QUANTITATIVE NONDESTRUCTIVE EVALUATION OF CEMENTITE IN STEEL AND WHITE CAST IRON BY FERROMAGNETIC PARAMETERS

I. Altpeter and P. Holler

Fraunhofer-Insitut fur Zerstorungsfreie
Prufverfahren (IzfP), Saarbrucken, FRG

INTRODUCTION

The change in the microstructure state (lattice defects, especially dislocations, precipitations and thus the stress fields) with the addition of alloying elements plays an important role for the strength and the toughness of a material. In order to determine these microstructural parameters, up to now the electron microscopy and similar methods are used. X-ray methods are normally used to determine residual stresses.

In this paper, the micromagnetic method has been chosen because it can be used for both nondestructive stress measurement and the material characterization. This micromagnetic method developed in the IzfP is called the 3MA-method (micromagnetic, multiparameter, microstructure and stress analysis based on its capability. The nondestructive characterization of materials by means of magneto-dynamic analysis is supported by the fact that material-specific lattice defects (i.e., dislocations) have the same linear dimensions as the Bloch-walls and thus there exists an analogy between the dislocations and the Bloch-walls, concerning their interaction with the precipitations and/or the residual stresses of the first, second and third kind.

The nondestructive determination of the cementite (Fe_3C) content is an important part of the total characterization of the microstructure in ferritic materials using magnetic testing methods because the cementite is represented as a phase in different material states, i.e. in the tempered martensitic states, in the ferritic/perlitic structure, and in the white cast iron.

The nature of the cementite phase (shape, volume, density) provides an important contribution to the increase in the yield strength. For example, in the as-delivered state (3.0 wt% Fe_3C, $R_{p0.2}$ = 470 PMa) of the steel SA 508 Cl.2, the contribution of the Fe_3C-precipitation for the increase in the yield strength is about 12% /1/.

According to the theories for the increase in strength /2/, in addition to the density and the kind and the shape of the cementite, grain size, sub-grain size, dislocation density and dissolved impurity atoms also contribute to the increase in the yield strength. In order to evaluate the yield strength nondestructively, it is necessary to know the microstructural characteristics, which are usually determined by microscopic methods. In order to be able to determine these

Review of Progress in Quantitative Nondestructive Evaluation, Vol. 9
Edited by D.O. Thompson and D.E. Chimenti
Plenum Press, New York, 1990

characteristic values nondestructively, it is important to develop a nondestructive testing method for the evaluation of the cementite content. The evaluation of the cementite content is also useful for the grading of steels and white cast iron. All the traditional methods for the evaluation of the cementite content, such as the Mossbauer spectroscopy /3/, the quantitative X-ray phase analysis /4/, the phase analysis by electrochemical dissolution of the matrix /5/, and the analysis of micrographs made by the transmission electron microscope or the light microscope /6/ are expensive, time consuming and, except the X-ray phase analysis and Mossbauer spectroscopy in backscattering technique, require the destruction of the testing component.

MEASURED RESULTS/DISCUSSION

In the two-phase material containing ferrite and cementite, the following interactions between the matrix and the second phase are possible:

First, there is the interaction between the Bloch-walls of the ferrite matrix and the cementite particle, which acts as a foreign body /7/. The interaction between the Bloch-walls of the ferrite matrix and the magnetic stray fields, which are caused by the cementite, is another possibility of interaction between the matrix and the inclusion. According to Stroppe et al. /8/, due to the permeability increment the ferrite/cementite interface represents a boundary surface at which free magnetic poles occur and lead to the formation of stray fields. In order to reduce the magneto-static energy, the cementite precipitations which are larger than the thickness of the Bloch-walls, form magnetic secondary structures. In the influencing region of these magnetic secondary structures, there is an interactions between them and the 90°-Bloch walls of the ferrite matrix and the residual stress fields as well as gradient fields of the second kind in the presence of the cementite. Such residual stress fields are due to the different thermal expansion coefficients between the ferrite and the cementite.

Since the cementite phase is ferromagnetic and has a Bloch wall structure of its own, an interaction between the Bloch walls of the ferrite matrix and the Bloch walls of the cementite is possible.

