



Structural and Magnetic Study of Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu Alloys

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Abstract

In this study, the microstructure and magnetic properties of martensite transformation induced were investigated in Fe based alloys (Fe-15,83%Mn-2,18%V ve Fe-18,50%Mn-2,27%Cu alloys). Micro structures were studied with Scanning Electron Microscope. The effect of the adding V and Cu in Fe-Mn alloy on the magnetic properties has been investigated by Mössbauer spectroscopy technique respectively. It was investigated that the adding of V and Cu to Fe-Mn alloy reveals the ferromagnetic and paramagnetic properties of alloy respectively. The other result of this study is the rate of Mn amount in Fe-Mn-X alloy. If the Mn rate in the Fe-Mn-X alloy is 18% or more, no α -martensitic transformation have been observed. Thus, this study showed that the additional atom has great importance on changing the magnetic properties of alloys.

Key Words

“Phase transformation, Magnetic properties, Mössbauer spectroscopy, Scanning electron microscope”

1. INTRODUCTION

Metal alloys have become materials that make life easier for human beings. Since the 19th century, Fe-based alloys have attracted the attention of material science. First studies have began in 1861 with Henry Sorby and continued with the German scientist Adolf Martens. The transformations in which the crystal structure changes with any physical effect and the neighborhoods of atoms in metal alloys are unchanged are called martensitic transformations. Adolf Martens first observed and is referred to by his name [Nishiyama(1978)]. Cotes et al.(2002) studied the magnetic properties of the Fe-13,7%Mn alloy with X-ray diffraction (XRD) and Mössbauer spectrometry(MS). The addition of third element to the Fe-Mn alloys because influences significantly their several physical and mechanical properties, Fe-Mn-X alloys have been intensively studied.

In 1998, J. H. Jun et al. studied the microstructural properties of Fe-Mn-Co alloys and their dependence on the martensitic phase transformation of the mechanical energetic vibration enhancement of the heat treatment by the material. In 2003, P. Marinelli et al. studied martensitic transformations in Fe-Mn-Co alloys depending on the temperature. Sarı et al. investigated the effect of martensitic transformation and magnetic properties of Mo and Co on Fe-Mn alloys. As a result of the studies, γ (fcc) \rightarrow ϵ (hcp) phase transformation occurring in Fe-Mn based alloys examined how the percentage of the added element affects the transformation and the defects occurring in the alloy. In this paper we study the magnetic properties and Austenite-Martensite Phase Transformation Fe-Mn-Cu and Fe-Mn-V alloys in the the experimental techniques of X-Ray diffraction (XRD), Mössbauer spectroscopy(MS) and scanning electron microscopy (SEM).

2. METHODS

In this study, Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu alloys were prepared at TUBITAK Gebze Research Center in Turkey. Fe, Mn, V and Fe, Mn, Cu elements which are in powder form in 99.9% purity are put together and melted at high temperature and poured into cylindrical rods of 1 cm diameter and 10 cm length. The composition of the alloys obtained in this way were obtained as Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu (% weight) by EDS (Electron Dispersion Spectroscopy) technique using IXRF system. Special slices from the ingots alloys were cut by using diamond saw. The samples were sealed into quartz tubes and then heat treated in the austenite γ - phase equilibrium region. Alloys were homogenized at 1100 °C for 12 hours and then subjected to slow cooling in the furnace.

For scanning electron microscopy (SEM) observations, the surfaces of the samples were first mechanically polished and afterwards the damaged surface layers were eliminated by etching in a solution composed of 40%HCL, 30%C₃H₈O₃ and 30%HNO₃ for 40 s. The microstructure characteristics of samples surfaces were investigated by SEM of Jeol - JSM 5600 at 30 kV.

Mössbauer spectroscopy was applied to study the magnetism and volume fractions of both the austenite and martensity phases. Specimens examined by SEM were used for Mössbauer spectroscopy measurements at room temperature. Then, the Mössbauer spectra values of these samples were obtained using a Normos-90 computer program. Finally, XRD spectrum of the samples were measured by X-ray diffraction method.

