IWSMT-10

Study on Microwave Sintering of Tungsten at Relatively Low Temperature

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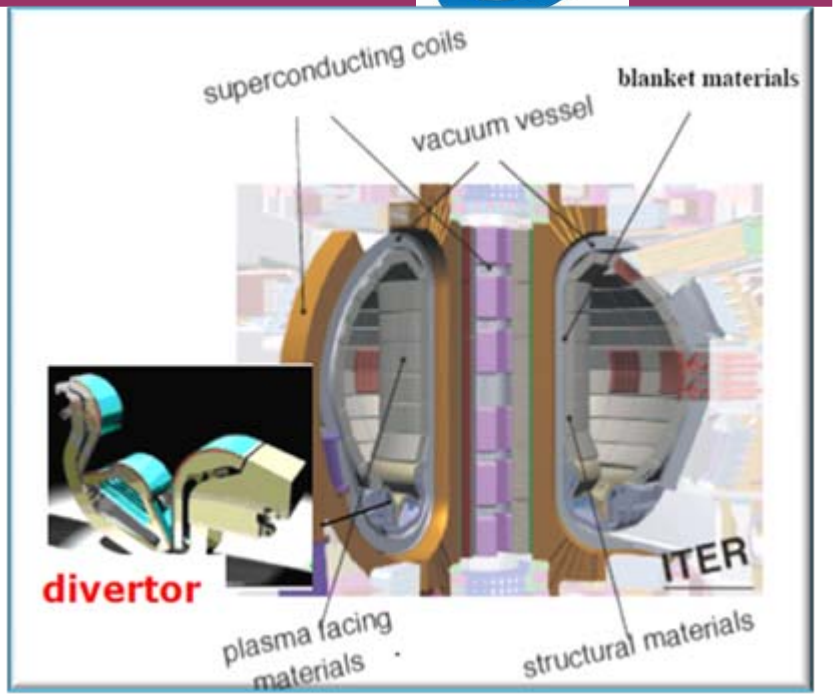
Outline

- Introduction
- Sample preparation
- Experimental results
- Conclusions

1. Introduction

Tungsten: promising solid spallation target and PFM materials in advanced fusion reactors (ITER , DEMO)



Advantage:

- low sputtering yield;
- less chemical erosion;
- low tritium retention ;
- good thermal-stability;……

Problem:
the high melting point (3683K) makes it difficult to prepare bulk W by melting and casting method.

High density W: Powder metallurgy method

High density Tungsten: Powder metallurgy method

- **Conventional resistance sintering method(heat transfer by radiation or conduction): high sintering temperature (T>2273K); Long soaking time; ⇨ Coarsen grain size**

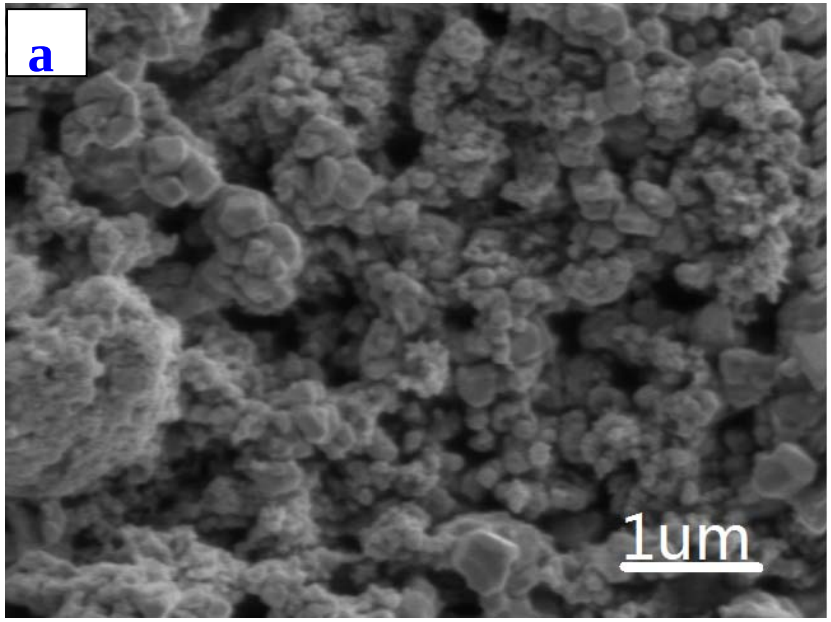
- **Microwave sintering: heated by eddy-current loss (electromagnetic energy transfers to heat)**

