

## Thermal Stability of $\text{Fe}_{85.5}\text{B}_{14.5}$ Metallic Glass

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### Abstract

The thermal stability and crystallization behaviour of metallic glass  $\text{Fe}_{85.5}\text{B}_{14.5}$  were investigated by Dynamic Temperature Resistivity Measurement (DTRM) technique from room temperature to 1050K in forward and reverse mode at a heating rate of 40 K/hr. The slope of Resistivity-Temperature-Curve (RTC) changes sign and magnitude at the temperature where the transition takes place.

Differential Thermal Analysis (DTA) was carried out at the heating rates of 10, 20, 30 & 40 K/min. A comparison of the results of DTRM and DTA measurements shows that crystallization in this alloy is two-stage process. Activation energy for two crystallization steps was calculated from DTA data, using various peak shift equations. The activation energy for first step was found to be lower than the second.

### 1. Introduction

Liquid-quenched amorphous alloys, the so-called metallic glasses, have attracted much attention all over the world due to their interesting chemical, physical and mechanical properties. The technical use of these materials depends, however, on their thermal stability and crystallization behaviour. The elucidation of the mechanism by which amorphous to crystallization transition takes place has been of concern to materials scientists. To understand this phenomenon and the thermal stability of these alloys, a number of methods are commonly used.

In this communication, we report the thermal stability of binary metallic glass  $\text{Fe}_{85.5}\text{B}_{14.5}$  as studied by dynamic temperature resistivity measurement (DTRM) and DTA methods. The glass composition was chosen, because, no such work has been carried out so far.

### 2. Experimental Details

The glass ribbon of composition  $\text{Fe}_{85.5}\text{B}_{14.5}$ , about 15 mm thick and 10 mm wide

was prepared by melt quenching, using the outer side of a single-roller casting apparatus in Ruhr University, Bochum, Germany. The measurements of electrical resistivity was made on a 6 cm sample by four-point probe method at a constant heating rate of 40 K/hr. Nitrogen gas was continuously flowed throughout experiment to prevent the oxidation of the sample. The data were normalized with respect to the value at room temperature value ( $\rho_T/\rho_o$ ). Differential Thermal Analysis (DTA) measurements were performed in Argon atmosphere using Shimadzu Thermal Analyzer Dt-40. Alumina powder was used as the reference material. The data were recorded at heating rates of 10, 20, 30 and 40 K/min from room temperature to about 900K.

### 3. Results and Discussion

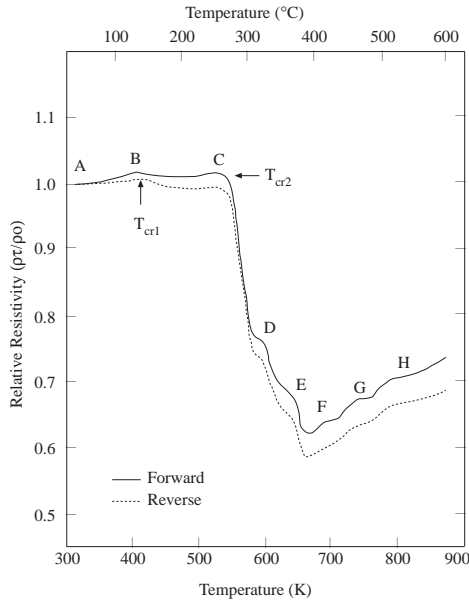
Figure 1 shows the graph between relative resistivity ( $\rho_T/\rho_o$ ) and temperature. The relative resistivity in this metallic glass increases with increasing temperature (point A), but when it reaches 413K (point B) it begins to decrease. This decrease continues up to 463K then it rises again up to 523K (point C). Beyond this it decreases sharply. The region from point A to C is suggested to be mostly amorphous and peak at point B (hereafter called first crystallization step i.e.  $T_{cr1}$ ) in this region is probably due to small amount of crystallization in the amorphous matrix. The  $T_{cr1}$  value observed by this method is comparable to, but slightly lower than, that measured by x-ray diffraction [1] (where diffraction lines, corresponding to a-Fe phase, appear in the amorphous halo). The sharp decrease at point C indicates the beginning of bulk crystallization (hereafter-called second crystallization i.e.  $T_{cr2}$ ). This  $T_{cr2}$  value is also in agreement with those obtained by x-ray diffraction, here the whole amorphous halo disappears and sharp diffraction lines appear. From this observation it is concluded that the crystallization in this glass sample is a two-stage process. This result is also evidenced from the work of Nakajima [2] on  $Fe_{86}B_{14}$  and  $Fe_{85}B_{15}$  amorphous alloys. They observed two exothermic peaks in DSC experiment on each alloy and two phases (a-Fe+amorphous and a-Fe+tetragonal  $Fe_3B$ ) in x-ray diffraction analysis. The DTA results in the present study also show two exothermic peaks at all heating rates. This again confirms the two stages of crystallization as observed in resistivity-temperature curve (RT-curve, Fig. 1).

The sudden fall of resistivity, which started from point C, continues up to 583K (point D) where a slight bend in its path occurs. The downward trend continues again up to 613K and another step occurs at 643K (point E), followed by a sharp decrease up to minimum value of RT-curve. This slight bend may be due to the formation of another phase as reported by Nakajima [2] to be  $Fe_2B$  in addition to a- $Fe_3B$ . The sharp decrease at point E is similar to our work [3] where splitting of  $Fe_3B$  takes place into  $\alpha$ -Fe and  $Fe_2B$ . Khan [1] also reported the formation of two phases ( $Fe_3B$  and  $Fe_2B$ ) at temperatures 570K and 580K, respectively.

