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Effect of Annealing Treatment Processes on Structural and Magnetic Properties for Some Invar Alloys

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Abstract

Invar alloys Fe-Ni content of 30% - 75% Ni were prepared by melting Iron and Nickel metal using an induction furnace. The crystal structure, grain size and lattice constant of the Fe-Ni alloy were studied by X-ray diffraction technique. The chemical composition and surface morphology of the F.c.c of Invar alloys were investigated by using Scanning electron Microscopy (SEM). Measurements of the saturation magnetization, coercivity, remnant ratio and magneto number were deduced from hysteresis curve. Vibrating-sample magnetometer (VSM) was used to investigate the effect of heat treatment on the area of hysteresis loop. The behavior of the hysteresis loop with annealing treatment due to the change in short-rang order, this change is related to atomic rearrangement such as short-range order. The change in the Curie temperature T_c as the function of the time demonstrated the similar behavior as in metallic glasses state, and was interpreted as a rearrangement atomic in the primitive cell.

Keywords: susceptibility, annealing treatment, Invar alloy, Curie temperature (Tc)

1. INTRODUCTION

Characterization of Fe-Ni alloys around the Invar composition of 30%, 50% and 75% Ni demonstrated a various anomalies in the physical properties [1]. Like larges magneto-striction, extremely low thermal expansion etc. that was explained as a major positive values of the magnetic volume with the coupling constant, therefore showed the variety of the interest from 3d-electron ferromagnetism material to many techniques applications [1]. Although much theoretical and experimental research effort, the Invar problems has been not completely understand. To enhance diffusion processes a specific technique has been used at low temperature in Fe30Ni70 alloy, for allowed the research of the system state to reach the equilibrium condition of the thermo dynamic in reasonable time, such special techniques are ion irradiation [2], neutron irradiation [3]. Indicate of the atomic inhomogeneity in Invar alloys Fe79Ni21 is produced from MÖssbauer measurements [4]. In a recent work [5] the increase of the line width showed in the spectra of MÖssbaure pecks for Ni70Ni30 alloy was annealing by high pressure processes. The reason of the increase as a result of the renew of the atomic rearrangement; in the other hand that cause another things such the short-range order [S.R.O] the two behaviors which agree with others researches [6].

These structure of atomic rearrangements states are considers as a result of the change in Curie temperature (Tc) for Fe-Ni alloys yields at different alloys when the irradiated with electrons and ions, for such compositions containing % Ni with concentrations between (30 and 50) [7]. For the Fe30Ni70 amorphous compound alloys which were subjected to the thermal treatments that processes cause increasing in the Curie temperature (Tc) approximately to 35°C were showed at low temperature, these explained as a result of locally rearrangement of the lattice atoms in the alloys [8]. On the new researches theoretical calculations for Fe-Ni alloys [9] demonstrate important change in magnetic properties as a result of local environment effects. The anomalies of Invar alloy has been interpreted as a result of the instability of the 3d-band ferromagnetism in f.c.c materials and alloy [10]. Result to found a sharp peak for the 3d-electron band at the top in f.c.c phase, which indicate, the ferromagnetic properties become unstable as a result of the decreasing in the number of the electrons in the outer shells according to the decreasing of the concentration Ni element when approach to 29 % [20].

Fe-Ni Invar alloys phase are crystal structure on the other hand if the concentration of Ni decrease below 29 at %, in that case the martensitic transformation occurs ie (γ Fcc and α bcc) as a result of boundary (FM) Invar to AF Invar order which occurs

For that things many crystallographic studies and local lattice distortion had been done [11]. In this work we intended to determine the structural change and alloys properties, such as Mc, magnetic susceptibility of $Ni_{30}Fe_{70}$ obtained by applied magnetic field at room temperature (RT) after annealing processes at 700 C° and by using fast thermal processing accompanied with rapid cooling rate. We have also determined the martensitic transformation of α phase and γ phase transition of $Ni_{30}Fe_{70}$ alloy with properties including Curie temperature (Tc). Martensitic transition (γ-α) in magnetic alloys which determines the Fermi levels position of the Invar alloy whether FM-Invar or anti-Invar (in a few alloys with the AF-Invar together martensitic do occur, attributed as a result of transition from FM (high Spin Hs) to AF (low Spin Ls) [16].

