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COMPARISON STUDY BEWTWEEN THE RECOVERY BEHAVIOR OF DEFORMED QUENCHED, PRE-ANNEALED AND PULSED LASER DAMAGED Fe$_{50}$Ni$_{50}$ ALLOY BY MAGNETIC MEASUREMENTS

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Abstract:

Through the magnetic measurements, isochronal annealing experiments in the temperature 25-950 ºC of deformed, quenched, pre-annealed and pulsed laser damage samples Fe50Ni50 alloy revealed the existence of three annealing stages I, II and III in the annealing spectrum of heavily cold-worked by observing the associated changes in maximum magnetic permeability (μmax) on the magnetic field(H) for different annealing temperature. The first annealing stage I appeared in the temperature range from125-500 ºC and it was activated by energy 1.05 eV. It was attributed to the short range order caused by the long range migration of vacancies. The second annealing stage II appeared in the temperature range from 550-750 ºC and it was activated by 1.82 eV, it is associated with an increase in μmax due to the dissociation of vacancy clusters formed during stage I. The third annealing stage III appeared in the temperature range from 750-825 ºC and it was activated by 3.04 eV, it was related to the climb motion of dislocation during the recrystallization process.

Keywords: Fe50Ni50; Ferromagnetic Alloy; Magnetic Properties; Plastic Deformation; Activation Energy; Order of Reaction.

1. Introduction

Attention, has recently been focused on the behavior of recovery of lattice defects in deformed, quenched, pre-annealed and pulsed laser damaged Fe-based alloys. These alloys are an important class of materials for present and future technologies applications [1-4]. Actually, the structure sensitive properties of these materials are directly dependent on such fundamental processes as creation of lattice defects, defect diffusion, and defect reaction in the alloy matrix. On the other hand, the changes in the structural sensitive properties Fe-based alloys have been observed by several author [4-9], during the annealing of irradiated or cold-worked alloys above room temperature. Results of some of those experiments have been explained on the basis of either solute segregation, changes in short-range order or precipitation of ordered phases. These processes cause microscopic homogeneities in the alloy matrix which lead to anomalous changes in the physical property measured. Moreover, the present binary alloy Fe$_{50}$Ni$_{50}$ of superstructure LI$_2$ type (Ni$_3$Fe)

[10-12], its prospect as soft magnetic material and good corrosion resistance [13-15] at elevated temperature is of additional interest. This binary system forms a long range ordered intermetallic phase superstructure LI$_2$ type (Ni$_3$Fe) which has FCC unit cell with Fe atoms occupy the corners and Ni atoms occupy the middle of the faces of the cube [16] while in the disordered phase, Ni and Fe atoms randomly occupy the FCC lattice sites. According to the constitution diagram of Fe-Ni alloy [16] the ordered and disordered phases coexist in equilibrium around 830 K, while below this temperature the ordered phase is stable. Moreover, in this class of intermetallic materials, unlike in pure metals or solid solution alloys, there exists a restriction imposed on the formation and movements of vacancies. This restriction is due to the thermal formation of vacancies on the sublattices concomitant thermal formation of antisite defects on both sublattices, and the coexistence of point defects due to deviations from the stochiometric composition [17,18]. Therefore, it is expected that the annealing behavior of physical parameters of intermetallic compounds after pre-annealed , quenching or pulsed laser are more complex than in pure metals on the other hand, since in intermetallic compounds the local order transformation caused by short-range or long-range ordering are induced by diffusion of point defects. Therefore, it is difficult to separate ordering effects from those due to vacancy or interstitial annealing. Although several studies [19, 20] have been made on the thermal formation and migration of point defects intermetallic compounds by various techniques, fundamental information on the temperature and composition dependence of point defects is still remains a challenging problem.

The aim of the present work is to study the behaviour of structural changes induced by lattice defects created in intermetallic compound Fe$_{50}$Ni$_{50}$ after quenching and pre-annealed, also to study the influence of additional lattice defects produced by plastic deformation and laser damaged on the behavior of isochronal annealing curves.

