ARCHIVES of FOUNDRY ENGINEERING

ISSN (1897-3310)
Volume 10
Special Issue 3/2010

189 – 194

36/3

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

Determination of bearing steel heat treatment with the use of the acoustic emission method

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Received 30.04.2010; accepted in revised form 30.05.2010

Abstract

A study on the control of an extremely important stage of the martensitic-bainitic austempering and obtaining the M-B structure in the 100CrMnSi6-4 steel with the use of the acoustic emission (AE) has been undertaken. In order to enrich retained austenite with carbon, steels are austempered at appropriately low temperatures. A martensitic transformation, resulting from diffusionless and displacive transformation is associated with significant AE signs. The strain energy produced during growth due to the shape change is reduced by plastic deformation. Predominant source of (AE) is the movement of dislocations in order to relieve internal stresses.The heat treatment was performed in a modern, purpose-constructed device which simultaneously records acoustic emission effects. The signals were recorded with the use of an AE analyzer 20–800 kHz, and they were received by means of a broadband piezoelectric transducer with the use of a specialist card with a sampling frequency of 1200 kHz. The results regarding a correlation of austempering temperature and the maximum number of AE events and dilatometric results have been presented. This parameter can be used for precise Ms temperature estimation. Basing on microstructural investigations, it has been found that previously formed martensite with midrib morphology also accelerates the bainitic transformation.

Keywords: heat treatment, bainite, acoustic emission, midrib, dilatometric analysis

1. Introduction

In recent years, highly durable steels of great industrial importance, containing significant fractions of retained austenite, have been developed. Steels with great amounts of carbon-enriched retained austenite are usually produced with the use of low-temperature transformations. This results in a microstructure containing bainitic plates with interlath retained austenite film. Recently, considering heat treatment, a production of steels with retained austenite, called ‘quenching and partitioning’, related to formation of duplex structures, has been widely discussed. Austempering, which induces lower bainite formation in the material, replaces two classical heat treatment processes. It is a cost-effective method which maintains strength and hardness of steels and may also improve their crack resistance. Austempering has been widely used in industry. Austempered elements show a weaker tendency towards changes in size than elements after customary quenching. As a result, this kind of treatment has been applied, among others, in the rolling bearing industry [1]. The SAE 52100 steel is usually used in a quenched and tempered form [2, 3]. Compared to a steel of martensitic structure, the bainitic structure in this steel provides a

higher wear resistance and hydrogen embrittlement [4, 5]. A combination of high strength and impact resistance requires optimal bainite and martensite fractions.

Despite many achievements in the technology of bearing steels, there is still a potential for improving their mechanical properties. A promising heat treatment is a combination of low-temperature isothermal martensitic transformation and subsequent bainitic transformation, resulting in a duplex, martensitic-bainitic (M-B) structure. After martensite formation, a typical bainitic transformation occurs. As a result of duplex (M-B) structure formation, increased strength and crack resistance are observed [6]. A suggested treatment method is based on phenomena occurring in high-carbon steels within the range of so-called 'Swing Back' [7-10]. Within this range of phase transition, it was shown that thin-plate isothermal martensite (called 'midrib'), forming during incubation, preceded and accelerated lower bainite nucleation [11-13].

Considering low-temperature treatment of bearing steel, poorly identifiable phase transitions occur due to overlapping phenomena and their volume fractions. Acoustic emission may be a promising solution here as there is a potential for obtaining a correlation of recorded AE signals, the microstructure and mechanical properties of the material that are formed during austempering. A result of local energy release in the process of twinning or dislocation movement with significant accelerations and delays is propagation of elastic waves inside the material or on its surface. A precision of measurements may be improved by proper recording and analysis of acoustic emission signals with the use of computer technology and advanced algorithms of signal analysis.

The aim of the paper was a presentation of a heat treatment conception utilizing carbon partitioning. A control of phenomena occurring during the transformation will be possible due to determination of acoustic parameters and frequency characteristics of elastic waves. In the suggested heat treatment, cooling to selected temperatures near MS was applied in order to achieve carbon partitioning with a purpose of enrichment austenite with carbon and, therefore, control the kinetics of midrib formation.

