MAGNETIC ESTIMATION OF LATTICE MICRODISTORTIONS IN HEAT-TREATED AND DEFORMED STRUCTURAL STEELS

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ABSTRACT

The paper presents results of estimating lattice microdistortions induced CLM (and, consequently, residual stresses) in structural steels being hardened by heat treatment or plastic deformation. Magnetic parameters, electrical resistivity and elastic wave velocity are shown to be unequivocally dependent on the CLM magnitude in steels subjected to plastic deformation by equal-channel angular pressing, hydroextrusion, drawing and controlled rolling. This enables us to state that it is essentially possible to estimate CLM in steels by measuring these parameters after deformation hardening.

The study of heat-treated steels has shown that it is the root-mean-square value of magnetic Barkhausen noise voltage that correlates best with lattice microdistortions occurring in steels under quenching from different temperatures. When CLM are estimated in steel products after martensite quenching followed by tempering, it is the most effective to use simultaneously coercivity (after low and medium tempering) and the rms value of magnetic Barkhausen noise voltage (after high tempering). The other parameters are less sensitive to CLM alterations in heat-treated steels.

Key words: steels, crystal lattice microdistortions, magnetic parameters

1. Introduction

Crystal lattice microdistortions CLM (and, consequently, residual stresses) occurring in structural materials while products are being made and used govern the lifetime of these products considerably. Therefore the determination of CLM is an urgent problem of nondestructive testing. For steel products, CLM estimation methods based on measuring the magnetic characteristics of the object under testing, such as coercive force, magnetic permeability, Magnetic Barkhausen noise parameters (MBN), are widely applied [1-5]. Hence it seems interesting to compare the effectiveness of using magnetic characteristics and the elastic wave propagation velocity determined by the parameters of the signal of electromagnetic-acoustic transduction to estimate CLM in products made of ferromagnetic structural steels subjected to various plastic deformation processes and martensite quenching followed by tempering.
2. Specimens and investigation techniques

Specimens made of structural steels of grades 09G2S, 65G, 70, U8A and pipe steels X70 and 35 were studied.

The effect of CLM caused by plastic deformation on the magnetic characteristics of structural steels was studied on specimens made of steels 09G2S (0.06 % C, 1.76 % Mn, 0.89 % Si), 70 (0.7 % C), U8A (0.75 % C) and controlled-rolling pipe steel Х70 similar in the composition to steel 09G2S. The 09G2S steel specimens were deformed by equal-channel angular extrusion (channel intersection angle 120º; extrusion temperature 350º С; 2 passes), the specimens made of patented steel 70 were drawn at room temperature from 2.6 mm to 0.45 mm in diameter without intermediate annealing, and the U8A specimens were deformed by hydroextrusion at room temperature from 18 mm to 8 mm in diameter also without intermediate annealings. The X70 steel specimens were cut out from a welded spiral pipe 1420 mm in diameter and 21.6 mm in wall thickness. The level of CLM in the specimens made of these steels was varied by annealing at temperatures ranging between 100 and 700º С.

The specimens made of steels 35 (0.36 % C), 65G (0.62 % C, 0.96 % Mn) and U8A were quenched for martensite and then tempered at temperatures ranging between 100 and 650º С.

The crystal lattice microdistortions in the specimens were determined from X-ray diffraction analysis along the (211) line of the α-phase with the use of monochromatized Кα -radiation of a chromium anode.

A permeameter circuit was applied to determine the magnetic characteristics of the specimens. A magnetic field of up to 600 A/cm was applied along the long specimen axis. The coercive force ($H_c$) and maximum magnetic permeability ($\mu_{\text{max}}$) were determined, as well as magnetization in a maximum applied field, which is approximately equal to saturation magnetization ($M_s$).

A resonance technique was used to determine elastic wave propagation velocity $V$ by electromagnetic-acoustic transduction (EMAT) with the use of through-type transducers. A zero symmetric mode of longitudinal normal elastic waves was used. The external polarising permanent magnetic field generated by an electromagnet and directed along the specimen axis was selected for each specimen by the maximum amplitude of the EMAT signal in resonance.

