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GUIDANCE AND ADVICE TO IMAGE ANALYSIS APPLIED IN MATERIALS SCIENCE

PRAKTYCZNE WSKAZÓWKI DO ANALIZY OBRAZU W BADANIACH MATERIAŁÓW INŻYNIERSKICH

A b s t r a c t

The paper summarizes selected examples from quantitative metallography of engineering materials and indicates potential errors which should be avoided. Problems in the areas of sample preparation (cutting, grinding, polishing and etching), microscopic observation, image collection, image processing, quantitative image analysis together with the interpretation of the results were shortly characterized.

Keywords: *metallography*, *sample preparation*, *image analysis*, *microstructure*

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

W artykule przedstawiono zestawienie wybranych przykładów z praktyki metalografii ilościowej materiałów inżynierskich wraz ze wskazaniami potencjalnych błędów, jakie można popełnić i jakich warto unikać. Scharakteryzowano pokrótce problemy, na które warto zwrócić uwagę podczas preparatyki próbek, akwizycji obrazu makro- i mikrostruktury, procedury przetwarzania obrazu, ilościowej analizy obrazu i interpretacji wyników.

Słowa kluczowe: *metalografia*, *preparatyka*, *analiza obrazu*, *mikrostruktura*

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1. Introduction

It is well known that the properties of materials are directly dependent on their structure. That is why it is reasonable to create a quantitative characterization of a structure which possesses desirable parameters. Microstructure generally consists of two or more phases with fundamentally different properties. The description of a material structure at the micro- and macro scale consists of size, shape and elongation of grains in the case of one-phase materials. The characterization of multiphase materials involves, besides grains description, a kind of phases, their volume fraction, distribution and position of each one in relation to others. In the case of both one- and multiphase materials, the depiction of their microstructure can also involve possible defects present in the materials, like nonmetallic inclusions, pores, cracks, etc.

Scientists can usually use only a part of a section taken through the sample of a given microstructure and then evaluate the measurable properties of the material (volume fractions, object number, etc.). This process is repeated on many sections in order to determine average values or other characteristics.

A typical analysis of a material structure requires the following stages [1]: preparation of the surface of the material (cutting, grinding, polishing and etching), microscopic observation and image collection, image processing, quantitative image analysis and interpretation of results.

Optical microscopes, scanning electron microscopes, transmission electron microscopes, computed tomography (CT) and magnetic resonance (MR) imaging are just some of possible methods applied to research of macro- and microstructure of materials. They enable to obtain 2D (two-dimensional) and 3D (three-dimensional) digital images of material microstructure [2]. The new diagnostic imaging methods (CT, MR, etc.) seem to be a real challenge for image analysis specialists. Obviously, results of image analysis are strongly dependent on the knowledge and experience of people carrying out the analysis.

Quantitative characterization with image analysis techniques is an important ingredient in predicting microstructure-property relations for heterogeneous materials.

2. Sample preparation

Proper preparation of metallographic specimens to determine microstructure and content requires that a rigid step-by-step process be followed. The final stages of metallographic sample preparation should produce a polished surface which will reveal the true microstructure. Each stage of metallographic preparation (sectioning, rough grinding, mounting, fine grinding, rough polishing and final polishing) is vital to the final result. Incorrect preparation can lead to an erroneous interpretation with potentially serious consequences. Mechanical polishing will always leave a layer of disturbed material on the surface of a specimen if the specimen is particularly susceptible to mechanical damage (or excessive force is used in the grinding and polishing stages) debris can become embedded in the surface and plastic deformation may exist below the surface. Ideally, there should be no scratches after polishing, but it is often hard to completely remove them all.

Electropolishing or chemical polishing can be used to remove this, leaving an undisturbed surface.

Microscopic examination and evaluation of the final surface of metallographic samples should be done before the next step of research activities. The presence of artifacts on the sample surface would lead to an incorrect interpretation of the image of analyzed particles after binarization (detection of carbides and partly scratches in Fig. 1b).

