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Study of Crystallization Process of $Fe_{78}Tm_2B_{20}$ and $Fe_{76}Tm_4B_{20}$ Alloys

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ABSTRACT
The amorphous to crystalline transformation in $Fe_{78}Tm_2B_{20}$ (Sample S1) and $Fe_{76}Tm_4B_{20}$ (Sample S2) alloys has been carried out using Differential Scanning Calorimetry (DSC). Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) of the samples are also discussed to confirm the complete crystallization after heating. DSC analysis of the samples S1 and S2 showed that on heating the samples upto 1000^0C, a sharp peak was observed at 545^0C and 791^0C respectively, indicating a primary crystalline phase appearing in the amorphous matrix. In sample S2 another sharp peak was observed at 896^0C, indicating further crystallization in the sample. Both the samples are completely crystallized when temperature is above 929^0C. In sample S2, the crystallization is a multi stage process where different crystalline phases grow in the amorphous matrix. SEM patterns of these samples showed that as cast samples are amorphous and the sample are completely crystallized when heated to 1000^0C. EDS patterns also confirmed the complete crystallization of the samples after heating to 1000^0C. Thus increase of Tm concentration in Fe-Tm-B alloy changes the process of crystallization.

I. INTRODUCTION

RE (Rare-Earth) containing alloys (usually Fe-RE-B) obtained in amorphous state by Melt - Spinning Technique and substantially annealed, have enhanced magnetic properties, compared to traditional permanent magnets. As the cost is lowered due to the substantial reduction of the rare-earth content, it therefore accounts for a new generation of permanent magnetic materials. The preferred charecteristics for permanent magnets are high saturation magnetization (M_s), high coercivity (H_c) and large energy product. Also, the hard magnetic nanocrystalline alloys with remanance enhancement provide greater flexibility in processing, especially with powder materials. These remanence enhanced nanocrystalline hard magnetic alloys may find many applications as permanent magnet components [1,2]. Thus, the development of rare earth iron-based permanent magnet has been a long standing goal of the permanent magnet industry.

The crystallization of amorphous alloys is diffusion controlled [3]. In the alloys with denser amorphous structure, the higher crystallization temperature (T_x) is expected. In this paper we present the amorphous to crystalline transformation in $Fe_{78}Tm_2B_{20}$ (Sample S1) and $Fe_{76}Tm_4B_{20}$ (Sample S2) alloys using Differential Scanning Calorimetry (DSC). The techniques of Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) are also used to confirm the crystallization of the heated samples.

II. EXPERIMENTAL

Amorphous ribbons of $Fe_{78}Tm_2B_{20}$ (Sample S1) and $Fe_{76}Tm_4B_{20}$ (Sample S2) alloys having a thickness of about 30 µm and width of 1 mm are produced by single roller melt spinning technique. The samples are obtained from other research labs. Samples of size 5 mm x 1 mm were cut and heat treated. For thermal analysis, a differential scanning calorimetry (DSC) was carried out. The samples of 2.603mg were taken and used for the DSC measurement. Amorphous samples were heated to 1000^0C at a constant heating rate of 20^0C/min. SEM and EDS of the amorphous and heated samples are also recorded at room temperature.

