4.3 SPECIMEN PREPARATIONS FOR MECHANICAL TESTING AND HYDROGEN PERMEATION OF NANO W-1Ti-0.5TiC —

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OBJECTIVE

The objective of this work is to prepare specimens for evaluating mechanical properties and hydrogen permeation of Nano W-1Ti-0.5TiC for fusion reactor materials applications.

SUMMARY

During the last reporting period, processing for making nano W with grain size near 100 nm was developed. The ongoing work in this reporting period is focused on evaluating mechanical properties and hydrogen permeation of the nano W alloys. So far, the specimen preparation for testing is being really difficult due to cracking problems during pressing and sintering, which originated from the special characteristics of the high energy milled nano W powders made in this project. Attempts have been made to make crack-free specimens for property evaluations of Nano W.

PROGRESS AND STATUS

Introduction

For fusion reactors, the material of choice must have well-established and high performance mechanical properties before and after irradiation. During the last reporting periods, we have developed processing for making nano W with grain size near 100 nm [1, 2]. First of all, the mechanical properties of the developed nano-W alloys at unirradiated state need to valid. However, the large samples after the processing showed some laminate cracks and radial cracks. For evaluating mechanical properties of nano W developed, the cracking problems of large samples have to be eliminated.

In this work, control of the processing parameters was investigated to eliminate cracking. Basically, the characteristics of nano-W powders with high specific surface area result in low and non-uniform density distributions in the large green compact to a great extent. Furthermore, the densification rate for the nano W powder made in this project can be really fast [1]. These lead to rapid and non-uniform shrinkage of the sintered compact, causing cracking. For achieving a high quality fully sintered testing specimen, the cracking problem may be solved via deceasing density inhomogeneity throughout the green compact and slow heating during sintering. The most sensitive parameters including compaction pressure, the heating rating of sintering, the pressing methods and the amount of binders were attempted for removing the cracks.

Experimental Procedure

Commercial tungsten powders with average particle size of ~ 100 nm were used as the raw materials. Commercially available Titanium Hydride (TiH_2) powders (average particle size of -45 µm) were used as the second phase element Ti and Titanium carbide (TiC) as the grain growth inhibitor. The powder W-1Ti-0.5TiC (in wt%) was milled in unique high energy ball milling machine [1] for 6 hours in a mixture of heptane and ethanol (15 vol%). Tungsten carbides (~ 1 mm) were used as the grinding media. Polyethylene glycol (PEG) and paraffin wax with different weight ratios were added as plastic binders and lubricants. The milled nano tungsten powders were dried in a vacuum desiccator and then were compacted using a uniaxial press and a cold isostatic press (CIP) at different pressures. The green compacts were then treated in hydrogen at different temperatures (250, 650, and up to 1100 ◦C) with different time durations and heating rates (1-10 ◦C/min) for debinding, reducing, and sintering.

Density measurements of the green and sintered specimens were conducted by simple geometric and mass measurements, as well as by the Archimedes technique using distilled water as the displacement

medium. The microstructures 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.

Results

Previously, only small samples for microstructural analysis and hardness testing were made. Figure 1(a) shows a photo of the small sample which is free of cracks. For measuring a wide range of properties of Nano W, large specimens with two shapes, rod and disc, have been made.

Rod specimens

Table 1 lists the process parameters tested for CIPed and uniaxial pressed nano W rods. At first, there were no plastic binders in the powers. Cracks were observed at each processing steps as shown in Table 2. As expected, the lower the pressure and the slower heating rate, the less and the finer the cracks. There were, however, still cracks for the pre-sintered rod (in Figure 1(b)) with a pressure of 50 MPa and a heating rate of 1 °C /min.

On uniaxial pressing, zinc stearate in ethanol as lubricant was coated on die wall. ~ 20 vol %, 45 vol % and 70 vol % plastic binder were added. For sintering, the heating rate was 1 °C /min. The observation of cracking during processing is give in Table 3. The higher amount of binders and the lower pressure, the smaller the crack size. The cracks moved from the end to the middle, indicating the decease of the density inhomogeneity. There was still very fine crack in the middle of the sample, see Figure 1(d) .
In short, we need to further adjust our processing to prepare high quality large samples for measuring mechanical properties.

