

**CONSOLIDATION OF HIERARCHY-STRUCTURED
NANOPOWDER AGGLOMERATES AND ITS APPLICATION TO
NET-SHAPING NANOPOWDER MATERIALS**

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The potential application of nanoscale materials as novel structural or functional engineering materials largely depends on the consolidation of powders into bulk nanoscale solids. In general, pressure-assisted sintering is known to be adequate for both reaching full density and preventing grain growth [1]. Besides the fabrication of nanomaterials into engineering components by hot pressing or Materials 2013, 6 4047 sinter-forging, another promising objective is the application of nanopowders to the net-shaping technique as powder injection molding (PIM). In this case, the consolidation of the net-shaped powders is only achieved by pressureless sintering. It was reported that Fe base nanopowder compacts underwent the densification process strongly depending on the state of powder agglomeration [2]. The consolidation was incomplete due to a severe influence of particle agglomeration. Consequently, full densification of nanopowder compact even at elevated temperature could be achieved by optimizing the agglomerate size through either high-pressure compaction or reducing agglomerate size. Based upon these findings, Lee et al. [3–5] suggested a new sintering concept for consolidation of nanoscale powders, so called nanopowder agglomerate sintering (NAS) process. The key concept of the NAS process is based on the optimization of structure design and full density processing of nanopowder into nanostructured micro-components. The kinetics of this process is found to be controlled by material transport through hierarchical interface structures of nanopowder agglomerates [5–7]. From the studies of densification process [2,5–8] and diffusion process [9–11] in Fe–Ni nanomaterials, it has been reported that the hierarchy-structured grain boundaries consisting of nano grain boundary and agglomerate boundary act as high diffusion paths for densification process. In particular, it was found that atoms diffuse much faster along agglomerate boundaries compared to nano grain boundary. This experimental finding provides a breakthrough for processing full density nanopowders by optimal design of the agglomerate structure, i.e., hierarchical interfaces in nanopowders. Moreover, based on this processing concept, Lee and his coworkers [3–5] fabricated nanograin structured Fe base PIM parts with full density and superior mechanical property owing to grain refinement effect. This is a good example or the

application of NAS process to processing a net-shaped powder material.

Another application example of NAS process is recently reported for the fabrication of full density Fe nanomaterial [12]. In this paper we review our recent investigations on the consolidation of hierarchy-structured nanopowder agglomerates in terms of the densification process and microstructural development and also introduce on their application studies for processing a net-shaped Fe base nanopowder parts using PIM technology and pressureless sintering. In addition, current research on low-temperature PIM using hierarchy-structured micro-nano powder feedstock is briefly addressed. 2. Optimal Processing of Iron Base Nanopowder Agglomerates Figure 1 shows a schematic illustration of densification process by controlling of agglomerate size [13]. The agglomerate powder compact exhibits a bimodal pore distribution comprised of nanoscale intra-agglomerate pores and micron scale inter-agglomerate pores. While the nano-sized intra-agglomerate pores are easily eliminated in the course of sintering, the micro-sized inter-agglomerate pores remain and restrain densification (Figure 1a). After controlling of agglomerate size distribution, the compact part has homogeneous- or narrow pore size distribution (Figure 1b) and can reach full densification after sintering. The structural modification of the agglomerated nanopowders by controlling agglomerate size might be a key technology to obtain smaller and narrower pore size distribution. This chapter describes the full density processing of net-shaped Fe base nanopowder agglomerates by controlling agglomerate size in terms of densification process and microstructure. Materials 2013, 6

Figure 1. Schematic illustration of pore size distribution in agglomerated nanocrystalline powder (a) without and (b) with controlling of distribution of agglomerate size [13].

