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Methodology to estimate the minimum number of experiments and key microstructural parameters in macroscopic strength properties evaluation

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A novel methodology, based on the theory of fuzzy sets, to obtain materials with pre-defined sets of strength properties has been analysed from the position of identifying the necessary and sufficient number of experiments needed to predict these macro characteristics and establishing which micro parameters significantly influence the macroscale results. The procedure to estimate, with a user-defined degree of accuracy, the minimum number of experiments and significant micro parameters has been tested and verified using experimental data, obtained from digital images of material microsections under different heat treatment conditions while analysing strength properties of reinforcing steel. The results confirm the possibility of using the developed methodologies for the performance properties evaluation of materials based on the minimum number of experiments and identification of the key grain-phase parameters.

1. Introduction

As it is commonly accepted in the literature (see e.g. [1]), the properties of any metallic material are determined by its chemical composition and internal structure. The internal structure, in turn, depends on the manufacturing technology and subsequent mechanical, chemical and thermal after-treatments. Thus, with a fixed chemical composition, and by controlling the internal metal structure, it is possible to obtain materials with desired combinations of physico-mechanical properties [1-3].

Nowadays, the task of manufacturing metallic materials with predetermined exploitation properties (mechanical, electrical, magnetic, chemical, etc.), so-called functional materials, is getting increasingly popular. Functional materials should have strictly defined and preferably customisable physical (and chemical) characteristics, that enable them to fulfil service requirements of structures and devices that are unattainable with other materials. The manufacturing of functional materials with optimal properties can be carried out by strengthening or suppressing (i.e. tuning) properties of already available materials; this process requires novel experimental and theoretical techniques. As an example of metallic functional materials, metal alloys with martensitic transformations and shape memory effects [4] can be considered. However, in order to manufacture novel metallic functional materials with desired operational properties, it is necessary to have a methodology, determining these properties depending on the internal structure of the metal.

In this paper, attention will be focused on designing the methodology, which, unlike the majority of currently accepted approaches, does not require a specific format of a constitutive relation, or other approximation function, linking microscopic (internal) characteristics and macroscopic effective behaviour of a material; instead it allows to estimate parameters of interest by formulating fuzzy relationships between micro- and macro characteristics. Further on it will be analysed which characteristics of the internal structure influence macroscopic properties significantly and which ones can potentially be neglected. The attention will also be focused on developing a methodology to estimate the minimum number of experiments that has to be performed for sufficiently accurate predictions. As an illustrative example of this methodology, medium-carbon steels, strengthened by a special heat treatment, will be analysed.

2. Methodology

Accepting the hypothesis that the macroscopic strength properties of a metallic material are determined by its internal structure, the problem can be split in the following sub-problems:

1. Develop a mathematical model of macroscopic (strength) properties integrated estimation based on the analysis of microstructural parameters.
2. Develop a methodology to establish the key parameters of the microstructure, that significantly affect the strength properties of metallic functional materials.
3. Offer an algorithm to estimate the minimum number of experiments necessary to predict strength properties of metallic functional materials.

In the following three sub-sections, the aforementioned sup-problems are described in more details.

2.1. A mathematical model of strength properties integrated estimation based on the analysis of microstructural parameters

The formulation and methodology of the integrated estimation of material's strength properties (sub-problem 1) has been analysed in detail in [5-6]. In particular, a new approach to the solution of this problem has been proposed, based on decomposition of the initial problem into several sub-steps. Initially, as already noted, a hypothesis has been introduced that the set of required physico-mechanical characteristics of a material is uniquely determined by its microstructure. In the case of metals and alloys, by microstructure we mean the grain-phase structure of the material. Note here, that an unambiguous conformity between parameters of the grain-phase microstructure and macroscopic parameters characterizing the performance characteristics of functional material cannot be guaranteed, thus one-to-one relations may not be possible to construct. This, however, is not an issue in the proposed methodology, where the framework is designed in micro-to-macro direction.

The determination of a material's properties can be reduced to two sub-tasks:

i. obtaining a grain-phase structure of the material, and
ii. an integrated estimation of its performance properties, depending on the parameters of the material's microstructure.

