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Kinetic characteristics of liquid phase sintering of mechanically activated W-15wt%Cu powder

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Abstract: The kinetic characteristics of W grain growth operated by diffusion controlled Oswald ripening (DOR) during liquid phase sintering were studied. A liquid phase sintering of W-15wt%Cu was carried out by pushing compacts into a furnace at the moment when the temperature increased to 1340°C for different sintering times. The results show that liquid phase sintering produces the compacts with considerably low relative density and inversely, rather high homogeneity. On the basis of the data extracted from the SEM images, the kinetic equation of W grain growth, $G^n = G_0^n + kt$, is determined in which the grain growth exponent n is 3 and the grain growth rate constant k is 0.15 $\mu m^3/s$. The cumulative normalized grain size distributions produced by different sintering times show self-similar. The cumulative distribution function is extracted from the curves by non-linear fitting. In addition, the sintering kinetic characteristics of W-15wt%Cu compacts were also investigated.

Key words: liquid phase sintering; mechanical alloying; grain growth; kinetics; W-Cu alloy

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1. Introduction

Liquid phase sintering is a net-shape fabrication technique used to consolidate ceramic and metal powders. The formation of liquid during heating enhances the diffusing and bonding process, leading to increasing sintering rates [1-2]. For the W-Cu system, Cu plays the most important role in liquid phase sintering. The formation of Cu pool throughout the structure causes a more intensive rearrangement of W particles [3] and furthermore, promotes the densification of W-Cu compacts. This phenomenon also called the second shrinkage has been observed by many researchers [4-5] by means of dilatometer. Cu liquid, however, gives a possibility that W atoms dissolve into Cu pool so that a remarkable W grain growth occurs through the diffusion controlled Oswald ripening (DOR), especially, for W-Cu composite powders with extremely fine W grains produced by the mechano-chemical process or mechanical alloying. This surprising phenomenon has been confirmed by Yu *et al*. [6] and they also indicate that the further investigation of DOR growth in W-Cu system should be conducted in terms of kinetic aspect.

Of late, Kim and Moon [7] have reported on the drastic W grain growth during solid phase sintering of W-20wt%Cu and W-30wt%Cu composite powders prepared by mechanical alloying. The W grain size increases from 20 to 30 nm (in as-milled powder at room temperature) to 200 nm at 1000°C. According to the authors [7], the possible reason of W grain growth during solid phase sintering may be the recovery and recrystallization of as-deformed W particles during the heat-up stage. However, they also notice that the W grain size hardly increases above 1100°C. Thus, for the purpose of investigation of W grain growth during liquid phase sintering only, the influence of solid phase sintering on W grain growth should be prevented to the utmost and whether the W grain size increases during liquid phase sintering should be examined closely. In this regard, one approach was used in the experiment below that the compacts were pushed into the furnace cavity at the moment when the temperature increased to a target value.

From the viewpoint of the conventional liquid phase sintering theory, the process of liquid sintering consists of three stages, which often overlap each other: (1) liquid flow and particle rearrangement; (2) solid dissolution and reprecipitation; and (3) solid state sintering and microstructure coarsening [8]. In most cases, the sintering kinetics of an alloy system has its own

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characteristics. Jia *et al*. [9] has studied the sintering kinetics of the Mo-Cu system and found that the process of liquid phase sintering had only two stages. The first stage corresponds to the second stage in the traditional theory and the second stage corresponds to the third stage. The characteristics of sintering kinetics should also be described by studying the shrinkage behavior of the compacts in the W-Cu system.

In this article, the kinetic characteristics of W grain growth during near-pure liquid phase sintering of mechanically activated W-15wt%Cu powder were investigated and the reason why the sintered compacts have homogeneous microstructure and low densities was explained. Besides, the characteristics of sintering kinetics were reported.

