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Research Article

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Study on the Behavior of the Recovery in Plastically Deformed Ferrite Steel (0.15 wt% C steel, 16 Cr-4 Mo)

Eman G. Takla

Physics Department, Faculty of Science, Cairo University, Giza, Egypt

Abstract Through the magnetic measurements, isochronal annealing experiments in the temperature range from 25 to 800 ºC of deformed ferrite steel 0.15 wt % C steel , 16 Cr-4 Mo revealed the existence of two annealing stages V and VI in the annealing spectrum of heavily cold-worked by observing the associated changes in maximum magnetic permeability (μ_{max}) and the magnetic coercivity (H_{cr}). The recovery stage V appeared above 450 °C and it was activated by an energy 1.8 e V and it was attributed to the dissociation of carbon-vacancy pairs. The recovery stage VI appeared above 700 ºC , it was activated by 3 eV, it was related to the climb motion of dislocation during the recrystallization process.

Keywords Ferrite steel - magnetic properties – annealing - vacancy formation

1. Introduction

Considerable efforts have been undertaken to investigate the behavior of the recovery of lattice defects in α –iron produced by particle radiation [1-4], cold-worked [5,6] or quenching [7,8]. Evidences were found for the existence of different recovery stages above room temperature was found by several authors [9-12].

Seeger [12] showed that the recovery stage III in α-iron is a self interstitial. The recovery stage (stage IV) appeared between 230 and 330 C observed by residual resistivity, positron lifetime and magnetic measurements [13] was ascribed to the free migration of mono-vacancy in α-iron above room temperature. In addition to this recovery stage , α- iron exhibits other high temperature recovery stages in the temperature range 330-700º C, namely stage V and stage VI [14]. Moreover, it is well known that the presence atoms seems to have a strong influence on the recovery process. Therefore, the aim of this work is mainly to study the role of Cr-atoms and Mo-atoms on the behavior of the recovery of the deformed Fe-25 Cr-Mo alloy. The maximum magnetic permeability (μ_{max}) and magnetic coercivity (H_{cr}) were used to investigate the nature of the defects participating during the various recovery processes.

Experimental Work

The test material, 0.15 wt% C steel, 16 Cr-4 Mo alloy, was prepared in the Institute for Ferrous Metallurgy, Moscow, by induction melting of α-iron with pure graphite, Cr, and Mo in an induction furnace under a helium atmosphere followed by suitable homogenization at 850 ^{o}C for 24 hours. The material was then shaped by extrusion into rods of 3 mm diameter followed by swaging at room temperature to wires of diameter 0.8 mm. This standard state of the samples was considered as heavily cold-worked samples. Chemical analysis of the sample was shown in Table 1.

Table 1: Chemical composition

Material	C	N	Cr	Mo
Concentration	0.15	0.06	16	4

The annealing spectrum of the sample was controlled by subjecting them to rectangular heat pulses using heat pulses using high-heat capacity furnace maintained at the annealing temperature. The sample temperature was measured with accuracy ± 0.5 ^{o}C. The sample was introduced at the core of magnetization coil and the cathode-ray technique was employed to obtain room temperature B-H curves at different magnetization fields. The maximum magnetic permeability was obtained from the relation $\mu_{max} = B/H$ which characterizes the magnetization of both reversible and irreversible domain-wall motion. The magnetic coercivity (H_{cr}) was obtained from the peak position of μ_{max}. The observation of the associated changes in maximum magnetic permeability (μ_{max}) are able to give information about the nature of defects participating in the various recovery process. This may help in clarifying some of the unresolved problems in the field of defect annealing in ferrite alloy above room temperature. The experimental results have been reported in normalized form of magnetic coercivity.

$$K = \frac{[\mathbf{H_{cr}}\ (\mathbf{T_a}) - \mathbf{H_{cr}}\ (\mathbf{0})]}{[\mathbf{H_{cr}}(\boldsymbol{i}) - \mathbf{H_{cr}}(\mathbf{0})]} = \frac{\Delta\, \boldsymbol{H_{cr}}\ (\boldsymbol{T_a})}{\Delta \mathbf{H_{cr}}\ (\mathbf{0})} \quad (1)$$

where H_{cr} (i) is the value of the magnetic coercivity of cold-worked sample, H_{cr} (T_a) and H_{cr}(0) are the values after annealing at temperature T_a and the value of fully annealed samples respectively. All measurements were carried at room temperature.