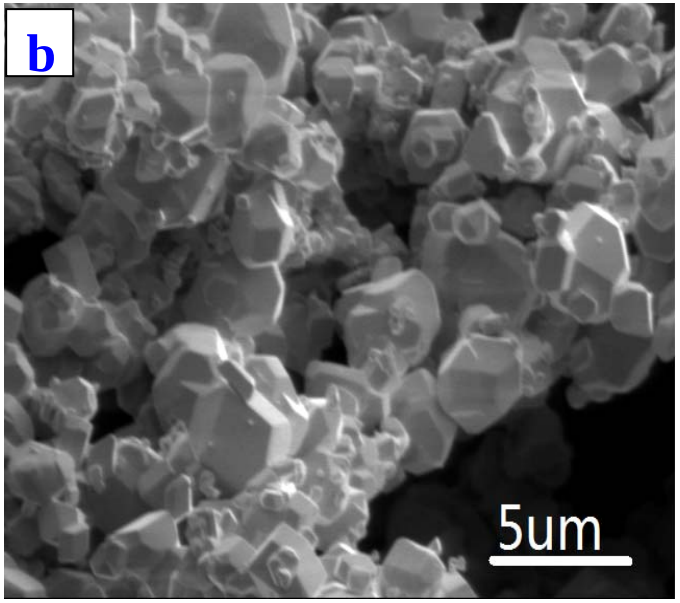
Advantage: higher heating rate, low sintering temperature, short sintering time and low energy consumption; ⇨ Finer grain size

Purpose : To prepare high density bulk tungsten using microwave sintering !

2. Sample preparation

To evaluate the influence of grain size on sintering properties, two kinds of W powder are used:





(a) Fine powder:initial size 20-100 nm ; spherical shape; agglomeration

(b) Coarse powder :initial size ~3um; cubical shape

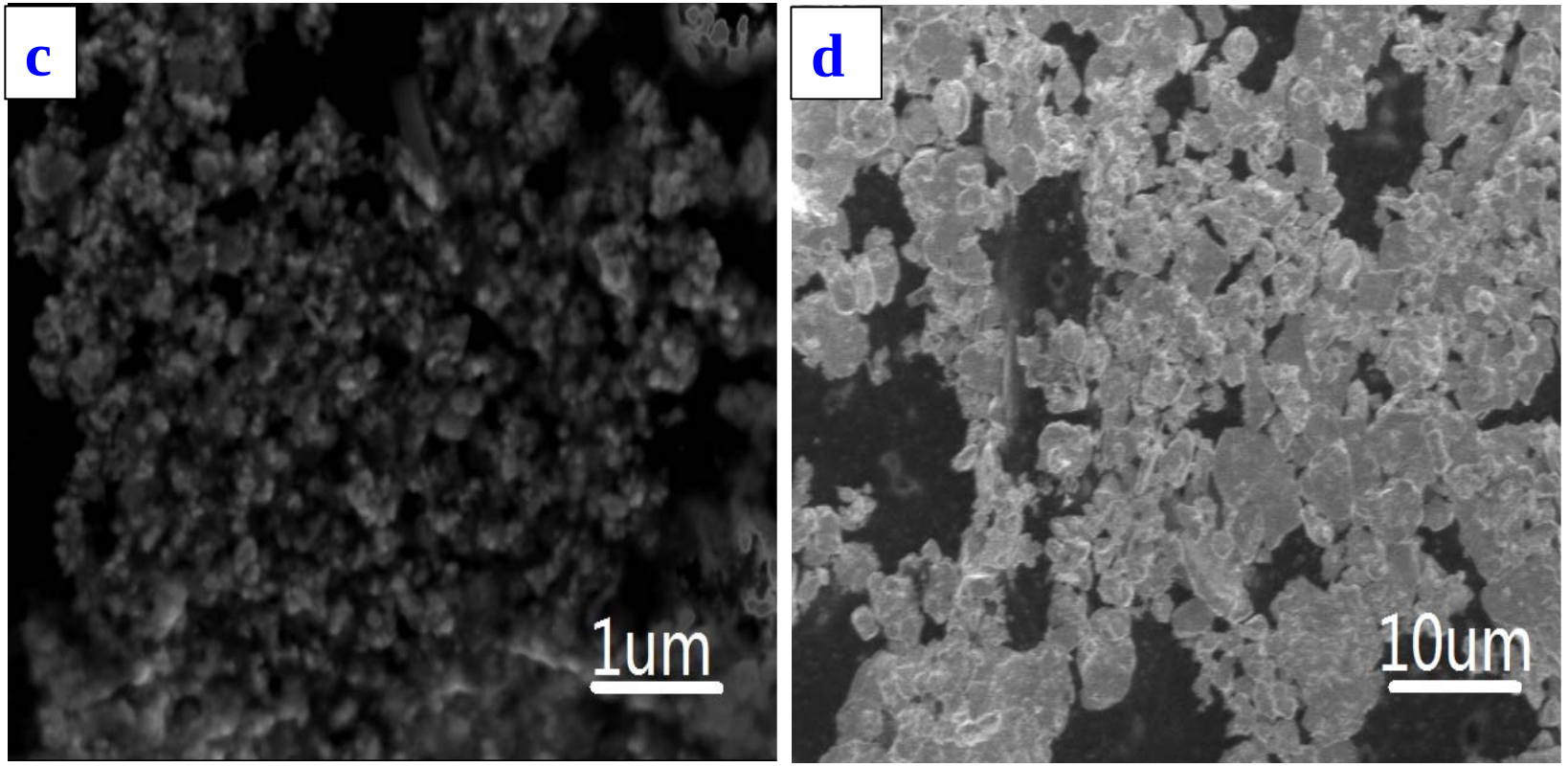
High energy ball milling: improve sintering activity

- **Carbide ball (WC-Co)**
- **Stainless steel ball**

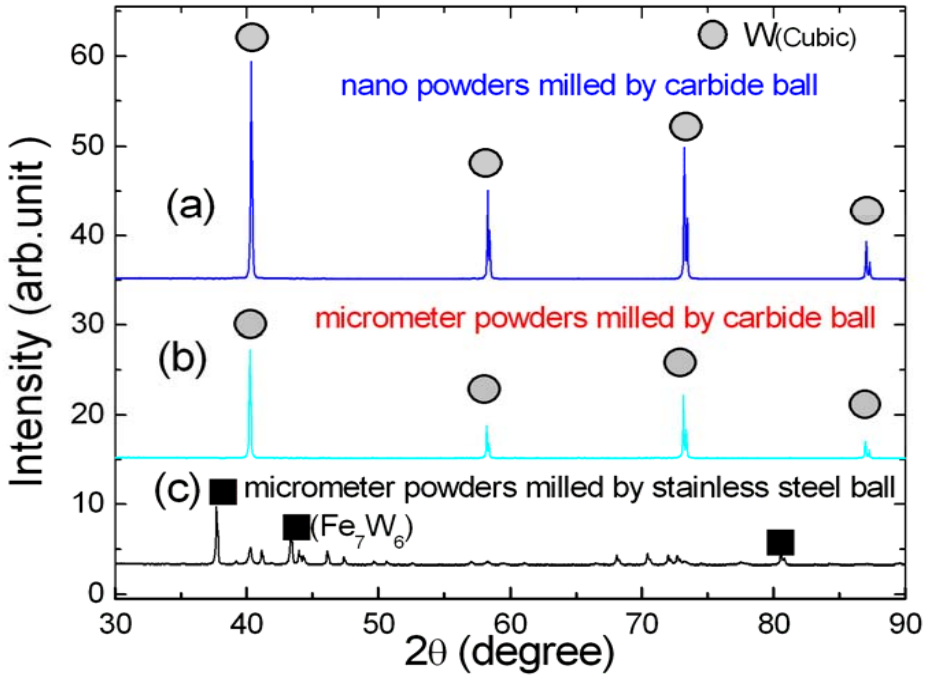
Table : Main milling parameters used in the present study