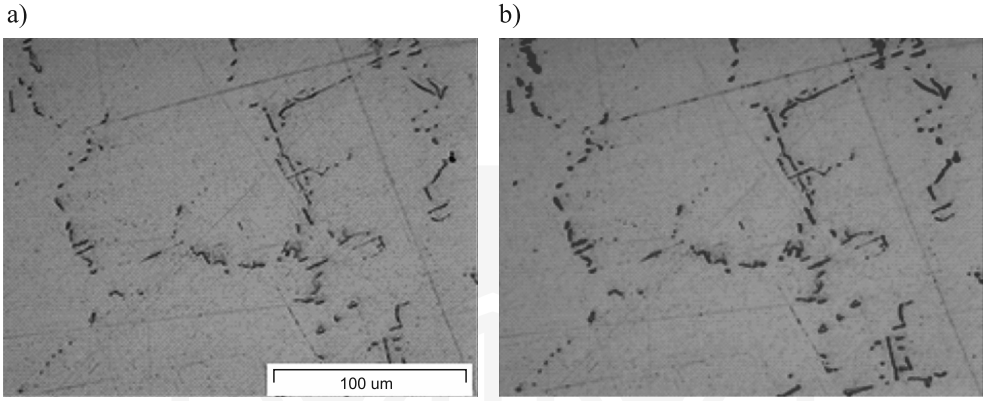

Fig. 1. Microstructure of Ni-based superalloy with carbides and visible scratches (preparation artifacts): a, b) LM, BF; c) LM, DF

To highlight surface defects, scratches or engraving a dark field illumination is mainly used. Examples of the microstructure image with scratches which are grooves in the surface of a sample, produced by the points of abrasive particles are presented in Figs. 1a, b. To enhance the contrast between the phases, different etchants and different etching methods can be used consecutively.

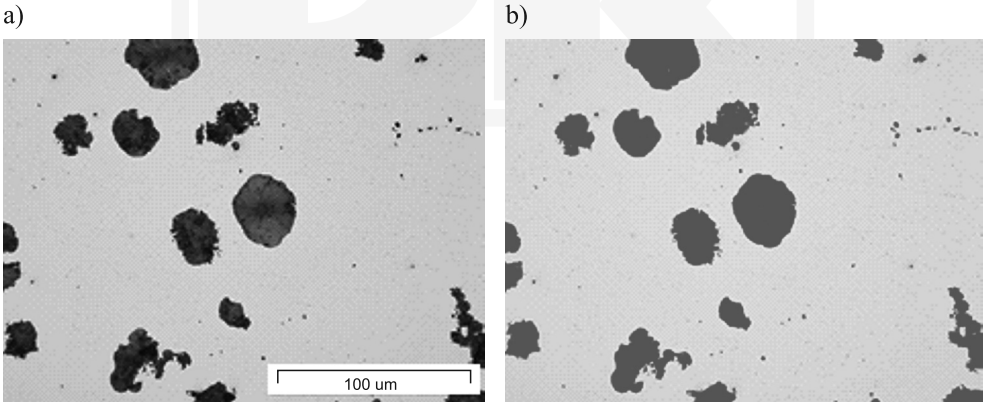

Fig. 2. Differences in the grey level of particles and background in a spheroidal grey cast iron: grey initial image, LM, BF (a) and binarization of graphite (b)

Polishing and etching of the investigated material often leads to significant differences in the intensity of light reflected from the parts of the area studied that are occupied by different phases. As a result, particles revealed on a cross-section appear to have a grey level distinguishing them from the background. Differences in the grey level of various phase constituents can be used to detect particles and conduct simple measurements on their geometrical features. An example is presented in Fig. 2.

Microscopic examination of a properly polished, unetched specimen will reveal only a few structural features such as pores, inclusions and cracks or other physical imperfections. Etching is used to highlight, and sometimes identify, microstructural features or phases present. Sometimes etching of a metallographic sample surface is not indispensable, because polishing of the sample surface makes it possible to obtain sufficient contrast required to perform selective detection of the investigated particles (example of pores in Fig. 3a, b). Some significant information about the position of each analyzed precipitate in relation to the others, and distribution of the precipitates in the microstructure could be missed exclusive of etching (Fig. 3c, d). Nevertheless, without etching the selective detection of some particles is possible (example of carbides in the Ni-based superalloy in Fig. 4).