2. Experimental procedure

Elemental W (99.5%, 4-6 μm) and Cu (99.9%, 75 μm) powders, both from Beijing Non-ferrous Metal Research Institute, China, were used as raw materials. For the purpose of research only, the mass ratio of W powders to Cu powders is 85:15. The mechanical activation of mixed powders was carried out in a high-energy ball mill (vibratory type) under the atmosphere of argon at room temperature for 28 h. The milling vessels and the balls were made of stainless steel and quenched steel, respectively. The milling media-to-powder mass ratio is 20:1. The milling environment was dry milling and the filling factor was 0.75.

Single action pressing was used to make the milled powder into a cylindrical shape compact under a pressure of 300 MPa and the holding time is 5 s. The sample size obtained is ϕ8 mm×5 mm. The green density of compacts is about 48% of the theoretical density. To determine the best temperature of sintering, some compacts were sintered in a resistive furnace (molybdenum wire type) for 10 min under the reducing atmosphere of hydrogen gas at 1330, 1340, 1350, and 1370°C, respectively. The heating rate was 10°C /min.

For the purpose of building the experimental kinetic equation of liquid phase sintering, other compacts were sintered in a continuous type furnace under the protective atmosphere of hydrogen at 1340°C for 5, 10, 20, 30, 40, 50, and 60 min, respectively. The compacts were pushed into the furnace cavity at the moment when the temperature increased up to 1340°C.

The green and sintered densities were measured by Archimedes' displacement method. The theoretical density of the W-15wt%Cu alloy was calculated according to the formula below:

$$\rho_{\text{W-15wt\%Cu}} = \frac{\rho_{\text{W}}\rho_{\text{Cu}}}{\rho_{\text{W}}w_{\text{Cu}} + \rho_{\text{Cu}}w_{\text{W}}} = \frac{19.30\times 8.96}{19.30\times 0.15 + 8.96\times 0.85} = 16.45\ \text{g}\cdot\ \text{cm}^{-3} \quad (1)$$

Scanning electron microscopy (SEM) was used to observe the microstructure of the sintered body. The mean size of W grains was obtained by two methods for the purpose of accuracy and comparison as well. One is the manual measurement according to the SEM images imported into the program of Image Tool on a computer. All subsequent computations were based on the manual measurement data. The other is calculated according to the formulae below:

$$D_{\text{grain}} = \xi_{\text{grain}}\sqrt{\frac{\rho_{\text{relative}}}{n}} \quad (2)$$

$$\xi_{\text{grain}} = 2\sqrt{\frac{f_{\text{volume}}\times l\times h}{\pi}} \quad (3)$$

where f_{volume}, l, and h are the solid volume fraction of W, length, and width of SEM view field, respectively; ξ_{grain}, ρ_{relative}, and n are the calculating parameter of W grains, relative density of the sintered compact to be measured and the number of W grains counted by Image Tool, respectively. For the W-15wt%Cu alloy, f_{volume} is 0.7246, and for the present study, ξ_{grain} calculated is 19.92.

3. Results and discussion

3.1. Densities of W-15%Cu sintered at different temperatures

Fig. 1 shows the relative densities of the W-15wt%Cu alloy sintered for 10 min at 1330, 1340, 1350, and 1370°C, respectively.

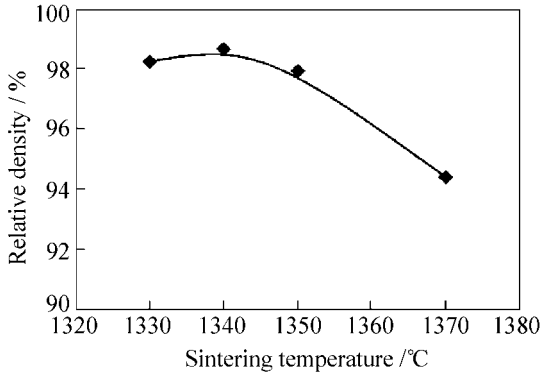

Fig. 1. Relative densities of W-15wt%Cu alloy at different temperatures.