Initial Material	Tungsten powder (20~100nm, 3um)
Milling time	5h
Milling medium	Ethanol
Grinding medium	Carbide or stainless balls (4-10mm in diameter)
Ball to powder ratio	10:1
Plate speed	240 rpm

Morphology of tungsten powder after ball milling

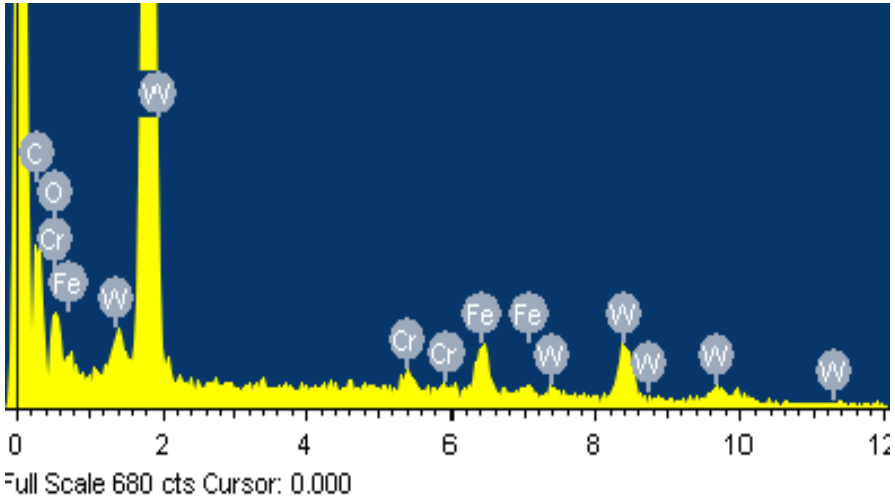


(c) Nano powder: the agglomeration is almost eliminated;
(d) Coarse powder: the surface is smoothened



XRD patterns of the ball milled W samples

- **Carbide ball: pure cubic W phase**
- **Stainless steel ball: XRD pattern very complex, main impurity phase is Fe_7W_6 alloy**