The influence of the cementite on the micromagnetic parameters was evaluated for steels with different microstructures, which include perlitic structure states with globular (Pg) and lamellar cementite formation (P_L), tempered martensitic structure states (M+A) and soft tempered martensitic states (M+W). The structure states differ concerning the size (between 0.28 µm and 3.4 µm and shape of the cementite particles and the degree of distribution.

The hysteresis and the magnetic Barkhausen noise curves for soft iron and massive sintered cementite specimen (low alloyed) indicate a distinctive difference for the two materials. The soft iron has a double-peak structure in the frequency range 500 Hz - 50 kHz in contrast to the cementite, where no such double peak has been observed. The coercive force for the cementite phase is 13 A/cm and for the soft iron is approximately 1 A/cm /9/.

Figure 1 shows the hysteresis curve and the magnetic Barkhausen amplitude M as a function of the magnetic field strength for soft iron and for two perlitic globular structure states with different cementite contents.

With the increasing cementite content, the double peak structure in the magnetic Barkhausen noise curve disappears. The magnetic field range between the two maxima becomes smaller and smaller. The phenomenon of appearance of the multiple-peak structures in the magnetic Barkhausen noise curves is fundamentally due to an additional contribution of the 90°-Bloch walls to the remagnetization process. According to the phase

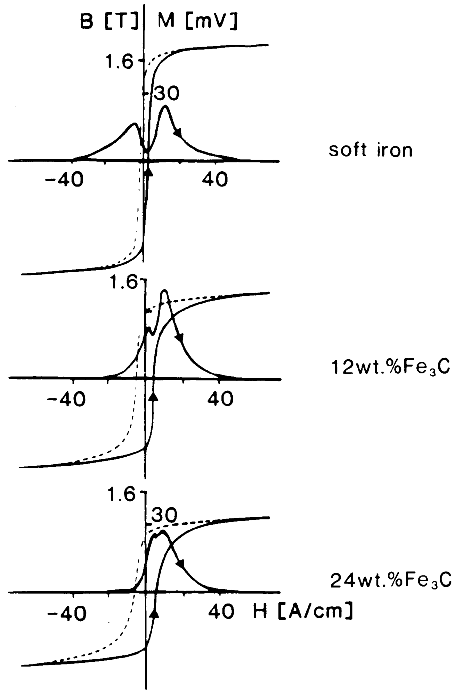

Fig. 1. Magnetic Barkhausen noise-hysteresis curves. Microstructure state: perlitic globular

theory /10/, the main noise activity of the 90°-Bloch walls lies in the knee region of the hysteresis curve, that is in the magnetic field range in which the multiple peaks are measured. These 90°-Bloch walls are magnetostrictively active and are stress-sensitive because of their large residual stress fields extension in contrast to 180°-BW. Due to this fact, they have a strong interaction with other, i.e. structure-dependent residual stress fields such as the residual stress fields of the second kind originating from the cementite precipitates. Due to this interaction, the 90°-Bloch-walls can be restricted or even hindered in their mobility. The main remagnetization thus moves to the 180°-Bloch walls whose main noise activity lies in the region of the coercive force.

With the increasing volume, the residual stress in the cementite phase is becoming smaller: by reasons of balance, the tensile stresses in the ferrite phase has to increase. Hence, the 90°-Bloch walls of the ferrite matrix are increasingly hindered and the density of movable 90°-Bloch walls decreases. This can be seen in the longitudinal magnetostriction curves.

Figure 2 shows such λ_L (H)-curves for two specimens with different cementite contents. With increasing cementite content, the maxima of the longitudinal magnetostriction curve become smaller. This points to the fact that the density of movable 90°-Bloch walls in the remagnetization process decreases. The hysteresis and the Barkhausen noise curves for the four structure states, (P_g), (P_L), M+W and M+A illustrate that the hysteresis curves differ clearly in the coercive force values and the Barkhausen noise curves differ clearly in the magnetic field ranges for the Boch-wall-nucleation. The Barkhausen noise curves themselves also differ clearly in their structure (single, double, triple peaks) in the frequency range 500 Hz - 50 kHz. The measurements show how much the shape, size and the degree of distribution of the cementite influence the macro- and microscopic magnetic parameters /11/.

