DEVELOPMENT OF NANOCRYSTALLINE FeNbCuSiB BASED ALLOY FOR SENSOR APPLICATION

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ABSTRACT

The new generation of FeNbCuSiB based alloys were prepared in the form of ribbons by rapid quenching route using melt spinning technique. Differential scanning calorimetry (DSC) and thermal electrical resistivity (TER) measurements indicated the generation of nanophase from the as-cast amorphous state. The ribbons prepared at optimum quenching wheel velocity of 32m/s exhibited low brittleness with superior soft magnetic properties in the as-cast state. The superior quality ribbon sample indicated ultrasoft magnetic properties at an annealing temperature of 790K. This was due to the generation of nanoparticles as evidenced from the transmission electron microscopy (TEM). Potentiodynamic polarization studies showed that the material exhibited good corrosion resistive properties around optimum annealing temperature of 790K.

Key Words : Nanocrystalline, Rapid quenching, Potentio-dynamic, Polarisation, Sensor.

INTRODUCTION

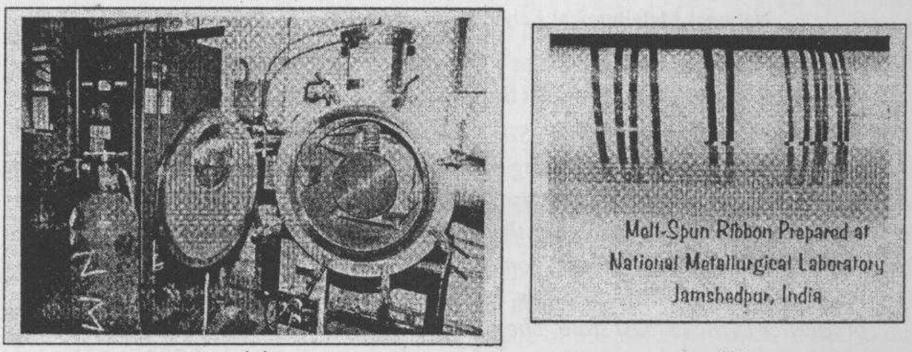
The classical soft magnetic materials primarily comprised of Si-steels and ferrites. With the advent of rapid solidification technology in early 70's, the Fe and Co-based amorphous alloys were introduced. However, these materials did not possess combination of advantageous properties like high saturation induction and high permeability, which are necessary for sensor application. Thus, in search of alloys having high saturation induction and permeability, a new generation of FeNbCuSiB based amorphous materials was introduced by Yoshizawa et al. [1]. These materials after optimum heat-treatment exhibited superior soft magnetic properties with coercivities less than lA/m, permeabilities of the order of 10^5 [2] and moderate saturation induction in their nanocrystalline state. Hence, the ultrasoft magnetic FeNbCuSiB based alloys which are generally prepared in the form of continuous amorphous ribbons by melt spinning technique have found applications in sensor cores, choke coils etc. As these materials are used as a sensor element in various application, therefore, they would be exposed to various hostile environments. Thus, their corrosion resistance properties need to be investigated.

The present work addresses the aspects of optimization of preparation process parameters and evaluation of magnetic and corrosion properties.

EXPERIMENTAL

Material preparation

The materials were prepared in the form of ribbons by melt spinning technique at National Metallurgical Laboratory, Jamshedpur. Figure 1a shows the photograph of the melt-spinner (Marko Materials, USA, Model: 2M) with a Cu-Be quenching wheel. The ribbons were prepared at wheel wheel velocity ranging between 27m/s to 40m/s. Long continuous ribbons were obtained as shown in figure 1b.



(a) (b)

Fig. 1 : Photograph of the (a) melt-spinning system (b) ribbon sample

Property evaluation

The crystallization behaviour of the alloys was carried out using Differential Scanning Calorimeter (Perkin-Elmer, DSC-7) and Thermal variation of electrical resistivity (Sinku-Riko, TER-2000) at a constant heating rate of lOK/min in an argon atmosphere. For magnetic property evaluation, coercivity and susceptibility was measured in open-flux configuration using Helmholtz coil. The microstructure of as-cast and heat-treated materials was observed using transmission electron microscope (Philips CM200). For the evaluation of mechanical property, fracture strength was measured [3]. The corrosion behaviour of the developed material was studied by potentiodynamic polarization studies using an electrolyte of NaCI solution in buffer media.

