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Densification and microstructural development during sintering of powder injection molded Fe micro–nanopowder

Joon-Phil Choi [a], Hyun-Gon Lyu [a], Won-Sik Lee [b], Jai-Sung Lee [a,*]

[a] Department of Metallurgy and Materials Science, Hanyang University, Ansan 426-791, Republic of Korea
[b] Eco Materials & Processing Department, Korea Institute of Industrial Technology, Incheon 406-840, Republic of Korea

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ABSTRACT

The sintering behavior of the powder injection molded Fe micro–nanopowder fabricated under low temperature and low pressure conditions of 70 °C and 4 MPa was investigated in terms of the microstructural development. It was found that the sintered density of the micro–nanopowder was remarkably increased in the temperature range of 800 to 900 °C by the nanopowder effect due to the enhancement of the material transport by grain boundary diffusion. The micrograins in the micro–nanopowder almost maintained their initial grain size until the sintered density reached 90% of the theoretical density (TD), even showing no drastic grain growth under the condition of full density of 97% TD after sintering at 1250 °C for 3 h. This means that the presence of the nanopowders not only increases material transport paths for rapid densification but also suppresses the grain growth. The experimental findings in this study provide the potential application of micro–nanopowders for processing the PIM products with full density and fine microstructure.

1. Introduction

Powder injection molding (PIM) is an attractive cost-effective process to manufacture net-shaped components for mass production. In this technology, sintering is critical for determining the mechanical and chemical properties of the final components [1]. In particular, the dimensional accuracy and surface roughness required for the final components are determined by sintering technology [2–4]. Much is known about the effect of high sintered density on the final quality of PIMed parts, which allows for good mechanical properties and corrosion resistance [1,5,6]. Studies of the sintering process in PIM have therefore been conducted to achieve full or near-full density by using different particle size distributions, to improve the packing density [7–9]. However, powder loading in feedstock decreases due to degradation of the compactibility and the packing density of the fine particles, as it results in the loss of control of the dimensional accuracy during sintering [1,10,11].

In our previous work, we developed feedstock for low temperature micro-PIM using a micro–nano-mixed powder [12–14]. It was found that including a low viscosity binder without a high density polymer appeared to achieve superior flowability at low temperature and relatively high powder loading up to 71 vol.%. The feedstock was especially optimized with 66 vol.% of powder loading with a nanopowder composition of 25%, showing interesting properties in the entire PIM process. Such good feedstock property was explained in part due to the solid lubrication effect of the nano-agglomerate powders [12].

Owing to the excellent feedstock performance, the authors [15] succeeded in powder injection molding of micro–nanopowders under low temperature and low pressure conditions of 70 °C and 4 MPa. According to the results, the nanopowders uniformly located at the interstitial space of the micropowders could consistently maintain the molded parts during the injection molding process, and therefore sufficient green strength could be obtained for successful demolding without using high density polymer binders. During the debinding process, we found that nanopowders formed a strong bridge by pre-sintering at temperatures below 500 °C, and the strength of the micro–nanopowder compact was much higher than that of the micropowder samples [14]. It was also found that nanopowders play an important role in the entire sintering process by enhancing densification but suppressing grain growth. However, it is not entirely understood how both competing sintering processes of densification and grain growth contribute to the enhanced sintering of micro–nanopowders. Furthermore, these above results conflict with earlier studies [16–18] indicating that sintering using a bimodal powder might not be successful due to the remaining stress, irregular microstructures, and differences in the driving force between the two powders. Thus, to understand this issue, the sintering behavior of the powder injection molded Fe micro–nanopowder, especially the effect of nanopowder on densification and grain growth, was investigated in terms of the microstructural development in this study.

2. Experimental procedure

The metal powders used for this study are shown in Fig. 1: (a) is carbonyl Fe powder (BASF, 98.3%, D_{50} = 4.1 μm) and (b) is Fe nanopowder

* Corresponding author. Tel.: +82 31 400 5225; fax: +82 31 406 5170.
E-mail address: jslee@hanyang.ac.kr (J.-S. Lee).

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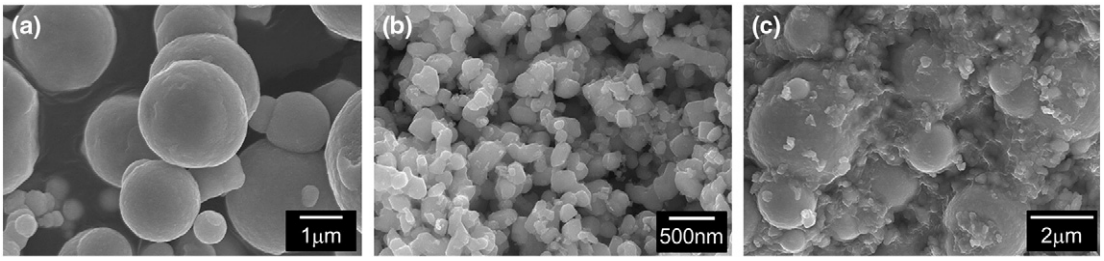

Fig. 1. SEM micrographs of (a) Fe micropowder, (b) Fe nanopowder and (c) feedstock.