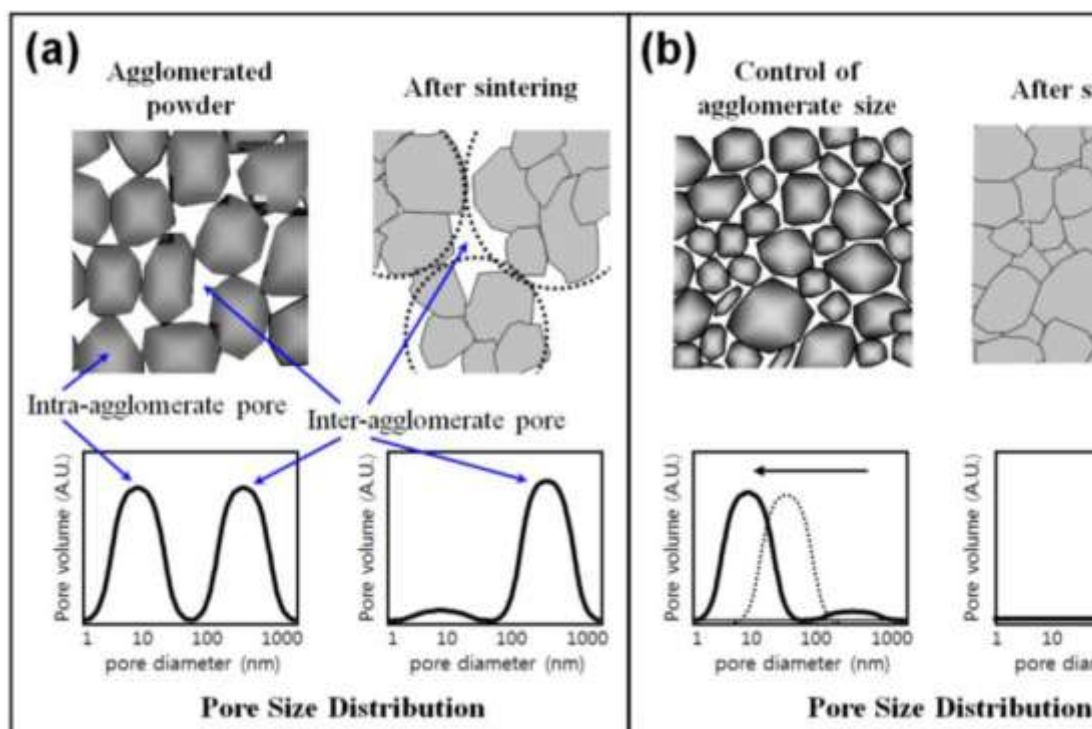
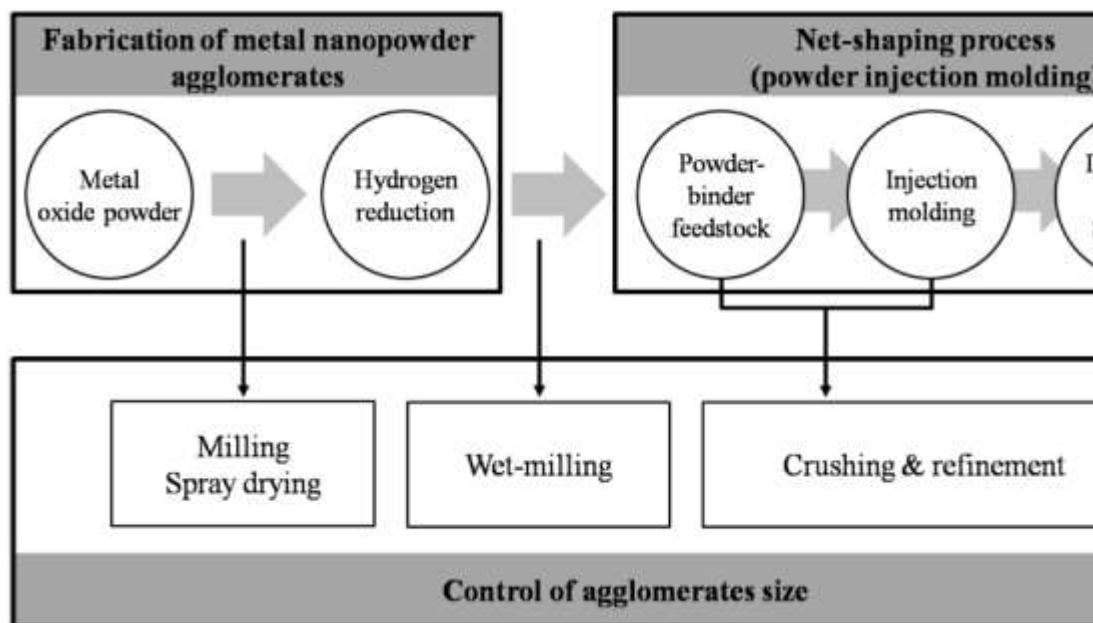


Figure 2 schematically represents the experimental procedure utilized for net-shaping process of Fe base nanopowder [3]. The process involves a two-step procedure consisting of Fe base nanopowder synthesis by hydrogen reduction of oxide precursors [14–17] and full densification process of PIM nanopowder by pressureless sintering. As depicted in the diagram, the large inter-agglomerate pores should be efficiently minimized by using different processing routes which can control the agglomerate size during the entire process. Intuitively, two methods for controlling agglomerate size and distribution are suggested; one is the wet milling of as-reduced nanopowder in dispersant containing solution [3,5,18] and the other is the mixing of nanopowder with the binder material for making PIM feedstock [3,5,18,19]. The wet-milling of nano-agglomerate powder in non-oxidation atmosphere can lead to increasing of packing density of agglomerate powder owing to improvement of particle size uniformity [20]. The maximum packing density for random packing of mono-sized spheres is predicted to be independent of the sphere size, and this prediction has been verified experimentally [21].

Figure 2. Experimental procedure for synthesis and net-shaping process of metal nanopowders produced by hydrogen reduction of ball-milled oxide powder [3].