3. RESULTS

3.1. Investigation of the Martensite Structure Formed in Fe-15,83%Mn-2,18%V and Fe- 18,50%Mn-2,27%Cu Alloys by Scanning Electron Microscopy (SEM)

Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu alloys were homogenized at 1100 °C for 12 hours and subjected to slow cooling process in the furnace. In the sample studied by SEM, ϵ bands were observed in γ phase with thermal effect. The structure observed in any region were carried out in Fig.1.

By examining the SEM image of the Fe-15,83%Mn-2,18%V alloy, it has been easily seen that the formed martensitic layers begin at the grain boundaries and finish at grain boundaries and have different orientations. The α' and ϵ bands in this structure are shown in Fig.1. Askeland (2003) noted that the atomic sequences within these Alloys, which are observed as the result of slow cooling, are identical, and that the orientation of atoms in each of these particles is different. SEM image of Fe-18,50%Mn-2,27%Cu alloy is show in Fig.2.

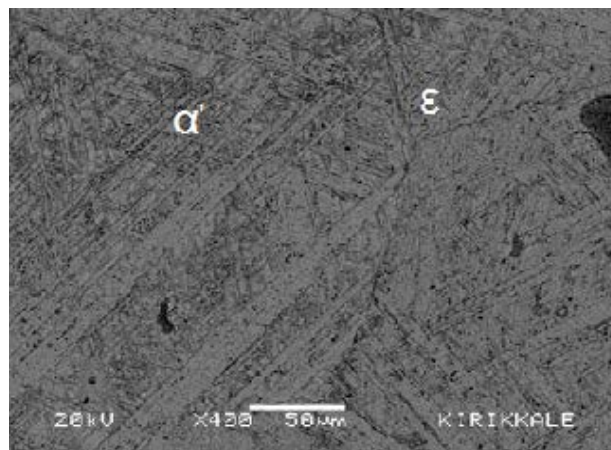


Fig. 1. SEM image of Fe-15,83%Mn-2,18%V alloy

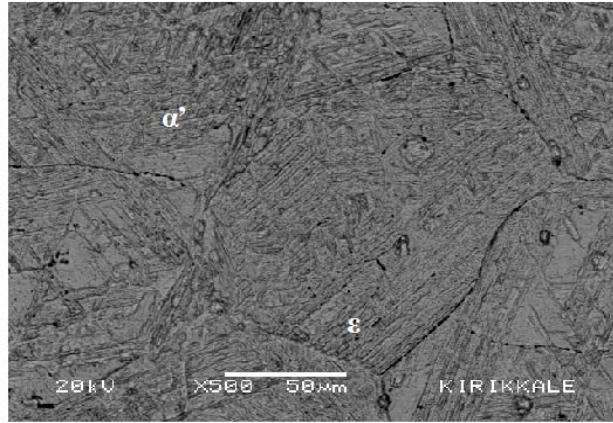


Fig. 2. SEM image of Fe-18,50%Mn-2,27%Cu alloy

By examining Fig.2, it has been observed that the slow-cooling samples were piled parallel to each other and formed ϵ martensite plates with different orientations. It was stated by Porter et al.(2009) that the growth of martensite plates is inhibited at the grain boundaries and that the number of martensite nuclei has no effect on the grain size, but the shape and size of the formed martensite plates is a function of the grain size. ϵ martensite plates have been introduced in Cotes et al.(2004) and Kajivara (1984) studies in which they form parallel plates in the grain. In the intersection regions of the martensite plates, a formation of α' (bcc) martensite is possible.