2. Experimental Work

The test material, Fe$_{50}$Ni$_{50}$ alloy, was prepared from high-purity Fe and Ni by induction melting followed by a suitable homogenization at 1200 ℃ under a helium atmosphere for 24 hours, then slowly cooled to room temperature. The material was shaped by extrusion into rods of 3mm diameter followed by swaging at room temperature to wires of 1mm diameter. The atomic absorption method was used in order to determine the composition of the alloy (Table 1) [21]. The wire sample was introduced as a core of a magnetization coil and the cathode ray technique was employed to obtain room-temperature B-H curves at different magnetizing fields. The maximum magnetic permeability was obtained from the relation: $\mu_{max} = (B/H)_{max}$ which characterizes the magnetization of both reversible and irreversible domain-wall motion. Plastic strain deformation was introduced on the samples by a locally conventional strain machine. It was measured by the dimensionless quantity: $\eta\% = [(\Delta L/L) \times 100)]$, where ΔL and L are the changes in length and the initial length of the sample respectively [22]. The quenching sample was performed by holding the specimen at 950 ℃, and then quenched in cold water with rate of quenching 3×10^3 K/s.

Table 1: Chemical composition of Fe$_{50}$Ni$_{50}$ alloy

Fe	Ni	Mn	Zn	Mg	Sn	Al	Cu
Major	Major	-	0.001	-	0.001	0.001	0.001

The most widely used lasers for Laser Induced Plasma Spectroscopy (LIPS) are Nd:YAG Lasers [type Brilliant B from Quantel] [23]. The laser system (Nd:YAG) delivers 650, 300 and 150 mJ per pulse at constant duration of 5 ns. The energy per pulse at the target surface was measured

using a set of glass sheet absorbers which reflects ~ 6.3% of the laser energy as measured by a power meter (Ophier model lz 02165). The target surface was fixed on a precise x-y translational stage at a distance 9.5cm from a quartz lens of focal length 10 cm. The laser spot area was calculated using a thermal paper, and it was found to be in a circular shape with diameter 2 mm. This enabled us to evaluate the fluencies at the target surface, which were in the range from 7 to 19 J/cm^2.

