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Association of polarized light metallography with quantitative image analysis for the characterization of microstructures

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Some alloys, such as titanium alloys, exhibit microstructures difficult to etch and consequently difficult to characterize quantitatively. A new method is presented to solve such problems, consisting of associating polarized light microscopy with the techniques of image analysis. The problems set and the solutions obtained by the method are discussed in the case of acicular α-titanium microstructures. The method can also be applied to other optically anisotropic materials.

1. Introduction

Quantitative metallography is currently used for the characterization of microstructures and has been proved to give reliable and precise results [1, 2]. One of the main difficulties with such analyses is that of obtaining an image with sufficient contrast by an appropriate etching method. Further treatment of the image relies on stereology. Some alloys, such as zirconium, uranium or titanium alloys are, however, difficult to etch and the resulting microstructures cannot give satisfactory digitalized images because their contrast is too low which prevents any correct threshold being reached. Such microstructures, e.g. of titanium alloys, have nevertheless been characterized but essentially qualitatively and with a vocabulary which is neither precise nor coherent from one user to another. These microstructures are furthermore numerous and very different, depending on the specimen history, i.e. the previous thermal and thermomechanical treatments. Qualitative characterization is consequently not sufficient to distinguish precisely and unequivocally two microstructures.

Optical methods such as polarized light or differential contrast in polarized light are currently used in metallography to enhance the contrast. Such methods allow the details of a microstructure to be distinguished with a criterion depending on the structural orientation. The observation in polarized light is, however, associated for the first time in this paper with image analysis to obtain a quantitative characterization of microstructures. In the first part of this paper, the basic concepts of polarized light microscopy and its applications in metallography are recalled and we show how it can be associated with image analysis to improve considerably the characterization of some metallic alloys. In the second part, the method devised is detailed and applied to titanium alloys which exhibit complex acicular microstructures. Such a method can, however, be extended to any alloy containing optically anisotropic phases.

2. Use of polarized light in metallography

2.1. Basic concepts of polarized light microscopy

The vibration directions of light are generally perpendicular to the direction of its propagation. Under given reflection conditions or when passing through given crystals, several vibration directions may be suppressed and others specifically favoured. Light is said to be linearly polarized if only the vibrations in a given plane subsist [3, 4]. Because human eyes do not distinguish polarized and normal light, a system for the observation in polarized light must be composed of a generator of polarized light (the polarizer) and a detector of polarization (the analyser). If the polarization planes of the analyser and the polarizer are perpendicular, no light will be transmitted and this situation is called "polarizer and analyser crossed". Light is integrally transmitted if their planes are parallel. The transmitted light intensity for any orientation is given by the Malus law [3].

Materials are generally optically classified into the two following groups.

(i) Isotropic materials: the velocity of light propagation in such materials is independent of its direction. The reflection coefficient is consequently independent of the polarization plane of the light.

(ii) Anisotropic materials: in such materials the velocity of light propagation depends on its direction. The reflection coefficient depends on the surface orientation and on the polarization plane of the incident light.

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Cubic crystals are isotropic materials whereas quadratic and hexagonal ones are anisotropic.

In opposition to isotropic media, the reflection index, n, of an anisotropic medium varies with the propagation direction of the light. The plot of n against the propagation directions forms what is called the indices ellipsoid. The three perpendicular axes of this ellipsoid correspond to the minimum, maximum and average index, respectively. These three values are called the principal indices of the anisotropic medium and correspond to crystallographic axes of the material.

Some anisotropic materials are called birefringent, meaning that a light ray emanating from an isotropic medium is divided into two refracted and 90° polarized rays on entering the anisotropic medium. For crystals having crystallographic axes of symmetry greater than two, the indices ellipsoid is of revolution around these axes which are then called optical axes. Crystals with one optical axis are called uniaxial crystals. If the incident light is parallel to the optical axis, the ellipse of the indices is circular and only one ray is refracted. The optical axis of uniaxial crystals is consequently their unique direction of unirefringence.

Polarized light can consequently be used to determine the orientation of uniaxial anisotropic materials in the following way: if the polarizer and the analyser are in crossed position, no light is transmitted. If a material which has the property to rotate the polarization plane is inserted between polarizer and analyser, the analyser and the polarizer will have to be turned through a given angle before light is again cut off completely. Accordingly, with the polarizer in the crossed position, grains of a polycrystalline aggregate should appear more or less bright according to their orientation and to their amount of optical anisotropy. Starting with one set of grains dark, the angle through which the stage has to be rotated to bring another set into extinction is a measurement of the crystallographic disorientation between the two sets [5, 6].

2.2. Application to metallography

Polarized light has been currently used in geology to identify rapidly the different components and phases existing in a material by the precise measurements of the refractive indices [4, 7]. In metallography, polarized light has been widely used for the identification of grains having parallel basal plane in zirconium, hafnium, beryllium, uranium and titanium alloys [5, 6, 8–14]. Most of these authors have reported crystallographic investigations in correlation with measurements, or only observations, in polarized light and have shown the precision of this optical method to determine structural relationships between or inside grains. This optical technique was, however, abandoned at the end of the fifties, probably because of the progress and extension of the possibilities of electronic transmission microscopy. Polarized light has been brought back into fashion by Nauer-Gerhardt and Bunge in recent papers which show the accuracy of such a technique in determining structural orientations in titanium [7, 15].