3.2. Investigation of Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu Alloys by X-ray Diffraction Method

The X-ray diffraction system is a suitable experimental method for investigating the properties of solid inorganic and crystalline materials, which are mostly composed of heavy elements. For X-ray diffraction measurements, 20 mg dust samples were prepared from the alloys. A X-ray beam of λ wavelength is sent onto this dust sample. So, angle and intensity values given in accordance with Bragg diffraction are poured on a graph to obtain intensity peaks corresponding to 2θ angles. After structure analysis is carried out by determining the planes providing these peaks (hkl). With this method, crystal lattice parameters, phase structures occurring at different temperatures and different phase components can be revealed easily. The results obtained for Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu alloys are given in Fig.3 and Fig.4.

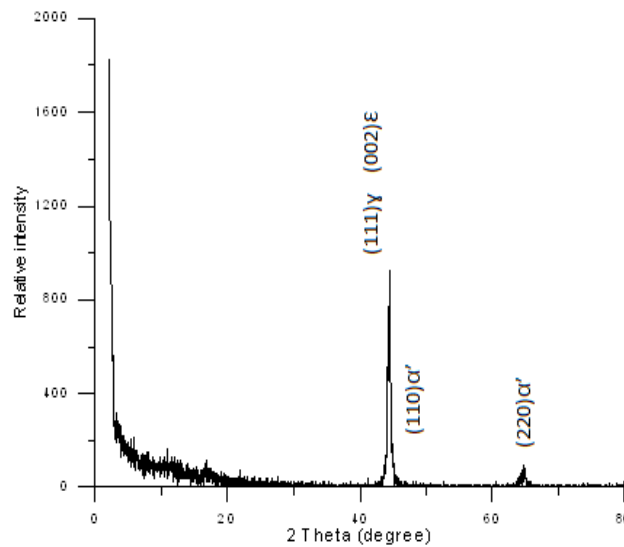


Fig. 3. X-ray diffraction pattern of Fe-15.83%Mn-2.18%V alloy

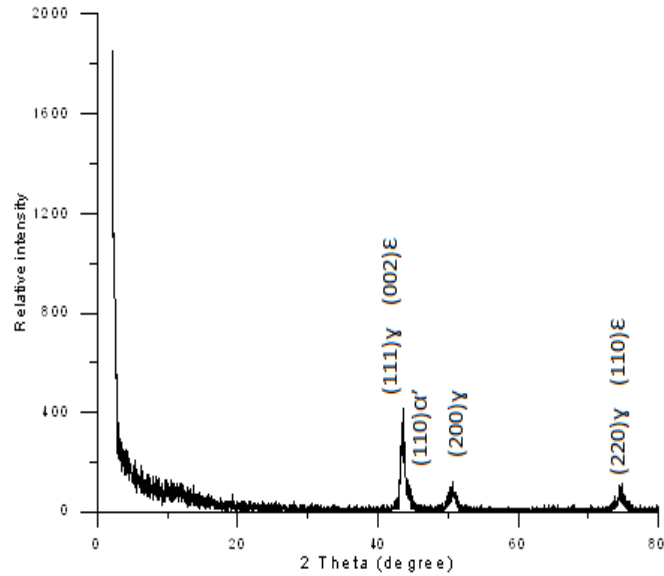


Fig. 4. X-ray diffraction pattern of Fe-18,50%Mn-2,27%Cu alloy

Comparing the X-ray diffraction graphs, the (111) γ and (002) ϵ peak of the Fe- 15,83%Mn-2,18%V alloy at $2\theta = 44,421^\circ$ are more intense than the same peaks in Fe-18,50%Mn-2,27%Cu alloy. In addition to these; sample alloy at $2\theta = 65^\circ$, there is α' peak. Unlike the other samples of Fe-18,50%Mn-2,27%Cu alloy, there are peaks at $2\theta = 50,62^\circ$ (200) γ and $2\theta = 74,80^\circ$ (220) γ , (110) ϵ . The lattice parameters α' (bcc) of the Fe-15,83% Mn-2,18 %V and Fe-18,50%Mn-2,27%Cu alloys are $a = 0,28406$ nm, $a = 0,28743$ nm, respectively.

