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Application of Ultrasonic Spectral Analysis for the Study of Steels

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Abstract: Ensuring high quality, specific structures, and defined mechanical properties of materials requires the widespread application of non-destructive methods for control and investigation of the structural elements of materials, in particular ultrasonic methods. The subject of this study is samples of low-carbon steel, in which equilibrium ferrite-pearlite structures with different grain sizes have been obtained through targeted heat treatments. Through quantitative metallographic analysis, the average grain size, the percentages of ferrite and pearlite, and the grain size distribution have been determined. The aim of this study is to investigate the structural elements of carbon steels using an ultrasonic method. Ultrasonic spectral analysis is employed, which is based on the changes in the frequency spectrum of ultrasonic waves after their interaction with the material. The way ultrasonic waves scatter and attenuate in a polycrystalline material provides information about the size, shape, and distribution of the structural elements that make up the material. This work analyzes the influence of grain size distribution and the quantitative assessment of different phases on the attenuation of ultrasonic wave

Keywords: Low carbon steel, Grain size distribution, Attenuation of ultrasonic waves, Ultrasonic spectral analysis

Introduction

Ultrasonic methods have shown good potential for studying the microstructure of polycrystalline materials. The propagation of ultrasonic waves in polycrystalline materials has been the subject of research in the past. Research in this area began with the work of Rayleigh, further developed by a number of researchers. Mason and McSkimmin (1947) indicated that differences in acoustic impedance at crystallite boundaries are a major source of ultrasonic scattering and attenuation in materials. Attenuation is determined by the energy losses of the ultrasonic waves, which include absorption and scattering contributions. Hirsekorn (1982), Stanke and Kino (1984), and Weaver (1990) developed theoretical models that relate ultrasonic attenuation to the microstructural features of polycrystalline materials, valid for all grain size to wavelength ratios and for polycrystalline materials with cubic symmetry. Attenuation models for grains of cubic symmetry were improved by Lifshits and Parkhomovski (1956) and Merkulov (1956).

A major contribution was made by Papadakis (1964, 1968) with several published papers on the subject. The author classifies scattering into three classical regions depending on the relationship between grain size (D) and wavelength (λ). For each region, the microstructure is assumed to be composed of spherical grains of uniform size filling the medium. A study by Smith (1982) shows the influence of grain size distribution on ultrasonic attenuation. Nicoletti and Anderson (1997) presented a deconvolution method to study the inverse problem of finding the grain size distribution from ultrasonic attenuation.

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In recent years, there has been significant progress in studies on the influence of crystallite distribution on the attenuation of ultrasonic waves. Arguelles and Turner (2017) investigated how a distribution of grain sizes in polycrystalline materials affects ultrasonic attenuation. They found that the frequency dependence of ultrasonic attenuation varies with the width of the grain size distribution, even when the average grain size remains constant. This means that neglecting the grain size distribution can lead to inaccurate estimations of grain size based solely on attenuation measurements.

Norouzian & Turner (2020) analyze 3D synthetic microstructures with lognormal grain distribution and demonstrate that the width of the distribution affects the frequency dependence of the attenuation, applying microstructure generators such as DREAM.3D. It can be concluded that the average crystallite size is not a sufficient parameter and dispersion should be taken into account. Studying steels S355, S275, Markja et al., 2021 found that grain size significantly affects the propagation and attenuation of ultrasonic waves: smaller grains generally increase the attenuation of ultrasound and are associated with higher strength, while larger grains reduce attenuation but are associated with lower strength.

In the paper Bonifazi et al., (2018), the effect of the grain size and other microstructural parameters on the ultrasonic propagation have been evaluated for an experimental carbon steel. The main results obtained from the study are that at high frequencies the attenuation coefficient follows Rayleigh's law of scattering and shows a maximum at a frequency value that increases with decreasing grain size; the frequency value corresponding to the attenuation maximum, converted to wavelength, can be compared to approximately 3 - 4 times the average grain size. Shan et al. (2018) proposed a new unified model of ultrasonic scattering corrected for lognormal grain distribution. Using the theory, the effects of the grain distribution widening on the ultrasonic scattering are analyzed for the longitudinal wave and the shear wave.