The magnetic Barkhausen noise parameters were determined with the application of a superimposed transducer. The root-mean-square value of MBN voltage ($U$) and the number of Barkhausen jumps ($N$) in a series of 10 magnetization reversal cycles were used as informative parameters. The transducer was placed in the middle of the specimen so that, during the measurement of MBN parameters, it can be magnetized along the long axis. The form of magnetizing current was close to triangular. The amplitude and frequency of magnetisation reversal current were selected experimentally by the maximum value of voltage $U$. For the materials tested, they were 120 mA and 20 Hz respectively. Measurements of $U$ and $N$ were made with transducer replacement for each specimen, the results being averaged thereafter. The maximum deviation of $U$ and $N$ from the average values did not exceed 6 %.

3. Results and discussion

3.1. The effect of crystal lattice microdistortions of plastically deformed steels on their magnetic characteristics

Figure 1a presents crystal lattice microdistortions, coercive force and maximum magnetic permeability as dependent on the annealing temperature $T_{\text{ann}}$ of plastically deformed steels 09G2S, 70, U8A and X70. Since the phase composition of these steels remains unchanged under annealing (as evidenced by constant values of their saturation magnetization), CLM and $H_c$ decrease monotonically due to the relaxation of internal stresses, and $\mu_{\text{max}}$ decreases. The behaviour of CLM in the steels that have undergone equal-channel angular extrusion and controlled rolling is worthy of notion: at annealing temperatures below $\approx 400^\circ$ С, the CLM of...
these steels decreases only slightly, and it is only at higher annealing temperatures that the intensity of CLM relaxation grows significantly.

Fig. 1: Crystal lattice microdistortions, coercive force and maximum magnetic permeability as functions of the annealing temperature of plastically deformed steels 09G2S, 70, U8A and X70 (a); the magnetic parameters of steels 09G2S, 70, U8A and X70 as functions of crystal lattice microdistortions (b).

The unique behaviour of the CLM dependences of $H_c$ and $\mu_{\text{max}}$ (see Fig. 1b) enables CLM in steels strain-hardened by different processes to be estimated by the coercimetric method or by measuring maximum magnetic permeability.

