Densification Behavior and Microstructural Development of Nano-agglomerate Powder during Singering

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Abstract

Densification behavior of nano-agglomerate powder during pressureless sintering of Fe-Ni nanopowder was investigated in terms of diffusion kinetics and microstructural development. To understand the role of agglomerate boundary for sintering process, densification kinetics of Fe-Ni nano-agglomerate powder with different agglomerate size was investigated. It was found that activation energy for densification was lower in the small-sized agglomerate powder. The increase in the volume fraction of inter-agglomerate boundary acting as high diffusion path might be responsible for the enhanced diffusion process.

Keywords : nanopowder, Fe-8wt%Ni, diffusion, agglomerate boundary, full densification

1. Introduction

Nanostructured materials are of enormous interest in scientific and technological field due to their unique properties caused by the reduced dimensions of the grains [1,2]. Powder metallurgical process by using nano-sized powder is the most promising processing route for fabricating nanostructured materials [3]. Recently, we reported on a new processing route for fabrication full densified Fe-40wt%Ni material by pressureless sintering [4]. Sintering behavior showed that the initial pore size distribution of agglomerates of nanopowder played a decisive role for full densification process. According to the result of radio tracer diffusion investigation [5,6], bimodal type of boundaries in Fe-Ni nanopowder consisting of nano-sized grain boundary and agglomerate boundary act as high diffusion paths for densification process.

This study was undertaken to examine the role of agglomerate boundary in sintering kinetics and microstructure development. For this study, sintering behavior of Fe-8wt%Ni nanopowder was investigated with the same particle size of 100 nm, but with different agglomerate size of 5 μm and 500 μm. On the basis of this result, the full densification process during pressureless sintering of nanopowder should be understood in terms of characteristics and microstructure of agglomerated nanopowder.

2. Experimental and Results

The Fe-8wt%Ni nanopowder having an average grain size of 100 nm was prepared by hydrogen reduction of ball-milled oxide mixture of Fe_2O_3 and NiO [7]. The agglomerated Fe-8wt%Ni nanopowders were classified in agglomerate size (500 μm and 5 μm) by a repeated sieving method. However, both powder samples were equal in particle size (100 nm). Thus, classified nanopowders were virtually identical, only differing in the agglomerate size. To eliminate inter-agglomerate pores, both powders were equally compacted to 72% of theoretical density (T.D.) with compacting pressure of 1.2 GPa. A sintering experiment was performed using a laser-photo dilatometer in the course of heat up to 1223K at different heating rates of 5~20K/min. The linear shrinkage during sintering was measured and the apparent activation energy for densification process at each sintering stage was calculated semi-empirically [4].

Fig. 1 (a) and (b) show the densification process of compacts which have 500 μm and 5 μm of agglomerate size during heat-up sintering at various heating rates. From these results, the parameters required to calculate activation energy on the basis of Eq. 1 were obtained and plotted in Fig. 1 (c) and (d) [4]:

$$\ln\left(\frac{\Phi}{T^2}\right) = \ln\left(\frac{CR}{Y^n Q}\right) - \frac{Q}{RT} \quad (1)$$

where Φ is the ratio of the heating rate, T is temperature, R is gas constant, Y is the identical value of shrinkage and Q is activation energy for densification.

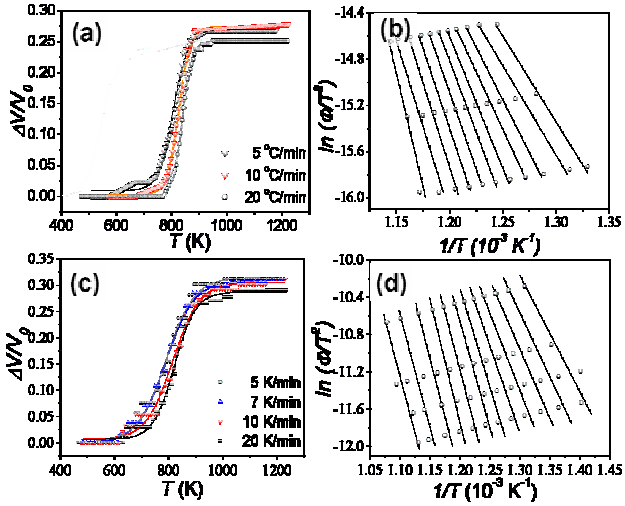

Fig. 1. Volume shrinkage as a function of various heating rates for Fe-Ni nanopowder with agglomerate size of (a) 500 μm and (c) 5 μm. Plots of the data according to Eq. (1) with agglomerate size of (b) 500 μm and (c) 5μm.

