

EFFECT OF DISLOCATION DENSITY ON THE ACTIVATION ENERGY OF RECOVERY OF COERCIVE FORCE FOR A FERROMAGNETIC MATERIAL

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الخلاصة

- يهتم هذا البحث بدراسة استرجاع قوة الممانعة المغناطيسية (Coercive force) بالمعالجة الحرارية لأحد المواد الفرومغناطيسية بعد تعرضها للتشوه اللدن (Plastic Deformation . . .) واختبار مدى تطابق النتائج العملية مع العلاقات النظرية التي تناقش هذه المشكلة . ولقد أظهرت نتائج هذه الدراسة ما يلي :
- (١) ان النتائج العملية تتطابق الى حد بعيد مع النظرية المراد اختبارها في شأن انخفاض قوة الممانعة المغناطيسية بالمعالجة الحرارية للصلب الفريتي (24 Cr Al x10) بعد تعرضه للاجهاد اللدن .
 - (٢) ان طاقة التفاعل اللازمة لعملية الاسترجاع تتناقص بتزايد درجة التشوه اللدن للعينات المختبره .
 - (٣) ان طاقة التفاعل تتغير مع درجة التشوه اللدن بصورة غير عادية في صورة حط مستقيم منكسر ، حيث لوحظ تناقص فجائي كبير في طاقة التفاعل عندما تحطت درجة التشوه اللدن ٣٠٪ .
 - (٤) ان النسيج التشوهي (Deformation Texture . .) والتركيب البنائي للانخلاعات (Dislocation Structure) يلعبان دوراً محسوساً في عملية الاسترجاع .

ABSTRACT

The applicability of the theory of the recovery of coercive force after plastic deformation of a ferromagnetic material has been investigated. It is found that the results obtained for the ferritic stainless steel 1x10 CrAl 24 are in good agreement with the theory discussed. The activation energy of the recovery process is found to be reduced on increasing the degree of plastic deformation. Also, the deformation texture and the dislocation structure play a dominant role in the recovery process.

INTRODUCTION

The influence of the microstructure, in particular internal stresses, on the coercive force of ferromagnetic materials has been known for a long time. In earlier work [1] the internal stresses were characterized by an average stress amplitude and their source remained unspecified. The internal stresses in large pure crystals are known to be due to line defects, namely dislocations [2]. They act as sources of internal stresses that decrease inversely with distance from the dislocation line. During plastic deformation of crystals these dislocations play a two fold role. Their movement constitutes the mechanism of glide, and an increase in their density together with their mutual interaction causes work hardening.

The interaction of magnetization and internal stresses is usually described in terms of the interaction potential between the domain walls and the dislocations. Vicena [3] and Trauble [4] have used this approach to calculate the relation between the coercive force and the dislocation density; the result obtained is:

$$H_c \sim \rho^n \quad (1)$$

where H_c = the coercive force
 ρ = dislocation density
 n = $\frac{1}{2}$ for pure metals and pure alloys

In order to correlate the coercive force directly with the amount of plastic deformation (ε), Kassem [5] has used the relationships between ε and ρ Hahn [6] and Koster et al. [7], together with equation (1) to deduce the relation between the coercive force of a plastically deformed material (H_{cp}) and ε . The deduced relation is:

$$H_{cp} \sim \varepsilon^{n/2} \quad (2)$$

On the assumption that the recovery process follows first-order reaction kinetics and that the overall grain structure of the cold deformed material remains essentially unchanged during such a process, Kassem [5] has deduced a relationship that gives the recovered coercive force of a plastically deformed ferromagnetic material. This correlation is based mainly on the relations (1) and (2) in addition to the theory of recovery given by Kuhlmann et al. [8]. The reported correlation is:

$$H_{cr} = N [K_1 \varepsilon^n - K_2 t \exp(-Q/RT)]^{\frac{1}{2}} \quad (3)$$

where N = the specific change in the coercive force per unit length of dislocation lines.

K_1, K_2 = constants
 t = annealing time
 Q = activation energy
 R = universal gas constant
 T = annealing temperature.

