Study of the Features of Damage Accumulation in Steel 12Cr18Ni10Ti at Low Temperatures Using Non-Destructive Testing Methods

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Abstract. The paper considers the features of damage accumulation during fatigue loading of 12Cr18Ni10Ti steel, in particular at low temperatures. The character of changes in the magnetic and acoustic characteristics, as well as the index of the fractal dimension of microstructures during the accumulation of damage to the metal structure, is studied. The correlation of the considered indicators is shown, which indicates the possibility of using indicators such as the acoustic parameter, magnetic characteristics (Hc/Br ratio, as well as the coercive force Hc) and the fractal dimension of the metal microstructure to assess structural damage, such as in laboratory conditions, and during the operation of individual parts and structural elements in a wide range of low temperatures. The use of the considered indicators in the complex will significantly increase the accuracy of forecasting metal damage and its residual life.

1. Introduction
The active development of strategically important territories of the Arctic and the Far North necessitates the reliable functioning of technical facilities under extreme climatic conditions, which significantly reduce the operational reliability and resource of mechanisms and structural elements, which can lead to serious accidents and economic consequences.

During operation, structural elements and mechanisms of the Northern version are subjected to various temperature and power influences, many structural elements operate in conditions of low and high cycle fatigue. Moreover, in the process of operation, cases of nucleation and development of a crack are often observed. The process of damage accumulation begins almost immediately after the application of a variable load, proceeds covertly, practically without visible external signs, and can reach up to 80% of the resource of the sample material. Hidden processes of material degradation lead to a change in physical and mechanical characteristics: elastic moduli, strength and other characteristics. The period of damage accumulation ends with the formation of macrocracks and the destruction of the structure. Such a crack can be detected visually or using flaw detection tools. With the functioning of structures and mechanisms at low temperatures, the appearance of microcracks in the material is unacceptable, because the work of crack propagation with decreasing temperature decreases several times [1, 2].

In this regard, the task is to assess the degree of degradation of the material during its operation even before the appearance of macrocracks, and to ensure constant monitoring of the structural state of the material of construction with the use of reliable diagnostic tools. A comparative analysis of modern methods of non-destructive testing shows that the most promising methods for monitoring the structural
state of the material at the stages of operation before macrodefects include physical acoustics, as well as the magnetic method for monitoring and diagnosing the material [3, 4, 5].

In recent years, approaches to nonlinear dynamics and fractal representations developed by V.S. Ivanova, A.G. Kolmakov, V.I. Trefilov, et al. [6–11], which make it possible to quantify the processes of structure degradation based on a quantitative criterion — fractal dimension.

2. Choice of material and design of a stand for tests
Of scientific interest is the study of the mechanisms of deformation and fracture during elastoplastic cyclic deformation of metastable austenitic steels, in particular steel 12Cr18Ni10Ti in connection with the peculiarities of phase transformations during deformation, which significantly affects the physicomechanical characteristics of the material and its resource.

Steel 12Cr18Ni10Ti is widely used for the manufacture of welded structures, vessels and individual parts operating at low temperatures, at high pressures and in aggressive environments.

The chemical composition of steel 12Cr18Ni10Ti is as follows (%): C - 0.11; Si - 0.7; Mn 0.16; Ni - 10.2; S is 0.012; P is 0.008; Cr - 18.16; Ti - 0.6; Fe is the basis.

For fatigue tests, samples were prepared whose geometry is shown in Figure 1 a. For testing, a specialized stand for fatigue testing was developed, the scheme of which is shown in Figure 1 b.

Test conditions: loading according to the cantilever bending diagram (cycle asymmetry coefficient \( R = -1 \)) at temperature \( t = +20 \) °C and \( t = -100 \) °C on a fatigue testing machine (Figure 1, b) taking into account the requirements of GOST 25.502-79. The frequency of the elastoplastic cyclic deformation was set using a frequency converter equal to 8.3 Hz (500 cycles/min).

The stress amplitude \( (\sigma_{\text{max}}) \) was calculated on the basis of the loading scheme, sample dimensions and mechanical characteristics of the material. During the tests, the following were recorded: the number of cycles \( N \) and the amplitude of the stresses in the cycle. The studies were carried out at a stress amplitude \( \sigma_{\text{max}} \) of 305 MPa.