2.3. Application of polarized light to image analysis

Some metals such as uranium are difficult to etch [11]. For other materials, such as titanium alloys, the etching solutions currently used give micrographs satisfactory for the illustration of the microstructures, but of too low a contrast for a correct quantitative analysis. Improvement of the etching solution could be one way to enhance the contrast but the probability of success is low. The photograph quality could also be improved or the principal lines of the micrographs reproduced with black ink on a transparent paper in order to obtain artificial, but very contrasted, images. The direct observation of specimen surfaces must anyway be preferred to such artificial methods which result in a loss of information through the photography as well as through the drawing.

Polarized light is a better performing method, which realizes directly on the surface of the specimen a considerable enhancement of the contrast by transforming a microstructure with grey tones into an image with a few very distinct colours. Each of the different colours corresponds to a family of grains the basal planes of which have the same orientation as the direction of the incident light. Polarized light consists, in fact, of a first threshold of the microstructure but with a structural criterion. The digital threshold of the optical signal transmitted from the image, which is performed by the image analyser is based on a light intensity criterion. The distinction between the phases is, therefore, based only on the difference in their contrast and brightness, and not really on the difference in their structural characteristics.

Accordingly, the threshold realized by polarized light allows us to first classify, with different colours, the grains or the colonies of lamellae having identical basal planes. The different colours can be further separated using the digital threshold in order to perform a quantitative analysis independently for each family of grains or colonies appearing with the same colour.

3. Quantitative metallography in polarized light and its application to titanium alloys

3.1. General method of analysis

A general method is proposed for the characterization of microstructures which can be revealed by polarized light, such as anisotropic uniaxial materials. This method will be detailed in the case of the complex acicular alpha microstructures of titanium alloys obtained after quenching from the β phase. Examples will be given for TA6Zr5D and TA6V alloys. The process chart of the general method is represented in Fig. 1. The method specifically used for titanium alloys is given in Section 3.3.

The specimen is installed on an optical microscope and an area is chosen on the surface. The appropriate magnification is set in order to obtain the correct level of observation and meaningful parameters, e.g. an overall view of the inside of a grain for the characterization of the microstructure or a higher magnification

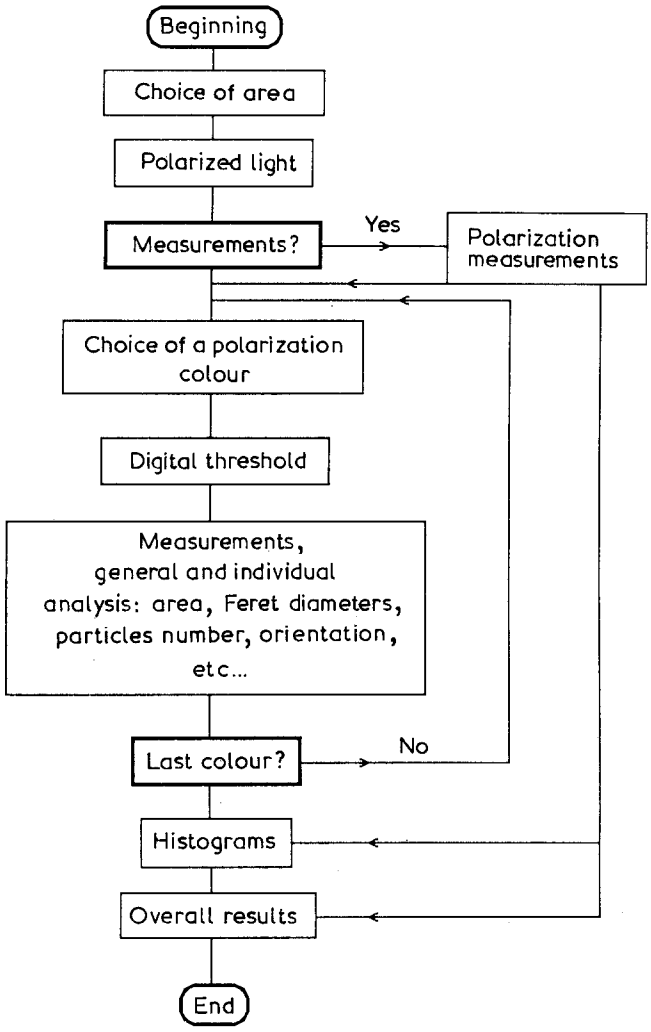


Figure 1 Process chart of the general method.

for the measurements of the lamella sizes. Polarized light is then used to enhance the contrast and to reveal the details of the microstructure. Grains or colonies having identical structural orientation appear with the same colour. Such an image, with high contrast, is ready for ordinary quantitative metallography. At this point, disorientation measurements can also be performed with adequate polarization equipment. Such measurements will indicate the structural relationships existing between different sets of colonies or grains.

One of the different polarization colours is then separated by digital threshold in order to be analysed independently. This separation can be manual, or automatic if the analyser is equipped for several grey levels. The digitalized image which is obtained after separation, corresponds to one polarization orientation and can be further transformed with appropriate morphological operations to enable the measurement of parameters such as the area, number of particles, particle dimensions or their orientation. The data obtained are stocked in files for further treatments and another polarization colour is chosen for analysis.

When the whole image has been treated, several histograms can be calculated from all or a part of the data. Such histograms and other overall results can be further correlated with the polarization measurements and with the evolution of the material during thermal or thermomechanical treatments.