2. EXPERIMENTAL PROCEDURE

The (VSM) was used to investigate the effect of heat treatment on Fe-Ni Invar alloys of composition (30%, 50% and 75% Ni). In this region the magnetic properties change very rapidly with Ni concentration and are very sensitive to the atomic arrangement. The behavior of the hysteresis loop in Fe-Ni alloys has been investigated for different states of annealing.

Investigate of morphology of the Fe-Ni samples and measured the particle size distribution had been done by using Scanning Electron Microscopy (SEM)

The experimental dependences of the ferromagnetic susceptibility of antiferromagnetic close to the critical temperature Below T_c, the behavior of the susceptibility depends on the direction of the applied field whether is perpendicular or parallel to the direction of sub lattice magnetization. For the case of antiferromagnetic as isotropic would not be has a similar case: when the applied field effect on the sub-lattice the produce magnetization would rotate in a favorable energetically orientation with the field as if perpendicular field the result would be only one susceptibility ($\chi\perp$). The respect orientation below T_c is a result of crystalline anisotropy. The anisotropy is also responsible for the slight difference in ($\chi \parallel$) and ($\chi\perp$) above T_c, where the perpendicular and parallel field not point to the direction of the axis (due to the anisotropy the sub lattice of the magnetization near T_c prefers to lie) [17]. In antiferromagnetic the susceptibility arises to a maximum a little above T_c, and the drops to ward T_c with a large maximum in it's slop at the critical point Fig. (1). The calculations of the Curie temperature (T_c) were produced from the drop of the curve of the magnetic susceptibility, according to Hartshorn bridge [12]. A perfect experimental curve determined in this method is shows in Fig. (2). Curie temperature (T_c) is determined by the linear extrapolation at intersection point of the steepest part of the curve and the back ground as shown in [13]. The magnetic properties of the using materials were determined at the room temperature (RT) by using Vibrating Sample Magnetometer (VSM) in an applied external magnetic field above to 20 Koe. Also by using the devices of Ferromagnetic resonance (FMR) device the measurement at 9.5 GHz obtain by (Bruker spectrometer at room temperature (RT)). The using Specimens in this work involve Fe 30at % Ni alloy was prepared by using arc melting method Fe and Ni metal was at the purity of 99.9%. The sample shapes of the was as a thin plate specimens and the diameters of the samples was 7×5 mm^2 and the thickness of it is 200 mm. The Specimens were annealed at 700°C for 3h after that it was quenched into the water and they put in small quartz tubes as a shape of encapsulated with (Ar) atmosphere. This method by using tube is easier than place inside the coil to measure T_c. The density of the magnetization M(H) according to quantum-mechanic system of volume V at low temperature (T=0) and by using uniform magnetic field H it can be defined by:

$$M(H) = -\frac{1}{v}\frac{\partial E0(H)}{\partial H} \dots\dots\dots (1)$$

Where $E_0(H)$ is the ground state energy equation (1) used in this work to measured (Ms). On the other hand if the system at the thermal equilibrium state the magnetization density at temperature (T) for excitation state $E_n(H)$ [14] can be determined by following equation.

$$M(H, T) = \frac{\sum_n Mn(H) e^{-En/K_B T}}{\sum_n e^{-En/K_B T}} \dots\dots\dots\dots (2)$$

Where $M_n(H) = -\frac{1}{v}\frac{\partial En(H)}{\partial H}$

This form of the equation at the thermodynamic system can be written as follow

$$M = -\frac{1}{v}\frac{\partial F}{\partial H}$$

Where F represent the magnetic Helmholtz free energy according to fundamental statistical mechanical rule as shown in $e^{-F/K_B T} = \sum_n e^{-En/K_B T}$

The susceptibility is defined as $\chi = \frac{\partial M}{\partial H} = -\frac{1}{v}\frac{\partial^2 F}{\partial H^2}$ ……….. (3) [14]

At higher temperature the magnetization was calculated from the curve of Fig. (1) by the last squares method, according to the formula

$$M(H)= M_o \left[cth \frac{\mu H}{KT} - \left(\frac{\mu H}{KT}\right)^{-1} \right] + \chi_o H$$

Where the first term represent the magnetization of the system of super paramagnetic particles μ (magnetic moment), T (Temperature), H (magnetic field) Mo (is the magnetization of a system in the stabiles state), K (is Boltzmann's constant). The second term takes account of the paraprocess in a system of stabilized super paramagnetic [22].

