

DYNAMIC COMPRESSION BEHAVIOR OF TUNGSTEN POWDERS CONSOLIDATED BY PLASMA PRESSURE COMPACTION (P²C™)

Sang H. Yoo, T.S. Sudarshan, Krupa Sethuram

Materials Modification Inc, 2929-P1 Eskridge Rd, Fairfax, VA, 22031

Ghatu Subhash

Michigan Technological University, Houghton, MI, 49931

R.J. Dowding

U.S. Army Research Laboratory, Aberdeen Proving Ground, MD, 21005

***Abstract** – It has been generally believed that the ductility of tungsten can be increased by decreasing grain size to ultra fine levels (~100 nm). Pure ultra fine grain tungsten powders of various average particle size (2.0mm, 0.7mm, and 0.16mm) were consolidated using the rapid consolidation technique, Plasma Pressure Compaction (P²C), to minimize grain growth. Preliminary results indicate that tungsten powders can be consolidated to high density without abnormal grain growth at 2000°C and 50 MPa with less than 3 min of isothermal holding time in the P²C process. The densities of consolidated specimens were measured using Archimedes' method, and the microstructure was analyzed using SEM. The mechanical behavior of the consolidated specimens was investigated using the compression Kolsky bar. Dynamic compression properties were examined to determine the effect of grain size, grain structure, and density on yield stress at various strain rates.*

INTRODUCTION

The mechanical properties of polycrystalline metals and alloys are very sensitive to their grain size. The Hall-Petch relationship has shown that by refining grain size at room temperature, the strength of a polycrystalline material can be effectively increased [1]. In addition, deformation studies have shown that ductile behavior is observed in polycrystalline metals with fine grain structure [2, 3]. However, the effect of tungsten grain size refinement on its flow and failure behavior has not been studied.

Polycrystalline pure tungsten is essentially brittle for various reasons at quasi-static and elevated strain rates. Impurity content, especially oxygen, plays a significant role. Plasma Pressure Compaction (P²C) is a field-activated consolidation technique that allows surface activation and rapid densification of various powders in a short cycle time (<15 minutes). Now that ultra fine-grained tungsten particles are available along with a field-activated fast consolidation technique, the restrictions of classical pure tungsten may be overcome.

EXPERIMENTAL PROCEDURE

Tungsten powders of average particle size 2.0 μm and 0.7 μm were obtained from Wah Chang (Albany OR), and 0.16 μm tungsten powder was synthesized at Materials Modification Inc. (Fairfax, VA), using a DC plasma synthesis system. Average particle size was determined using a Horiba laser scattering particle size analyzer, LA-910.

In the P²C process, the tungsten powders were first loaded into a graphite die and compressed by graphite punches to establish a current path. In the initial stage of consolidation, pulsed voltage (50 ms on-time, 50 ms off-time, 0-1500 amp, 0-4 V, 3 min duration) was applied across the powder compacts to generate interparticle plasma that activates the particle surfaces by removing the oxide and contaminant layers. Subsequently, the power output mode is changed to apply a continuous DC voltage to densify the powder compact by resistance sintering at 2000 °C and 50 MPa for <3 minute. The temperature of the graphite die surface was measured using an optical pyrometer.

The consolidated tungsten samples (2.5 cm diameter \times 1 cm long) were taken out of the graphite die and their surfaces were ground to remove carbon contamination. After measuring the final densities using the Archimedes method, samples were cut in half, polished, and then etched for SEM characterization.

For mechanical testing, cylindrical test specimens of diameter 2.5 mm and length 4.5 mm were prepared by cutting consolidated samples using electro-discharge machining (EDM). High strain rate experiments were performed using a Split Hopkinson Pressure Bar (SHPB) at room temperature.

RESULTS AND DISCUSSION

Processing parameters and resulting densities of P²C consolidated tungsten powders are shown in Table 1. Faster densification is expected as particle size is reduced from 2.0 to 0.16 μm because the small tungsten particles have high surface area and more energy per unit volume, which contributes to faster sintering. However, comparison of the consolidated samples shows that density does not increase steadily with decrease in average particle size in the P²C process. As average particle size was reduced from 2.0 μm to 0.16 μm , the density remained more or less constant at the same processing conditions. The high surface energy available in the finest particle size probably did not promote sintering because of agglomeration, oxidation, and contamination.

An SEM picture of the PP20 sample (0.16 μm) is shown in the Figure 1. The microstructure indicates that significant grain growth occurred due to pore-grain boundary separation. Pores along grain boundaries as well as inside grains can be observed clearly in this micrograph. Grain boundary mobility must be controlled in order to minimize grain growth in the ultrafine tungsten powders.

Table 1. Effect of P²C parameters on densities of the consolidated samples.

Powders	Sample ID	Temp. °C	Hold Time min	Pressure MPa	Density %
W (2.0μm)	PP 12	2000	<3	50	94%
W (0.7μm)	PP 21	2000	<3	50	95%
W (0.16μm)	PP 20	2000	<3	50	93%

Figure 1 SEM picture of PP20 specimen consolidated from 0.16μm tungsten powders.

Most of the 2.0μm and 0.7μm tungsten specimens failed prematurely and pulverized upon impact at high strain rates (1000-5000 s⁻¹). Such deformation behavior is expected at the high strain rates because polycrystalline tungsten has been found to be extremely brittle [4]. The P²C consolidated tungsten specimens also exhibited strain rate sensitive behavior; as the strain rate increases, the flow stress also increased. The strain rate sensitivity is typical of polycrystalline tungsten and its composites [5] as well as tungsten single crystals [6].

Ductility and flow stress increased dramatically as initial particle size is reduced to 0.16 μm . As shown in Figure 2, true strain and flow stress increased to 0.3 and 2250 MPa, respectively at 4000/s. Flow stress may increase by decreasing grain size as predicted by the Hall-Petch relation due to grain boundary strengthening. However, such high strain has not been previously observed in randomly-oriented polycrystalline tungsten. Further efforts are required to investigate the deformation mechanisms of tungsten.

Figure 2 True stress-true strain curves of tungsten specimens (0.16 μm) at high strain rates.

SUMMARY

- Tungsten powders were consolidated rapidly by Plasma Pressure Compaction within 3 minutes of isothermal holding time at 2000 °C and 50 MPa.
- High ductility and flow stress can be obtained by rapidly consolidating ultra fine particles to high density.
- Further study is required to investigate high strain rate ductility and deformation mechanisms of tungsten consolidated from the ultra fine particles.

REFERENCES

- [1] J.C.M. Li and Y.T. Chou, Metallurgical Transactions, **1**, p1145, 1970.
- [2] J. Karch, R. Birringer, and H. Gleiter, Nature, **330**, p556, 1987
- [3] A.H. Chokshi, A. Rosen, J. Karch, and H. Gleiter, Scripta Metallurgica, **23**, p1679, 1989.
- [5] D. Hull, Acta Metallurgica et Materialia. **8**, 11, p769, 1960.
- [6] J.W. Christian, Metallurgical Transactions A, **12A**, p1237, 1993.