III. RESULTS AND DISCUSSION

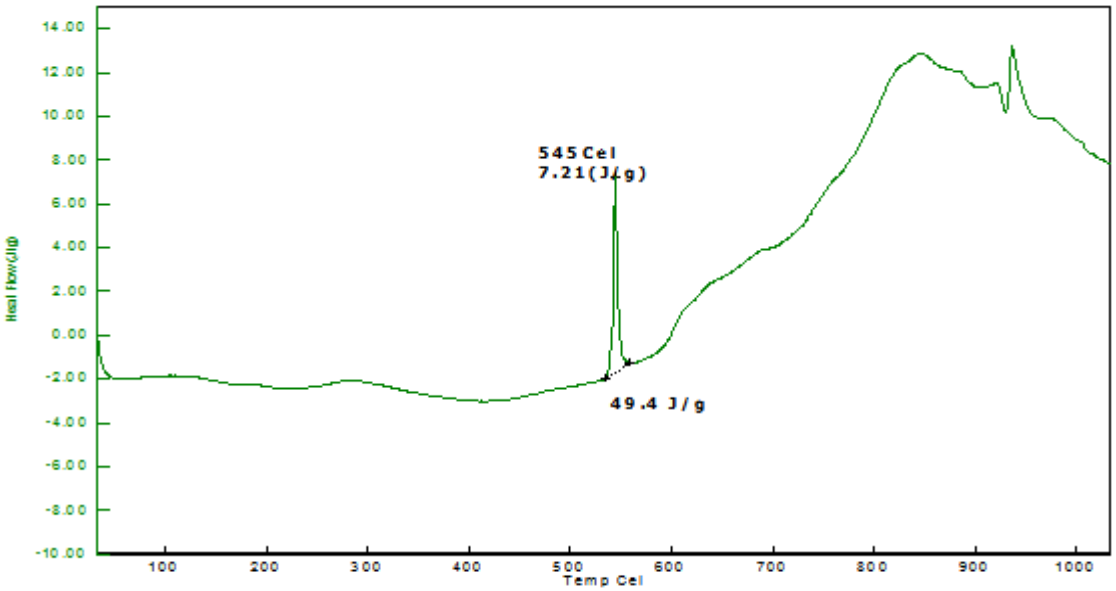

Figure 1 DSC curve of Amorphous $Fe_{78}Tm_2B_{20}$ alloy at a heating rate of 20^0C/min.

Figure 1 shows the DSC curve of fresh amorphous $Fe_{78}Tm_2B_{20}$ alloy in the temperature range 30^0C - 1000^0C at a heating rate of 20^0C/min. The DSC curve shows a sharp peak at 545^0C and a broad hump with a small sharp peak in the temperature range 800^0C - 1000^0C. The first sharp peak at 545^0C shows phase transformation from amorphous to crystalline indicating the primary crystallization of the amorphous sample where a primary crystalline phase α-Fe may grow in the amorphous matrix. The broader curve with a small sharp peak may indicate secondary crystallization of the sample after which the sample is completely crystallized. A detailed study on the confirmation of the phases in the primary and secondary crystallization of the sample will be reported later.

Figure 2 shows the DSC curve of fresh amorphous $Fe_{76}Tm_4B_{20}$ alloy in the temperature range 30^0C - 1000^0C at a heating rate of 20^0C/min. DSC analysis showed that on heating the sample upto 1000^0C, a sharp peak was observed at 791^0C, indicating a primary crystalline phase appearing in the amorphous matrix. Another sharp peak was observed at 896^0C showing further crystallization of the sample. Minor peaks are observed at 853^0C and at 929^0C. The sample is completely crystallized at 929^0C. Thus, in amorphous $Fe_{76}Tm_4B_{20}$ alloy, the crystallization is a multi stage process where different crystalline phases may grow in the amorphous matrix. The crystallization process is thermally activated and generally proceeds in various stages. Thus increase of Tm concentration in Fe-Tm-B alloy changes the process of crystallization.

