

## Sm<sub>2</sub>Fe<sub>17</sub> Prepared by Calciothermic Reduction-Diffusion Using Different Iron Powders

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### Abstract

*This paper compares the effect of using different types of iron powders for the preparation of Sm<sub>2</sub>Fe<sub>17</sub> by calciothermic reduction-diffusion (CRD). Three types of iron powder were used: carbonyl, sponge and water atomized. The results show that, when immediately nitrogenated after the CRD process, Sm<sub>2</sub>Fe<sub>17</sub> prepared from sponge and water atomized iron powders yield Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub>-magnets with a high degree of texture. However, after a suitable treatment with hydrogen followed by nitrogenation, Sm<sub>2</sub>Fe<sub>17</sub>-powders made from Carbonyl iron produce magnets with the best quality regarding coercivity, remanence and degree of texture.*

**Keywords :** permanent magnets, SmFeN, calciothermic reduction-diffusion, Iron powder

### 1. Introduction

Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub> was first synthesized by Coey and co-workers in 1990 [1]. Different techniques can be used for the preparation of the parent Sm<sub>2</sub>Fe<sub>17</sub> alloy. Among them are: melting, mechanical alloying and calciothermic reduction-diffusion (CRD). The CRD technique presents some advantages over the others, and they can be summarized as follows:

- It uses samarium oxide as raw material, which is cheaper than the metallic samarium used in the melting process
- It produces the alloy in powder form
- Its main reaction occurs at moderate temperatures, which leads to less loss of samarium due to evaporation

Most of the reports on the preparation of Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub> magnetic materials using alloy powders prepared by CRD have neglected the influence of raw materials (iron, samarium oxide and calcium) and process parameters on the final magnetic properties of the nitrogenated Sm<sub>2</sub>Fe<sub>17</sub> alloys [2].

This work is intended to evaluate the differences in magnetic properties of Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub>-magnets produced via CRD, using three different commercial iron powders: carbonyl, sponge and water atomized.

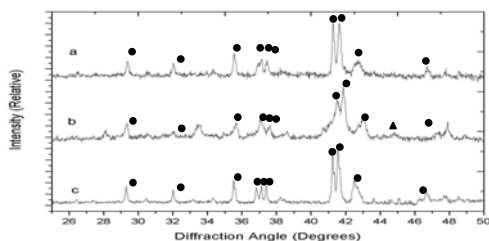
### 2. Experimental and Results

The powders used had the following particle sizes: 90 % < 10 μm for carbonyl iron powder, 90 % < 60 μm for

sponge iron powder and 90 % < 45 μm for water atomized iron powder. The iron powders were mixed individually with samarium oxide and metallic calcium. The mixtures were placed in a stainless steel retort, and heated to 1000°C [3] in argon atmosphere to initiate the CRD process. For the carbonyl iron powder, the CRD time took 3h, for the water atomized iron powder, it took 6h and for the sponge iron powder, it took 10h. These different periods of time were chosen in order to completely interdiffuse iron and samarium. The temperature of the furnace was then decreased to 450°C in order to perform the nitrogenation step. The atmosphere used was N<sub>2</sub>. The nitrogenation time was chosen according to the respective particle size in order to fully nitrogenate the particles. After the nitrogenation, the powders were washed with water and with 2% acetic acid solution. This last step was carried out in order to remove the undesired reaction products such as CaO and traces of Sm<sub>2</sub>O<sub>3</sub>. The powders were then milled until their particle sizes became smaller than 25 μm.

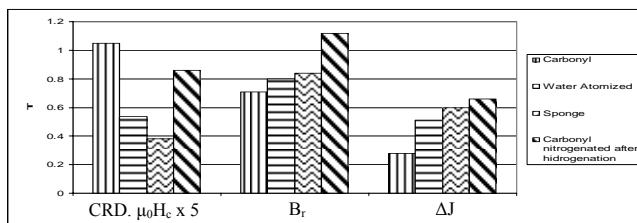
Figure 1 shows the X-ray Diffraction (XRD) patterns, using α Cu radiation, of the materials. They show the peaks corresponding to Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub>, although the powder produced from sponge iron presented a small amount of α iron. This may happen because even by using moderated temperatures the extended time necessary for nitrogenation of large particles can cause free α-iron to form as reported by Katter et al [4].

In addition, the peaks in the pattern of the material produced with sponge iron powder are not as well defined as in the other patterns. This could be attributed to the fact that the nitrogenation was not completely uniform in this alloy.



**Fig. 1. X-ray diffraction pattern of  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  using Cu radiation produced by calciothermic reduction-diffusion (CRD) with a) water atomized iron powder, b) sponge iron powder c) carbonyl iron powder. ● Indicate  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  peaks and ▲ indicate  $\alpha\text{-Fe}$  peaks.**

Figure 2 shows the magnetic properties of  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  magnets, measured in a Vibrating Sample Magnetometer (VSM), produced from powder immediately nitrogenated after CRD. For the alloy produced with carbonyl iron powder, the remanence was 0.71 T and intrinsic coercivity 0.21 T. The difference in polarization at 1.5 T in easy and hard directions, that is taken as a measure of the degree of texture is 0.32 T. The alloy produced with sponge iron powder has a higher remanence (0.84 T) but its coercivity is smaller (0.076 T). The difference in polarization in easy and hard directions, however, is much higher, 0.6 T. The sample prepared with water atomized iron powder has intermediary values: the remanence was 0.80 T, coercivity 0.11 T and polarization difference 0.51 T.



**Fig. 2. Magnetic properties of  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  produced by CRD.  $\mu_0 H_c$  – coercivity;  $B_r$  – remanence;  $\Delta J$  – difference in polarization. Note that coercivity is multiplied by 5.**

The difference in polarization and remanence are higher for the  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  produced with sponge iron and the coercivity is higher for the one produced with carbonyl iron. The poor texture in the magnet produced with carbonyl iron is attributed to its finer microstructure which leads to a non-monocrystalline powder after milling.

In order to improve the texture in the  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  produced with carbonyl iron hydrogen decrepitation (HD) [5] was performed during the treatment. For that, the furnace temperature was decreased to 150°C after CRD and  $\text{H}_2$  was introduced into the retort for 1h. Then, the nitrogenation was allowed to take place under the previous conditions. Figure 2 shows the magnetic properties for the  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  magnet produced with carbonyl iron using this

cycle. The remanence increases to 1.12T and the polarization difference increases to 0.66T. This means that the magnet has the highest texture among the three alloys. The same treatment has been applied to the  $\text{Sm}_2\text{Fe}_{17}\text{N}_3$  produced with sponge iron powder but it did not lead to further increase in magnetic properties.

### 3. Summary

In this study, it was possible to produce  $\text{Sm}_2\text{Fe}_{17}$  using the different iron powders: carbonyl, sponge, and water atomized. The processing conditions, however, had to be different in order to obtain the formation of pure intermetallic  $\text{Sm}_2\text{Fe}_{17}$ . High degrees of texture were found in the alloys prepared with sponge and water atomized powder. This is probably due to their coarser microstructure that may result in more monocrystalline particles after milling than the one prepared with carbonyl iron powder. The alloy produced with carbonyl iron powder did not present a high degree of texture when it was nitrogenated immediately after the CRD. However, it was possible to increase it by introducing hydrogen to the powder before nitrogenation. The magnet produced with this powder presented the highest degree of texture, coercivity and remanence.

### 4. References

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