From Fig. 1, little difference between relative densities can be observed within 1330-1350°C. All the sintered bodies within this temperature range have a relative density of more than 97%. The relative density of the sintered body reaches the maximum value of 98.66% at 1340°C. The decrease in relative density is also shown in the curve with continuous increase of

sintering temperature up to 1350°C. Therefore, the optimal sintering temperature of the W-15wt%Cu compact is determined to be 1340°C.

According to the theory of sintering mechanism, the possible reason why the relative density decreases with continuous increase of sintering temperature can be explained. Before the escape of liquid phase Cu from interconnected pores because of its viscose flow and capillary force, solid phase sintering has taken place among W particles with high activation when the sintering temperature reaches a certain value. Meanwhile, certain amount of closed pores are formed. These closed pores cannot be eliminated by volume diffusion and surface diffusion during the subsequent solid phase sintering stage so that the densification degree of the sintered body decreases [16].

3.2. Heating rate, homogeneity, and density

Of late, Kim *et al*. [10] have reported the effect of heating rate on the microstructural homogeneity of sintered W-15wt%Cu alloys. The experimental results show that the homogeneity index of sintered compacts with the heating rate of 20°C/min is 9.68, lower than that of the sintered compacts with the heating rate of 5°C /min, 13.52, and meanwhile, the relative densities of the sintered compacts show almost the same value in about 97%. This means that the sintered compacts with a high heating rate have more homogeneous microstructure than those with a low heating rate and within a certain range of heating rates, high relative densities can be reached. The possible reasons are explained on the basis of the mechanisms of solid phase sintering. By milling the mixture of W and Cu powders, W particles are embedded in Cu phase [3] and furthermore, the agglomerates of W-Cu particles are formed. The agglomerated composite powders experience a densification process mainly through the diffusion of Cu phase, which can be extruded to the surfaces of agglomerates during the heat-up stage [10] from room temperature to the Cu melting point (1083°C). Kim *et al*. [11] and Ryu *et al*. [4] also pointed out that the segregation of Cu phase was a dominant aspect of the microstructure evolution during the solid phase sintering. The segregated Cu phase can lead to the formation of larger Cu pool during the liquid phase sintering and eventually deplete the homogeneity of the final microstructure. According to the experiment by Kim *et al*. [10], however, the segregation of Cu phase can be effectively inhibited because of the relatively short time for the diffusion of Cu, if compacts are sintered at a high heating rate.

A liquid phase sintering was simulated by pushing compacts into the furnace cavity at the moment when the temperature increased to 1340°C. By this method, the effect of heating rate on the microstructural homogeneity of sintered compacts can be neglected because the solid phase sintering is jumped. Fig. 2 shows the comparison in microstructure between the compacts sintered at 1340°C for 10 min, with solid phase sintering and without solid phase sintering (Figs. 2(a) and 2(b)), respectively. Obviously, the homogeneity of the sintered compact without heat-up stage is higher than that with heat-up stage. This is in a good agreement with the experiment carried out by Kim *et al*. [10]. However, the relative density of the sintered compact without heat-up stage is considerably lower than that with heat-up stage. The former is 98.66% and the latter is 81.18% as shown in Table 1. Other relative densities with a value less than 87% can also be evident in Table 1.

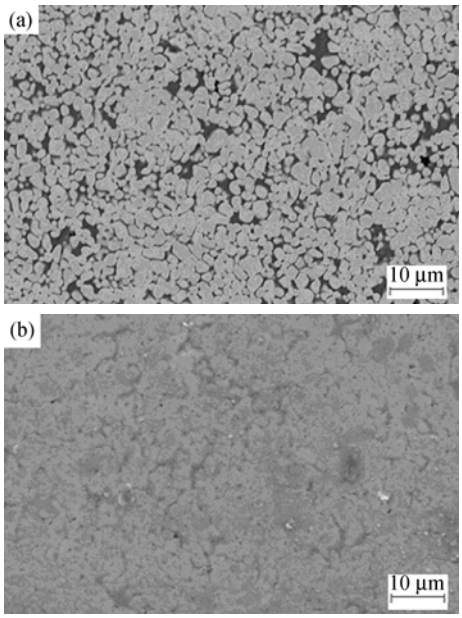

Fig. 2. Microstructures of W-15wt%Cu compacts sintered at 1340°C for 10 min with the heat-up stage (a) and without the heat-up stage (b).