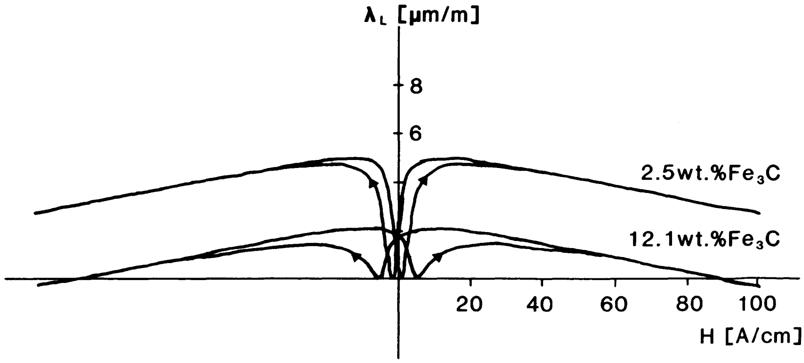

Fig. 2. Longitudinal Magnetostriction

According to the theoretical considerations by Laszlo /12/, differences in the cementite shape (globular or lamellar) and the size cause different residual stress fields σ_{II}. For that, the overlap of the residual stress fields in the presence of single carbide precipitates depends essentially upon the shape and the size of the precipitates. But also the degree of dispersion plays an important role. According to the theoretical considerations, the mutual influence of the overlap of residual stress fields in the environment of single carbide precipitations for the structure states with lamellar cementite (lamellar perlite and white cast iron), for the same degree of dispersion provided, is larger than in structure states with globular cementite. Due to the greater restriction of the stress-sensitive 90°-Bloch-wall-movements, in these structure states, the main remagnetization is concentrated on non-stress-sensitive Bloch walls in the region of the corresponding coercive forces, which leads to single-peak structures in the magnetic Barkhausen noise curves. The restriction of the 90°-Bloch-wall-movement in perlitic steels with lamellar cementite can also be explained by the formation of secondary structures in the ferrite/cementite interface. In the case of the lamellar cementite precipitates, these secondary structures are certainly larger than in the case of the globular precipitations. The greater restriction of the 90°-Bloch-wall-movements in the ferrite matrix of perlitic steels with lamellar cementite also explains that the coercive force values are higher compared to perlitic steels with globular cementite. The magnetic behavior of the white cast iron specimens differs fundamentally from that of the steel specimens. Nearly all the magnetic parameters show a different characteristic behavior below and above 2% carbon i.e. steels and cast irons respectively /13/.

The Barkhausen noise amplitude at H_C, $M(H_C)$, increases with the cementite content, the slope above 2% carbon being essentially greater than the one below 2% carbon. The greater increase in the Barkhausen noise amplitude points to an active noise contribution originating from the cementite phase. H_C is found to increase with cementite for steels whereas it decreases with cementite for cast irons. The different bahavior of the magnetic parameters above and below 2% carbon can be derived from the fact that there are different phase contents in the white cast iron specimens than in the ferritic/perlitic steel grades. In the ferritic/perlitic steel grades, the cementite lies in the perlite; in the white cast iron a part of the cementite is bound within the perlite and the rest of it in the ledeburite.

When the two phase material is heated above the Curie temperature of the cementite phase, cementite-specific changes in the magnetic parameters can be seen. In the ferromagnetic materials, increasing or lowering of the temperature leads to a change in the magnetic energy conditions in the region of a Bloch wall. This can be attributed to the following three reasons: (1) the magnetic material constants I_S (saturation magnetization), K_1 (crystal anisotropy constant) and λ_L (magnetostriction constant) depend upon the temperature; (2) the thermal expansion in a polycrystal leads to a change in the form of anisotropy and to the distribution of microstresses; and (3) the local distribution of the magnetically active defects changes due to the diffusion of lattice atoms /14/. The last reason has been neglected in the boundary conditions which are chosen here.

