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Materials Characterization Through Quantitative Digital Image Analysis

Joel Philliber, Bonnie Antoun, Brian Somerday, Nancy Yang

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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Materials Characterization Through Quantitative Digital Image Analysis

Joel Philliber, Brian Somerday
Engineered Materials Department

Bonnie Antoun
Materials Mechanics Department

and

Nancy Yang
Analytical Materials Science
Sandia National Laboratories
Livermore, CA 94550

ABSTRACT

A digital image analysis system has been developed to allow advanced quantitative measurement of microstructural features. This capability is maintained as part of the microscopy facility at Sandia, Livermore. The system records images digitally, eliminating the use of film. Images obtained from other sources may also be imported into the system. Subsequent digital image processing enhances image appearance through the contrast and brightness adjustments. The system measures a variety of user-defined microstructural features—including area fraction, particle size and spatial distributions, grain sizes and orientations of elongated particles. These measurements are made in a semi-automatic mode through the use of macro programs and a computer controlled translation stage. A routine has been developed to create large montages of 50+ separate images. Individual image frames are matched to the nearest pixel to create seamless montages. Results from three different studies are presented to illustrate the capabilities of the system.

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Materials Characterization Through Quantitative Digital Image Analysis

Introduction

Sandia has recently purchased an optical microscope with a color digital camera and image analysis software. This system is part of the microscopy facility and is available for any projects which require quantitative image analysis. As shown in Figure 1, the system includes a Zeiss Axioplan 2 microscope and a 233 Mhz computer. Images are collected and stored electronically, eliminating the need for polaroid film. Electronic images of a polished metallographic mount are acquired more quickly and with less expense than is possible with conventional polaroid film. Also, electronic images are easily enhanced to optimize the contrast and brightness of an image, and can be subsequently analyzed to quantitatively measure microstructural features.

Figure 1. Zeiss image analysis system.

Image files obtained from other sources can be easily imported into the computer for analysis. Some projects may require analysis at a magnification that is higher than what is typically used for optical microscopy. For these cases, electronic images acquired

from a scanning electron microscope (SEM), or scanned images from a transmission electron microscope (TEM), may be imported into the computer for analysis. This capability greatly expands the range of analyses that can be performed.

A large number of microstructural features are measured with the image analysis system. In general, two types of measurements are made—region and field properties. Region measurements are associated with each individual feature (or particle) within the microstructure. For example, a database can be generated to list the area, perimeter, spatial location, average diameter, aspect ratio, orientation and several other measures of each specific particle. Field measurements are related to an entire field of view. These field measures include area fraction and total number of particle in addition to several other parameters. While the image analysis system measures features visible in a polished plane, geometric relations allow volume properties (e.g. void volume fraction, and average particle diameter) to be calculated.

Image processing routines are used to alter the image with the goal of obtaining more accurate measurements. For instance, particles that contain holes may be “filled in” to measure overall area outlined by a particular phase. Also, routines exist to separate connected regions (e.g. spheres which overlap in an image can be isolated to obtain an accurate number count). Qualifiers may be put on measurements such that only particles greater (or smaller) than a particular size are measured. Shape qualifiers may also be used to measure only those particles which are roughly circular in shape. The operator has the option of selecting which particles are to be measured and which are to be omitted.

Grain sizes can be measured, providing that the grain boundaries have enough contrast to allow them to be thresholded. Individual grains are often distinguished by eye; but computer routines have a difficult time separating them from the rest of the microstructure. In this situation, a graphics plane is used to manually trace over the grain boundaries, and then measurements are made on the graphics plane to get an accurate measure of grain sizes.

The Zeiss image analysis system has the ability to control stage movements and measurement functions through macro-programs. Through collaboration with researchers at Georgia Institute of Technology, a routine has been developed to allow montages of several images to be semi-automatically acquired and analyzed. The size of the montage is essentially limited only by the size of the computer hard disk onto which the images are stored. However, montages of up to 100 separate images are easily generated. As an example, a 3x3 montage is shown in Figure 2. This montage consists of nine separate images which were acquired at a resolution of $0.209\text{ }\mu\text{m}/\text{pixel}$ (50X objective lens) and then “pasted” back together. The matching between individual images is within approximately 10 pixels.

Figure 2. 3x3 montage acquired with 50X objective lens.

Some applications require exact matching between individual montage frames. A separate routine has been developed to allow matching of consecutively acquired images to an accuracy of 1 pixel. Operator input is used to match each frame to the

previously acquired image to the nearest pixel. The result of this process is a large montage with no discontinuities at the individual frame edges.

