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Using the Processing, Properties and Characterization of Brass to Teach the Differences Between Crystal Structure and Microstructure

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Introduction

This paper discusses using the materials characterization techniques of optical microscopy and scanning electron microscopy to clarify the fundamental concept of scale in microstructure, a challenging concept for undergraduate and beginning graduate students. The activities described are used in Materials Engineering at San Jose State University in a required undergraduate laboratory course, Principles of Scanning Electron Microscopy (MatE 143). The course primarily focuses on teaching the theory and operation of the scanning electron microscope (SEM). It also provides a forum to examine materials on different scales (from Angstrom to centimeter) using complementary techniques, and thus improve student understanding of the relative scale of different microstructural features. The students enrolled in the class vary in pre-requisite knowledge, but all have had an introductory materials science course and are at least familiar with the concepts of crystal structure and polycrystalline materials microstructure. In this paper we discuss the learning module developed for the course as well as some preliminary results on the effectiveness of the materials in promoting student mastery of the learning objectives. The α-brass materials system was used in this work because of the ease with which the microstructure can be altered. Thus much of the discussion that follows relates to a bulk polycrystalline metallic system.

Crystal Structure and Microstructure

Crystal structure and microstructure are important in materials science and engineering, as the key determinants of materials properties and performance. Crystal structure describes the periodic arrangement of individual atoms or molecules and generally refers to nanometer scale arrangements. Microstructure describes the order of a material on a larger scale (nanometers to centimeters), and has features that often include disruptions of ideal crystalline order. Microstructure is a broad descriptive term, encompassing many concepts, including phases, grain boundaries, dislocations, twinning, subgrains and crystallites, intra- and inter-granular strain, crystal orientation and texture.

The difference in the two concepts lies in the scale of atomic arrangement being described. Crystal structure describes the basic structural building block of a crystalline solid. Specifically, every possible crystal structure can be uniquely described according to its particular atomic or molecular spacing in the x, y and z direction, as well as the angular relationships between the axes α, β and γ. The crystal structure system allows classification of different solids according to their basic atomic arrangement.

If the order of atomic arrangement continues over a long range, at least hundreds to thousands of atomic radii, the material is classified as a crystalline material. Crystalline phases are distinctly identified by their composition and crystal structure.

In polycrystalline materials, the atomic order is broken, and boundaries exist between different regions of the same phase, or of different phases. These different regions may be referred to as grains, sub-grains, or crystallites, depending on the scale of the variation as well as on the definition of the boundary. The interruptions to atomic order may be visualized in terms of the orientation of the crystal plane where it intersects a surface. When the crystal is unperturbed, the plane orientation, indicated by a vector normal to the plane, continues unchanged throughout the observed surface. Crystal orientation changes of between 1 to 10 degrees create very thin (nanometer wide) regions called low angle grain boundaries (LAGBs), and where they occur, they separate a crystal into subgrains, or crystallites. High angle grain boundaries (HAGBs), with misorientation between 10 to 15 degrees, are a discrete change of crystal orientation. These regions are defined as the demarcation between grains. Grain boundaries render a material a poly-crystal. The long-range collection of grains, grain boundaries and other defects in a material sample are collectively termed "microstructure".

It may confuse novice learners that HAGBs and LAGBs may each be detected or imaged by some characterization techniques and not by others. For example, optical microscopy of a polished and etched polycrystalline metallographic specimen can reveal a grain structure defined by high angle grain boundaries, while XRD analysis may reveal a crystallite size on the same sample that is much smaller and impossible to visualize optically. Care must be taken in comparing such data, as the XRD crystallite size refers to the size of a coherently scattering domain rather than to higher angle "grain boundary." The concept of crystallite size can be very important in modern engineering materials such as epitaxial thins films, nanomaterials, and electronic and optical devices made from single crystals. Thus a deeper understanding of the scale of various crystalline microstructures is important to the materials engineer and scientist.

Student Comprehension of Crystal Structure and Microstructure

In introductory materials science, students learn about crystals as areas of repeating atomic units of identical orientation interrupted by disordered grain boundaries. This simple model is reinforced by characterization techniques like metallography. Crystal grains and grain boundaries are revealed through sample preparation (grinding, polishing and etching), and viewed under magnification. The macroscopically observable structure is treated as a mirror of the underlying atomic arrangement. The nuance of crystallite orientation is absent from this model.

