“Let’s show ‘em what we’re made of”

might be an effective battle cry for human warfighters, but when considering the materials on which these warfighters rely, relating material composition to properties and performance has been an extremely difficult task. Knowing that relationship, however, is crucial for those using computational design to make better, more reliable materials for specific Naval applications. In the last few years, breakthroughs in 3D characterization, which uses information from experimentally measured images, and analysis of the reconstructed material microstructures have resulted from the availability of faster and more accurate measurement tools. Researchers in the Multifunctional Materials Branch of NRL’s Materials Science and Technology Division, using serial sectioning techniques that they devised, along with optical microscopy and electron backscatter diffraction (EBSD), collect and analyze 3D data from a variety of polycrystalline alloy microstructures to relate structure to properties and attempt to correlate such phenomena as material failure, corrosion behavior, mechanical response, and phase transformations to microstructure. Recent data sampling of a large reconstructed volume from a titanium alloy revealed a correlation between crystallography, applied load, and mechanical response. These data are applied to predictive models and simulations that in turn drive the development of materials that will function as designed under real-world conditions.

Structure–Property Relationships in a 3D Polycrystalline Microstructure

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This article provides a brief review of the three-dimensional materials characterization, analysis, and simulation tools developed in the Multifunctional Materials Branch of the Naval Research Laboratory. This innovative research combines serial sectioning, electron backscatter diffraction (EBSD), and finite element modeling to examine the role of microstructure on material behavior. An emphasis of this research is the determination of structure–property correlations in polycrystalline microstructures, as well as the identification of critical microstructural features for failure in polycrystalline microstructures. Recently, using data sampling from a large reconstructed volume of a titanium alloy, a correlation between crystallography and mechanical response was observed. This type of data is used as input for the efficient computational design of Naval materials and structures.

INTRODUCTION

To design new Naval materials that meet specific performance criteria, an in-depth understanding of the relationships between processing, microstructure, properties, and performance is required. It is now well known that the three-dimensional (3D) microstructure of materials dictates their mechanical performance and physical properties, and to develop accurate predictive models of processing and performance of advanced Naval materials, it is critical to understand the morphology and evolution of real 3D microstructures.

In recent years, 3D characterization and analysis of material microstructures have advanced rapidly, as the speed and accuracy of computational and measurement tools have increased. Three-dimensional microstructures, reconstructed from experimentally measured images, can now be used as input for simulations of mechanical response, corrosion behavior, and phase transformations.

Techniques developed in the Multifunctional Materials Branch at NRL have been applied to a variety of polycrystalline alloy microstructures for the collection and analysis of 3D data, and the application of this data to predictive models and simulations to develop an understanding of microstructure–property relationships. The overall goal of this research is to provide a framework for efficient and accurate design of materials by developing the tools for the prediction of material response under service conditions. Recently, to develop a general framework for understanding the relationships between material microstructure and response, a beta titanium alloy has been investigated at the micro-scale to study structure–property correlations and determine critical microstructural features that cause initiation of failure.

THREE-DIMENSIONAL MICROSTRUCTURE RECONSTRUCTION

The three-dimensional microstructure of the beta titanium alloy (Timet 21s) was reconstructed using serial sectioning with optical microscopy and electron backscatter diffraction (EBSD). The microstructure of beta titanium is prototypical of many metallic systems, including stainless steels and high-performance aluminum alloys, thus investigation of this microstructure provides important insights into the behavior of many alloy systems of interest to the Navy. In the serial sectioning process, a fixed amount of material is removed from the sample surface through automated polishing. The sample is then etched to reveal contrast between the microstructural features, and finally images are collected using light optical microscopy. The serial sectioning process is repeated multiple times with a practical limit of a few hundred sections. The process results in a “stack” of images, which can be reconstructed to create the 3D microstructure.

