Vibroacoustic diagnostics of surface electron beam alloying process of ferritic stainless steel

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Abstract. Controlling the process of surface electron beam alloying substantially complicates the instability of the parameters of the microsecond pulse of the electron beam and the process of its interaction with the processed material, which leads to fairly significant random changes that occur spontaneously, regardless of the control system. In this situation, it is proposed to use the method of acoustic emission, which has long established itself as an effective tool in the study of phase transformations and plastic deformation. Under the action of a low-energy high-current electron beam, a vibroacoustic wave is caused by the effect of thermoelasticity in a thin surface layer heated up to the evaporation temperature of a substance in a vacuum. It is experimentally shown that a process with high vibroacoustic activity occurs upon irradiation of plates of pre-nitrided steel 08Cr17Ti with a deposited Nb70Hf22Ti8 film in the range 11-22 kHz after the 10th millisecond. The source of this vibroacoustic signal is the appearance of a martensitic component in the near-surface layer of the sample, caused by the formation of niobium-based nitride. Tracking changes in the effective value of the vibroacoustic signal allows us to select rational irradiation modes for electron-beam surface alloying, assuming, on the one hand, the maximum possible power supply, on the other hand, the limitations caused by the possibility of evaporation of the previously deposited film with the alloying components.

1. Introduction

Numerous studies of the processes of surface alloying of structural and tool materials with the help of concentrated energy fluxes have shown the possibility of a significant effect on their physical and mechanical properties, including obtained experimental results that showed the expediency of creating wear-resistant near-surface layers by means of electron-beam technology using the source of a wide-aperture low-energy high-current electron beam (LEHCEB) «RITM» [1]. Such layers were obtained by initiating of exothermic chemical reactions between the base material and the film deposited on it with the formation of new phase components [2-4].

At the same time, the control of the surface alloying process aggravates a sufficiently large number of control factors, which are determined primarily by the thickness of the surface layer, in which the energy sufficient for its heat treatment (melting and partial evaporation) must be introduced. This is the value of the charging voltage, on which the specific energy of the beam depends, the thickness of the film with the alloying components deposited using magnetron sputtering, the number of processing cycles, etc. Their optimization was carried out, based on the data of metallographic, X-ray structural and spectral studies [5].
However, the instability of the parameters of the electron beam and the process of its interaction with the material being processed leads to rather significant random changes that occur spontaneously, regardless of the control system. Therefore, it is rather difficult to ensure a strict repeatability of the technological process of surface alloying, especially since there is no single parameter that reflects its kinetics.

When pulsed by electrons, the surface layer of the substrate with a doped coating deposited on it undergoes pulsed heating which causes the volume expansion of the material and the corresponding movement of the elastic wave [6, 7], which is accompanied by significant structural changes, enhanced by processes of substance transfer, associated with a change in the defective structure of the material [8]. The source of excitation of stress waves can be the effect of thermoelasticity, as well as the recoil impulse caused by evaporation of the material in the irradiation zone. An equally important factor is the quasi-static thermoelastic stresses appearing in the surface layers under pulsed heating [9], the range of which extends to hundreds of micrometers, and the value can exceed the yield strength of the material.

In this situation, it is advisable to use the acoustic emission method, which has long proved itself as an effective tool for studying phase transformations and plastic deformation [10].

This work presents an attempt to relate the processes occurring in nitrided steel sheet during the process of surface alloying with a change in the parameters of the vibroacoustic signal that occurs when exposed to LEHCEB.

2. Experimental setup
In order to remove extraneous noise as much as possible, the working table of the «RITM-SP» unit was replaced by a sample of AISI 439 (08Cr17Ti) sheet of nickel-free stainless ferritic steel. The choice of this steel as a starting material was due to the high chromium content and the low carbon content, in which practically no carbides of the alloying elements were formed, with the exception of a small amount of titanium carbides. This makes it possible to keep the chromium in a solid solution and to ensure a high solubility of nitrogen, which will subsequently be necessary for the formation of special nitrides during the surface alloying process.

To achieve effect of corrosion and wear resistance, the steel was preliminarily subjected to low-temperature nitriding using a two-stage vacuum-arc gas discharge [11]. The treatment was carried out at a temperature of 500°C for 60 minutes, which made it possible to create on the surface of a chemically-heat treated layer with a thickness of about 40 μm without the formation of chromium nitride Cr2N. The near-surface layer acquires a martensitic-austenite structure. At the same time, an increase in the average mass fraction of nitrogen leads to stabilization of the residual austenite.

