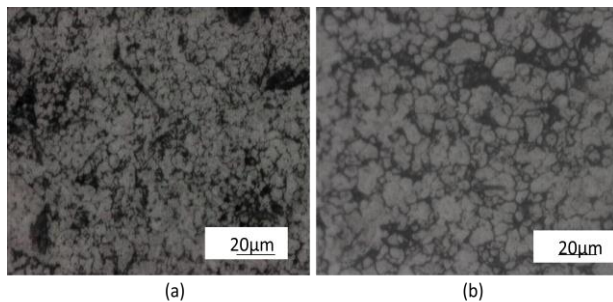
Table .3 The density of sintered samples which are compacted at 200 MPa

This facilitated atomic mobility during the sintering process resulting in rapid densification, (b) higher energy (originating from the significant strain of the particles due to high impact forces during milling) associated with the particles [1].

2.3 Metallographic study and Mechanical properties:

By observing the micrographs, it was known that better sintering was done with higher temperature and higher soaking time. Grain size was increased with soaking time [Fig.2]. Hardness and transverse rupture strength values are shown in table.4 & 5 respectively. Hardness and bending strength decreased with increasing the grain size of W although the relative density increased slightly at the same time [6]. And the samples were got fractured in brittle manner [17].

Fig.2 Micrographs of samples which are having 50% milling powder and compaction pressure is 200MPa (a) at



(a) 1 hr soaking time (b) 2hr soaking time

2.4. Capillary rise: The behavior of capillary rise was observed for the sintered compacts which were sintered at 1550°C. Capillary rise was increased with decrease in density [18]. Thus it was less for the sintered W compact which was having 50 % of MM compared to pure W (0% MM) as shown in fig 3.

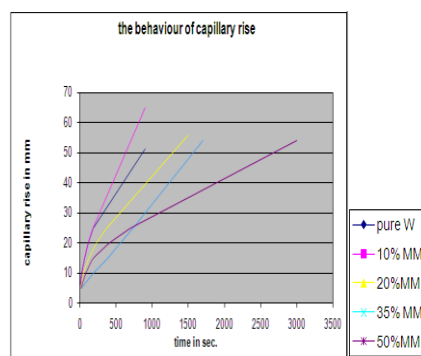


Fig.3. Behaviour of capillary rise

3. CONCLUSIONS

Sintering temperature increases the densification kinetics. Density was increased with the addition of milling powder due to the reduction in particle size and higher specific surface energy (because of the increased surface area of the high-energy milled powder) and the strain energy (because of the strain of the particles during high-energy milling) associated with the particles.

Hardness depends on sintered density. According to the results, pure W has lesser density thus it is having less hardness. It was observed in the samples which were sintered at 1470 and 1550° C. When the W samples with different percentage of milling powder were sintered at same temperature and at different soaking times (1hr and 2hrs) , hardness was decreased with grain size increment even though density increased.

Bending strength or transverse rupture strength was also decreased with increase in grain size. It was due to the increase in soaking time. Capillary rise was decreased with the increase of sintered density.

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REFERENCES

1. G. Prabhu *, Amitava Chakraborty, Bijoy Sarma, "Microwave sintering of tungsten" Int. Journal of Refractory Metals & Hard Materials 27 (2009) 545–548.
2. S. Broglu, Tubitak – MRC, Gebze kocaeli, Turkey "Effects of tungsten powder size and mixing technology on properties and structure of heavy alloys".
3. T.P. Bagchi, N. Arvind Kumar, Bijoy Sarma, N. Maitra " Problem solving in sintering of tungsten heavy alloy product — a case study" , Materials Chemistry and Physics 67 (2001).
4. Powder Metallurgy Technology, G. S. Upadhyaya, Department of Materials and Metallurgical Engineering Indian Institute of Technology, Kanpur, India, Published by Cambridge International Science Publishing.
5. M.Amirjan, K. Zangeneh-Madar , N. Parvin "Evaluation of microstructure and contiguity of W/Cu composites prepared by coated tungsten powders" Int. Journal of Refractory Metals & Hard Materials 27 (2009) 729–733.
6. Zhangjian Zhou, Gerald Pintsuk, Jochen Linke, Takeshi Hirai, Manfred Rödiger, Yao Ma, Changchun Ge "Transient high heat load tests on pure ultra-fine grained tungsten fabricated by resistance sintering under ultra-high pressure" Fusion Engineering and Design 85 (2010) 115–12.
7. Vivek K. Gupta, Dang-Hyok Yoon, Harry M. Meyer III, Jian Luo "Thin intergranular films and solid-state activated sintering in nickel-doped tungsten" Acta Materialia 55 (2007) 3131–3142.
8. Hideo Nakajima "Fabrication, properties and application of porous metals with directional pores" Progress in Materials Science 52 (2007) 1091–1173.
9. Alan Lawleya, Thomas F. Murphy "Metallography of powder metallurgy materials" Materials Characterization 51 (2003) 315– 327.

10. TONY PIOTROWSKI AND DANTE , J. ACCINNO
"Metallography of the Precious Metals" Previously published in METALLOGRAPHY 10:243-289 (1977).
11. C. Selcuk, J.V. Wood, "Reactive sintering of porous tungsten: A cost effective sustainable technique for the manufacturing of high current density cathodes to be used in flash lamps", Journal of Materials processing Technology 170(2005) page no.471-476.
12. Yuttannat Boonyongmaneerat, "Mechanical properties of partially sintered materials", Materials Science and Engineering 44:452-453 (2007) ,page no.773-780.
13. Powder Metallurgy Science, Technology and Applications by P.C. Angelo, R. Subramanian (2008).
14. R.L. Orban et al. (University of Cluj-Napoca, Cluj-Napoca, Romania, Refractory metals, "Activity of W powder- a controlling parameter for inter connected pores in sintered porous tungsten"- Abstract.
15. Tungsten properties, chemistry, technology of the element, alloys by Erik Lassmer, Wolf-Dieter Schubert. pages-276,285,302.
16. ASM Handbook -7th volume-Powder metal Technology and Applications.
17. ASTM designation: B528-83a "Standard Test method for Transverse Rupture Strength of Sintered Metal Powder Specimens".
18. Suresh Thokchom¹, Partha Ghosh² and Somnath Ghosh¹ "Effect of water absorption, Porosity and Sorptivity on durability of Geopolymer mortars", ARPN Journal of Engineering and Applied Sciences.