Figure 1 illustrates the serial sectioning laboratory at NRL, where the data were collected. For this study, sectioning was performed on a semiautomatic polisher, which was calibrated to remove 1.5 μm of material

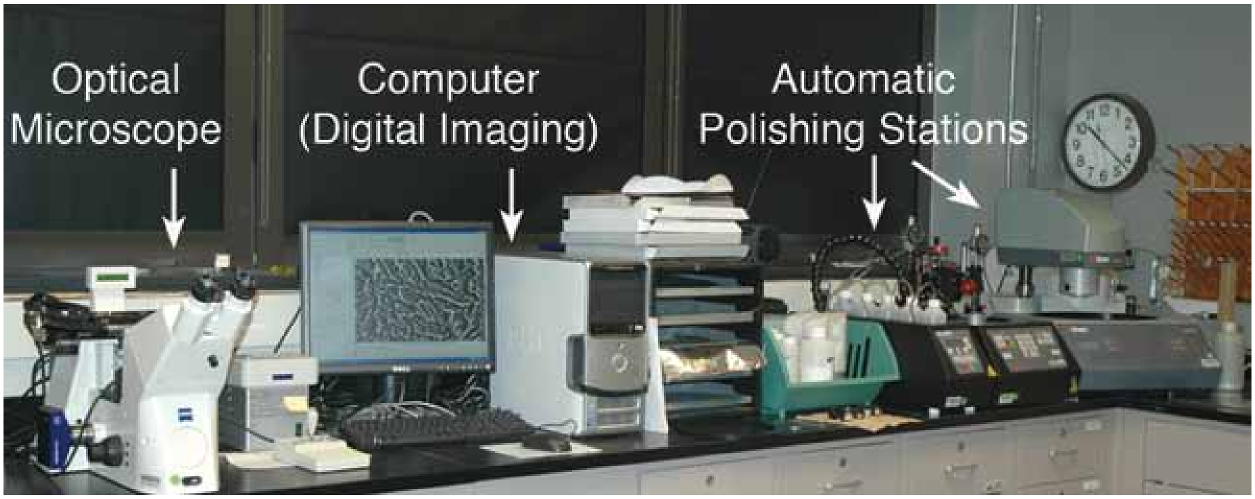

FIGURE 1
Serial Sectioning Laboratory setup at NRL.

per section. After final polishing and etching, a series of tiled light optical micrographs was taken for each section at 500× magnification (see Fig. 2). These image tiles are stitched together to form a single image of that section, representing a large field of view (approximately 1 mm by 0.5 mm), while simultaneously maintaining a high image resolution (<0.6 µm per pixel). Over 200 such image montages were collected and aligned using fiducial marks placed at the edges of the region of interest, resulting in a dataset measuring approximately 1 mm by 0.5 mm by 0.3 mm.

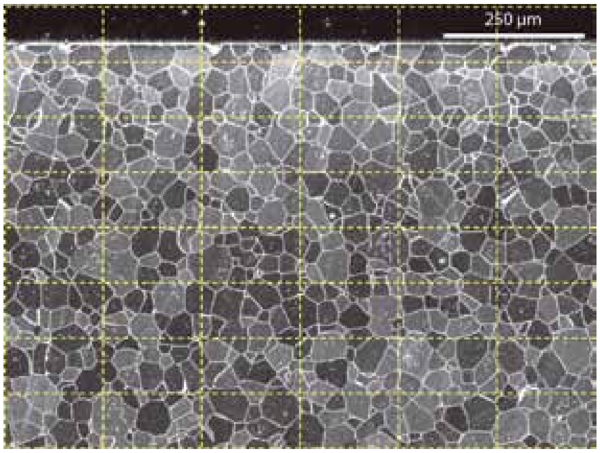

FIGURE 2
Micrograph showing beta titanium grains, from one of the 200 sections that make up the 3D dataset. The yellow lines approximate the size of the individual image tiles that were stitched together to form this image.