A multiple choice quiz was developed to measure conceptual understanding of structure. It included questions on equilibrium crystal orientation within and across grains, and the effect of cold working and heat treatment on that orientation. The quiz was administered to an

undergraduate introductory materials class, at the close of the Fall 2012 semester. The quiz questions are shown in Table I.

The students who took the quiz are from all engineering majors except electrical engineering. The average overall score was surprisingly low (48%). The poor performance was likely influenced by the quiz being voluntary and ungraded. It was also given at the end of the semester, when students were subject to a number of other distractions. The results suggest student difficulty with the nuances of microstructure and crystal structure, but are not conclusive. We plan to repeat the quiz, for credit and during the microstructure and heat treating portion of the class.

The Electron Backscatter Diffraction Technique

Intra-granular atomic arrangement can be determined by Electron Backscatter Diffraction (EBSD), a technique performed in conjunction with scanning electron microscopy. In EBSD, the top atomic layers of a highly polished material diffract the incident electron beam. As in transmission electron microscopy, the resulting electron diffraction pattern at any point is uniquely determined by the crystal structure that the beam is incident upon. Computerized methods allow the patterns to be easily indexed to the corresponding crystal structure and orientation. This technique was previously constrained by the time required to process the patterns and derive the structure. Computing power was the barrier to automating this process. Now, it is possible to collect and process many patterns per second using computer automation, allowing high resolution scans. EBSD maps each point on a sample's surface with its specific crystal orientation. The internal variations in crystal orientation may be combined with metallography, electron microscopy and elemental characterization to present crystal structure and microstructure in easy-to-conceptualize image form.

To correctly interpret the results of EBSD, the students must, of course, be able to systematically understand the instrument operation, experimental techniques and resulting data. General experimental factors include systematic errors, and the correct interpretation of the role of errors due to instrumentation. Concepts specific to the characterization techniques themselves must also be mastered. For example, in SEM, the beam interaction volume affects the size of the sampled region. For accurate EBSD results, the surface must be carefully prepared and free of deformation to avoid artifacts.

The α-Brass System

The present work derives from experiments performed as part of a graduate research project, with the objective of characterizing alpha-brass (α-brass), a common polycrystalline system, via several methods and comparing the results.

Question 1. In general, the change in crystal orientation across a grain boundary is:

a) less than 10 degrees
b) more than 15 degrees (42%)
c) 45 degrees
d) there is no change in orientation

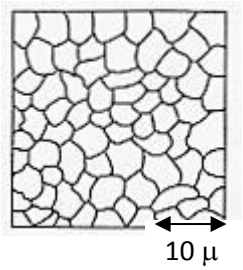
Question 2. In general, the change in crystal orientation within a grain is:

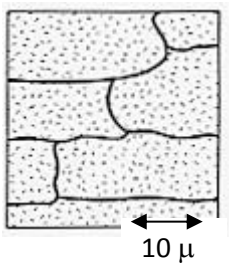
a) less than 10 degrees (41%)
b) more than 15 degrees
c) 45 degrees
d) there is no change in orientation

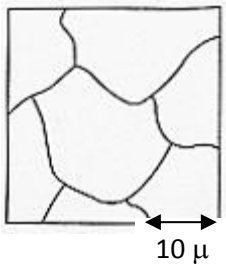
Question 3. After cold-working, a metal's microstructure can best be described as having:

a) large equiaxed grains
b) strain free grains
c) deformed grains (64%)

Question 4. After cold working and recrystallization, under optical microscopy, a metal's microstructure will most likely resemble:

a) 

(55%) b) 

c) 

Question 5. After cold-working, the atoms within a grain will exhibit (select all that might be true):

a) long range order
b) short range order (43%)
c) disorder within each grain (33%)
d) no change from their equilibrium position

Question 6. After recrystallization, the atomic arrangement within a grain could best be described as having (select all that might be true):

a) long range order (45%)
b) short range order
c) disorder within each grain
d) no change from their equilibrium position (0%)

Question 7. After cold working, a metal will (select all that might be true):

a) have anisotropic properties
b) have increased dislocation density
c) have sub-grains delineated by low angle grain boundaries
d) all of the above (55%)

Table I. Concepts quiz given to introductory materials class. The correct answer, and the percentage of students who selected that answer, is shown in bold text.