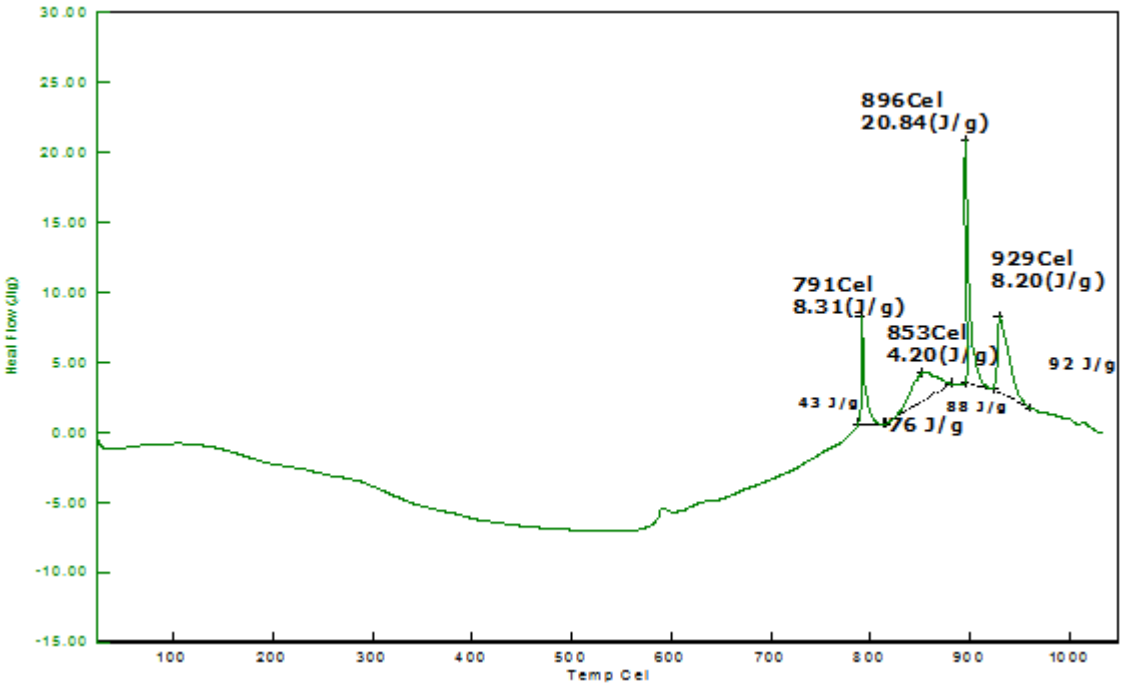

Figure 2 DSC curve of Amorphous $Fe_{76}Tm_4B_{20}$ alloy at a heating rate of 20^0C/min.

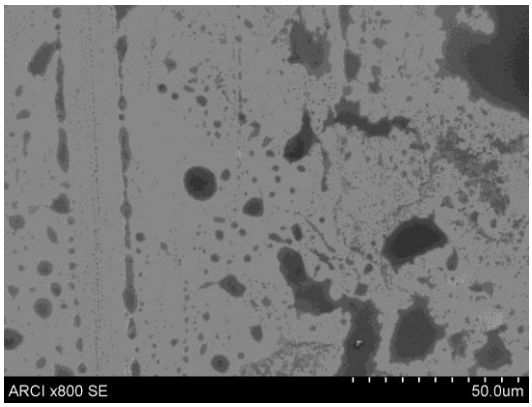

Fig. 3 SEM photo of as cast sample (S1)

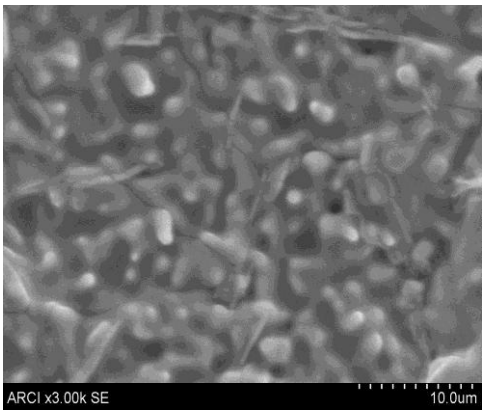

Fig. 4 SEM photo of heated sample (s1)

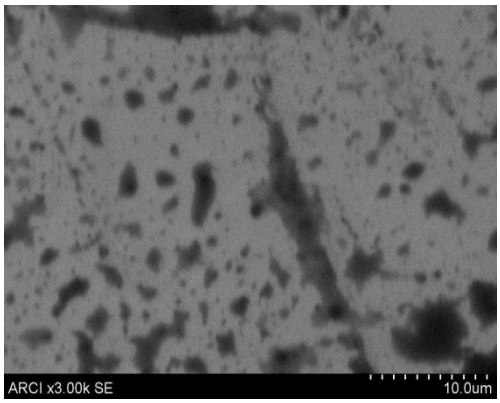

Fig. 5 SEM photo of the as cast sample (S2)