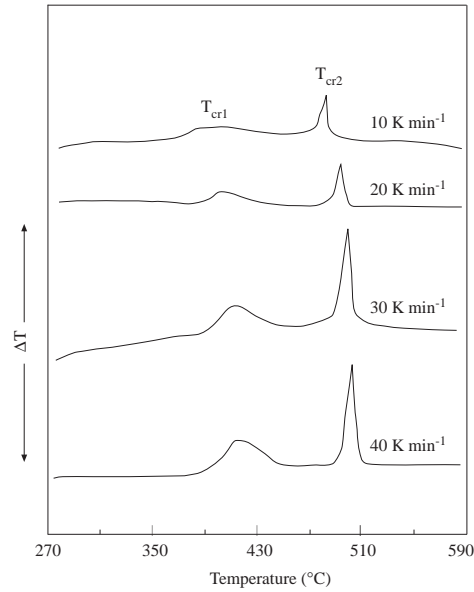
Further heating of glass, from minimum value onward, shows an increase in resistivity up to the last temperature. However, some humps are observed at points F, G and H as shown in Fig. 1. The reason for these humps is not yet clear. However, these might be due to some rearrangements in the crystalline phases not detected in x-ray work [1].

Similar results are observed in the reverse mode of resistivity measurements.

Figure 2 shows the DTA signals for the investigated glass at heating rates of 10, 20, 30 and 40 K/min. All the DTA curves show two exotherms corresponding to first ( $T_{cr1}$ ) and second ( $T_{cr2}$ ) crystallization stages. Using Kissinger, Augus-Bennett and Takhor equations [4], DTA data were plotted from the shift of DTA peaks at different heating rates (Fig. 3). Least-square-fit method was used for finding the slope of the curves, from which the apparent activation energies for both crystallization stages ( $T_{cr1}$  &  $T_{cr2}$ ) respectively, were calculated and are listed in Table 1. The lowering of activation energy for the first crystallization stage as compared to the second is due to the reduced driving force required for this type of reaction [5]. The values so obtained are comparable with those of the earlier investigation of Von Heimendahl [6].



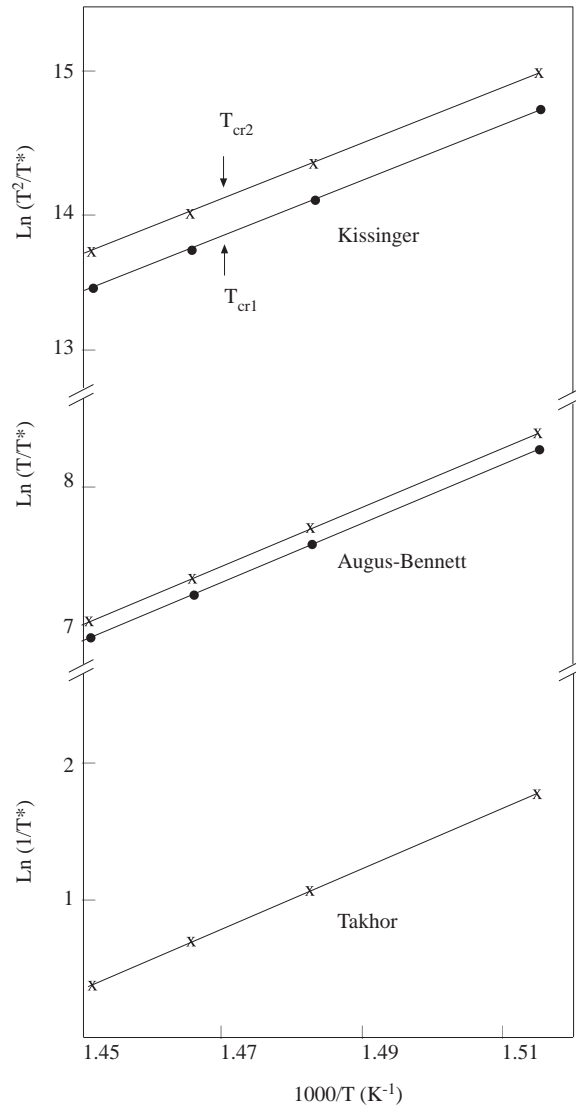
**Figure 1.** Relative resistivity change of metallic glass ribbon  $Fe_{85.5}B_{14.5}$  as a function of temperature at a heating rate of 40 K/hr.



**Figure 2.** DTA signals for  $Fe_{85.5}B_{14.5}$  metallic glass at different heating rates.

**Table 1.** Activation energy for first and second crystallization.

Method	Equation	Data	$Q (T_{cr1})$ kJ mol <sup>-1</sup>	$Q (T_{cr2})$ kJ mol <sup>-1</sup>
Kissinger[4]	$T^2/T^* = Ae^{-Q/RT}$	DTA	170±1	173±1
Augus-Bennett[4]	$T/T^* = Ae^{-Q/RT}$	DTA	176±1	178±1
Takhor[4]	$1/T^* = Ae^{-Q/RT}$	DTA	181±1	181±1
$T^* =$ Heating Rate	$Q =$ Activation Energy			



**Figure 3.** Kissinger, Augus-Bennett and Takhor plots of DTA peak-temperatures  $T_{cr1}$  and  $T_{cr2}$  for metallic glass  $Fe_{85.5}B_{14.5}$

#### 4. Conclusion

It is concluded that various steps present in the RT-curve correspond to various phase transitions. The two-stage crystallization process, detected in the present study, is also supported by findings of others by DSC and x-ray diffraction on similar type of alloys.

### Acknowledgment

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