Kim *et al*. [11] have described the densification behavior of W-20wt%Cu and W-30wt%Cu composite powders prepared by mechanical alloying during the conventional liquid phase sintering. A double (two-stage) rearrangement theoretical model is elucidated that the first rearrangement happens within 1000-1083°C and the second rearrangement takes place above 1100°C. Furthermore, they imagine that the first rearrangement (in the solid phase sintering stage) and the second rearrangement (in the liquid phase sintering stage) contributes to the densification process and microstructural homogeneity of sintered compacts, respectively. Ryu *et al*. [4] have researched the shrinkage behavior of a W-30wt%Cu compact by a dilatometer. According to the authors [11], two shrinkage stages

appear during solid phase sintering, first stage with the shrinkage of about 5% between 200 and 600°C, the second stage at 850°C, and around 85% of total shrinkage occurs through solid phase sintering. They also indicate that solid phase sintering as well as liquid phase sintering plays an important role in the sintering mechanism of nanocomposite W-Cu powders, and the dominant mechanism is solid phase sintering.

Table 1. Relative densities and the mean sizes of W grains in W-15wt%Cu alloys sintered for different holding times at 1340°C

W-15wt%Cu alloys	Relative density / %	*n*	Mean size of W grains / μm	
			By formulae	Manual measurement
$A_{5\ min}$	78.01	603	0.72	0.73
$A_{10\ min}$	81.18	264	1.10	1.10
$A_{20\ min}$	83.03	235	1.18	1.13
$A_{30\ min}$	83.52	232	1.20	1.25
$A_{40\ min}$	85.52	219	1.24	1.45
$A_{50\ min}$	86.18	201	1.30	1.52
$A_{60\ min}$	86.60	181	1.38	1.67

Note: *n* is the number of W grains. The relative density of the compacts is 50%±1%, and the mean size of W grains is 20-40 nm.

On the basis of the experimental results and the discussion above, two quantitative relationships can be concluded. One is the relationship between density (*D*) and heating rate (*h*) and the other is the relationship between homogeneity (*H*) and heating rate. Considering that the sintering process is gradually transformed into solid phase sintering as the heating rate approaches 0°C/min and in turn, the sintering process is gradually transformed into liquid phase sintering as the heating rate approaches +∞, and in addition, the homogeneity increases with the increase of time that the liquid phase sintering accounts for the total sintering process. The density increases with the increase of time that the solid phase sintering accounts for the total sintering process, the following quantitative relationships may be implied:

$$f: h \rightarrow D \tag{4}$$

$$f: h \rightarrow H \tag{5}$$

Hence, for practical application and a particular sintering process, the value of heating rate should be well determined in advance.

3.3. Grain growth during liquid phase sintering

Fig. 3 shows the SEM images of W-15wt%Cu compacts sintered at 1340°C for various holding times. Two phases can be observed from Fig. 3. The round gray grains with the size of about 1 μm and the irregular black areas are composed of W and Cu, respectively. W grains connect with each other through sintering neck and in some area little agglomeration of W grains can still be observed (Fig. 3(b)). According to the images shown in Fig. 3, the mean size of W grains with different holding times is measured, which is shown in Table 1. The significant grain growth of W can be evident from Table 1, from the initial size of 20-40 nm to 1.67 μm with the increasing sintering time.

Yu *et al*. [6] have researched the grain growth mechanism for the W-20wt%Cu and W-25wt%Cu alloy sintered at 1350°C. The results show that the diffusion controlled Ostwald ripening (DOR) contributes to the W grain growth during liquid phase sintering. They also indicate that the DOR growth is basically because of the solution-reprecipitation process. Apart from the intrinsic solubility effect (the solubility of W in Cu melt is 10^{-5}at% and that is enough to initiate the DOR growth [12]), an excess local dissolution of nanosized W grains in Cu melts because of the capillary effect may additionally favor the DOR growth. Meanwhile, they think that this explanation needs to be examined closely by further investigation on DOR growth in terms of kinetics.

