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# ACOUSTIC EMISSION DURING AUSTENITE DECOMPOSITION INTO LOWER BAINITE WITH MIDRIB

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Phenomena accompanying bainitic transformation in the range of swing back were investigated by using acoustic methods and microscopic analyses. It has been found out that midribs are probably formed in the first few dozen seconds. Plates of a midrib are a twinned thin-plate martensite. While forming midribs, sources of displacement are activated. Rapid local deformations exceeding the limit of plasticity produce large acoustic effects. Midribs are the first elements of the formed sheaves of bainite and privileged sites for bainite nucleation. The tests were made by using specimens of high-carbon steel with 1.1C wt.%, where a very clear range of the accelerated start of bainite transformation was observed. This range was determined earlier by using dilatometric measurements. It was stated that morphology of microstructures of lower bainite with a midrib (LBM) shows butterfly-shaped arrangement of plates, which is induced by midribs. An outline of the acoustic spectrum corresponds with a characteristic of the continuous precipitation.

**Key words**: thermal treatment of metals, austenite phase transformations in steels, acoustic emission monitoring of phase transformations in solids.

## 1. Introduction

With the development of science and technologies, requirements for materials, among which metals are the majority, still increase. Therefore, more effective methods of improving their quality and achieving suitable parameters are being sought. Thermal treatment of metals is one of the basic processing methods. This type of treatment, despite intensive technological development, has no coherent theoretical basis for the related phenomena. Knowledge of the phenomena is an indispensable condition for modelling the materials. The research on the events running during decomposition of overcooled austenite at a constant temperature for eutectoid steel was first made by BAIN and DAVENPORT in 1930, who initiated further studies on the austenite isothermal decomposition [1]. Bainite transformation is the least recognized of all the austenite transformations in steels. Bainite is formed in the range of temperature dividing diffusive transitions and the non-diffusive martensite transition. While lowering temperature, the rate of transition for the overcooled austenite decreases. For a given range there is an area of high stability of the overcooled austenite and a minimal rate of its decomposition. In steels containing more than 0.6C wt.%, changes in the kinetics were observed, including the range of temperatures near $M_s$. This phenomenon is characterized by a distinct acceleration of the transformation start [2–4]. It has been stated that this fact is connected with bainitic transformation and can be seen clearly up to app. 25% of the transition. Curves related with further stages of the transformation show only insignificant symptoms of the abnormal kinetics. The reasons for this phenomenon have not been fully explained so far. In particular, no mathematical description has been even attempted. A pioneer research on modelling in this field has been made by T.Z. WOŹNIAK [5, 6].

The accelerated start of the transformation is induced by forming midribs which, in turn, initiate further run of the bainitic transition. OKAMOTO and OKA [2, 3] suggested that midrib was a thin-plate isothermal martensite formed before the lower bainite nucleation. Even though the nature of a midrib is still not fully understood, it is agreed that a thin plate of midrib is the first area of a new phase, and a plane of midrib is the one on which shearing takes place, i.e. a habit plane. Midribs are also observed in plate martensite and are considered as one of the sub-structural features distinguishing this type of martensite [7]. A single thin-plate carbon-free ferrite was also observed in bainite sheaves. This type of ferrite, called by SPANOS *et al.* [8] a "spine", is similar to a midrib in many aspects. Controversies concerning the nature of the midrib have not been settled yet and the studies on it are far from final solution. It has been found that "spine" is a feature generally observed in a structure of lower bainite [9], which is similar to an isothermal midrib observed by OKAMOTO and OKA [2, 3].

Gathering information by using acoustic methods is widely practiced in studies on the properties and structure of the matter, and contributes to identifying the processes taking place in it. Only the processes during which accumulation and release of energy are observed can be a source of acoustic emission (AE). One of the main reasons of generating acoustic energy in metals is the motion of displacements, especially the motion with significant accelerations or delays. The generation of the acoustic energy (AE) signals by displacement movements is a subject of many studies. The received AE signal is deformed by the medium suppression and by a multiple reflection on the material boundaries. Due to computer techniques, the accuracy of measurements has considerably increased and the selected parameters of the AE signals can be comprehensively analysed [10, 11].

It is anticipated that midribs should to induce acoustic effects because their growth rate is as fast as that in the case of martensite and may be limited only by the velocity of sound in metal.

## 2. Method of the research

In the research, a model material of hypereutectoid carbon steel of 1.1 pct was used. The steel was supplied in the form of cylindrical rods of size 30 × 50 mm in a softened state and from one industrial batch cast. The analyses of chemical composition have been made on the appliance Spectrolab and are presented in Table 1.

