The magnetic properties of martensitic phase in Fe–Ni–Mn alloys: Mössbauer spectroscopy observation

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It has been well established that martensitic phase transformation occurs athermally, however, it also forms isothermally in some Fe-based alloys below room temperature. Apart from kinetic differences of the transformation, morphologies of the martensite phases also differ [1]. A strong relation between austenite— martensite phase transformation and its magnetic behavior in Fe based alloys can be established. Despite the paramagnetic behavior of austenite, martensite phase can show either ferromagnetic or antiferromagnetic behavior [2, 3]. The physical properties of isothermal and athermal α (bcc)—martensite in Fe–Ni–Mn alloys have been extensively studied using different characterisation techniques [4–10].

Mössbauer spectroscopy is one of the widely used methods to study the phase transition behavior in metals and alloys. With this method, the magnetic ordering of austenite and martensite phases, volume fraction of martensite, dependent on various external factors, and its internal magnetic field can be accurately determined by the resonance absorption effect created by gamma rays which are generated by a change in energy levels of atomic nuclei. The internal magnetic field affected by neighboring atoms can be determined in metals and alloys. In additon, distortion in crystal lattice and electron orbits causing potential gradient and the isomer shift responsible for the interaction occuring between the nucleus and *s*-electrons can also be determined by this method. Information on short-range atomic interaction in metals and alloys can be consequently derived [1]. Mössbauer spectroscopy provides the absorption areas for different components corresponding to the austenite and the martensite phases in Fe-based alloys. Generally, the area fraction for *i*-th component, F_i, can be expressed by, where A is the total resonance area, A_i the resonance area of *i*-th phase, n_i the number fraction of Fe atoms, and f_i the Debye-Waller factor for *i*-th phase. Accordingly, if one could assume that the Debye-Waller factors are equal in all phases, or if one could obtain the Debye-Waller factors, one could estimate the number fractions, which may be used to determine the volume fractions taking into account a

correction due to the Fe densities in the different phases [1, 11].

In literature, the characteristics of athermal and isothermal martensitic phase transformations have been observed using different techniques in Fe–Ni–Mn alloys [4–10]. In some Fe based alloys, austenite phase exhibits paramagnetic behavior, which is shown by a single absorption line spectrum whereas martensite phase shows either an antiferromagnetic or ferromagnetic behavior, which is characterized by six absorption lines [12, 13]. Although there are several Mössbauer studies on athermal martensitic transformation in this alloy, there is no work reported on an isothermal martensite yet. Therefore, the present study was aimed to compare the magnetic properties of athermal and isothermal martensitic products formed in an Fe–Ni–Mn alloy, by using the Mössbauer spectroscopy technique.

Fe–Ni–Mn alloys were prepared by vacuum induction melting. Samples were austenized at 1100 °C in vacuum for 12 h and furnace cooled to room temperature. Martensite was formed by subzero cooling in liquid nitrogen. Thin foils for Mössbauer spectroscopy were prepared by mechanical and chemical thinning procedures. The Mössbauer spectroscopy was carried out by using a 50 m Ci ^{57}Co source diffused in Rh. Thin foils for transmission electron microscope (TEM) observations were electropolished by using a double-jet polishing technique with a solution of 10 ml of 2-butoxy ethanol, 50 ml of perchloric acid and 300 ml of ethanol, using 35–40 V (DC) at room temperature. Microscopy was performed using a JEOL 3010 TEM operated at 300 kV.

TEM observations showed that the Fe-%25Ni-%5Mn alloy is austenite at room temperature and it exhibits both athermal and isothermal martensitic transformation below room temparature. However, there is no phase transformation in Fe-31.5%Ni-10%Mn alloy. In this study, Mössbauer spectra of austenite Fe-%25Ni-%5Mn and Fe-31.5%Ni-10%Mn; mössbauer spectra of athermal and isothermal martensite phases and volume fraction of martensite and