3.2. Problems met in the quantitative metallography of titanium alloys

Titanium alloys belong to materials for which there exists only a qualitative characterization of the different microstructures, with moreover a non-precise vocabulary. The acicular α microstructures have been chosen because of their complexity and consequently the difficulty to characterize them.

We have distinguished the following three levels of observation in order to separate the different parameters to be measured.

1. An overall observation of the grains distribution is necessary to measure the size of the original β-grain, which is an important parameter in the transformation process and for the mechanical properties.
2. The observation of one grain separately is appropriate to characterize the microstructure by the distribution of the α colonies and their relative orientations.
3. A higher magnification would be necessary to focus on an α colony in order to measure some dimensions of the α lamellae more precisely. These parameters are important in the evolution of the material during thermal and thermomechanical treatments.

In this paper, we have principally applied the method at the second level of observation, inside a grain, for the following reasons.

(i) The application of polarized light to distinguish between several families of similar orientation is more successful at this level of observation.

(ii) This level of observation gives more numerous parameters to characterize the microstructures.

(iii) The existing micrographs of the inside of a grain can be very complex and their analysis was until now mostly qualitative.

(iv) Once the method was set, its generalization to the two other levels of observation could be easily considered. The success of the first one is strongly dependent on the chemical etching of the specimen and the third one can be achieved with a scanning electron microscope equipped with image analysis.

As previously said, titanium alloy micrographs which are available in the literature [16] are satisfactory as microstructure illustrations but it is very difficult to perform a correct threshold on such images in order to analyse in one grain the different α lamellae obtained after quenching. We will give two examples of the problems met when attempting to analyse quantitatively these microstructures.

1. Fig. 2a and b, represent two micrographs of the same area as observed with an optical microscope. Fig. 2a represents the original microstructure and is very similar to other typical microstructures of titanium alloys called Widmanstätten microstructures. Fig. 2b represents the artificial etching of Fig. 2a. The colonies of parallel α lamellae have been schematically represented, with several parallel straight lines in order to give simply their orientation. Ink etching was the only way to use micrographs similar to that of Fig. 2a for image analysis. However, such transformation is obviously too long for an automatic characterization.

2. On another observation level, Fig. 3 illustrates the difficulty of distinguishing β-grain boundaries on microstructures of titanium alloys even if the macrostructure appears well defined. The two artificial etchings correspond to two possible criteria for the apparent definition of β-grain boundaries. They symbolize two digitalized images which would be interesting to obtain after threshold by the image analyser in order to analyse the size distribution of the original β grains. The granulometries calculated from these two images would give completely different results. The definition of grain boundaries is a problem currently met in image analysis. The lack of contrast of the microstructure of Fig. 3a would result, in total ambiguity, in a digitalized image similar to Fig. 3b or c. This second example is quite significant and leads to two remarks: (i) image analysis cannot give more results than those contained in the image which is used. In certain cases, such as ceramics, some complex methods have nevertheless been set to solve this problem [17]; (ii) artificial etching methods must be used very carefully. For example, some authors have proposed etching methods including heat treatments in order to well define the β grains by the precipitation of another phase at the boundaries [18–20]. The results are satisfying but the material structure will no doubt be altered by the heat treatments.

The two examples of Figs 2 and 3 illustrate the difficulties met in the quantitative characterization of the microstructures of titanium alloys. We will now show that the association of polarized light with image analysis can solve a great many of these problems.

3.3. Analysis programme for titanium alloys with acicular α microstructures

The α phase of titanium is hexagonal compact, and consequently belongs to anisotropic uniaxial materials which react to polarized light. Polarized light has been largely used between 1950 and 1960 to investigate the microstructures of titanium alloys [6, 8, 13, 14]. Several authors have used polarized light alone and in correlation to other crystallographic techniques to determine the structural orientations in such microstructures, e.g. the orientation of the basal planes of the α-colonies.

In one original β grain, the α lamellae which are formed follow the characteristics of the martensitic transformation, i.e. given relationships between the original β grain and the α lamellae formed are observed. For these alloys, the following Burgers relationships have been determined [13, 21–23]

$$\{1\,1\,0\}_\beta \parallel \{0\,0\,0\,1\}_\alpha \quad (1)$$

$$\langle 1\,1\,1\rangle_\beta \parallel \langle 1\,1\,\bar{2}\,0\rangle_\alpha \quad (2)$$

Consequently, the basal planes of the hexagonal acicular α phase are parallel to the $(1\,1\,0)_\beta$ planes of the