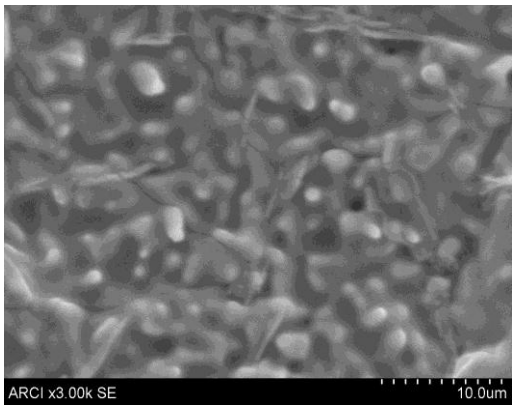

Fig. 6 SEM photo of the heated sample (S2)

Figures 3 and 4 show SEM micrographs of the as cast (fresh) and heated samples of $Fe_{78}Tm_2B_{20}$ (Sample S1) respectively. Fig. 3 shows that the sample is amorphous. Fig. 4 shows that the sample is crystallized. Similarly, figures 5 and 6 show SEM micrographs of as cast and heated samples of $Fe_{76}Tm_4B_{20}$ (Sample S2).

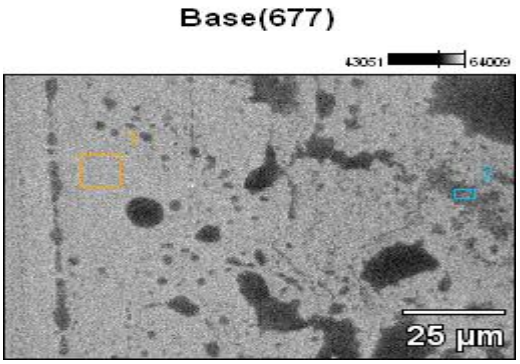

Fig. 7 EDS photo of the sample (S1) Before heating

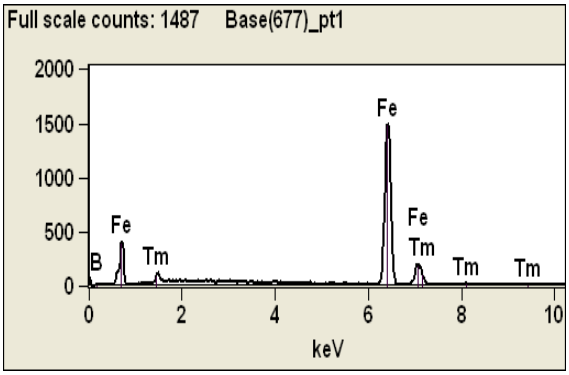

Fig. 8 EDS spectrum of the sample (S1) before heating

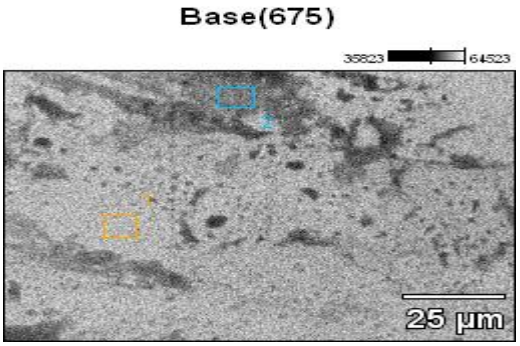

Fig. 9 EDS photo of the as cast sample (S2)