3. RESULT AND DISCUSSION

The variation of the magnetic properties of Fe-Ni were investigated for samples with (30%, 50% and 75% by weight Ni) and sintered at 700˚C for 3h are shown in Fig. (1). The Curie point (Tc) measured in the (VSM) was found to be 725k. the results are in agreement with Tc measurement reported by ASM international [19] and Ram [18]. For the samples there appears to be phase transitions occur at 692k with 30% Ni, others wise (Tc) was found to at 745k with 50% Ni and the other phase at 750k with 75% Ni. The results are in general agreement with those of [21]. The magnetic properties for different samples are listed in table (1). X-ray patterns were obtained at room temperature using a philips diffract meter and Cu (k α) radiation. The diffraction pattern is show in Fig. (3) shows the powder diffraction pattern obtained. It may be seen that the diffraction pattern is consistent with a single phase material with B2 structure. In the present work a systematic study of the hysteresis loop as a function of magnetic field was made for Fe-Ni alloys with composition (30%, 50% and 75% Ni). The saturation magnetization was 86, 65 (emu / g) for ($Fe_{70}Ni_{30}$, $Fe_{50}Ni_{50}$) respectively.

The change in the area of the hysteresis loop with composition for Fe-Ni alloys is particularly large near 50% Ni see Fig (4). This occurs presumably because of a strong competition between ferromagnetism (related to positive exchange interaction between Fe-Ni and Ni-Ni pairs) and anti-ferromagnetism (favored by negative exchange interaction between Fe-Fe pair) [15, 20]. Frustration arises with antiferromagnetic interactions wherever the geometry of the lattice is such that the neighbor of a given atom are themselves neighbors of each other. Consider a f.c.c system all the spins on the corner sides will be parallel to each other and all the spins in the center positions will be antiparallel. The distance between the corner and face centered positions is a⁄√2 which is identical to the separation of the face centered position themselves. However the exchange interaction will negative and positive for the two identical respectively therefor the system is frustrated. Therefore, this composition (50 at % Ni) is expected to be very sensitive to structural change like short range order or clustering. From the hysterics curves the value of the Coercivity (Hc), the saturation magnetization (Ms), remint ratio (Mr / Ms), magneton number (nB) of Fe-Ni are given in table (1). The morphology of the (Fe-Ni) Invar alloys and there precursors were determine by using Scanning Electron Microscopy (SEM) technique. The measurement of grain size distribution of the samples has been done by using the same technique. The range of the grain size of the Fe-Ni is (□100-500 nm) as show in Fig. (6) scanning electron microscopy (SEM) was used to investigate the surface morphology of the particles and grain size distribution for the Fe-Ni Invar alloy at difference concentration of Nickel. Measurement of the Curie temperature the result presented in Fig. (5), show that depends on the thermal equilibrium treatment for Invar alloy. Fig. (7) shows the susceptibility of Invar Fe-Ni obtained by applied magnetic field after annealing process and at room temperature.

Table 1: Show the Magnetic Properties and Structural for Fe-Ni Invar alloys at Difference Nickel Concentrations

Fe-Nix	Ms emu/g	Mr emu/g	Hc (oe)	Mr/Ms	nB(μB)	Average atomic mass (amu)	Lattice constant (A˚)	Malting temp. (k)	Curie temp. (k)	Debye temp. (k)	Structure
X=30	81.28	77.93	1028.99	0.96	3.414	57.88	3.586	1710	692	407.8	fcc phase
X=50	50.10	29.50	926.03	0.59	2.263	56.31	3.586	15.50	745	315	fcc phase
X=75	41.46	19.62	1000.42	0.47	1.961	55.99	3.586	1773	750	285	bcc phase