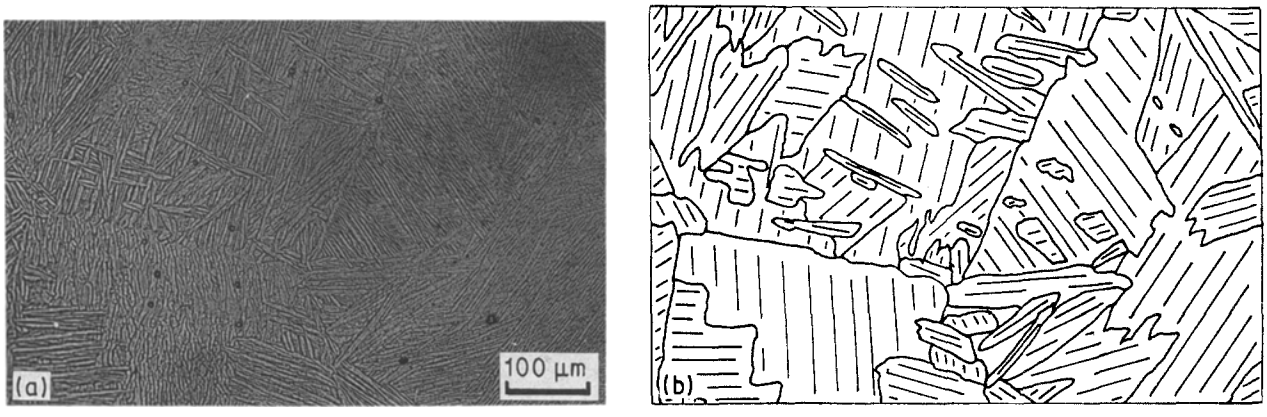
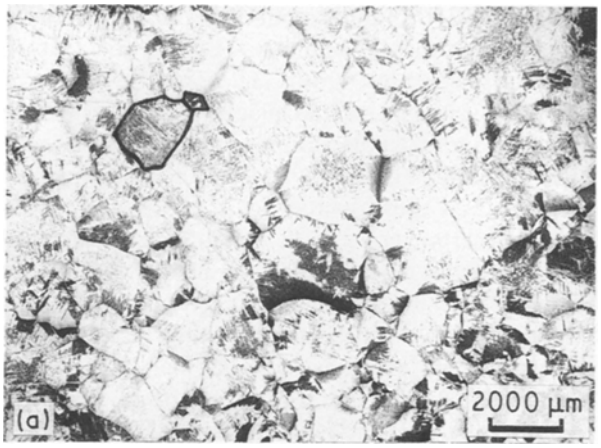
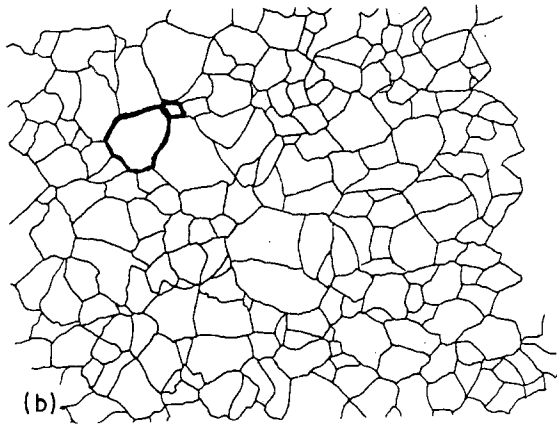


Figure 2 Illustration of artificial etching in order to reveal the α colonies and their schematic orientation. (a) Original microstructure of a TA6Zr5D alloy. (b) Ink etching of (a).





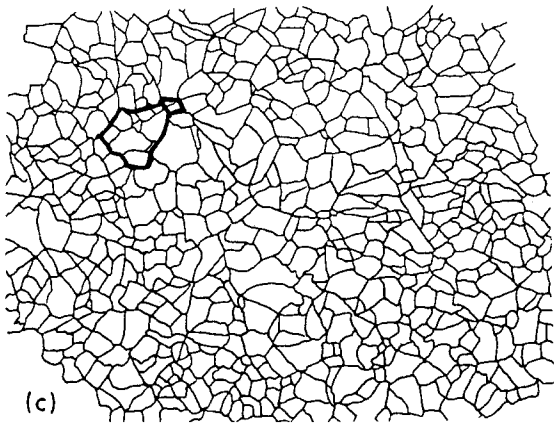


Figure 3 Illustration of artificial etching in order to obtain initial β grain size. (a) Original microstructure of a TA6Zr5D alloy. (b, c) Ink etchings of (a) corresponding to two different criteria for the definitions of the grain boundaries.

body centred cubic (b c c) crystalline structure of the initial β grain. These compact $\{1\,1\,0\}_\beta$ planes are either parallel or oriented at 60° or 90° to each other. Each of these planes results in two different variants for the α phase corresponding to the two possible directions of the relationships (2). Twelve orientations are consequently equally possible for such $\langle 1\,1\,\bar{2}\,0\rangle_\beta$ directions. Williams *et al.* [13] have noticed that after surface etching, α colonies with nearly parallel basal planes are undistinguished by polarized light and consequently appear with the same colour. Polarization and crystallographic investigations which have been performed in conjunction with such observations have revealed that the six-fold axes of two α colonies of nearly parallel basal planes can be oriented at 10°32′ from each other. Such an orientation difference results from the two different $\langle 1\,1\,1\rangle_\alpha$ directions in the initial β grains. A recent study by Bowen and Clark [24] reports the different orientations of α colonies *c*-axes measured by crystallographic techniques. Such measurements confirm the observations of Williams *et al.* These measurements represent the same structural orientation determinations that can be performed with polarized light. The precision of polarized light for such structural investigations has also been underlined for titanium in a recent paper of Nauer-Gerhardt and Bunge [15].

Fig. 4 shows an example of images which could be obtained with polarized light on a titanium alloy with acicular α microstructure. This image has been obtained with the same specimen as the micrograph in Fig. 2. A coloured filter has also been used in order to enhance the contrast between the three colours that can be observed. Such colours result from the different orientations between the $\{1\,1\,0\}_\beta$ planes in the initial β grain. Such microstructures can be perfectly quantitatively characterized, directly from the surface of the specimen, with the method presented here.