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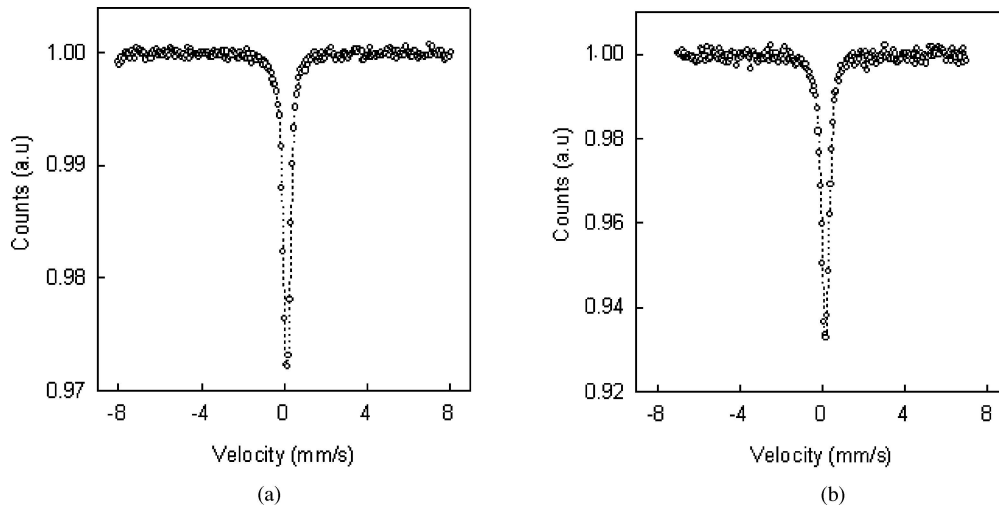* Author to whom all correspondence should be addressed.

Figure 1 Mössbauer spectra of austenitic (a) Fe-%31.5Ni-%10Mn, and (b) Fe-%25Ni-%5Mn thin foil.
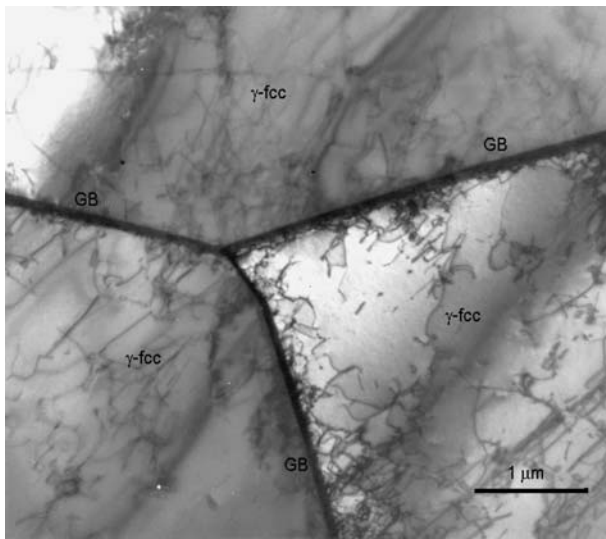

Figure 2 Transmission electron micrograph showing austenite structure with the dislocations (GB refers to grain baundary).

retained austenite in an Fe-%25Ni-%5Mn alloy are presented. The internal hyperfine magnetic field of the martensite and isomery shift of phases were also observed in Fe-%25Ni-%5Mn alloy.

