Amorphous to crystalline transformation and structural studies in amorphous $Fe_{78}Dy_2B_{20}$ and $Fe_{74}Dy_6B_{20}$ alloys

B. Bhanu Prasad

Department of Sciences & Humanities, M.V.S.R. Engineering College, Nadergul, Hyderabad

***Abstract*- Phase transformations and structural studies have been done on amorphous $Fe_{78}Dy_2B_{20}$ (S1) and $Fe_{74}Dy_6B_{20}$ (S2) alloys. Four Probe Electrical Resistivity (FPER), Differential Scanning Calorimetry (DSC) and X-Ray Diffraction (XRD) were used to characterize the samples. Resistance versus Temperature curve of the fresh sample $Fe_{78}Dy_2B_{20}$ (S1) showed a sudden drop around 800 K indicating the amorphous to crystalline transformation of the sample. The resistivity of the sample S1 at room temperature was found to be 244.3 μΩ-cm. The DSC curve of sample S1 showed an exothermic sharp peak around 551.6^{0}C (824.6 K) indicating the amorphous to crystalline transformation. Thus, sample S1 showed single step crystallization. Sample S2 showed multi step (growth of different phases) crystallization. XRD studies showed the presence of α-Fe and BCC Fe_3B phases in the completely crystallized S1 sample.**

Index terms- four probe electrical resistivity, differential scanning calorimetry, x-ray diffraction, phase transformation, room temperature, electrical resistance

INTRODUCTION

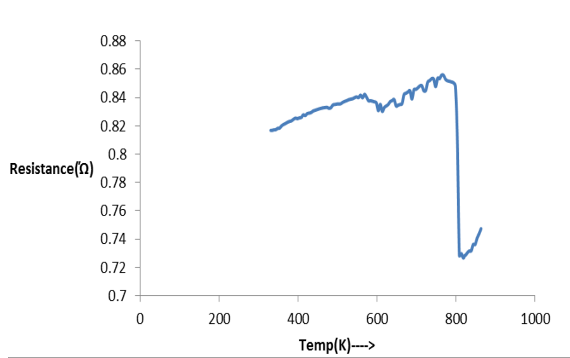
Some studies have been done on amorphous systems containing Transition Metal (TM), Rare-Earth (RE) and Metalloid Elements (ME), particularly with compositions such as (TM)80-x (RE)x (ME)20. Most of these show high saturation induction, high coercivity and reasonably high resistivity. These are used in making permanent magnets. The use of rare earth iron group alloys has increased significantly in a number of industrial fields over the past few decades. The demand of these magnets is increasing because these magnets are indispensable for high performance motors in electric vehicles. These magnets possess sufficient thermal stability for several more applications [1-4]. The thermal stability of these amorphous alloys is a subject of considerable interest, since the properties of these engineering materials may be significantly changed by the onset of crystallization and crystallization is associated with nucleation and growth.

In this paper, the temperature dependence of electrical resistivity, amorphous to crystalline transformation and structure of amorphous $Fe_{78}Dy_2B_{20}$ (S1) and $Fe_{74}Dy_6B_{20}$ (S2) alloys using FPER, DSC and XRD is discussed.

EXPERIMENTAL

Amorphous ribbons of $Fe_{78}Dy_2B_{20}$ (S1) and $Fe_{74}Dy_6B_{20}$ (S2) alloys are procured commercially which are prepared by the melt- spinning method. The ribbons were about 1 mm wide and about 30µm thick. The amorphous state of the ribbons was initially confirmed by taking XRD for the fresh samples. The Electrical Resistance of these alloys is measured as a function of temperature between 300 K and 850 K using FPER. DSC is used in the temperature range 300C - 10000C (heating rate 200C/min.) to study the crystallization behavior of the samples. XRD patterns were recorded for the completely crystallized samples at room temperature.

RESULTS AND DISCUSSION



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-273 -73 127 327 527 727
Temperature in Centigrade--------→

Figure 1 Variation of Resistance (Ω) with Temperature (K) of amorphous $Fe_{78}Dy_2B_{20}$(S1)

Figure 1 shows the resistance versus temperature curve of amorphous $Fe78Dy_2B_{20}$ (S1) in the temperature range 300 K – 900 K. In Fig. 1, the Electrical Resistance of the fresh sample S1 as a function of temperature shows a sharp drop in the resistance around 800 K, indicating the amorphous to crystalline transformation of the sample. The resistivity of the sample S1 at room temperature is found to be 244.3 μΩ-cm. The phase transformation studies have been done using Differential Scanning Calorimetry (DSC) in the temperature range 300C – 10000C. Figures 2 and 3 show the DSC curves of samples S1 and S2 for a heating rate of 200C/min. The DSC curve of sample S1 shows an exothermic sharp peak around 551.60C (824.6 K) indicating the amorphous to crystalline transformation which is reflected as a sharp drop in the resistance versus temperature curve of the fresh S1 sample. Thus, sample S1 shows single step crystallization. Sample S2 shows multi step (growth of different phases) crystallization. As shown in Table 1, the peak temperature (Tp) and the completion of crystallization temperature (Tx) of samples S1 and S2 are 824.6 K, 929.5 K, 950.6 K and 940.6 K, 995.0 K, respectively. Thus, as the concentration of Dy in Fe-B increases, the crystallization temperature increases.