Fig. 5 shows the process chart of the analysis program that has been used to apply the method to titanium alloys with acicular alpha microstructures. The program illustrating the method has been divided into six independent modules for the following reasons:

1. Such organization allows a rapid modification of only one part of the program independently. It allows, furthermore, an easy adjunction of another module for additional measurements or operations.
2. Such organization allows a perfect portability to other programming languages or other types of analyser. Particularly, this organization in modules is very well adapted for structured and efficient languages such as C or Pascal.
3. Because of the previous remark, we will not describe in detail the equipment nor the programming language which have been used to set up this program

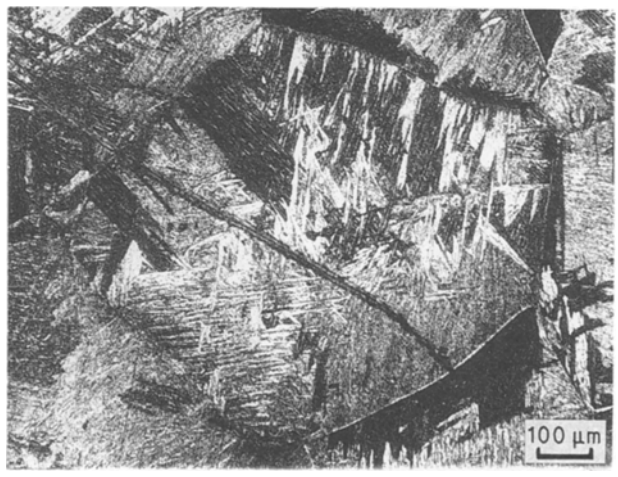


Figure 4 Microstructure of TA6Zr5D alloy as observed with polarized light.

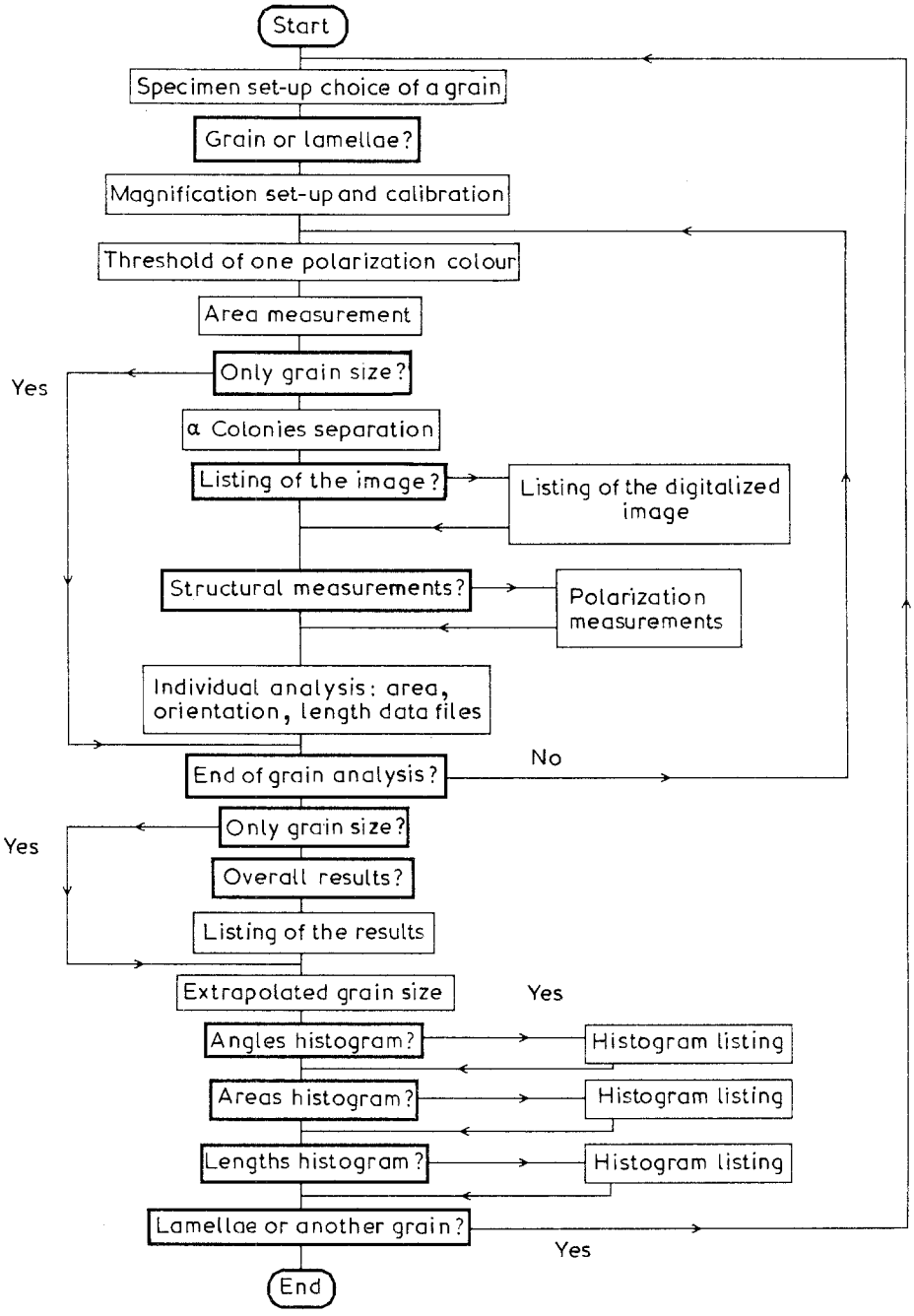


Figure 5 Process chart of the method applied to titanium alloys.

of application of the method to titanium alloys. The available memory on the analyser we have used was quite limited, but the organization into six modules has allowed compensation for this lack of capacity.

