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COMPLEX ESTIMATION OF STRENGTH PROPERTIES OF FUNCTIONAL MATERIALS ON THE BASIS OF THE ANALYSIS OF GRAIN-PHASE STRUCTURE PARAMETERS

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The technique allows analysis using grain-phase structure of the functional material to evaluate its performance, particularly strength properties. The technique is based on the use of linguistic variable in the process of comprehensive evaluation. An example of estimating the strength properties of steel reinforcement, subject to special heat treatment to obtain the desired grain-phase structure.

Keywords: grain-phase structure, microsection, mechanical properties, the theory of fuzzy sets, material hardness, toughness.

Introduction. Recent years have seen a sharp increase of the role of so-called functional materials [1] notable for their preset combination of performance characteristics (strength, plasticity, heat resistance, etc.), which are achieved through application of novel production technologies and/or special processes of subsequent physical-mechanical treatment of materials. However, this calls for the development of new tools for automatic analysis of complex grain-phase structures at the meso- or microscale responsible for providing a require combination of properties at the macroscale [2].

Nowadays, the task of analyzing microstructure of materials by using microsection images, micrographs taken through atomic-force microscopy, scanning probe microscopes, X-ray diffraction topography, or other techniques, is very important for it would result in further development of automatic inspection methods, thus facilitating the design of new promising processes for producing functional materials and methods for predicting their performance [3].

The objective of this work is to elaborate an effective methodology of complex estimation whereby one would be able to assess final strength properties of materials without using any material model or, for example, any empirical relations similar to the Hall–Petch law [4, 5]. The proposed methodology in itself does imply setting an certain approximation function or providing a way of approaching the result in the form of, say, the least-squares technique, but it permits assessing the sought-for quantities through deriving fuzzy relations between the grain-phase structure parameters and performance characteristics of a material.

Problem Statement for Complex Estimation. Assume that there is a digital microsection photograph of a functional material to be studied. Analyzing this micrograph one can distinguish the main parameters of the grain-phase structure, including the phase state parameters, such as volume concentration of phases, and the grain structure parameters, such as mean grain size, grain size variation coefficient, grain anisotropy, volume fraction of grains, and others. The number of selected grain-phase structure parameters is denoted by a variable k.

It is required to assess, based on the analysis of the grain-phase structure parameters of a functional material, the performance characteristics of this material, in particular its strength properties. The number of parameters that represent the required set of performance properties is assumed to be equal to r. Clearly, each set depends on some initially unknown group of parameters of the grain-phase structure of the material. Therefore, it is necessary to elaborate a general procedure of how to derive the sought-for relations using a limited data array. This in turn requires the development of an automated system for analyzing the grain-phase structure parameters from a microsection image, and a methodology of complex estimation of performance characteristics of a material under study.

Analysis of Grain-Phase Structure Parameters. Within the framework of tackling the problem of microstructure analysis, the image segmentation methods are advanced. Segmentation is an important step in the process of the micrograph analysis, for it permits finding grain boundaries, identifying grains as objects for further analysis. Recently, a rapid progress has been observed regarding nonlinear methods of analysis of the image brightness function gradients, similar to the methods proposed by Lee or Previtt [6, 7], continuous segmentation techniques [8], threshold clustering methods [9–11], ridge segmentation methods [12–14], and fractal methods [15].

When solving the problems of quantitative microstructure analysis (quantitative metallography for steels), researchers actively use various parameters of the grain structure, such as: grain area, boundary perimeter, lengths of the grain major and minor axes (average and dispersion), roundness, elongation, compactness, etc. Automation of image segmentation enables one to obtain these parameters directly, i.e., without using the conventional manual processing methods, e.g., the linear intercept method.

In addition, thermomechanical properties of a material essentially depend not only on the grain structure parameters, but also on the phase composition and the presence of dislocation substructures inside a grain. An intensive plastic deformation can give rise to a steady fragmented substructure stabilized by secondary carbide particles [16]. The influence of grain boundaries and triple junctions should be also kept in mind [4]. Note that automation of revealing these features in micrographs would essentially increase the accuracy of prediction of material properties.