The second sub-task (ii), in which an integrated estimation of the physico-mechanical (macroscopic) characteristics from a set of the grain-phase (microscopic) structure of the material can be performed, has been analysed in details in [5-6], however for readers' benefits, the main aspects of the methodology are presented below.

As mentioned above, the methodology, contrary to the majority of currently accepted approaches, does not require a specific format of constitutive relation or other approximation function, instead it builds fuzzy relationships between microscopic characteristics of the metals grain-phase structure and macroscopic effective performance properties. These fuzzy relationships are built for all (n) available experiments (here microsections and measurements of performance characteristics corresponding to each of them):

$$S^i = m^i \times M^i, \quad i = 1..n$$

Mathematically, m^i and M^i are special[1] fuzzy sets containing parameters of the grain-phase microstructure and macroscopic performance characteristics for the ith experiment respectively, and $\times$ represents Cartesian product of fuzzy sets.

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[1] Elements of special fuzzy sets can have a different nature and thus different dimensions, unlike conventional fuzzy sets, where elements belong to the same physical space and are of same type with the same dimensions.

As the next step, an operator $\boldsymbol{F}$, linking material's macroscopic effective performance characteristics (e.g. strength), and parameters, describing the materials microstructure can be constructed as:

$$\boldsymbol{F} = \cup_{i=1}^{l} S^i$$

with the operator $\cup_{i=1}^{l} S^i$ referring to the standard fuzzy union of membership matrices: $f^i = \max(s_i)$.

Once constructed, for available experiments, the operator $\boldsymbol{F}$ is then used to determine the set of unknown macroscopic parameters $\widetilde{M}$ via the relation

$$\widetilde{M} = \widetilde{m} \circ \boldsymbol{F}$$

where $\widetilde{m}$ refers to the set of microscopic parameters, corresponding to the set of unknown macrostructural characteristics, and $\circ$ represents the maximin product[2].

Note, that the number of parameters in both sets of microscopic parameters and macroscopic characteristics can be different: for example in [5,6] the main parameters of the grain-phase structure were identified as volume fractions of phases - ferritic-carbide mixture, martensite, bainite, the mean grain size, coefficient of grain size variation, degree of grain anisotropy and volume proportion of grains (seven in total), and the macroscopic performance characteristics where the Vickers hardness and the impact strength (two in total).

In this paper, to slightly simplify the mathematical procedure, for the theoretical example only five microstructural parameters were considered (volume fractions of phases - ferritic-carbide mixture, martensite, bainite, the mean grain size and degree of grain anisotropy) and only one macroscopic strength characteristic: the Vickers hardness. For the practical example, all seven microstructural parameters were taken into account. However, as this methodology is design to be mathematically generic, other mechanical (as well as chemical or metallurgical) parameters and their combinations can also be considered, both in micro- and macroscopic levels.

2.2. A methodology to estimate the minimum number of experiments needed to predict strength properties of metallic functional materials

In order to define the minimum number of experiments needed to determine the strength properties of materials, it is required first to estimate the error resulting from solving the problem for a certain given number of experiments.

For every experiment, the parameters of the microstructure and values of corresponding determined strength characteristics are determined. In this paper, the strength characteristic has been chosen to be the Vickers hardness HV_{30}, but note that the methodology for estimating the minimum number of experiments remains the same, if the strength characteristic is chosen differently or if more than one strength parameter is desired.

Consider the total number of available experiments to be n. In order to estimate the accuracy of calculations, assume one of the available experimentally obtained Vickers hardness as a reference with its value denoted by HV_{30}^{ex}; the remaining experimental values of strength, using the technique described in Section 2.1, will result in determination of the theoretical value of strength HV_{30}^{theor}.

Now the relative computational error can be defined as

$$. \tag{1}$$

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[2] An ordinary product of matrices [7], where min and max are substituted for the multiplication and addition operations respectively.

$$\delta_{HV} = \left| \frac{HV_{30}^{theor} - HV_{30}^{ex}}{HV_{30}^{ex}} \right| \cdot 100\%. \tag{1}$$

In order to determine the required number of experiments, ensuring acceptable accuracy, let's consider conducting all possible combinations of numerical experiments to find the required performance characteristics of the reference sample, starting from conducting only two (out of n available), i.e. $i = 2$, experiments, then conducting three experiments (out of n available), i.e. $i = 3$, etc. until $i = n$.