For the above three reasons, we will here present and discuss only the efficiency of the method and the application of the results that can be obtained with the program. The method is at the present time being practically applied to follow the variations of several characteristics of some metallic alloys during thermal and thermomechanical treatments.

The method applied to titanium alloys and illustrated by the process chart in Fig. 5 can be detailed as follows. After the specimen has been set up, a grain is chosen in an interesting surface area. Polarized light enables easier recognition of grain boundaries by the change in colour and orientation of the α colonies at the boundaries. Once a grain has been chosen and the appropriate magnification set so that the whole grain is viewed in the analyser screen, one polarization colour is separated by digital threshold and the area of the resulting image is measured. Such a digitalized image represents α colonies of parallel basal planes. A first quantitative analysis can be realized. If necessary, colonies of parallel α lamellae can then be separated, e.g. with the light pen functions. Such an operation is the only one which should be manually performed, apart from entering some data from the keyboard. Although particle separation is a classical stereological problem, it has not been possible to apply the existing programs found in the literature [25] to the separation of α colonies. The manual separation can be rapidly and easily performed by switching several times between the optical and the digitalized images. Fig. 6 represents an illustration of the images that can be obtained after such separation. The three images correspond to the three polarization colours observed

Figure 6 Digitalized images obtained after manual separation of colonies of α-lamellae for the three colours of polarization as observed in Fig. 2.

in Fig. 2. Individual analysis is further performed on such images and for each colour, its area, the maximum Feret diameter and its orientation are measured. The orientation is obtained by measuring the Feret diameters at 0° and 90° and by calculating the angle of the α colony with the X-axis by its sine or cosine depending on the compared values of the three Feret diameters. In order to minimize the errors in the direction measurements, we have used sine or cosine function by selecting the maximal Feret diameter in the 0° or 90° direction, respectively. All the data are kept in files for further treatments. When the images for the three polarization colours have been analysed, the overall results and the histograms can be calculated and listed.

The same programme can be used for different purposes. On the analyser that has been used, and with the available programming language, this possibility to choose between options has been realized with several tests. For a more structured and efficient programming language such as C, such a possibility would be realized naturally by a set of individual functions used or not in a main program. These options correspond to the three levels of observation as follows.

1. The program can be used for only β grain size determination. In this case, for the three polarization colours, only the area of the digitalized image is measured. No colony separation nor individual analysis is performed. The whole grain area is then extrapolated by adding the three values for the three polarization colours. Such β grain size measurement is not rapid and would need many measurements to obtain statistically valuable measures. However it allows an estimation of the β grain size and location of such measurements and/or the analysed grain within the β grain size distribution that can be achieved with other current techniques.

2. Lamellar analysis can also be performed with the program: in this case a test at the end of the program allows one to go back to the beginning and a higher magnification must be set. The same operations and measurements as previously described are then performed without polarized light. For a more rapid execution, a supplementary module can, however, be added after the test. Such a module would include the set-up of a high magnification and only a few new

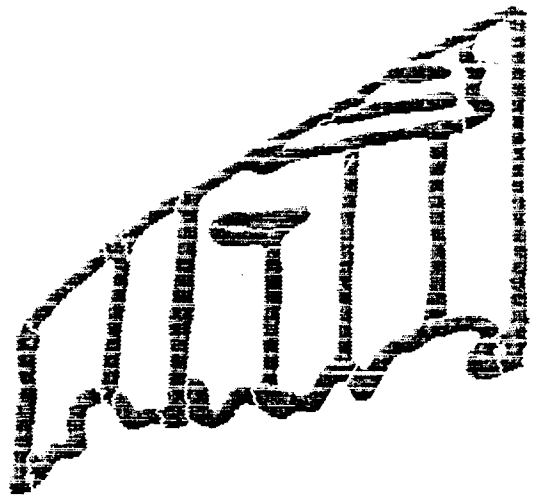


Figure 7 Ideal digitalized image of a colony of α lamellae obtained after ink etching.

operations (such as the mean chord measurement), if a digitalized image, as in Fig. 7, is to be obtained.

3. At the end of the program, the test can also be used to go back to the beginning and analyse another grain. Such a possibility can be used to study the comparative disorientations and the sizes of the α colonies in the near-neighbours of a given grain.

We have detailed up to now the concepts that have been used to set up the method and the program for its application to the microstructures of titanium alloys. The following part will present some quantitative results which have been obtained either with artificial images or directly from specimen surfaces.

4. Quantitative results

The program was set up for the first time with artificial images such as that shown in Fig. 8 in order to confirm that valuable results are obtained with the different modules.

Fig. 8a represents an ideal micrograph in three colours similar to those obtained with microstructures of titanium alloys after achieving a threshold with polarized light. Fig. 8a is an ideal representation of images similar to Fig. 4. Fig. 8b to d represent the digitalized images obtained after manual separation of the colonies of α lamellae from the three colours independently. We have called these colours grey levels because of the analogy between the optical threshold and the numerical threshold in image analysis.