(D_{50} = 100 nm) fabricated by ball milling and a hydrogen reduction process [19]. Fe micropowder of 75 vol.% and nanopowder of 25 vol.% were mixed with a binder composed of paraffin wax and stearic acid in a twin screw mixer (C.W. Brabender Instruments Inc., Plasti-Corder) at 70 °C and 60 rpm. Feedstock with a designed powder loading of 66 vol.% was prepared in which the 34 vol.% binder in the feedstock was composed of 75 vol.% paraffin wax and 25 vol.% stearic acid (Fig. 1(c)) [14,15]. The composition and powder loading of the micro–nanopowder were obtained from our previous study on the mixing and molding behavior of Fe micro–nanopowder feedstock [12,13]. The obtained feedstock was crushed into particles smaller than 1 mm, and then injection molded into a small double gear component at 70 °C under a pressure of 4 MPa [14,15], as shown in Fig. 2. During the injection molding, the hold pressure was fixed at 3 MPa to prevent the generation of insufficient filling, shrinkage voids or cracks. The injection molding machine used in this study was a laboratory-scale screw type machine (HPL2-007-NN-14, ANC Corp., Korea).

After the injection molding process, the subsequent heat treatment processes of debinding and sintering were carried out according to the heat treatment cycles shown in Fig. 3. In this study the debinding process was carried out during the thermal process using a multi-step schedule designed on the basis of thermogravimetric analysis [13] of the feedstock and melt wicking debinding using fine Al_2O_3 powder [4,20]. The binder was removed by a process involving a thermal and wicking method below the temperature of 500 °C in hydrogen atmosphere at different heating rates from 1 to 10 °C/min. The sintering experiment was then conducted in two ways, via heat-up sintering in a temperature range of 500 to 1250 °C, and isothermally at 1250 °C for 3 h. Both the sintering processes were carried out in a hydrogen atmosphere (H_2, 99.999% purity) with a heating rate of 10 °C/min. The flow rate of hydrogen gas was 1.5 L/min during the entire heat treatment process including debinding and sintering. For a comparison study, the same sintering experiment was performed for Fe micropowder compacts with 66% of the theoretical density prepared by static molding process under the same temperature condition as the injection molding process [13].

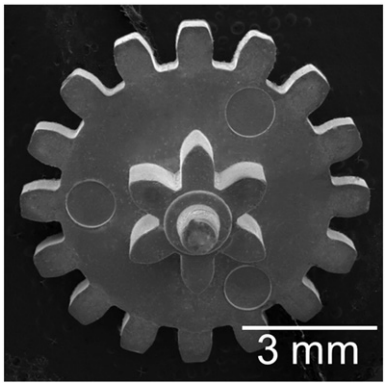

Fig. 2. SEM micrographs of injection molded part.

The density of the sintered samples was measured by Archimedes' method using distilled water, and their surface was thinly coated with wax to prevent penetration of water to the open pores of the samples before measurement. The microstructure of the sintered specimens was examined with a scanning electron microscope (SEM, HITACHI S-4800), and grain size analysis was performed using the linear-intercept method [21] from SEM micrographs taken randomly at the surface of the specimens using the relationship $G = 1.5L$, where L is the average intercept length, by counting at least 1000 grains for each specimen. In addition, the grain size measurement to compare the grain growth behavior of micro- and micro–nanopowder was separately conducted by counting the number of grains >1 μm in the sintered component based on the results above.

3. Results and discussion

3.1. Microstructural feature of the fully densified micro–nanopowder PIM part

It is well known that packing density can be increased by introducing a bimodal distribution of particles, and it may lead to higher sintered density without assisting sintering procedures such as high temperature–pressure or application of sintering aids [1,17]. However, it has also been reported that the sintered density of bimodal powder mixture has not increased due to the difference of the driving force between coarse and fine powders when used in powder injection molding [17,18]. At composition rich in coarse powders, the fine powders have a negligible effect on sintering due to the formation of a skeletal structure of the coarse powders [16,17]. In contrast, at the majority of the fine powders in bimodal mixtures, the densification of the fine particle network is degraded by the addition of coarse powders which generate stresses leading to cracks and pore growth [17,22].

In previous work [15], however, the sintering of Fe micro–nanopowder was successfully conducted at 1250 °C for 3 h in H_2 atmosphere. The microstructure of the sintered parts is shown in Fig. 4(a)

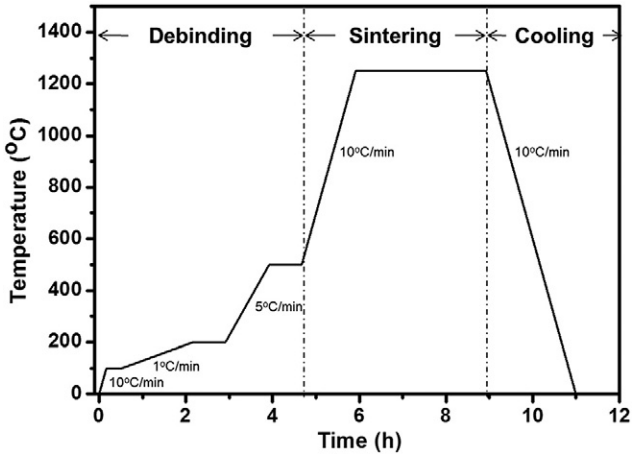

Fig. 3. Schematic diagram of the heat treatment cycle of debinding and sintering.