3. Results and Discussion

The results of isochronal annealing observed in the present work for pulsed laser, pre-annealed (T_a =850 ℃, t_a= 2 h), deformed (η =10%, t_a = 10 minutes) and quenched (T_q= 950 ℃) $Fe_{50}Ni_{50}$ alloy in the temperature range from 25 to 950 ℃ (see Figs. 1-4) [21-24]. The relative changes in the maximum magnetic permeability of the four samples revealed the presence of three annealing stages, stage I, stage II and stage III in the temperature range from 25-950 ℃. The isochronal annealing at stage I range from 125 to 500 ℃. Therefore the interstitial atoms migration could be definitely excluded in this temperature [25-29]. From this consideration, stage I could be attributed the structural change in the alloy matrix by short-range ordering promoted by migration of vacancies, formed by plastic deformation, quenching and laser damage $Fe_{50}Ni_{50}$ alloy to deep traps or sinks. During isochronal annealing of the disordered $Fe_{50}Ni_{50}$ alloy of structure below equilibrium level each vacancy jump induced a certain increase of local order. The contribution of vacancy migration, leading to an increase in short range order, has also been suggested by Sharma et al. [30] and Nakata et al. [31] in annealing studies of irradiated Fe-Cr-Ni alloy. When the vacancy become mobile, it performs a certain number of jumps before annihilation or trapping clusters and can promote the evolution of the local atomic order of the alloy towards its equilibrium state. The activation energy of stage I (1.05 eV) is of the same order of magnitude as that required for free mono-vacancy migration in α-Fe [24]. Beside the annealing process during this stage was controlled by reaction kinetics of an order greater than one.
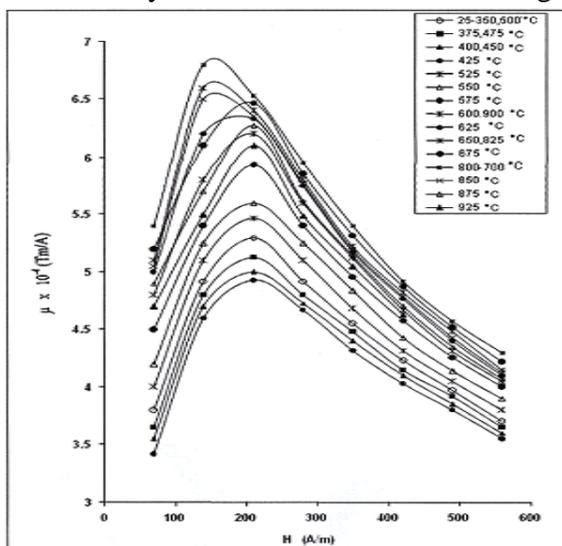
Figure 1: Effect of isochronal annealing temperature on the dependence of magnetic permeability μ, on the magnetic field H, of pulsed laser damaged $Fe_{50}Ni_{50}$ sample for different annealing temperatures.
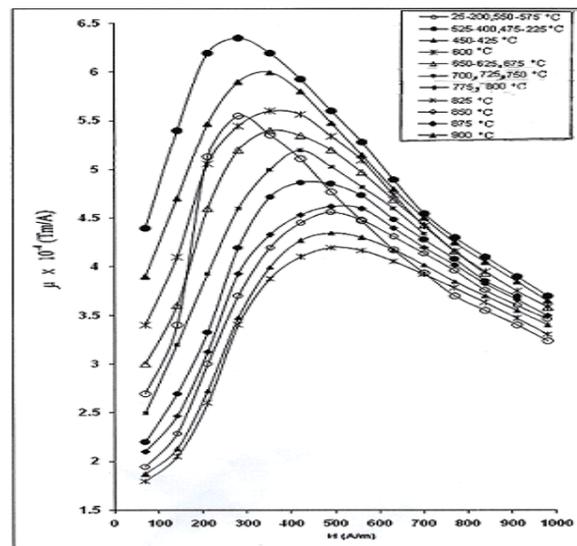
Figure 2: Effect of isochronal annealing temperature on the dependence of magnetic permeability μ, on the magnetic field H, for pre-annealed (T_a = 850 ℃ , t_a = 2 h) $Fe_{50}Ni_{50}$ sample for different annealing temperatures.
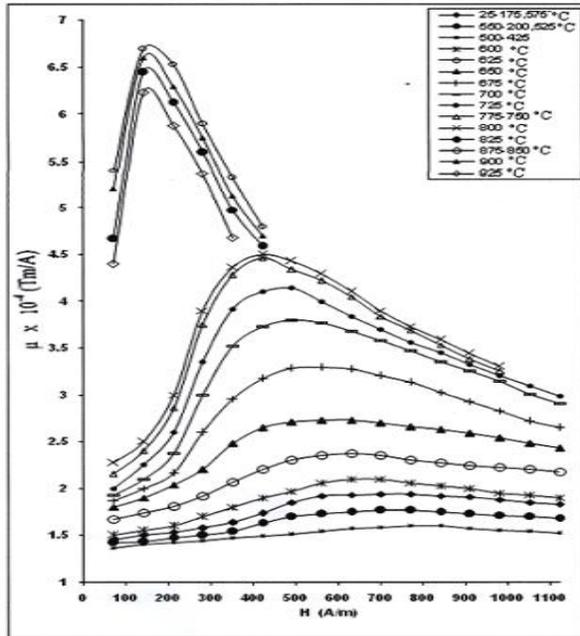
Figure 3: Effect of isochronal annealing temperature on the dependence of magnetic permeability μ, on the magnetic field H, of deformed ($\eta = 10\%$, $t_a = 10$ minutes) $Fe_{50}Ni_{50}$ sample for different annealing temperatures [1].
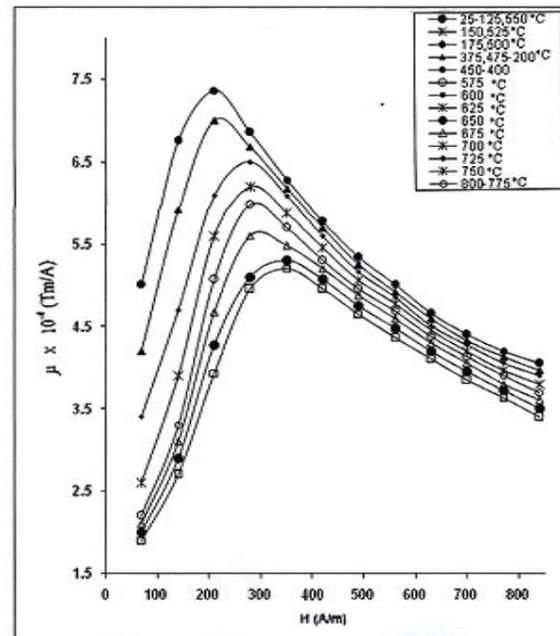