**Table 1.** Chemical composition of steels.

| C [%] | Si [%] | Mn [%] | Cr [%] | Ni [%] | Mo [%] | Cu [%] | V [%] | P [%] | S [%] | $M_s$ [°C] |
|---|---|---|---|---|---|---|---|---|---|---|
| 1.090 | 0.370 | 0.209 | 0.077 | 0.050 | 0.013 | 0.132 | 0.0023 | 0.004 | 0.016 | 116.6 ± 23.5 |

The austenitizing of dilatomatric specimens was carried out in a chamber oven at 950°C during 30 min with accuracy of recording of 5°C. Such conditions caused the dissolution of carbides in austenite. The tests on acoustic effects were carried out during austempering at temperatures from 100°C to 150°C, i.e. in the range of the lower bainite with a midrib formation. For this purpose, a special stand was made to carry out austempering with recording acoustic effects that occur during the process.

a) b)

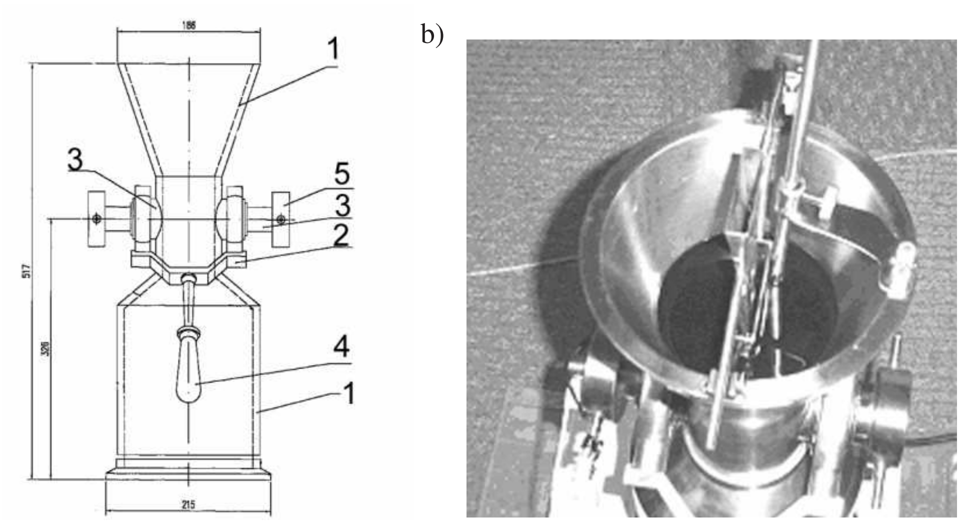



Fig. 1. a) A diagram of the experimental set-up for austempering in oil with a possibility of recording acoustic effects, 1 – elements of body and basis structure, 2 – link of movable mandrels – 3 with leverage – 4, 5 – cooling system. b) Top view of the experimental set-up, presenting the specimen transporting mechanism to the movable mandrels after throwing the tested specimens into oil.

The holding time at temperature of isothermal transformation was dependent on the time of the acoustic effects. Temperatures were measured using a thermocouple.

The research was carried out on cylindrical specimens 36 mm in diameter and 2-mm thick. After heating the specimen up to the temperature of austenitizing, it was thrown to a special mechanism that allows adjusting it between clamping mandrels. While constructing the set-up, it was taken into consideration that the dependence of AE signal on the place of the signal generation can be neglected for inconsiderable distances from the transducer (not more than 200 mm) [12].

An acoustic sensor was installed on one of the two movable mandrels and connected to the analyzer. To obtain a fast change of temperature during austempering, orifices were made in the leading mechanism. It provided a maximal contact of the specimen with a cooling agent. The size of the appliance was designed so that the maximal increase in the oil temperature, being a result of cooling a specimen from the temperature of 950°C, should not exceed 1°C.

## 3. A characteristic of the equipment for recording acoustic emission

Reaching the material surface, the excited elastic waves were transformed into the electric signal by using a piezoelectric transducer. For electro-acoustic analysis, it is necessary to measure the amplitude and duration time of the impulses with the accuracy from microseconds to decimal fractions of a second. Depending on the possibilities of the measurement equipment, the signal mentioned above can be analysed in many ways.

In our studies, in order to record and analyse EA, we used an experimental set-up installed at the IPPT PAN and presented in Fig. 2. It is equipped with the following elements:

1. A wide-band acoustic emission sensor of type WD, produced by Physical Acoustics Corp.
2. A single-channel analyzer of acoustic emission with the transmission band 1–1000 kHz equipped with: software, a set of active filters and an output of rms value.
3. A PC Card of Analogue to Digital Converter of type Adlink 9112 (12 bits).
4. PC with Pentium IV.
5. Software to collect the data (with options to record and analyze them).

Pre-trials of EA interception with the use of specimens of steel 1H18N9T allowed us to establish correct working parameters for the system.

The measuring set can co-operate with a computer configured in the environment of MS Windows XP.

Signals received by the piezoelectric sensor were recorded by using the analyzer. The recording head reacted on variable tensions applied perpendicularly to its front. Sensitivity of the head was defined as 63 dB with reference to the calibrating signal, being a pressure wave with amplitude of 1 μbar. The head responded to the wide-band frequency waves and practically recorded vibrations of frequency from 1 kHz to 1 MHz.

Configuration for recording the RMS value

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| EA Sensor<br>1 kHz–1000 kHz | → | Low-noise<br>Amplifier | → | High-pass Filter<br>10 kHz | → | RMS<br>Converter | → | AD Transducer<br>Sampling<br>1 kHz | → | Computer<br>Software |

Fig. 2. Block diagram of the experimental set-up used for AE measurement.

Considering technical possibilities of the whole path of the signal treatment, the effective band of the recorded acoustic signal frequencies was 10–700 kHz [12].

Electroacoustic signals were recorded in PC memory by using the card ADLINK 9112. Sampling frequency of the card Adlink 9112 was 1 kHz. The signal recorded by the appliance described above was a binary file with DAT enhance. Analysis of the recorded Acoustic Emission activity was focused on tracing of energy changes in the signal. This led the authors to control the intensity of the phase transition processes what was the aim of investigation. The registered AE signal has the form of a wideband noise with the remarkably seen maximum in the time domain. Therefore a hardware RMS value conversion was chosen as the most efficient method of signal characterisation. Wider discussion of the strategies of multi-source AE signal analysis can be found in [11].