The main advantage of a montage is that a relatively large area of the sample (on the order of millimeters) can be examined at a high magnification. When a sample does not have a uniform microstructure, large areas have to be analyzed to eliminate any local variations in the size or spatial distribution of the measured particles. This often occurs with samples such as metal matrix composites where the spatial distribution of reinforcements is non-uniform. The montage allows large areas to be examined at a resolution that is sufficiently high to accurately measure the relevant features of individual particles (size, aspect ratio, spatial distribution...). This technique is also necessary in samples where a gradient in the size or density of particles is present—such as a tensile sample where void density is measured as a function of distance from the fracture surface.

System Specifications

The automated stage controller installed on the Zeiss Axioplan 2 microscope allows stage translations to be controlled either manually, using a joystick, or automatically through the image analysis software. Four objective lenses are currently installed on the microscope—5X, 10X, 20X and 50X. A Diagnostic Instruments Spot 3-color camera acquires images with 8 bits of color depth in each of three colors (red, blue and green) to give a total color depth of 24 bits. Color depth refers to the number of brightness levels. Eight bits of color depth corresponds to 256 distinct level of brightness (*i.e.* 256 grey levels for a black and white image). The size of the digitally captured images is 1312 x 1033 pixels. Image resolution ranges from 2.09 $\mu\text{m}/\text{pixel}$ for the 5X objective to 0.209 $\mu\text{m}/\text{pixel}$ for the 50X objective. The memory required for the images is approximately 1.3 MB for a grey scale TIFF image. Color TIFF images require 3.9 MB of memory since separate red, blue and green images are stored. Other file formats can be used to reduce the storage requirements. The 233 MHz computer uses the Windows 95 operating system and is connected to the computer network for transferring saved images. The image analysis software is Zeiss KS400 version 3.0. Powerful macro routines can automate most of the measurement and stage control functions. Digital

images from other sources (SEM, scanned images, ...) are easily imported into the KS400 software to allow image processing and analysis.

Example of Image Analysis

This section outlines the process of acquiring a single digital image and measuring the area fraction of a particular phase. Some of the capabilities of the system are highlighted along with potential sources of error in the analysis. There are three major steps to image analysis. The first step is to acquire the image and store it in the computer memory. The second step is to perform image processing operations. These operations include altering the image contrast and brightness, correcting for artifacts such as uneven illumination effects, and thresholding the image to convert it to a binary black and white image. The final step in the process is to define the measurements of interest and make the measurements.

The current example examines an alloy which is a two-phase mixture of copper and Kovar (a nickel-iron alloy with a low coefficient of thermal expansion). Figure 3 shows the acquired color image. The original colors of the sample are reproduced in the image. The copper phase appears as copper colored (orange) regions, while the Kovar phase is yellowish-white. Software provided with the camera allows for adjustments of the color balance. Colors in the image can be matched to the original colors of the sample if exact color matching is required. However, for most applications, color information is not necessary and the images are converted to grey level images for memory considerations.

Converting the color image in Figure 3 to a grey scale equivalent produces the grey scale image shown in Figure 4a. Image processing enhances the appearance of the image. Two more common image processing operations are to optimize the contrast and brightness of the image, and to remove any effects of uneven illumination from the light source.

Figure 3. Color image acquired from Zeiss Image Analysis System.

The contrast and brightness are adjusted with the menu window shown in Figure 5. Two histograms are present with Input representing that of the original image and Output representing that of the enhanced image. Each histogram has an x-axis that shows the 256 different grey levels while the y-axis shows the relative number of pixels in the image that are at that particular grey level. In order to fully enhance the contrast, the range of grey levels in the Output image should span the entire 256 levels. However, using the full range of grey levels may produce an image that appears to have too much contrast. The contrast should be adjusted to the point that the different microstructural features in the image are easily distinguished from one another. The range of grey levels in the output histogram is adjusted left or right to make the image darker or brighter.

Figure 4. Image from Figure 3. a) Converted to a grey scale image. b) Increased contrast. c) Corrected for effects of non-uniform illumination.

Figure 5. Contrast menu from KS400 software.

Figure 4b shows the image from Figure 3 with enhanced contrast. Enhancing the contrast often exacerbates a dark “halo” near the edges of the image. This effect is a result of non-uniform illumination and must be removed prior to performing the threshold operation. Otherwise, the lighter phase near the edge of the image may have the same grey level as the darker phase near the center of the image. It is then difficult to separate the phases using the threshold routine. Illumination artifacts are removed by using an electronic filter to blur the image so that no details from the enhanced image remain except for the overall grey level. This blurred image represents only the illumination pattern in the image. Subtracting the illumination pattern from the enhanced image produces a uniformly illuminated image, as shown in Figure 4c.