This theoretically deduced relationship has been verified experimentally for pure iron, pure nickel and a pure Fe/Cr solid solution [5]. During the evaluation of the correlation (3), it was found that the activation energy (Q) is dependent on the amount of plastic deformation (ε) given to the material as follows:

$$Q = RT \left[\ln \left(\frac{C + \varepsilon \tan \beta}{K_2 N^2} \right) \right] \quad (4)$$

where β and C are empirically determined constants.

The present investigation deals with the applicability of the above mentioned correlations to a technical ferritic stainless steel x10 CrAl 24 and the calculation of its activation energy of recovery.

EXPERIMENTAL WORK

The studies were performed with a technical ferritic stainless steel x10 CrAl 24 having the chemical composition given in Table 1.

The steel was available in the form of vacuum induction melted ingots (250 x 250 x 500 mm³) which were hot rolled to 3.5 mm plates and then annealed for 10 minutes at 870°C in hydrogen. Finally, a deformation of 5-90% was applied by cold rolling.

The specimens used for measuring the coercive force were 60 x 6 mm² in area and 3.5 mm to 0.35 mm in thickness according to the degree of cold deformation. The coercive force was measured at 20°C by using a precision coercimeter of the type "Firma Foerster, Reutlingen - West Germany" [9 -10]. The accuracy in measurements of the coercive force was better than 0.01 Oe in the range where the values of H_c is greater than 0.20 Oe [9 - 11]. The methods of calibration and measurement as well as the precautions undertaken to avoid any temperature change during experimentation are described elsewhere [10-13].

To initiate and further observe the recovery process, the cold rolled specimens were subjected to isothermal heat treatment. The specimens were annealed in

Table 1: The Chemical Composition of the Steel x10 CrAl 24

C	N	Mn	Si	Al	Cr	P	S	Fe
0.12	0.02	0.35	1.72	1.44	23.9	0.035	0.026	72.389

argon at a temperatures from above 300°C up to 450°C the annealing time varied between 5 min and 240 min.

To measure the dislocation density in particular specimens, at least eight enlarged electron micrographs were used, taken from different and representative regions of the specimens. A square grid was drawn on each micrograph. The total projected length of dislocations was derived from the number of intercepts with the two sets of parallel lines. Using the expression derived by Smith and Guttman [14] and the grid technique developed by Keh and Bain [15] the dislocation density was estimated.

RESULTS AND DISCUSSION

Electronmicroscopy

In order to assess the effect of plastic deformation and annealing on the coercive force of the steel x10 CrAl 24, direct observation of the dislocation configuration in different deformed and annealed states and also an estimate of the dislocation density were essential, so that a truly detailed study of the effect of the microstructure could be carried out.

Figure 1 shows the undeformed initial state of the steel after recrystallization at 870°C/10 min. It can be observed that the dislocation configuration consists mainly of distinct lines, which are statistically distributed in the grain structure of the steel. It was also found that the dislocation density varies from one region to the other, and that the dislocation density of the recrystallized state was relatively low. The observed configuration is typical for recrystallized and undeformed crystalline materials [16] [17].

When the deformation just exceeded 5%, a marked increase in the dislocations was observable and the dislocation lines were found to be distributed statistically on the slip planes (Figure 2). Also, dislocation clusters were sometimes observed.

At higher percentage deformations, the characteristic cell structure of a body-centered cubic metal was detected as shown in Figure 3 and 4. Evaluation of the results obtained by electron microscopy has

shown that the dislocation structures developed with an increased degree of deformation are in good agreement with the data reported in the literature [13-19].

The relation between the measured increase in the dislocation density ($\Delta\rho$) and the amount of plastic

