AFRL-ML-WP-TP-2006-432

A COMPARISON OF OPTICAL AND SEM BSE IMAGING TECHNIQUES FOR QUANTIFYING ALPHA-BETA TITANIUM ALLOY MICROSTRUCTURES (PREPRINT)

Jonathan Miller

MAY 2006

Approved for public release; distribution is unlimited.

STINFO COPY

MATERIALS AND MANUFACTURING DIRECTORATE
AIR FORCE RESEARCH LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750

REPORT DOCUMENTATION PAGE			*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE ***(DD-MM-YY)***	**2. REPORT TYPE**	**3. DATES COVERED** ***(From - To)***
May 2006	Journal Article Preprint	N/A

4. TITLE AND SUBTITLE A COMPARISON OF OPTICAL AND SEM BSE IMAGING TECHNIQUES FOR QUANTIFYING ALPHA-BETA TITANIUM ALLOY MICROSTRUCTURES (PREPRINT)	**5a. CONTRACT NUMBER** In-house **5b. GRANT NUMBER** **5c. PROGRAM ELEMENT NUMBER** N/A
6. AUTHOR(S) Jonathan Miller	**5d. PROJECT NUMBER** N/A **5e. TASK NUMBER** N/A **5f. WORK UNIT NUMBER** N/A
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Processing Section, Metals Branch (AFRL/MLLMP) Metals, Ceramics, and Nondestructive Evaluation Division Materials and Manufacturing Directorate Air Force Research Laboratory, Air Force Materiel Command Wright-Patterson Air Force Base, OH 45433-7750	**8. PERFORMING ORGANIZATION REPORT NUMBER** AFRL-ML-WP-TP-2006-432
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Materials and Manufacturing Directorate Air Force Research Laboratory Air Force Materiel Command Wright-Patterson AFB, OH 45433-7750	**10. SPONSORING/MONITORING AGENCY ACRONYM(S)** AFRL-ML-WP **11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)** AFRL-ML-WP-TP-2006-432

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

This work has been submitted to Microscopy and Analysis, John Wiley and Sons Ltd., publisher. This is a work of the U.S. Government and is not subject to copyright protection in the United States.

PAO Case Number: AFRL/WS 06-1165, 03 May 2006.

14. ABSTRACT

Quantitative metallography is often used to confirm the proper processing of aerospace metallic materials. A microstructural feature of great importance for titanium alloys processed in the alpha-beta phase field is the volume fraction of primary alpha. Standard methods of measuring delineated featured within a microstructure have been established previous, such as ASTM E-112 for grain size and ASTM E-562 for fraction of secondary phase. An accepted standard, however, for imaging technique has not been established to determine the quantity of primary alpha in alpha-beta titanium alloys, and metallurgists in industry and academia often favor different imaging techniques. In the present work, the volume fraction of primary alpha was measured using both optical microscopy and SEM backscatter electron (BSE) techniques. A comparison of measurements from images from both techniques indicated that the volume fraction of primary alpha was essentially equivalent.

15. SUBJECT TERMS

Titanium, microstructure, quantitative metallography, alpha phase

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT:**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON** (Monitor)
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Jonathan Miller
Unclassified	Unclassified	Unclassified	SAR	14	**19b. TELEPHONE NUMBER** *(Include Area Code)* N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

A comparison of optical and SEM BSE imaging techniques for quantifying alpha/beta titanium alloy microstructures

J.D. Miller[1]

[1] Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLMP, Wright-Patterson AFB, OH 45433-7817, United States

BIOGRAPHY

Mr. Miller received his B.S. in Chemical Engineering at the University of Dayton, Dayton, OH. His background is in materials research for aerospace alloys. He is currently a civilian working in the Air Force Research Laboratory's Metals, Ceramics, and NDE Division.

ABSTRACT

Quantitative metallography is often used to confirm the proper processing of aerospace metallic materials. A microstructural feature of great importance for titanium alloys processed in the alpha-beta phase field is the volume fraction of primary alpha. Standard methods of measuring delineated featured within a microstructure have been established previous, such as ASTM E-112 for grain size and ASTM E-562 for fraction of secondary phase. An accepted standard, however, for imaging technique has not been established to determine the quantity of primary alpha in alpha-beta titanium alloys, and metallurgists in industry and academia often favor different imaging techniques. In the present work, the volume fraction of primary alpha was measured using both optical microscopy and SEM backscatter electron (BSE) techniques. A comparison of measurements from images from both techniques indicated that the volume fraction of primary alpha was essentially equivalent.