Measuring attenuation coefficients at different frequencies with a variety of ultrasonic sensors and devices leads to accumulation of errors due to non-uniformity of conditions: differences in contact layers, in the efforts to establish contact, diffraction losses, etc. Another approach to studying the structure of materials is ultrasonic spectral analysis, which is based on the change in the spectrum of pulse signals used in ultrasonic measurement practice. Ultrasonic spectral analysis is a non-destructive method used to evaluate the grain structure of polycrystalline materials by analyzing the frequency spectrum of ultrasonic waves (Adler, 1993). The way in which ultrasonic waves are scattered in a polycrystalline material provides information about the microstructure’s elements. Pulse-echo or transmission techniques using direct contact or immersion modes have been used (Buckin et al., 2002).

Ultrasonic spectral analysis was performed on samples of AISI type 316 stainless steel and modified ferritic steel 9Cr–1Mo (Kumar et al., 2000). Spectral analysis was performed on the RF signal corresponding to the first echo from the back wall obtained with a 25 MHz longitudinal beam transducer. The peak frequency shift and the change in the full width at half maximum of the auto power spectrum were correlated with the average grain size and the yield strength.

Stella et al. (2009) used spectral analysis and conventional ultrasonic techniques to characterize the degree of sensitization in AISI 304 stainless steel. Sensitization, which is a heating process by which carbides are formed, increases the attenuation of ultrasonic waves due to the deposition of carbides at grain boundaries. The authors found that the combination of attenuation and spectral analysis is suitable for assessing the degree of sensitization. Artificial aging of welded joints of API 5L X52 steel pipeline was investigated using ultrasonic spectral analysis (Vargas-Arista et al., 2009). It was found that the attenuation of ultrasonic waves is a more sensitive parameter for identifying the change in microstructure due to aging.

Improved ultrasonic spectroscopy methods (Wang and Cao, 2001) for characterization of dispersed materials aim to increase accuracy and reduce sources of error. The technique involves analyzing spectral characteristics of reflected and transmitted ultrasonic waves to determine phase velocity and attenuation, which are frequency dependent in dispersive media. The research of Peters and Petit (2003) described a broad band spectroscopy method for ultrasound wave velocity and attenuation measurement in dispersive media. This allowed measurements over a wide frequency spectrum in highly absorbing and highly dispersive media.

The purpose of the current research is to investigate the microstructural elements of low carbon steels by ultrasonic spectral analysis. Methods for evaluating the attenuation of ultrasonic waves in polycrystalline structures are compared in order to find the best approach for the study.

Experimentals

Materials

This section describes the details of the test samples, ultrasonic measurement method and measurement procedure. The chemical composition of the studied steel grade is: C - 0.17-0.2%, Si - 0.17-0.37%, Mn - 0.35 - 0.65%, P << 0.035%, S << 0.035%. The low carbon unalloyed steel samples were heat treated at temperatures T_a from 800°C to 1150°C and slowly cooled (100°C/hour) to obtain ferritic-pearlitic structures.

By varying the austenitization temperature (T_a) and the holding time at this temperature, microstructures with different crystallite sizes were obtained. Metallographic studies were carried out using an optical microscope with reflected light with the requirements of regulatory documents.The microstructure was revealed with reagent Nital - 3% solution of nitric acid in ethyl alcohol. A 5MP digital microscope eyepiece camera type Hayear was installed on the microscope. An object-micrometer sample OMP -GOST 7513 -75 was used for calibration. The type and features of the microstructure, mean grain size of the ferrite ($\overline{D}_f$) and pearlite ($\overline{D}_p$) and the amount of pearlite were determined by quantitative metallographic analysis. Table 1 presents the heat treatment and the results of metallographic analysis.