After every tenth optical micrograph was collected, EBSD was used to measure the crystallographic orientation of each grain. This technique allows the user to scan an electron beam over a polished surface and measure the crystallographic orientation at each point. This results in a “map” of the crystallography of the specimen. The alignment of these EBSD images to the optical micrographs (and, thus, to the final 3D reconstruction) was accomplished by a semi-automatic alignment routine that matched the position of the center of area of a grain in the optical micrographs of a section with the equivalent center of area of the same grain in the EBSD map for that section. After the two images (EBSD map and optical micrograph) were aligned, the measured crystallographic orientations were then corrected for the EBSD image rotation, and an average crystallographic orientation was assigned to each grain in the 3D reconstruction.

The reconstructed 3D dataset for the beta titanium alloy is shown in Fig. 3. The dataset consists of over 4300 individual grains, and for each grain, the true 3D size, shape, and crystallography have been measured. From this data, a number of microstructural param-

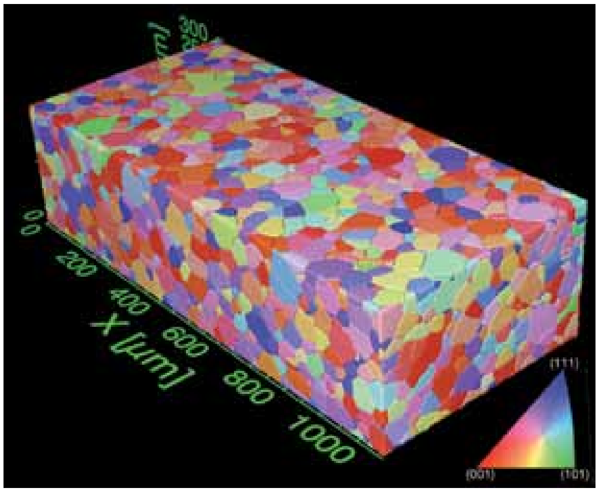

FIGURE 3
3D reconstruction of the beta titanium microstructure. Each grain is colored according to the crystallographic direction parallel to the z-axis (legend at lower right).

eters can be measured or derived, including the true 3D grain size distribution, number of grain neighbors or grain faces, grain boundary curvatures, crystallographic texture, and crystallographic interface normal distributions.[1]

3D IMAGE-BASED FINITE ELEMENT ANALYSIS

In addition to the microstructural and crystallographic information that can be obtained from the 3D dataset, the reconstructed volume can be used as input into simulations of phase transformations, grain growth, or local mechanical response. One of the challenges in producing predictive simulations of materials performance is that typically very little is known about the initial conditions of the microstructure. Many researchers use algorithms to create a virtual microstructure that appears similar to real microstructures, but often these virtual microstructures lack important microstructural details that significantly affect the results of the models. By using the actual 3D representation of the microstructure as our initial condition for simulations, however, we have a perfect representation of the initial state of the microstructure with no additional assumptions or approximations.

The 3D reconstructed microstructures just described have been used as input for image-based finite element modeling (FEM) of mechanical response. Although computation power currently limits FEM simulations based on crystal plasticity to smaller datasets (consisting of up to about a few hundred grains), the results from these simulations are very powerful in that they can be used to determine critical microstructural features where local plasticity failure is likely to initiate.

For ease of computation and analysis, a subvolume containing approximately 100 grains was selected from the larger dataset, and is shown in Fig. 4(a). For this specimen, data were sampled from the original high-resolution dataset so that every third voxel (i.e., volumetric pixel) in the x- and y-directions and every second voxel in the z-direction were represented, to create a volume measuring 136 by 128 by 137 µm, represented by approximately 200,000 voxels. As shown in Fig. 4(b), an FEM mesh was generated that consisted of eight-noded brick elements, with each element corresponding to one voxel in the sampled microstructure. The finite element simulations were performed using ABAQUS™ finite element software with customized anisotropic linear elasticity and crystal plasticity constitutive relationships for the body centered cubic (bcc) beta titanium.