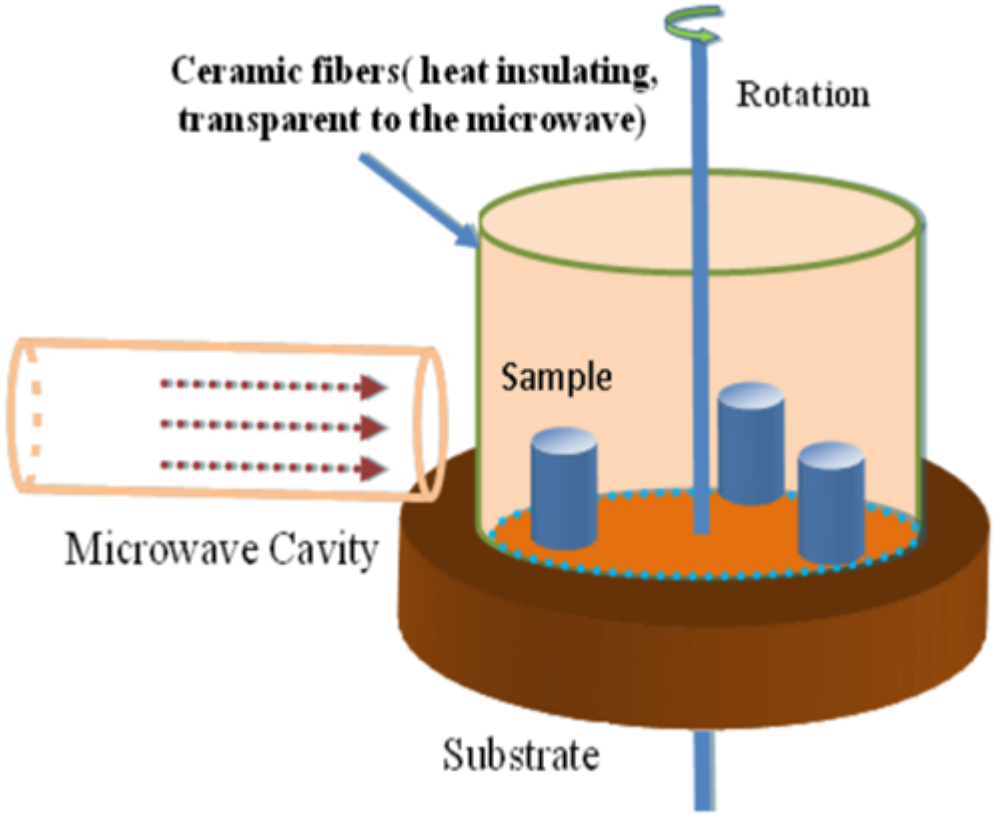


EDX analysis evidences the existence of impurity elements : Fe and Cr

the stainless steel balls **are not suitable** for milling the tungsten powders.

Microwave sintering

Schematic diagram of the microwave furnace



Main parameters of microwave furnace

sintering space: Φ150 × 160 (mm)