## 4. Results of the research

Structural objects of characteristic “butterfly” morphology were searched for at transformation temperatures of 140°C and 160°C, which makes the shapes most similar to the model ones found in literature. Considering the results of the equations presented by T. Z. WOŹNIAK [5, 6], the micrograph analysis was carried out with times when the 1st stage of the transformation was predominant, Fig. 3.

A lower bainite with midrib (LBM) is formed at the 1st stage of the reaction, and a lower “classic” bainite (LB) – at the 2nd stage. Such two-stage transformation was identified by using kinetic tests [15, 16], which can be observed in the micrographs, Fig. 4.

Basing on metallographic research for larger volume fractions, it is difficult to assess whether the observed sheaves show a morphology of LBM or LB. These differences are revealed using electron microscopy. After austempering in the range of LBM, electron micrographs show light trail-shaped midribs, Fig. 5.

In our research generally occurred selective etching of midribs in thin areas of foils, which made the research more difficult. The etched midrib at the side of a sheave, Fig. 6, may suggest that it is, according to the GONG and GU classification [17], the so-called “bainite with no tails” and that the bainite growth takes place on one side of the midrib.

The micrographs of bainite sheaves are similar to the images observed by OKAMOTO and OKA [3] in their research using electron microscopy with accelerating voltage of 200 kV. Due to diffraction areas surrounding the midrib, the existence of ferrite and cementite was confirmed, Fig. 7.

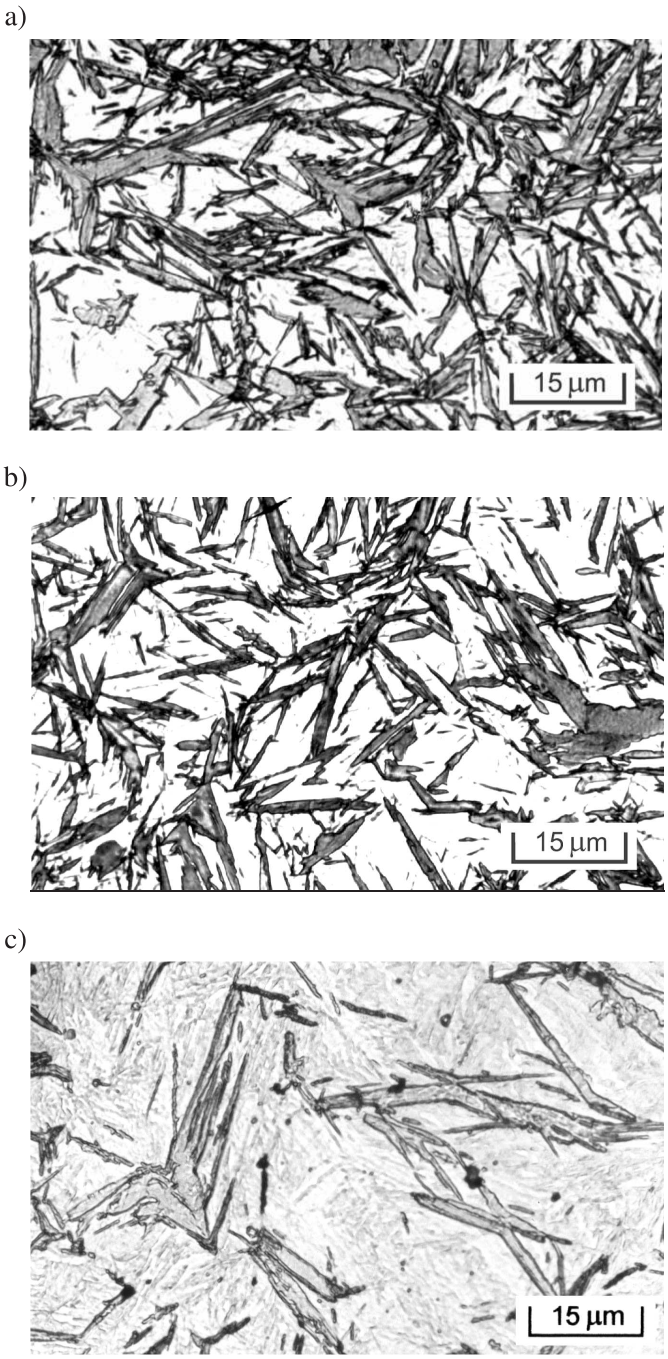



Fig. 3. Selected fragments of the structural elements of butterfly bainite in the 1st stage of the transformation, after austempering with different temperatures, etched using Villela's reagent: a) 130°C with holding time of 8439 s, b) 140°C with holding time of 34930 s, c) 160°C with holding time of 1800 s.

