4.2 EFFECT OF GREEN DENSITY AND FAST SINTERING ON GRAIN GROWTH OF NANO W — X. Wang and Z. Z. Fang (University of Utah)

OBJECTIVE

The objective of this research is to evaluate the effect of green density and fast sintering (rapid heating and high pressure) on the grain growth of nano W and W-TiC powders produced by a high energy planetary ball milling process.

SUMMARY

Two novel processing techniques were developed for inhibiting the grain growth of W during sintering of nano W and W alloy powders; one of which is a hot-pressing green compaction technique for increasing the relative density of green compacts of the nano powders. The second is a fast sintering technique for applying a combination of rapid heating and high pressure during sintering. Nano W and W-TiC powders were used to investigate the effect of green density and fast sintering on grain growth. The following preliminary results were obtained:

(1) The maximum obtainable green density of cold-pressed samples was 38% of the theoretical full density at 800 MPa, while the hot-pressed sample exhibited an 18% increase in green density (56% TD).
(2) Higher green density enabled the samples to be fully sintered at lower temperature.
(3) Near fully dense bodies with a grain size smaller than 300 nm were obtained via fast sintering.

PROGRESS AND STATUS

Introduction

From a structural viewpoint, the major concerns in the development of W alloys for future fusion reactors are their high ductile-to-brittle transition temperature (DBTT) and low fracture toughness. Nanostructuring of tungsten and tungsten alloys could provide a solution to address those concerns [1-4]. In the past, severe plastic deformation processes, such as high-pressure torsion (HPT) have been investigated to obtain nanoscale grain structure. These processes, however, are not suitable for producing industrial-scale finished products. Therefore in this work, we developed new strategies following powder metallurgy routes.

Our previous work [8] has shown that a high energy milled nano tungsten powder can be sintered to 99% relative density at 1100°C under hydrogen, but the final grain size after sintering is larger than 2 micrometers, far beyond the nanoscale. The initial grain growth takes place rapidly when the relative density of the material is very low at low temperatures. Increasing green density and/or lowering sintering temperature will effectively minimize this part of the grain growth. However, the milled nanosized tungsten powders are difficult to press into green compact with only 35-40% relative density using conventional approaches, which makes the material prone to rapid grain growth before densification.

For controlling grain growth of nano W, a novel method to compact nano W powders has been recently developed. The compaction is being done at a moderate temperature that is above its DBTT, but below temperatures that would cause rapid grain growth. This process is expected to yield green compacts with high relative densities, which would significantly improve the chance of obtaining full densification with minimum grain growth during subsequent sintering.

Additionally, a modified rapid omni-directional compaction (m-ROC) process has been investigated for sintering the nano W powders. The ROC process is known to be very effective for consolidating metal or ceramic powders to full density [9]. The m-ROC, which combines rapid, heating (>200°C/min) with a high pressure (up to 1.0 GPa), offers one of the most promising approaches for limiting grain growth while achieving full densification [10]. Rapid heating minimizes sample exposure in the temperature range for rapid coarsening of grains, which results from grain/particle coalescence and is controlled by surface diffusion [11]. The use of high pressure aids the densification process at relatively low temperature.

W-TiC nano powder was processed together with the nano W powder to study the effect of green density and fast sintering on grain growth. This preliminary investigation effectively demonstrated that the W grains grew slowly under higher relative green density and faster sintering.

Experimental Procedure

Improving green density

Nano W and W-1TiC powders were prepared by using a unique high-energy planetary ball milling (HEPM) process. The powders were milled for 6 hours in heptane. The particle size of most of the particles was in the range of 10–55nm. The average W crystallite size was determined to be 12 nm by using X-ray diffraction (XRD) and the Stokes and Wilson formula for grain size determination from peak broadening. Milled nano tungsten powders were compacted using a uniaxial press with heaters shown schematically in the Fig. 1. This hot-pressing system includes a die assembly, a heating device, and a gas control system. The die components can operate at temperatures, up to ~700°C. During compaction, the die assembly is heated under a cover gas of flowing argon. When the temperature reaches a point above the DBTT, high pressures are applied. Sintering experiments to validate this method were carried out at 1100°C for 1 h in a hydrogen atmosphere using a tube furnace. Our previous results have shown that the HEPM process significantly enhances the sinterability of the nanosized tungsten powders, achieving near-full density at 1100°C but with final grain size larger than 2 μm.