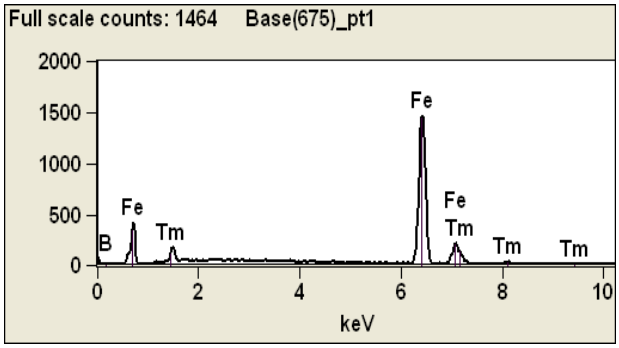

Fig. 10 (a)EDS spectra of the as cast sample(S2)

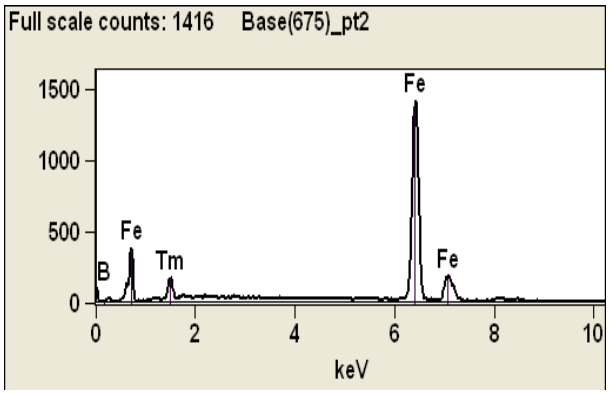

Fig. 10 (b)EDS spectra of the as cast sample (S2)

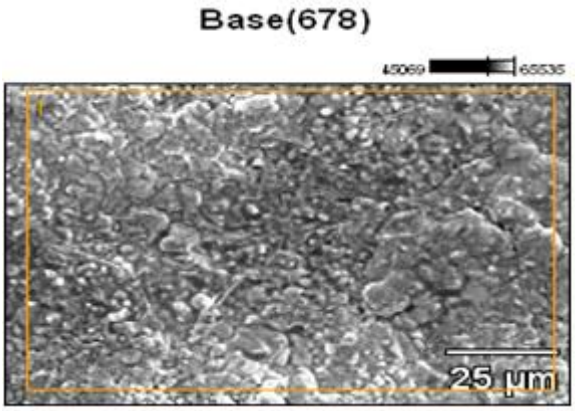

Fig. 11 EDS photo of the sample (S1) after heating

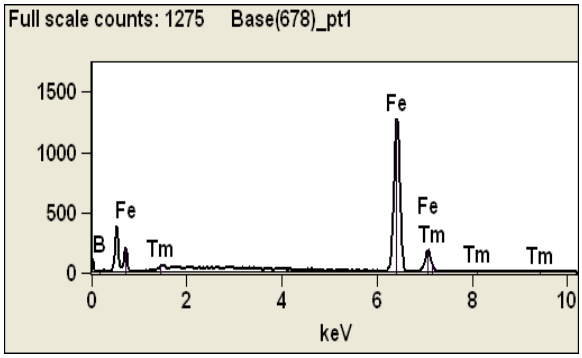

Fig. 12 EDS spectrum of the sample (S1) after heating

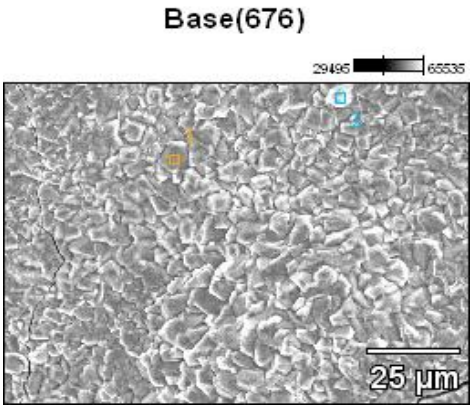

Fig.13 EDS photo of the heated Sample (S2)