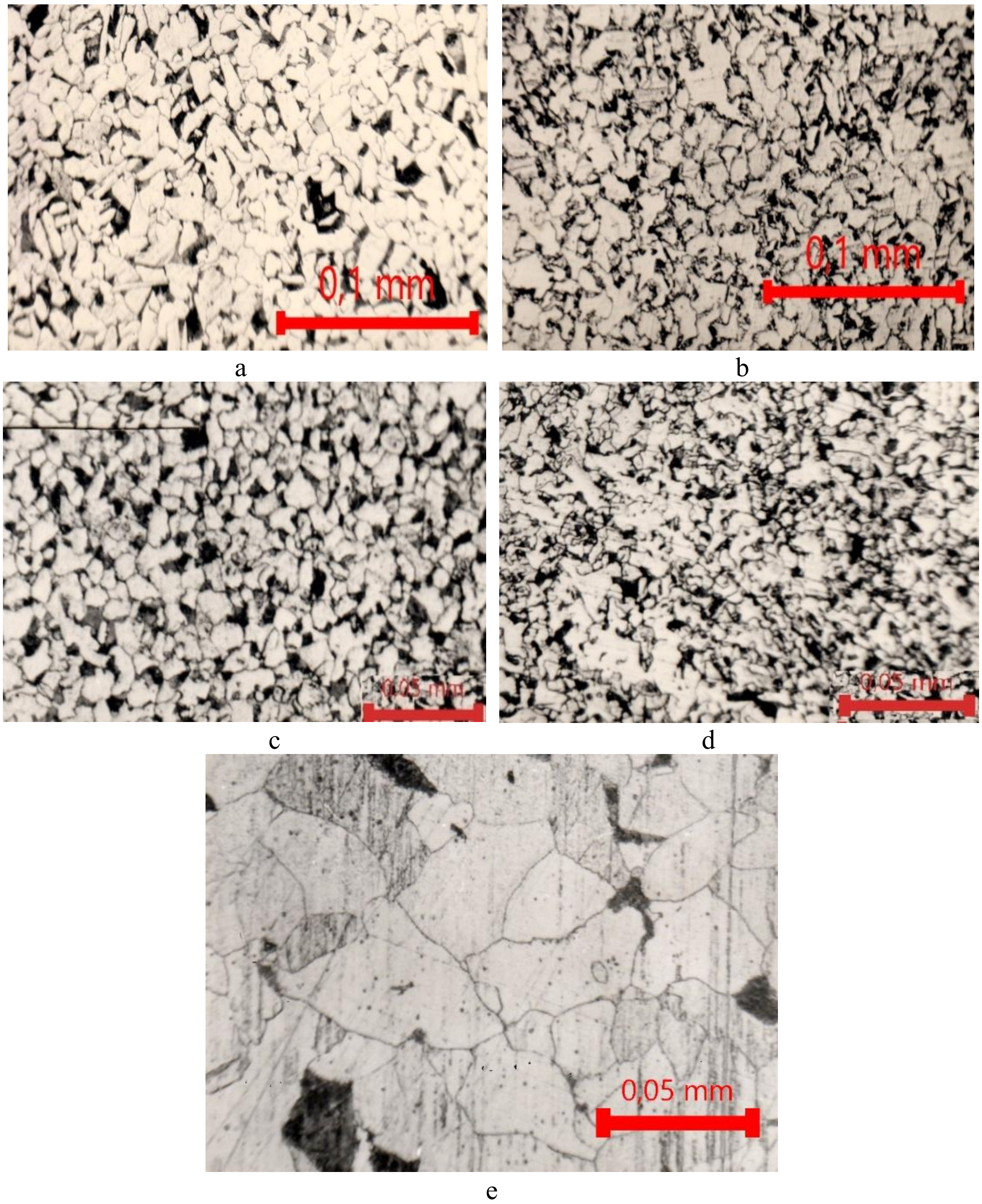


Figure 1. Metallographic photographs of microstructures - samples №0 (a), №1 (b), №2 (c), №3(d), №4 (e)

Figure 1 shows metallographic photographs of some of the investigated microstructures. The microstructure of the sample in the initial state is shown in Figure 1a. It is relatively non-uniform, with the crystals having an irregular shape acquired during the initial technological processing. After heating at temperatures in the austenitic region of 850-900 °C and cooling, an equiaxed ferrite-pearlite structure was obtained – Figures 1 b and c. Figure 1d shows an uneven grain structure obtained after heating at 950 °C. Areas with small grains, as well as areas with agglomerated structure, can be observed. Therefore, the results of this group will be discussed in detail in future works. After prolonged heating at 1150 ° C the structure consists of large ferrite and pearlite crystals - Figure 1 e.