The ratio $M_{Max}(T)/M_{Max}(RT)$ (ratio between the Barkhausen noise maximum at the corresponding measuring temperature and the Barkhausen noise maximum at room temperature) decreases in the temperature interval (40°C $\leq T \leq$ 240°C) by 10% for ferrite and by 100% for cementite /9/. With increasing temperature, the coupling of adjoining spins, that is the spontaneous magnetization, decreases continuously. At the "critical" or Curie temperature, the thermal motion is so intensive that any order by the Weiss' field is prevented, and therefore the spontaneous magnetization disappears entirely in cementite. The transition from the ferromagnetic state to the paramagnetic state leads to the decomposition of the Bloch-wall range structure, and hence, to the decrease in the Barkhausen noise intensity.

In the magnetic Barkhausen noise, the non-alloyed white cast iron specimens show a temperature dependence which is nearly the same as the one for the compact cementite specimen /11/. With increasing temperature, the maximum in the Barkhausen noise amplitude decreases with increasing cementite content in the specimen. This behavior indicates that cementite contributes actively as a ferromagnetic second phase to the Barkhausen noise. The rest of the noise activity above the Curie temperature is only due to the contribution of the ferrite. The ferromagnetic regions above the Curie temperature of cementite in the white cast iron can be compared with ferromagnetic sponges /14/, because above the Curie temperature all the portions of the cementite phase such as eutectic cementite, eutectoid cementite and secondary cementite have a paramagnetic state ($\mu = 1$) and the rest of the ferritic phase structure consists only of isolated island regions. In the boundary surfaces of the ferrite matrix dipole configurations are developed and consequently internal stray fields are formed. These stray fields partly determine the remagnetization behavior and explain the coercive force values, which increase with the temperature.

The microstructure states in steels are characterized by typical $M_{Max}(T)$-curves. Two different classes can be distinguished. One class for the structure states with lamellar cementite, and the other for the globular cementite. The structure states with lamellar cementite are characterized by a continuously decreasing $M_{Max}(T)$-curve. All the curves of the structure states with globular cementite have a maximum in the temperature range between the room temperature and the corresponding Curie temperature of cementite. The soft tempered martensitic structure states are additionally characterized by a minimum in the region of the Curie temperature (Fig. 3). The position of this minimum depends upon the alloy content of the corresponding cementite phase /15/.

Because of their high affinity towards carbon, alloying elements such as CR or Mn are preferably dissolved in the cementite phase.

The solubility of Cr or Mn in the carbide phase depends decisively upon the speed and the temperature at which the structure change occurs, which in turn depends upon the corresponding structure state. A comparison with Mossbauer-spectroscopic data proves that the temperatures of the $M_{Max}(T)$-minima correlate with the Curie temperatures of the corresponding cementite phase /9/.

Considering the residual stress models of Haigh and Laszlo /12/, the change in residual volume stresses of the second kind, $\sigma_{V,\ II}$ with the temperature in the two phase ferrite-cementite material can be theoretically estimated for globular precipitates. According to these estimations, the average residual volume stresses decrease in the ferrite matrix proceeding from room temperature values. Supposing that only the ferrite phase reacts magnetically to stress changes, the residual stress course can also be seen in the $M_{Max}(T)$-curves in the temperature range $T \leq T_{Curie}$. This assumption is certainly justified, when the stress dependence of M_{Max} is considered for soft iron and white cast iron. M_{Max} in soft iron is very sensitive to stress alterations, whereas M_{Max} in white cast iron with 70 wt% Fe_3C shows only a very small stress dependence.