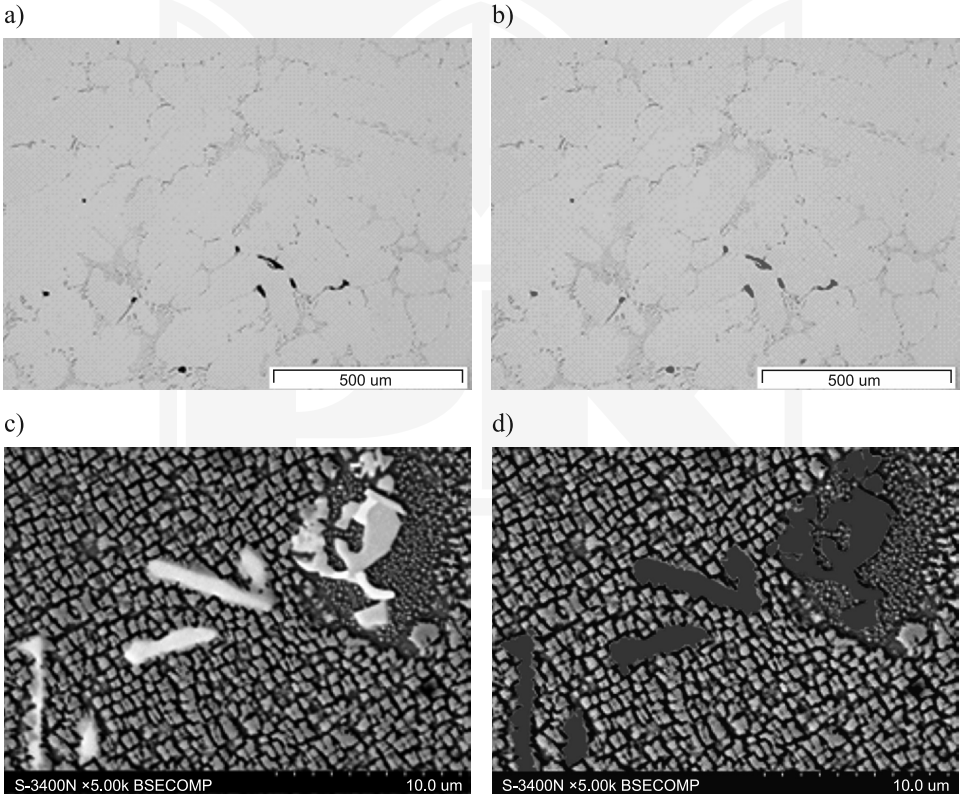

Fig. 3. Detection of pores on the unetched sample (a, b – LM, BF) and carbides on the etched sample (c, d – SEM, BSE) in Ni-based superalloy

3. Microscopic observation

Techniques of light (optical) microscopy (LM) and scanning electron microscopy (SEM) are mostly applied for the visualization of microstructures. Correct sample preparation process is not the only factor that can influence the measurement results of particles occurring in a material. The applied technique of observation and conditions of acquisition are significant for easier and less time-consuming image threshold and mathematical morphological operations (Fig. 4).

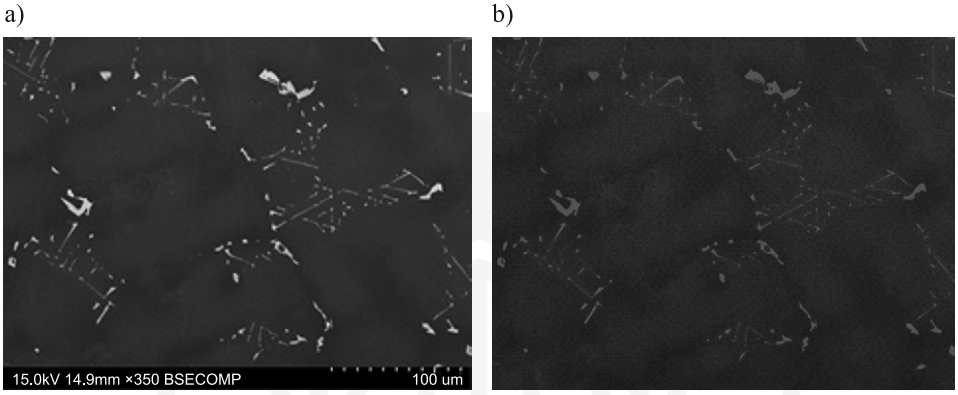

Fig. 4. Carbides precipitates in the Ni-based superalloy at 350×: grey initial image, SEM, BSE (a) and binarization of carbides obtained by means of an image analysis program (b)

The most common imaging mode collects low-energy ($<$ 50 eV) secondary electrons that are ejected from the *k*-shell of specimen atoms. Backscattered electrons (BSE) consist of high-energy electrons originating in the electron beam that are reflected or back-scattered out of the specimen interaction volume. Since heavy elements (high atomic number) backscatter electrons more strongly than light elements (low atomic number), and thus appear brighter in the image, BSEs are used to detect contrast between areas with different chemical compositions. Contrast changes in images taken in scanning electron microscope result from a change in accelerating voltage, spot size (probe current) and tilt angle.