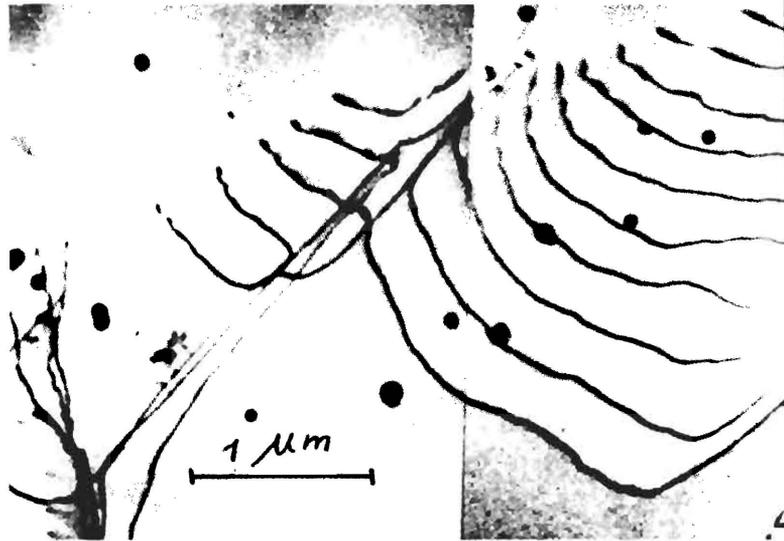


Figure 1. Transmission Electron Micrograph showing the Dislocation Structure in the Recrystallized and Undeformed Steel x 10 CrAl 24.

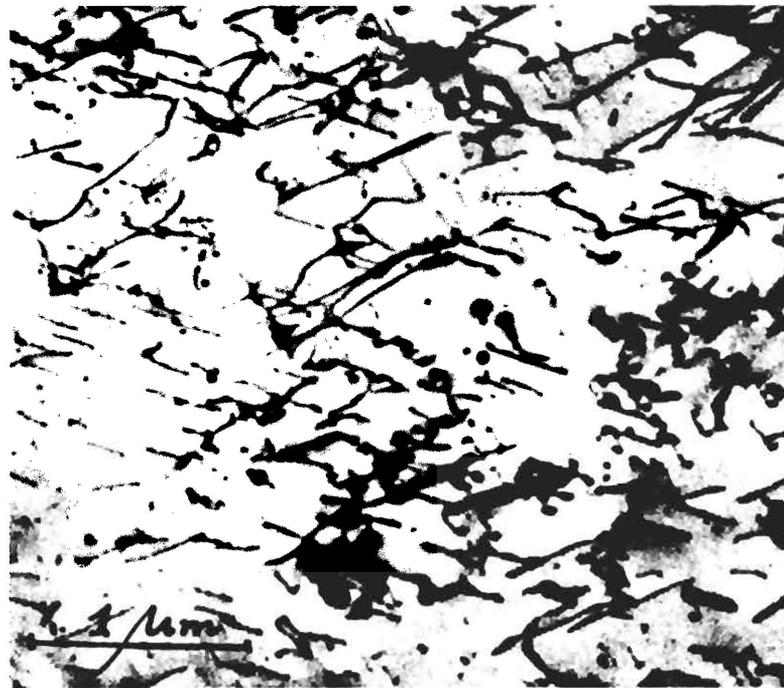


Figure 2. Transmission Electron Micrograph showing the Multiplication of Dislocations after 5% Plastic Deformation of Steel x 10 Cr Al 24.

deformation (ϵ) is presented in Figure 5, which can be mathematically expressed as:

$$\Delta \rho = 5.4 \times 10^9 \epsilon^{0.754} \quad (5)$$

The value of the exponent $n=0.754$ is greater than its value ($n=\frac{1}{2}$) in the case of pure metals and pure alloys [5]. Such a result is acceptable in view of the existence of fine precipitated phases in the microstructure of the steel examined [20]. These precipitates enlarge the rate of multiplication of dislocations during the deformation as compared with pure metals and pure alloys [21].

In order to calculate the activation energies of recovery an estimate of the dislocation density of some of the annealed and deformed states was required. The measured values are listed in Table 2.

Table 2: The Dislocation Density of Some Deformed and Annealed States

Annealing temperature [°C]	Annealing time (min)	ϵ (%)	$\rho \epsilon$ (cm ⁻²)	ρ_t (cm ⁻²)
375	30	30	6.8×10^{10}	6.5×10^{10}
		70	15.8×10^{10}	14.5×10^{10}
400	30	30	6.8×10^{10}	6.3×10^{10}
		70	15.8×10^{10}	13.8×10^{10}

Coercive Force Measurements

The results of the coercive force measurements are presented in Figures 6-8. Figure 6 shows the variation of the coercive force (H_{cp}) with the degree of plastic deformation, i.e. with the change in the dislocation density ($\Delta \rho$). It can be seen that the coercive force of a plastically deformed specimen (H_{cp}) varies exponentially with the degree of plastic deformation as:

$$H_{cp} = 2.78 \times \epsilon^{0.377} \quad (6)$$

Equation 6 shows that the exponent of ϵ is 0.377, which represents half the value of the exponent n in Equation 5. This result verifies the theoretical relationship given by Equation 2.