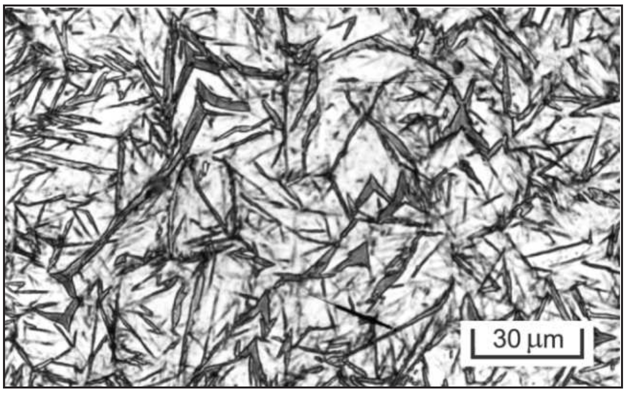



Fig. 4. Optical micrographs after austempering at 160°C, treatment with Vilella's reagent, holding time of 15300 s, volume fraction of bainite 55%.

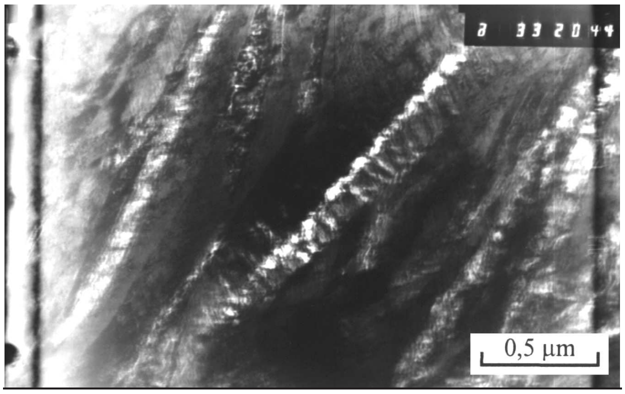



Fig. 5. Microstructure after austempering at 160°C during 4 hours, with characteristic sheaves of bainite with midribs.

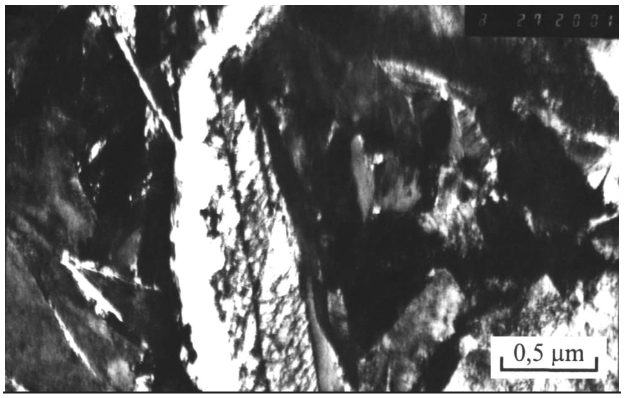



Fig. 6. A microstructure of bainite sheaves of butterfly morphology with the etched midrib after austempering at 160°C, 3000 s.

a) b)

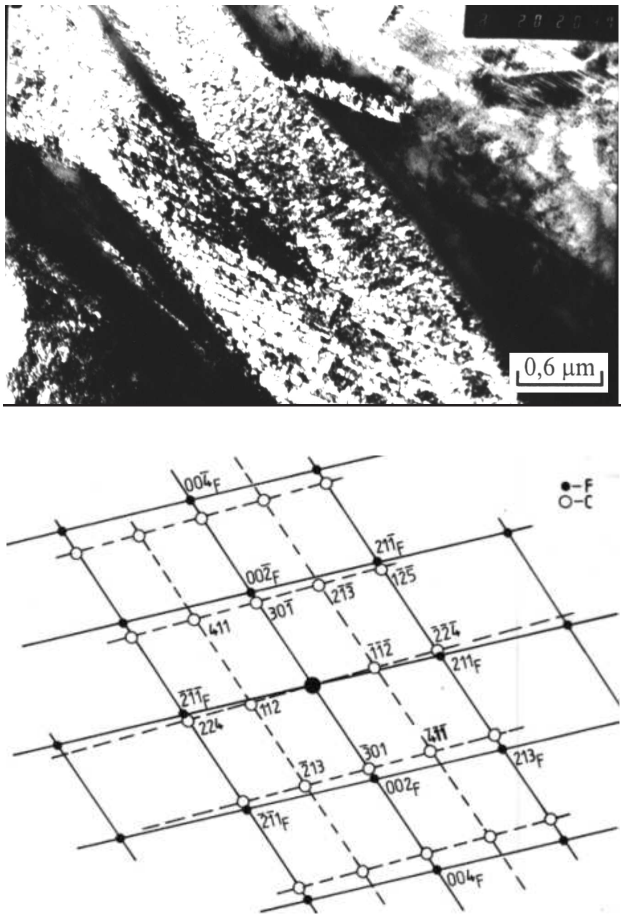



Fig. 7. A microstructure after austempering at 160°C after 10 min. a) Sub-structure of a sheave with secondary platelets, b) solution of the diffraction pattern, axis plane strip $[120]_F$.

Sheaves of bainite have a structure of clearly noticeable secondary plates. Sheaves of lower bainite of morphology very similar to the one shown in Fig. 8 were observed earlier by SPANOS *et al.* [8].

