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EFFECT OF ISOTHERMAL ANNEALING ON THE MAGNETIC PROPERTIES OF COLD-ROLLED LOW-CARBON STEEL WITH MAGNETIC HYSTERESIS-LOOP MEASUREMENTS

VPLIV IZOTERMNEGA ŽARJENJA NA MAGNETNE LASTNOSTI HLADNO VALJANEGA MALOOGLJIČNEGA JEKLA Z MERITVAMI MAGNETNE HISTEREZNE ZANKE

Amir Ansaripour, Hossein Monajatizadeh, Jamshid Amighian

Department of Materials Engineering, Najafabad Branch, Islamic Azad University, P.O. Box 517, Isfahan, Iran
ansaripour@outlook.com

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The magnetic properties of ferritic steels including the coercive field and the remanent induction are sensitive to microstructural features such as the grain size and the dislocation density. In this paper the effect of isothermal-annealing treatments at different temperatures on the coercive field and the remanent induction of industrially cold-rolled low-carbon steel samples was investigated. The specimens were annealed at low (500 °C) and high temperatures (640 °C) in order to promote the recovery and recrystallization, respectively. A hysteresis-loop measuring apparatus, specifically designed and assembled for plate samples was used under different annealing conditions. Furthermore, microstructural changes were investigated using light microscopy and hardness measurement and were compared with the data extracted from the device. The result showed that the variation in measured H_c is an appropriate magnetic parameter for following the microstructural evolutions such as recovery and recrystallization.

Keywords: non-destructive testing, annealing, magnetic hysteresis

Magnetne lastnosti feritnih jekel, vključno s koercitivnim poljem in remanentno indukcijo, so občutljive za pojave v mikrostrukturi, kot sta velikost zrn in gostota dislokacij. V tem članku je bil preiskovan vpliv obdelave z izotermnim žarjenjem na koercitivno polje in remanentno indukcijo industrijsko hladno valjanih vzorcev malooglјičnega jekla pri različnih temperaturah preiskovanja. Vzorci so bili žarjeni pri nizki (500 °C) in visoki temperaturi (640 °C) z namenom, da bi pospešili popravo in rekristalizacijo. Uporabljena je bila naprava za merjenje histerezne zanke, posebno prirejena za ploščate vzorce v različnih razmerah žarjenja. Spremembe mikrostrukture so bile preiskovane s svetlobnim mikroskopom in izmerjena trdota je bila primerjana s podatki, dobljenimi iz naprave. Rezultati so pokazali, da je spreminjanje izmerjenega H_c pravi magnetni parameter za spremljanje razvoja mikrostrukture, kot sta poprava in rekristalizacija.

Ključne besede: neporušne preiskave, žarjenje, magnetna histereza

1 INTRODUCTION

It is well known that work-hardened steel sheets require an annealing process, during which the material softens and recovers its ductility and formability.[1] The main softening mechanisms in metallic materials are recovery and recrystallization. The former leads to an annihilation of dislocations and their re-arrangement into low-energy sub-boundaries and the latter results in a nucleation and growth of new strain-free grains.[2,3] Numerous studies have been published regarding the study of the recovery and recrystallization kinetics using different methods such as the thermal analysis, TEP (thermo-electrical power), X-ray diffraction, metallography and, in recent years, magnetic techniques.[4,5] Among them, the last set of techniques is potentially able to be used non-destructively. This is of great importance because different amounts of softened material at various points of the steel coil that are inherently characteristic of box annealing, may lead to a variation in the mechanical properties and formability of sheets.[6] An application of a magnetic measuring technique on a production line of steel coils can give an appropriate evaluation of the variation in the mechanical properties along a coil.

Magnetic properties of steels depend on microstructural features such as dislocation density, grain and sub-grains size, precipitates and other structural defects.[7–9] The first two parameters are affected mainly during the annealing process[3]. Little has been published about the evaluation of annealing cold-rolled low-carbon steels by means of magnetic parameters. Martinez et al.[10–12] surveyed the recovery and the onset of recrystallization, using non-destructive magnetic tests, in low-carbon steels. They measured the coercive force (H_c) and found that this parameter is sensitive to the evolution of microstructure during annealing, using equation $H_c \propto \sqrt{\rho/d}$, in which ρ is the dislocation density and d is the average grain diameter. However, the annealing progress in the form of a recrystallization volume fraction and a grain growth still needs to be examined.

