Quantitative metallography of two-phase titanium alloys

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Abstract

The paper presents the results of investigations of the two-phase Ti–6Al–2Mo–2Cr–Fe alloy microstructure by means of different methods: special techniques of light and scanning electron microscopy. It has been found that for the evaluation of the grain size and lamella colonies of the α phase, an observation in polarized light with a lambda filter gives the best results. The algorithm of the image transformations enabling detection of boundaries of the lamella colonies is presented. It employs color image separation into basic colors according to the CMYK model. Also, a fully automatic way is presented for the preparation of images obtained by means of a scanning electron microscope for quantitative analysis. © 2001 Elsevier Science Inc. All rights reserved.

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1. Introduction

Owing to their exceptional properties such as: good specific strength, creep resistance and corrosion resistance in the majority of aggressive media and biological inertness, titanium alloys are widely used in aeronautics, astronautics, chemical industry, food industry, power industry and medicine [1]. Depending on their chemical composition, titanium alloys have α, α+β or β microstructure. Predominately, parts are manufactured from two-phase alloys α+β. In titanium alloys, particularly in two-phase ones, there is a great diversification of the microstructural morphology: the grain size and substructure, which is controlled by the chemical composition, methods and conditions of melting, crystallization, plastic working and thermal treatment. The microstructures of titanium alloys in relation to the morphology of their components can be classified into two groups:

- fine-grained microstructure of α and β phase for single-phase alloys or their mixtures for two-phase alloys,
- coarse-grained microstructure of polyhedral grains of α or β phase for single-phase alloys or ones characterized by colonies of a lamellar α and β phase placed within the boundaries of the big grains of the former β phase for the two-phase alloys [1,2].

The analysis of the literature [3–7] shows the complexity of issues concerning the microstructure–mechanical properties relationships in titanium alloys. The variety of microstructures obtained in titanium alloys under the influence of technological parameters shows extensive possibilities of changes in their properties. A high level of mechanical proper-
ties of titanium alloys can be reached providing that it is possible to obtain a homogenous fine-grained microstructure. The necessity of grain size reduction creates a number of difficulties during the processing of semifinished products from titanium alloys. Irreversible changes of the microstructure (in the form of an excessive grain growth) present in the case of exceeding the allotropic transformation temperature during plastic working or thermal treatment are the main problems.

An analysis of the published quantitative relations between the mechanical properties and the geometrical characteristics of the titanium alloy microstructures leads to the conclusion that the basic parameter hitherto used for the grain size description has been the mean grain diameter [8]. Currently it is thought that a few parameters determined using a surface method describe the microstructure in a better way [9,10]. Hence one may state that the development of optimum methods and criteria of microstructural shaping must use quantitative descriptions of the microstructural properties. Only stereological parameters determined by an image analysis and quantitative metallography allow one to compare objectively the material microstructure after different technological processes and to look for relationships between the microstructure and the mechanical properties of the alloys.

2. Material

A Ti–6Al–2Mo–2Cr–Fe titanium alloy with the two-phase $\alpha + \beta$ type microstructure was used in the experiments. The chemical composition of the alloy is presented in Table 1. In order to obtain a homogenous coarse-grained initial microstructure the alloy was subjected to preannealing at 1273 K for 1 h and then it was furnace cooled after annealing. That type of annealing resulted in forming an initial material with a mean grain equivalent diameter equal to about 0.5 mm. To diversify the content of the structural components and the microstructural parameters the specimens were annealed isothermally for 1 h at temperatures over the range of 1073–1243 K. After annealing the specimens were cooled in air. Specimens for the microstructural examinations were prepared by electrolytic polishing and etching in Kroll’s reagent.

3. Experimental procedure

The $\alpha + \beta$ two-phase titanium alloys are characterized by a granular microstructure consisting of grains of the former $\beta$ phase on the boundaries of which there is an $\alpha$ phase network (Fig. 1). Inside the grains there are colonies of $\alpha$ phase lamellae, nucleating in the phase transformation process, mainly on the grain boundaries. The lamellae inside the colonies are placed in a parallel way, forming the texture within the grains. A complete quantitative description of this type of microstructure should include data about all three elements: grains, colonies and lamellae. Due to considerable differences in the size and lack of features unambiguously distinguishing these microstructural elements from one another, it is not possible to apply one observation technique to determine the stereological parameters of grains, colonies and lamellae of the phases $\alpha$ and $\beta$ simultaneously. Detection based on the image texture [11,12] is also impossible, because at magnifications at which colonies and grains are visible, lamellae cannot be differentiated. In this paper, for the evaluation of the sizes of grains and colonies, images obtained by means of special techniques of light microscopy were used, whereas the quantitative characterization of the $\alpha$ phase lamellae was carried out on images obtained by means of scanning electron microscopy.