4. CONCLUSIONS

In summary, Measurements of the value of the Magnetic-susceptibility as a function of temperature had been measured for the (30%, 50% and 75% Ni Invar alloys). The magnetic locally ferromagnetic parts are found when the samples annealing treatment at (700 k) Curie temperature, also in this range the Invar characterization change very strong with the increase of the atomic concentration and that behavior also was very sensitive to the atomic rearrangements in the alloys. Annealing processes at the low temperature such as 340°C cause increasing the Curie temperature (T_c) of Invar alloys, the explanation of these criteria has been interrupted as a result to the rearrangement of the atoms for a small range, associated with the change of the chemical short-range order (S.R.O). For more details to determine other properties such as saturation magnetization (MS) volume magneto striation and magnetic anisotropy, that results could refers to the real nature of magnetic structure obtain in this paper. The X-ray indicated that the samples (i.e Invar alloys) where chemically disorder into B2 structure. It would be extremely interesting to repeat these studies on samples which have $L2_1$ structure. In this way it would be possible to assess the effects of atomic order on the occurrence of magnetic and superconductivity and possibly understand the physical underlying mechanisms in this stage the two effects in the Fe-Ni amorphous alloys difficult to separate it. In this research the effect due to the change in the chemical order has been shown. Also the relaxation kinetic is very similar in amorphous alloy and metallic glasses states. The similarity between two properties is real surprising, due to the structures of the crystalline alloy and amorphous metals are different. Our measurement for the Curie temperature (T_c) the results show in Fig. (3), denoted that the Curie temperature depends on thermal treatment processes for the Invar alloy.

Finally, in this work it is important to make a good quantitative relation between the local chemical and Curie temperature (T_c) we need for theoretical work

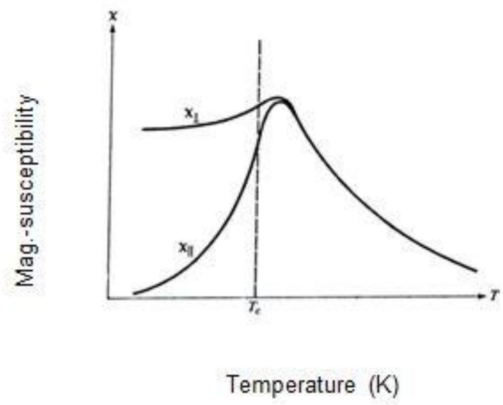

Fig. 1. The susceptibility of an antiferromagnetic vs. the temperature at the critical temperature below (Tc)

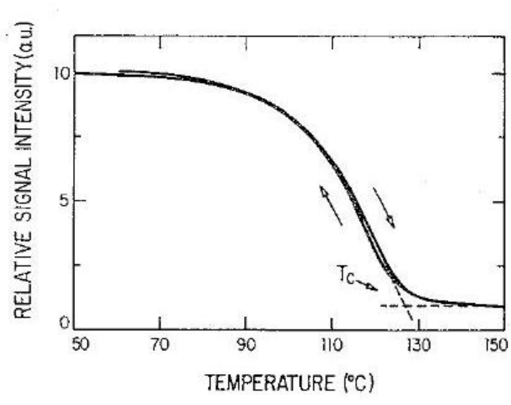

Fig. 2. the behavior of the typical experimental curve shows by using method to determine Tc.

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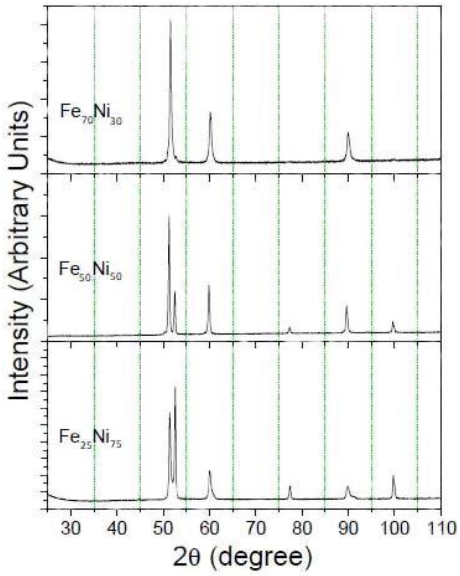

Fig. 3. X-ray Intensities versus scattering angle (2θ) of Fe-Ni Invar alloy.