Table 1. Heat treatment of steel samples

Code	Heat treatment	Ta,ºC	t, h	$\overline{D}_f$, mm	$\overline{D}_p$, mm	Pearlite %
0	Initial state	-	-	0,019	0,001	8
1	Annealing	850	2	0.018	0.0098	8
2	Annealing	900	2	0.0010	0.0075	5.5
3	Annealing	950	2	irregular	grain	structure
4	Ageing	1150	10	0.0485	0.013	7

Method

Ultrasonic Spectral Analysis

The method for studying materials by ultrasonic spectral analysis is based on the analysis of the spectrum of ultrasonic signals that propagate in materials with a specific micro and macrostructure (American Society for Nondestructive Testing. Ultrasonic testing, 1991), (Schmerr, 2016). When an ultrasonic pulse passes through materials with different structures, the shape of the signal changes, which change contains information about the structure. The signal P(t) and its spectrum S(f) at each point of the medium are related by Fourier transformation, using the dependencies (Smith, 1997), (Skuchik, 1976):

$$P(t) = \int_{-\infty}^{\infty} S(f) e^{i2\pi f t} df \quad (1)$$

$$S(f) = \int_{-\infty}^{\infty} P(t) e^{-i2\pi f t} dt, \quad (2)$$

where *S(f)* is the spectrum of the signal *P(t)*, *f* - current frequency, *t* - time.

Methodology

The electroacoustic path in ultrasonic material testing consists of a radio pulse generator, to which is connected a radiating piezoelectric system, a contact layer (between the sensor and the medium), a receiving piezoelectric system, a broadband amplifier and an analyzer of the spectrum of the transmitted signal. The conversion of the electrical signal into an ultrasonic signal is described by the expression:

$$S(f)_{x=0} = S_g(f) S_T(f) \quad (3)$$

Where $S(f)_{x=0}$ is the spectrum of the ultrasonic signal entering the controlled material, $S_g(f)$ is the spectrum of the generating signal and $S_T(f)$ is the spectrum of the transmitting acoustic system. The spectrum of the received transmitted or reflected signal is recorded,

$$S \;\; (x, f) \;\; = S_g(f) S_T(f) S_R(f) S_m(x, f) = S_a(f) S_m(x, f) \quad (4)$$

Where $S_R(f)$ is the spectrum of the receiving acoustic system, $S_a(f) = S_g(f) S_T(f) S_R(f)$ is the spectral characteristic of all the acoustic system.

$$S_m(x, f) = Ce^{-\alpha \;\; (f)x} \quad (4a)$$

The expression (4a) represents the attenuation $\alpha(f)$ in the material, which can be considered as a filter with certain frequency properties, where *x* is the acoustic path.

Equipment

Experimental measurements were obtained by contact method with pulse echo technique. The apparatus consists of a computerized ultrasonic apparatus UT expert, which includes a generator and receiver of ultrasonic signals integrated in a computer. A piezoelectric transducer with an operating frequency of 15 MHz is used, which is an emitter and receiver of ultrasonic signals. Silicone gel was used as a contact medium.

The apparatus allows measuring the propagation time of ultrasonic waves and recording echo signals with the possibility of additional signal processing. To achieve higher accuracy in measurements, constant conditions for generating acoustic signals, uniform contact fluid and equal pressure force on the sensor, etc. are maintained. In this method, the two consecutive echo signals from the back wall, measured on a sample of known thickness, are employed to determine the ultrasonic parameters - velocity and attenuation. The transit time of ultrasonic waves is measured with an accuracy of the order of 10 ns.

Experimentation and Results

To study the attenuation of materials in this work, two procedures were used:

I. Comparison of the spectral characteristics of the tested polycrystalline material and a reference sample.
II. Determination of the attenuation of the tested materials from the spectra of successive echo signals

Comparison of the Spectral Characteristics of the Tested Polycrystalline Material and a Reference Sample

In the first approach, a fine-grained material of pure iron with an attenuation coefficient is used as a reference.

$$S_t(x,f) = S_a(f)Ce^{-\alpha_t(f)x} \quad (5)$$
$$S_{ref}(x,f) = S_a(f)C_1e^{-\alpha_{ref}(f)x} \quad (6)$$

where S_t and S_{ref} are the spectrum characteristic of the tested and reference sample.
The attenuation coefficients of the test and reference samples are denoted by $\alpha_t(f)$ and $\alpha_{ref}(f)$, respectively. The ratio of the spectra (7) depends on the attenuation coefficients and the acoustic path x.