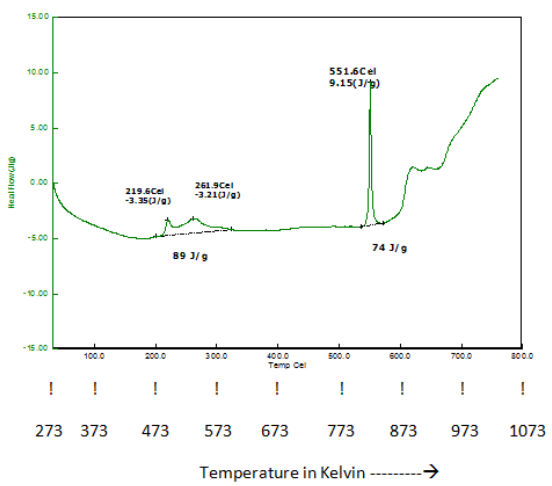

Figure 2 DSC curve of amorphous $Fe_{78}Dy_2B_{20}$(S1)

Figure 4 shows the XRD pattern of the fresh and the crystallized samples of S1. In Fig. 4, at $2\theta=46^0$ a sharp peak is observed in the crystallized S1 sample indicating a crystalline phase α-F e. In Fig. 4, there is another small peak around $2\theta=65^0$ indicating the growth of another phase BCC Fe_3B [5]. Thus, as the concentration of Dy in Fe-B increases the crystallization process becomes complex or multi-stage crystallization may take place where different phases may grow in addition to α–Fe. Table 2 shows the crystalline phases, lattice parameters, cell volume and crystallite size of the crystallized sample S1.

Table 1

Composition, Onset of Crystallization Temperature (T_{onset}) and Completion of Crystallization Crystallization Temperature ($T_{completion}$) of amorphous $Fe_{78}Dy_2B_{20}$ (S1) and $Fe_{74}Dy_6B_{20}$(S2) alloys

Composition	Peak Temperature T_p T (K)	Crystallization Temperaure T_x T (K)
$Fe_{78}Dy_2B_{20}$ (S1)	824.6	940.6
$Fe_{74}Dy_6B_{20}$ (S2)	929.5, 950.6	995.0

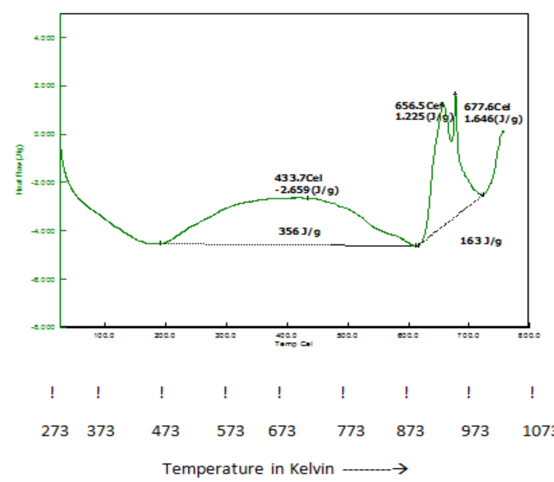

Figure 3 DSC curve of amorphous $Fe_{74}Dy_6B_{20}$(S2)

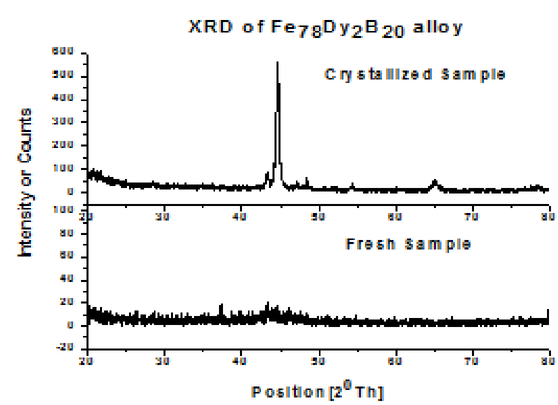

Figure 4 XRD pattern of amorphous and crystallized $Fe_{78}Dy_2B_{20}$(S1) recorded at room temperature

Table 2 Crystalline phases, lattice parameters, cell volume and crystallite size of crystallized $Fe_{78}Dy_2B_{20}$ alloy

Element	Phases obtained	2003 JCPDS Inter National Centre for diffraction data	Lattice parameter(A^0)			Cell Volume (A^0)	Crystallite size
		Card No.	aA^0	b A^0	c A^0		
Fe_{78} Dy_2B_{20}	Cubic α-Fe	65-4899	2.867			23.55	6.307 x10^{-10}m
	A small amount of orthorhombic FeB Phase	03-0957	4.053	5.495	2.946	65.611	
	A small amount of Tetragonal Fe_2B Phase	35-1340	8.62		4.27	317.27	
	A small amount of Tetragonal Fe_3B Phase	33-0644	8.62		4.27	317.27	

CONCLUSIONS

The resistance versus temperature curve of the given amorphous $Fe_{78}Dy_2B_{20}$(S1) alloy showed a drop around 800 K due to massive nucleation and growth of a primary crystalline phase. The crystallization temperature of the samples $Fe_{78}Dy_2B_{20}$(S1) and $Fe_{74}Dy_6B_{20}$(S2) increases with increase in the concentration of Dy in Fe-B. The sample S1 showed single-step crystallization. Sample with 6% Dy in Fe-B (S2) showed multi-step crystallization. From XRD studies, it is confirmed that in the completely crystallized sample S1, a sharp peak due the presence of α-Fe phase and small peaks due to the presence of other phases appear.

ACKNOWLEDGEMENTS

The author B. Bhanu Prasad acknowledge the encouragement given by the Management, the Principal, Head of Sciences & Humanities and Staff of M.V.S.R. Engineering College, Nadergul, Hyderabad in completing this work.

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