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Materials Science Rocks! Using Geology Specimens to Teach Microstructures and Error Analysis

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Introduction

Structure-property-processing-performance relationships are central to the discipline of materials science and engineering (MSE). Undergraduate MSE curriculum often focuses on engineering materials, such as steels, technical ceramics, and synthetic polymers, to teach about microstructural features and standard test methods. For instance, the well-known Hall-Petch equation relates the strength and hardness of a metal to its grain size. Students can examine this relationship in a lab by testing brass annealed under different conditions. The students measure the material's grain size using optical micrographs and test the mechanical properties through tensile or hardness testing.

Today, there are hundreds of thousands of different materials used for a wide variety of purposes, including engineered and natural materials. Throughout civilization, humans have used materials found in their natural environment, including animal skins, mud, wood, and rocks. Modern buildings are often constructed with wood frames, clay tiles (made from mud), and stone. Many characteristics, such as crystallography and microscopic structures, are shared between natural and modern engineering materials. For instance, grains are visible in stainless steel microstructures (Figure 1a) and naturally occurring aeolian sandstone (Figure 1b). The MSE curriculum should teach students about the full spectrum of materials used in engineering, including natural materials.

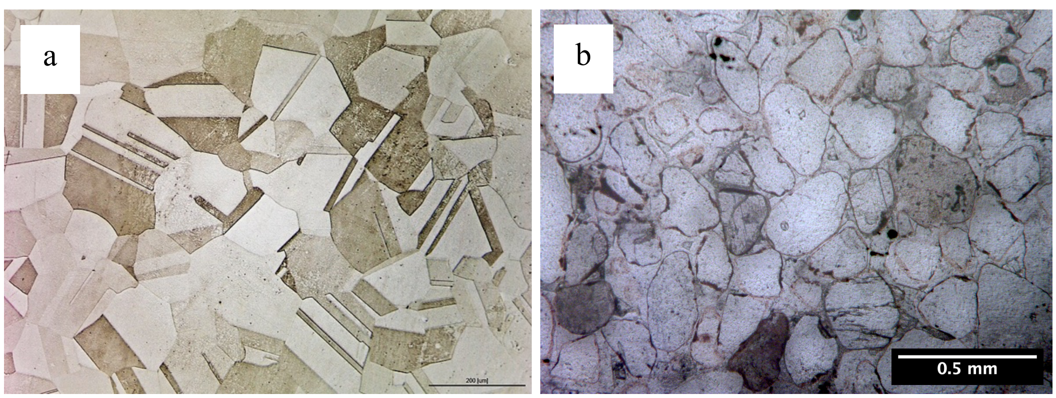

Figure 1. Microstructures of (a) stainless steel[1] and (b) aeolian sandstone.

This paper reports on a geology-based laboratory module for an introductory MSE course. This lab occurred at the beginning of the term, so it could only require minimal course content. The aim was to introduce students to MSE concepts, such as quantifying microstructures, while reinforcing measurement error principles taught in prerequisite courses. The learning goals for the lab were to:

- Calculate measurement errors,
- Analyze feature sizes and size distributions, and
- Evaluate sources of uncertainty in microstructural analysis.

Geological Specimens

Depending on their formation, rocks can be classified as igneous, sedimentary, or metamorphic. Much like manmade alloys, igneous rocks form from a melt, whereas metamorphic rocks form from existing rocks that are exposed to high temperatures and pressures that cause physical and chemical changes. On the other hand, sedimentary rocks form from compaction and cementation of rock particles ("sediments") through weathering and erosion of existing rocks. The structure of sedimentary rocks is related to their underlying sediments and may contain fossils or layered, elliptical grains called "ooids" [2]. This lab investigated sedimentary and igneous rocks, with ironstone and scoria emerging as the most promising candidates.

Ironstone is an iron-rich sedimentary rock with a minimum composition of 15% Fe by weight [3]. Historically, ironstone contained sufficiently high grades of iron that could be smelted to extract iron metal. However, the minimum composition for smelting has increased from 28% Fe in the mid-twentieth century to 60% Fe for modern ore [3]. Lower-grade ironstone is now used as a building material. Most ironstones contain minerals such as oxides, silicates, and carbonates [2]. Some of these structures may be visible in optical microscopy. For instance, quartz (SiO_2) appears grayish, whereas the iron-containing compound is often oxidized and appears dark red.

Scoria is an igneous rock that forms when a volcano ejects magma. The magma may have significant amounts of trapped gasses that can form vesicles and bubbles [4]. Considerable variations in the pore structure and vesicle sizes can lead to a wide range of densities. Visually, the rock appears dark, and the open vesicles can be easily seen using optical microscopes.

Petrographic thin samples are commonly analyzed using optical microscopy. A standard sample preparation method [5] creates thin sections cut from a larger rock, mounted to a glass slide, and polished to a standard thickness of 30 μm. Some materials, such as ironstone and volcanic scoria, can be easily viewed using basic planar or stereographic microscopy methods. However, many geologic materials are viewed with polarizers and filters using transmitted optical microscopy. For instance, quartz grains are difficult to distinguish under unpolarized light, as all grains are transparent and have the same refractive index. However, polarized light interacts differently with the birefringent grains, which have differing crystallographic orientations, causing the grains to have varying contrast. Microscope features such as retardation plates can further enhance the images and distinguishing features. Nikon's MicroscopyU shows examples of phyllite and oolite, Figures 8 and 9 of Ref. [6], respectively, under differing illumination conditions. However, some microscopes may not have sufficient capabilities, or the operators may not have adequate expertise. Thus, some geologic materials are more suitable for introductory courses or outreach opportunities than other materials.