$$\frac{S_t(f)}{S_{ref}(f)} = \frac{C}{C_1}e^{(\alpha_{ref}-\alpha_t)x} \quad (7)$$

After taking the logarithm of (7) can be obtained

$$\ln\frac{S_t}{S_{ref}} = (\alpha_{ref} - \alpha_t)x + K \quad (8)$$

Where $K=\ln(C/C_1)$ is the term corresponding to the scaling factor.

$$\alpha_t(f) = \alpha_{ref}(f) - \frac{1}{x}\left(\ln\frac{S_t}{S_{ref}} - K\right) \quad (9)$$

Determination of the Attenuation of Tested Materials From Spectra of Successive Echo Signals

In the second procedure, the attenuation of ultrasonic waves is determined based on data from measurements of the spectra of the first (10) and second (11) bottom echoes from the controlled sample:

$$S_L(L,f) = S_a(f)e^{-\alpha(f)L} \quad (10)$$

$$S_{2L}(2L,f) = S_a(f)e^{-\alpha(f)2L} \quad (11)$$

The ratio of the spectra is (12)

$$\frac{S_L(L,f)}{S_{2L}(2L,f)} = \frac{e^{-\alpha(f)L}}{e^{-\alpha(f)2L}} = e^{\alpha(f)L} \qquad (12)$$

After conversion, we obtain

$$\alpha(f) = \frac{1}{L}\ln\frac{S_L}{S_{2L}} \qquad (14)$$

The dependence of the attenuation of ultrasonic waves on the frequency according to (Truell, 2013) can be represented as the sum of absorption and attenuation:

$$\alpha(f) = \sum_i \ \alpha_{abs,i}.f^{m,i} + \sum_k \ \alpha_{scat,k}f^{n,k} \qquad (15)$$

where $\alpha_{abs,i}$ and $\alpha_{scat,k}$ are coefficients taking into account the losses from absorption and scattering, m,i and n,k are the power coefficients determining the frequency dependences in the absorption and scattering of ultrasonic waves in the material.

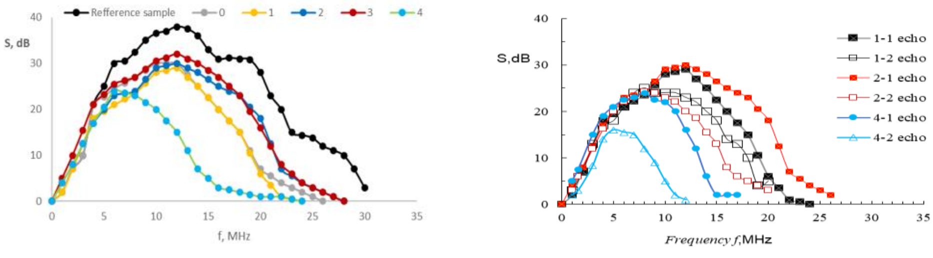
In the frequency range up to 12 MHz, the attenuation coefficient of ultrasonic waves is the sum of absorption and Rayleigh scattering (Papadakis, 1968) and is written

$$\alpha(f) = \alpha_1 f + \alpha_4 f^4 = \alpha_1 f + C_R\bar{D}^3 f^4 \qquad (16)$$

Where C_R is a structural coefficient depending on the elastic properties of the materials, $\bar{D}$ is the average grain diameter. The division into regions in (16) depends on the relation (D/λ), λ- wavelength. For the studied materials, the relation for Rayleigh-type attenuation D/λ <0.2 is fulfilled.