For the solution-reprecipitation process, the isothermal grain growth kinetic equation relates the mean grain size to time as follows [1]:

$$G^n = G_0^n + kt \tag{6}$$

where *G* is the mean grain size after the sintering time *t*; G_0 the initial grain size; *k* the grain growth rate constant; *n* and *t* are the grain growth exponent and the sintering time, respectively. For the diffusion controlled grain growth, *n*=3 [2].

For the W-Cu powders prepared by mechanical alloying, the crystalline size is extremely fine. Thus, the component G_0^n can be ignored and Eq. (6) can be simplified as

$$G^n = kt \tag{7}$$

then

$$\ln G = k' + \frac{1}{n}\ln t \tag{8}$$

where $k' = \frac{1}{n}\ln k$. On the basis of the data in Table 1, the plot of the natural logarithm of sintering time *versus* that of the grain size is given in Fig. 4. It is easily extracted from the linear fit of the plot that $n = 3$ and $k' = -2.2$. Hence, $k = 0.15\ \mu m^3/s$. The result gives an argument that the DOR operates the W grain growth during liquid phase sintering of W-15wt%Cu prepared by mechanical alloying and is in good agreement with the assumption by Eremenko *et al*. [12] and Prokushev *et al*. [13] in terms of kinetics.

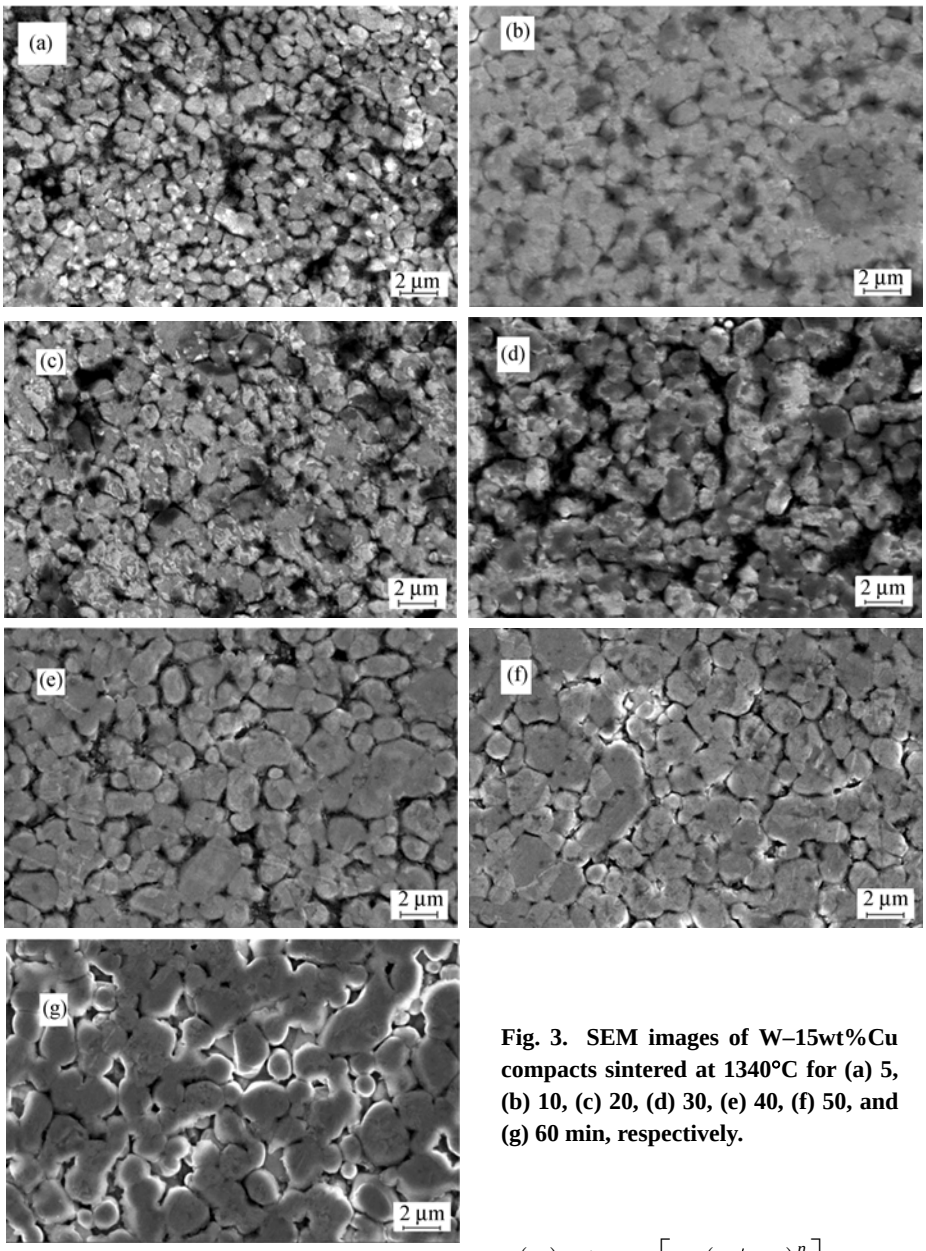

Fig. 3. SEM images of W–15wt%Cu compacts sintered at 1340°C for (a) 5, (b) 10, (c) 20, (d) 30, (e) 40, (f) 50, and (g) 60 min, respectively.