Figure 4(c) is a contour plot of the response of the beta titanium microstructure subvolume. This plot shows the cumulative shear strain as a result of 0.7% applied uniaxial strain in the x-direction, which allows for qualitative visualization of the areas in the microstructure with high local stresses. Plotting the data in this fashion, and relating mechanical response to microstructural features (by mapping back to the 3D reconstruction) allows for identification of features

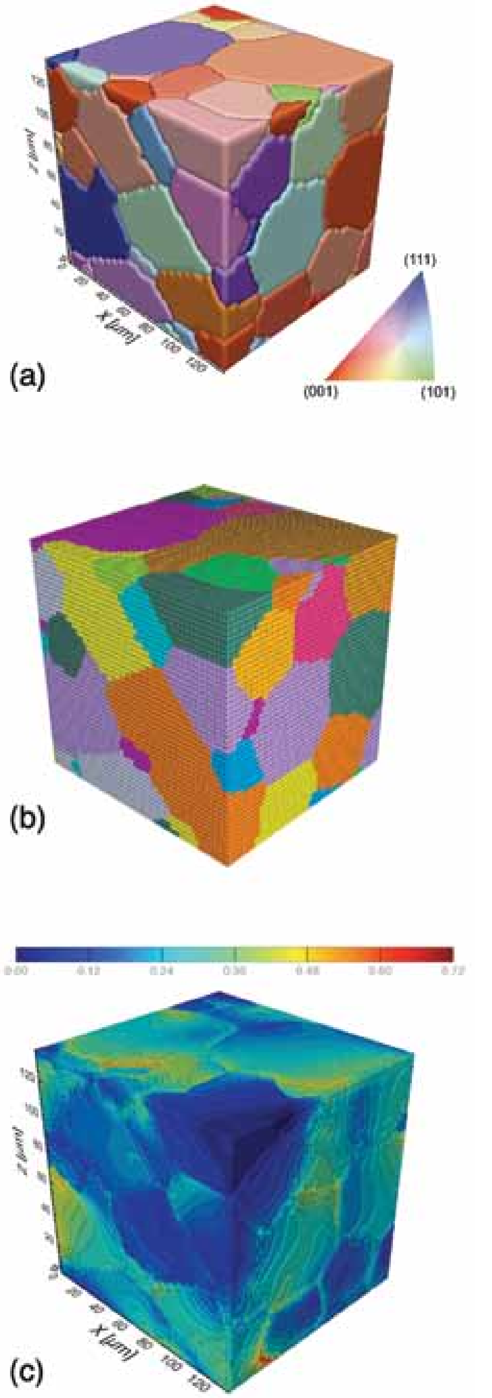

FIGURE 4
(a) Reconstruction of a subset of the titanium microstructure. (b) Finite element mesh of the reconstructed subset. (c) Contour plot of cumulative shear strain in the titanium subset, as a result of a uniaxial tensile load applied in the global x-direction. Figure adapted from Ref. 3.

where failure initiates, and can be used to aid in materials design. One example of such a critical microstructural feature identified by this technique is the initiation of plastic flow at grain boundaries between grains with a high degree of misorientation.[2]

DATA SAMPLING FOR LARGE-SCALE ANALYSIS

Because of the large amounts of memory and computation time required for the simulation of mechanical response of a volume as large as that shown in Fig. 3, it is prudent to sample multiple smaller subvolumes from the larger volume and analyze the responses of these subvolumes in combination to elucidate specific correlations. To investigate the relationship between grain orientation and mechanical response, five subvolumes, each containing approximately 100 grains, were selected from the larger volume. Figure 5 shows the location of these subvolumes within the reconstructed microstructure. The mechanical response of each subvolume was simulated separately, applying the same loading conditions.

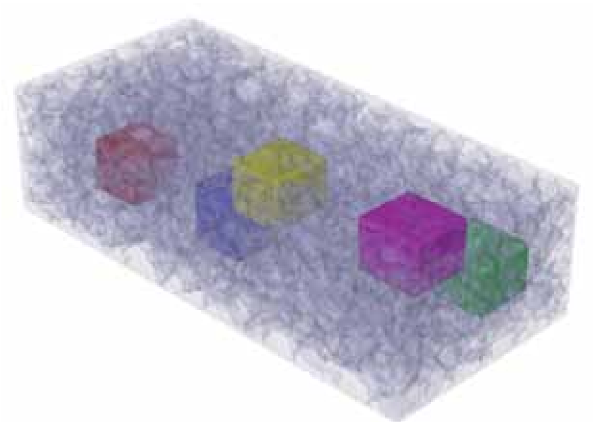

FIGURE 5
3D reconstruction of polycrystalline beta titanium microstructure, showing the location of five randomly selected subvolumes.