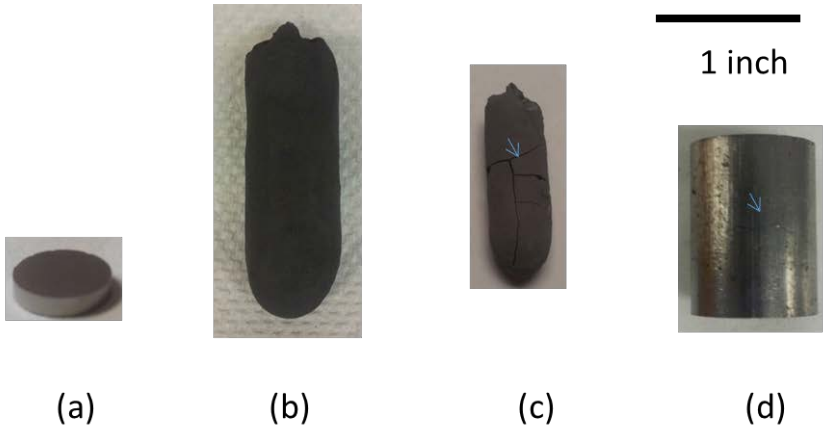

Figure 1. W rods (a) small specimen with height ~3 mm, sintered at 1100°C for 1 hour, Heating Rate=10°C/min. (b) CIPed under 100 MPa (b) CIPed (b) and then sintered at 1000°C. (d) 70 vol% binder, uniaxial pressed 80 MP, sintered at 1000°C.

Table 1. Process parameters tested for CIPed rods

CIPing pressure (MPa)	Heating rates (°C /min)
300	10
150	5
100	1
50	

Table 2. Observations of cracking for CIPed rods

CIPing pressure (MPa)	Heating rate °C /min	After CIPing	After reduction at 650 °C for 2h	Crack after pre sinter
300	10	N	Y	Y
300	1	N	Y	
100	1	N	Y	
50	1	N	N	Y

Table 3. Observations of cracking for uniaxial pressed rods

binder vol%,	After debinding	after reducing at 650C	Crack after pre sinter?
20%	Y		
45%	N	Y	
70%	N	N	Y

Disc specimens

The nano-W alloys are expected to have good resistance to radiation damage such as hydrogen blistering. 1" thin nano-W disc specimens are being made for measuring tritium permeating [3]. Figure 2 shows the typical crack occurred after sintering the nano W powders. Experiments have indicated that the crack appears in the low density region of the green compact. This should be improved by increasing the flow-ability of the nano W powders, such as granulation. Figure 3 demonstrates the morphology of fracture surface of the sintered specimens with grain size of ~ 200 nm.

Figure 2. Nano W disc sintered at 1100°C for 1 hour.

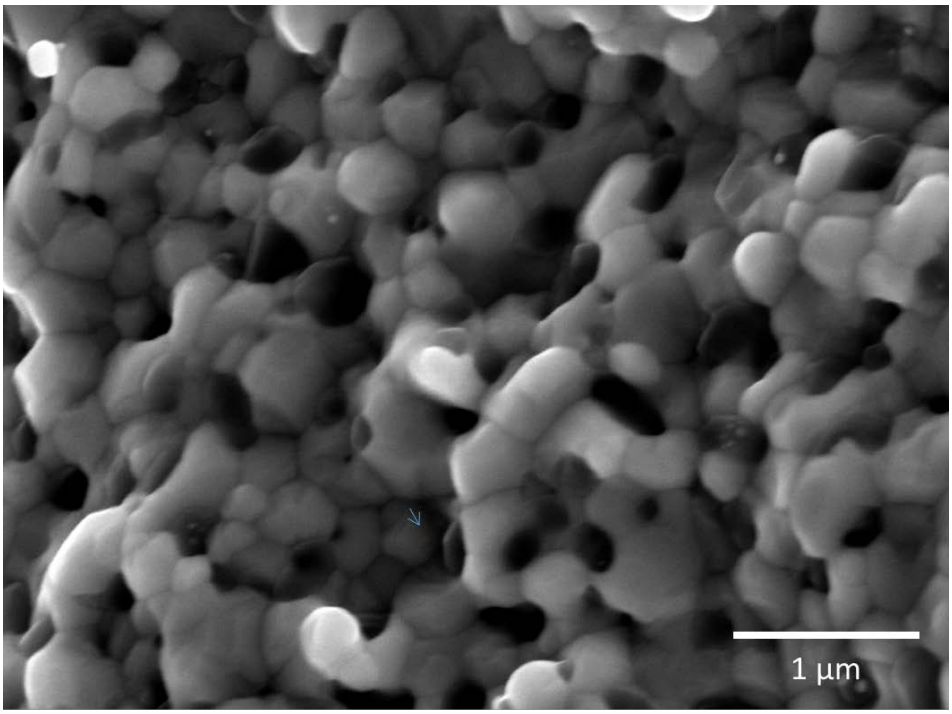

Figure 3. SEM micrograph of Nano W disc sintered at 1100°C for 1 hour.

FUTURE WORK

The processes will be further optimized to achieve high quality test specimens of the nano W alloys. Then the properties of nano W (including mechanical properties and hydrogen permeation) will be evaluated.

REFERENCES

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[3] D. Buchenauer, et al. "Development of plasma driven permeation experiment for TPE". Fusion Engineering and Design, 2014, In press.