Phase Transformations in Alloy Steel Containing Boron Using Dilatometry and Acoustic Emission

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The tests were carried out for the selected 27MnCrB5-2 alloy steel with the addition of boron. The paper presents the kinetics of the ferritic transformation occurring under continuous cooling conditions. The temperatures of the beginning and end of the transformation of supercooled austenite were determined using a dilatometer. In addition, a waveguide and an acoustic sensor were installed in the device to receive acoustic emission signals. The results in the form of dilatometric curves were additionally confirmed by the obtained acoustic emission signals. Based on the obtained microstructural results, it can be concluded that in the hardening process of 27MnCrB5-2 steel at the applied temperatures, complex transformations of austenite into ferrite and bainite occur. The use of acoustic methods in the field of phase transformation issues creates the possibility of their application in industry.

Keywords: Steel 27MnCrB5; dilatometric tests; acoustic emission (AE); microstructure.

INTRODUCTION

Despite the enormous development of science, there is still a need to expand knowledge about materials, an important group of which are metals. Heat treatment of metals and their alloys is used to improve their mechanical and physicochemical properties to meet the needs and operational requirements^{1, 2}. Properly selected and carried out heat treatment conditions leading to the desired transformation have a fundamental impact on the properties of finished alloy steel materials^{$\overline{3}-5$}. The consequences of heat treatments are changes in the microstructure, which in turn leads to changes in the properties of materials. 27MnCrB5-2 alloy steel with the addition of boron is a steel widely used in many industries. Due to its mechanical properties such as high strength, hardness and good hardenability, it is used for demanding machine and device parts. Heat-treated steel can have a mixed martensitic-bainitic structure or in the form of lamellar ferrite and Widmanstätten ferrite in a bainite matrix.

In recent years, the development of measurement techniques has led to the creation of various research methods. This has enabled precise chemical and physical analyses of materials with different properties. Dilatometric tests have gained particular attention, and due to their versatility and nature, they are used in many fields of science and industry. Dilatometry is used in the aviation, automotive, construction, chemical, and ceramic industries. A dilatometer allows for the examination of dimensional changes in various materials, including metals, polymers, ceramics, glasses, and other construction materials⁶⁻⁹. In the study of metals and their alloys, dilatometric tests enable the determination of the beginning and end of phase transformations that occur as a result of the assumed thermal treatments¹⁰. The aim of these tests is to determine the coefficient of linear thermal expansion^{11, 12}. Dilatometric tests are an important tool in the study and design of materials and processes.

The acoustic emission method is a widely used technique for studying the properties and structure of matter, enabling the understanding of processes occurring on both macro and microscopic scale¹³. The acoustic emission method is also a technique for monitoring various engineering objects, such as structures and tanks. It is a non-destructive method and does not affect the condition of the tested material. The sources of AE signals during monitoring of objects are micro and macro cracks, corrosion processes, gas leakage in leaky pressure tanks. AE is a phenomenon that consists in the formation of elastic waves on the surface or inside the tested material as a result of local release of internal energy. This method can also be used in the process of monitoring phase transformations occurring in steels during their heat treatment^{14, 15}. The sources generating AE signals in steels are considered to be the movement of dislocations as a result of plastic deformation, the formation of twins, and displacement-type phase transformations^{16, 17}. Research using AE is conducted in many laboratories to monitor and understand various physical phenomena^{18, 19}. Other interesting research methods can also be applied to study phase transformations. In particular, thermodynamic simulation²⁰ should be mentioned, which aims to achieve holistic management of the phase assemblages of alkali-activated materials (AAMs). Another method for analyzing phase transformations could be molecular dynamics simulation²¹ which is based on interatomic interaction potentials.

Studies of phase transformations in steels, using dilatometry and acoustic emission, have a significant impact on better understanding and optimization of heat treatment. These analyses are key to providing the high strength and durability materials that are desired today. These studies are not only an analytical tool, but also an element of materials engineering, enabling development and innovation in the design of modern construction materials.

The aim of the authors' work was to investigate phase transformations in boron steel using a dilatometer with an integrated sensor for measuring acoustic emission. Additionally, an analysis was performed on the dilatometric curves and the resulting spectrograms of the acoustic emission signal energy spectra.

RESEARCH METHODOLOGY

Phase transformations occurring during the decomposition of supercooled austenite in 27MnCrB5-2 steel with boron addition were investigated. The chemical composition of the steel is presented in Table 1.