KEYWORDS

Titanium, microstructure, quantitative metallography, alpha phase

ACKNOWLEDGEMENTS

This work was conducted as a joint effort of the Metals Processing Group at the Air Force Research Laboratory and Ladish Company in support of the Titanium Modeling Program of the Air Force Materials Affordability Initiative.

AUTHOR DETAILS

Address: Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLMP, Wright-Patterson AFB, OH 45433-7817, United States. Tel.: +1 937 255 3233; fax: +1 937 255 0445. Email address: jonathan.miller@wpafb.af.mil (J.D. Miller).

INTRODUCTION

Quantitative metallography is commonly used to verify the proper heat treatment of aerospace alloys such as those based on titanium. Academic institutions and research laboratories typically use backscattered-electron (BSE) imaging of as-polished surfaces, while industrial quality-control laboratories favor optical images of etched surfaces, primarily due to cost and time considerations. Because of the often-different drivers in the different environments, it can be challenging to try to compare the different results from the two techniques. Hence, the question arises as to whether the two measurement techniques yield statistically equivalent results. In this regard, it is well-known that some etchants preferentially attack grain and inter-phase boundaries and others preferentially coat or tint various phases. In addition, etchants may affect one phase in a two-phase microstructure; for example, the alpha phase of alpha-beta titanium is preferentially attacked by Kroll's reagent. In the present work, quantitative analysis of the primary-alpha volume fraction in the same region of several samples of Ti-6Al-4V was performed with both optical and SEM approaches in order to determine if there is a systematic difference between the two measurement techniques.

The assessment of imaging technique is critical to ensure proper measurement of microstructural features. BSE imaging of polished Ti-6Al-4V samples and optical imaging of the same samples appropriately etched with Kroll's reagent showed equivalent measurements by the ASMT point count method. When image analysis is used and thresholding is required, sample preparation and imaging can be even more critical. BSE shows the challenge of shading primary alpha both light and dark depending on crystal orientation as well as phase edge blurring as a function of voltage. Optical imaging of etched samples will show similar challenges as noted in the ASTM point count method, but repeatable measurements can be achieved with appropriate etching methods.

MATERIALS AND METHODS

A major issue with measurement of percent primary alpha in alpha-beta titanium alloys is the area of sample that must be measured to determine the correct average value. A study was undertaken to assess the affect of magnification and quantity of images on measured volume fraction of primary alpha. It was determined that while BSE imaging is often conducted at relatively high magnifications, it is more appropriate to image alpha-beta titanium alloys at 200X and perform measurements on a minimum of three adjacent locations to determine an accurate primary alpha volume fraction. Slight variations in primary alpha can be seen in alpha-beta titanium alloys as a function of segregation.

Three samples of alpha/beta forged Ti-6Al-4V were prepared using standard metallographic techniques (grinding using 240, 320, and 400 SiC papers, followed by polishing using 15, 9, 3, 1, and 0.5 micron diamond paste with a activated colloidal silica final polish) to yield an as-polished surface suitable for scanning electron microscopy. Fiducial marks comprising micro-hardness indents were placed on each specimen to locate specific regions of interest. BSE images were then taken from the specified regions. Following SEM imaging, each sample was lightly etched three times (5 sec., 10 sec., and 15 sec.) to successively deeper depths using Kroll's reagent (composed of 100 mL distilled water, 5 mL nitric acid, and 3 mL hydrofluoric acid), and optical images were taken in the same regions as the SEM BSE photomicrographs. The volume fraction of primary alpha in each set of backscatter and optical images was measured via high-resolution-grid point counting per ASTM E-562 and compared. The results from the two imaging techniques were then compared.