As it follows from the aforesaid, in recent times the quantitative metallography has widely accepted the intelligent technologies which are based on the use of computer-aided pattern recognition methods and provide support to an expert for decision making during his analysis of complex microstructures. Within this work, for the purpose of solving the problems of phase classification and grain segmentation we employed the automated system [17] that permits computing, to a specified accuracy, the grain-phase structure parameters from a digital photograph of a microsection.

Methodology of Complex Estimation of Performance Parameters of a Functional Material. To achieve the objective as stated above, let us define fuzzy relations S^i between the grain-phase structure parameters and the performance characteristics of of a functional material [18–20].

The number of available experiments (the microsections and the respective measurements of performance characteristics) is assumed to be l. Then,

$$S^i = A^i \times B^i, \qquad i = 1, \ldots, l, \tag{1}$$

where A^i is the fuzzy set containing the grain-phase structure parameters for the ith experiment, B^i is the fuzzy set consisting of the performance characteristics for the ith experiment, and $\times$ is the sign of the Cartesian product of fuzzy sets [20].

Note that (1) defines a fuzzy relation that can be represented in a matrix form [20], where mnth term of this matrix, which has been determined for the element (a_m, b_n), is governed by the rules of cross-product for fuzzy sets [20]; $m = 1, \ldots, d$, $n = 1, \ldots, w$.

Keep in mind that the number of pairs of elements in the fuzzy set A^i is d, while the total number of element pairs in the fuzzy set B^i is w; they are given by

$$d = \sum_{l=1}^{k} p_l, \qquad w = \sum_{l=1}^{r} t_l. \tag{2}$$

Thus, for each ith experiment ($i=1, \ldots, l$) we have defined the relation S^i between the fuzzy sets A^i and B^i, which can be written, in the general form, as follows:

$$A^i = \left(\underset{p_1-\text{pair of elements}}{FCM}, \underset{p_2-\text{pair of elements}}{M}, \ldots, \underset{p_k-\text{pair of elements}}{TR} \right),$$

$$B^i = \left(\underset{t_1-\text{pair of elements}}{b_1}, \underset{t_2-\text{pair of elements}}{b_2}, \ldots, \underset{p_r-\text{pair of elements}}{b_r} \right).$$

A relation between an arbitrary set (to be tested) A^{test}, which describes the grain-phase structure of a functional material, and the induced (by it) set B^{test}, which represents the performance characteristics of the same material, can be written as [20]

$$B^{test} = A^{test} \circ S. \tag{3}$$

Here $\circ$ is the sign of the maximin product (the maximin product is defined as a usual product of matrices [21], where min and max are put in place of multiplication and addition operations, respectively).

Here

$$S = S^i. \tag{4}$$

Note that B^{test} will be derived in the form of a fuzzy set. If we are interested to have B^{test} in the form of a crisp set (i.e., each performance characteristic should be represented as a uniquely defined scalar quantity), we will have to solve the problem of identifying a crisp representative of the fuzzy number for each characteristic [20]. There are more than one method for handling this problem, e.g., those discussed in [20].

If the plot of the membership function is described by a continuous non-unimodal function, the most unbiased selection of this representative will be, in our opinion, an analog of median [21] for random quantities, which can be defined as follows:

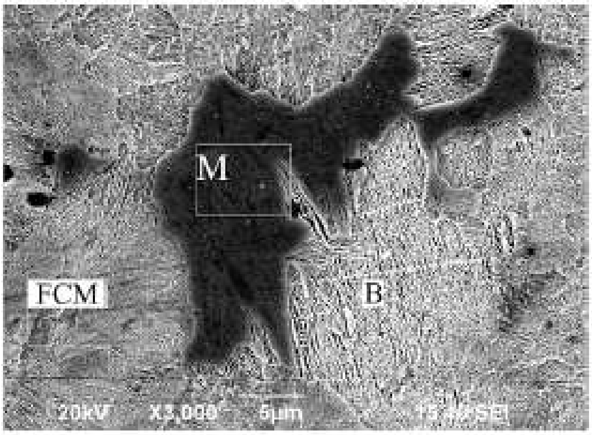
$$\int_{a_i^{\min}}^{a_i^*} \mu_{A_i}(u)du = \int_{a_i^*}^{a_i^{\max}} \mu_{A_i}(u)du, \tag{5}$$

where A_i is the fuzzy set corresponding to the ith performance characteristic, a_i^* is the crisp representative of the fuzzy set A_i, $a_i^{\min}$ is the lower limit of the A_i fuzzy set carrier, $a_i^{\max}$ is the upper limit of the A_i fuzzy set carrier, and $\mu_{A_i}(u)$ is the membership function of the fuzzy set A_i. In other words, for the crisp representative of the fuzzy set we select the particular value of its carrier, which divides the area under the membership function plot into two almost equal parts.