To visualize the complex 3D interactions within the microstructure, the scalar mean effective stress for each individual grain was calculated and plotted vs the crystallographic orientation of the grain in each of the five subvolumes. Figure 6 shows one such plot, for the case of uniaxial tensile strain applied in the x-direction. In this figure, the location of each data point on the unit triangle corresponds to the crystallographic direction in that grain that is aligned with the loading axis. (For example, data for a grain with its <001> axis aligned with the global x-direction would be plotted in the lower left corner of the unit triangle.) The value of effective stress is indicated by the color of each data point, according to the scale bar in the figure, with the lower stresses shown in blue and the higher stresses in red. A correlation between crystallography and mechanical response can be seen from this plot; grains with a <001> direction aligned with the loading axis have a lower effective stress in response to the applied load, whereas grains with <101> and <111> directions aligned with the loading axis have a higher effective stress.

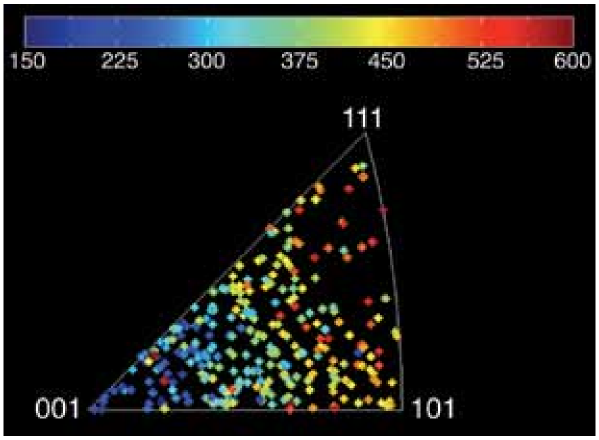

FIGURE 6
Mean effective stress (MPa) vs crystallographic axis aligned with the loading direction for the five subvolumes under uniaxial tension in the global x-direction.

By sampling the data in this manner, it was possible to increase the number of data points without increasing the size of the model beyond the computational limits for the simulation. This method allows for calculation of properties for a statistically significant number of grains, while keeping data sets and file sizes manageable. This type of data can be used to build reliable statistical structure–property correlations that can guide design of materials intended for specific Naval applications.

SUMMARY AND CONCLUSIONS

Three-dimensional materials characterization, analysis, and simulation tools developed in the Multifunctional Materials Branch have been applied to examine the role of microstructure on material behavior. These tools are used to determine structure–property correlations in Naval materials, and to facilitate efficient computational materials design. In this study, this is demonstrated for a titanium alloy, where data sampling from a large reconstructed volume was used to reveal a correlation between crystallography, applied load, and mechanical response.

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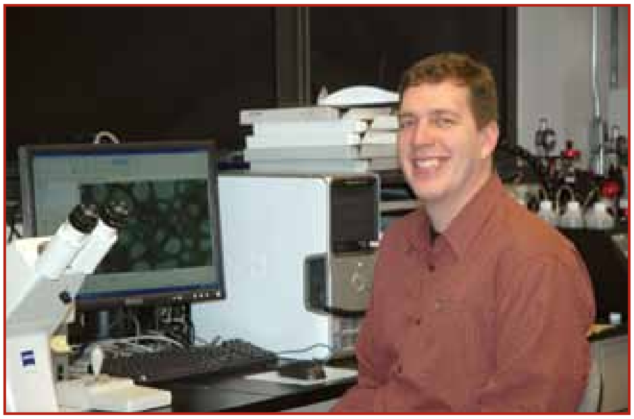

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