Experimental Results

Experiments were performed and ultrasonic signals passing through the studied samples were recorded. After Fourier transformation, the spectra of ultrasonic signals were obtained. Figures 2a and b, respectively, show the spectra of the ultrasonic signals by the two procedures. The following notations are used in Figure 2a: spectrum of a reference sample, from 0 to 4 are the spectra of the studied samples. With increasing grain size, changes are observed in the spectral characteristics of the ultrasonic signals, expressed in a depletion of the spectrum at high frequencies and a shift of the maximum value of the spectrum to lower frequencies. Figure 2b illustrates some of the results obtained by the second procedure. Spectra of two consecutive echoes of samples with the most distinct average crystallite size are shown: №1 ($\bar{D}_f = 18\ \mu m$) , №2 ($\bar{D}_f = 10\ \mu m$) and №4 ($\bar{D}_f = 48\ \mu m$).



a b

Figure 2. Spectra of ultrasonic signals: a) spectral characteristics of the tested materials and a reference sample; b) spectra of successive echo signals

Figure 3 show the dependences of the attenuation coefficients on the frequency, determined by procedures I and II, as well as the approximated dependences by the least square's method for samples with different average grain diameters. The differences between the attenuation values by the two procedures are small for fine-grained steels and more significant for coarse-grained steels at frequencies greater than 10 MHz. Figure 3d illustrates the variations of the averaged values of attenuation coefficients for samples № 1, № 2, and № 4. Greater attenuation is observed for samples with larger grains, which is in accordance with many authors (Merkulov, 1956), (Bonifazi et al., 2018).

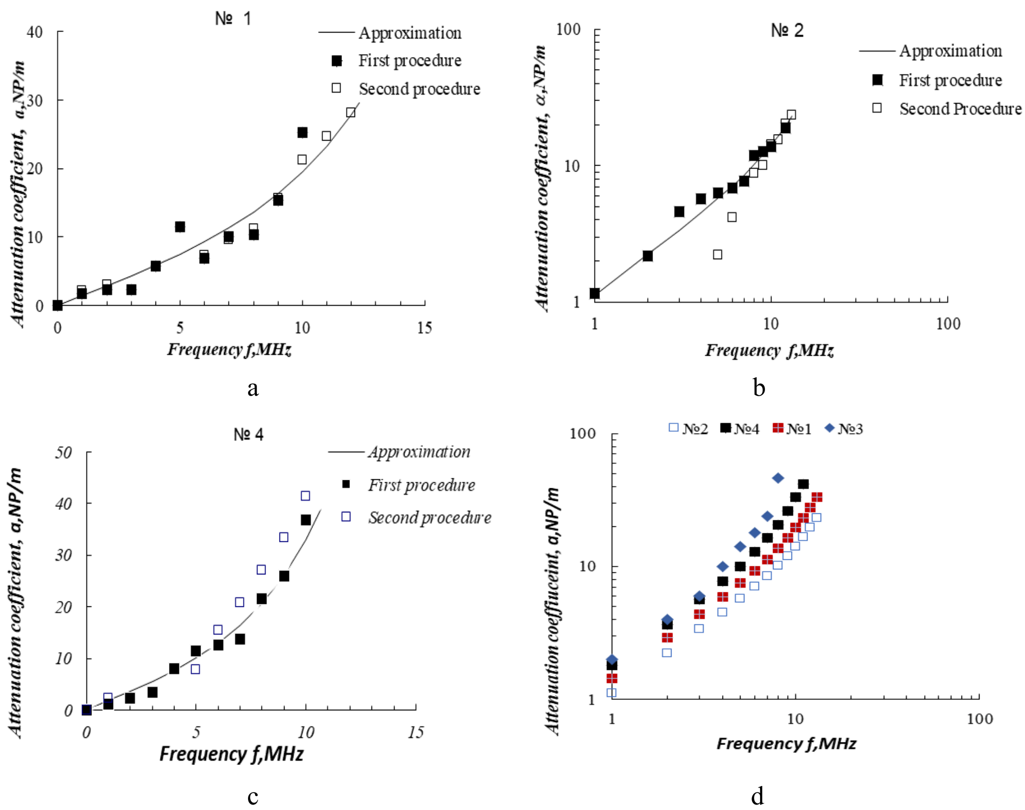


Figure 3. Attenuation coefficients according to procedure I and II for samples №1 (a), №2 (b), № 4 (c) and average attenuation coefficients (d)

Table 2 presents the coefficients that determine the fraction of absorption (α_1) and scattering (α_4) for the studied samples, determined by the least squares' method.