The number of possible combinations of i conducted experiments is equal to $\binom{n}{i}$, where $i = 2, 3, \ldots n$. Here $\binom{n}{i}$, the number of combinations from n to i, which is determined by the usual formulation [8]

$$\binom{n}{i} = \frac{n!}{i!(n-i)!} \tag{2}$$

Each number of conducted experiments i would lead to a specific relative error. Defining the desired accuracy and the following maximum used-defined error as δ_{HV}^{*}, the minimum number of experiment i^{*} can be determined from

$$\delta_n^{i^{*}} \leq \delta_{HV}^{*} \tag{3}$$

with i^{*} representing the minimum value of i, ensuring satisfaction of relation (3); δ_n^{i} is an averaged computational error for all possible combinations of experiments.

2.3. An algorithm to determine microstructural parameters significantly affecting strength properties of the functional material

In order to determine the most influential microscopic parameters affecting the macroscopic properties, the process starts from identification of all available microstructural parameters that can be chosen to evaluate the Vickers hardness of steel. Several recent studies (see e.g. [9-14]), have reported an existence of relationships between microscopic properties of a material, in particular its grain-phase structure, and macro properties. Analysing the above-mentioned studies, it can be concluded that the Vickers hardness for steels is mainly determined by the phase composition and the characteristic grain size and morphology.

Following from the aforementioned conclusion, the next step is the determination of micro parameters which can be used to estimate the Vickers hardness, based on the available experimental data. Using the computer pattern recognition [15], it is possible to estimate values of some microstructural parameters from microsections images. Thus, to evaluate the Vickers hardness, the values of the volume fractions of phases, the mean grain size, the coefficient of grain size variation and the volume content of grains can be used.

To determine parameters that significantly affect the analysed macroscopic characteristic, a slightly modified approach to the one described in Section 2.2 can be employed.

Consider, as before, the total number of available experiments to be n. To estimate the accuracy of calculations for each of the selected microstructural parameters, assume one of the available experiments as the reference one, i.e. the value of the strength property for this experiment is denoted by HV_{30}^{ex}, all the remaining experiments following the methodology presented in Section 2.1, result in determining the value of the HV_{30}^{theor} for each of the parameters. Now, following the procedure in Section 2.2, the relative error for each j-th microstructure parameter can be estimated as

$$\delta_{\text{HV}}^{\text{j}} = \left| \frac{\text{HV}_{30}^{\text{theor, j}} - \text{HV}_{30}^{\text{ex}}}{\text{HV}_{30}^{\text{ex}}} \right| \cdot 100\% \,. \tag{4}$$

Note that each microstructural parameter will result to its own relative error. For a pre-determined required accuracy (i.e. user pre-defined relative error δ_{HV}^{**}), the significance of the j-th parameter can be determined as:

$$\delta_{HV}^{j*} \leq \delta_{HV}^{**}, \tag{5}$$

where δ_{HV}^{j*} is an averaged error for j-th parameter for all possible experiments.

The proposed above methodology for determining the parameters of the microstructure, which significantly affects the macroscopic characteristics of the functional material, is simple and sufficiently clear. However, it has a drawback: while evaluating the computational error for each j-th microstructural parameter all other microstructural parameters, potentially also affecting the strength, are not taken into account.

To address this, a new (corrected) methodology has been proposed. In this case, the value of the HV_{30}^{theor} can be determined on the basis of all the microscopic parameters, with the exception of the tested parameter. If the computation error (4) is found to be insignificant, then the corresponding testes parameter can be ignored in determining the required strength characteristic. In other words, the significance of the j-th parameter is determined from the relation:

$$\delta_{HV}^{j**} \geq \delta_{HV}^{**} \tag{6}$$

Thus, if the relation (6) is not satisfied, then the influence of the jth microstructural parameter can be neglected in determining the required strength characteristic.

3. Selecting the microstructural parameters and the number of experiments affecting the strength properties of metallic functional materials: practical examples

In the Section below both problems (the choice of microstructural parameters and the minimum number of experiment) will be tested. The attention first will be focused on the methodology to determine key micro-parameter influencing macro-properties, followed by the determination of the minimum number of experiments needed to predict these macro-parameters with a desired accuracy.