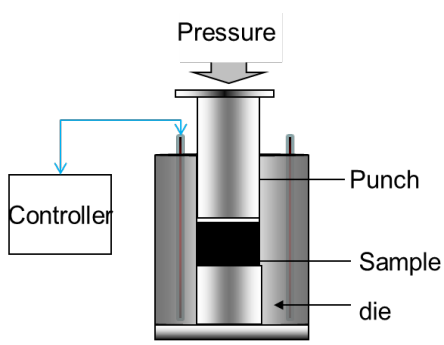

Figure 1. Schematic for the novel green compaction technique with temperatures up to 700 °C.

Fast sintering

Figure 2 shows the schematic diagram of the modified rapid omni-directional compaction (m-ROC) process. The milled powder was first compacted at 250 MPa into cylindrical samples. The samples were introduced into a graphite cell of the m-ROC system. The processing was carried out at 1100°C with ultrahigh isostatic pressure of 800 MPa. During the m-ROC processing, the samples were heated in situ using resistance heating. In m-ROC processing, the pressure medium conveys the isostatic pressure.

Characterization

After sintering, the densities of the samples were measured by using Archimedes method. The microstructural changes of the fractured surface of the sintered samples were observed using Scanning Electron Microscopy (SEM). Tungsten grain sizes were determined from the SEM micrographs by using the linear intercept method.

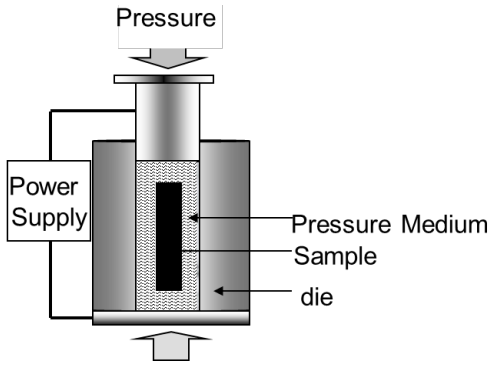

Figure 2. Schematic diagram of apparatus for modified rapid omni-directional compaction (m-ROC) process

RESULTS

Effect of green density

Figure 3 shows the relative green density of hot pressed samples as a function of temperature at 800 MPa. As shown, a continuous increase in green density was observed above 250°C. Below 250°C, the relative density varies slightly. A significant increase of 18% in density was achieved at 450°C. These results indicated that the DBTT for the milled nano W powder is between 200-300°C.

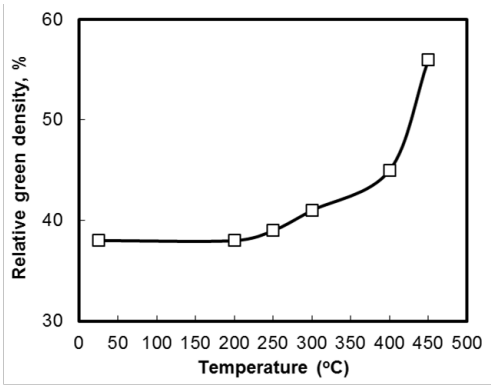

Figure 3. The effect of pressing temperature on the relative green density of nano W powder

The effect of green density on the W grain growth and densification is shown in Fig. 4. It can be seen that at the same sintering temperature, the higher the relative green density, the smaller the grain size and the higher the sintered density are. As the densification rate is enhanced by higher density in the green compacts, sintering temperature, as well as sintering time, can be reduced more and grain growth further suppressed. Incrementally increasing the green density of W-TiC alloys indicates promise for obtaining W grain sizes < 100 nm.