To conduct fatigue tests at low temperatures, a cryochamber was used to ensure uniform cooling of the sample and automatic maintenance of the test temperature due to the flow of chilled air from a bath with liquid nitrogen. The temperature in the working zone of the sample was monitored using pt100 temperature sensors (Figure 1, b).

3. Methods of processing acoustic signals and microstructure images
Preliminary tests were carried out to estimate the number of cycles until sample failure \( (N_\Sigma) \). The study of the structures of materials, registration of the parameters of the ultrasonic signal and magnetic
characteristics was carried out before the start of testing and after operating time with a step of ~ 10% of the limit number of cycles \( N_{\Sigma} \).

The microstructure was studied using a KYENCE-VHX 1000 optical microscope. The polished surface was etched in a 10% oxalic acid solution. To assess the fractal dimension of microstructures \( D_h \), software was developed in the MATLAB environment [6].

The study of material structures, registration of the parameters of the ultrasonic signal and magnetic characteristics was carried out in the zone with the highest stresses, where microcracks were subsequently detected in the material.

For acoustic measurements, the «Astron» measuring and computing acoustic complex (MCC) was used [12]. The device is designed for non-destructive testing of the physicomехanical characteristics of solids by measuring the propagation time of ultrasonic vibrations (ultrasonic testing) and measuring the attenuation coefficient of the received signal. The device allows measuring the propagation time of elastic impulses with an accuracy of \( 10^{-9} \) s. The error in measuring the attenuation coefficient \( (K_{zat}) \) is 5%.

MCC “Astron” is able to work both in separate and in a separately combined mode with various types of elastic waves. A longitudinal wave sensor was used in the work. The nominal frequency of the sensors is 5 MHz. Glycerin was used as a contact liquid, the viscosity level of which at low temperatures retains its efficiency.

We used pulsed excitation and reception of elastic pulses. As a reference pulse, the first reflected pulse was used, relative to which the velocity and attenuation were measured by comparing its characteristics with the characteristics of subsequent pulses. This allows one to take into account only the effects associated with the path of an elastic impulse in a controlled medium [12–15].

The speed of the ultrasonic wave \( C \) when passing through the test sample was determined by the following formula:

\[
C = \frac{2L}{t},
\]

where \( L \) is the thickness of the sample, m; \( t \) is the transit time of the corresponding type of wave through the sample, s. The speed measurement error is ± 2 m / s.

The attenuation coefficient \( K_{zat} \) was determined by comparing the amplitude range of the second reflected pulse compared to the first reference:

\[
K_{zat} = \frac{1}{L} \cdot \frac{20 \log A_2}{A_1},
\]

where \( A_2 \) is the amplitude of the second reflected pulse, \( A_1 \) is the amplitude of the first reflected pulse.

As an acoustic characteristic, materials taking into account the change in the propagation velocity of ultrasonic waves \( (C_1 \ and \ C_2 - \text{transverse with longitudinal and transverse polarization and } C_3 - \text{longitudinal, relative to the sample}) \) used the dimensionless parameter \( D \), determined by the formula:

\[
D = \frac{C_1 + C_2}{C_3}.
\]

Damage (W) of the material was determined on the basis of acoustic measurement data according to the technique shown in [16].

To evaluate the magnetic characteristics, a magnetic metal analyzer/coercimeter MA-412MM was used. The following magnetic characteristics were evaluated: residual magnetization \( B_r \), coercive force \( H_c \), maximum induction \( B_s \), ratio \( H_c/B_r \).

At each stage, at least 5 measurements, both acoustic and magnetic characteristics, were carried out, followed by statistical processing of the obtained data.

The results obtained at different stages of testing were recorded with a special database of test parameters (DB) and were used later as initial data for training an artificial neural network.
4. Experimental research results
The microstructures of the material under study at different stages of production and with different damage are shown in Figure 2 at test temperatures \( t = +20\,^\circ\text{C} \) and \( t = -100\,^\circ\text{C} \).

![Figure 2](image)

**Figure 2.** Microstructures (x300) at different stages of operating time and with different damage \( W \).

Based on the analysis of microstructures with different operating time and damage \( W \) and the estimation of fractal dimension \( D_f \) from the images of microstructures, the dependences of the fractal dimension of the microstructure of the working area of the sample \( D_f \) on the damage of the metal \( W \) at temperatures \( t = +20\,^\circ\text{C} \) and \( t = -100\,^\circ\text{C} \) (Figure 3).