Brass is a particularly versatile system. Its microstructure is easy to manipulate via cold working and heat treating, using readily available laboratory equipment (rolling mill and furnaces). By such treatments it can be made to demonstrate essentially all fundamental metallurgical principles, including work hardening, recovery, recrystallization, grain growth, twinning, etc. These characteristics make it a very common, and useful, system for teaching materials science. The α-brass was cold-work and recrystallized, and then characterized with metallography (consisting of optical microscopy and hardness measurements) and electron back-scatter diffraction (EBSD). The processing and characterization steps are shown in flow-chart form in Figure 1. The specific processing steps were:

- Anneal as-received brass, at 425°C for 60 minutes, then 45 minutes at 300°C, in air. This step removes any strain and texture that may be present in the as-received material specimens.
- Cold roll specimens to 40% reduction in thickness
- Anneal at 425°C and 550°C for 1 hour to achieve recrystallization and grain growth.

The resultant properties of the α-brass followed the usual expectation for cold work and recrystallization. Specific results are given in the discussion of the EBSD results, but are briefly summarized in Table II.

EBSD Characterization Module of α-Brass for a Scanning Electron Microscopy Class

The α-brass experiment and characterization results were incorporated into teaching and laboratory modules for the Materials Engineering 143 Electron Microscopy class. The goals of the modules were to introduce EBSD as a materials characterization technique, and to help students distinguish between the concepts of microstructure and crystallography in visual and intuitive ways. The specific learning objectives are listed in Table III. The lecture topics are listed in Table IV.

Since most of the students in the class had an introduction to materials course, the lecture first reviewed the basic concepts (crystal structure and microstructure, and cold work, recovery recrystallization and grain growth) then went on to discuss the phenomena in more detail, and specifically in terms of effect on grain and crystal structure.

The EBSD technique was presented as a materials characterization tool, and as a way to understand the above concepts. In the experiment (Table V) the students performed EBSD scans on annealed, cold-rolled and recrystallized α-brass and compared their results to a set of reference data consisting of optical and SEM micrographs of etched α-brass, and hardness data (Figures 2,3 and 4). By combining multiple modes of characterization within the context of material structure, we hoped to make a meaningful experiment for the students, while demonstrating the significance of the "black-box" EBSD technique.