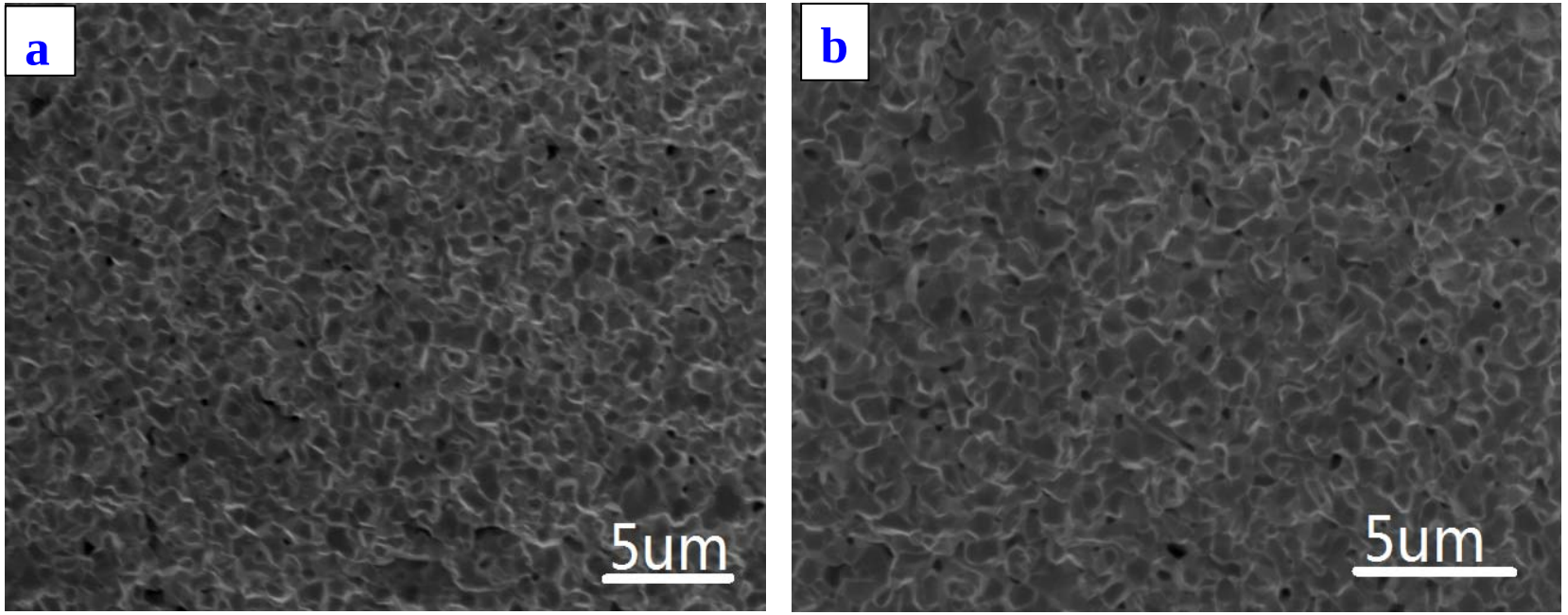
maximum Temperature: 1600℃

Microwave frequency: 2.450GHz

reducing atmosphere (5%H_2 and 95%Ar).

3. Experimental results

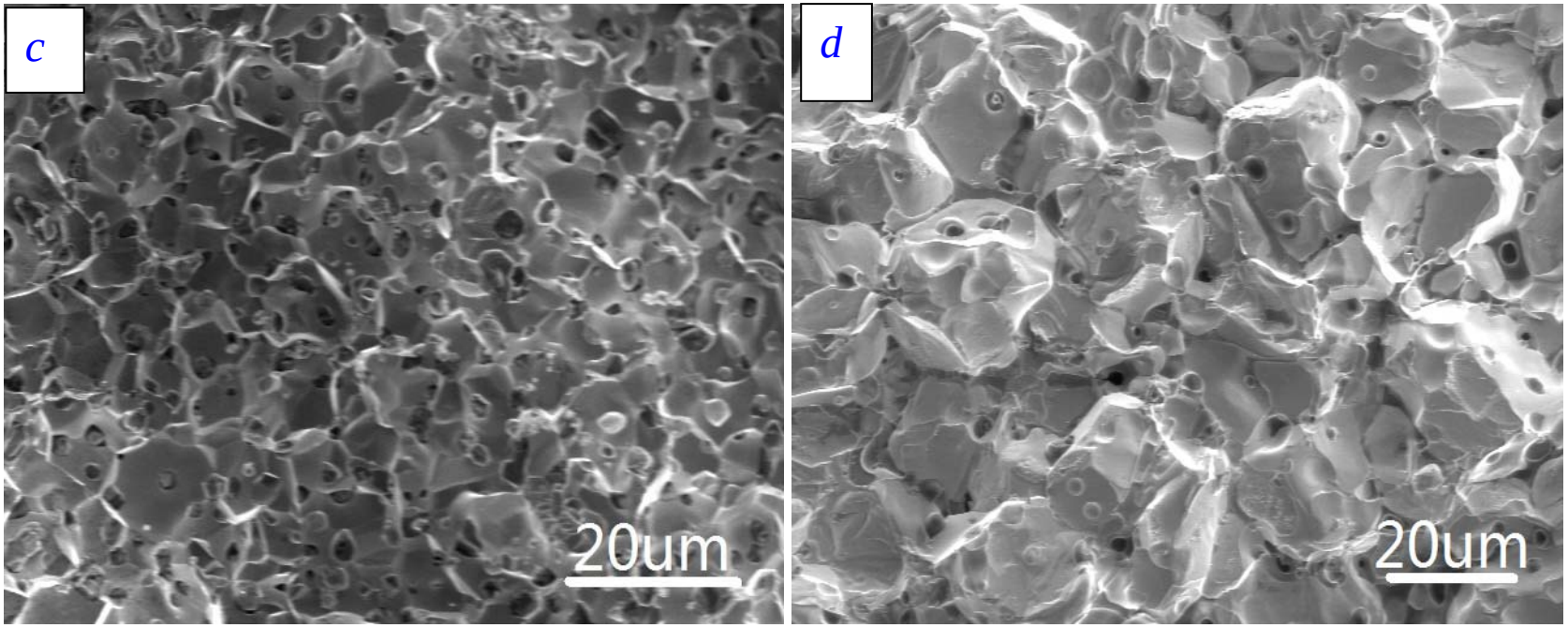
SEM images of fractured surface of the sintered W samples(Carbide ball):
a few small pores are seen