![Figure 3](image)

**Figure 3.** Dependence of the fractal dimension of the microstructure \( D_f \) on damage \( W \) for steel 12Cr18Ni10Ti for test temperatures.

An analysis of the obtained dependences allows us to talk about the relationship between the fractal dimension of the microstructure in the working zone of the sample and the value of the metal damage. At a low temperature \( t = -100\,^\circ\text{C} \), the nature of the dependence in question does not change, however, the values of the fractal dimension of the microstructure are slightly higher. As studies have shown, the values of the fractal dimension of the structure during the test also depend on the initial structure of the material.

When studying the features of changes in the parameters of ultrasonic waves (surface, longitudinal and transverse), it was found that the most informative is the pattern of change in the acoustic parameter \( D \). The obtained dependences of the acoustic parameter \( D \) on the damage to the material \( W \) for temperatures \( t = +20\,^\circ\text{C} \) and \( t = -100\,^\circ\text{C} \) are shown in Figure 4.

As can be seen from figure 4, the obtained dependence of the acoustic parameter \( D \) on the damage value, along with the index of the fractal dimension of the microstructure (Figure 3), has a definite relationship with the damage values of the sample material. On the obtained dependences, 3 characteristic stages can be distinguished, however, with a decrease in temperature, the character of the dependence changes. So, at a temperature of \( t = +20\,^\circ\text{C} \), an increase in the parameter \( D \) is observed, while at \( t = -100\,^\circ\text{C} \) its decrease is observed during the accumulation of damage to the material \( W \).
Figure 4. The dependence of the acoustic parameter $D$ on the value material damage $W$ for temperatures $t = +20^\circ C$ (a) and $t = -100^\circ C$ (b).

The results obtained indicate the need to take into account, along with parameter $D$, other acoustic parameters (velocity and attenuation coefficient of waves in the material) to determine the stage of destruction of the material and its damage taking into account the test temperature.

In figure 5 shows the nature of the change in the magnetic characteristics of 12Cr18Ni10Ti steel during fatigue tests at different test temperatures.

Figure 5. Dependences of the magnetic characteristics of 12Cr18Ni10Ti steel on material damage $W$ at temperatures: a) $t = +20^\circ C$ and b) $t = -100^\circ C$.

An analysis of the magnetic characteristics indicates their mutual correlation, and the $H_c/Br$ ratio, as well as the coercive force $H_c$ for which the most intense change is observed in the entire temperature range under consideration, can be taken as the most informative. At low temperatures, there is such a feature as an intensive growth of all magnetic characteristics in the initial stages of the test, which also indicates the intensity of phase transformations in the material. At a temperature of $t = +20^\circ C$, the most intense increase in magnetic characteristics is observed at the stage of prefracture of the sample.

Based on numerous informative parameters (Figures 3–5) that characterize the damage to the material during loading, it is necessary to use tools to assess the damage to the material on the basis of the whole complex of heterogeneous diagnostic information. Taking into account all the identified indicators in the complex will make it possible to identify the stage of degradation and destruction of the material and its residual life.
5. Conclusion
1. In the course of experimental studies, the dependence of the fractal dimension of the metal microstructure (DF) on the parameter of its damage is shown. The nature of the dependence is preserved over the entire temperature range under consideration, namely, a monotonic increase in the fractal dimension of the microstructure during the accumulation of metal damage is observed.
2. To predict the damage to the metal, the acoustic parameter $D$ can be used, which reflects the change in the propagation velocity of elastic waves in the metal. However, in connection with the peculiarities of the change in the considered parameter at room and low temperatures (the nature of the dependence changes), it is additionally necessary to take into account the change in all parameters of the elastic waves in aggregate to increase the accuracy of predicting metal damage.
3. An analysis of the magnetic characteristics showed that the $H_c/Br$ ratio, as well as the coercive force $H_c$, for which the most intense change is observed in the entire temperature range under consideration, can be taken as the most informative. At low temperatures, there is an intensive growth of all magnetic characteristics at the initial stages of the test, which also indicates the intensity of phase transformations in the material, since at room temperature the change in magnetic characteristics is less intense.
4. Based on numerous informative parameters characterizing the damage to the material during fatigue loading, it is necessary to use tools to assess the damage to the material on the basis of the whole complex of heterogeneous diagnostic information in order to increase the accuracy of the stage of degradation and destruction of the material and its residual life.

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