In the range of swing back there is observed a double decomposition of meta-stable austenite and the rate of the decomposition is the higher the lower the temperature of the transformation [15, 16]. At the 1st stage of the reaction predominate thicker plates of lower bainite with midribs as the centres of their nucleation. At the 2nd stage of the reaction, a "classic" lower bainite prevails, generally nucleating on grain boundaries. Basing on the earlier kinetic analyses, the parameters of heat treatment were adjusted to the simultaneous recording of the acoustic effects during austempering. For this purpose, a reference material emitting reference acoustic signals was selected at the beginning. In the reference material made of steel 1H18N9, no phase transitions took place, and the acoustic image was at the level of background, Fig. 8. While conducting the ba-

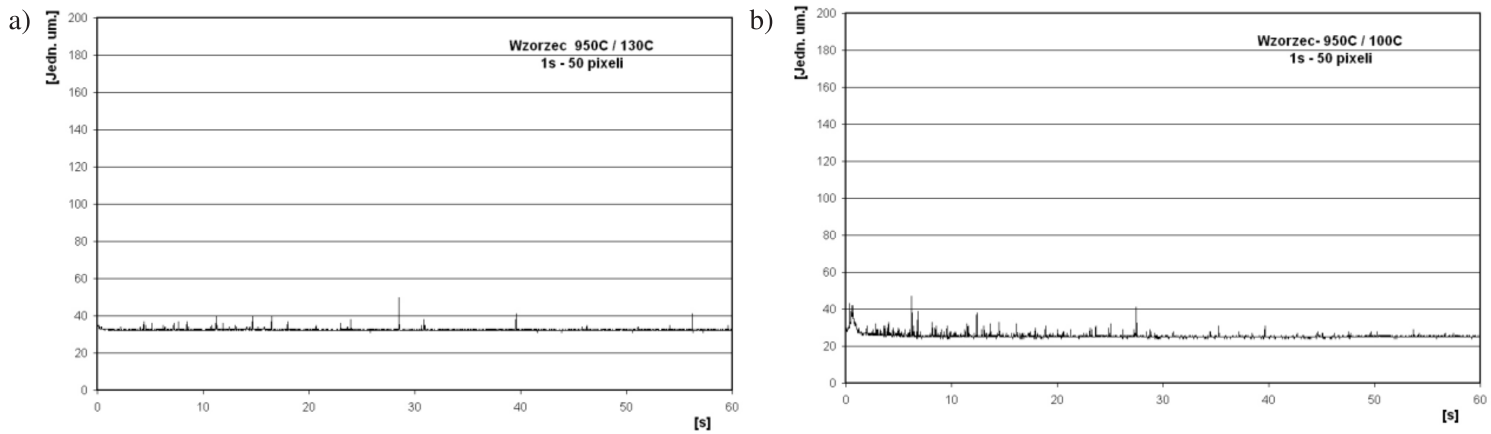



Fig. 8. AE spectra on pattern specimens made of austenitic steel 1H18N9, at temperatures of cooling medium: a) 100°C, b) 130°C, with graphic compressions 1 pix. – 0.02 s.

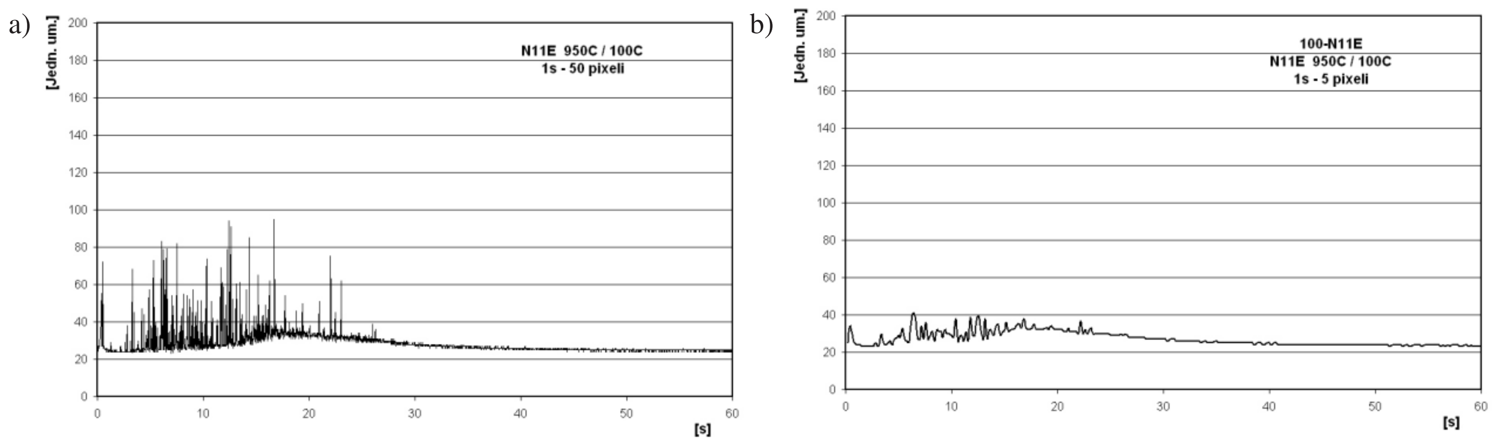
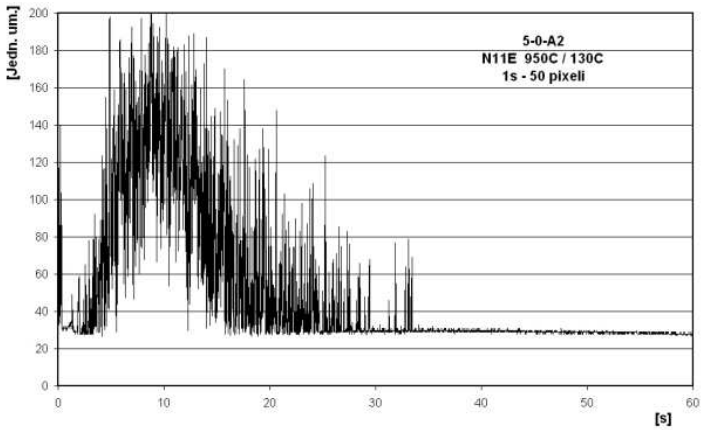



