Powder Technology 310 (2017) 264–271

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Spark plasma sintering of pure tungsten powder: Densification kinetics and grain growth

Shenghua Deng, Tiechui Yuan *, Ruidi Li *, Fanhao Zeng, Guanghong Liu, Xiang Zhou

State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, PR China

ARTICLE INFO

Article history:
Received 21 November 2016
Received in revised form 11 January 2017
Accepted 21 January 2017
Available online 24 January 2017

Keywords:
Sintering
Spark plasma sintering
Tungsten
Densification mechanism
Grain growth

ABSTRACT

The densification kinetics and grain growth behavior of an undoped tungsten powder during spark plasma sintering (SPS) were investigated under the pressure of 40 MPa and constant heating rate of 100 °C min^{-1}. Two stages of the sintering process were clearly identified: densification without grain growth at the low temperatures (1200–1450 °C) and grain growth without much further densification at higher temperatures (1500–2000 °C). A creep model was applied to determine the densification mechanisms involved in the densification stage, which can be elucidated by evaluating the stress exponent (n) and the apparent activation energy (Q_d) from the densification rate law. It shows that a boundary diffusion governs the densification process at low effective compaction stresses ($n = 1.5$, $Q_d = 140.57 \pm 12$ kJ mol^{-1}), while grain boundary diffusion and dislocation motion both operate at higher effective compaction stresses ($n = 3$, $Q_d = 302.48 \pm 24$ kJ mol^{-1}), which is confirmed by transmission electron microscopy observation. During the final-stage of sintering, the fast grain growth mechanism was suggested as surface diffusion.

© 2017 Published by Elsevier B.V.

1. Introduction

Owing to its high melting point, excellent thermal conductivity, low thermal expansion and superior mechanical properties at high temperatures, tungsten is suitable for many engineering applications such as lighting filaments, heating source, electronic devices, military and aerospace use, etc. Traditionally, tungsten powder is processed by powder metallurgy route [1] which requires high sintering temperatures and long soaking times, and produces coarse microstructure often. Thus, more efficient sintering techniques have been pursued for consolidating tungsten.

Spark plasma sintering (SPS) is an advanced sintering method which can provide rapid densification of sintered powder. SPS closely resembles hot-pressing (HP) technology, but differs in the heating source. Unlike HP, a pulsed direct current is applied through the die and thus acts as the heating source by the Joule effect in SPS process. Owing to its unique heating mode, SPS offers a shorter soak time, higher heating rate and lower sintering temperature compared with traditional sintering technique [2]. Therefore, SPS is quite suitable for producing of various difficult-to-cult materials and refractory metal, especially tungsten with high density and fine grained microstructures.

In previous works, the sintering behavior of tungsten powder has been studied using SPS to achieve densification. Autissie [3] obtained pure tungsten sample with a relative density of 95.2% under the condition of 1900 °C for 20 min and 100 MPa by SPS. Atwani [4] studied the sinter-ability of tungsten powder at different temperatures, pressures and annealing times by SPS. A fine grain (4.97 μm) tungsten sample was acquired at 1500 °C for 5 min under the pressure of 200 MPa. The above researches, which confirmed that high density tungsten with a fine structure can be obtained at low sintering temperature using SPS, have not yet investigate on the rate controlling mechanisms of densification. Deep comprehension of the control mechanisms during SPS densification, which could provide theoretical guidance for microstructural controlling and process optimization, is considered to be very important. Although there have been many studies that examined densification mechanisms of tungsten powder, most of them focused on pressureless sintering [5–9]. Little attention has been paid to the densification mechanisms of the pressure-assisted sintering, especially SPS. On the densification kinetics, Karpinos [10] investigated the densification behaviors of tungsten during HP by a diffusion model, suggesting that densification is controlled chiefly by plastic deformation in the density range 0.6–0.8, while diffusional creep becomes the dominant factor at higher densities. Recently, Gao [11] studied the densification mechanisms of tungsten during SPS by two spheres shrinkage model, indicating that grain boundary diffusion accounted for the dominant densification mechanism by estimating the activation energy of 277 ± 15 kJ mol^{-1} (1250–1500 °C). However, in above studies, the densification behaviors of HP and SPS are based on pressureless sintering analytical model, which neglected the efficacy of pressure. In fact, the pressure plays an important role in the densification of HP and SPS. It is generally recognized that the

* Corresponding authors.
E-mail addresses: tiechuiyuan@csu.edu.cn (T. Yuan), liruidi@csu.edu.cn (R. Li).

http://dx.doi.org/10.1016/j.powtec.2017.01.050
0032-5910/© 2017 Published by Elsevier B.V.

application of external pressure increases the driving force for densification in pressure-assisted sintering [12]. Thus, the study of densification law of tungsten powder during SPS considering the pressure effect is regarded as necessary.

In the present paper, the tungsten powders were consolidated by SPS under temperature 1100–2000 °C, soaking time 2–10 min and fixed pressure 40 MPa for the purpose of studying the densification kinetics and grain growth. To formulate hypotheses about the mechanisms controlling the densification of tungsten powder involved in SPS, we consider pressure as one of the main driving force of densification during pressure-assisted sintering. Hence, a new analytical model suggested by Bernard-Granger and Guizard [13,14] was applied.