Results

The magnetic permeability (μ) measured at room temperature as a function of the magnetizing field (H) after isochronal annealing of the heavily cold-worked 0.15 wt% C steel, 16 Cr-4 Mo alloy by heat pulses of 15 minutes is presented in Fig. 1. The curves were characterized by pronounced peak values in magnetic permeability (μ_{max}). These peaks shifted their positions to lower magnetic fields (H_{cr}) as the annealing temperature increased.

The dependence of both maximum magnetic permeability (μ_{max}) and the magnetic coercivity (H_{cr}) on the annealing temperature is illustrated in Fig. 2. It is clear that μ_{max} successively increases with the increase in the annealing temperature, while H_{cr} decreases.

The relative changes in magnetic coercivity, $K = [\Delta H_{cr}(T_a)/\Delta H_{cr}(0)]$ of two different isochronally annealed samples (t_a = 15 and 20 minutes) revealed the presence of two annealing stages V and VI in the temperature range 450-800 º C, Fig. 3a. The demonstration of these annealing stages could be made better distinguished by plotting the recovery spectrum of the magnetic coercivity as depicted in Fig. 3b for heavily cold-worked samples of 0.15 wt % C, 16 Cr-4 Mo alloy.

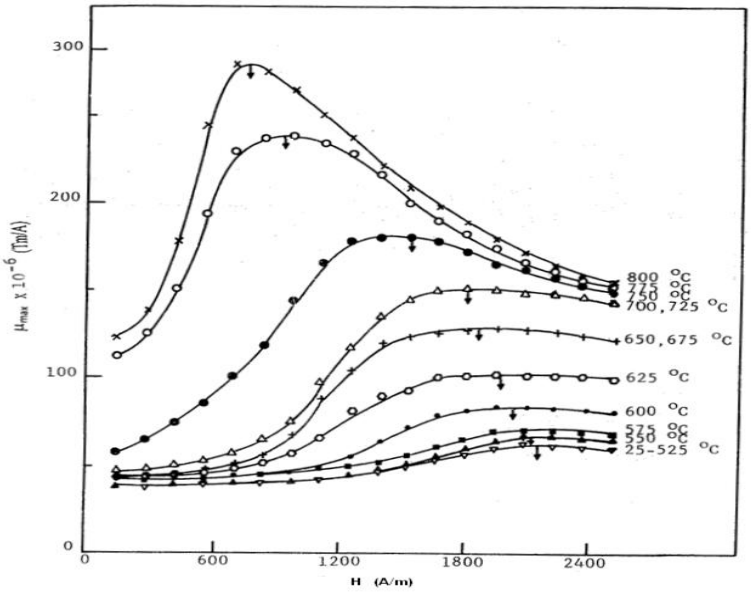

Figure 1: Effect of isochronal annealing temperature (T_a) on the dependence of the magnetic permeability (μ) on the magnetic field (H). (The arrows represent the values of magnetic coercivity (H_{cr}).