Figure 4: Effect of isochronal annealing temperature on the dependence of magnetic permeability μ, on the magnetic field H, for quenched ($T_q = 950$ ºC) $Fe_{50}Ni_{50}$ sample for different annealing temperatures [2].

These results support the idea that the change in structural ordering by short-range order is controlled by free vacancy migration, with the possible formation of vacancy clusters or complex aggregates [11,24]. Therefore, the increase in concentration of vacancies would enhance the change in local order by short-range ordering during this stage. The second annealing stage II centered around 600 ºC. This annealing stage observed in the present work in pre-annealed, quenched and laser damaged samples (see Figs. 5-8) could be attributed to the dissociation of vacancy collasters or complex aggregates formed during stage I or after quenching or laser damage, resulting in a release of free mono-vacancy. The vacancy migrates further to a deeper trap or falls into sinks during the migration process with annealing temperature rising from 550-750 ºC. The activation energy of this stage (1.82 eV) had the same order of magnitude as that required for the dissociation of vacancy clusters in Fe and Ni alloys [32,33]. Moreover, the recovery process during this stage was largely controlled by a second order reaction kinetics. This implied that this stage of recovery might be due to a bimolecular reaction presumably the dissociation of vacancy clusters, formed during the first stage by normal self-diffusion of vacancies.
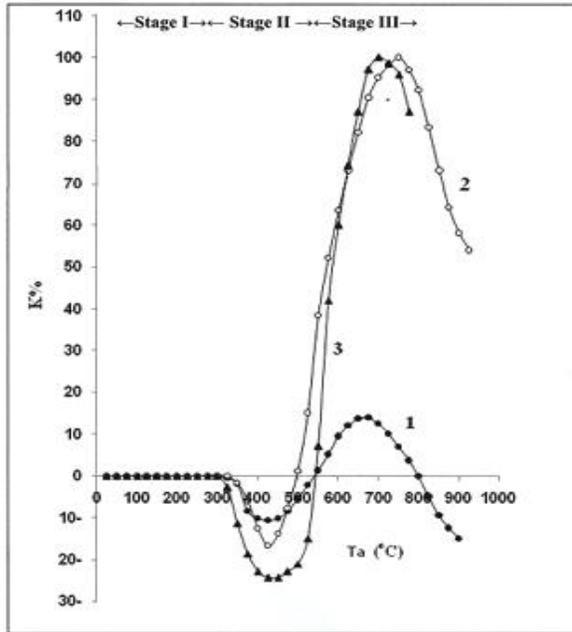
Figure 5: Relative change in the maximum magnetic permeability (K%) of: (1) pre-annealed ($T_a = 850$ ºC, $t_a = 2$ h), (2) pulsed laser, and (3) quenched ($T_q = 950$ ºC) $Fe_{50}Ni_{50}$ samples with the annealing temperature.
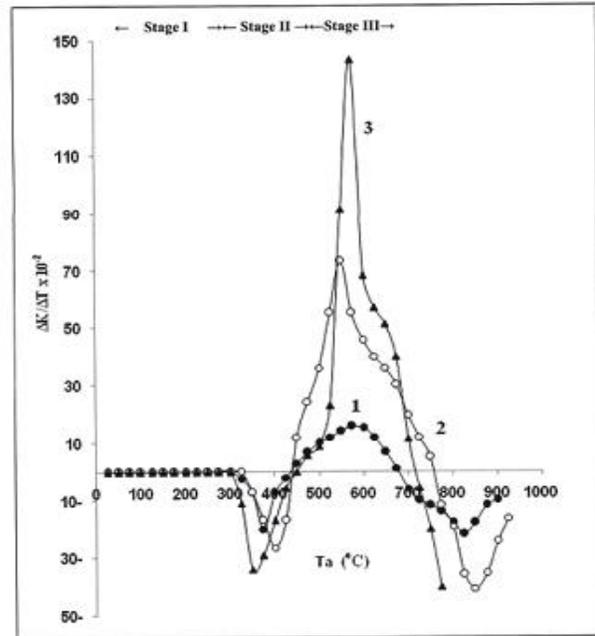