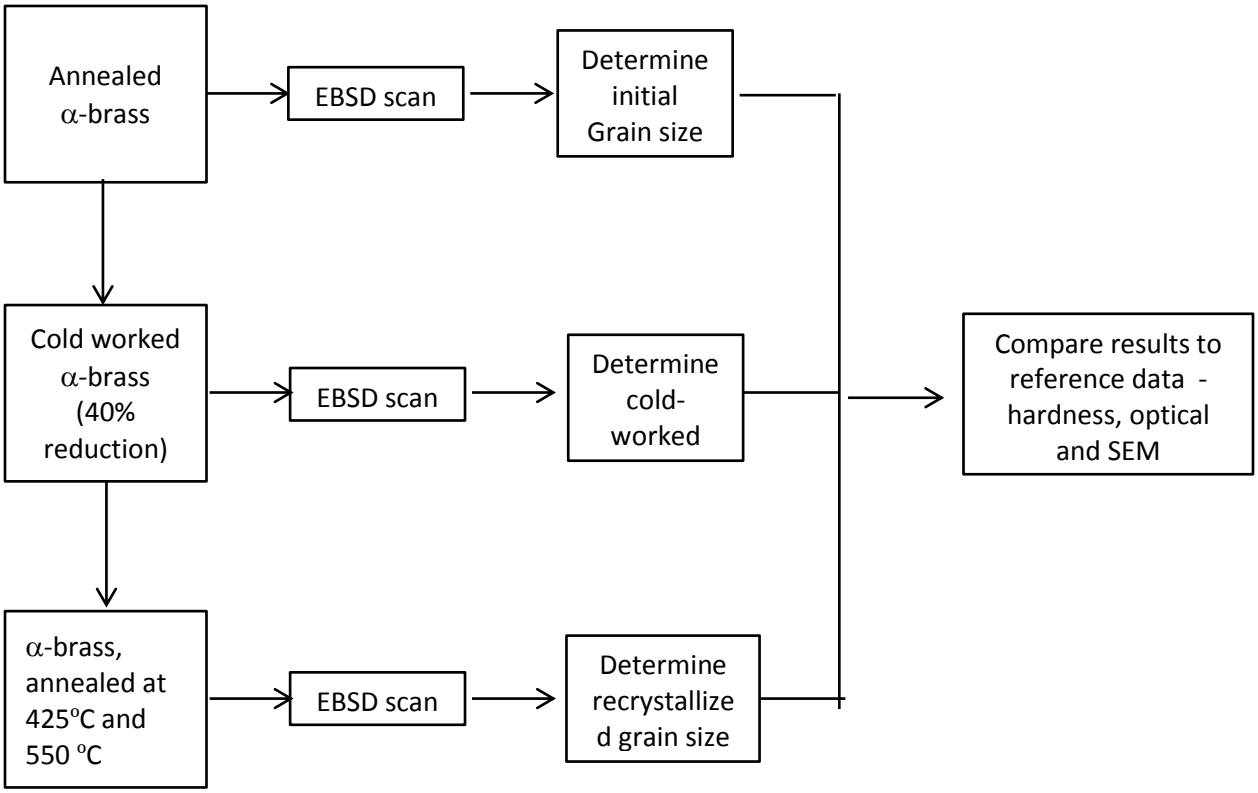


Figure 1. Flowchart of α-brass processing and characterization.

	Hardness	**Grain size by optical microscopy**
As-received α-brass, annealed	Low	Large
Cold worked	Increases with degree of cold work	Elongated grains, with increased dislocation density indicated by visible strain lines
Recrystallized	Decreases with increasing time and temperature	Large, equiaxed, strain-free grains

Table II. Metallographic characterization results.

After completion of the EBSD Brass Characterization Module in MatE 143, students will be able to:

- Produce EBSD grain orientation plots by defining the angular threshold
- Produce grain size distribution plots
- Articulate assumptions inherent in using EBSD

- Define grains and grain boundaries
- Explain the concept of low and high angle grain boundaries

- Identify the transitions between recovery, recrystallization and grain growth
- Correlate physical properties with microstructure

Table III. Learning objectives for the MatE 143 Principles of Scanning Electron Microscopy class module.

- Review Crystal Structure and Microstructure
- Define and illustrate low and high angle grain boundaries

- Review General Concepts of Cold Work, Recovery, Recrystallization and Grain Growth
- Effect on Mechanical Properties
- Discuss Effect of Recovery, Recrystallization and Grain Growth on Crystal Structure and Microstructure

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- Introduce EBSD Technique (Operation covered in Lab)
- Review Experiment
    - Process flow for Alpha brass samples
    - EBSD Scans
    - Produce orientation and grain size distribution plots
    - Compare to Provided Optical and SEM Micrographs, Hardness

Table IV. Lecture topics for the EBSD Brass Characterization module.

- Demonstrate EBSD system operation
- Students perform EBSD data acquisition scans on a-brass samples:
    - Pre-cold worked brass sample
    - Cold worked brass (40% reduction in thickness)
    - Annealed brass (1 hour at 425ºC, 550ºC)
- Using EBSD system software, analyze data by preparing orientation maps, and grain size distribution plots.
- In written laboratory report, consider the following questions:
    - From the reference data, can you determine the temperature at which recrystallization starts? When is recrystallization complete?
    - Compare the Orientation maps for the pre-cold worked and 40% cold worked samples. Describe the changes in the microstructure that occur with cold work. Describe the changes in crystal structure that occur with cold work.
    - Compare Orientation maps of 40% cold worked and heat treated at 425ºC and 550ºC. When does recrystallization begin? Describe how orientation changes occur in the cold worked material upon heating.
    - How do the changes in the orientation correlate with the hardness?
    - Compare the grain size for the 4 samples. How does the grain size correlate with the hardness?

Table V. Experimental steps for the EBSD Brass Characterization module.