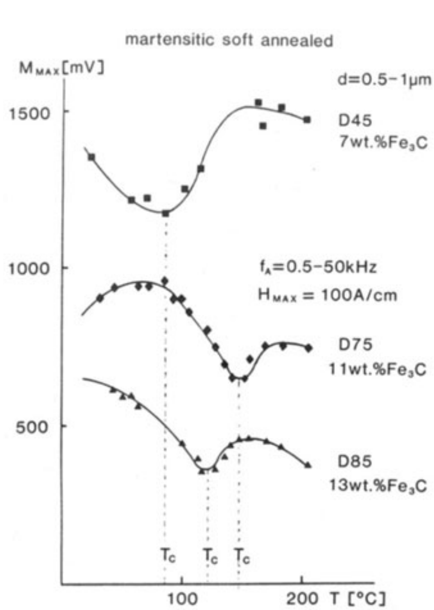

Fig. 3. Maximum of Barkhausen noise amplitude as a function of temperature

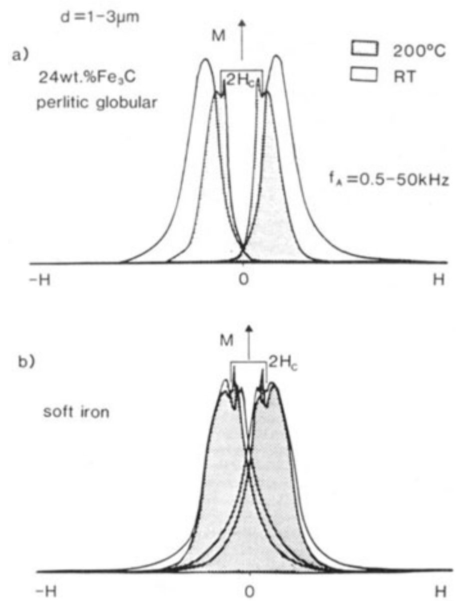

Fig. 4. Magnetic Barkhausen noise for perlitic state (a) and soft iron (b) at 200°C and room temperature

A comparison of the M(H)-curves at room temperature and $T = T_{Curie}$ shows clearly that at higher magnetic fields, cementite contributes actively to the noise, in the steel specimens also. As figure 4 shows for the case of a P_g-structure state, at $T > T_{Curie}$ and the M(H)-curve is strongly constricted and the double-peak structure typical for soft iron is observed; the magnetic field strengths of the remaining Barkhausen noise maxima correlate well with the magnetic field strengths of soft iron.

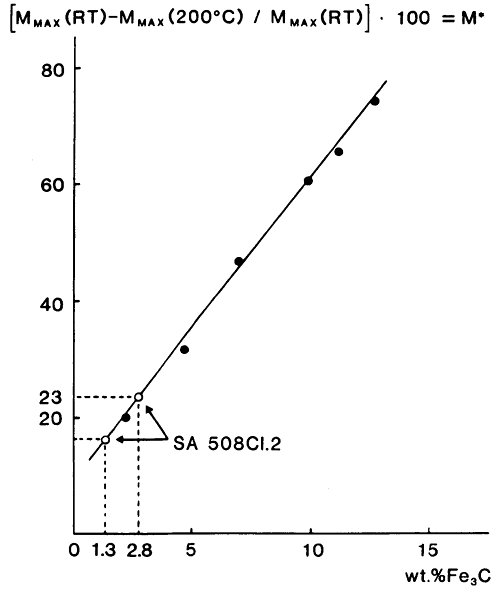

Fig. 5. Normalized amplitude M* of magnetic Barkhausen noise as a function of cementite content

In the globular perlite structure states, the ratio $\Delta M^{25\%}(RT)/\Delta M^{25\%}(200°C)$, that is the ratio of the full width of the curve at 25% of the maximum amplitude at room temperature to the full width of the curve at 25% of the maximum amplitude at 200°C, can be used as a cementite-sensitive parameter /9/.

It has been found that the parameter M* given by $M_{Max}(RT)-M_{Max}(200°C/M_{Max}(RT)$ can be used to estimate the cementite content in tempered martensite structure. A calibration curve has been generated (Fig. 5), which gives the variations in M* with cementite content. This calibration curve has been applied to estimate the cementite content in two SA 508 Cl.2 steel specimens having different cementite content. A good agreement between the cementite content estimated from the calibration curve (1.3 and 2.8%) to that obtained by transmission electron microscopy (1.2 and 2.9%) has been found in these specimens.

CONCLUSION

The micromagnetic measurements at room temperature have shown that the multiple-peak-structure in the magnetic Barkhausen noise strongly depends on the cementite morphology and its content.