REFERENCES

1. Suryanarayana, C. Nanocrystalline materials. *Int. Mater. Rev.* 1995, 40, 41–64.
2. Knorr, P.; Nam, J.G.; Lee, J.S. Sintering behavior of nanocrystalline γ -Ni-Fe powders. *Metall. Mater. Trans.* 2000, 31, 503–510.
3. Lee, J.S.; Cha, B.H.; Kang, Y.S. Processing of net-shaped nanocrystalline Fe–Ni material. *Adv. Eng. Mater.* 2005, 7, 467–473.
4. Cha, B.H.; Lee, J.S. Surface roughness and sintering characteristics of Fe–8 wt % Ni component fabricated by PIM. *J. Kor. Powder Metall. Inst.* 2009, 16, 342–350.
5. Lee, J.S.; Cha, B.H.; You, W.K. Full-density nanopowder agglomerate sintering of injection molded iron-nickel. *Int. J. Powder Metall.* 2010, 46, 27–34.
6. Lee, J.S.; Kang, H.G.; Kang, Y.S. The Importance of Agglomerate-Size Control in Full-Density Processing of Nanopowder by Pressureless Sintering. In *Proceedings of the World Congress on Powder Metallurgy & Particulate Materials 2008*, Washington, DC, USA, 8–12 June 2008.
7. Lee, J.S.; You, W.K.; Cha, B.H. Hierarchical structured nanomaterial fabricated by nanopowder agglomerate sintering. *Mater. Sci. Forum* 2010, 638–642, 93–97.
8. Lee, J.S.; Kang, Y.S. Processing of bulk nanostructure Ni-Fe materials. *Scr. Mater.* 2001, 44, 1591–1594.
9. Divinski, S.V.; Hisker, F.; Kang, Y.-S.; Lee, J.S.; Herzig, Chr. 59Fe grain boundary diffusion in nanostructured γ -Fe–Ni: Part I: Radiotracer experiments and

Monte-Carlo simulation in the type-A and B kinetic regimes. Z. Metallkd. 2002, 93, 256–264.

10. Divinski, S.V.; Hisker, F.; Kang, Y.-S.; Lee, J.S.; Herzig, Chr. 59Fe grain boundary diffusion in nanostructured γ -Fe-Ni part: Effect of bimodal microstructure on diffusion behavior in type-C kinetic regime, Z. Metallkd. 2002, 93, 265–272.

11. Divinski, S.V.; Hisker, F.; Kang, Y.S.; Lee, J.S.; Herzig, Chr. Tracer diffusion of ^{63}Ni in nano- γ -FeNi produced by powder metallurgical method: Systematic investigations in the C, B, and A diffusion regimes. Interface Sci. 2003, 11, 67–80.

12. Lee, J.S.; Yun, J.C.; Choi, J.P.; Lee, G.Y. Consolidation of iron nanopowder by nanopowder-agglomerate sintering at elevated sintering. J. Kor. Powder Metall. Inst. 2013, 20, 1–6.

13. Lee, J.S.; Kang, Y.S.; Kwon, S.K.; Cha, B.H.; Qin, X.Y. A new processing route for net-shaped nanoparticulate materials. Adv. Powder Technol. 2004, 15, 639–655.

14. Qin, X.Y.; Lee, J.S.; Nam, J.G.; Kim, B.S. Synthesis and microstructural characterization of nanostructured γ -Ni-Fe powder. Nanostr. Mater. 1995, 11, 383–397.

15. Kim, B.S.; Lee, J.S.; Sekino, T.; Choa, Y.H.; Niihara, K. Hydrogen reduction behavior of NiO dispersoid during processing of Al_2O_3 -Ni nanocomposites. Scr. Mater. 2001, 44, 2121–2125.

16. Jung, S.S.; Kang, Y.S.; Lee, J.S. The optimization of hydrogen reduction process for mass production of Fe–8 wt % Ni nanoalloy powder. Mater. Sci. Forum 2007, 534–536, 153–156. Materials 2013, 6 4063

17. Jung, S.S.; Lee, J.S. In-situ kinetic study of hydrogen reduction of Fe_2O_3 for the production of Fe nanopowder. Mater. Trans. 2009, 50, 2270–2276.

18. Lee, J.S.; Yun, J.C.; You, W.K. Full Density Processing of Pure Iron Nanomaterial by Nanopowder Agglomerate Sintering and Related Properties. In Proceeding of the World Congress PM 2010, Florence, Italy, 10–14 October 2010.

19. Nam, H.Y.; Kwon, S.K.; Kang, Y.S.; Lee, J.S. Sintering behavior of the PIMed Fe–50 wt % Ni nanoalloy powder. Mater. Sci. Forum 2004, 449–452, 1141–1144.

20. Rahaman, M.N. Ceramic Processing and Sintering; Marcel Dekker, Inc.: New York, NY, USA, 1995.

21. Cha, B.H. Sintering Behavior of Net-shaped Nanocrystalline Fe–Ni Powder Material and Its Mechanical Properties, Ph.D. Thesis, Hanyang University, Hanyang, Korea, 2009.