2. Material and work methodology

The experiments were conducted with the use of 100CrMnSi6-4 steel (EN ISO 683-17:1999) that contained 0,95 wt. pct carbon, 1,10 wt. pct manganese; 1,47 wt. pct chromium; 0,57 wt. pct silicon. The material, used for production of rings and rolling parts of bearings, shows high purity. The investigated steel was delivered as rolled bars in a softened state, 46 mm in diameter, from the same casting batch. In this research, the aus***tenitizing*** temperature of 950°C for 30 min was used. Then the specimens underwent isothermal ***holding*** at 100-180 °C within the time range 0–5000 s, followed by quenching to the ambient temperature. For this purpose, a special set-up was designed that allowed for both austempering and recording acoustic effects during the process. Signals were recorded by means of the AE SIGNAL ANALYZER 10/20 kHz - 800 kHz. The signals were received by means of a broadband piezoelectric AE transducer WD type (20 kHz - 900 kHz) connected to a pair of differential inputs of a low noise preamplifier. The AE signals were saved in a PC memory by means of the ADLINK 9812 card at 1200 kHz . The maximum recording time was 4 minutes. As a data format, a bipolar 5V option was applied. The differential input impedance is 1 MOhm and a total differential amplification of the device: 72 dB (x4000) in the 0.01–0.8 MHz band. The noise root-mean-square value at the preamplifier input was approximately 12 μV. The best setting for low power AE signals was 500 divisions in the AE measuring system, which corresponded to the total amplification of 66 dB (x2000). The analysis of AE signals was performed with the use of special software designed for recording, spectrogram plotting, and the root-mean-square value determining and signal analysing. The software has a graphics window designed for displaying maximum numerical signal values in consecutive time frames every 15 milliseconds. For spectrogram plotting, the STFT (Short Time Fourier Transform) algorithm with the Hamming window was used. Each frame is obtained from 17640 signal samples.

The MS temperature of the steel was measured with the use of an Adamel Lhomargy LK 02 dilatometer. Cylindrical specimens 12 mm in length and 2 mm in diameter were heated to 950 °C and then rapidly cooled. The formation of martensite during cooling was detected for six specimens. For determination of MS temperatures, the so-called theoretical martensite-start temperature, expressions that can be described by simple linear equations provided by Steven and Haynes (Ms-S) [14], Andrews-linear (Ms-A) [15], Grange and Stewart (Ms-G) [16], Bohemen and Sietsma (Ms-B) [17] were applied.

The metallographic study of the transformed specimens was carried out to identify the transformation products formed at various temperatures. A scanning electron microscope (SEM) Hitachi S-2600N was used in order to demonstrate the presence of bainite. For all photographs with the use of SEM, the magnification was x3000. The microsection etching was performed by means of a Nital reagent.

3. Description of resutls

Sample spectrograms of AE signals, obtained during austempering, are presented in Fig. 1. The analysis of the number of AE events was performed with the use of specially designed software. This software analyzes AE events that exceed the background noise level. In the measurement procedure, an AE event was recorded when the unit level exceeded the discrimination level of established 5000 units. Next, a sum of event numbers corresponding to each austempering temperature 30–190°C was calculated. Mean values of event numbers occurring during austempering for various transformation conditions are presented in Fig. 2. At 50–100°C, the nature of changes in acoustic emission signal intensity is comparable, proving their athermal character.

