Microstructual Study on Consolidation of Fe Nanopowder Synthesized by Plasma Arc Discharge Process

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1. Introduction

Fe nanopowder synthesized by plasma arc discharge (PAD) process has a fine powder size (20~200 nm in size) without powder agglomeration and surface passivated layer on the metallic powder. In author's previous study, it has been proven that PADed Fe nanopowder could be easily densified at relatively low sintering temperature by even conventional pressureless sintering process. In this study, the consolidation process of PADed Fe nanopowder has been investigated in terms of microstructural development. And the innovative route for consolidation of metallic nanopowder was suggested by the discussion of the relationship between sinterability and powder characterisitcs.

2. Experimental Procedure

The Fe nanopowders were uniaxially pressed with 175 MPa in a cylindrical compaction die, to form disk shape specimens. The green densities of each compacted bodies were 43%T.D. The volume shrinkage of compacted bodies were measured by using laser opto-dilatometry system. The compacted bodies were heated up to 800℃ in hydrogen atmosphere with various heating rates of 5, 10, 20 and 30℃/min. For comparison, micron size Fe powder (5-10 μm, 99.9%) was compacted with 200 MPa (47%T.D. of green density) and sintered at 1200℃ for 2 hours in H_2 atmosphere. The microstructures of each sintered samples were observed by FE-SEM and TEM, and the pore distribution in compaction and sintered bodies were measured by porosimetry during the densification process.

3. Results and Discussion

The densification behaviors of PADed Fe nanopowders with the different heating rates are depicted in Fig. 1. In the result, the sintering behavior started at 280℃ and finished at 560℃ for 5℃/min. heating rate condition. And the shrinkage divided into two steps, namely, the rapid densification at low temperature region and the densification retardization at higher temperature range. The transition temperature of densification rate is 380℃ and the green density is around 70%T.D. at this point.

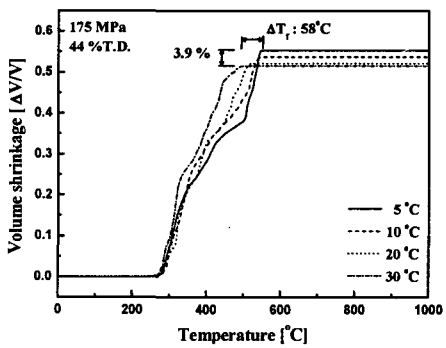

Fig. 1. Volume shrinkage during sintering of PADed Fe nanopowder with heating rates.

With increasing of the heating rates, the finishing temperature of densification and the volume shrinkage decreased at the same time. The sintering densities for each samples are in the range from 83 %T.D. to 91 %T.D. after heating up. The highest sintering density is 90.5 %T.D. at lowest heating rate of 5℃/min condition.

Fig. 2 shows the microstructures of fracture surface for sintered body with elevating temperature. The fracture

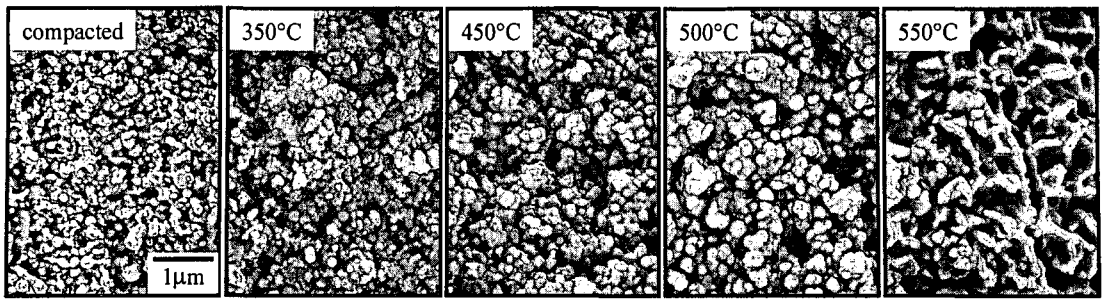

Fig. 2. Fracture microstructure of compacted and sintered PADed Fe nanopowder with temperatures.

surface of compaction body represented the powders are homogeneously dispersed and there are no large pores in the compaction body. The measured pore size was around 60～70 nm and it shows the mono-modal distribution in Fig. 3. But with increasing of sintering temperature, the powders contact each other and form a agglomeration with volume shrinkage. The pore size doesn't change but the total pore volume decreases with elevating temperature. Full densification over 98%T.D. could be obtained for the sintered specimen at 560℃ for 1 hrs. and the average grain size was around 200nm.

The PADed nanopowder has higher sintering density than micron size powder (80 %T.D.) at even low temperature as a half of sinteringtemperature for micron powder. In micron size powder sample, the grains remarkably grew up to 50 μm due to relatively high sintering temperature. And the Vickers hardness of PADed nanopowder was around 250 Hv, which is 3 times higher than that of micron size powder.

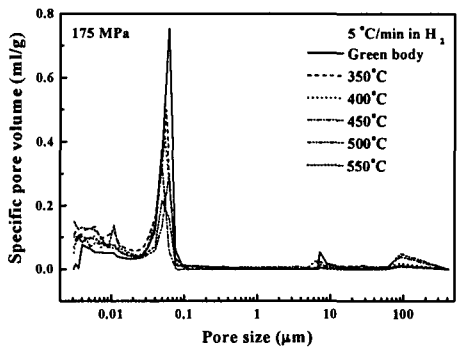

Fig. 3. Pore size distribution of PADed Fe nanopowder with elevating temperature.

4. Conclusion

The sintering process of PADed Fe nanopowder has been studied with microstructural change. The densification occurred from 280℃ to 560℃ temperature range and consisted of first rapid densification and second densification retardization. During the densification process, the homogeneous pore distribution plays an important role for full densification of 98.5%T.D. and inhibiting grain growth under 250 nm in size.

Acknowledgement

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