In the present work, a measuring direct pickup coil and a Hall-sensor system were applied to the plate samples to estimate the magnetic properties of the steels that have different microstructures consisting of cold-worked and also fully and partially recrystallized grains. The aim was to find the effect of the microstructure change during annealing on H_c and B_r and to follow the non-destructive characterization of the microstructural changes produced due to the recovery and recrystallization during the annealing of the cold-rolled low-carbon steel presented above.

2 EXPERIMENTAL PROCEDURE

2.1 Materials

The composition of the studied steel is shown in **Table 1**. The samples selected from the industrially produced coils were cold rolled to a final thickness of 0.8 mm through a 75 % reduction.

Table 1: Chemical composition of the samples (mass fractions, *w*/%)
Tabela 1: Kemijska sestava vzorcev (masni deleži, *w*/%)

C	Si	Mn	Al	N (10^{-6})	Fe
0.044	0.008	0.229	0.049	25	balance

The samples were isothermally annealed in the laboratory heat-treatment furnace, in which they were heated up in an argon atmosphere at a rate of 10 °C/h according to the cycle shown in **Table 2**. Then the samples were cooled in air to ambient temperature.

2.2 Hardness and metallographic measurement

Superficial Rockwell-hardness (HR30T) measurements were performed on the sheet of the annealed samples with a steel indenter in the form of a sphere with a 1.58 mm (1/16 in.) diameter subjected to a load of 30 kg. The full load was applied for 30 s according to ASTM E18, while the Vickers-hardness measurements were carried out on the transverse section according to ASTM E92. Each sample was measured five times and the average values were calculated.

Longitudinal sections of the investigated steels were prepared for microscopic examinations with the standard metallographic technique. Having been ground and polished, the metallographic samples were etched with 3 % Nital and examined using an optical microscope.

2.3 Magnetic measuring system

The magnetic measuring system consisted of a U-shaped magnetizing (Fe-Si laminated) core with an excitation coil (200 turns), supplied with a sinusoidal (0.5 Hz) exciting current. The field was measured using a Hall probe placed on the surface of the sample and the direct pickup coil (50 turns) that was wound around the specimen, as depicted in **Figure 1**. The latter was used to compare and validate the flux density measured with the coil. The system is based on a PC, through which an input-output A/D card transfers the signals of the measured magnetic-field strength (H) and the flux density (B).

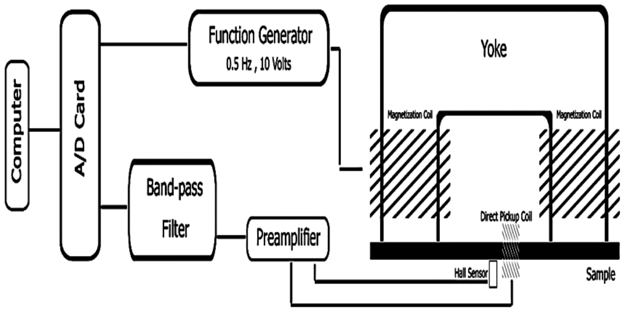

Figure 1: Schematic diagram of the system for measuring magnetic parameters
Slika 1: Shematski prikaz sistema za merjenje magnetnih parametrov

The field in a tested sample was evaluated to be proportional to the exciting current (I) and calculated from equation 1:

$$H = \frac{NI}{l} \quad (1)$$

where N is the number of the exciting-coil turns, and l is the effective magnetic path.

The magnetic induction signal was obtained with an integration of the induced voltage on a 50-turn encircling coil wound around the samples. The flux density (B) was obtained after integrating the pickup-coil induced voltage, and calculated from equation 2:

$$B = \frac{NI}{N_p S} \int V \mathrm{d}t = \frac{1}{N_p S F_s} \sum_{i=1}^{N} Vi \quad (2)$$

where N_P is the number of pickup coil turns, S is the total section area of the pickup coil, F_s is the sampling frequency and V is the voltage induced in the pickup coil.