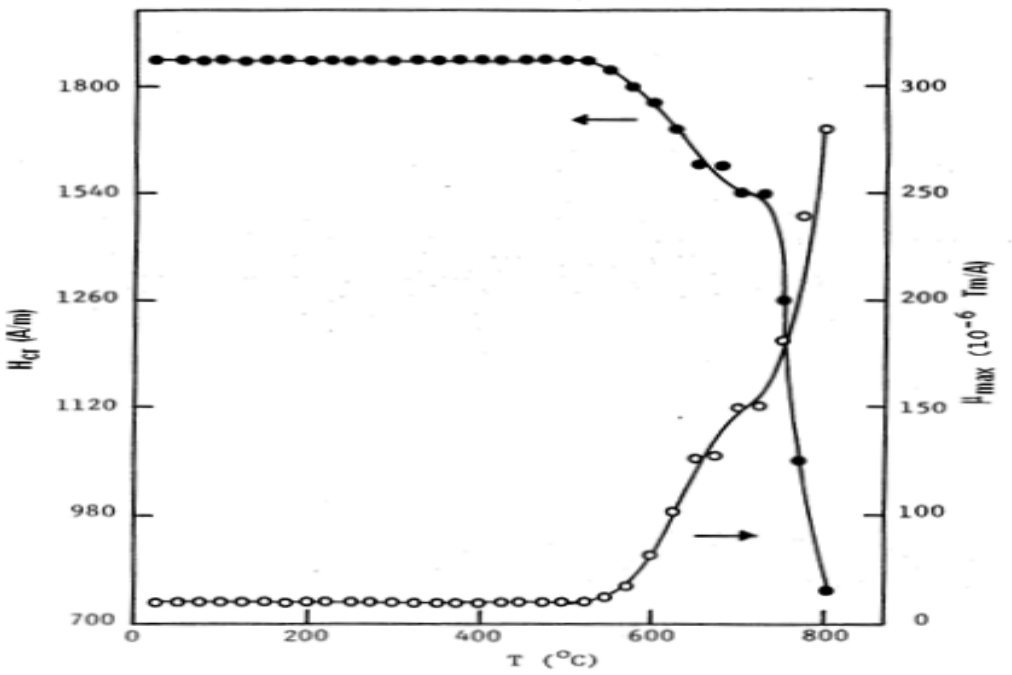

Figure 2: Effect of isochronal annealing temperature (T_a) on the maximum magnetic permeability (μ_{max}) and magnetic coercivity (H_{cr}) of the ferrite steel 16 Cr-4 Mo alloy, t_a = 15 minutes.

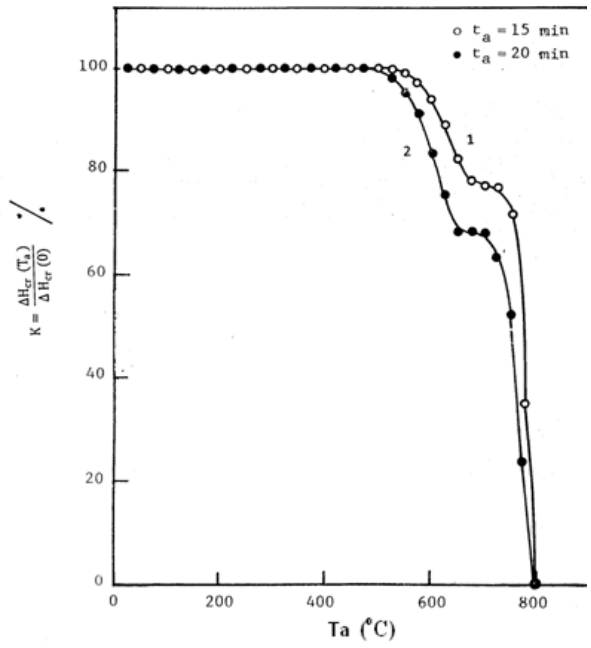

Figure 3a: Relative changes in magnetic coercivity, [$K = \Delta H_{cr}(T_a)/\Delta H_{cr}(0)$] with annealing temperature (T_a).

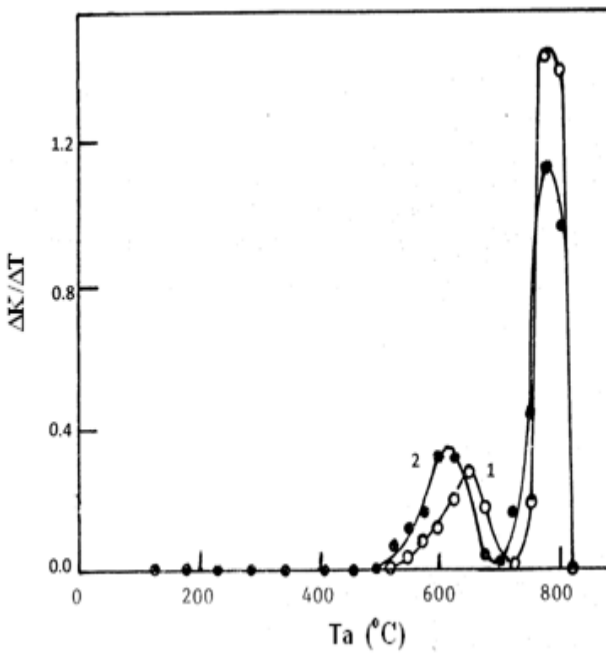

Figure 3b: The annealing spectrum of magnetic coercivity of cold-worked ferrite alloy 0.15 wt% C steel, 16 Cr-4 Mo alloy. 1) t_a = 15 minutes, and 2) t_a= 20 minutes.

