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Refinement of Nanocrystalline Fe-Ni Agglomerate Powder by Hydrogen Reduction of Size-controlled Fe_2O_3-NiO Powder

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1. Introduction

Recently it has been reported that bulk nanocrystalline Fe-Ni alloy can be fabricated by pressureless sintering of Fe-Ni nanoalloy powder synthesized by hydrogen reduction of Fe_2O_3-NiO ball milled agglomerate powder.[1~2] The result of sintering behavior showed that the initial pore size distribution of nano-agglomerate Fe-Ni powder played a decisive role for full densification process.[3] Namely, the bimodal type of pore size distribution consisting of nanoscale- and microscale pores in agglomerate nanopowder was found to suppress the densification process of Fe-Ni agglomerate powder. As a consequence, fully densified Fe-Ni nanopowder could be obtained by reducing microscale large pores. For this, in the present study we investigated how to reduce the large size pores by controlling the Fe-Ni agglomerate size. The agglomerate size was also controlled by hydrogen reduction process depending on the powder characteristics of Fe_2O_3-NiO.

2. Experimental

Commercial Fe_2O_3 powder(1㎛, 99.9%) and NiO powder (7㎛, 99.9%) were used as the starting materials. Mixing of the precursor powders was done by high energy ball milling in attritor. After ball milling, agglomerated oxide powder was crushed and dispersed by sonomilling treatment in methyl alcohol, and finally classified in size by centrifugal method (2000, 4000, and 8000 rpm for 20 min.). Hydrogen reduction of each oxide powders was performed at a heating rate of 10°C/min up to 600°C and its behavior was studied by TG and hygrometry. Powder characteristics were analyzed by BET, SEM and TEM.

3. Results, discussion and conclusion

Ball-milled powder of Fe_2O_3-NiO powder was in an agglomerate form as 20 ㎛ in size which consists of about 20 nm in average crystallite size. After sonomilling and centrifugal classification treatment of the oxide powder, we had three agglomerate size groups of 70, 150 and 250 nm, respectively. With decreasing oxide agglomerate size, the reduction process was accomplished in lower temperature range. After hydrogen reduction, the particle size was 50, 100 and 150 nm, respectively. This implies that the microstructure of Fe-Ni nanoalloy powder strongly depends on that of initial oxide agglomerate.

4. References

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