3.2. The effect of tempering temperature on crystal lattice microdistortions and the properties of quenched steels

CLM in deformed steels result mainly from the increasing density of crystal structure defects, the phase composition being unaffected. It is more difficult to estimate CLM in steel products hardened by quenching followed by tempering, since the magnetic behaviour of heat-treated steel are affected not only by dislocation density variation, as is the case with plastic deformation, but also by changes in the granular structure of the material and its phase
composition.
Figure 2 presents crystal lattice microdistortions, magnetic characteristics, magnetic Barkhausen composition. In the first stage of tempering, CLM remain unchanged up to \( T_{\text{tem}} \approx 100\ldots160^\circ\text{C} \) for all the steels, whereas they decrease with a further rise in the tempering temperature. This behaviour of the \( T_{\text{tem}} \)-dependences of CLM results from two effects: quenching distortions caused by the \( \gamma \rightarrow \alpha \) transformation and non-uniform metal cooling during quenching decrease practically linearly as \( T_{\text{tem}} \) rises, and the so-called coherent distortions caused by the formation of finely dispersed carbides, whose lattice is coherent with martensite, increase at tempering temperatures ranging between 20 and 350\(^\circ\)C [6]. Residual austenite decomposition occurring in the first stage of tempering also contributes to a decrease in CLM. Residual austenite decomposition is evidenced by increasing saturation magnetization at tempering temperatures exceeding 160\(^\circ\)C (Fig. 2). According to the measurements of \( M_s \), austenite decomposition is completed at \( T_{\text{tem}} \approx 250^\circ\text{C} \) in the steels studied. It follows from Fig. 2 that the above-described structural-phase transformations in the first stage of tempering are accompanied by decreasing \( H_c \), electrical resistivity \( \rho \) and increasing \( \mu_{\text{max}} \) and \( V \). These transformations cause significant changes in MBN voltage only in the case of steel 35, for which \( U \) tends to grow (Fig. 2). For steels 65G and U8A, MBN voltage remains unchanged at tempering temperatures ranging between 20 and 250\(^\circ\)C. The number of Barkhausen jumps \( N \) linearly decreases for steels 35 and U8A, whereas it remains practically constant in this temperature range for steel 65G. When the tempering temperature exceeds 250\(^\circ\)C, there are a further decrease of quenching distortions and some growth of coherent distortions [6]. According to [6], at \( T_{\text{tem}} \approx 350^\circ\text{C} \), coherent distortions start to decrease in carbon steels due to the onset of carbide crystal isolation, and there arise dispersion distortions resulting from the fact that the specific volume of isolated carbides differs from those of the \( \alpha \)-phase and carbides coherently connected with the matrix. The cumulative effect of these factors results in a further monotonic decrease in CLM as the tempering temperature rises to 400\(^\circ\)C. The growth of carbide content in the second stage of tempering is evidenced by decreasing saturation magnetization of the U8A and 65G steels with growing \( T_{\text{tem}} \) (Fig. 2). For steel 35, some decrease in saturation magnetization is observed only at \( T_{\text{tem}} > 450^\circ\text{C} \). At tempering temperatures ranging between 250 and 400\(^\circ\)C there is a further decrease in \( H_c \), \( N \) and \( \rho \) and a further increase in \( \mu_{\text{max}} \) and \( V \) for all the steels, with the rms value of MBN voltage increasing for steel 35. Besides, for steels 65G and U8A tempered at 350…400\(^\circ\)C the values of \( H_c \) and \( \mu_{\text{max}} \) vary insignificantly due to the onset of cementite particle coagulation. It follows from the reported results that, in the first two stages of tempering, it is only for steel 35 that MBN parameters demonstrate tangible changes with varied \( T_{\text{tem}} \) and, consequently, CLM. In the third stage of tempering (\( T_{\text{tem}} > 400^\circ\text{C} \)), CLM are mainly due to dispersion distortions, with their maximum falling on \( T_{\text{tem}} \approx 450^\circ\text{C} \) [6]. For the U8A and 65G steels, as CLM decreases monotonically, the coercive force values have a maximum pronounced to a different extent in proximity to \( T_{\text{tem}} \approx 500^\circ\text{C} \) (Fig. 2). That is, according to the inclusion theory [7], at \( T_{\text{tem}} \approx 500^\circ\text{C} \), the size of carbide inclusions becomes comparable to the thickness of domain walls in these steels. For steel 35, the dependence \( H_c(T_{\text{tem}}) \) has no extrema due to a lower carbon content than in the other steels and, consequently, a smaller number of carbides. At the same time, at \( T_{\text{tem}} \approx 500^\circ\text{C} \) there are minima on the dependences \( \mu_{\text{max}}(T_{\text{tem}}) \) for all the steels studied. The structural transformations in the range 400…610\(^\circ\)C result in lower values of \( N \) and \( \rho \) and higher values of \( U \). Thus, the processes of cementite isolation and coagulation do not make the dependences \( N(T_{\text{tem}}) \), \( \rho(T_{\text{tem}}) \) and \( U(T_{\text{tem}}) \) non-unique, as distinct from the coercive force and maximum magnetic permeability.
Fig. 2: The effect of tempering temperature on crystal lattice microdistortions, magnetic characteristics, electrical resistivity and elastic wave propagation velocity for steels 35 (○), 65G (■) and U8A (▲).

For steels 35 and 65G, the elastic wave propagation velocity increases further at $T_{\text{tem}}$ exceeding 400°C, whereas, for the steel with higher carbon content (U8A), $V$ decreases a little as the tempering temperature varies from 450 to 500°C (Fig. 2). This may be because in the U8A steel there appears the greatest quantity of cementite, whose Young’s modulus is 20 % lower than that of solid solutions of carbon in iron [8], the velocity of the elastic wave mode excited in our experiments being proportional to the square root of Young’s modulus [9]. Besides, as is obvious from Fig. 2, the value of $V$ is affected by cementite only in high-carbon steel and only after coagulation of precipitated carbide.

Referring to Fig. 3, for steel 35, on the CLM-dependence of $H_c$ there is no extremum related to cementite coagulation; however, for the CLM range 0.05…0.15 %, the coercive force is almost...
not sensitive to changes in CLM. For steels 65G and U8A, the unique CLM-dependence of $H_c$ is observed only at CLM exceeding 0.16 %, that is, at tempering temperatures below 450° C. At CLM < 0.16 %, the CLM-dependences of $H_c$ are non-unique for the high-carbon steels. Thus the coercimetric technique for estimating CLM is inapplicable to products made of carbon steels after tempering accompanied by coagulation of carbide precipitations.