- **(a) nano powder sample sintered at 1500C (30min): 1 μm; density: 95%**
- **(b) coarse powder sample sintered at 1500 K (30min):1~2 μm; density: 93.5%**

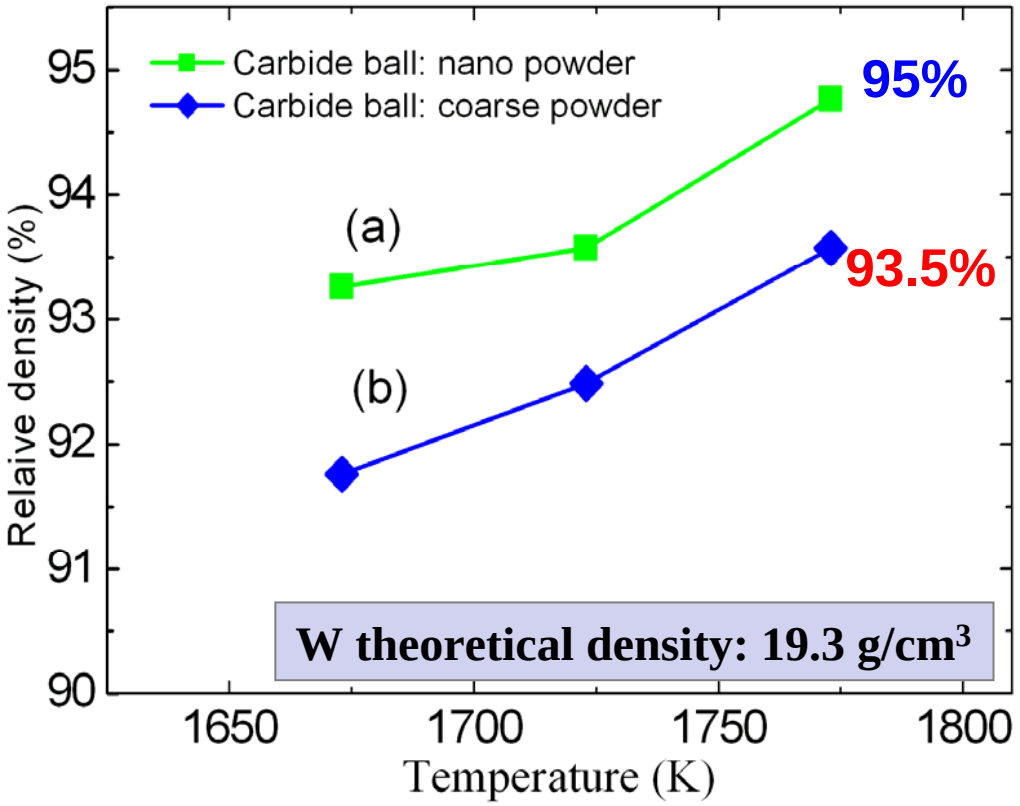
Mircowave sintering is suitable method to fabricate high density bulk W with fine grain size.