If the fuzzy set carrier is a discrete quantity, the crisp representative of the fuzzy number can be defined in a similar way to the definition of mathematic expectation [21] for discrete random quantities:

$$a_i^* = \sum_{l=1}^{r} \left(\frac{\mu_{A_i}(a_i^l)}{\sum_{l=1}^{r} \mu_{A_i}(a_i^l)} \right) a_i^l, \tag{6}$$

where a_i^l is the lth element of the carrier of the fuzzy set A_i, $\mu_{A_i}(a_i^{l})$ is the membership function of the lth element of the carrier of the fuzzy set A_i, and r is the number of pairs in the fuzzy set A_i.



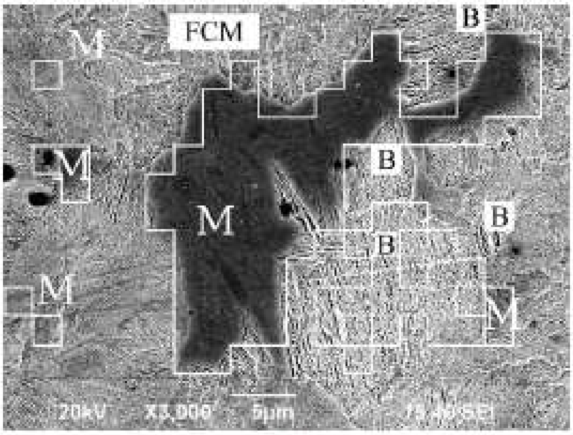


Fig. 1. Microstructure of steel upon heating to 930℃, holding for 3 min, and cooling with a rate of 20 ℃/s [the results of analysis by an expert (a) and analysis by an automated system (b)].

If the plot of the membership function of the fuzzy set has a distinct unimodal trend, then for the crisp representative of the fuzzy set we can select the carrier value that corresponds to the maximum value of the membership function:

$$a_l^* = (a_l^* \in S(A_l) | \mu_{A_l}(a_l^*) \geq \mu_{A_l}(a_l), \ a_l \in S(A_l)), \tag{7}$$