The next step in determining the area fraction of the second phase is to threshold the image. Thresholding involves conversion of the image to a binary black and white image where the phase to be measured is white (i.e. grey level of 256) while the rest of the image is black (grey level equals 1). The thresholding menu is shown in Figure 6. Ideally, there will be sufficient contrast between the phases that each phase will have a distinct peak on the histogram. The peak corresponding to the phase of interest is bounded by the upper (red) and lower (blue) range markers on the histogram. When the threshold operation is performed, the pixels with grey levels between the two markers become white, while all others are black. The binary image is shown in Figure 7 where the phase to be measured (kovar) is white. Analysis by the KS400 software indicates that the white area (kovar phase) occupies 68.5% of the image area. The thresholding operation is probably the most important step in the entire image analysis routine. Most grey level images do not have a sharp boundary at the interface between two different phases. Instead, a boundary *region* is present over a distance of several pixels where the grey level gradually transitions from one grey level to the other. Small changes in the threshold parameters alter the size of the regions of interest by changing the specific location in the boundary region where the particle is defined in the binary image.

An extreme example of thresholding sensitivity is illustrated with the images in Figure 8. This image is from a metal matrix composite which contains acicular reinforcing particles of random orientations. The images in Figure 8 are two different binary representations of the same grey level image. Image 8a was obtained using threshold values of 1 for the lower range (i.e. the red line in the histogram) and 154 for the upper range (blue line). Image 8b used values of 1 and 157. The area fraction of both images was measured, with image 8a having 6.508% white area and image 8b having 7.277%. In both measurements, regions consisting of fewer than 5 pixels were considered to be "noise" and were not included in the measurements. For this example, changing the threshold level by just three grey levels (out of a total of 256) produces a minimal effect on appearance of the binary images. However, the measured area fractions have a relative difference of approximately 10%.

Figure 6. Threshold menu from KS400 software.

Figure 7. Image from Figure 4c which has been converted to a binary black and white image using the threshold parameters shown in Figure 6. The white area occupies 68.5% of the total area.

8a: Threshold=154

8b: Threshold=157

Figure 8. Two images which have different threshold levels. Image 8a has a threshold level from 0-154 and an area% of 6.508. Image 8b has a threshold image from 0-157 and an area% of 7.277.

The relatively large variation in measured area fractions with small changes in threshold values is associated with the geometry of the particles in the image. When large numbers of small particles are present, the number of pixels at the edge of the particles represent a large fraction of the total number of pixels associated with the particles. Increasing the radius of the particles by one pixel uniformly around their edge adds a large number of pixels to the measured area. As the example illustrates, this may have a significant effect on the measured area fraction.

When making multiple measurements of a single sample, it is important to have consistent focus levels, image contrast, and threshold levels in each frame. Otherwise each measurement (or set of measurements) will give different values. Also, each operator may tend to use slightly different threshold conditions. The measurements should not be considered to be an absolute measure of area fractions (or other microstructural features). Samples measured by different operators may give slightly different absolute values; however, the *trends* between multiple samples should be consistent between different operators. For most applications, the measurements should have a relative accuracy of between 5 and 10 percent. The accuracy between different operators can be improved by performing several test measurements to determine specific lighting conditions and threshold levels that will be used by all operators.

Case Studies

The Zeiss Image Analysis System has been used for three different projects. The first study examined variation of the size of relatively large microstructural features. These measurements are analogous to grain size determinations. The second project involved analysis of strengthening particles in an aluminum alloy. The final project related microvoid features to their geometric location within test sample. These case studies illustrate the kinds of the measurements that can be made with the image analysis system.

Case Study 1: Variability of LENS Melt Features

Laser Engineered Net Shaping (LENS™) is a unique solid freeform fabrication process in which a solid metallic part is built by using a laser to melt powder particles. A computer CAD file governs the movement of the laser such that a complex part can be created by successive deposition of molten material in consecutive melt paths and layers. A typical LENS™ microstructure is shown in Figure 9, which shows a cross section of a part. A periodic structure is evident with alternating layers that are oriented 90° to one another. Distinct “scallop” features are evident in the layers containing melt pool traces which are into and out of the plane of the paper. The “scallop” outlined in yellow represents the shape of a single melt path except for the part which was re-melted by a subsequent path to its left.

Figure 9. Typical LENS microstructure. One of the scallops representing a melt path in and out of the plane of the paper is highlighted in yellow. The layers immediately above and below the yellow scallop correspond to melt pools running left to right.