Table 1. Chemical composition of alloy steel 27MnCrB5-2,
element content in wt%

С	Mn	Si	Cr	Cu	В	Р	S
0.27	1.28	0.25	0.51	0.20	0.003	0.014	0.01

In order to determine the dilatometric curves, a modified quenching dilatometer was used. The device enabled the measurement of the absolute change in sample length, specific variables, and the coefficient of thermal expansion as a function of temperature and time. The change in the length of the sample was recorded using a measuring system and its detection was carried out by means of a linear displacement sensor LVDT (Linear Variable Differential Transformer). The design of the device ensures the highest accuracy, repeatability, and stability. The dilatometer comprises a measurement and control unit, along with a computer system equipped with precise software for recording and processing results in the form of dilatometric curves. The sample was heated by a furnace, which includes thermal shields, a fan, power and control connections, and a heating element. A waveguide was installed in the measurement system where the test sample was placed, such that one end of the waveguide contacts the sample. An ultrasonic sensor for recording acoustic emission signals generated during the heat treatment process was mounted on the other end of the waveguide, located outside the device. During the hardening process, the waveguide does not exceed 50 °C, which is within the safe operating range of the sensor. Additionally, the waveguide is made of 4H13 steel, which has been tested to exclude phase transformations within the temperature range in which the waveguide is operated. The acoustic emission signal was recorded using a differential, wideband WD sensor (20-900 kHz). The modified dilatometer that enables the measurement of acoustic emission signals is shown in Figure 1.



Figure 1. Diagram of the Linseis L75HX 1000 dilatometer with the waveguide and sensor for receiving AE signals marked

For the dilatometric studies, samples of 27MnCrB5-2 steel were prepared in the shape of a cylinder with a diameter of 10 mm and a length of 20 mm. The test samples were austenitized at a temperature of 940 $^{\circ}$ C and held at this temperature for 900 seconds. They were then cooled in air at a rate of 50 (K/min) under an argon atmosphere. The austenitizing parameters were designed

to allow for the dissolution of a significant amount of carbides and to lower the Ms temperature. The heat treatment parameters were determined based on the TTT (time-temperature-transformation) curve for the studied steel grade. These conditions facilitated the increase of retained austenite during the hardening of the steel and reduced material distortions. During the recording of the dilatometric curves, acoustic emission signals from the occurring phase transformations during the hardening of the alloy steel were registered. The signal was recorded using the original measurement setup. The schematic of the measurement device is shown in Fig. 2.



Figure 2. Schematic of the setup for recording AE signals

The AE signal from the transformations was recorded using a differential, wideband acoustic sensor. The sensor was connected to a waveguide integrated with the dilatometer and to an analyzer used for receiving and recording the signal. An analyzer was used for recording the AE signal. The instrument was designed to work with the wideband AE sensor from Physical Acoustics. The analyzer allows for the cyclic recording of approximately 100 ms from each successive second of the signal generated during the phase transformations of steel alloys. The sensor was connected to a low-noise amplifier. The noise level in the operating band did not exceed 50 microvolts peak-to-peak. The amplifier enabled signal amplification in the range of 20 to 60 dB. A high-pass filter connected to the amplifier's output allowed for the elimination of background noise from the measurement system. The amplified signal was converted to a .wav file and saved to a PC. The research apparatus was equipped with software for analyzing the recorded signal. The Spectrum program was used for the graphical presentation of the AE signal in the form of a spectrogram in the time-frequency domain. To generate the spectrogram in the Spectrum program, the STFT (Short Time Fourier Transform) algorithm with a Hamming window was used. The resolution of the graph was 0.6 kHz/1 s, and the maximum displayed frequency was 500 kHz.

RESULTS AND DISCUSSION

As a result of the isothermal hardening process of the alloy steel, the AE signal was recorded. Figure 3 shows the relationship between the root mean square (RMS) value of the AE signal and time for the measurements performed. Based on these relationships, it is possible to determine the duration of the isothermal transformation and identify two phases.



Figure 3. Comparison of the time courses of the RMS values of the AE signal recorded during the hardening process of 27MnCrB5-2 alloy steel

The results of the AE measurements, showing the spectral images of the acoustic emission signal from different phase transformations, are presented in Fig. 4.



Figure 4. Spectrograms of the AE signal energy spectrum, with the horizontal axis representing the signal recording time and the vertical axis representing the signal frequency

The results of the acoustic emission signals and the spectral images indicate that two physical phenomena emitting acoustic signals occur in the studied material during its heat treatment. The images show that at the beginning of the hardening process, no peaks are observed, indicating the level of the acoustic background. Only after about 400 seconds can the first events be seen, which gradually increase in intensity and last until 800 seconds, after which they diminish. This signal characteristic indicates the occurrence of the first physical phenomenon, i.e., the first phase transformation. Based on literature data⁴, it can be inferred that under such heat treatment conditions, the transformation of austenite to ferrite occurs first. It can also be observed that the maximum spectral density falls within the range of 100-300 kHz. Subsequently, single acoustic emission events are observed, and this state lasts until 1800 seconds. After this time, another phenomenon occurs, with an increase in the intensity and power of the signal. Acoustic effects from the phase transformation continue until 2400 seconds. In the subsequent stage, the power of the spectrum decreases, and single emission events are observed again. The signal power decreases to the level of the acoustic background. The nature of the acoustic emission signals and the presented spectrogram indicates the occurrence of another phase transformation, which could be the bainitic transformation. The measuring instrument allows for the cyclic recording of approximately 100 milliseconds from each successive second of the signal generated during the phase transformations of metal alloys. These conditions enable a longer recording of the signal, even up to 3600 seconds. However, with longer recording times, some data loss occurs, which is why the signal characteristics shown in the above figures are not as intense, though they still indicate the beginning and end of the phase transformation.