Fig. 9. AE spectra below temperature $M_s$ while austempering at 100°C, with different graphic compressions: a) 1 pix. – 0.02 s, b) 1 pix. – 0.2 s.

a)


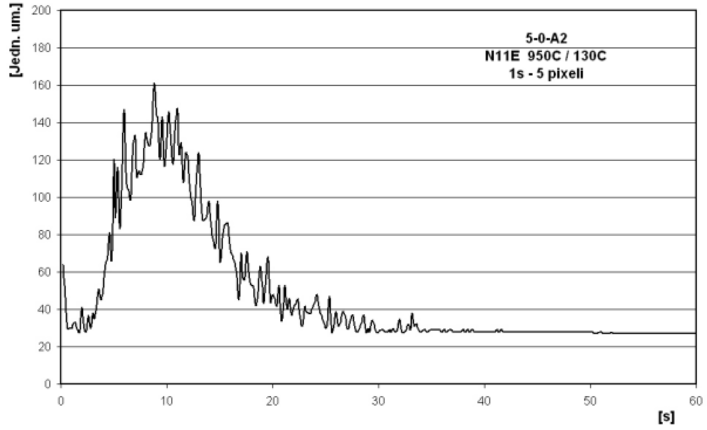
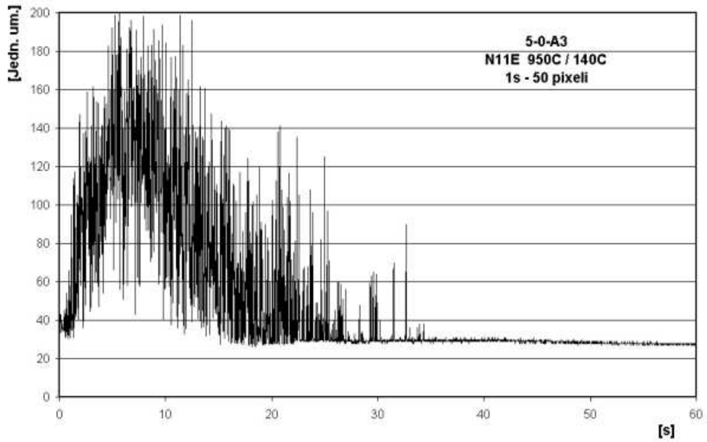

b)



Fig. 10. AE spectra above $M_s$ during austempering at 130°C, with different graphic compressions: a) 1 pix. – 0.02 s, b) 1 pix. – 0.2 s.

a)


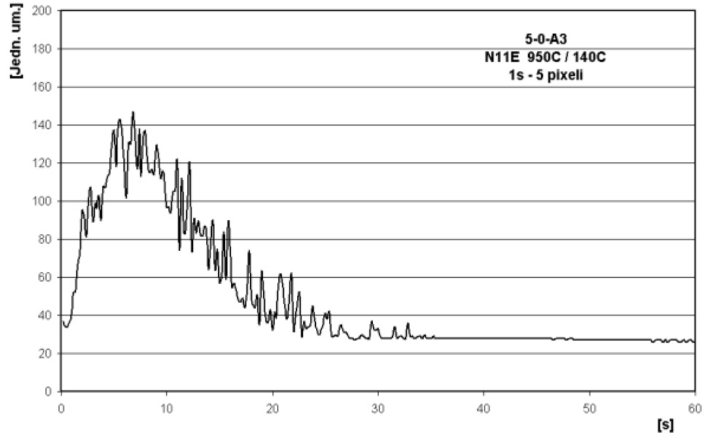

b)



Fig. 11. AE spectra above $M_s$ during austempering at 140°C, with different graphic compressions: a) 1 pix. – 0.02 s, b) 1 pix. – 0.2 s.