(a)

(b)

Figure 2. Optical micrographs of α-brass provided to students as reference data. a) Pre-cold worked and b) cold worked to 40% thickness reduction

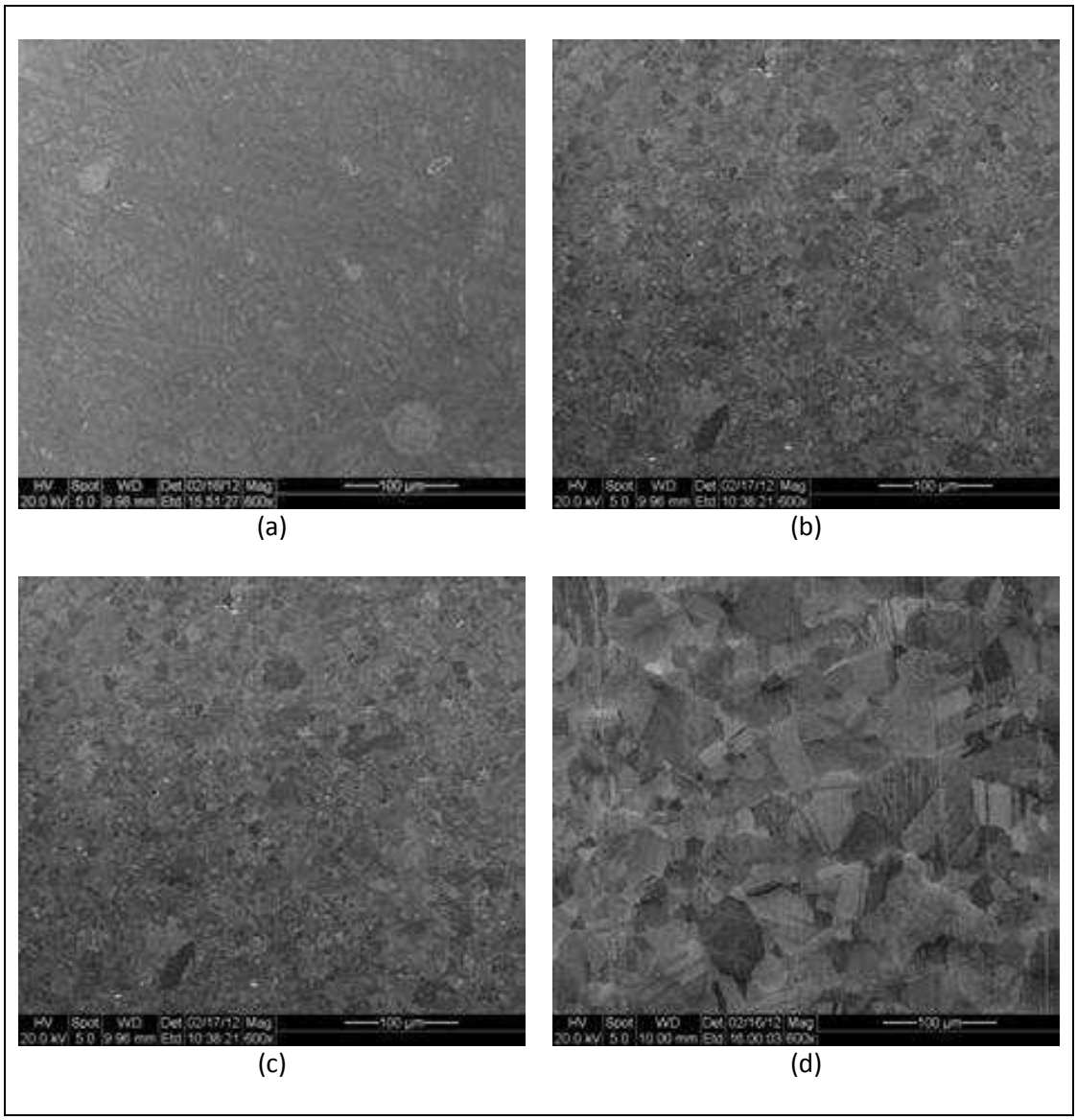

Figure 3. Electron micrographs of α-brass provided to students as reference data. a) Pre-cold worked, b) cold worked to 40% thickness reduction, c) and d) annealed 1 hour at 425 and 550°C respectively.