Table I shows the area measured for each grey level before manual separation. This value represents the surface covered by the α lamellae having nearly parallel basal planes. In addition to the histograms, several general parameters have also been determined, such as the minimum and maximum values of the area and the length, which have been measured for the colonies of α lamellae in each grey level. These values are reported in Table II.

Fig. 9a to c represent the three histograms calculated by the program. Length and area histograms are in good agreement with the expected results.

From the orientation measurements, we observe two large peaks on the histogram for the angles 0° and 90°. Perpendicular lamellae are observed. Their number is near to that measured. However, no lamellae are observed which are nearly parallel to the 0° direction. This inexact result is partly due to the angle determination we have used which can lead to some errors. The importance of these errors increases when the ratio, length of the lamella over width of the lamella, decreases. When this ratio is near 1, the direction is determined arbitrarily by the analyser. In such cases the operator would give a direction parallel to the adjacent lamellae, whereas they could appear perpendicular in the histogram. An improvement in the angle measurements can be realized by using the "rose of directions'; with this method, the accuracy of the angle determination is about 2° to 5°. However, such a method is at the present time still quite time-consuming.

Similar results have been obtained afterwards directly from the specimen surfaces observed with an

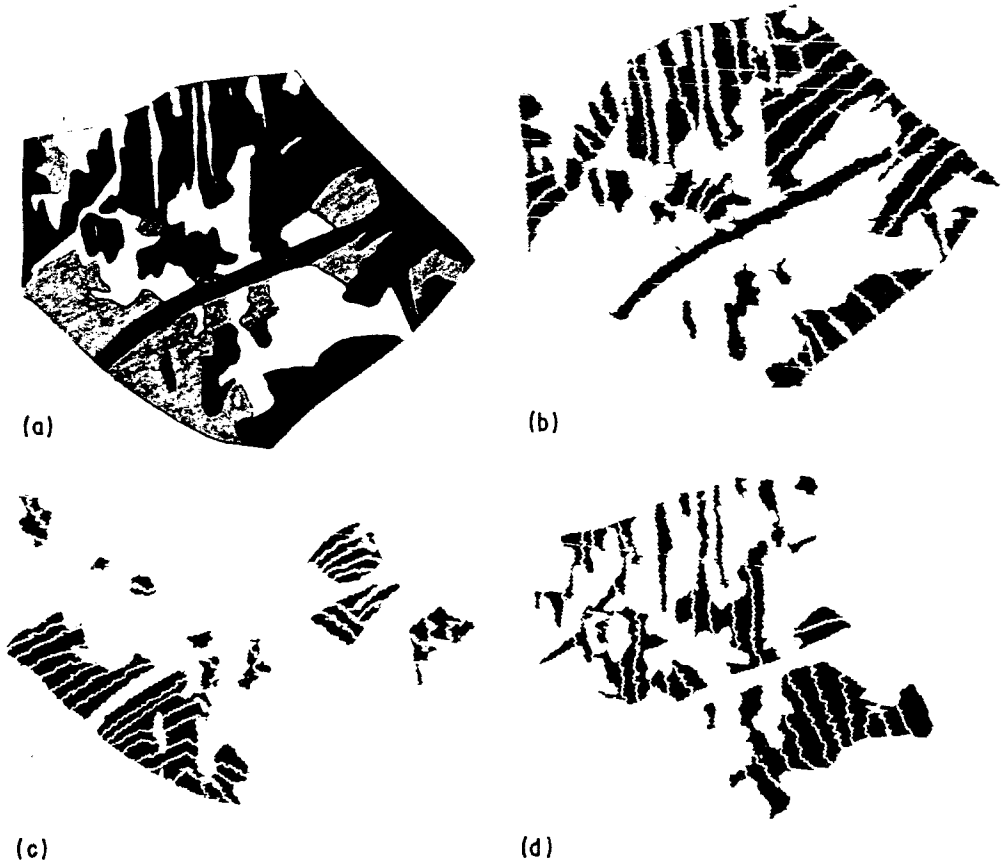


Figure 8 (a) Artificial image of an initial β grain (TA6Zr5D alloy). (b to d) Digitalized images corresponding to the three colours of polarization, obtained after threshold and manual separation. (b) Shade of grey I1 = 1. (c) Shade of grey I1 = 2. (d) Shade of Grey I1 = 3.

TABLE I Area measurements for the three grey levels

Grey level	1	2	3	Extrapolated grain area
Area (arb. units)	775	335	457	1568

optical microscope connected to the images analyser and equipped with polarized light [26].

5. Discussion

5.1. Characterization of the grain microstructure

The program allows the characterization of complex microstructures such as β-quenched titanium alloys by the distribution of sizes and orientations of α colonies within an initial β grain. The changes of the histograms should characterize the evolution of the microstructure during thermal and thermomechanical treatments. Such changes are being investigated to study the transformation products for several quenching rates, as well as the effects of deformation temperatures and/or mechanical test conditions on the size of the α colonies or initial β grains.