3.1. Justification of the choice of microstructural parameters

As noted above, the main parameters of the grain-phase structure affecting the strength characteristics of metallic functional materials include: volume fractions of phases (ferritic-carbide mixture (FCM), martensite (M) and bainite (B)), the mean grain size, coefficient of grain size variation, the degree of anisotropy of grains and the volume fraction of grains. In sub-section below, a theoretical example and an experimental example (see [16] for the data) will be performed to demonstrate the methodology discussed in Section 2.3 and justify the choice of the most significant microstructural parameters influencing macroscopic responses.

For illustrative purpose, some parameters obtained in experiments reported in [16] were slightly modified and extra values were added to the theoretical example. Note, that the actual un-amended experimental data were also analysed further in the experimental example.

3.1.1. Theoretical example. It is assumed that the total number of available experiments is 10. In Table 1 the microstructural characteristics of the grain-phase structure (here the mean grain size, the degree of anisotropy, and the volume fractions of the phases) and the

macroscopic Vickers hardness obtained for each experiment are presented. To demonstrate the methodology, assume the Vickers hardness for the 5th experiment unknown (denoted by " $*$ " in Table 1).

Table 1: Microstructural characteristics and the macroscopic Vickers hardness for theoretical example

Number of the experiment	Mean grain size, μm	Degree of anisotropy	Volume fraction of FCM, %	Volume fraction of B, %	Volume fraction of M, %	Vickers hardness
1	0.1	0.98	98	1	1	400
2	0.15	0.93	85	10	5	410
3	0.15	0.81	80	15	5	440
4	0.2	0.17	75	20	5	500
5	0.2	0.43	70	25	5	530*
6	0.25	0.89	67	28	5	560
7	0.3	0.57	60	30	10	590
8	0.3	0.53	50	35	15	650
9	0.3	0.42	30	40	30	710
10	0.35	0.41	10	50	40	800

Based on 9 experiments (excluding experiment No. 5), the values of the theoretical Vickers hardness for the 5th experiment for each characteristic of the grain-phase structure (see 5 microscopic characteristics in Table 1) were calculated by the method described in Section 2.1 and the methodology presented in Section 2. The results of the calculations are summarized in Table 2a.

Table 2a: Assessments of microstructural parameters, simplified methodology (theoretical example)

Microstructural characteristics	Vickers hardness	Relative error δ_{HV}^{j*}
Mean grain size	553.4	4.42
Degree of anisotropy	574.0	8.30
Volume fraction of FCM	552.4	4.23
Volume fraction of B	558.4	5.36
Volume fraction of M	541.8	2.23

As can be concluded from Table 2a, the greatest value of an average relative error δ_{HV}^{j*} in calculating the Vickers hardness corresponds to the second characteristic (degree of anisotropy of grains).

If the required accuracy is set to be 95% and the acceptable error thus is $\delta_{HV}^{**} = 5\%$ then, based on condition (5), the degree of anisotropy and the volume fraction of bainite (B) for calculating the strength characteristic (Vickers hardness) can be neglected. Note that the volume fraction of bainite produces considerably smaller error compared to the degree of anisotropy.

As a next the step the corrected methodology of Section 2.2 has been implemented. The results obtained (Table 2b) correspond to the results given in Table 2a:

Table 2b: Assessments of microstructural parameters, improved methodology (theoretical example)

Microstructural characteristics	Vickers hardness	Relative error δ_{HV}^{j**}
Mean grain size	557.6	5.21
Degree of anisotropy	549.1	3.60
Volume fraction of FCM	557.6	5.21
Volume fraction of B	557.5	5.21
Volume fraction of M	560.7	5.70

As follows from Table 2b, the degree of anisotropy for estimating the Vickers hardness shows much less influence than the rest of parameters and thus can be neglected. These results confirm and improve the results of the initial, simplified, methodology presented in Table 2a and clarify the influence of bainite. As an outcome of this example the new, corrected methodology will be used in the remainder of the paper.

3.1.2. Experimental example. The results of this example are based on the data of [16], where the heat treated reinforcing steel with given strength properties was analysed.