3 RESULTS AND DISCUSSION

Figure 2 shows the evolution of the microstructures of the samples during isothermal annealing at 500 °C. As can be seen in the figure, the structures consist of elongated grains along the rolling direction and microbands

Table 2: Annealing cycles used in this study
Tabela 2: Uporabljeni cikli žarjenja

L (500 °C)	L1	L2	L3	L4	L5	L6	L7	L8	L9	L10
H (640 °C)	H1	H2	H3	H4	H5	H6	H7	H8	H9	H10
Annealing time (s)	100	300	600	1200	1800	2700	3600	5400	7200	10800

*L: Low-temperature annealing, *H: High-temperature annealing

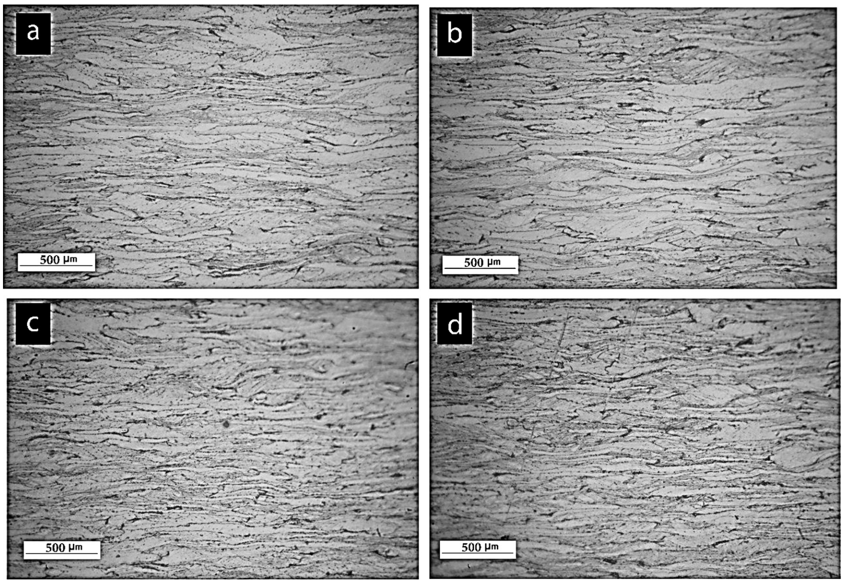

Figure 2: Light micrographs showing the microstructures of the samples after the isothermal annealing at 500 °C: a) 100 s, b) 600 s, c) 1800 s, d) 10800 s
Slika 2: Posnetki mikrostrukture vzorcev po izotermnem žarjenju pri 500 °C: a) 100 s, b) 600 s, c) 1800 s, d) 10800 s

are evident inside them. The soaking time increased up to 10800 s does not noticeably change this structure.

As can be seen, **Figure 3a** reveals the initially deformed grains elongated in the rolling direction without any signs of recrystallization but, during the annealing carried out at 640 °C after about 600 s of soaking (**Figure 3b**), newly recrystallized grains take place. A more increased soaking time (**Figure 3c**), increases the fraction of recrystallized grains, with nearly 90 % of the structure recrystallized after 1800 s. The recrystallization process is completed after 2700 s and the grain growth (**Figure 3d**), is observed after 10800 s.

Figure 4 illustrates the evolution of the hardness of a sample during isothermal annealing. It can be seen in the figure that the hardness is approximately insensitive to the annealing temperature at 500 °C when the softening of the material is governed by the recovery. The Vickers-hardness tests carried out on the transverse section of the sheet give the same type of result. As an example, HV for L1 is 220 and after a 10800 s soaking time at 500 °C it only decreases to 212 HV. At 640 °C after a 300 s soaking time, when the static recrystallization is started, a sudden decrease in the hardness is observed. As the hardness tests of the present steel do not give any infor-