Austenitic Fe-25%Ni-5%Mn and Fe-%31.5Ni-%10Mn alloys are characterized by a single absorption line spectrum because of their paramagnetic nature as presented in Fig. 1. A TEM micrograph of grain boundaries in an austenitic sample with the dislocations in a Fe-25%Ni-5%Mn alloy is also shown Fig. 2. In Fe-25%-5%Mn alloy, an athermal type of martensitic transformation was observed at around $-140\,^{\circ}$C in the austenitic structure. A TEM study of this microstructure was carried out in order to define its morphology. The reverse quenching experiment, holding at liquid nitrogen temperature and immediately up quenching into boiling water, indicated athermal transformation in this alloy. A change from paramagnetic austenite phase to ferromagnetic or antiferromagnetic athermal martensite phase is characterized by six absorption lines and retained austenite spectrums of Fe-%25Ni-%5Mn as shown in Fig. 3, which was found to contain 20.34% of transformed martensite on examination of spectrum lines. The hyperfine internal magnetic field of the athermal martensite was found to be 32.99 Tesla and also the wedge shape athermal martensites are shown Fig. 4. For isothermal martensitic transformation, the specimen was subjected to liquid nitrogen treatment for 50 hr at $-196\,^{\circ}$C and Mössbauer spectra were
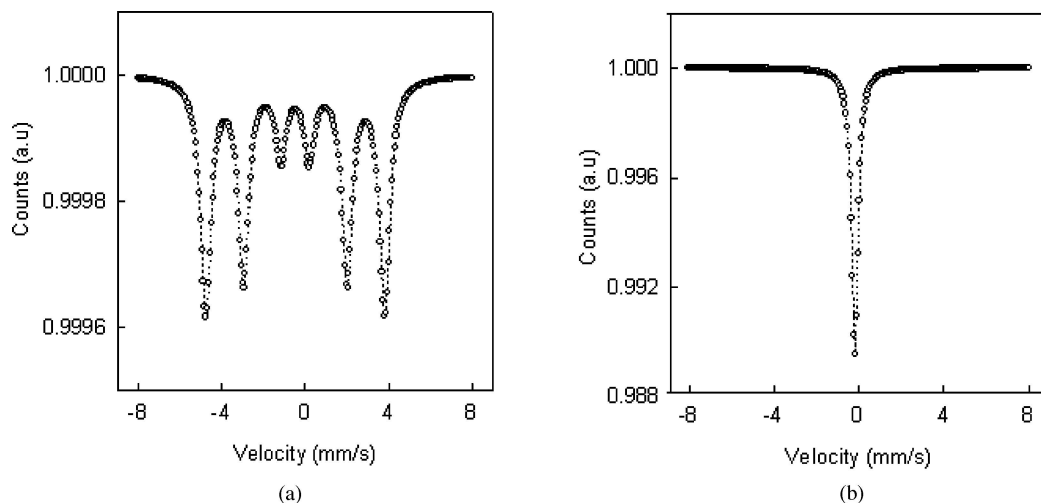

Figure 3 Mössbauer spectra of Fe-%25Ni-%5Mn thin foil (a) athermal martensite and (b) retained austenite.
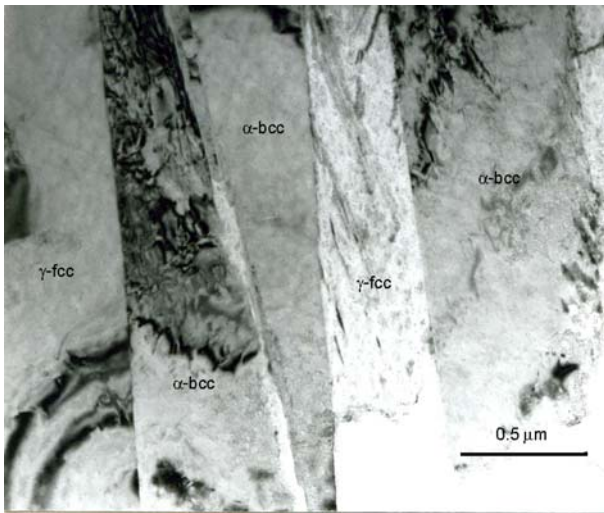
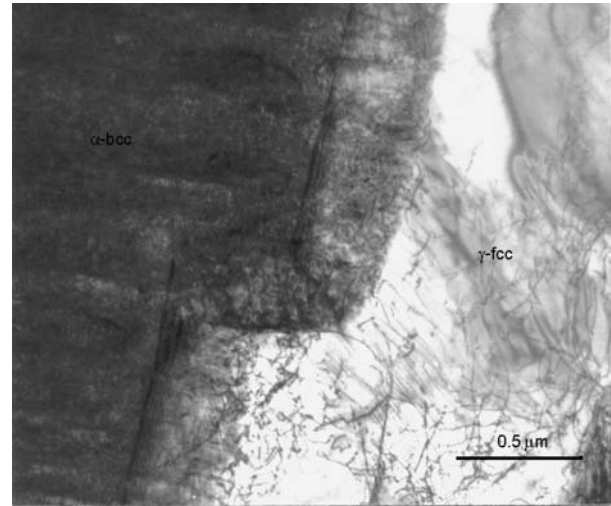*Figure 4* Transmission electron micrograph showing athermal martensite plates formed in the austenite matrix.