Figures 7 and 8 show the variation of the square of the coercive force (H_{cp}^2) of the annealed specimens with annealing time (t) at different annealing temperatures (T_a) and various degrees of plastic deformation (ϵ). The plotted results obeyed the theory as stated in Equation 3. The value of the specific change

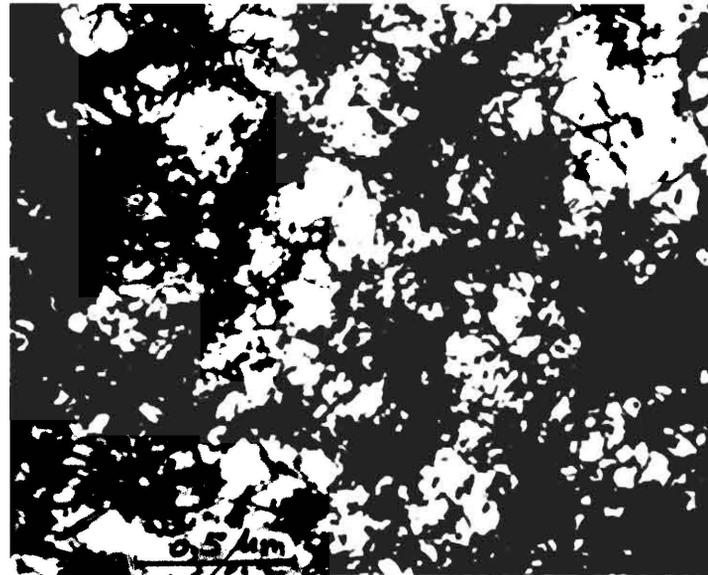


Figure 3. Transmission Electron Micrograph showing the Typical Cell Structure of Dislocations after 10% Plastic Deformation of the Steel x 10 CrAl 24.

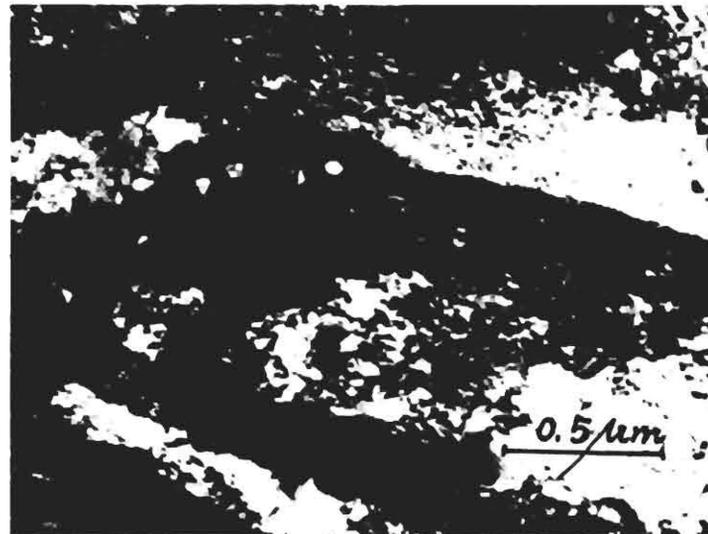


Figure 4. Transmission Electron Micrograph showing Local Inhomogeneities in the Dislocation Structure after 40% Plastic Deformation of Steel x CrAl 24.

in the coercive force per unit length of dislocation lines (N) was also estimated and found to equal $3.80 \times 10^{-5} \text{ Oe cm}^{-1}$.

Activation Energies

On the basis of the isothermal annealing curves the activation energies for the recovery process were calculated by the method proposed by Kassem [5]. Figure 9 represents the relation between $(N^2 K_1^{-1} H_{cp}^2$

– H_{cr}^2) and the annealing time (t). Such a plot allows the calculation:

$$\begin{aligned} \tan \theta &= -N^2 K_2 \exp(-Q/RT) \\ &= \rho_r - \rho_e \end{aligned}$$

i.e. a determination of the change in the density of dislocation during the recovery process as reported in the literature [5].