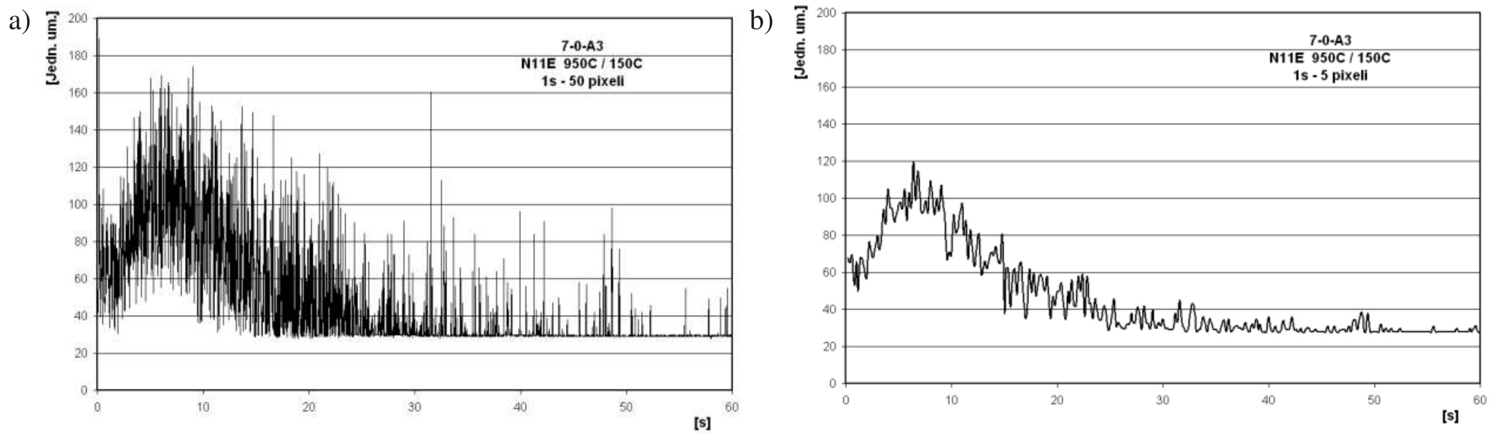



Fig. 12. AE spectra above $M_s$ during austempering at 150°C, with different graphic compressions: a) 1 pix. – 0.02 s, b) 1 pix. – 0.2 s.

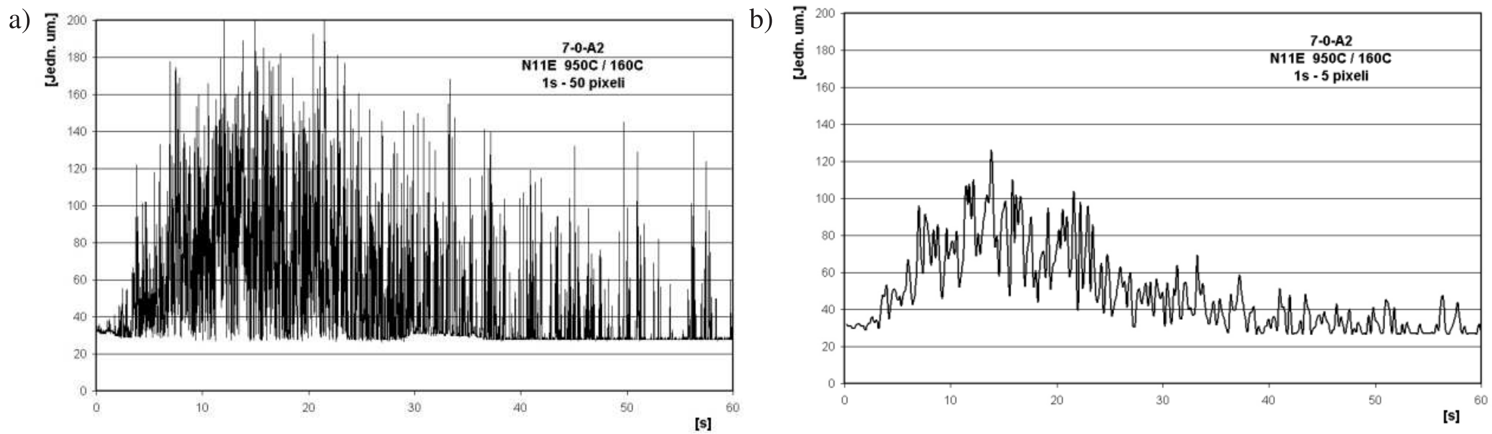



Fig. 13. AE spectra above $M_s$ during austempering at 160°C, with different graphic compressions: a) 1 pix. – 0.02 s, b) 1 pix. – 0.2 s.

sic studies, acoustic effects were recorded during austempering below $M_s$, Fig. 10, and above $M_s$, Figs. 10–13. The level of acoustic signals below $M_s$ is relatively insignificant, although the athermal martensitic transformation occurs intensely in this range of temperatures. Above this temperature, within the scope of where the martensitic transformation is not observable yet, very intensive acoustic effects occur, which are the higher the closer to $M_s$ the temperature of the transformation is, Fig. 10. At 160°C, the acoustic emission (AE) spectrum is less intense, which results in a volume decrease even to the level of background, Fig. 13. Diagrams presented in Figs. 8a–13a, made with higher resolution, were narrowed by the method of graphical processing in order to save space.

## 5. Discussion

Butterfly microstructures were observed in other materials by many researchers [18]. However, they usually occurred in the martensitic range. Details concerning substructure and crystallography have not been fully recognized so far. The butterfly martensite consists of two plates with an obtuse angle between them. The plates do not grow across the whole grain as it is in a case of lenticular or plate martensite. A similar situation is for bainite nucleating on midribs and being also of butterfly morphology [16, 19]. Butterfly martensite is formed under a specific pair of relationships, where planes: the habit one $(252)_f$ and $(2\bar{5}2)_f$ make an angle of app. 140°.

The growth of midrib changes the transformation space. The deformation of the shape is a strain with an invariant plane (IPS), with a considerable shear component 0.22 and a dilatation strain (app. 0.03) directed perpendicularly to the habit plane, i.e. the invariant plane [20]. A relatively small dilatometric strain is used in dilatometric experiments to analyze kinetics of the transformations. A shear component connected with a change in shape is not detected dilatometrically. Strains connected with lattice shearing during the midrib formation induce considerable acoustic effects.