Figure 6 Transmission electron micrograph showing isothermal martensite plates formed in the austenite matrix (α-bcc refers to isothermal martensite; γ-fcc refers to austenite).

obtained for these conditions at room temperature, which is given in Fig. 5. On examination of the curves, it was found that the specimen treated isothermally for 50 hr at liquid nitrogen temperature yields 5.62% martensite phase and the internal hyperfine magnetic field of the martensite phase is 31.94 T. The martensite and retained austenite percentages of these samples were determined and are given in Table I with the calculated isomery shifts of austenite and martensite peaks and also the internal magnetic fields of the martensite phase. Fig. 6 shows a TEM micrograph of

isothermal martensite formed with the dislocations of the matrix austenite at room temperature.

In conclusion, Mössbauer spectroscopy was used to determine percentages of both athermal and isothermal martensite formed in austenite phase. It was found that the observed difference in the athermal and isothermal martensite volume changes and hyperfine internal magnetic fields can be explained in terms of nucleation and growth behavior of Fe-25%Ni-5%Mn alloy. Isothermal heat treatment for a long period of time creates high density of dislocations, as in athermal process,

TABLE I Mössbauer spectroscopy results of austenite, martensite and retained austenite phases

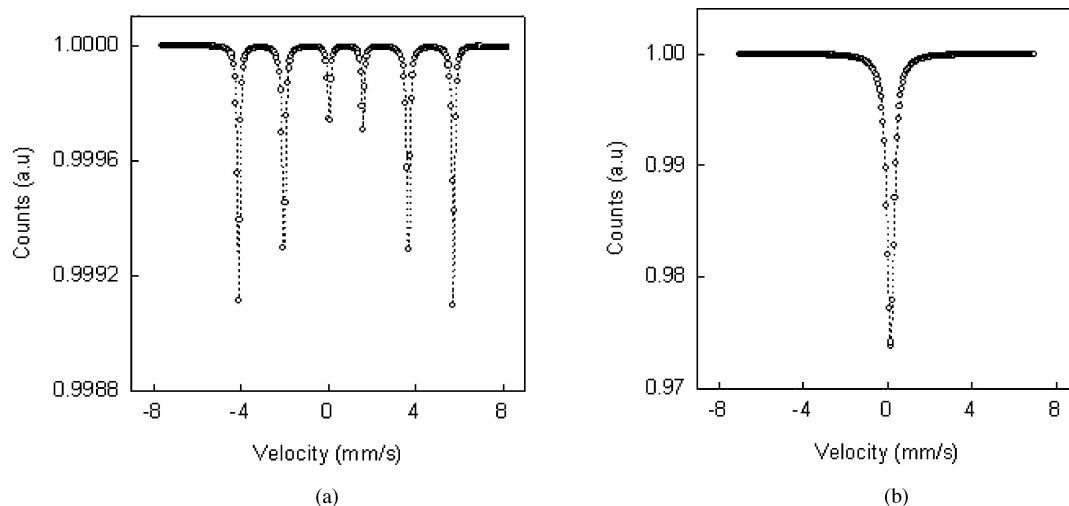| | %Austenite | %Martensite | Isomery shifts (mm/s) | | Internal magnetic field (T) |
			Austenite	Martensite	
Fe-%31.5Ni-%10Mn	100	–	$0.14 \pm 0.11 \times 10^{-2}$	–	–
Fe-%25Ni-%5Mn	100	–	$0.16 \pm 0.18 \times 10^{-2}$	–	–
Fe-%25Ni-%5Mn Athermally	79.66	20.34	$-0.17 \pm 0.10 \times 10^{-2}$	$-0.14 \pm 0.10 \times 10^{-2}$	32.99
Fe-%25Ni-%5Mn Isothermally-50 h at $-196\,^{\circ}C$	94.38	5.62	$0.15 \pm 0.10 \times 10^{-2}$	$0.20 \pm 0.10 \times 10^{-2}$	31.94

(a)

(b)

Figure 5 Mössbauer spectra of Fe-%25Ni-%5Mn thin foil: (a) isothermal martensite and (b) retained austenite.

and those dislocations could assist the nucleation of martensite, in that dislocations produce initial martensite embrios for isothermal martensitic transformation. And also, unlike austenite, which has a paramagnetic behavior, martensite phases showed ferromagnetic or antiferromagnetic behavior.

Acknowledgments

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