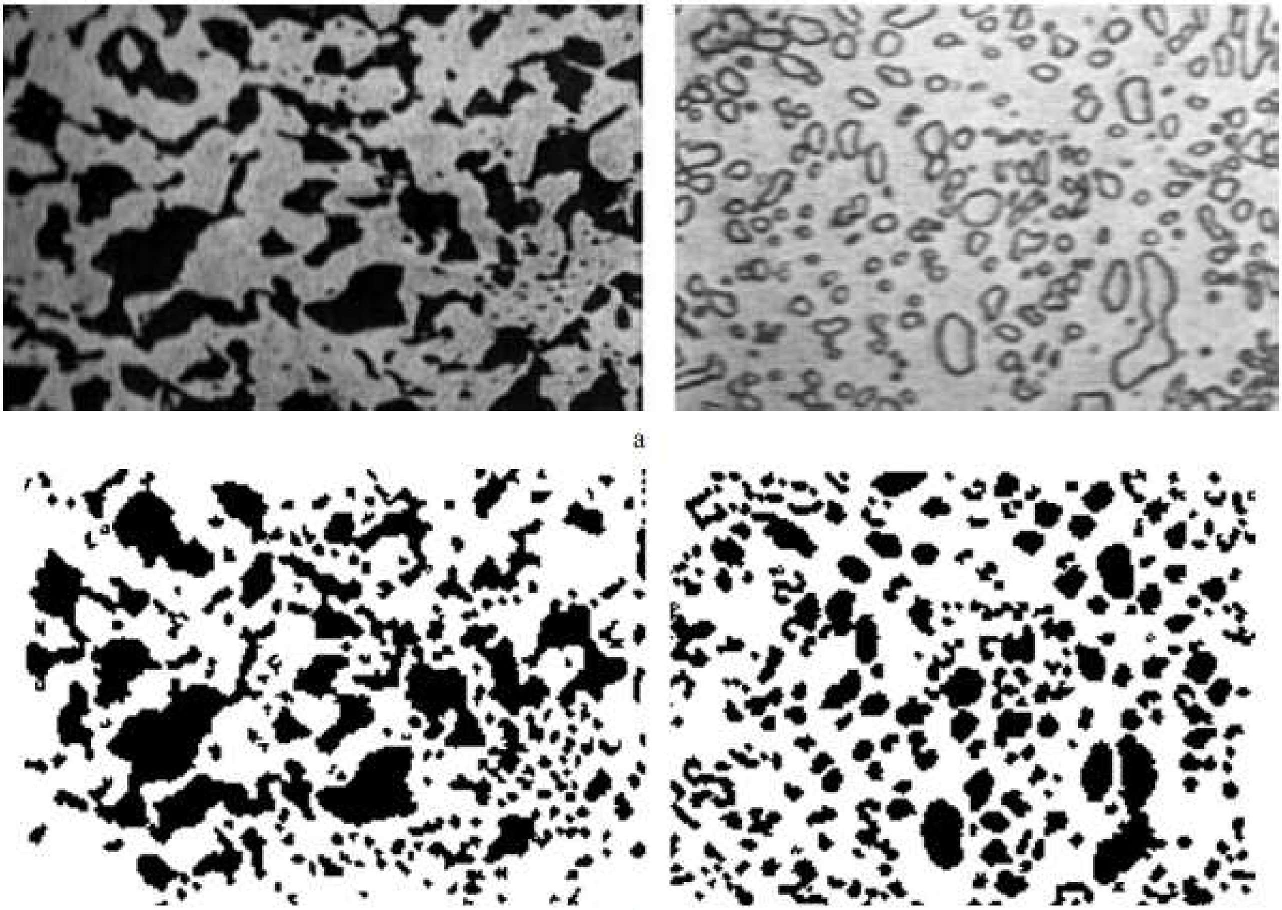
where $S(A_l)$ is the carrier of the fuzzy set A_l.

Example of Assessment of Strength Properties of a Heat-Treated Steel. For a demonstrating example we consider producing a re-bar steel with specified strength properties through heat treatment under the conditions as studied at the Research Institute for Nanosteels, Nosov Magnitogorsk State Technical University (Russian Federation) [22]. To reveal qualitative and quantitative characteristics of the structure being formed, we used a Mod. GLEEBLE 3500 testing system with a Meiji Techno optical microscope, applying Thixomet PRO image analysis software, as well as with a Mod. JSM 6490 LV scanning electron microscope. Figures 1 and 2 show examples of grain-phase structures produced under various heat treatment conditions. It is seen that under the conditions studied there arise three phases: ferrite-carbide mixture (FCM), martensite (M), and bainite (B), whose volume concentrations dictate the material strength properties. In addition to the phase composition, the grain structure parameters (the examples are given in Fig. 2) have also a strong influence on the performance characteristics of the material at hand. The grain shape and size are seen to vary within a wide range, which undoubtedly have an effect on the strength properties of steel. The following grain-phase structure parameters have been selected as the main ones: volume of the FCM, B, and M phases, mean grain size, grain size variation coefficient, grain anisotropy, and volume fraction of grains. The parameters of the grain-phase structure were computed using an automated system [17].

As performance characteristics of the functional material under consideration, we selected the Vickers hardness (HV_{30}) and impact toughness (KC). The Vickers hardness tests were carried out with a load of 30 kg, using a Mod. M4C075G3 (Emco Test) third-generation universal hardness testing machine with a high-resolution camera, and automatic measurements by Brinell, Rockwell, and Vickers methods.

Table 1 summarizes the results of processing of experimental data of the analysis of the grain-phase structure and performance characteristics of the tested steel, for seven specimens prepared in various heat treatment modes (cooling rates).