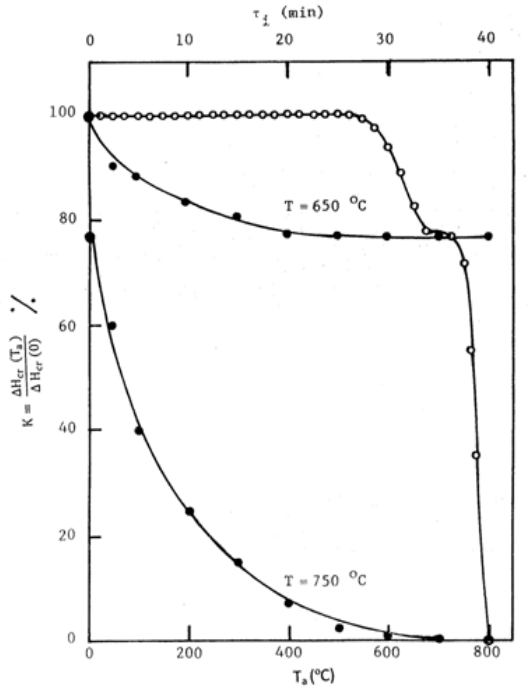

Figure 4: Isochronal and isothermal annealing of the magnetic coercivity in two annealing stages V (T_a = 650 ºC) and VI (T_a = 750 ºC).(●) Isochronal annealing and (○) Isothermal annealing.

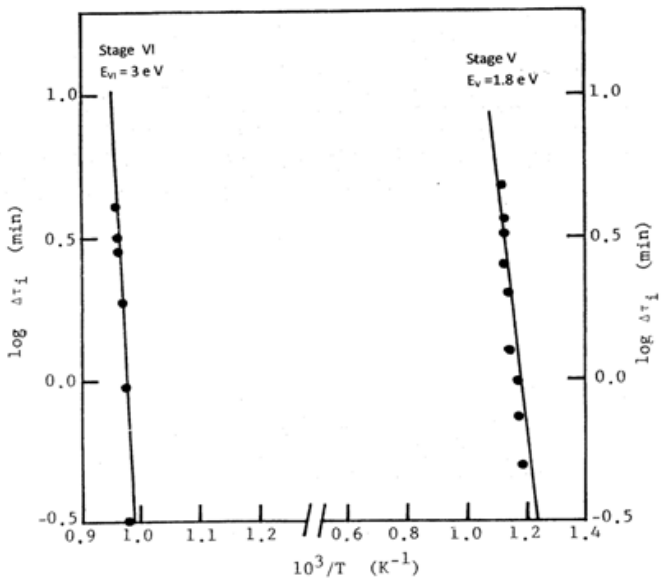

Figure 5: Activation energy determination using Meechan-Brinkman method for the stages V and VI.

In order to study the annealing kinetics of the observed recovery stages, the activation energy (E) was determined by Meechan and Brinkman method [15]. It was found that the activation energy for stage V is 1.8 e V but for stage VI is 3 e V (see Figs. 4 and 5).

Discussion

The isochronal annealing of heavily cold-worked 0.15 wt% C steel 16 Cr-4 Mo alloy in temperature range 25-800 ºC revealed the existence of two annealing stages V and VI. The recovery stage V observed at temperature higher than 450 ºC might be attributed to the dissociation of carbon-vacancy pairs or complex formed during plastic deformation resulting in a release of free mono-vacancies bounded to vacancy agglomerates [1,6]. This process was expected to decrease the density of pinning sites for the motion of the magnetic domain walls in the matrix[(16)] resulting in the decrease of H_{cr} and the increase in μ_{max} (Fig. 2). The presently determined activation energy of this annealing stage being about 1.8 eV (Fig. 5) has the same order of magnitude of the required for the dissociation of carbon-vacancy pairs in carbon-doped iron[1,17]. The activation energy of the dissociation process was considered to be equal to the sum of migration of energy of vacancy free defect and the binding energy of interstitial carbon atoms [18,19]. Comparing the present value of carbon-vacancy dissociation energy (about 1.8 eV) and vacancy migration energy (1.1 eV) (Refs. 9,17) , the carbon-vacancy binding energy is easily seen to be about 0.7 eV. A value that agreed reasonably well with previously published value for the dissociation of carbon-vacancy pairs [1,16,18] in α-iron.

The second annealing stage VI was inferred to dislocation climb in 0.15 wt% C steel, 16 Cr-4 Mo alloy. It should be pointed out here that above 700 ºC in cold-worked samples, only large clusters of dislocation loops and dislocation tangles [19,20] with which the magnetic domain walls can attract, remain in the matrix. Therefore, stage VI recovery appearing at temperature above 700 ºC, could be attributed to a recrystallization phenomenon [6]. The activation energy of 3 eV obtained here for recrystallization in cold-worked in ferromagnetic materials [20,21].

The high experimental value of the activation energy for recrystallization in alloy could be attributed to the pinning of dislocations by Mo atoms during plastic deformation.

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