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APPLICATION OF ASTM STANDARDS IN QUANTITATIVE MICROSTRUCTURE EVALUATION

WYKORZYSTANIE NORM ASTM DO ILOŚCIOWEJ OCENY MIKROSTURKTURY

A b s t r a c t

Quantitative microstructure characterization is currently required in R&D as well as in routine quality control of materials and products. Every step: preparation of the laboratory and specimens, measurements and interpretation of the results etc. can be a source of serious errors. Application of the ASTM guidelines can minimize these problems and improve international circulation of the results. The paper characterizes selected ASTM standards devoted to laboratory practice and analyses the complexity of microstructural materials characterization.

Keywords: *ASTM*, *image analysis*, *metallography*, *microstructure*, *standardization*

S t r e s z c z e n i e

Ilościowa ocena mikrostruktury jest obecnie powszechnie wymagana zarówno w działalności R&D, jak i podczas rutynowej kontroli jakości materiałów i wyrobów. Każdy krok: przygotowanie laboratorium i próbek, pomiary oraz interpretacja wyników etc. może być źródłem poważnych błędów. Wykorzystanie zaleceń ASTM może zminimalizować te problemy i ułatwić międzynarodową wymianę wyników. W artykule scharakteryzowano wybrane normy ASTM poświęcone praktyce laboratoryjnej oraz przeanalizowano złożoność problemu ilościowej charakterystyki mikrostruktury materiałów.

Słowa kluczowe: *ASTM*, *analiza obrazu*, *metalografia*, *mikrostruktura*, *normalizacja*

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1. Introduction

The question: *Why should we quantify materials microstructure?* is neither simple not naïve. One can define two major groups dealing with materials: producers and customers. The producer supplies materials to the customer, often in the form of semi products, and is interested mainly in production costs, technology and properties required by the customer. The customer, on the other hand, (the one who can use a given material for further production of several goods) is interested in the price and properties of the materials offered by producers. Please, note that nobody is interested in microstructure. The above demonstrated discussion is usually very surprising for students who devote a lot of time to study microstructures of different materials and methods of how to test them. The importance of materials microstructure becomes quite obvious if we discover that materials engineering is based on a simple theorem that in fact cannot be found even in advanced textbooks:

Two materials with ***identical microstructure*** *have* ***identical properties*** *irrespective of their technological history.*

If one considers the above rule it will be clear that quantitative parameters of the microstructure can be successfully used in quality control and assessment of structure-property relationships. Basic rules of microstructure quantification have been defined and well known for decades [1]. Unfortunately, only a very limited number of structure-property relationships has been fixed and verified [2]. Some of them are presented in Table 1. It is noteworthy that even if the relation is well known, we often have no precise, quantitative formula which describes it.

Table 1

Well defined structure-property relationships

Structure-property relationship	Quantitative relation
Effect of grain size on yield point (Hall-Petch relation)	Yes
Effect of pearlite content on properties of annealed carbon steel	Yes
Effect of hard phase content on wear resistance	Yes
Effect of porosity on mechanical properties of sintered materials	No
Effect of graphite shape on mechanical properties of cast iron	No
Effect of non-metallic inclusions on mechanical properties of steel	Partially
Effect of dislocation density on plastic deformation	No
Effect of nonhomogeneity of wear resistance on cast alloys	No

The above analysis demonstrates that the problem of practical application of microstructural data is still far from final solving. In most cases, one uses these data for quality control. The de facto world standards in this area are set by ASTM and therefore the possibility of building a complete set of procedures based on ASTM documents is analysed in the subsequent part of this paper.

2. Laboratory preparation

In this template, styles are created to facilitate writing. Depending on what you want to write, you have to choose the right style.

Table 2

Selected standards for laboratory preparation

Number of the standard	Title of the standard
ASTM E7 – 15	Standard Terminology Relating to Metallography
ASTM E3 – 11	Standard Guide for Preparation of Metallographic Specimens
ASTM E768 – 99(2010)e1	Standard Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel
ASTM E883 – 11	Standard Guide for Reflected–Light Photomicrography
ASTM E1951 – 14	Standard Guide for Calibrating Reticles and Light Microscope Magnifications
ASTM E2014 – 11	Standard Guide on Metallographic Laboratory Safety
ASTM E691 – 15	Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

To ensure precise communication, standard terminology related to metallography (ASTM E7) and preparation of metallographic specimens (ASTM E3) are standardized. For automatic image analysis, which allows for much more effective microstructure quantification, one needs specimens prepared with higher precision. These requirements emerged later and therefore instead of thorough revision of the previous standard, a completely new procedure has been prepared (ASTM E768). Most of the test is performed with the use of reflected light microscopy. Guidelines concerning the proper choice of microscopes and their calibration can be found in the ASTM E883 and ASTM E1951 standards. There are also some standards, not listed here, which are devoted to macro and micro etching leading to better appearance of the microstructural features tested.

Ensuring safety work conditions is of the highest importance in a metallographic laboratory due to the machining of specimens made of hard materials and the presence of hazardous chemicals. When organizing a laboratory, one can find some help in the appropriate AST standard (E2014).

Subtle differences in specimen preparation, etching, image acquisition and subsequent analysis often lead to significant scatter in the results obtained in different laboratories. Objective validation of test procedures used in metallography is very limited and in many cases even impossible. This is caused by the lack of alternative source of microstructural information. For example, fine precipitations can be observed only by means of advanced methods of transmission electron microscopy (TEM), and one has no other possibility to observe these precipitations. Consequently, we cannot verify correctness of shape and size characteristics evaluated with the help of TEM. Comparative measurements performed during interlaboratory tests are the best and sometimes the only possible solution. This methodology is also standardized (ASTM E691).