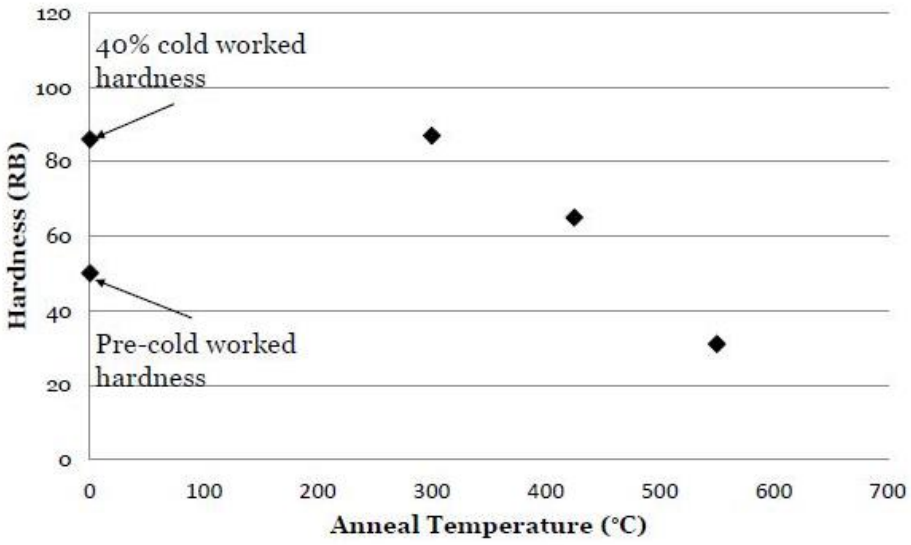

Figure 4. Rockwell B Hardness of α-brass provided to students as reference data.

EBSD Results

The Brass Characterization module was used in the Spring 2012 SEM class, at the end of the semester. To perform the experiment, the class used an FEI Quanta 200 scanning electron microscope, an EDAX characteristic x-ray spectroscopy system, and an EDAX EBSD system. By the end of the semester the students were all proficient in the use of the SEM system, so the EBSD data collection and analysis were performed without any difficulty.

Orientation and grain size for the samples, as derived from the EBSD data, are shown in Figures 5 and 6. The raw data from a scan consists of the crystal orientation at each point. Therefore, it is straightforward to create an orientation map. First, a color is specified for each direction normal to the crystal orientation. For example, as seen in the legend for the present maps (Figure 5e), [100] is specified by red, [101] by green and [111] by blue. Then, the appropriate color is plotted and displayed by a computer program at each point on the scanned area. To obtain the grain size distribution, grain conditions are defined by the user, by specifying, for example, the degree of crystal misorientation required for a grain boundary.

The orientation maps for the pre-cold worked and both annealed samples compare well with the optical and electron micrographs. Grain boundaries, and the presence of annealing twins, are obvious from all three types of images. The EBSD map adds the additional dimension of orientation; preferred orientation and the angular relation of annealing twins are readily identifiable.

The cold-worked sample required a bit more interpretation to compare the three types of images. From the optical micrograph of the etched a-brass, while strain marks are visible, the clearly

delineated grain boundaries suggest that the original grain structure is maintained after cold work. Due to the contrast mechanisms in the SEM, the grain boundaries are less defined in the electron micrograph, but the SEM image again suggests grain structure. However the orientation map reveals that the original microstructure has been fairly nearly obliterated by the cold work, leaving instead a collection of crystallites.

This same conclusion can be reached by interpreting the same EBSD data as grain size distributions. The pre-cold worked sample has a grain size between 10 and 100 microns; the majority of grains are one micron or less for the cold-worked sample. With recrystallization and grain growth, the grain size again approaches the 10 to 100 micron range.

Pedagogical Results

Student improvement was measured by a quiz of preliminary understanding, given before the start of the module, and by a report written after the completion of the module. The best initial understanding was demonstrated by graduate students. This is as expected; more experience should translate to better concept mastery. In the written laboratory report, the students were able to draw appropriate conclusions from the EBSD and conventional metallographic data. All students showed improvement in concept mastery following completion of the lecture, experiment and written assignment.

Conclusions and Future Work

Overall we judged the module to be a success. It appeared to improve students' conceptual understanding of crystal structure and microstructure. Based on this initial work, we have identified a number of further areas of exploration.

The SEM class size is small (14 students or less), and of mixed makeup, composed of undergraduates, graduates, material engineering and other engineering and science majors. Since many undergraduates transfer to our university after completing their lower division classwork at community college, the students have frequently taken their introductory materials class at another school. Additionally, they may or may not have had additional undergraduate or graduate materials classes. We plan to repeat the baseline quiz with the introductory materials class, as well as with SEM class, to see if results are consistent over several groups, and to allow meaningful statistical analysis. Additionally in regards to the baseline quiz, we plan to refine the questions, as well as administer it as a mandatory, for credit assignment. This will be in conjunction with repeating the module with the SEM class.