According to the previous studies on diffusion controlled coarsening [14], the grain size distribution appears to be self-similar after the grain growth evolves from the initial state to the final steady state. The steady state is assumed to be achieved when the mean grain size becomes larger by 5 times than the initial [15]. By analyzing the data from Table 1, it can be concluded that the W grain growth is in steady state when the isothermal sintering time is longer than 5 min. For multiple-phase systems in which the DOR mechanism operates, the cumulative intercept grain size distribution $F(G)$ can be described by

$$F(G) = 1 - \exp\left[-a\left(G/G_{\mathrm{M}}\right)^n\right] \tag{9}$$

where a and n are constants; G is the actual grain size and G_{M} is the mean grain size.

Fig. 5 shows the cumulative intercept grain size distribution for W grain contained in W-15wt%Cu sintered compacts. Since W grains growth proceeds in steady state when sintering for 5 min, the curve of cumulative frequency against normalized grain size (the ratio of the actual grain size G to the mean grain size G_{M}), which is obtained from Eq. (9) by non-linear fitting on the basis of the data from sintering for 5 min,

should be consistent with the scattered dots plotted by the data from other sintering time. As can be evident from Fig. 5, this assumption is clearly confirmed and the cumulative plots give an idea of the similarity of the distributions, *i.e.* the scattered dots fall on the same cumulative curve. From the non-linear fitting curve, the *a* and *n* values in Eq. (9) can be extracted, they are 1.44 and 2.0, respectively.

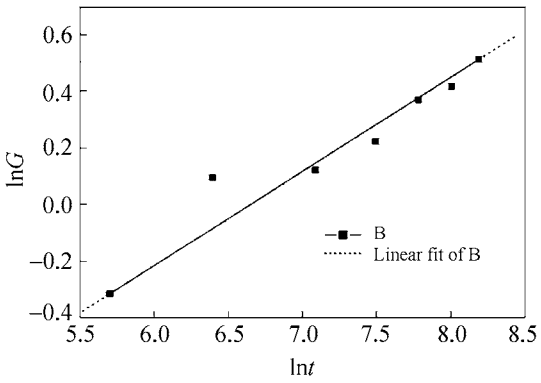

Fig. 4. Natural logarithm of W grain size plotted as a function of that of sintering time for W-15wt%Cu alloys sintered at 1340°C. B stands for the experimental curve.