Fig. 3: Magnetic parameters, electrical resistivity and elastic wave propagation velocity as dependent on the relative value of crystal lattice microdistortions for steels 35 (○), 65G (■) and U8A (▲).

For steel 35, $\mu_{\text{max}}$ demonstrates a 3-times monotonic decrease as CLM grow from 0.05 to 0.24 % (Fig. 3). For steel 65G, all the variations of $\mu_{\text{max}}$, namely, a decrease from 600 to 150, occur when CLM range between 0.15 and 0.28 %. Beyond this range, the maximum magnetic permeability is insensitive to CLM variations. In the case of steel U8A, an increase in CLM from 0.14 to 0.4 % entails a decrease in the maximum magnetic permeability, and with CLM below 0.14 %, that is, after tempering causing carbide coagulation, there is a non-unique CLM dependence of $\mu_{\text{max}}$. Consequently, maximum magnetic permeability is also inapplicable as a test parameter when estimating CLM in products made of high-carbon steels after quenching and high tempering.

The electrical resistivity of all the steels increases monotonically with growing crystal lattice microdistortions. In the case of steel 65G, when CLM grow from 0.05 to 0.31 %, electrical resistivity grows by a factor of 1.5 and more. For medium-carbon steel 35, this dependence is
also monotonic, $\rho$ grows linearly with CLM, the change of $\rho$, however, being only by 20 % (Fig.2.) The electrical resistivity of eutectoid steel U8A increases about 1.2 times in the CLM range 0.05…0.37 %, and it varies much more intensively with a further growth of CLM. Thus, for all the steels studied, electrical resistivity varies uniquely with CLM. However, the estimation error for $\rho$ is rather large, and in the case of steel 35 the variation of electrical resistivity with CLM does not exceed the measurement error. Besides, it must me taken into consideration that steels have a high temperature coefficient of resistance: the elevation of temperature by $10^\circ$ C raises the electrical resistivity of the steels by about 7 % [10].

It is obvious from Fig. 3 that the rms value of MBN voltage decreases monotonically with increasing CLM for all the steels. Yet, significant changes in $U$ are observed for the high-carbon steels only at CLM not exceeding 0.20 %. Thus the rms value of MBN voltage is fairly sensitive to the crystal lattice microdistortions of the heat-treated high-carbon steels in the CLM range where the coercimetric technique is inapplicable. In the instance of heat-treated medium-carbon steel, $U$ is fairly sensitive to CLM changes in the whole range of these changes.

The number of Barkhausen jumps linearly decreases in the whole range of CLM changes for both medium-carbon and high-carbon steels. However, the change in $N$ is small, namely, about 20 % (Fig. 3).

The CLM-dependences of $V$ are unique for the steels with 0.36 and 0.62 % carbon, whereas the elastic wave propagation velocity behaves nonmonotonically with increasing CLM for steel U8A, namely, it increases within the CLM range 0.05…0.11 %, i.e. after high tempering, and it decreases notably with a further growth of CLM, i.e. at lower tempering temperatures (Fig. 3).

It follows from the results reported that it is impossible to estimate crystal lattice microdistortions in carbon steels in the whole range of tempering temperatures by a single magnetic parameter, especially in steels with high carbon content. It is the most effective, when estimating CLM, to use simultaneously the coercive force and the rms value of MBN voltage, namely, the coercimetric method is applicable at tempering temperatures below about 300…350$^\circ$ C, and the measurement of the rms value of MBN voltage can be applied at higher $T_{tem}$.

4. Conclusion

The unique CLM dependences of the magnetic parameters of steels undergoing plastic deformation by equal-channel angular extrusion, hydroextrusion, drawing and controlled rolling principally enable crystal lattice microdistortions in strain-hardened steels to be estimated by measuring such parameters as coercive force and maximum magnetic permeability. Investigations made on heat-treated steels have demonstrated that it is the rms value of magnetic Barkhausen noise voltage that has the best correlation with crystal lattice microdistortions arising in steels quenched from various temperatures. When crystal lattice microdistortions are to be estimated in steel products after martensite quenching followed by tempering, it can best be done through the simultaneous use of coercive force (after low and medium tempering) and the root-mean-square value of MBN voltage (after high tempering). The other parameters are less sensitive to changes in crystal lattice microdistortions in heat-treated steels.

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5. References