TABLE II Minima and maxima of area and length measurements (arb. units)

	Grey level		
	1	2	3
Area			
min	2.54	0.07	1.41
max	73.30	17.25	26.97
Length			
min	2.87	0.29	2.32
max	34.73	11.98	17.25

For a given specimen, the disorientation at grain boundaries can also be analysed by focusing on the boundary between the grains instead of the whole grain. Such analysis could give information about the α lamellae transformation near grain boundaries.

For a given grain, the near-neighbours can also be characterized in order to evaluate their relative influence on the α lamellae transformation.

5.2. Lamella size

For this measurement, a higher magnification, or even scanning electron microscopy (SEM) in the case of

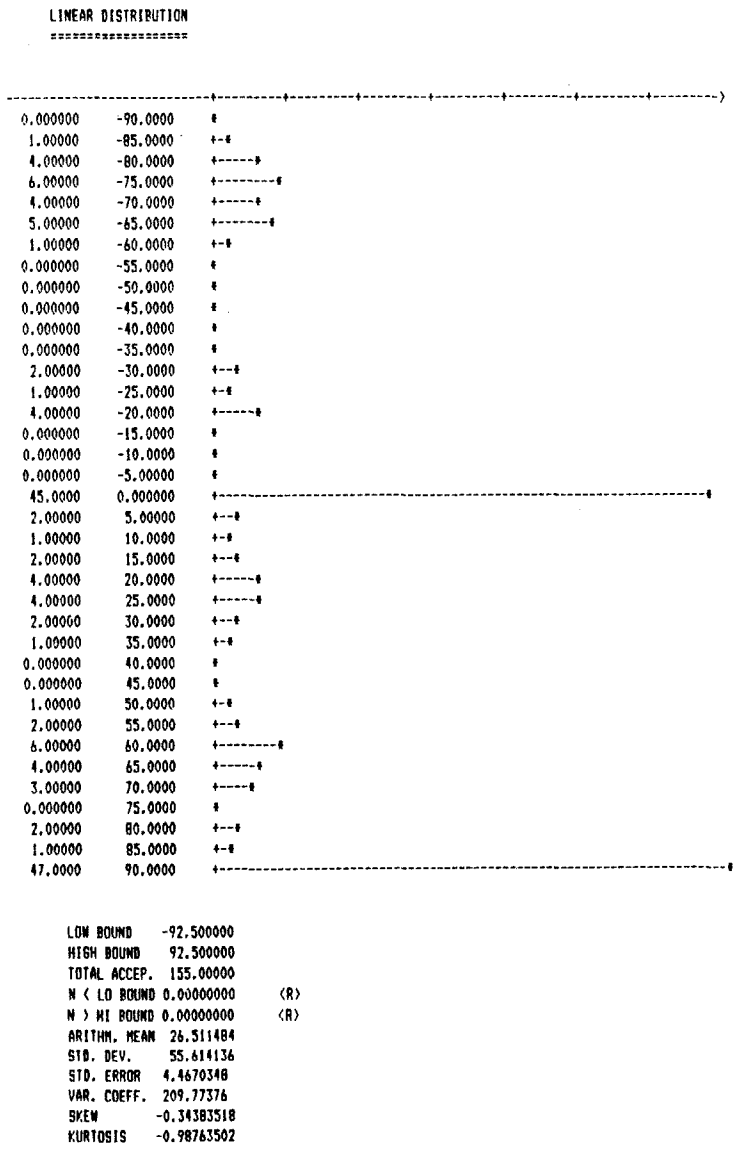


Figure 9 Histograms of (a) angles, (b) areas and (c) lengths as obtained from Fig. 8.

Figure 9 Continued

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      VAR. COEFF.   73.086342
      SKEW           2.1021633
      KURTOSIS       8.8864489
```

(c)

very fine lamellae (e.g. for high cooling rates), is necessary. The same program can be used or another appropriate module can be added. If a correct threshold can be performed, the simple measurement of the mean chord will easily give the α lamella size which is an important parameter to characterize the structural evolution during thermal and thermomechanical treatments [27].

5.3. Initial grain size

The grain size is an important parameter for the production of materials with good mechanical properties. In the case of titanium alloys, the size of the initial β grain is usually manually measured and a distribution in size can be obtained [28]. With the program, it is until now only possible to extrapolate the initial β grain size by adding the separate area values for the three polarization colours. The value is precise, but quite long to obtain for a control measurement. However, it locates the analysed grain within the size distribution which can be obtained by other methods.

If an appropriate etching solution could be found which allows unequivocal and distinct revelation of the β grain boundaries, as on the artificial images in Fig. 3, β grain size determination would be very easy to perform with image analysis.

5.4. Portability of the analysis method

The method of analysis is portable in the large definition of this word. The method can indeed be used to

characterize several types of microstructure of alloys with optically anisotropic phases. The architecture of the process chart does not depend on the analyser nor on the programming language. Its organization in modules is, furthermore, very adapted to structured and efficient languages such as C or Pascal.

5.5. Supplementary measurements in polarized light

Supplementary measurements in polarized light can be performed in parallel to the quantitative metallography. Such measurements are directly related to the structural orientations of the microstructure [15]. A further correlation could thus be achieved between the morphological parameters, the structural orientations and the material properties during thermal and thermomechanical treatments.

6. Conclusion

The association of an old-fashioned metallographic method with a modern technique of image treatment has allowed the automatic quantification of the characterization of some microstructures. The quantitative characterization which can be obtained should be associated with all the possibilities offered by polarized light in order to correlate the structural characteristics to the evolution of materials properties.

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