RESULTS AND DISCUSSION

For the optical images, the contrast and darkness of microstructural features changed substantially as the material was etched for increasing periods of time. As expected, the etchant attacked the phase boundaries first, revealing the general microstructure. Once all the boundaries were revealed, the etchant attacked regions within the boundaries resulting in artifacts that did not correspond to microstructural features per se and hence may have skewed quantitative metallography measurements. For this reason, there was an optimum time that the material should be exposed to the etchant, but this time was dependent on the microstructural features and properties of each specific sample, making it difficult to know the etching time *a priori*. Thus, the measured volume fraction of a given microstructural feature in an optical image for an alpha/beta titanium alloy varied depending on the etching time. Typically, the volume fraction of primary alpha was underestimated for short etching times because all of the boundaries were not revealed, and primary alpha particles were mistaken for difficult-to-resolve, fine transformed beta phase. In particular, 14.4 pct primary alpha was measured for the first succession of light etch and optical microscopy (Fig 1).

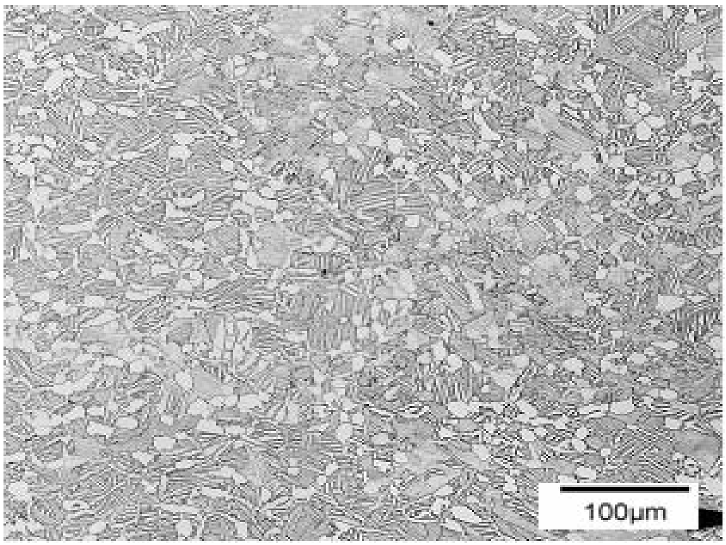

Figure 1: Optical micrograph of a lightly etched surface of a specific location of an alpha-beta titanium alloy

Following two successions of light etch and optical microscopy, all grain boundaries were revealed, and a clear measurement of percent primary alpha could be measured. With the appropriate etching time, the measured percent of primary alpha in an optical image, specifically 23.2 pct primary alpha, was indeed the percent of primary alpha on the etched surface (Fig 2).

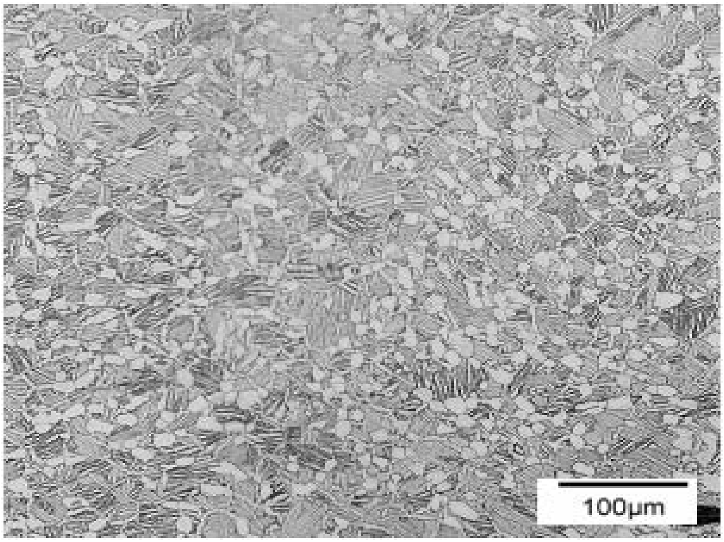

Figure 2: Optical micrograph of an under-etched surface of a specific location of an alpha-beta titanium alloy

For long etching times, the volume fraction was overestimated because etching artifacts unrelated to microstructure appeared as primary alpha globules, thus increasing the apparent volume fraction of primary alpha. Specifically, 23.6 pct primary alpha was measured after three successions of etch and optical microscopy (Fig 3).