4. Image processing

The term image processing is used to describe operations that are performed on images of microstructures in order to correct them or to make them more accessible for quantitative analysis. Image processing consists of the following steps: image acquisition, digital processing, threshold operations, mathematical morphological operations, manually made corrections (if they are necessary) and measurements. It is a concept based on digital images. Opportunities for image transformations and for measurements depend on the applied software [3].

Image segmentation is an essential task in the fields of image processing and computer vision. It is a process of partitioning of digital images and is used to locate the boundaries into a finite number of meaning full regions which are easier to analyze [4]. The simplest method for image segmentation (enhancement and object detection) is thresholding. The output of the thresholding process is a binary image whose grey level value 0 (black) will indicate a pixel belonging to a print, legend, drawing or target and a grey level value 1 (white) will indicate the background [5].

Preparation of an image representing such a microstructure is a very important factor of a reliable description of a real microstructure. Note that samples of microstructure are usually taken by means of microstructure sampling and converted into the electronic form. The characterization of microstructures usually requires an analysis of a large number of complicated images with each of them containing microstructural elements required as well as some artifacts produced by the imaging technique. This justifies the need for automation and explains the reasons for computer-aided procedures in the characterization of materials.

5. Quantitative image analysis

Modern computer aided methods used for the quantitative description of microstructures take advantage of the progress made in recent years in the field of image processing, mathematical morphology and quantitative stereology [5]. One of the most important issues is a selection of proper magnification for quantitative image analysis. It is essential to take into consideration the magnification at which the images for this analysis were registered. The examples presented in Figs. 4 and 5 can help to understand the effect of applied magnification on the obtained results of quantitative analysis. The example of carbide image registered at 350 times magnification is presented in Fig. 4. In this case, the area fraction

a) b)

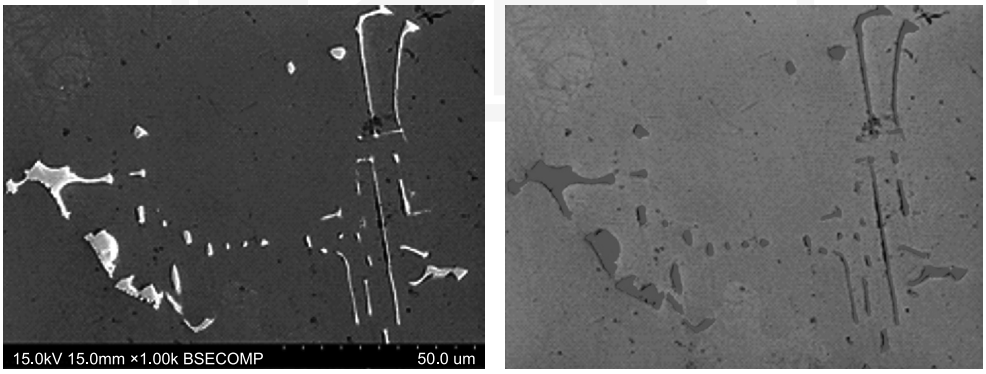

Fig. 5. Carbides precipitates in the Ni-based superalloy at 1000×: grey initial image, SEM, BSE (a) and binarization of carbides obtained by means of an image analysis program (b)

of carbides is equal to 2.28%. On the same material sample, another image with carbides was registered using 1000 times magnification (Fig. 5). At higher magnification you obtain the result of area fraction for carbides amounting to 3.66%. This result is overestimated.

6. Interpretation of results

Each quantitative analysis of a material microstructure should end with proper interpretation of the obtained results. A relative error of the carried out measurements should be estimated. Moreover, a minimal number of measured fields of view should be estimated on the basis of a statistical test (for example the goodness of fit Kolmogorov-Smirnov test of distributions). In the similar way, the repeatability of results of the obtained measurements for selected parameters should be tested.

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