To verify the proposed methodology of assessing performance characteristics of functional materials, specimens Nos. 1, 2, 3, 4, 6, and 7 (a total of six specimens) are selected as input data, while specimen No. 5 is taken as the one to be tested. As an example, we will demonstrate the form of A^1 and B^1. Note that the membership functions of an arbitrary element c_i of carriers of the fuzzy sets A or B will be found from the relationship: $1-|(c_i - c_i^r)/c_i|$ (c_i^r is the value of the respective carrier element with the membership function equal to 1). For



a

b

Fig. 2. Input photographs of microsections (a) and grains identified by an automated system (b).

instance, the membership function of the second element of the carrier of the fuzzy set B^1 is $1-|(412-400)/400|=1-0.03=0.97$. Noteworthy is that the carrier element with the membership function equal to 1 is 0, while the membership function of the element c_i is determined subjectively based on the physical meaning:

A^1 = (1.0/100, 0.6/60, 0.8/80, 0.7/70, 0/50, 0.4/40, 0.1/10, 1.0/0, 0.9/10, 0.8/20, 0.8/20, 0.7/30, 0.7/30, 1.0/0, 1.0/0, 0.7/30, 0.9/10, 0.8/20, 0.8/20, 0.7/30, 0/90, 1.0/0.1, 1.0/0.1, 0.4/0.16, 0/0.26, 0/0.25, 0/0.21, 0.7/0.13, 1.0/0.11, 0.64/0.07, 0.55/0.06, 0.45/0.17, 0.27/0.03, 0.55/0.06, 0.64/0.07, 1.0/0.98, 0.84/1.14, 0.89/1.09, 0.80/1.18, 0.72/1.25, 0.88/1.1, 0.90/1.08, 1.0/0.87, 0.30/0.26, 0.51/0.44, 0.87/0.98, 0.43/0.37, 0.91/0.79, 0.95/0.91);

B^1 = (1.0/400, 0.97/412, 0.91/436, 0.83/469, 0.62/554, 0.46/617, 0/800, 1.0/54.3, 0.97/55.7, 0.92/58.5, 0.82/64.0, 0.97/55.7, 0.92/50.1, 0.82/44.6).

It should be pointed out that the fuzzy relations S^i, $i=1, \ldots, 6$ for the example at hand are the matrices with dimensions 49×14.

By combining the fuzzy relations S^i, $i=1, \ldots, 6$ by formula (4), we will arrive at a fuzzy relation (matrix) between an arbitrary set describing the grain-phase structure of a functional material (in the example considered it is the structure of specimen No. 5) and the induced set describing the performance characteristics of the material. Define A^5 (based on Table 1):

A^5 = (0/100, 0.8/60, 0.4/80, 0.6/70, 1.0/50, 0.8/40, 0.2/10, 0.7/0, 0.8/10, 0.9/20, 0.9/20, 1.0/30, 1.3/30, 0.7/0, 0.8/0, 0.9/30, 0.9/10, 1.0/20, 1.0/20, 0.9/30, 0/90, 0.4/10, 0.4/0.10, 0.64/0.16, 0.96/0.26, 1.0/0.25, 0.84/0.21, 0.52/0.13, 0/0.11, 0/0.07, 0/0.06, 0/0.17, 1.0/0.03, 0/0.06, 0/0.07, 0.78/0.98, 0.91/1.14, 0.87/1.09, 0.94/1.18, 1.0/1.25, 0.88/1.1, 0.86/1.08, 0/0.87, 0.73/0.26, 0.82/0.44, 0/0.98, 1.0/0.37, 0/0.79, 0/0.91).