As a result of the conducted dilatometric tests, dilatometric curves were obtained as a function of time and temperature, which are used to analyze the phase transformations occurring in the tested material. The dilatometric curve from the hardening process of alloy steel with the addition of boron 27MnCrB5-2 is shown in Fig. 5.



Figure 5. Fragment of the curve from the dilatometric test during cooling of 27MnCrB5-2 steel

As observed on the dilatometric curve shown in Figure 5, transformations occur within a specific temperature range. The first change in the slope of the curve, where the cooling curve loses its linear character, can be seen at higher temperatures around 700 °C, indicating the beginning of the ferritic transformation. This means that structural changes in the material began, which concluded upon reaching a temperature of about 640 °C. Another change in the curve can be observed in the temperature range of 550–450 °C. These conditions indicate the occurrence of the bainitic transformation.

The microstructural analysis of the 27MnCrB5-2 alloy steel subjected to hardening was carried out using a Phenom XL scanning electron microscope. Microscopic examinations will enable comparison of microstructure images after hardening and after etching the structure with reagent Vilella, analysis of the surface condition and assessment of its morphology. The results of microstructural examinations are presented in Fig. 6.

Problems with identifying phase transformations during the heat treatment of steel arise from the complexity of thermodynamic processes occurring within the volume of the hardened material. The results of dilatometric tests and the acoustic emission signal presented above proved to be effective for tracking physical phenomena. Events with different energies indicating phase transformations were detected in the recorded AE signal. There are several different approaches to studying the AE in solids, metals, and their alloys. The most significant include studies on atomic structure reorganization, closely related to impurity diffusion. Another focus is on dislocation studies, generally relating to dislocation density, multiplication, and lattice geometry. The main sources of AE in metals and their alloys are considered to be dislocation movement due to plastic deformation, twinning, and displacive phase transformations^{16, 17, 22, 23}. During phase transformations in heat-treated steels, an AE signal is generated. However, strong, effective sources of this signal are not always present in the steel. When phase transformation involves very slow, diffusive atom movements, the AE signal energy is very low. In contrast, when the crystal lattice of the cooled material



Figure 6. Images of the microstructure after hardening of the 27MnCrB5-2 alloy steel, after etching the matrix with Vilella reagent, SEM microscope. Visible white-etched acicular ferrite and wedge-shaped plates of Widmanstätten ferrite in the bainite matrix

reorganizes, causing atom position changes, a high-frequency, high-energy signal is generated. This mechanism is not fully understood, so tracking the dynamics of the AE signal associated with phase transformations can provide valuable information about the nature and stages of these transformations. Since dislocations are a significant source of AE in metals, the literature contains many studies on this topic. These studies cover dislocation mobility, generation mechanisms, interactions, blocking, and annihilation. For example, Eshelby's work²⁴ from 1962, where the author attributed AE generation to dislocation acceleration and deceleration processes. He first formulated the relationship between the emitted elastic energy and changes in dislocation velocity or the oscillatory movement of a single kink along the dislocation line. Dislocation movement in metals was also studied in²⁵, confirming its significant role in AE generation. Pawełek et al.^{24, 26} noted that collective dislocation movement could be an AE signal source in metal studies. They observed that dislocation movement within the material is related to the acceleration and synchronized annihilation of dislocations, occurring both internally and on the material's surface. AE research has been conducted in many laboratories by researchers using various investigative methods^{14, 27-30}. In the case of the 27MnCrB5-2 alloy steel cooled continuously in air, AE signals were also recorded. The formed Widmanstätten ferrite and bainite, due to their transformation nature, emit AE signals. Monitoring heat treatment processes is crucial for the utility values of final industrial products. By tracking the kinetics of supercooled austenite decomposition, industrial processes can be regulated and improved. Optimizing these processes can enable economic time use without compromising product quality. Utilizing methods that control the start and end of phase transformations can thus contribute to the future optimization of industrial processes.

CONCLUSIONS

As a result of the conducted studies of phase transformations in 27MnCrB5-2 alloy steel using research methods such as dilatometry and acoustic emission, the following conclusions can be drawn:

1. As a result of continuous air cooling of 27MnCrB5-2 alloy steel with the addition of boron using dilatometric tests, it can be seen that the transformation product was Widmanstätten ferrite and bainite.

2. The results obtained using the acoustic emission method indicate, analogously to dilatometric tests, the occurrence of two phase transformations.

3. The duration of transformations in the tested steel depends strictly on the hardening temperature of the steel.

4. The dominant range of the AE signal spectrum generated during the tests was in the range of 100-300 kHz. In addition, the maximum spectral density was observed at a frequency of 180-200 kHz.

5. A large population of events with different energy values was identified in the tested AE signal.

6. The acoustic emission method can be successfully used to assess the kinetics of complex phase transformations occurring during steel hardening.

7. Control of the phase transformation process may facilitate obtaining steel with more favorable functional properties in the future.

8. The use of methods that enable control of the beginning and end of phase changes using acoustic emission can be used in the industry. In addition, it can contribute to the optimization of industrial processes.

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