In this example the total number of available experiments is 7. In Table 3 all microstructural characteristics of the grain-phase structure and the Vickers hardness obtained for each experiment are presented. The microstructural characteristics in this case are slightly different than the ones discussed in the theoretical example: the mean grain size, grain size variation coefficient, the degree of anisotropy, the volume content of grains and the volume fractions of the phases. Again the macroscopic characteristics of the 5th experiment is assumed to be unknown (denoted by " $*$ " in Table 3).

Table 3: Microstructural characteristics and the macroscopic Vickers hardness for experimental example

Number of the experiment	Volume fraction of FCM, %	Volume fraction of B, %	Volume fraction of M, %	Mean grain size, μm	Grain size variation coefficient	Degree of anisotropy	Volume fraction of grains	Vickers hardness
1	100	0	0	0.10	0.11	0.98	0.87	400
2	60	10	30	0.10	0.07	1.14	0.26	412
3	70	20	10	0.16	0.06	1.09	0.44	436
4	70	20	10	0.26	0.17	1.18	0.98	469
5	50	30	20	0.25	0.03	1.25	0.37	554[*]
6	40	30	30	0.21	0.06	1.10	0.79	617
7	10	0	90	0.13	0.07	1.08	0.91	800

Based on 6 experiments (excluding experiment No. 5), the values of the theoretical Vickers hardness HV_{30}^{theor} for the 5th experiment, following the procedure introduced in Section 2.1, are summarized in Table 4.

Table 4: Assessments of microstructural parameters, improved methodology (experimental example)

Microstructural characteristics	Vickers hardness	Relative error δ_{HV}^{j**}
Volume fraction of FCM	526.0	5.05
Volume fraction of B	524.9	5.25
Volume fraction of M	526.0	5.05
Mean grain size	527.1	4.86
Grain size variation coefficient	526.0	5.05
Degree of anisotropy	523.0	5.60
Volume fraction of grains	526.0	5.05

Analysing the results, it can be concluded that for estimation of the Vickers hardness, the mean grain size shows the smallest effect on the overall results, and, as follows from equation (6), and assuming $\delta_{HV}^{**} = 5\%$, it can be neglected.

The analysed examples verified the methodology to exclude microstructural parameters of the grain-phase structure that are of insufficient influence on the macro-characteristics performance properties of functional materials.

3.2. Estimation of the minimum number of experiments needed to determine strength properties of metallic functional materials: practical examples

This Section focuses on the determination of the minimum number of experiments, needed to determine the Vickers hardness in the context of discussed above examples. The methodology, to determine this minimum number is presented in Section 2.2 above.

3.2.1. Theoretical example (continued). Following the results presented in Table 2, in the case of the required accuracy of 95% and thus $\delta_{HV}^{**} = 5\%$, the degree of anisotropy of the grains in predicting the Vickers strength can be neglected. Thus the degree of anisotropy was not taken into account in further computation for this example.

Following the methodology, discussed in Section 2.1, and taking into account the possible combinations of different number of experiments, the theoretical values of the Vickers hardness HV_{30}^{theor} are summarised in Table 5.

Table 5: Assessments of number of experiment (theoretical example)

Amount of conducted experiments (i)	Number of combinations $\binom{n}{i}$	Vickers hardness	Relative error δ_{HV}^{j*}
1	9	559.0	5.47
2	36	552.3	4.21
3	84	551.1	3.98
4	126	550.5	3.87
5	126	550.0	3.77
6	84	549.8	3.74
7	36	549.6	3.70
8	9	549.3	3.64
9	1	549.1	3.60

As can be seen from Table 5, the relative error δ_{HV}^{j*} is monotonically decreasing with increasing the amount of experiments, thus by setting the error threshold, the user can identify the minimum number of experiments to be conducted.

3.2.2. Experimental example (continued). According to Table 4, allowing the error to be $\delta_{HV}^{**} = 5\%$, the mean grain size can be neglected and will not be considered in the computations. Table 6 summarises the values of Vickers hardness for the tested 5th experiment, and the relative computational errors for different numbers of experiments (taking into account their possible combinations).