3.3. Investigation of Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu Alloys by Mössbauer Spectroscopy Method

Mössbauer spectrometry is widely used in studying phase transformations of metal and metal alloys. With this method, the magnetic properties of austenite and martensite structures, the volume fractions of martensite crystals formed by different effects, and the internal magnetic field of the martensite phase can be determined. The Mössbauer spectra taken to determine the magnetic properties of the martensite structure observed in Fe-15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu alloys are given in Fig.5 and Fig.6.

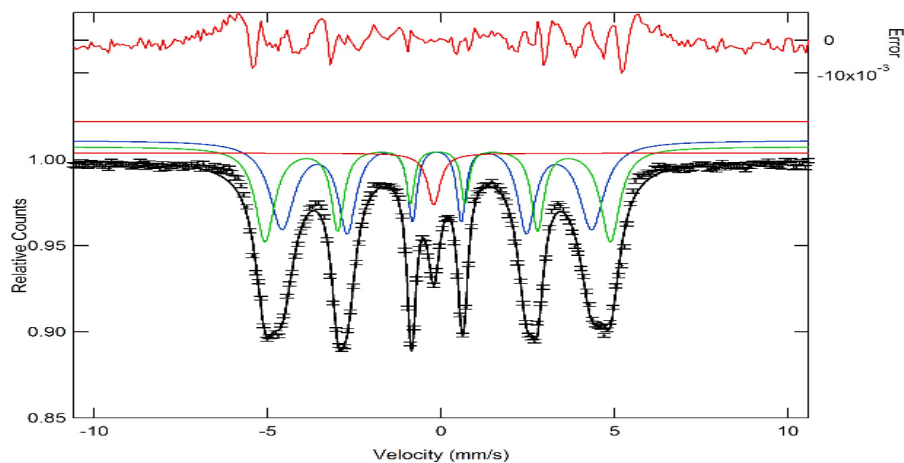


Fig. 5. Mössbauer spectrum of Fe-15,83%Mn-2,18%V alloy

As seen in the Fig.5, Fe-15,83%Mn-2,18%V alloy gives 6 (six) peaks. It is well known in Mössbauer spectroscopy that these six peaks indicates that the material represent ferromagnetic properties (or antiferromagnetic). In the studies Cotes et al.(2002) and Sumiyama et al. (1981) carried out, although the austenite (γ) and martensite (ϵ) phases of Fe-Mn alloys generally showed paramagnetic properties, α' phase is ferromagnetic. Therefore, the sextets belong to α' martensite phase in Fig.5.

In Fe-based alloys, the austenite structure is usually paramagnetic, and this phase is characterized by a single absorption line. Austenite structure shows paramagnetic properties in Fe-18,50%Mn-2,27%Cu alloy in Fig.6.