Figure 6: The annealing spectrum of the maximum magnetic permeability (K%) of: (1) pre-annealed ($T_a = 850$ ºC, $t_a = 2$ h), (2) pulsed laser, and (3)quenched ($T_q = 950$ ºC) $Fe_{50}Ni_{50}$ samples with the annealing temperature.

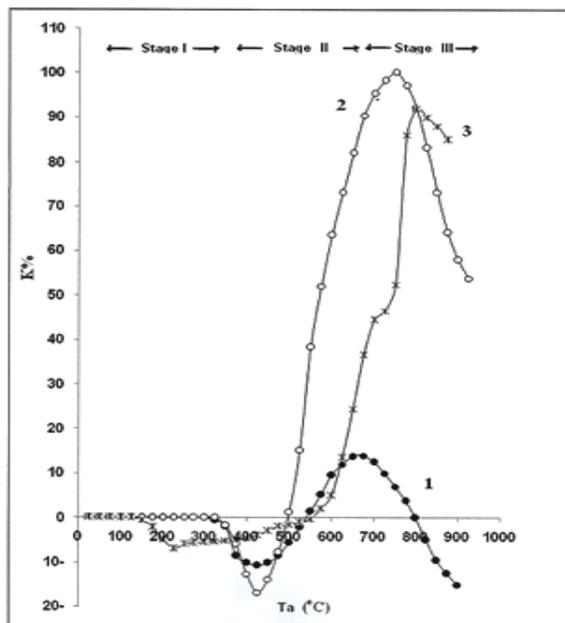Figure 7: Relative change in the maximum magnetic permeability (K%) of: (1) pre-annealed ($T_a = 850$ ºC, $t_a = 2$ h), (2) pulsed laser and (3) deformed ($\eta = 10\%$, $t_a = 10$ minutes) $Fe_{50}Ni_{50}$ samples with the annealing temperature.

Figure 8: The annealing spectrum of the maximum magnetic permeability (K%) of: (1) pre-annealed ($T_a = 850$ ºC, $t_a = 2$ h), (2) pulsed laser and (3) deformed ($\eta = 10\%$, $t_a = 10$ minutes) $Fe_{50}Ni_{50}$ samples with the annealing temperature.