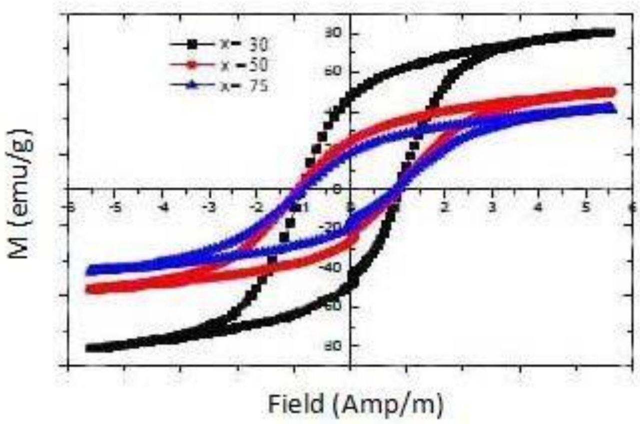

Fig. 4. Hysteresis loop for Fe-Ni as a function of temperature at (x= 30%, 5%, 75% Ni) .

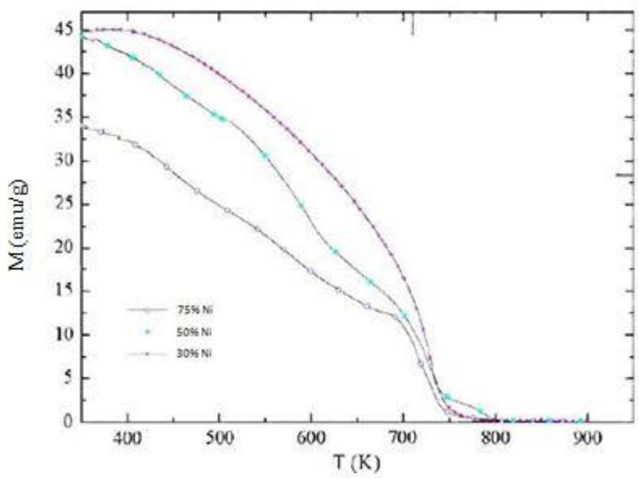

Fig. 5. Shows Curie temperature measurement for Fe-Ni samples with (30%, 50% and 75% at Ni).

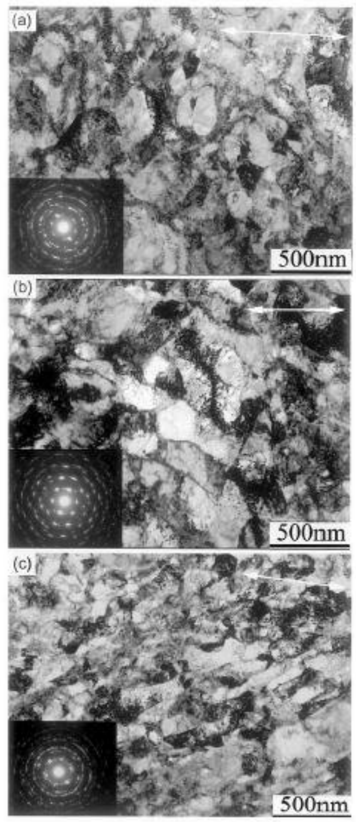

Fig. 6. Scanning Electron Microgarghs of Fe-Ni Invar alloy at a: x= 30% Ni, b:= x= 50% Ni and c: x= 75% Ni.

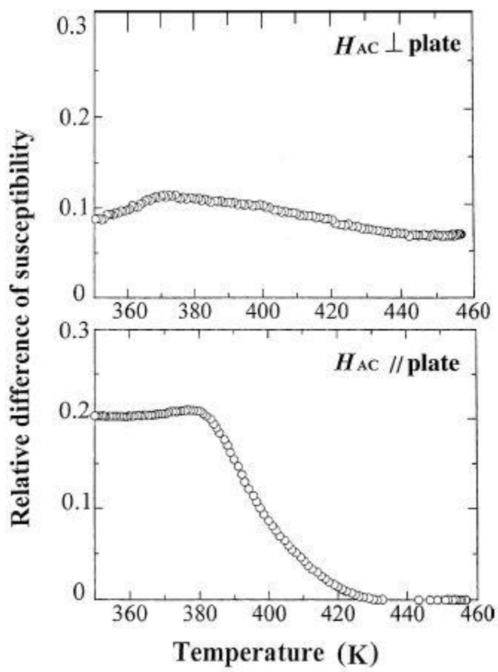

Fig. 7. The differences magnetic-susceptibility behavior with temperature for specimen before and after the annealing processes

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