The Optimization of Hydrogen Reduction Process for Mass Production of Fe-8wt%Ni Nanoalloy Powder

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1. Introduction

Mechano-chemical process including ball-milling and hydrogen reduction is widely used for fabrication of nanosized metal powder. It is very important to optimize hydrogen reduction process, because the properties of final Powder Metallurgy parts are greatly influenced by reduced nanopowder. Hydrogen reduction depends on various process variables such as temperature, time, quantity and packing shape of powder and flow rate of hydrogen. Hence, in order to obtain completely reduced powder of which grain growth is inhibited, it requires to the optimization of hydrogen reduction by controlling the process variables. Conventional investigation methods used to optimize hydrogen reduction were to analyze the behavior of vapor using TG and DTA. However, the results from TG and DTA using a little amount of powder have shown the difficulties to estimate the hydrogen reduction behavior for mass production.

In this sense, more effective method to optimize of hydrogen reduction process for mass production has been required and the authors tried to trace the behavior of vapor occurred during hydrogen reduction process by attaching hygrometry to the production system. For the measurements, reduction temperature and time which influence on the properties of reduced powder more than the others were varied as process variables.

2. Experimental

A mixture of iron oxide and nickel oxide with a composition of Fe-8wt%Ni was prepared by blending α-Fe_2O_3 (99.9%, 1 ㎛) and NiO (99.9%, 7 ㎛) powders. In order to reduce grain size, high-energy ball milling was performed in a stainless steel attritor at a speed of 300 rpm for 10 h. The powder-to-ball mass ratio was 1:50 with a powder mass of 100 g. Methyl alcohol was added as a PCA (Process Control Agent). After ball milling, the powders were dried in oven for 6 h at 60℃ and sieved down 100 mesh. Also, the powders (BM) were dry-milled to homogenize of agglomerate size for 2 h and then sieved down 400 mesh. Then the dry-milled (DM) powders were reduced at 450℃, 500℃, 550℃. The behavior of vapor generated during reduction was measured by hygrometry.

In the powder characterizations, phase identification and reduction percentage were analyzed by XRD. The specific surface area of powder was measured by BET. Finally, microstructures, degree of agglomeration and grain size of reduced nanopowder were observed by SEM and TEM.

3. Results, discussion and conclusion

Although reduction process was completed, vapor was continually detected by hygrometry analysis. Also, it was found that reduction at every temperature conditions was completed at 140 min. From XRD analysis, it was confirmed that powder was completely reduced. Average grain size of powder reduced at 450℃, 500℃ and 550℃ was 100 nm, 150 nm and 200 nm respectively by SEM and TEM.

From the hygrometry curves of the powders reduced at 450℃, 500℃ and 550℃, the highest reduction rate were observed at 60 min, 51 min and 43 min. At this time, the average grain size was 70 nm, 90 nm and 120 nm (Fig. 1), respectively. Moreover, it was confirmed that powders were reduced over 90% (Fig. 2).

According to above results, it can be confirmed that hydrogen reduction behavior for mass production was quite differ from that of laboratory scale. The difference can be expressed by the trapped vapor in the powder, which was not released easily when they are generated. Therefore, although reduction was completed, reduction time from hygrometry result was delayed over 80 min for all temperature conditions and grain growth occurred in this experiment. Conclusively, new process variables are required to optimize of hydrogen reduction process for mass production.

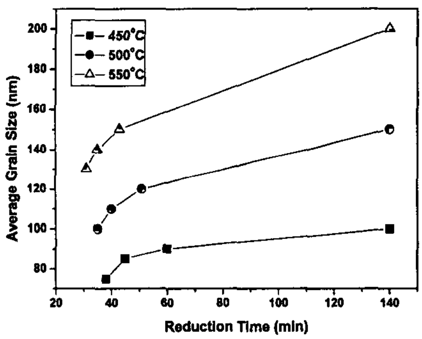

Fig. 1. Average grain size with various temperature and time.

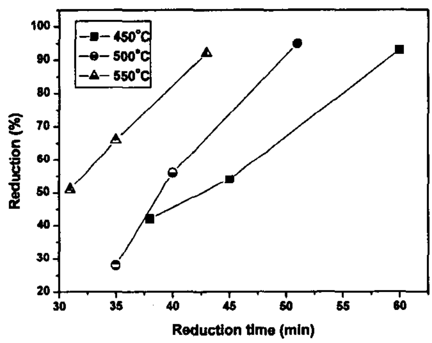

Fig. 2. Reduction percentage with various temperature and time.