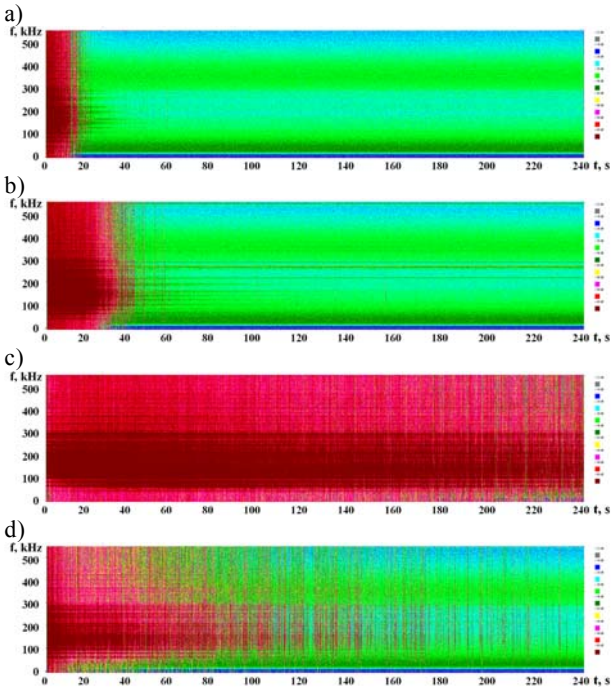


Fig. 1. Spectrograms of acoustic emission signals for the ŁH15SG steel after austempering at: a) 30 ˚C, b) 100 ˚C, c) 160 ˚C, d) 190 °C.The x-axis indicates the signal recording time (240 s), while the y-axis presents the recorded signal frequency in the linear scale of 0–600 kHz. The colours encode the chemical element value from the power spectral density of the recorded signal in a 9-grade scale. The lowest signal power is marked dark-blue, while the highest is dark-red

When the temperature is increased to 130–190°C, the isothermal course of martensitic transformation is activated, which is seen in the spectrogram (Fig. 1) as an explicit acoustic beam. The dominant spectral range is still within 100-300 kHz. At 130˚C and 160˚C, the numbers of events are comparable, but at 160˚C, the event emission rate is lower due to the longer emission time.

At the M_S, determined as the dilatometric curve deviation from the straight line which is tangent to this curve, a maximum number of acoustic events was observed, Fig. 2. Due to a large number of AE events, it is at 157 °C when a macroscopic effect of elongation changes resulting from midrib formation, occurs. Midribs form significantly earlier and at 190°C, despite their large amounts, elongation changes are not observed yet. Lack of full correlation between AE and dilatometric results is also affected by a time factor. Emission effects were investigated under isothermal conditions. At 160°C, the beginning of signal emission is delayed in comparison with other temperatures despite the maximum summary number of the signals at this temperature. During continuous cooling, only the beginning of the dilatometric curve deviation at 160 °C is observed.

Full lines in Fig. 3 denote a relative fraction related to a different maximum event number at each temperature. In the range of 20–100 °C, acoustic emission is the fastest and completes in a 10 to 20 seconds. A temperature rise results in a longer emission time. The longest AE time occurs at 160 °C and at this temperature, there is also the largest number of acoustic emission signals observed, which is seen in Fig. 2. A number of maximum AE events comparable to that observed at 160 °C also occurs at 130 °C. However, at 130 °C, 90% of event emission is completed in approximately 100 s. A comparison of the TTAE curves and dilatometric results suggests that the dilatometric method determines only a relatively advanced stage of the transformation associated with midrib formation. Also, mathematical relations described by Bohemen et al. are worth consideration as the calculations with the use of their equations are closest to the maximum number of events (Fig. 2) and to the area of the maximum stability of austenite in Fig. 5. A temperature rise above the M_S-B results in both reduction in the AE event number and the duration of events.

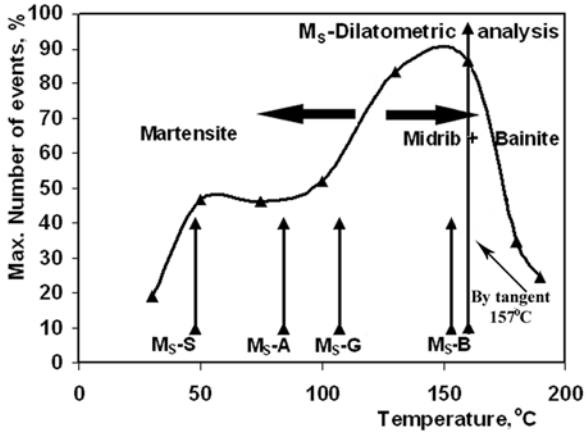


Fig. 2. A distribution of the sum of acoustic event numbers during austempering. In the y-axis, 100% corresponds to $5x10^7$ events. In the figure, the M_S calculation results with the use of Steven's (Ms-S), Andrews' (Ms-A), Grange's (Ms-G), Bohemen's (Ms-B) [14-17] equations are also presented