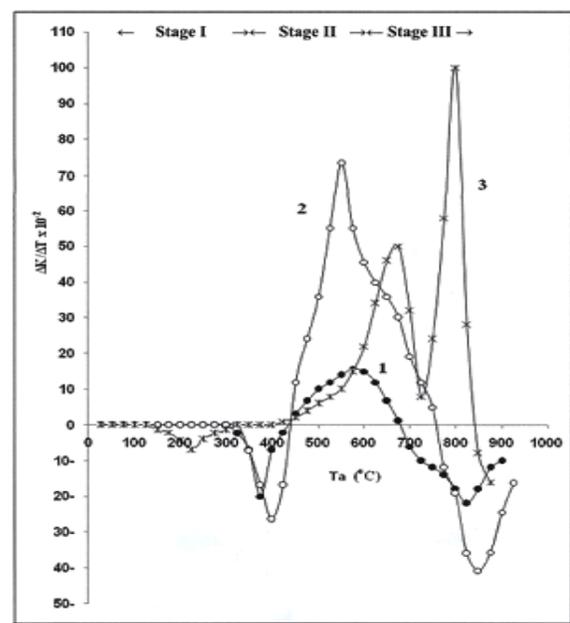The higher temperature annealing stage (stage III) observed in the present work only during the isochronal annealing of deformed $Fe_{50}Ni_{50}$ sample. In the temperature range from 750 to 825 ℃ could be attributed to recrystallization phenomena. The observed increase of μ_{max}, when recrystallization started in the deformed alloy matrix, was thought to be due to the release of some of the dislocations forming the cell boundaries through a process of climb by edge of dislocation [34]. The subsequent removal of these dislocations settles down the density of pinning sides for the motion of magnetic domain walls in the matrix which consequently decrease the load on the magnetic domain. This process should give first-order Kinetics which was actually observed in this stage (Table 2) [34]. The activation energy for stage III obtained 3.04 eV (see Fig.9). Finally, the observed decrease in the maximum magnetic permeability in all samples (pre-annealed, deformed, quenched and laser damaged) in the temperature range from 825 to 900 ℃ (see Figs. 5-8). This decrease is most probably due to the precipitation of $FeNi_3$ phase. The precipitation of this phase causes a marked expansion of the material, leading to an increase in the location density in the alloy matrix [35-37]. This process seemed to impose a heavily pinning action on the magnetic domain walls, preventing them from normal detachment from fixation points and leak out the magnetic pressure exerted by the magnetic field on domain walls.

Table 2 represents the activation energies for the three stages I, II and III of deformed $Fe_{50}Ni_{50}$ sample (η = 10 %) using Meechan-Brinkman method [38] and the order of reaction for the three stages I, II and III of $Fe_{50}Ni_{50}$ alloy using Damask and Dienes method [39].

Table 2:

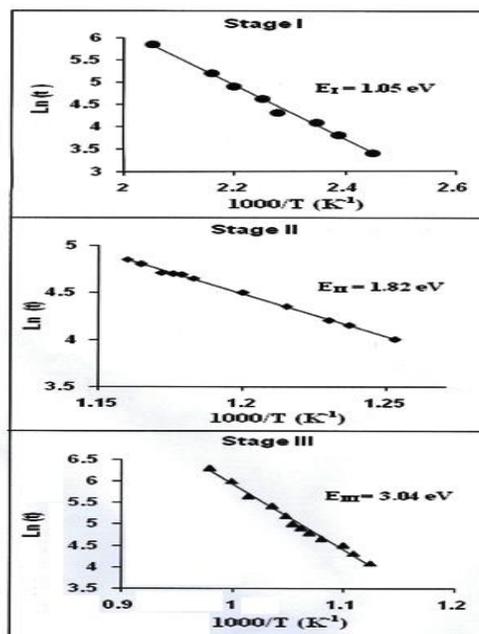| Stage	Activation energy (eV)	Order of reaction (γ)
I	1.05	1
II	1.82	2
III	3.04	1

Figure 9: The activation energies for the three stages I, II and III of deformed $Fe_{50}Ni_{50}$ sample (η = 10 %) using (Meechan-Brinkman method) [38].

Acknowledgements

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