DEVELOPMENT OF RARE EARTH BASED MAGNETIC ALLOY POWDERS BY REDUCTION-DIFFUSION METHOD

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Abstract

Samarium-cobalt based magnetic alloy powders have been prepared by calciothermic reduction of samarium oxide in presence of cobalt under argon atmosphere. The resultant alloy powders were subjected to decalcification. The effect of processing parameters in obtaining the desired magnetic phases and on residual calcium has been studied. The products have been characterized by various microscopic and spectroscopic techniques. Higher samarium content and lower calcium content in the powder improve the magnetic properties of the final product.

Introduction

Permanent magnets based on rare earth-transition metal intermetallics are the basis of ever increasing number of commercial and scientific applications, including electric motors, NMR scanners, wind-mills, compact discs, actuators for robotics and flight control, etc [1-3]. The advantage of permanent magnets in these applications is their ability to exhibit high level, constant magnetic fluxes without applying an external magnetic field or electrical current. Samarium-cobalt based magnets with high energy product and excellent coercive force, are ideally compact and suitable to highly efficient machine and components in which higher operation temperature, higher corrosion and oxidation resistance are crucial [4]. Iron-neodymium-boron (Nd-Fe-B) and other rare earth-iron/cobalt-based intermetallics have been investigated due to their superior magnetic properties [5-7]. However, corrosion resistance is a primary concern with Nd-Fe-B type of magnet and it has limitations to be used at higher temperature. In view of growing indigenous demand of samarium-cobalt based permanent magnets, namely SmCo5 and Sm2Co17 for specific applications, attention has been paid to prepare this magnetic material. In this paper the preparation of Sm-Co alloy powders by reduction-diffusion process followed by decalcification has been discussed.

Experimental

$\text{Sm}_2\text{O}_3$ powder (supplied by IREL: 95-96% pure) was mixed with calculated amount of Co (purchased from local vendor: 98.5 % pure) and Ca granules in a glove box. The charge was taken in a crucible and heated to high temperatures (1000-1200°C) under inert atmosphere in a resistance-heating furnace. After required duration of heating (3-10 hrs) the furnace was cooled and the charge was subjected to leaching for removal of Ca/CaO. The leaching was performed initially by water wash and followed by organic acid. The product was characterized by XRD (for phase identification), SEM (for morphology and size of the powder), EDX, XRF (for chemical composition) and
ICP-AES (for residual calcium). The powder was used to make a bar magnet and its magnetic properties were measured. The process has been developed to produce the powder in one kg batch scale with residual Ca $\sim$ 2000 ppm.

**Results and Discussion**

The desired phases were obtained when the reduction was carried out with more than 50% stoichiometric excess of Ca. 5-15% excess $\text{Sm}_2\text{O}_3$ was found to be necessary to get the desired phases. The reduction was incomplete when it was carried out below $1050^\circ\text{C}$.

Almost 95% CaO was removed by leaching the product with water. For further decrease of residual calcium, leaching with buffer solution containing ammonia and organic acid was necessary. Fine control of pH was found necessary to bring down the calcium content below 2000 ppm in the final product without disturbing the magnetic phases. Improper leaching results in loss of rare earth values. Fig. 1 (a) and (b) are the XRD patterns of the leached SmCo5 and Sm2Co17 powders, respectively.

Typical SEM image and the corresponding EDX pattern of the decalcined powder is shown in Fig.2 (a) and 2 (b) respectively.

The energy product improves with lower Ca content and higher Sm content. Fig 3 (a) shows a magnetization curve of a product which has 27% Sm and 5000 ppm Ca, whereas Fig. 3 (b) represents the curve of a product containing 37% Sm and 2000 ppm Ca. Definitely the product with higher Sm and lower Ca shows much better magnetic properties.
better residual magnetization and energy product, which are required for a good permanent magnet.

Fig. 3 (a): Magnetization curve of a product containing 27% Sm and 5000 ppm Ca

Fig. 3 (b): Magnetization curve of a product containing 37% Sm and 2000 ppm Ca

Conclusion

Preparation of Sm-Co alloy powder by reduction-diffusion process has been optimized. Leaching conditions were also standardized for successful removal of calcium from the products. Improper leaching resulted in loss of Sm. XRD pattern showed the formation of required phases. The oxygen and calcium contents in the final products are 0.5 and 0.2 wt % respectively which are well within the permissible limit for magnet preparation. The magnetic properties improve with increase in Sm content in the final product.

Acknowledgement

The authors are grateful to Dr. D. K. Singh for technical inputs during leaching, Dr. M. Anitha for chemical analysis and Shri Bhaskar Paul for SEM analysis.

References