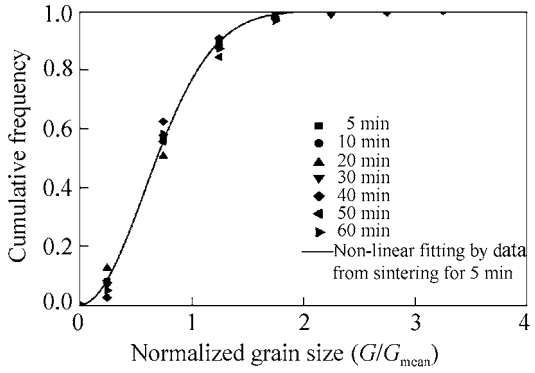

Fig. 5. Normalized two-dimensional grain intercept distribution for W grains contained in W-15wt%Cu compacts sintered at 1340°C for various keeping times shows self-similar distribution.

3.4. Characteristics of sintering kinetics at an optimal sintering temperature

In general, the process of liquid phase sintering consists of three stages, which often overlap each other. The first stage is the formation of liquid phase and the rearrangement of particles. Solid particles can flow because of the effect of surface capillary force of liquid, if there is no link among particles. Consequently, the particle rearrangement takes place and the sintering compact shrinks accordingly. This is the process of densification and its speed is considerately high. The second stage is called solution and precipitation. The coarsening of microstructure is regarded as the general characteristic of this stage. During this stage, fine particles and burrs on the surface of coarse particles dissolve into liquid phase. At the same time, on the surface of large particles, fine grains precipitate. Thus, large particles become larger. Meanwhile, the shape of particles turns out to be spherical. This process leads to shrinkage and densification of the sintering compact. Comparing with the first stage, the speed of densification becomes obviously low. The last stage of liquid phase sintering is termed as the formation of solid skeleton or solid bounding. In this stage, particles contact with each other and solid-solid bounding occurs by diffusion. With the formation of solid skeleton, liquid phase begins to infiltrate into the pores contained in the solid skeleton. The densification process slows down because of the stiffness of the solid skeleton. It is generally considered that a process similar to solid phase sintering possesses the dominant position in the third stage of liquid phase sintering.

The densification of liquid phase sintering can be quantitatively described by a densification parameter as below:

$$\alpha = \frac{\rho_s - \rho_g}{\rho_t - \rho_g} \times 100\% \tag{10}$$

where ρ_s is the sintering density, ρ_t the theoretical density, and ρ_g the green density [9].

On the basis of the data shown in Table 1, the curve of densification parameters against sintering time is plotted in Fig. 6. This curve demonstrates the shrinkage process of W-15wt%Cu compacts during the near-pure liquid phase sintering at 1340°C. It should be noticed that this experiment is carried out under the condition of taking out the solid phase sintering stage approximately and the isothermal holding time range from just 5 to 60 min. The initial stage of the near-pure liquid phase sintering from 0 to 5 min cannot be analyzed in this study. Hence, it can be observed from Fig. 6 that the densification process is performed with rather low speed and the three stages for liquid phase sintering can hardly be discerned. The result seems to imply that the initial stage of the near-pure liquid phase sintering from 0 to 5 min plays the most important role in the densification process. This supposition needs to be further checked in terms of kinetics.

According to the conventional liquid phase sintering theory, the first stage of shrinkage process can be described by the following equation:

$$\frac{\Delta L}{L_0} = \frac{1}{3}\frac{\Delta V}{V_0} = Kr^{-1} \cdot t^{1+x} = K' \cdot t^{1+x} \tag{11}$$

where $\frac{\Delta L}{L_0}$ is the linear shrinkage, $\frac{\Delta V}{V_0}$ is the volume shrinkage, x is a revising value for the geometric

shape of pores which is less than 1, r is the initial particle size, K and K' are the parameters related to temperature and interface energy, *etc*.