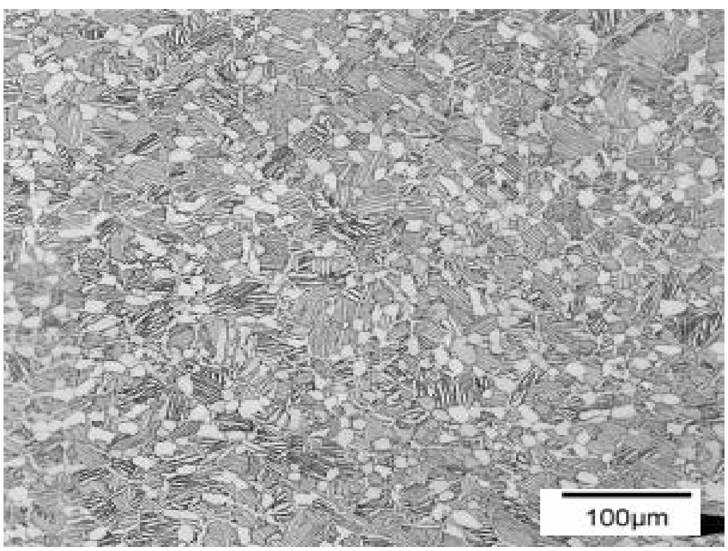

Figure 3: Optical micrograph of an appropriately etched surface of a specific location of an alpha-beta titanium alloy

It is expected that any further etching will increase the ambiguity in microstructural features in this particular region. Additionally, the percent of primary alpha increased with increasing exposure time. As the exposure time increased, the rate of growth decreased. This decline in rate of percent alpha growth can be explained by the kinetics of the etchant with the metal. The surface will only appear burnt after long etching times, as the etchant begins to attack the alpha phase rather than the grain boundaries. These observations agree with measurements for each etching time.

SEM BSE images are considered to measure the area fraction exactly, as the penetration depth is typically on the order of nanometers and the particle sizes on the order of micrometers. However, due to the finite penetration depth of the electron beam, SEM BSE measurements of the volume fraction of primary alpha were affected by the voltage of the beam.

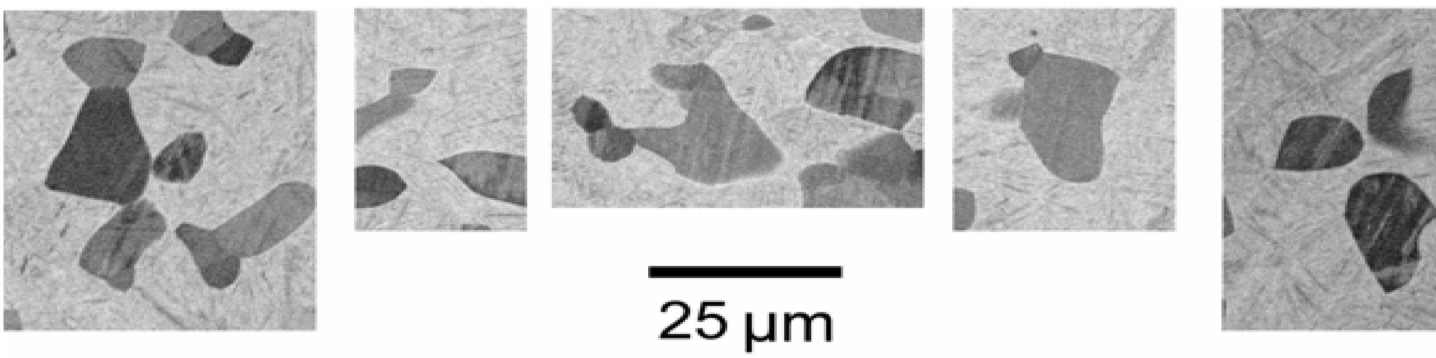

Figure 4: BSE micrograph showing the increase (from left to right) in the blurring of grain boundaries due to the penetration depth of the beam

When imaging was done with a high voltage, a blurring affect occurred at the edges of particles due to averaging through the penetration depth, leading to an overestimation of the area fraction of primary alpha (Fig 4). This error is small and accounts for less than 1 pct error of the final measurement. In particular, if the sectioned surface of a spherical alpha particle lies above its diametral plane, the area of the sectioned surface will be *less* than the area just below the surface when using low-to-moderate voltage. On the other hand, when the sectioned surface of a spherical particle was below the diametral plane, the sectioned area on the surface was greater than the sub-surface and no blurring occurred. Overall, the area fraction of primary alpha was slightly overestimated under SEM BSE techniques when a high voltage was used.