Table 6: Assessments of number of experiment (experimental example)

Amount of conducted experiments (i)	Number of combinations $\binom{n}{i}$	Vickers hardness	Relative error δ_{HV}^{j*}
1	6	499.3	9.87
2	15	507.9	8.32
3	20	515.5	6.95
4	15	520.8	5.99
5	6	524.1	5.40
6	1	527.1	4.86

For this experimental example, similar to the theoretical one, the relative error δ_{HV}^{j*} is monotonically decreasing with increasing the amount of experiments. Setting the user-defined accuracy the minimum number of conducted experiment can be identified. For example: setting

the required accuracy to be 95%, and thus $\delta_{HV}^* = 5$ %, and following (3), the minimum number of experiments necessary to predict the desired characteristic with a given accuracy is 5. However, if the desired accuracy is lower, e.g. $\delta_{HV}^* = 7$ %, then 2 experiments are enough.

Analysing the above examples it can be concluded that the proposed approach makes it possible to estimate the minimum number of experiments necessary to predict the operational properties of metallic functional materials.

The following has to be noted, however: for both (i) the task of selecting the main parameters of the grain-phase structure and (ii) the task of estimating the minimum number of experiments, all analysed microstructural parameters should ideally lie within characteristic bounds. In this case, an interpolation problem is being solved. If one (or more) of the microstructural characteristics in an analysed material sample is outside the specified bounds, the problem becomes an extrapolation problem and the accuracy of predicted properties of the functional material in this case deteriorates significantly, see Table 7.

Table 7: Assessments of number of experiment – interpolation and extrapolation problems (experimental example)

Experiments with known parameters / experiment with unknown parameters	Vickers hardness	Relative error δ_{HV}^{j*}
1,2,3,4,6,7 / 5 (554*)	527.1	4.86
1,2,3,4,5 / 7 (800*)	506.2	36.73
1,2,3,4,5,6 / 7 (800*)	514.6	35.68

In this Table 7 experimental data reported in Table 4 are analysed. The results show that for the case of microstructural parameters lying within characteristic bounds (first row in Table 10), the accuracy of predicted macrostructural parameter is high (i.e. the error is low) $\delta_{HV}^{j*} = 4.86$ %. Note that this row corresponds to the case of known parameters from experiments 1, 2, 3, 4, 6, 7 and 5th experiment assumed to be with unknown macroproperties. All microparameters of experiment 5 lie within bounds of previously recorded values (e.g. the volume fraction of FCM, measured 50%, lie inside bounds of recorded FCM ∈ [10,100], etc.).

However, for the case of microstructural parameters occurring outside the characteristic bounds, and thus for the case of extrapolation problem, the predicted macroproperties are of much less accurate nature – see Table 10 row 2 (and 3), when experiments 1,2,3,4,5 were considered known and experiment 7 unknown. Note here a different value of $HV_{30}^{ex} = 800$. In this case not all microstructural parameters lie within recorded bounds of the other five experiment (e.g. the volume fraction of FCM for 7th experiment was measured 10%, however, recorded bounds in known five experiments are FCM ∈ [50,100] and the same goes for the volume fraction of M: for 7th experiment it was measured 90%, however, recorded bounds of the other five experiments are M ∈ [0,30]).

Moreover, the further away from the characteristics limits the microstructural parameter, the less accurate the predicted properties: the second row (known experimental FCM ∈ [50,100]) results in $\delta_{HV}^{j*} = 36.75\%$, and the third row (known experimental FCM ∈ [40,100]) $\delta_{HV}^{j*} = 35.63\%$; also taking more known experiments into account (third row in Table 10), helps increase the accuracy.

4. Conclusion

The methodology, designed to evaluate the macroscopic properties of a material based on the analysis of the microscopic parameters of its microstructure, is discussed and illustrated on practical example of medium-carbon steels, strengthened by a special heat treatment. Particular attention has been given to the selection of the key parameters of the microstructure, sufficiently influencing macroscopic properties. A procedure to estimate the minimum number of experiments to estimate macroscopic properties with the required accuracy has also been discussed.

The obtained results confirm the possibility of using the developed method for an integrated evaluation of the performance properties of materials based on the parameters of its grain-phase microstructure.

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