RESULTS AND DISCUSSION

DSC studies showed that crystallisation temperature changed with the wheel velocity. Figure 1a shows the representative DSC plots for two wheel surface velocities, Vs. Similar change in crystallisation temperature was also observed in resistivity study (Fig. 2b). The broad exothermic peak in DSC study indicated the transformation from amorphous to nanocrystalline phase. The rise in resistivity at the onset of crystallisation temperature was due to the scattering of electrons by the nanoparticles. The onset of crystallisation, T_x increased with the increase in quenching wheel velocity upto 32m/s, above which no further change in crystallization temperature was observed. The low onset temperature at low wheel velocity (<32m/s) was due to the low cooling rate that generated nucleation centers for the crystallisation in as-cast state. The results of the magnetic measurements for samples with different quenching rates in shown in Table-1. The decrease in coercivity and increase in susceptibility i.e., enhanced soft magnetic properties with increasing wheel velocity indicated the lesser long range ordering existed at higher quenching rate. The variation of fracture strength with the quenching rate is shown in Table-1. The materials prepared at high quenching rate had high amorphous volume fraction with lower crystallinity and lesser grain boundary as weak links that led to higher fracture strength i.e a more ductile ribbon.

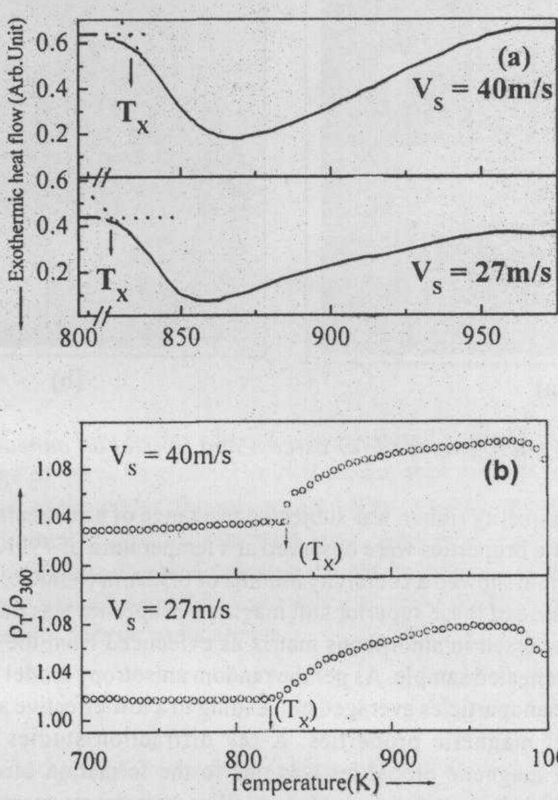
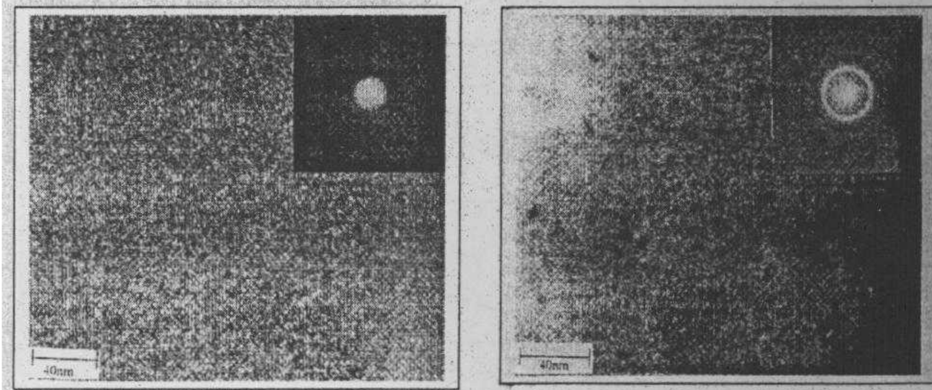


Fig. 2 : (a) DSC plots (b) Thermal electrical resistivity of ribbons prepared at different wheel velocities

Table-1: Effect of quenching wheel velocities on magnetic properties and fracture strain

Wheel Speed (m/s)	Ribbons Thickness ± 2 (μm)	Coercivity (Hc) (A/m)	Initial suscept. (χi)(X10^3)	Fracture Strain λ_f(X10^{-2})
27	36	3.73	4.82	0.54
32	34	1.75	6.52	0.94
36	33	2.07	7.02	1.11
40	28	1.88	8.59	1.13

The ribbons prepared at a quenching wheel speed of 32m/s exhibited not only low brittleness but also superior soft magnetic properties. The lesser pitting on the ribbon surface at this optimum wheel speed was the cause of such superiority in the properties. Therefore, ribbons prepared at 32m/s was considered for further investigation. The as-cast ribbon was in amorphous state as indicated by TEM micrograph shown in figure 3a.