Next, a metallic coating of Nb70Hf22Ti8 alloy was applied to a number of plates by magnetron sputtering, which was subsequently used as a surface alloying material. Irradiation of the LEHCEB causes dissociation of iron nitrides. The released nitrogen atoms enter an exothermic chemical reaction with the coating material to form a refractory nitride phase based on Nb and Hf, which are strong nitride-forming agents. The presence of hafnium, which has a greater heat of nitride formation than that of niobium (406 kJ/mole vs. 238), significantly stabilizes the process.

To register the vibroacoustic signal, the plate was connected to the accelerometer KD35 using a waveguide in the form of a copper wire with a cross-section of 2.5 mm². The circuit of the channel is shown in Figure1. The recording and processing of vibroacoustic information was carried out with the help of the software complex "nkRecorder".

The vibroacoustic signal, passing through the elastic system, undergoes transformations in accordance with the amplitude-frequency characteristic of the observation channel [12]. If the spectrum of the primary pulse has a continuous structure covering the frequency range up to 0.25 MHz (with a pulse duration of about 5 μs), then the spectrum of the converted signal registered in the computer's memory will have a discrete appearance. When vibrations pass through an extended elastic system, the acoustic signal decays due to internal friction of materials and contact friction at the joints of individual parts [13]. In the present case, the components in the zone up to 40 kHz stand out quite
well against the background of interference. The low-frequency components up to 1 kHz were also
excluded from consideration, since they included all kinds of noise caused by the operation of the
“RITM-SP” unit.

**Figure 1.** The object of the study placed in the unit for surface alloying "RITM-SP" and the channel
circuit for recording vibroacoustic signals: 1 – test sample; 2 – wire waveguide; 3 – receiving plate; 4
– accelerometer KD-35 with a magnet; 5 – preamplifier PM-3; 6 – amplifier VSV-003; 7 – ADC
E440; 8 – the recording computer.

To obtain information about the processes taking place on the irradiated sample, the recorded
vibroacoustic signal was subjected to time and frequency analysis. The spectrum of vibration signals
obtained for different time intervals from the moment of pulse occurrence were compared, and the
records of effective signal values in different frequency bands were compared also. The width of the
analyzed frequency bands approximately corresponded to the octave bandwidth.

It makes no sense to express vibro-acceleration in absolute units, since it is measured at a long
distance from the controlled object. If the gain is constant in the observation channel to compare the
parameters of the vibroacoustic signal received at different times or under different conditions, it is
enough to estimate it in mV, fixed in the computer program.

3. Results and discussion

The operating pulses were applied at different values of the charging voltage of the generator.
Effective amplitude values of these signals in octave bands with central frequencies of 16 and 32 kHz
were taken as the parameters of the vibroacoustic signal, reflecting the kinetics of the processes on the
work piece surface. The main energy of the vibroacoustic signal is concentrated on the time interval of
up to 20 ms. The high-frequency component of 32 kHz, the amplitude of which is 4 times smaller
compared to an octave of 16 kHz, decays faster.

The amplitude of the vibroacoustic signal at a high frequency with a charging voltage of 16 to 20
kV varies little with the growth of the charging voltage. The sharp growth starts at 22 kV. For a
voltage of 25 kV in an octave of 32 kHz, the signal amplitude is 3-6 times higher than the signal at 16
kV. A sharp increase in the amplitude of the vibroacoustic signal at large values of the charge voltage
can be attributed to the intensification of the process of evaporation of the metal from the surface of
the sample and, as a consequence, to the enhancement of the recoil pulse associated with the
expansion of the vapor.

Comparison of vibroacoustic signals during irradiation of ordinary and nitrided steel plates on
which a continuous layer of austenite is formed (figure 2a) showed that the character of the response
to the impulse action does not change, despite a significant change in the structure of the metal in the
surface layer.

The next stage of the experiment was the investigation of the influence of changes in the structure
of the near-surface layer that arise after irradiation of a steel plate with a layer of a film containing Nb
and Hf deposited on it, having good mutual solubility, on the vibroacoustic signal. After irradiating
such a sample with an electron beam, an exothermic chemical reaction is triggered to form a nitride
phase. The content of niobium in the near-surface layer of the processed samples was approximately 0.5% according to X-ray spectral analysis. The consequence of the reaction of the formation of a new nitride phase is the depletion of the austenite component with nitrogen, which, under conditions of rapid cooling after pulsed heating by electrons, causes a martensitic transformation (figure 2b).

![Diffractogram](image)

**Figure 2.** a) Diffractogram from a sample of pre-nitrided steel after treatment with an electron beam at a charging voltage of 22 kV, b) the same after carrying out surface alloying.