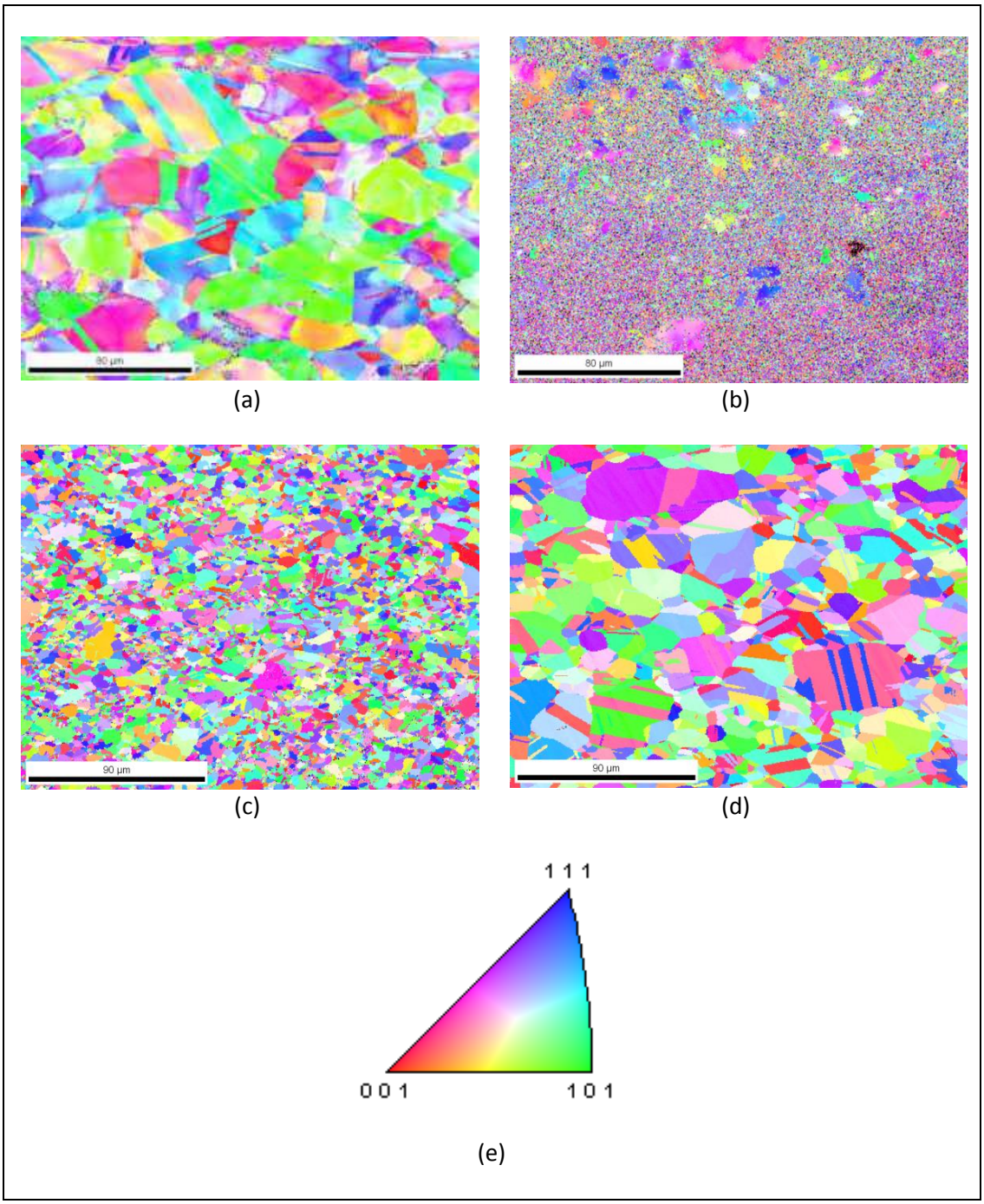

Figure 5. Orientation Maps of α-Brass determined from EBSD scan. a) Pre-cold worked, b) cold worked to 40% thickness reduction, c), and d) annealed 1 hour at 425 and 550°C respectively. The map legend is shown in e).