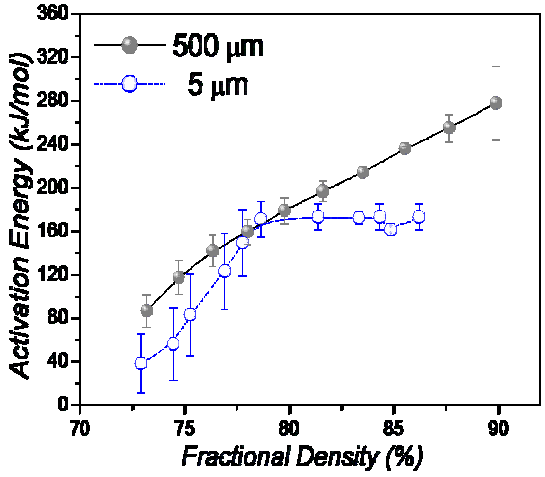

Fig. 2. Apparent activation energies for densification of agglomerated Fe-Ni nanopowder with different agglomerate size of 500 μm and 5 μm.

Fig. 2 compares the dependence of apparent activation energies for densification process on fractional sintered density, which is derived from the slops of the plots in Fig. 1(a) and (d). Apparent activation energies for the nanopowder compact with 500 μm of agglomerate size increased from 80 to 280 kJ/mol as densification proceeded from 72 to 90% T.D. On the other hand, in the case of 5 μm of agglomerate size, activation energy increased gradually from 40 to 170 kJ/mol with sintering from 72 % to about 80 % T.D. and thereafter retained 170 kJ/mol even above 80% T.D. Considering the agglomerate size in both powder samples, it is expected that the large size agglomerate sample (500 μm) requires higher activation energy for sintering of nanoparticles in the agglomerate compared to the small agglomerate sample (5 μm). This is because the sintering process takes place mainly and dominantly inside the agglomerates. Namely, densification and grain growth can occur simultaneously until the grain size reaches the agglomerate size. Therefore the large agglomerate sample showing a great change of activation energy (80 to 280 kJ/mol) is thought to undergo densification process by various diffusion processes from surface (60 kJ/mol), interface (180 kJ/mol) to volume diffusion (280 kJ/mol) [5,6].

This argument might be reasonable when the activation energy for densification process is compared with that for diffusion process. This implies that the sintering kinetics controlled by densification and grain growth inside the agglomerate can be explained in terms of all kinds of diffusion mechanisms. On the contrary, it seems that the sintering of the small agglomerate sample (5 μm) is enhanced owing to larger fraction of inter-agglomerate boundary. Consequently, the optimum sintering route for nanopowder is considered by reducing the agglomerate size, i. e., enlarging the agglomerate boundary volume, which provides faster diffusion path [6].

3. Summary

Densification behavior of Fe-8wt%Ni nanopowder with different agglomerate size of 5 μm and 500 μm was investigated in terms of diffusion kinetics and microstructural development. The apparent activation energy for densification was lower in small-sized agglomerate powder. The increase in the volume fraction of inter-agglomerate boundary acting as a faster diffusion path for sintering might be responsible for the enhanced diffusion process. As a consequence, the optimum sintering route for Fe-8wt%Ni nanopowder could be considered by reducing the agglomerate size.

4. References

1. H. Gleiter, Prog. Mater. Sci., **33**, 223(1989).
2. C. Suryanarayana, Int. Met. Rev., **40**, 41(1995).
3. J.R. Groza, Nanostr. Mater., **12**, 987(1999).
4. J.S. Lee and Y.S. Kang, Scripta Mater., **44**, 591(2001).
5. S.V. Divinski, F. Hisker, Y.S. Kang, J.S. Lee, Chr. Herzig, Z. Metallkd., **93**, 265(2002).
6. S.V. Divinski, F. Hisker, Y.S. Kang, J.S. Lee, Chr. Herzig, Interface Sci., **11**, 67(2003).
7. J.S. Lee, B.H. Cha and Y.S. Kang, Adv. Engin. Mater., **7[6]**, 467(2005).