The light-grey areas in Fig. 4a represent retained austenite γ, while martensite is shown in the others. The above results show that within this range, a characteristic change of the transformation occurs. It is seen that γ retains in blocks. In Figs. 4 and 5, a microstructures obtained after austempering at the temperature near M_S are presented. According to the calculations with the use of equations by Bohemen and Sietsma [17], the M_S is 153°C, which corresponds to the observed changes in signal emission. This means that the maximum number of events in the shortest emission time occurs at a temperature slightly above the M_S. At the transformation temperature of 160 °C, lower bainite plates with a midrib, marked as LBM in Figs. 4 b) and 5, are observed. The specimen exhibits a microstructure consisting of a small amount of bainite (black 'needles'), martensite and retained austenite in blocks.Carbon diffusion from the areas of bainite to austenite leads to stabilization of austenite at the boundaries of plates marked as P. The type of retained austenite (γ_R), located

between bainitic ferrite plates, is very fine and stable due to the enrichment with carbon. In the optical metallographic pictures, due to the fine structure, it is difficult to distinguish the film-type morphology of the retained austenite.

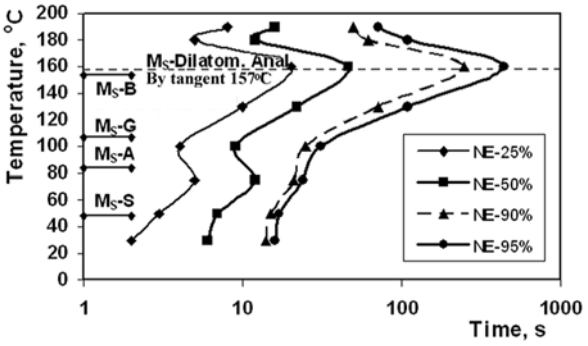
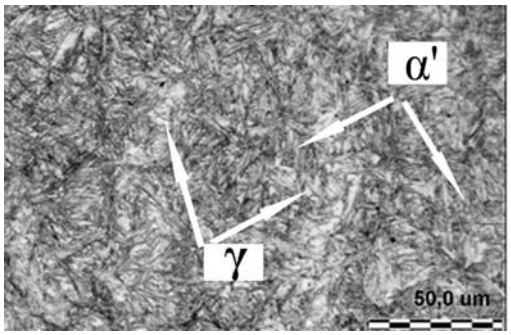


Fig. 3. The time-temperature-acoustic emission (TTEA) curve during austempering together with the experimental calculation results of the M_S temperature

a)



b)

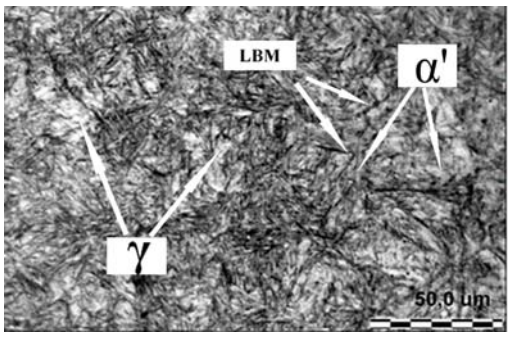
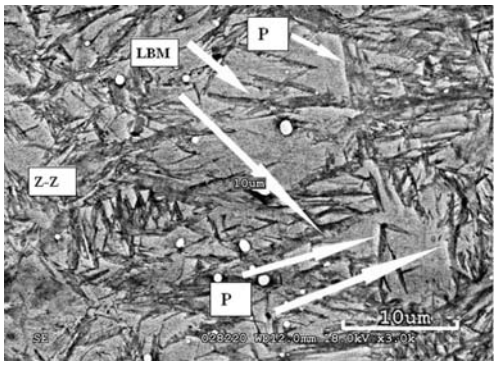