2. Experimental procedure

2.1. Materials and processing

A pure commercial tungsten powder (purity >99.9%) with an average particle size of 1 μm (FSSS) was used as the raw powder in the present study. The scanning electron microscopy (SEM) morphology of tungsten powder is shown in Fig. 1.

The raw powder was placed in a 20 mm internal diameter graphite module which had been lined with thick graphitic sheet previously for easy removal. Then the module was loaded into the SPS apparatus (HP D25, FCT Systeme GmbH, Rauenstein, Germany). Axial pressure of 16 MPa was applied on the powder bed at the beginning and then increased to 40 MPa. After that, sintering step was performed in vacuum with a heating rate 100 °C min^{-1}. As soon as the desire temperature (1200–2000 °C) reached, the dwell times were chosen as: 2 min, 4 min, 6 min, 8 min and 10 min respectively. At the cooling step, the axial pressure was released to 16 MPa and maintained until room temperature. A typical sintering program was illustrated in Fig. 2.

For all the SPS experiments, temperature was measured with infrared radiation pyrometer focused on the top punch, which cannot detect temperature below 420 °C. The deviations of measured temperature and real temperature of powder compact were neglect. The experiment data, including temperature, pressure and displacement of piston, were recorded by system for each second. The variation of the height of the powder bed was determined from the piston displacement and then corrected by subtracting the thermal expansion of the graphite module and graphitic sheet ($\Delta h'$). To achieve this, a blank cycle was performed by submitting an empty graphite die to a complete heating cycle. Therefore, the instantaneous height (the height at a certain moment) of the powder bed (h) can be calculated, $h = (h_f + \Delta h_t - \Delta h') - \Delta h$, where h_f is the final height of the sintered compact, Δh_t the total height variation (the variation between initial height and final height, the initial height is the height of powder bed at 420 °C) and Δh the instantaneous

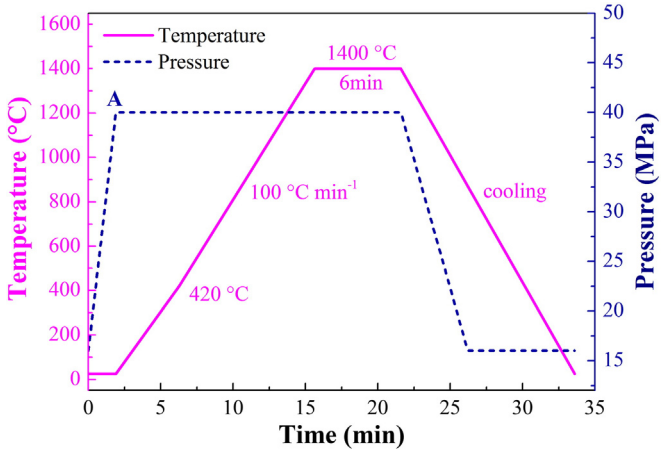

Fig. 2. Sintering curve of the samples sintered by spark plasma sintering at 1400 °C for 6 min.

height variation of the powder bed. Finally, the instantaneous relative density (D) can be calculated from the sample height variation, as follows:

$$D = \left(\frac{h_f}{h}\right) D_f \quad (1)$$

where D_f is the final relative density of sintered samples.

2.2. Material characterization

The density of sintered specimens was obtained via Archimedes method after sanding off the graphitic surface and carbide layer. The final relative density (D_f) was determined as the ratio between the final density and the theoretical density of tungsten (19.25 g/cm^3). Microstructural observations of the raw powder and sintered samples were achieved using SEM (JSM-6360LV, Jeol Ltd., Tokyo, Japan). For each sample, the average grain size was determined by statistical method, which considering at least 150 grains from SEM images of fracture surface. Transmission electron microscopy (TEM, JEM-2100F, Jeol Ltd., Tokyo, Japan) was carried out to study the possible mechanisms involved in the densification of the obtained samples. The thin foil samples for TEM, with a diameter of 3 mm, were grounded by sandpaper and thinned down to perforation by twin-Jet Electropolisher (MTP-1A, SHANGHAI JIAODA Inc., Shanghai, China).

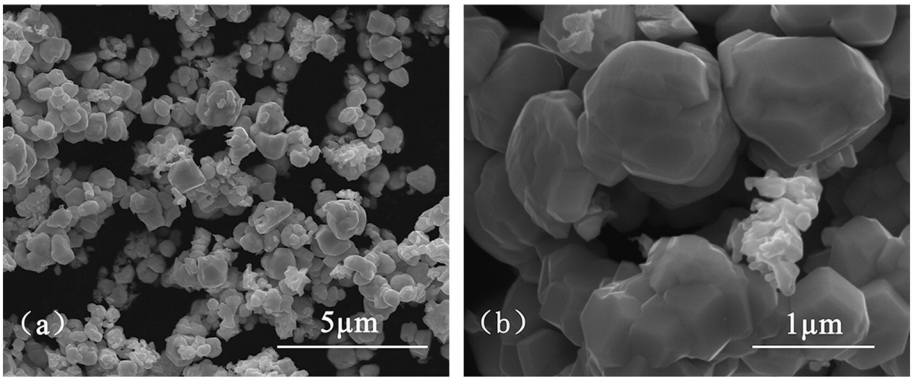

Fig. 1. SEM images of raw powder at (a) low and (b) high magnifications.

Download English Version:

https://daneshyari.com/en/article/4915045

Download Persian Version:

https://daneshyari.com/article/4915045

Daneshyari.com