TABLE 1. Data of the Analysis of the Grain-Phase Structure and Performance Characteristics of Investigated Steel

Specimen No.	Cooling rate, ℃/c	Grain structure parameters (grain size in μm)	Phase composition parameters, %	Hardness, HV_{30}	Impact toughness KC, J/cm^2
1	10	Mean grain size: 0.10 Grain size variation coefficient: 0.11 Grain anisotropy: 0.98 Volume fraction of grains: 0.87	FCM: 100	400	54.3
2	20	Mean grain size: 0.10 Grain size variation coefficient: 0.07 Grain anisotropy: 1.14 Volume fraction of grains: 0.26	FCM: 60 Bainite: 10 Martensite: 30	412	55.7
3	25	Mean grain size: 0.16 Grain size variation coefficient: 0.06 Grain anisotropy: 1.09 Volume fraction of grains: 0.44	FCM: 70 Bainite: 20 Martensite: 10	436	58.5
4	30	Mean grain size: 0.26 Grain size variation coefficient: 0.17 Grain anisotropy: 1.18 Volume fraction of grains: 0.98	FCM: 70 Bainite: 20 Martensite: 10	469	64.0
5	40	Mean grain size: 0.25 Grain size variation coefficient: 0.03 Grain anisotropy: 1.25 Volume fraction of grains: 0.37	FCM: 50 Bainite: 30 Martensite: 20	554	55.7
6	50	Mean grain size: 0.21 Grain size variation coefficient: 0.06 Grain anisotropy: 1.1 Volume fraction of grains: 0.79	FCM: 40 Bainite: 30 Martensite: 30	617	50.1
7	60	Mean grain size: 0.13 Grain size variation coefficient: 0.07 Grain anisotropy: 1.08 Volume fraction of grains: 0.91	FCM: 10 Martensite: 90	800	44.6

The induced (by this set) fuzzy set B^5 that represents the material performance characteristics is defined from relation (4) in the form:

B^5 = (0.94/400, 0.94/412, 0.94/436, 1.0/469, 0.9/554, 1.0/617, 0.91/800, 0.94/54,3, 0.94/55,7, 0.94/58.5, 1.0/64.0, 0.93/55.7, 1.0/50.1, 0.91/44.6).

If the values of the performance characteristics are to be substituted as scalar quantities, a problem of determination of the crisp representative of the fuzzy number has to be solved separately for each characteristic [in our case, they are the Vickers hardness (HV_{30}) and impact toughness (KC)]. This can be done in different ways [from relations (5), (6), or (7)].

For instance, by using relations (6), we can obtain HV_{30} = 525.66 and KC = 54.79 J/cm^2. In this case, the experimental values (Table 1) as as follows: HV_{30} = 554 and KC = 55.7 J/cm^2.

Thus, the calculation error is evaluates to be

$$\delta_{HV} = \frac{HV_{30}^{t} - HV_{30}^{e}}{HV_{30}^{e}} \cdot 100\% = 5.12\%, \qquad \delta_{KC} = \frac{KC^{t} - KC^{e}}{KC^{e}} \cdot 100\% = 1.64\%.$$

TABLE 2. Predicted Strength Characteristics of Specimen No. 5 for Different Number of Specimens to be Used

Number of specimens	Calculated hardness HV_{30}	Relative error (HV_{30}), %	Calculated impact toughness KC, J/cm^2	Relative error (KC), %
3	517.81	6.53	54.61	1.96
4	520.76	6.01	54.63	1.92
5	522.01	5.77	54.65	1.88
6	525.66	5.12	54.79	1.63

Of special interest is to assess the required number of experiments in order to achieve the necessary accuracy. Let us carry out a few numerical experiments to find the sought-for performance characteristics of specimen No. 5. The results obtained are summarized in Table 2.

The results obtained demonstrate that, as expected, the larger the number of experiments, the higher the accuracy of the predicted results. The smallest possible number of experiments needed should be found by way of trial, depending on the required accuracy of the complex estimation.

Conclusions. A methodology has been elaborated, which enables one to assess, based on the analysis of the grain-phase structure parameters of a functional material, the performance characteristics of this material, in particular its strength properties. A special feature of the proposed methodology is the application of the fuzzy-set theory to define multiple relationships between a preset combination of strength properties and the grain-phase structure parameters as computed from a photograph of a material microsection. As an example, we assessed strength properties of a re-bar steel produced through heat treatment under different holding and cooling conditions.

The results obtained confirm suitability of the elaborated methodology for the complex estimation of strength properties of a material by its grain-phase structure parameters. It should be kept in mind that further research has to be undertaken in order to assess the influence of various grain-phase structure parameters on the strength properties and to study convergence of the proposed methodology for different combinations of microstructure parameters.

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