Variations of laser power and other processing parameters during deposition can result in non-uniform melt pools as evidenced by variations in the areas of the “scallop”. This may ultimately lead to voids and inhomogeneities in the microstructure that can degrade the mechanical properties. Recent work has focused on developing a feedback control loop where the melt pool is monitored for size and temperature. This information is used to control the power source to maintain uniform deposition conditions. This case study involves the characterization of bulk LENS™ samples fabricated with and without closed loop feedback control.

A 4x5 frame montage is acquired from a controlled and an uncontrolled sample using the 5x objective lens. This corresponds to roughly 1 cm² of area on the actual samples. The scale of the montages does not warrant pixel to pixel matching of successive frames. Thus, the frames match with an accuracy of 10-20 pixels. The contrast of the scallops is not sufficient to allow thresholding with the KS400 software. Instead, a transparency is placed over the montages and the scallops are manually traced. The transparencies are scanned and imported into the KS400 software to measure the characteristics of the scallops. Figure 10 shows the scanned representation of the sample used to make measurements. The measured features include the area, the maximum horizontal dimension (width), the maximum vertical dimension (height) and the center of gravity for each scallop. The sizes of ~200 individual scallops are analyzed in each sample.

An example of the data collected is shown in Figure 11. The maximum vertical distance of each scallop (height) is plotted against the vertical position within the sample. The results show that the control sample (SCC3) has a less height variation within a layer than the uncontrolled sample (SC). However, the average heights of the “scallop” vary more as a function of distance from the substrate in sample (SCC3) than in sample (SC). This example illustrates that both measures of size, as well as the location of features can be easily collected and related to one another.

Figure 10. Outlines of LENS scallops used for determining size variation.

Figure 11. Height of LENS scallops as a function of vertical position in the sample. The non-control sample is labeled SC and control sample is labeled SCC3. Within each row of scallops, the range of height values is lower for the control sample.

Case Study 2: Damage Evolution Determination in Al alloys

Damage nucleation measurements are made for 6061-T6 aluminum and 5086-O aluminum. The strengthening precipitates in the two alloys cause different mechanical responses to applied loads—most notably in the strain hardening characteristics. The physical process of damage nucleation varies with material, microstructure and loading conditions. Analysis of void nucleation will further elucidate the effects of microstructural features and mechanical behavior on damage evolution. Also, the measurements will provide the constitutive material model developers with the information necessary to develop void nucleation models for these materials.

Interrupted tensile, compression and torsion tests were conducted to strain levels of 25%, 50% and 75% of the tensile failure strain in both alloys. Measurements of damage, in the form of debonded particles and cracked particles, are being made as a function of loading condition and strain level. After testing, the specimens are sectioned and polished on a plane that is perpendicular to the fracture surface. Some of the Mg_2Si particles react with the polishing medium, producing the visual effect that the particle has been pulled out, or that there is a void, but this can easily be ruled out by viewing at higher magnification.

In the 6061-T6 alloy, two different types of reinforcing particles are present, Mg_2Si and AlFeSi . The particles have different morphologies and grey levels, making them easily distinguished from one another as shown in Figure 12. The measurements that are of interest to this project include the number density of each type of particle, the number density of cracked particles, the number density of debonded particles, the average size of virgin particles and the average size of damaged particles. These measurements are compared to baseline data taken from untested samples.

As an example of the image analysis process, the measurements for an untested 6061-T6 specimen are reported. In order to generate adequate statistics on the particles, 25 frames were acquired and pasted together to within +/- one pixel of accuracy. The original frames were acquired with a 50X objective lens to ensure adequate resolution of the particles. An area corresponding to 1.3 mm x 1.0 mm was measured. The contrast

of each frame was adjusted and the uneven illumination effects were corrected on each grey level image prior to pasting into the montage. The montage was sufficiently homogeneous that one threshold setting was able to delineate the particles over the entire area. A threshold setting of 13 - 108 was used for the Mg_2Si particles and 119 - 152 for the AlFeSi particles.

The table in Figure 13 shows a sample of the measurements from the specimen. Although averages are reported, the values for each particle are stored in a data file. Also note that the maximum and minimum dimensions of the particles can be measured, allowing the aspect ratio of each particle to be calculated. This example demonstrates that a large number of particles can be analyzed to obtain a good statistical distribution.