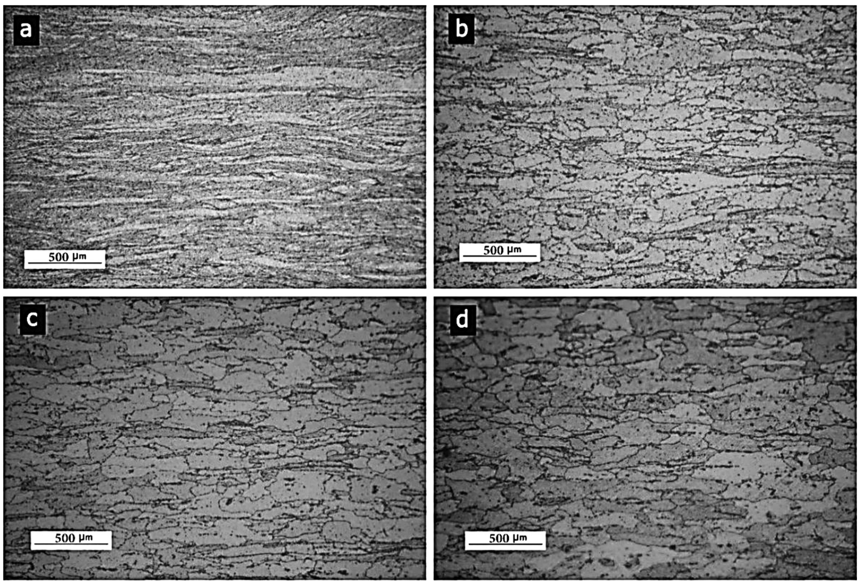

Figure 3: Light micrographs showing the microstructures of the samples after the isothermal annealing at 640 °C: a) 100 s, b) 600 s, c) 1800 s, d) 10800 s
Slika 3: Posnetki mikrostrukture vzorcev po izotermnem žarjenju pri 640 °C: a) 100 s, b) 600 s, c) 1800 s, d) 10800 s

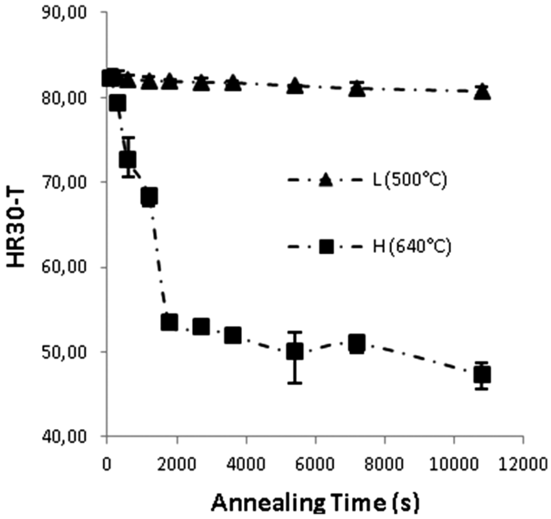

Figure 4: Evolution of the hardness as a function of the annealing time and temperature
Slika 4: Razvoj trdote v odvisnosti od časa in temperature žarjenja

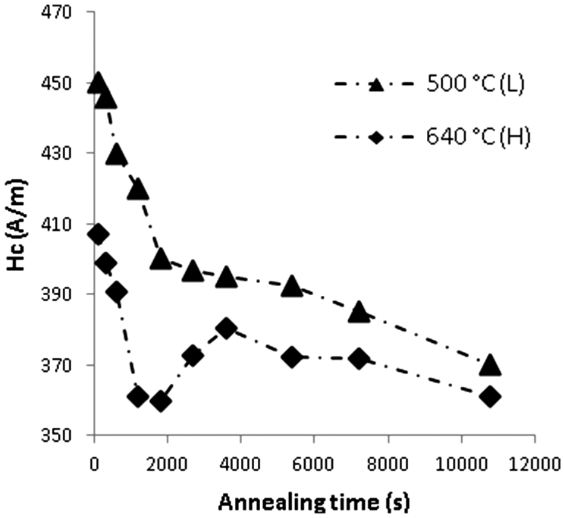

Figure 6: Evolution of the coercive field (H_c) with the annealing time
Slika 6: Razvoj koercitivnega polja (H_c) s časom žarjenja

mation about a possible recovery taking place during the annealing carried out at the lowest temperatures, magnetic methods were applied in order to get a higher degree of resolution and investigate this phenomenon.

The effect of increasing the annealing time in the hysteresis loops is shown in **Figure 5**. The *B–H* loops are found to become steeper with the annealing, providing lower H_c, and higher B_r values.

The general shape of the curves shows a knee in the middle where the difference between the curves is more pronounced, as shown more clearly in the right-hand corner of **Figure 5**. Above and below the knees, the curves become approximately coincident.

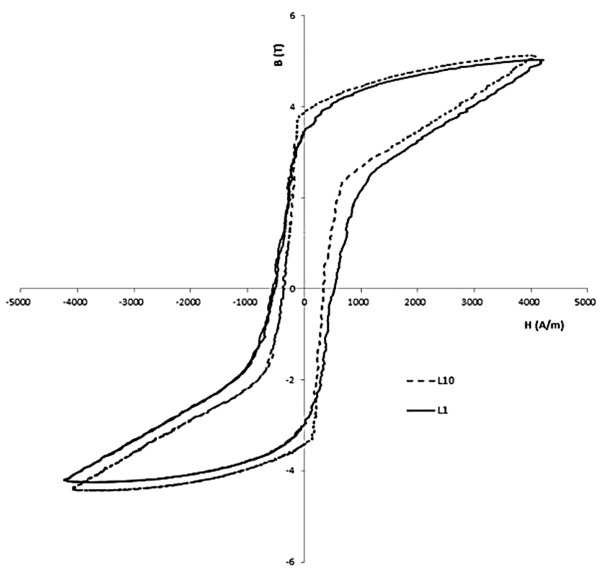

Figure 5: Hysteresis *B–H* loops showing the effect of the annealing treatment at 500 °C
Slika 5: Histerezna zanka *B–H* prikazuje vpliv žarjenja pri 500 °C