SEM images of fractured surface of the sintered W samples (Stainless steel ball): dark point: impurity



- **(c) coarse powder sample sinteredat 1400C (30min):**
- **(d) coarse powder sample sintered at 1500 C (30min)**

few pores are observed: the high density; grain rapildly grows :20 μ m; Reason: the introduction of low melting point metals promote the densification process.

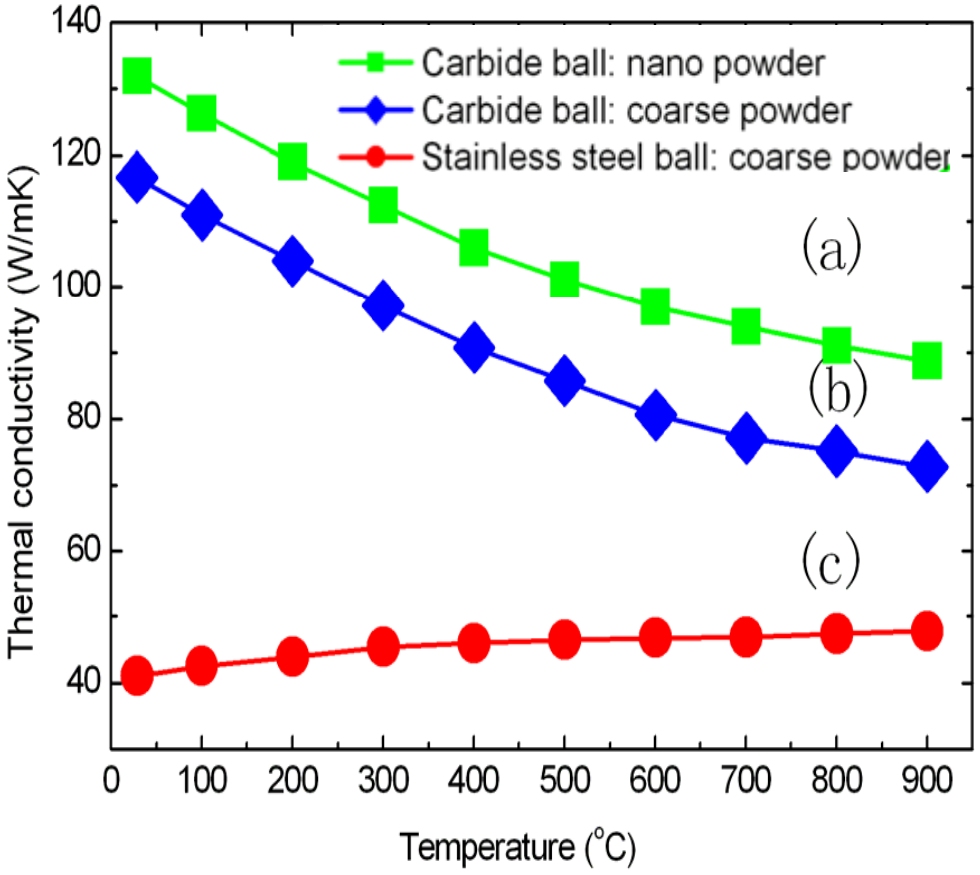


Temperature dependence of sintering density of bulk W

(1)W-density increases with elevation of sintering temperature(maximum: 95%);

(2) high density shows higher sintering activity of nanometer powders;

Influence of preparation routes on thermal conductivity of bulk W



(1) nano powder sample shows the highest thermal conductivity among the three samples (max: ~140 W/mK);

(2) The introduction of impurities results in lower thermal conductivity

4. Conclusion

◆**High-density bulk tungsten with fine grain size can be fabricated by microwave sintering at low temperatures (1400-1500C) with a short soaking time (30min);**

◆ **Nano-Tungsten powder shows higher sintering activity compared with coarse powder;**

◆**The highest density and thermal conductivity can reach to 95% and 140 W/mK, respectively.**

Acknowledgement

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Thank you!