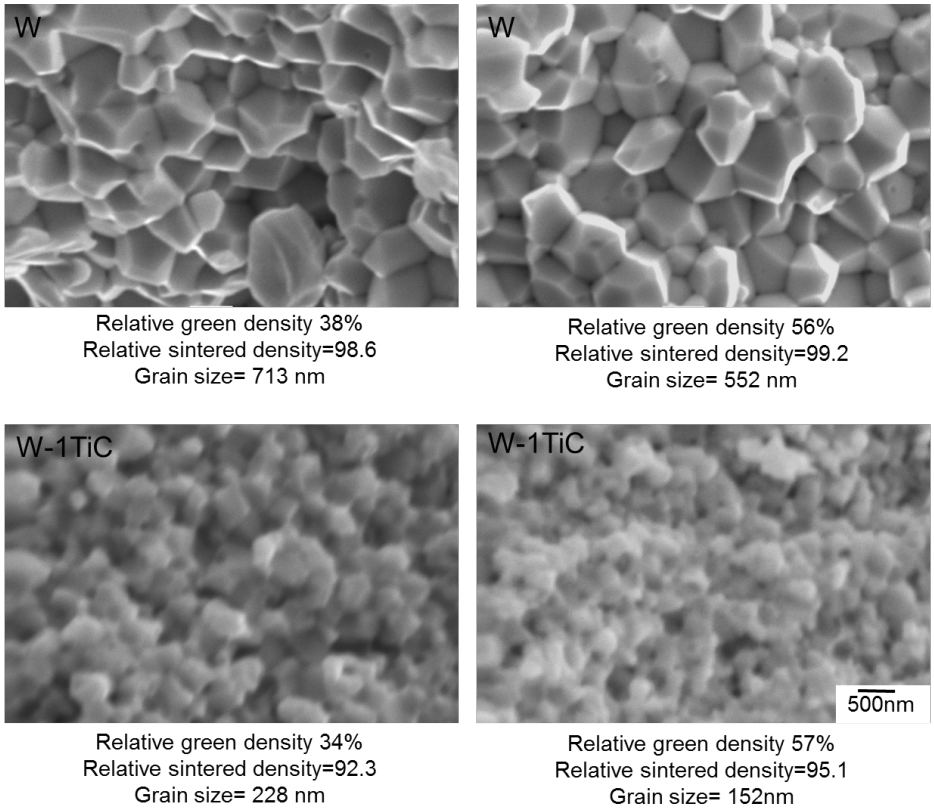

Figure 4. SEM images of fracture surface of sintered nano W and W-1TiC 1100 °C for 1h.

Effect of fast sintering

Preliminary Investigations on the consolidation of nano W alloy powders by the m-ROC have shown that the rapid heating (>200°C/min) and high pressure are beneficial to achieving small grain size. Figure 5 shows two micrographs from a W sample and a W-1TiC after pressing in the m-ROC with a heating rate of ~ 300°C/min and a maximum temperature of about 1200°C for 2 min. Compared to the W grain size measured from a sample sintered at 1200°C for 5 min in a tube furnace (not shown, but measured at 1 μm), the W grain size by m-ROC is much smaller, Fig. 5.

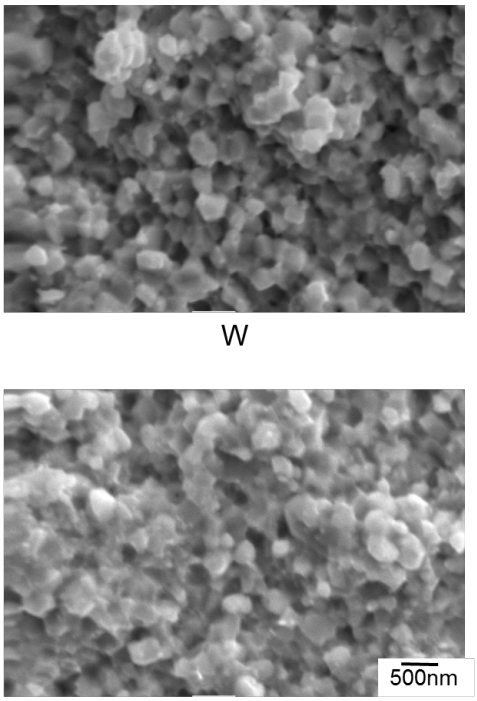

Figure 5. SEM images of fracture surface of sintered nano W- and W-1TiC by M-ROC.

FUTURE WORK

The processes discussed will be further optimized to achieve high density and minimized grain size after sintering of the nano W and W alloy powders. Additionally, mechanical properties of sintered samples will be evaluated.

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