Case Study 3: Void Damage Evolution in 304L Stainless Steel

The mechanism of fracture in 304L stainless steel under many mechanical and environmental conditions involves the nucleation, growth, and coalescence of microvoids. Modeling of microvoid fracture requires inputs that are derived from experimental characterization of the damage process. The objective of this study is to quantify microvoid damage as a function of position in the test specimen, applied strain/hydrostatic stress, temperature and hydrogen content for low-sulfur 304L stainless steel.

Different mechanical and environmental effects are examined by altering the testing variables and the condition of the notched tensile specimens. Hydrostatic tensile stress in the notched region is varied by the altering the notch radius. The dependence of void damage on applied strain is assessed by interrupting the experiments at different applied axial displacements. Hydrogen content effects are examined by charging the notched tensile specimens in hydrogen gas until a uniform concentration of hydrogen is established. The effect of temperature is examined by heating the specimens during tensile testing.

Figure 12. Sample image from 6061-T6 montage. Darker particles are Mg_2Si and lighter particles are $AlFeSi$. The different grey levels of the particles allows each population of particles to be analyzed independently. Note that the $AlFeSi$ particles are aligned with the rolling direction.

Particle	# of Particles Analyzed	Average Particle Area (μm) ²	Area Percent (%)	Avg. Max. Dimension (μm)	Avg. Min. Dimension (μm)	Number Density (#/mm ²)
Mg_2Si	504	6.17	0.21	3.36	1.90	375
$AlFeSi$	3678	3.09	0.76	2.95	1.33	2737

Figure 13. Sample of measurements from different particle populations.

Microvoid damage is produced in a controlled manner using cylindrical tensile specimens that are machined with circumferential notches having a circular profile. Microvoid damage evolves in the notched region due to the high triaxial tensile stress that develops as a result of geometrical constraint (Figure 14). Tensile tests are interrupted at prescribed fractions of the fracture displacement ($\Delta L_{\text{fracture}}$). The notched regions are sectioned and polished parallel to the tensile axis to reveal the microvoid damage. The measurements of interest include the area fraction, absolute area, number density and size distribution of microvoids; and the inter-void distances.

Figure 14. Schematic of cross-section of notched tensile bar showing location of microvoids in area of high stress triaxiality.

An example of the measurements is presented for sharp notched specimens (notch ligament radius/notch profile radius = 3.2) tested at 800°C without exposure to hydrogen. The area fraction of the microvoids is measured as a function of position across the notch ligament diameter. Five fields of view are acquired across the minimum diameter of the notch ligament using the 20X objective lens. Each frame covers an area of $\sim 550 \mu\text{m} \times 700 \mu\text{m}$.

A color image is acquired for each field of view, which is then converted to a grey-scale image. Contrast enhancement is not necessary since the grey level distribution of the pixels is essentially binary, *viz*, black voids and a white matrix. Non-uniform illumination effects are corrected.

Conversion of the grey level image to a binary black and white image requires careful analysis. The automatic threshold routine selected an upper-bound threshold value of about 180, which is considerably below the peak on the histogram that corresponded to the matrix phase. Increasing the upper-bound threshold parameter results in a noticeable increase in the number of smaller voids in the binary black and white image. Comparison of the binary image to the original grey level image shows that an upper-bound threshold level of about 220 captures the majority of the smaller voids. However, this value also increases the measured area of many of the larger voids. Clearly, a trade-off exists between including the smaller voids and increasing the area of the larger voids. In order to make the measurements consistent, a single upper threshold setting of 220 is used for all specimens. Many fields of view near the edges of the samples and in those specimens with smaller strains consist mainly of smaller voids which would not be captured with a lower threshold setting.

Figure 15 shows a representative sample of measurements of microvoid area as a function of distance across the minimum notch ligament diameter. Values are shown for three different strain levels. Two grey level micrographs are also included, corresponding to the mid-diameter fields of view at the lowest and highest strain levels. The results are consistent with theoretical expectations—microvoid damage increases as

a function of increasing strain, and is highest near the center of the specimen where the highest hydrostatic tensile stress is located.

Figure 15. Plot showing microvoid area as a function of location for three different strain levels. Also shown are photomicrographs illustrating the damage present at the highest and lowest strain levels.

Summary

This report demonstrates the capabilities of the Zeiss Image Analysis System. The combination of the remote controlled stage and the powerful macro programs allows large areas of samples to be easily analyzed. The measurement software allows numerous microstructural features to be quantified in a semi-automatic manner. This system enables a new level of microstructural characterization by measuring a myriad of features over relatively large areas at high magnifications. The automation capabilities of the system further extends the usefulness of the technique by allowing substantially more measures than could realistically be accomplished by more traditional point counting methods.

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