Table 2. Absorption and scattering coefficients

	First procedure		Second Procedure		
	α_1, Np/m.MHz	α_4, Np/m^4.MHz^4	α_1, Np/m.MHz	α_4, Np/m.MHz^4	C_R Np/m^4
1	1,2	2,2.10^{-4}	1,35	1,5.10^{-4}	1,8-2,1.10^{11}
2	1,24	1,9. 10^{-3}	1,14	2,197.10^{-4}	3,5-4,204.10^{11}
3	1,09	1,17.10^{-3}	1,35	5,23. 10^{-4}	4,3-4,677.10^{11}
4	1,5	2,2.10^{-3}	2,4	1,1.10^{-4}	1,0-1,3.10^{10}

Modeling of Attenuation of Ultrasonic Waves

Modeling of Attenuation of Ultrasonic Waves in a Single-Phase Polycrystalline Material

The attenuation coefficient of ultrasonic waves when taking into account the size distribution of crystallites (Nicoletti, 1992) is expressed by:

$$\alpha(f) = \int_0^{\infty} \beta(f, D) F_f(D) dD \tag{17}$$

Where $F_f(D)dD$ is the number of ferrite grains, $\beta(f, D)$ is function that describes the attenuation of ultrasonic waves in the entire range of changes in the grain diameter D and frequency *f*.

In this work, the attenuation of ultrasonic waves is modeled using an auxiliary function that combines experimentally obtained data in different ranges of variation of the parameter D/λ. The proposed model follows the theory of ultrasonic wave scatter and assumes a weak material anisotropy. The function $\beta(f, D)$ represents the kernel of the transformation (17) and has the following form:

$$\beta(f, D) = C_R D^3 f^4 g_1\left(\frac{D,f}{c_L}\right) + C_s D \quad f^2 g_2\left(\frac{D,f}{c_L}\right) + C_d g_3\left(\frac{D,f}{c_L}\right) \tag{18}$$

Where $g_1(x)$, $g_2(x)$, $g_3(x)$ are functions, that are equal to 1 within Rayleigh's, stochastic and diffusion ranges, respectively, and are zero out of them, D is the diameter of the crystallites, c_L is the velocity of the longitudinal ultrasonic wave. The division into regions depending on is based on literature data (Papadakis,) for the Rayleigh region ($D/\lambda<0,2$), for stochastic scattering ($0,2<D/\lambda<2,5$) and diffuse scattering ($2,5< D/\lambda$). The modeling uses the coefficients obtained from the real experiment (Table 2).

Modeling of Attenuation of Ultrasonic Waves in a Two-Phase Polycrystalline Material

The attenuation of ultrasonic waves in a two-phase polycrystalline material such as investigated low-carbon steel is modeled taking into account the influence of two types of scatterers (ferrite and pearlite grains) and the pearlite content. In this case, the attenuation coefficient is represented in the following form

$$\alpha(f) = \alpha_f(f) + \alpha_p(f) \tag{19}$$

The grain distribution function in the two-phase polycrystalline material can be expressed by

$$F(D) = qF_1(D) + (1-q)F_2(D) \tag{20}$$

Where q is the percentage content of one phase, and $F_1(D)$ and $F_2(D)$ are the distributions of ferrite and pearlite grains, respectively.

The grain distributions of the studied samples, obtained by quantitative metallography methods, are shown in Figure 4 for the samples №1, №2, №4.

According to literature data (Gür, 2005), (Wiskel, 2015), (de Araújo, 2009), the differences in the values of longitudinal wave velocities in pearlite and ferrite in low-carbon steel are small, and the percentage content of pearlite is up to 8%. The attenuation coefficient is given by