Using the data obtained by electronmicroscopy (Table 2) and the determined values of $\tan \theta$ and N, the activation energy Q of the recovery process can be calculated. An example of the calculated values degrees of plastic deformation is given in Table 3.

Table 3. Activation Energy of the Recovery Process

Degree of Deformation (%)	Activation Energy eV
00.00	$Q_0 = 2.795$
30.00	$Q_{30} = 2.580$
70.00	$Q_{70} = 2.064$

From the values listed in Table 3, it can be seen that an increase in the degree of plastic deformation reduces the energy required for the recovery process.

From a study of the effect of plastic deformation on the activation energy as given by Equation 4, it was found that the relation between $\tan \theta$ and ϵ consisted of a broken straight line as shown in Figure 10. An attempt was made to understand and clarify the abnormal form of the correlation between $\tan \theta$ and ϵ , but an excessive amount of work would be required to explain the behaviour.

Some preliminary experiments were carried out in order to give an idea about the texture of the rolled specimens. It was found that, as the degree of deformation exceeds 30% a strong preferred orientation was obvious (with an inclination of about 30° to the normal of the test sheet). Moreover, it was observed electron microscopically that, with an increasing degree of deformation the dislocation lines form a homogeneous cell structure all over the specimen. As soon as the plastic deformation exceeded 30%, the cell structure was converted to one of inhomogeneous localities distributed in the matrix (Figure 4). Therefore it may be primarily said that the dislocation structure and the texture of the matrix played an appreciable role in the abnormal behavior shown in Figure 10.

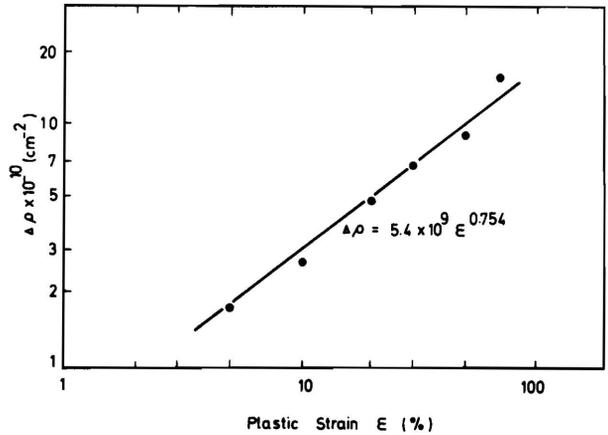


Figure 5. Increase in Dislocation Density with Extent of Plastic Deformation for the Steel x10 CrAl 24.

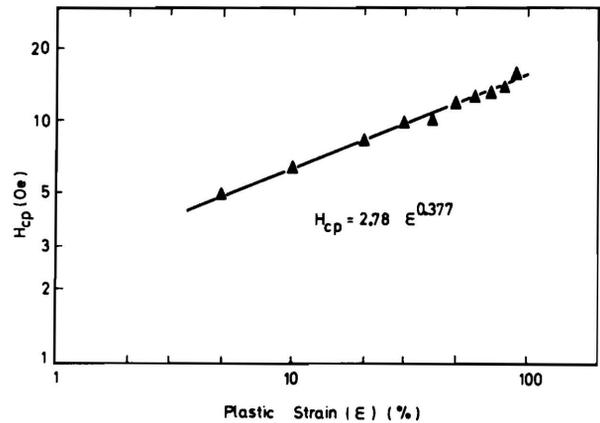


Figure 6. Variation of Coercive Force with the Degree of Elastic Deformation of the Steel x 10 CrAl 24.