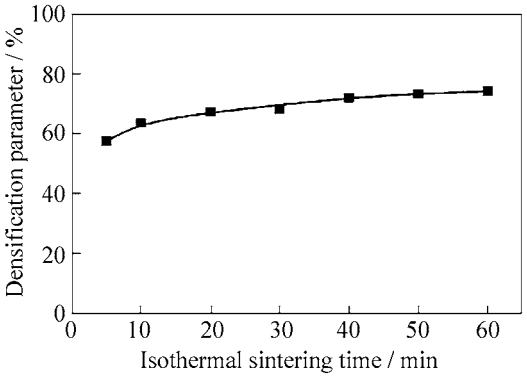

Fig. 6 Densification parameters of W-15wt%Cu compacts sintered at 1340°C for various sintering times.

And for the second stage, the shrinkage kinetic equation is expressed as

$$\frac{\Delta L}{L_0}=\frac{1}{3}\frac{\Delta V}{V_0}=K''\cdot t^{\frac{1}{3}} \tag{12}$$

where K'', the same as K and K', is a parameter. If Eqs. (11) and (12) are transformed into linear equations, for Eq. (11), the slope should be more than 1 and for Eq. (12), the slope equals 1/3. By this method, the first and second stages can be distinguished.

On the basis of the data in Fig. 6, the plot related the linear shrinkage to the sintering time is made, as can be observed in Fig. 7. The graph shows that only one stage can be found in this near-pure liquid phase sintering. The slope of the linear fit of B extracted from the plot is 0.06. This implies that the stage is neither the first nor the second and perhaps the first two stages have been finished during the sintering time from 0 to 5 min. This agrees with the analytical results obtained from Fig. 6.

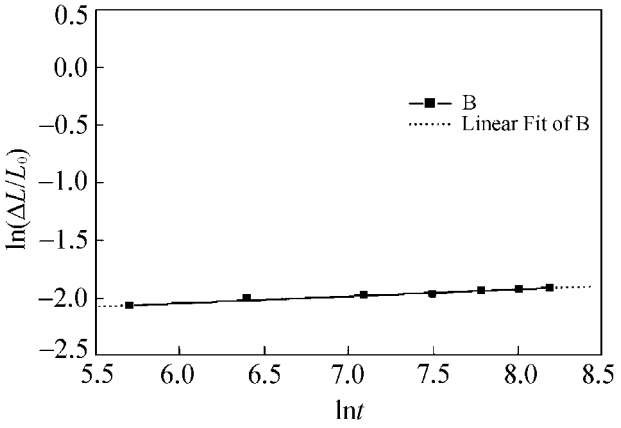

Fig. 7. Relationship between linear shrinkage and sintering time during the near-pure liquid phase sintering of W-15wt%Cu compacts. The slope of the linear fit of B extracted from the plot is 0.06. Solid B stands for the experimental curve.

4. Conclusions

The present study focuses on the kinetic characteristics of near-pure liquid phase sintering of the W-15wt%Cu powders prepared by mechanical alloying, including the kinetic characteristic of W grain growth and the shrinkage process of the sintered compacts. For purpose of research on liquid phase sintering only, a method of near-pure liquid phase sintering at 1340°C is used. By this method, sintered compacts with very high homogeneity but considerably low density are produced. This implies that liquid phase sintering contributes more to the homogeneity but little to the densification. During liquid phase sintering, W grains undergo a remarkable growth. The kinetic equation of W grain growth with $n=3$ and $k=0.15\ \mu m^3/s$ is determined. In addition, the cumulative intercept grain size distributions for W show self-similar regardless of the sintering time. The cumulative distribution equation with a=1.44 and n=2.0 is also solved. These results prove that the DOR operates W grain growth during liquid phase sintering.

By investigation on the shrinkage behavior of W−15wt%Cu sintered at 1340°C for the sintering time from 5 min to 60 min, the shrinkage kinetic process is described. The results show that only one liquid phase sintering stage is found under the current conditions. The slope of linear fit plot is much smaller than the theoretical value for the second stage as well as the first stage. Perhaps the first two stages have been finished during the sintering time from 0 min to 5 min. This explanation should be examined closely by further investigation on the initial stage (0-5 min or so) of liquid phase sintering also in terms of the kinetic aspect.

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