$$\alpha(f) = \int_0^\infty \beta(f, D) F \quad (D) dD \tag{21}$$

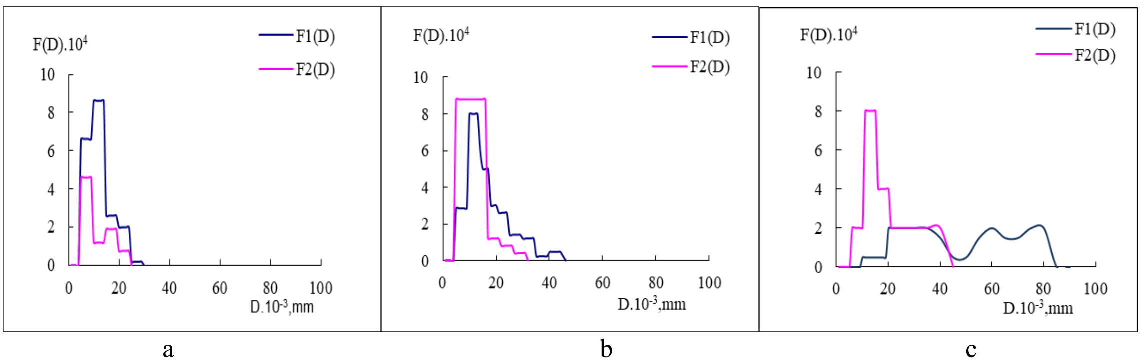


Figure 4. Distributions of ferrite $F_1(D)$ and pearlite $F_2(D)$ grains of samples №1, №2, №4

Figure 5 shows the results of calculations of the attenuation coefficients, considering the distribution of only ferrite crystals by (17) and that of two phases (ferrite and pearlite grains) in conformity with (21). When using the model that considers the distributions of the existing phases, the results for the attenuation are changed by 4-5%. A larger amount of pearlite and scattering particles will probably lead to a significant change in the results.

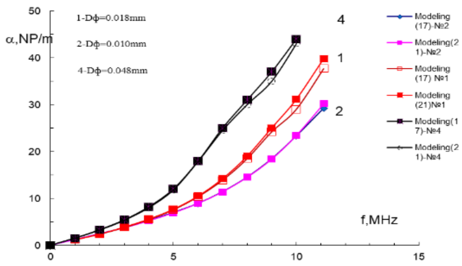


Figure 5. Attenuation coefficients taking into account the distribution of ferrite crystals according to (17) and the distribution of the two phases (21)

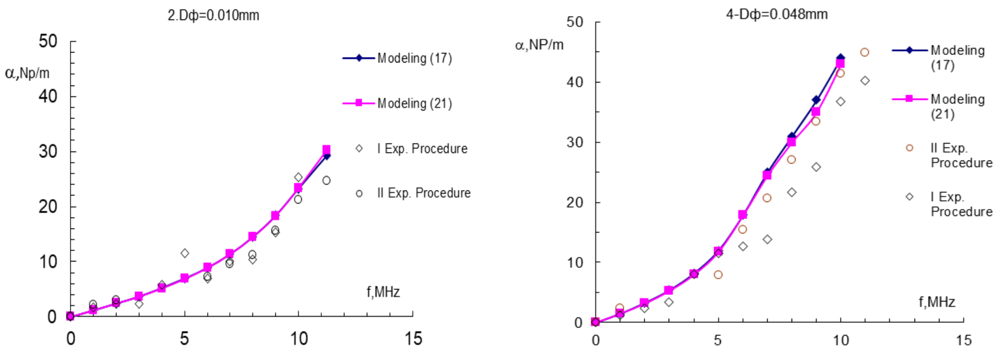


Figure 6. Comparison of experiments using procedures I and II with modeling for samples 2,4.

Figure 6 compares the attenuation coefficients from the calculations and experiments only for some of the samples with the most different grain sizes No. 2 and 4. The least squares method can be used to quantify the differences between the modeling results and the experiments. The smallest differences are for sample No. 2, with an average ferrite grain size of 10 µm, and the largest are for the coarse-grained structure No. 4. Qualitative correspondence of the experiments with the calculations is observed. The model allows analysis of the grain size distribution using the spectrum of the ultrasonic signal.

Conclusion

The work uses ultrasonic spectral analysis to determine the dependences of attenuation coefficients on the frequency for low-carbon steels. For the fine-grained structures, the procedure using a reference sample is more suitable. The coefficients considering the absorption and scattering of ultrasonic waves in the frequency range up to 12 MHz are found. A model, which takes into account the actually obtained distributions of ferrite and pearlite grains, is employed to estimate the attenuation. Qualitative agreement between experiments and calculations is found. The discrepancy between the them is probably due to the inaccuracy of the model in the frequency range used in the study.

Recommendations

The presented research can provide new knowledge to students and engineers to put into practice ultrasonic spectral analyses for non-destructive evaluation of materials.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest.

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