(a) **(b)**

Fig. 3 : TEM micrographs of (a) as-cast and (b) ribbon annealed at 775K

The superior quality ribbon was subjected to a range of heat-treatment temperatures till ultrasoft magnetic properties were observed at a temperature of 790K. At this optimum temperature the sample showed a coercivity minima of 0.32A/m (~5mOe) and susceptibility of $\sim 2.1\times10^5$. The cause of these superior soft magnetic properties was due to the formation of nanoparticles dispersed in amorphous matrix as evidenced from the TEM micrograph of 775K (Fig.-3b) annealed sample. As per the random anisotropy model [4], the individual anisotropies of the nanoparticles averaged out leading to a low effective anisotropy thereby improving the soft magnetic properties. X-ray diffraction studies showed that the degradation in soft magnetic properties was due to the formation of crystalline phases like Fe-borides, which had strong magnetocrystalline anisotropy energy.

The corrosion behaviour of the prepared alloy was studied by electrochemical technique. The potentiodynamic polarization plots of as-cast and heat-treated ribbons are shown in figure 4. Curves (a) and (b) represent the electrochemical behaviour at two different molar strength of NaCl solution. At a low halide strength of 0.01M, the as-cast alloy figure 4a(i) showed a passivation tendency around +200mV(SCE) while at higher concentration of 1.0M such passivation tendency was absent from as-cast alloy figure 4a(ii). Annealing at 775K, the material was still passive at 0.01M halide concentration figure 4b(i) and the material also showed passivation tendency at 1.0M figure 4b(ii) of halide concentration indicating that the material was still better corrosion resistant than as-cast material. This annealing temperature (775K) was close to the optimum heat treatment temperature of 790K around which ultrasoft magnetic properties were obtained.

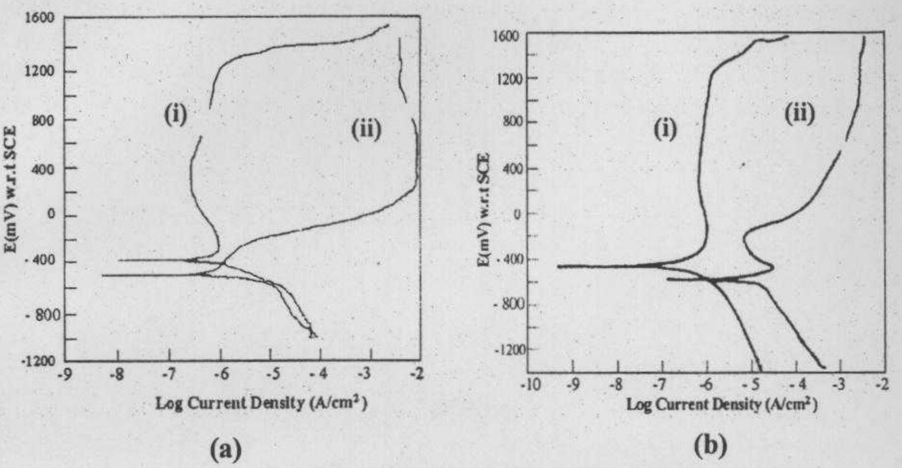


Fig. 4 : The potentiodynamic polarization plots of (a) as-cast and (b) heat-treated ribbon

CONCLUSIONS

The FeNbCuSiB based alloy ribbons were prepared in the form of ribbons by melt-spinning at a range of quenching wheel velocities. Superior soft magnetic properties and low brittleness were observed for ribbons prepared at an optimum quenching wheel velocity of 32m/s. The ultrasoft magnetic properties were generated at an optimum annealing temperature of 790K due to the formation of nanoparticles. Around this annealing temperature the material also exhibited superior corrosion resistance making the alloy a potential candidate for sensor applications.

REFERENCES

[1] *Y.Yoshizawa,S.Oguma,K.Yamauchi. J.Appl.phys 64 (1988) 6044*

[2] *G..Herzer."Nanocrystalline soft magnetic alloys" in Handbook of Magnetic materials,vol:10 edited by K.H.J.Buschow,1997.Elsevier Science B.V*

[3] *F.E.Luborsky, J.L.Walter, Mater.Sci.Engg 35(1978)255*

[4] *G.Herzer. IEEE Trans. Magn. 26 (1990) 1397*