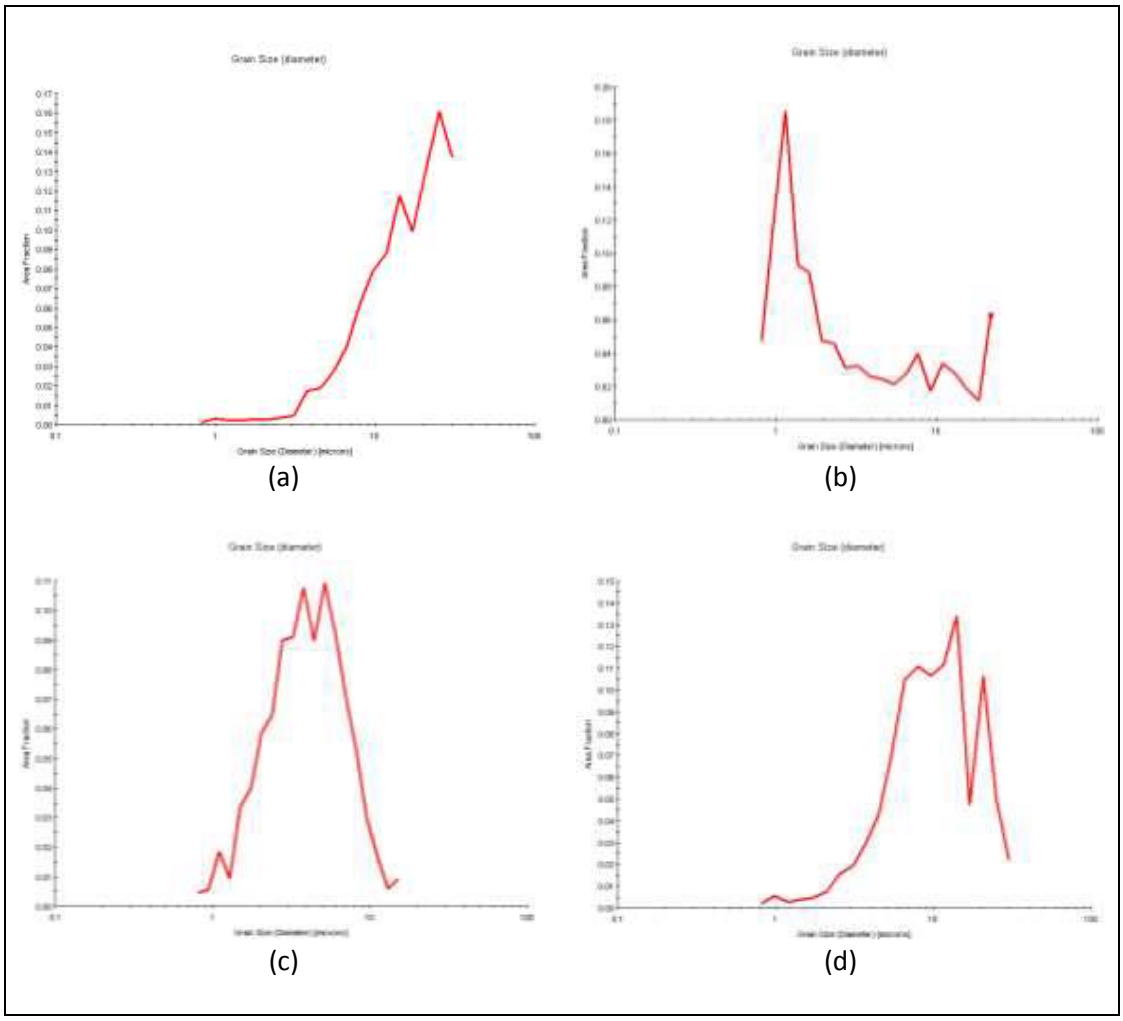

Figure 6. Grain Size Distribution of α-Brass determined from EBSD scan. a) Pre-cold worked, b) cold worked to 40% thickness reduction, c), and d) annealed 1 hour at 425 and 550°C respectively.

Based on the success of this effort, we hope to expand the work to two additional Materials Engineering classes, the X-ray Diffraction laboratory class (MatE 144) and Metals and Alloys (MatE 154). These classes both deal with crystal structure and grain structure, and are an appropriate forum for using materials characterization to explore cold-worked and annealed brass. For the Metals and Alloys class, the EBSD data could be presented as part of a brass metallography lab. For X-Ray Diffraction, a unit might be developed in which students determined the instrumental peak broadening, and, correcting for that, determined crystallite size and compared to the EBSD results.

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