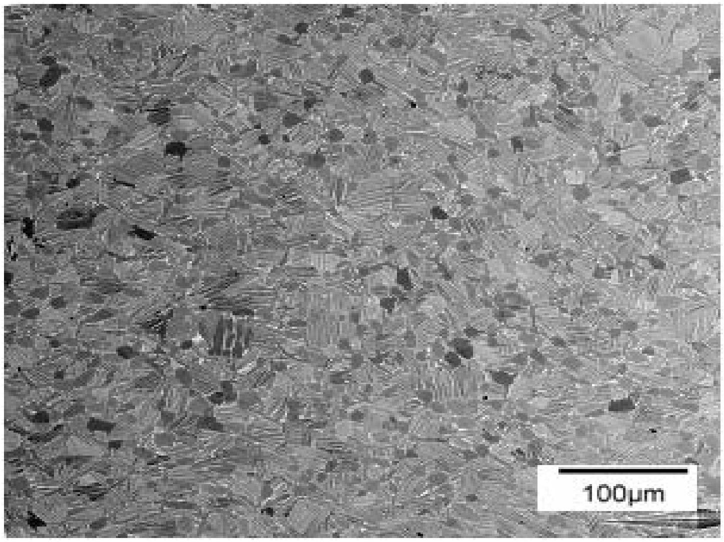

Figure 5: BSE micrograph of an as-polished surface of a specific location of an alpha-beta titanium alloy

In particular, 23.9 pct primary alpha was measured for the as-polished and BSE imaged case (Fig 5). By reducing the voltage, the penetration depth was reduced and the tendency for overestimating the volume fraction was reduced.

By comparing the measured percent of primary alpha for the optical micrographs of the three etching conditions and the BSE micrograph of the as-polished condition, the effect of etching-reagent exposure time on quantitative metallography measurements could be assessed, and the most appropriately etched micrograph could then be compared to the BSE micrograph of the same location on the sample (Table 1). The measured percent of primary alpha was essentially equivalent for the appropriately etched micrograph and the BSE micrograph. This comparison validated that an appropriately-etched optical micrograph yields equivalent quantitative results as an as-polished BSE micrograph.

CONCLUSIONS

From this work, it was determined that optical microscopy of a properly etched surface yields a statistically equivalent quantitative measurement of primary alpha volume fraction compared to measurements on as-polished surfaces using SEM BSE microscopy. However, care must be taken with optical techniques because the etchant can attack different regions of the surface at different rates. Therefore, a quantitative measurement performed optically is valid if the region is a representative region of the entire specimen and does not contain any suspect etching artifacts. Additionally, it is critical to understand the appropriate procedure for volume fraction measurement according to the ASTM specification. The findings above show that the two different techniques to collect a micrograph yield equivalent results, not thata single micrograph is sufficient to measure the volume fraction of a sample. Thus, an appropriate number of representative micrographs are still necessary to yield statistically relevant measurements, as outlined in the specification.

FIGURE CAPTIONS

Figure 1: Optical micrograph of a lightly etched surface of a specific location of an alpha-beta titanium alloy

Figure 2: Optical micrograph of an under-etched surface of a specific location of an alpha-beta titanium alloy

Figure 3: Optical micrograph of an appropriately etched surface of a specific location of an alpha-beta titanium alloy

Figure 4: BSE micrograph showing the increase (from left to right) in the blurring of grain boundaries due to the penetration depth of the beam

Figure 5: BSE micrograph of an as-polished surface of a specific location of an alpha-beta titanium alloy

REFERENCES

1. S.L. Semiatin, S.L. Knisley, P.N. Fagin, F. Zhang, and D.R. Barker: *Metall. Mater. Trans. A*, 2003, vol. 34, pp. 2377-86.
2. H.E. Exner and S Weinbruch: *ASM International*, 2004. “Scanning Electron Microscopy”. pp 355-367
3. E.E. Underwood: *ASM Handbook*. “Quantitative Metallography”
4. F.J. Humphreys: *Journal of Microscopy*, “Quantitative metallography by electron backscattered diffraction”. V195, Pt 3 Sept 1999, P170-185.
5. M. Dallair and D. Furrer: *Advanced Materials and Processes*, 2004. “Quantitative Metallography of Titanium Alloys”. 162:1212 25-28.