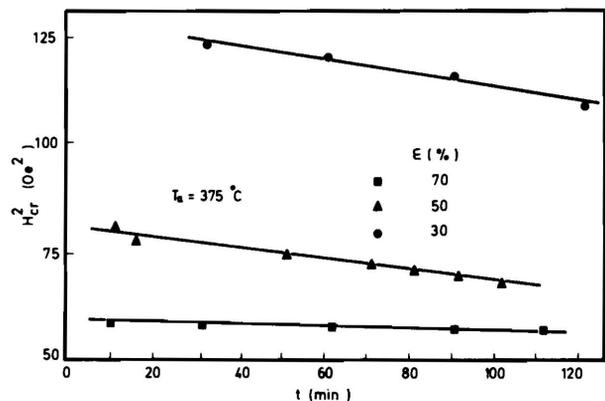


Figure 7. Relation between H_{cr}^2 and Annealing Time for Different Percentages of Plastic Deformation for the Steel x 10 CrAl 24.

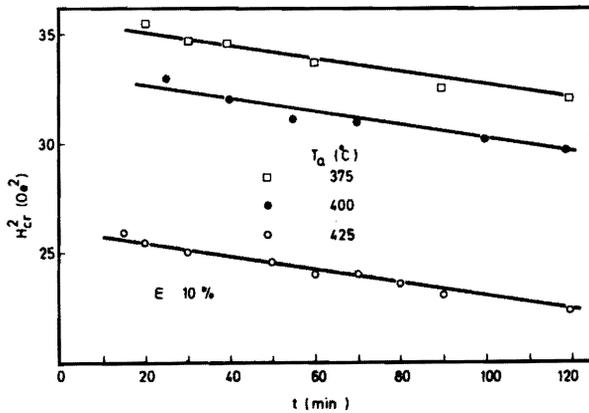


Figure 8. Relation between H_{cr}^2 and the Annealing Time at $\epsilon = 10\%$ at various Annealing Temperatures.

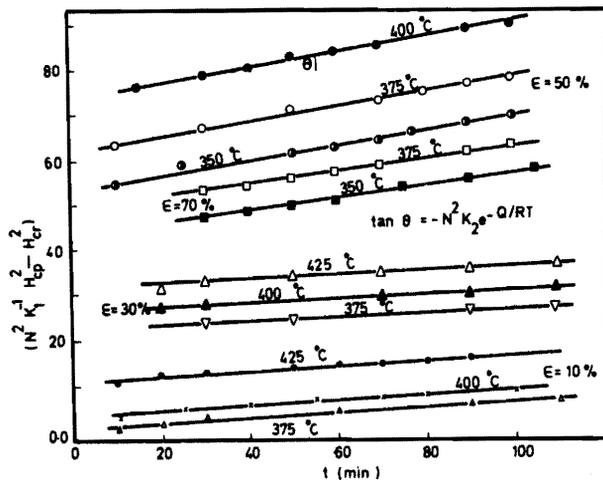


Figure 9. Plot of $(N^2 K_1^{-1} H_{cp}^2 - H_{cr}^2)$ as a Function of Annealing Time.

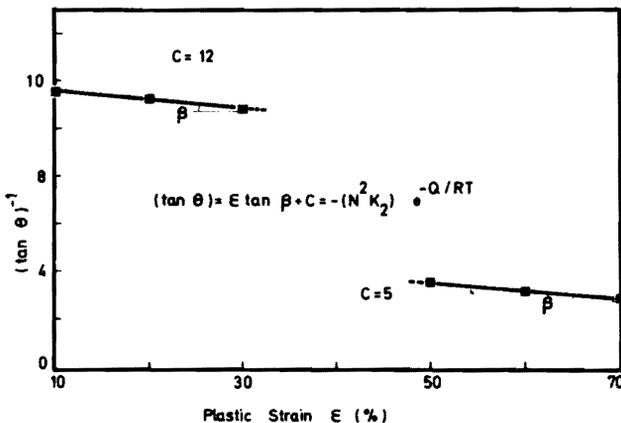


Figure 10. Plot of $\tan \theta$ as a Function of Percentage Plastic Deformation.

CONCLUSIONS

From the data obtained in the present investigation, it can be concluded that:

1. There is a good agreement between the present results and the theory of recovery of the coercive force after plastic deformation.
2. The activation energy of the recovery process is found to be reduced by increasing the degree of plastic deformation.
3. The activation energy varies with the degree of plastic deformation in an abnormal manner through a broken straight line (there was a sharp change in the activation energy between 30% and 50% plastic deformation).
4. The deformation texture and the dislocation structure are found to play an appreciable role in the recovery process.

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