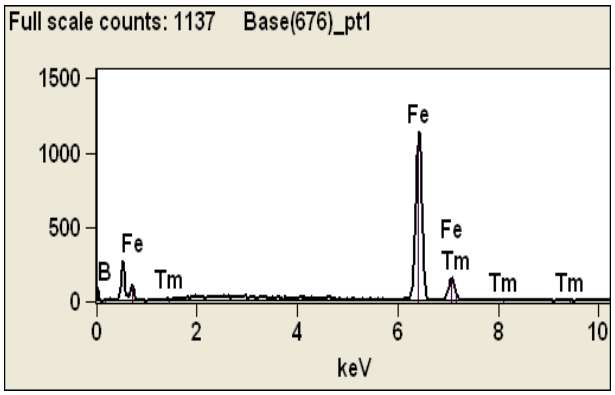

Fig.14 (a)EDS spectrum of the heated sample (S2)

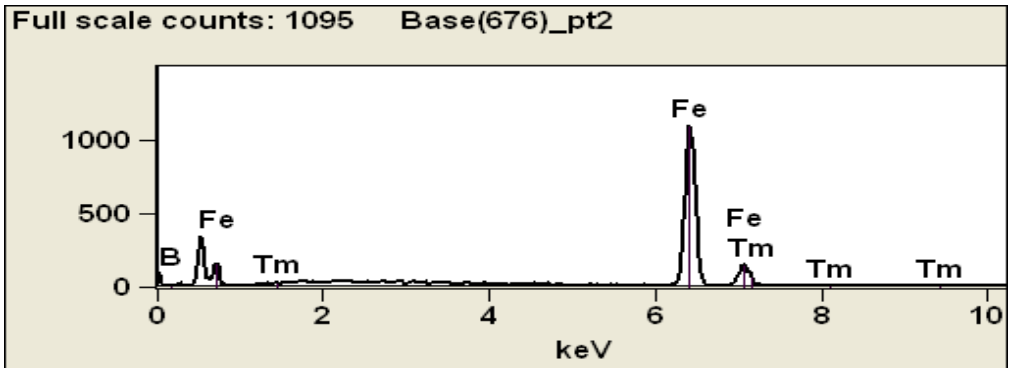

Fig.14 (b) EDS spectrum of the heated sample (S2)

Figure 7 shows the EDS photo of as cast (Fresh) sample (S1) indicating the amorphous nature. Figure 8 shows the EDS spectrum of point 1(the orange square in Fig. 7). Figure 9 shows the EDS photo of as cast (Fresh) sample (S2) indicating the amorphous nature. Figure 10(a) shows the EDS spectrum of point 1(the orange square in Fig. 9). Similarly, Figure 10(b) shows the EDS spectrum of point 2(the blue square in Fig. 9). Figure 11 shows the EDS photo of the heated sample (S1) indicating the crystalline nature. Figure 12 shows the EDS spectrum of point 1(the orange square in Fig. 11). Figure 13 shows the EDS photo of the heated sample (S2) indicating the crystalline nature. Figure 14(a) shows the EDS spectrum of point 1(the orange square in Fig. 9). Similarly, Figure 14(b) shows the EDS spectrum of point 2(the blue square in Fig. 9).

IV. CONCLUSIONS

DSC analysis of the samples S1 and S2 showed that on heating the samples upto 1000^0C, a sharp peak was observed at 545^0C and 791^0C respectively, indicating a primary crystalline phase appearing in the amorphous matrix. In sample S2 another sharp peak was observed at 896^0C, indicating further crystallization in the sample. Both the samples are completely crystallized when temperature is above 929^0C. In sample S2, the crystallization is a multi stage process where different crystalline phases grow in the amorphous matrix. SEM patterns of these samples showed that as cast samples are amorphous and the sample are completely crystallized when heated to 1000^0C. EDS patterns also confirmed the complete crystallization of the samples after heating to 1000^0C. Thus increase of Tm concentration in Fe-Tm-B alloy changes the process of crystallization.

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