The initial examinations of the Ti–6Al–2Mo–2Cr–Fe alloy microstructure by means of light microscopy showed that the contrast between the $\alpha$ phase lamella colonies was too small for image analysis methods to be used (Fig. 2a). One may try to achieve improvement of the contrast through the application

![Fig. 1. Diagram of microstructure of two-phase (\(\alpha + \beta\)) titanium alloys.](image_url)
of special techniques of light microscopy, e.g. by observation in dark field or polarized light. The methods of observation in polarized light can be applied when the material examined is characterized by optical anisotropy. This phenomenon occurs in the alloy investigated as a result of the lamellar structure of the \( \alpha \) phase colony. Observations carried out in dark field gave better results than in bright field (Fig. 2b), whereas good results were obtained only during the observation in polarized light (Fig. 2c). By changing the setting angle of the polarizer against the analyzer during the observation in polarized light it is possible to obtain images on which the same microstructural element has a different gray level. However, for image analysis needs, recording at least several images of the microstructure from the same place on a specimen is required. Applications and transformations of the images obtained this way are described in the literature [13].

The difficulties connected with the necessity of recording many images can be eliminated thanks to the application a lambda filter during the observation in polarized light (Fig. 3). The microstructural image obtained in this way is in pseudocolors. Such an image can be subjected to color separation according to the selected model of colors (e.g. RGB, CMYK, HSB and HLS). The investigations of the titanium alloy microstructures showed that the best results are achieved using the CMYK model (C = cyan, M = magenta, dark field gave better results than in bright field (Fig. 2b), whereas good results were obtained only during the observation in polarized light (Fig. 2c). By changing the setting angle of the polarizer against the analyzer during the observation in polarized light it is possible to obtain images on which the same microstructural element has a different gray level. However, for image analysis needs, recording at least several images of the microstructure from the same place on a specimen is required. Applications and transformations of the images obtained this way are described in the literature [13].

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Fig. 4. Algorithm of image transformations using colors separation.
Y = yellow, K = black). After separation into basic colors in accordance with this model, four gray images are obtained with greater difference in contrast than in images recorded only in polarized light (Fig 4).

The image transformations for measurements of the α phase lamella colony size included: smoothing of the initial color image, separation into four constituent gray images (CMYK), edge detection (gradient), thresholding, removing of fine elements of images and adding of four binary images (Fig. 4). The algorithm presented enables appropriate detection of about 80% of the colony boundaries. Unfortunately, fully automatic analysis and the measurement of the α phase lamella colonies is not possible. The final image requires manual corrections based on filling parts of the colony boundaries or removing needless boundaries. Algorithms of the grain boundary reconstruction in the case of the microstructure in question do not yield good final results because of the complex shape of colonies.

Due to the required magnification range, the image acquisition for the evaluation of the Ti–6Al–2Mo–2Cr–Fe alloy grain size was carried out by means of an Olympus SZ×9 stereoscopic microscope with an attachment for work in polarized light. The grain size was evaluated in binary images obtained in the same way, from which boundaries not being grain boundaries (a boundary between colonies is not always a grain boundary, but the grain boundary is always a colony boundary) were removed manually.

The quantitative characteristics of lamellae was performed on the images from a scanning electron microscope. These images are characterized by a considerable contrast between the α phase lamellae and the β phase matrix, which significantly facilitates a qualitative characterization of the α phase. However, the preparation of the images for measurements requires the removal of the noise occurring mainly in the β phase region.

The image transformation before the measurement included: smoothing by a mean filter, histogram normalization, thresholding and removing fine image elements (skeletonization, cutting skeleton ends and reconstruction). The image transformation scheme is presented in Fig. 5.

In many studies concerning the analysis of the relationships between the properties and parameters of titanium alloy microstructures (e.g. [8,14]) the authors limit themselves to measuring the mean grain diameter. This constitutes a considerable simplification of the microstructural description. In the present paper, the binary images obtained using the described research methodology enabled a comprehensive microstructural evaluation.

Fig. 5. Diagram of the transformation of images from a scanning electron microscope.
minimum diameter, Feret diameter along the $x$ and $y$ axes, elongation as the ratio of the maximum to minimum diameter, mean volume for grains and lamella colonies calculated on the basis of equation presented in Refs. [9,10], mean equivalent diameter, shape factor, specific surface, $S_v$. Complete research results and their detailed analysis are presented in Ref. [15].

4. Final remarks

Image analysis of a titanium alloy microstructure is a complex problem. The investigations conducted in this study showed that the quantitative evaluation of the microstructure present in two-phase titanium alloys required the application of different methods and research techniques: light microscopy using special techniques and scanning electron microscopy. The best effects were achieved by making observations in polarized light with a lambda filter. The image transformation algorithms presented speeds up and facilitates preparation of images for quantitative measurements. However, fully automatic image analysis of titanium alloy microstructures obtained by light microscopy is not possible. In case of the $\alpha$ phase lamellae with images from scanning electron microscopy the measurement procedures can be made fully automatically.

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