The transformation of the austenitic structure into the martensitic one related with lattice shear is connected with considerable acoustic emission (AE) symptoms [14]. The individual plates of a new phase that nucleate very fast within merely $10^{-8}$ – $10^{-6}$ s, induce rapid strains exceeding the yield point and release sources of new displacements. Apart from that, plates of martensite are able to twin, which is an additional source of the acoustic emission (AE). Those strongly localized processes lead finally to considerable shearing and dilatation strains which make the main AE source [14].

Acoustic effects are also related to an inhibiting distance of displacements that increases in proportion to displacement velocities. Kinetic energy, however, increases in proportion to the square of the velocity. Thus, while inhibiting the displacement that moves with a higher velocity, the concentration of the dissipated energy of vibrations per atom increases, which induces a more intense acoustic effect (AE) [14]. Therefore, there are significantly more acoustic effects while forming midribs than while forming further sheaves of bainite. The emission frequency of acoustic impulses is in direct proportion to the frequency of displacing movement initiation, and the amplitude of these waves is proportional to the kinetic energy dissipated as a result of displacement inhibition, i.e. to their velocities and inhibiting distances.

When the temperature of austempering gets closer to the martensite start temperature ($M_s$), the transformation rate accelerates rapidly. It is a result of the rapid formation of midrib plates during early stages of austenite decomposition where acoustic effects were observed. Basing on acoustic studies, it cannot be assumed as correct what other authors [21] say that strains induced by bainite growths form isothermal martensite only in further order. An actual situation is opposite, because the original sub-structural elements are not sub-plates of bainite but the plates making midribs.

Nucleation of both midribs and bainite sub-plates is a consequence of spontaneous dissociation of the displacement effects occurring in the matrix phase. Nucleation cannot take place until overcooling is large enough, because it forms structural defects. Since the displacements undergo slips, the mechanism of nucleation does not require diffusion. The only barrier of nucleation is the resistance of the displacement slip. Deformations related with the process of displacement lead to a new crystalline structure. Considering the possibilities of acoustic effects generation, the situations with long delays or accelerations and high velocities of displacements are particularly important. For displacement velocity $v_0 = (0.1 - 0.5)\, v_s$, the inhibition distance is of the order of a few dozen of lattice parameters [14]. With such a velocity, the whole kinetic energy of the stopping displacement changes into the additional oscillation energy of atoms situated next to the displacement line and is shifted onto the atoms in further distance.

In his kinetic model, BHADESHIA [22] considers the growth of bainitic ferrite as the shear growth. Despite such a mechanism of transformation, the acoustic effects are at the level of background. You can distinguish three separate stages of bainite transformation. At first: a sub-plate nucleates on austenite grain boundary and grows longer. Its growth is stopped by plastic deformation inside the austenite. So, the plates increase only up to a very limited size. After each sub-plate of bainite has grown, a partition of carbon into the residual austenite takes place. Then new sub-plates nucleate at the top of former ones and thus, as the process goes on, the structure of the whole sheave develops. New sub-plates are preferably formed near the tops of the already existing plates; nucleation in neighbouring locations is of much lower rate. Therefore, a general shape of the sheave is considered in three dimensions, with the growth limited only by austenite grain or by twin boundaries. The average rate of the sheave elongation is lower than the rate of sub-plate growth, owing to the delay in the nucleation of consecutive sub-plates. Thus, the total kinetics is considered mainly as the one controlled by nucleation rate. Although bainite grows rapidly, its elongation rate is much lower than the martensite growth rate, which is app. $10^6$µm/s. The elongation rate for sub-plate is about 75 $\mu ms^{-1}$, so it is many orders of magnitude lower than the rate of martensite growth. The movement of interfaces is relatively slower, even though it is also a slip. This is probably caused by plastic deformation during the growth of bainite. This is the reason why the level of acoustic effects coming from the growth of bainite sub-plates is too low and within the background level. It was stated that, with the velocity of displacements $v_0 \approx 0.01 v_s$ ($v_s$ – velocity of sound in the medium), the length of slowing space is of order b, i.e. of the lattice parameter.

## 6. Conclusions

Fast development of technology in all disciplines of science creates much wider possibilities in metallurgical research. Thanks to it, alloys of more and more diversified and developed properties can be produced. Acoustic methods can be perfectly complementary to the metallographic study to recognize micro-structures. The structure of metals and alloys is a main factor deciding on their properties.

The experiments revealed that the obtained acoustic image is a result of structural transformations taking place just at the start of transformation, when thin-plate martensite, i.e. the midrib, is formed. The processes running during the formation of midribs generate signals of extremely high energy. You can recognize that acoustic signals permit the identification of specific events, inducing the so-called effect of swing back in the kinetics. It is a result of midrib formation preceding proper bainitic transformation. The results proved the validity of the earlier developed model of the bainitic transformation in the range of swing back. In this model, simultaneous austenite decomposition into two products was assumed as an effect of bainite transition. The first product is formed just on midribs, the existence of which has been additionally confirmed by acoustic tests.

The recording of AE signals during austempering required constructing a special experimental set-up. First trials of AE signal monitoring consisted in generating a data file received by a head while realising the procedures on a pattern material made of austenitic steel with no martensitic transformation.

The results of EA analysis indicated the usefulness of the undertaken actions. It can be assumed that the microstructure will be better identified when the spectral characteristics of the recorded signals is recognised more precisely. The analysis of the emission caused by phase transformations may be enlarged by advanced computation tools, such as algorithms of the digital signal analysis. This type of analysis would require applying e.g. fast Fourier transformations, signal filtering or a wave analysis.

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