Both the formation of nitrides (although their amount is insignificant) and martensite in the near-surface layer of the sample is accompanied by a change in volume and promotes plastic deformation, which can be a sufficiently strong source of a vibroacoustic signal. The peculiarities of the effects of irradiation of such samples inevitably appeared in the character of the change in its parameters.

Figures 3 and 4 shows the changes in the effective amplitude when irradiation of the steel samples and the same sample with coating in octaves of 16 and 32 kHz. If we compare the upper graph in figure 5 with the graphs in figure 3, we see that the analogy is traced only on the first 10 ms. After the 10th millisecond, there is a sharp increase in the amplitude to values that were not previously observed when irradiating steel plates without a deposited film. Its value is more than 4 times higher than the previously recorded maximums in this frequency range and in the whole considered frequency range of the signal amplitudes in the region of the maximum corresponding to the 15th millisecond exceed the amplitudes corresponding to the initial phase of the process. This fact shows the occurrence of the process with high vibroacoustic activity after the 10th millisecond.

![Graphs](image)

**Figure 3.** Change in effective values of the amplitude of a vibroacoustic signal when the working pulse is applied to a steel plate with a charging voltage of 22 kV in octaves 16 (top) and 32 (bottom) kHz.
It is interesting that the high-frequency vibroacoustic signal in the octave of 32 kHz (the lower graph), which maximum effective amplitude was observed in the region of the 5th millisecond, differs from its analogs when uncoated plates are irradiated (figure 4) with increased amplitude values. At frequencies above 26 kHz, the amplitude of the beginning of the process exceeds the amplitude in the vicinity of the maximum of the vibroacoustic signal for an octave of 16 kHz. This excess is more noticeable for spectral maximums. For example, in the region of 30 kHz, the amplitude of the vibroacoustic signal for the initial phase of the process is three times bigger than the amplitude in the vicinity of the 15th millisecond.

Attention is also drawn to the wave-like nature of the signal attenuation appearing in both octaves. Local maximums at attenuation were observed on the 15th, 24th and 35th milliseconds. Figure 5 shows three specters of a vibroacoustic signal in the frequency range 11-22 kHz and higher frequencies 24-40 kHz. These specters were plotted for intervals located in the region of three maximums in an octave of 16 and 32 kHz (the numbers show the positions of the sections on the record for spectral analysis). On all the graphs, the spectral maximums retain their position on the frequency axis. This suggests that the excitation of vibroacoustic signals occurs predominantly at the natural frequencies of the elastic system of the measuring channel. The amplitude at high frequencies decays faster with time.

It can be assumed that such behavior of the vibroacoustic signal is associated with a martensitic transformation that takes place in several stages and is caused by the chemical reaction of formation of a niobium-based nitride phase against shock compression. When cooling under nonequilibrium conditions, a martensite of deformation is formed in the external elastic field. The formation of this martensite is well described by the wave model [14], which based on the concept of the stage of growth of a new phase in reconstructive martensitic transformations as a deformation transformation of a lattice propagating in a wave mode. It assumes that the transition to a new structural state is associated with overcoming a certain energy barrier and becomes possible if the level of deformation exceeds a certain threshold value, which in our case is reached in 5-7 ms after the supply of the operating pulse.

The restructuring of the austenite lattice is accompanied by the release of heat and an abrupt change in volume. The growing martensite crystal, starting from the moment of nucleation, exerts a thermal, electrical and mechanical effect on the interphase region $\gamma$-$\alpha$, causing a deviation of its state from the equilibrium one. A nonequilibrium region of $\gamma$-$\alpha$ can be considered as an active medium capable of generating or amplifying waves that cause its change. Such a mechanism is a positive feedback mechanism that converts part of the energy liberated during the process of transformation into the energy of displacement of atoms, which in turn is capable of causing a new wave of martensitic transformation, which could be observed during the above experiments.

**Figure 4.** Change in the effective values of the amplitude of the vibroacoustic signal under the action of a working pulse on a steel plate with a film layer of NbHfTi at a charge voltage of 22 kV in octaves 16 (top) and 32 (bottom) kHz.
4. Conclusion
Monitoring of the vibroacoustic signal makes it possible to observe the course of transformations occurring during surface electron-beam alloying, including estimating the sufficiency of the power of the supplied electron radiation pulses.

Monitoring of changes in the effective value of the vibroacoustic signal allows us to select rational irradiation modes for electron-beam surface doping, assuming, on the one hand, the maximum possible power supply, on the other – the limitations caused by the possibility of evaporation of the previously deposited film with the doping components.

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