Fig. 4. Optical microscopic pictures of the specimens after isothermal transformation with holding time of 1450 s at different temperatures: a) 100ºC, b) 160ºC, etching with Nital reagent, holding time: 1450 s. The symbols α′, γ, LBM stand for martensite, retained austenite and bainite, respectively

The carbon enrichment in austenite at the temperatures above M_S lead to the formation of midrib which accelerate of bainite transformation. Microstructural observations of bainite confirmed the occurrence of a number of plates of butterfly morphology containing midribs. The formation of the butterfly shape is preceded by the formation of two intersected midribs that are the thin-plate martensite.. In Fig. 5a), martensite laths are symmetrically arranged and form characteristic zigzagged arrays (Z-Z) with a midrib. At a higher transformation temperature, i.e. 190°C, a number of acoustic emission events markedly declines, which is confirmed by a reduced number of LBM plates in the microstructure photographs. Fig. 5b). This is a result of a higher number of transitional carbides in bainite plates which consume excessive carbon. Thus, lack of austenite enrichment with carbon, above the M_S, does not promote midrib formation and emission of acoustic signals.

a)



b)

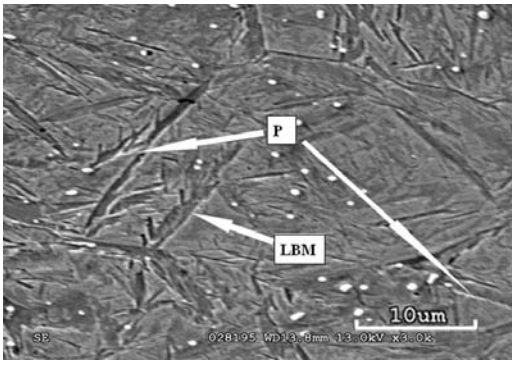


Fig. 5. Scanning electron micrographs of specimens isothermally treated with the holding time of 1450 s at temperatures: a) 160°C, b) 190°C; etched with a Nital reagent. The dark plates are lower bainite with midrib LBM and light places around the plates are untransformed retained austenite P. In the micrograph 5a), plate martensite with a characteristic morphology Z-Z is seen

4. Discussion

For years, most researchers that studied martensite focused on a transformation process during continuous cooling. Decomposition of austenite into martensite was thought to stop when a stable temperature was maintained. Martensite was believed to form at a certain critical temperature and continue its growth only during further cooling. A time factor was thought to

have a little effect on the kinetics of martensitic transformation. The studies suggest that each type of transformation products has a separate C-curve [18]. In late 1940s and early 1950s, Kurdjumow and Maksomova [19] discovered isothermal martensitic transformation. In some steels, both martensite types (twinned plate and lath) may form simultaneously, while in others they have separate C-curves. In 1980s, Okamoto [20] confirmed isothermal twinned plate martensite formation at 100 ^{o}C in the Fe-1.8%C steel. Later, Jicheng, Zhao and Zhanpeng Jin reviewed the TTT diagrams together with the data of phase transformations below the M_S [21].

Contrary to steel tempering, transitional carbide precipitation in the Q&P process is essential. In the Q&P process, a high supersaturation of martensite with carbon may force transitional carbide formation. At the same temperature, this process occurs more frequently in martensite than in growing bainite because bainitic ferrite grows at smaller carbon content than initially in austenite. Thus, the inhibition of carbide formation will be a critical factor affecting the microstructure in the Q&P process.