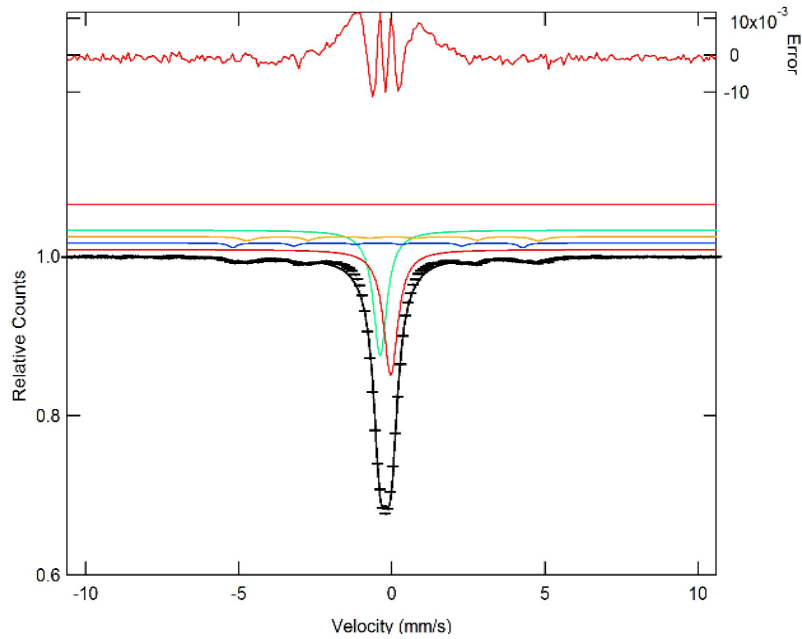


Fig. 6. Mössbauer Spectrum of Fe-18,50Mn-2,27Cu alloy

According to Mössbauer results, we have found out that these two alloys have two sextets and singlets as shown by Mijovilovich et al. (2000).

The formation percentages of austenite and martensite structures obtained by Mössbauer spectrometry method, hyperfine magnetic field values of martensite structure and isomer shifts values of austenite and martensite phases are given in Table 1.

Table 1. Some Mössbauer parameters of alloys

| Samples | Structure | H_{eff} (T)(± 0.1) | LS.(mms ¹)(± 0.004) | W(mm s ⁻¹)(± 0.02) | Area (%) |
|-------------------------|------------|--------------------------------------|---------------------------------------|--------------------------------------|----------|
| Fe-%15,83Mn- %2,18V | Austenite | - | -0.1053 | 0.4482 | 6.015 |
| | Sextet 1 | 30.862 | 0.0051 | 0.2794 | 36.871 |
| | Sextet 2 | 27.671 | -0.0185 | 0.3222 | 57.114 |
| Fe-%18,50Mn- %2,27Cu | Austenite1 | - | 0.0734 | 0.5648 | 48.936 |
| | Austenite2 | - | -0.2717 | 0.5353 | 43.846 |
| | Sextet 1 | 29.498 | 0.1457 | 0.5177 | 5.1797 |
| | Sextet 2 | 29.357 | -0.3478 | 0.3084 | 2.0384 |

When the results obtained from the Mössbauer spectrometer in Table 1 are evaluated, with increasing amount of martensite formed, the internal magnetic field value (H_{eff}) changes. The values of these parameters, determined from the Mössbauer spectroscopy, are directly dependent on the Fe core in the absorber. The values of the internal magnetic field in the Fe core are changes greatly depending on the number and distance of the atoms neighboring to the Fe core. As Sarı et al. (2009) and Cotes et al.(2002) demonstrated each Fe atom neighboring to the Fe core is raising the internal magnetic field value of the Fe core; Fe atoms (C, Ni, Mn, Al, Cr) which are not adjacent to the Fe core, reduces the value of the internal magnetic field.

4. DISCUSSION AND CONCLUSION

In this study, crystallographic and magnetic properties of martensite crystals formed in Fe- 15,83%Mn-2,18%V and Fe-18,50%Mn-2,27%Cu alloys were investigated. Occuring microstructural changes were investigated by scanning electron microscopy (SEM) and it was found that martensite transformation occurred. In the SEM analysis shown in Fig. 1 and Fig.2, the ϵ martensite was formed as parallel plates and ended at the grain boundary. The austenite grains are clearly visible in alloys. The grain sizes of Fe-15,83%Mn-2,18%V and Fe- 18,50%Mn-2,27%Cu alloys are different from each other. The results of the Mössbauer studies show that the martensite formation is greater in the Fe-15.83% Mn-2.18 % V alloy than in the Fe-18.50% Mn-2.27% Cu alloy. This result is also compatible with the SEM observations. In the Mössbauer observation, Fe-15,83%Mn-2,18%V alloy gives six peaks showing ferromagnetic character. The phase formed in Fe-18,50%Mn-2,27%Cu alloy shows a paramagnetic (single peak) property. Fe-18,50%Mn-2,27%Cu alloy have a low martensite transformation, indicating that the element of Cu is an element stabilizing element. In Sarı&Kırındı (2011) studies have shown that α -martensite transformation was not observed when the Mn rate in Fe-Mn-X alloys were more than 18%. Therefore, the results in this study are consistent with the literature studies.

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