By heating the two-phase-material above the Curie temperature of the corresponding cementite phase, cementite-specific changes in the Barkhausen noise curve such as the decrease in the Barkhausen noise maxima, the broadening of the Barkhausen noise curve ΔM, changes in the coercive force could be observed. On the one hand, these changes in the magnetic parameters can be understood from the decomposition of the average residual volume stresses of the second kind in the ferrite matrix, on the other hand, they can also be understood from the lack of active noise contribution from the cementite phase. The parameters derived from the magnetic Barkhausen noise can be used to determine the cementite content, when the microstructure state is known. When the steel grade is known, the microstructure state can be detected from the pattern of the Barkhausen noise curve by means of simple pattern recognition. When the steel grade is unknown, the coercive force derived from the Barkhausen noise curve must be used as an additional parameter for characterization of the microstructure state.

ACKNOWLEDGEMENT

This work has been carried out under the research support of the Deutsche Forschungsgemeinschaft.

REFERENCES

1. Herz, K., Kuppler, D., Deimel, P., Theiner, W., Bestimmung der Mikrogefuge von Druckehalterstahlen mit magnetisch induzierten MeBgroBen. Proj. - Nr. 150, 334, 3. Zwischenbericht der MPA-Stuttgart 1981.
2. Brown, L. M., Ham, R. K., Dislocation - Particle Interactions in, Strengthening Methods in Crystals. Hrsg., Kelly, A., Nicholson, R. B., Appl. Sci. Publ. LTD London 1971.
3. Ron, M., Iron-carbon and iron-nitrogen systems in, Applications of Mossbauer spectroscopy edited by Cohen, (1980), S. 329.
4. Faber, F., Hartmann, U., Quantitative rontgenographische Phasenanalyse am System, -Fe/ -Fe/Fe_3C Harterei-Techn. Mitt. 28 (1973).
5. Kretschmer, M. Schmolke, G., Koch, K. H., Phasenanalyse mikrolegierter Stahle, Hoesch Huttenwerke AG, ESTEL-Berichte aus Forschung and Entwicklung unserer Werke H. 2 (1975) 53.
6. Puskeppel, A., Harsdorff, M., Anwendung der quantitativen Bildanalyse bei der Auswertung elektronenmikroskopischer Hellfeld- und Dunkelfeldaufnahmen in, IMANCO, Fortschritte der quantitativen Bildanalyse, (1975), S. 20.
7. Dijkstra, L. J., Wert, C., Phys. Rev. 79, (1950), S. 979.
8. Kruger, F. D., Veit, P., Stoppe, H., Spektrale Auswertung von Barkhausenrauschsignalen, Wiss. Techn. Univ., "Otto von Guericke" Magdeburg 31 (1987) Heft 8, S. 1-1.
9. Altpeter, I., Kern, R., Holler, P., Einflub ferromagnetischer 2ter Phasen (Karbide) auf die Eigenspannungsmessung und Gefugecharakterisierung bei Nutzung magnetischer und magnetoelastischer MeBgroBen IzfP-Bericht Nr. 880324-TW.
10. Seeger, A., Moderne Probleme der Metallphysik, Berlin - Heidelberg - New York, Springer Verlag (1966).
11. Altpeter, I., Kern, R., Holler, P., Characterization of cementite in steel and white cast iron by micromagnetic nondestructive methods, Oktober (1988), 3rd International Symposium on Nondestructive Characterization of Materials, 3, 6, October 1988, Saarbrucken.
12. Laszlo, F., JISI 147 (1943) S. 173.
13. Theiner, W. A., Altpeter, I., Kern, R., Determination of sub-surface microstructure states by micromagnetic ndt, presented at the 2nd International Symposium on Nondestructive Characterization of Materials, Montreal, Canada, July 1986.
14. Kneller, E., Ferromagnetismus, Berlin, Springer Verlag (1962).
15. Jellinghaus, W., Weitere Messungen zu den physikalischen Eigenschaften des Zementits. Archiv fur das Eisenhuttenwesen 38 (1967) S. 488.