In quenched steels, carbon partitioning between martensite and retained austenite is usually neglected. Small transitional carbides in martensite are usually considered harmless so the emphasis on understanding the transitional processes related to their replacement with cementite was greater than on the initiation of transitional carbide precipitation. During tempering, the excessive carbon in martensite is normally eliminated via a different mechanism, i.e. carbide precipitation. Carbide formation limits carbon diffusion because these carbon atoms are no longer available for enrichment of austenite. The metastable equilibrium between ferrite and austenite will not be possible if carbides, which provide a stable equilibrium between ferrite and iron carbides, are formed. In the paraequilibrium, chemical compositions of ferrite and austenite are unequivocally determined and they meet the condition of one common tangent. According to this condition, chemical potentials of both carbon and iron are equal in both phases ((μ_C^{α}= μ_C^{γ} and μ_{Fe}^{α}= μ_{Fe}^{γ}). In case of stationary or constrained interface, the metastable equilibrium α/γ is called 'a constrained paraequilibrium' or CPE. This condition may be met by an infinite set of phase compositions of austenite and both tangents to the curves of free energy of ferrite and austenite have to intersect the carbon axis at one point [22].

Thus, the carbide precipitation processes, which may occur during low-temperature heat treatment, must be considered in the control of heat treatment processes and the analysis of acoustic emission phenomena. As the chemical potential of carbon is far higher in martensite during cooling than in austenite, carbon nucleation is more probable in ferrite than in austenite [23]. A privileged location of carbide formation is also the interface α/γ. Cementite formation may be eliminated or delayed with the use of silicon additions which are present in the investigated steel [24].Silicon inhibits cementite formation and delays the transition from early tempering stages, where ε or η carbides are present, to its second stage with the F_3C carbide.

The results of acoustic emission investigations suggest that at 160 °C there is a specific combination of phenomena. On one hand, the rate of acoustic emission dN/dt is relatively low, while on the other hand, the summary number of sounds is the highest. This results in the longest duration of acoustic emission. It should be assumed that this process is controlled by several factors. One of them is carbon diffusion in austenite and its enrichment, which promotes the M_S decrease and midrib formation. The longest periods of midrib formation at this temperature result from processes of austenite enrichment with carbon and a limited process of transitional carbide (which binds excessive carbon) precipitation. Transitional carbides formed at that stage additionally provide a higher carbon fraction in remaining austenite. A factor that promotes the longest and intensified acoustic activity at 160 °C is the interaction of clustered point defects (vacancies) with partial dislocations. The attachment of clustered vacancies to partial dislocations leads to midrib nucleation delay [25].

5. Conclusions

1. A methodology of an optimal austempering temperature selection by means of measurements of ultrasound signals that form during ***isothermal heat treatment*** has been developed.
2. At the M_S, determined as the dilatometric curve deviation from the straight line which is tangent to this curve, a maximum number of acoustic events was observed.
3. Basing on the measurements of a sum of acoustic emission events, the M_S for steels can be precisely determined, assuming that it corresponds to below the mark their maximum number. Regardless of the temperature changes, the dominant spectral signal range is constant and placed within 100-300 kHz.
4. Such a precise estimation of the martensite formation start is not available with the use of classical dilatometric methods that are based on the macroscopic effects of geometric alterations. The suggested method utilizes the micrometric changes.
5. The most intense phase of isothermal martensitic transformation occurs at 130-160°C. Above 130°C, lower bainite plates with a midrib (marked as LBM) and the areas around the plates suggesting their enrichment with carbon have been observed.

Acknowledgements

This work was carried out with financial support from the Polish Ministry of Science and Higher Education under project number R15 010 02, the Statury Studies at K. Wielki University in Bydgoszcz and project number POIG.01.01.02-14-100/09-00 within Operational Programme Innovative Economy 2007-2013, co-financed by European Regional Development Fund. The author wishes to acknowledge Prof. J. Jeleńkowski and K. Rozniatowski, MSc, PhD, Department of Material Science at Warsaw University of Technology, for valuable discussions on the idea of applying the acoustic emission method for the analysis of transformations during heat treatment processes, Z. Ranachowski MSc, PhD from Institute of Fundamental Technological Research, Polish Academy of Sciences, for fruitful cooperation regarding the methodology of signal recording,

Mikołaj Popławski MSc, PhD, Poznan University of Technology, Faculty of Mechanical Engineering and Management for his assistance with dilatometry measurements.

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