3. Application of image analysis

ASTM standards are prepared in a form similar to a scientific paper. The contents describe the scope and terminology, apparatus and specimen preparation, the measurement procedure, interpretation of the results and report as well as appendixes with exemplary case histories etc. Usually, the standards are based on well known and possibly simple methods. At the end one can find a list of references. The structure of ASTM standards is flexible and can differ in some elements from one standard to another.

Table 3

Selected standards for determining microstructure characteristics

Number of the standard	Title of the standard
ASTM E112 – 13	Standard Test Methods for Determining Average Grain Size (image analysis methods described in E1382)
ASTM E1382 – 97(2015)	Standard Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis
ASTM E562 – 11	Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count (image analysis methods described in E1245)
ASTM E45 – 13	Standard Test Methods for Determining the Inclusion Content of Steel (image analysis in E1245)
ASTM E1245 – 03(2016)	Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
ASTM E2109 – 01(2014)	Standard Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings
ASTM E1268 – 01(2016)	Standard Practice for Assessing the Degree of Banding or Orientation of Microstructures
ASTM E2283 – 08(2014)	Standard Practice for Extreme Value Analysis of Nonmetallic Inclusions in Steel and Other Microstructural Features
ASTM E1181 – 02(2015)	Standard Test Methods for Characterizing Duplex Grain Sizes
ASTM E930 – 99(2015)	Standard Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)
ASTM E2627 – 13	Standard Practice for Determining Average Grain Size Using Electron Backscatter Diffraction (EBSD) in Fully Recrystallized Polycrystalline Materials
ASTM A247 – 16a	Standard Test Method for Evaluating the Microstructure of Graphite in Iron Castings (image analysis methods described in E2567)
ASTM E2567 – 16a	Standard Test Method for Determining Nodularity and Nodule Count in Ductile Iron Using Image Analysis

It is obvious that automated methods are newer than manual ones. Similarly, standards devoted to image analysis were introduced later and, consequently, their numbers are higher, as can be seen in Table 3 (for example, E112 and E1382). However, this is not always the case. In some standards, for example E1181 or E1268, both types of measurements: manual and automatic are defined.

As shown in Table 3, most standard methods are devoted to analysis of grain size and nonmetallic inclusions. Quantification of other microstructural features is only partially covered by standard procedures or one observes a complete lack of standardization. In such a case, it is necessary to develop a new procedure that can be later added as a new standard. New procedures, usually apply image analysis and some guidelines for practice in this field can be found in dedicated literature [3, 4].

Metallurgy seems to be the most advanced in the standardization of microstructure characterization. However, other areas of application, like medicine, textiles or cement industry can be found among the standards (Table 4).

Table 4

Selected standards for microstructure characterization in areas different than metallurgy

Number of the standard	Title of the standard
ASTM D7879 – 13	Standard Test Method for Determining Flax Fiber Widths Using Image Analysis
ASTM E1695 – 95(2013)	Standard Test Method for Measurement of Computed Tomography (CT) System Performance
ASTM F1854 – 15	Standard Test Method for Stereological Evaluation of Porous Coatings on Medical Implants
ASTM C1356 – 07(2012)	Standard Test Method for Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point-Count Procedure

Test procedures in the ASTM standards are based on well-known and documented methods. Such a solution is safe; however, it can petrify small errors or uncertainties that have been corrected in more recent works. Fortunately, the ASTM standards are revised every few years and there is a possibility to significantly upgrade the procedure. The year of last revision is always a part of number of a given standard (see Tables 2–4).

4. Discussion

ASTM provides a very large set of 25,338 standards and publications devoted to materials. The subset related to test methods consists of 13,628 items. Such a huge collection makes finding the appropriate document difficult, even with the help of an electronic search system. When looking for a suitable document, one should keep in mind that access to full text documents is always paid. Nevertheless, ASTM provides the largest collection of documents related to microstructure evaluation. Moreover, many of them constitute a de facto world standard.

24 ASTM standards listed in this paper demonstrate the above mentioned wide range of documents that allow for the organisation of a laboratory that will be internationally accepted as a source of microstructural information.

In spite of enormous progress in image analysis methods, laborious and time consuming methods are still widely represented within the standard procedures. Within the automated methods, processing of the image leading to a binary image suitable for measurements is the weakest and practically not fixed point. The user has to evaluate their own procedures for feature detection and this step lies outside the standard. On the other hand, it is decisive for the final results. Therefore, it is recommended to perform interlaboratory tests in order to verify and fix the correctness of the elaborated procedure.

The ASTM standards give guidelines for test procedures but it is the user's responsibility to fix details and evaluate the whole procedure. Many details, especially concerning specimen preparation cannot be standardized due to the practically countless number of alloys, ceramics, plastics or composites. Fortunately, there are other sources of information which give answers to practical problems [5].

To summarize, the ASTM standards provide a huge set of information and rules suitable for microstructures evaluation. However, not all the problems are covered by these standards and many details have to be fixed on the basis of other sources of information or the user's own experience.

Miscellaneous

Numbers, names and basic information cocerning the contents of the ASTM Standards are available free of charge on the ASTM web site at www.astm.org.

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