The evolutions of H_c and B_r with the annealing progress are illustrated in **Figures 6** and **7**. Although both magnetic parameters show an inverse trend, it is observed that an increase in the annealing time from 100 s to 10800 s decreases H_c up to 50 A/m, while the corresponding drop in B_r represents no meaningful change in the high-temperature isothermal annealing. This confirms that H_c is more sensitive to the microstructural evolution during annealing.

A comparison between **Figures 4** and **6** shows that at 500 °C, when the recovery is the governing factor[4] both magnetic parameters are sensitive to the microstructural evolution, whereas the hardness is insensitive to it.

At 640 °C and after about a 1000 s soaking time the rate of decrease in H_c is relatively diminished due to the start of the recrystallization, as can be seen in **Figure 6** (*H*). To explain this diminution, one may refer to the effect of the grain size on H_c according to relation ($H_c \propto$

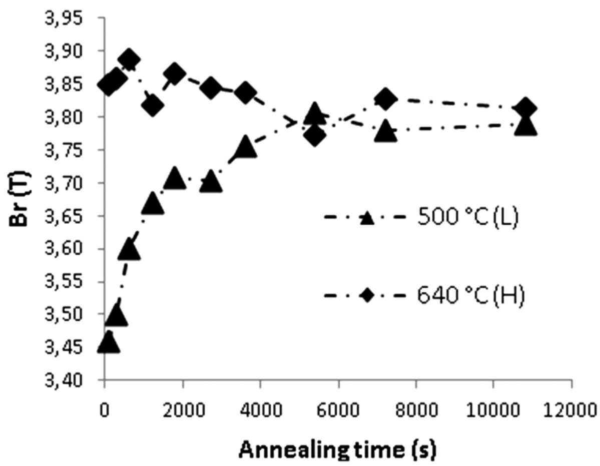

Figure 7: Evolution of the remanent induction (B_r) with the annealing time
Slika 7: Razvoj remanentne indukcije (B_r) s časom žarjenja

$\sqrt{\rho/d}$). When the grain size is reduced, H_c increases[9–19]. The domain-wall motion and rotation under the magnetic field is affected by the microstructural parameters such as grain boundaries, in addition to the dislocation density and inclusions. Grain boundaries present obstacles to the domain-wall motion acting as the pinning centers for the domain walls. As the grain size increases, the number of grains and the total length of grain boundaries decrease and, therefore, the pinning effect of the domain-wall motion decreases.[11–19] The start of the recrystallization leads to a decrease in the average grain size and, thereafter, an increase in the pinning effect of grain boundaries on the domain-wall motion. This is opposite to the effect of an annihilation of dislocations during domain-wall motion and, hence, the rate of decrease in H_c diminishes due to the resultant competition between these two phenomena. At the end of the 1800 s period, the contribution of the pinning effect due to the decreasing average grain size becomes higher than the softening effect of the dislocation annihilation and H_c starts to increase, as depicted above. Due to the growth of the recrystallized grains, this resistance to the domain-wall motion is decreased and H_c starts to decrease again.

These results show that H_c is sensitive enough to the microstructural changes such as recovery and recrystallization.

4 CONCLUSIONS

In the present work, magnetic hysteresis loops of cold-rolled steel-plate samples were measured at different times after isothermal annealing. Two parameters, the coercive force (H_c) and the remnant induction (B_r), in the hysteresis loop were analyzed and compared with the hardness measurement. It was found that both parameters decrease with the increasing time of annealing and the progressing recovery process. Also, while hardness is not sensitive to the microstructural changes during recovery, H_c can be useful for its non-destructive characterization. Moreover, H_c accurately characterizes various stages of recrystallization during the isothermal annealing of cold-rolled low-carbon steel and it can be used as a non-destructive tool for controlling the annealing progress in steel sheets.

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