Experimental Setup

This work investigated four commercially-sourced petrographic thin sections. The students' assignment focused on ironstone[7], but the authors also evaluated three other petrographic slides: volcanic scoria, aeolian sandstone, and oolitic limestone. For reference, the four petrographic thin sections were purchased from Northern Geological Supplies Limited in August 2024 for a total of £103, including international shipping to the United States. The samples were imaged using Nikon Optiphot planar optical microscopes in the department's undergraduate teaching laboratory. The microscopes are equipped for reflected and transmitted light microscopy, with polarizers and Nomarski prisms available in reflection geometry. A low-cost, hand-held USB microscope was also utilized to evaluate the four samples.

ImageJ [8] was used to measure particle sizes from the petrographs. For a single particle, the maximum Feret diameter was measured manually using the straight segment tool. The image was adjusted before measuring the particle size distribution. The image was converted to 8-bit greyscale, and the Threshold setting was applied to convert it to black and white. The Erode and Fill Holes functions were also used to clarify boundaries and remove speckles, respectively. The area and Feret diameter were selected within the Set Measurement function. When the Measure Particles function was applied, the settings were selected to measure particles between 100 and Infinity pixel units with the Exclude on Edges and Overlay options. The table of resulting particle sizes was copied into Excel for analysis. The area-weighted average Feret diameter was calculated as

$$\bar{d}_A = \frac{\sum_{i=1}^{N} A_i L_i}{\sum_{i=1}^{N} A_i}, \qquad \text{Eq. 1}$$

where A_i is the area of particle i and L_i is the Feret diameter of the particle. The area-weighted cumulative size distribution was plotted, and the students determined the tenth, fiftieth, and ninetieth percentiles of Feret diameter, d_{10}, d_{50}, and d_{90}, respectively [9].

Laboratory information was communicated with students through a lab manual and course web page. For this lab module, students watched pre-lab videos that introduced them to the lab and completed a pre-lab quiz based on the videos. The 50-minute lab sections were supervised by teaching assistants who introduced students to the lab space and discussed safety policies and procedures; the remainder of the time was open for imaging with optical microscopes. The online course materials also included instructional videos on using ImageJ and completing the grain size analysis. The prompts for students' analysis are given in Appendix A; additional course materials, including solutions, are available upon request from the authors.

Petrography Results

For the lab, students captured ironstone images using reflected light microscopy, similar to that from Figure 2a, investigating different regions and magnification levels. The Feret diameter of a single particle (Figure 2b) was measured five times. In this example, the selected particle's average Feret diameter was 0.159 mm, with a standard deviation of 0.002 mm. However, the sample includes particles with a range of sizes. Students also created a cumulative size distribution using all the particles from Figure 2a. They compared the variation in their single-particle measurement with the variation quantified by the d_{10}, d_{50}, and d_{90} of all particles. These results are not included in this publication as they contain the answers to the lab.

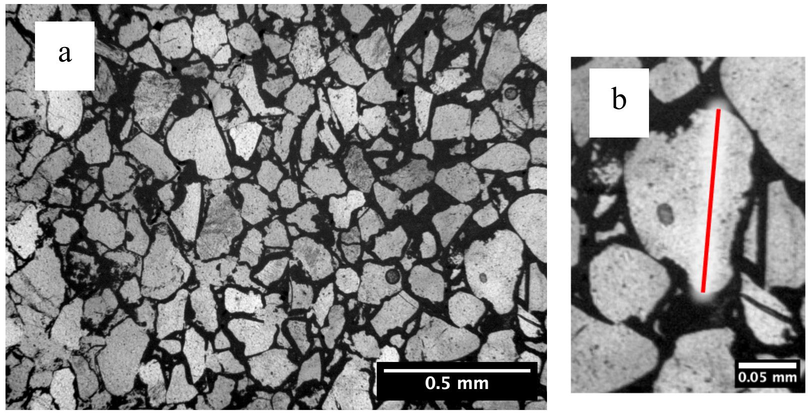

Figure 2. Reflected light optical microscopy of ironstone showing many particles (a) and the Feret diameter measurement of a single particle (b). The Feret diameter is indicated by the red line extending across the particle's longest dimension.

Ironstone was easily visualized under all imaging modes investigated. The sediment particles were visible in both reflected and transmitted light microscopy, Figure 2a and Figure 3b, respectively. The low-cost USB stereomicroscope also resolved the particles (Figure 3a). These petrographs are consistent with ironstone primarily composed of quartz particles, with a smaller volume fraction of iron oxide compounds that cement the grains together. The birefringence of the silica grains is apparent in Figures 3c and 3d since rotating the filter caused the grains to change color and brightness. The dark matrix is the iron oxide. The reddish hue of the iron oxide is visible in the USB microscope, whereas it appears gray in all planar microscope images. This discrepancy is likely due to an error in the camera's white balance.

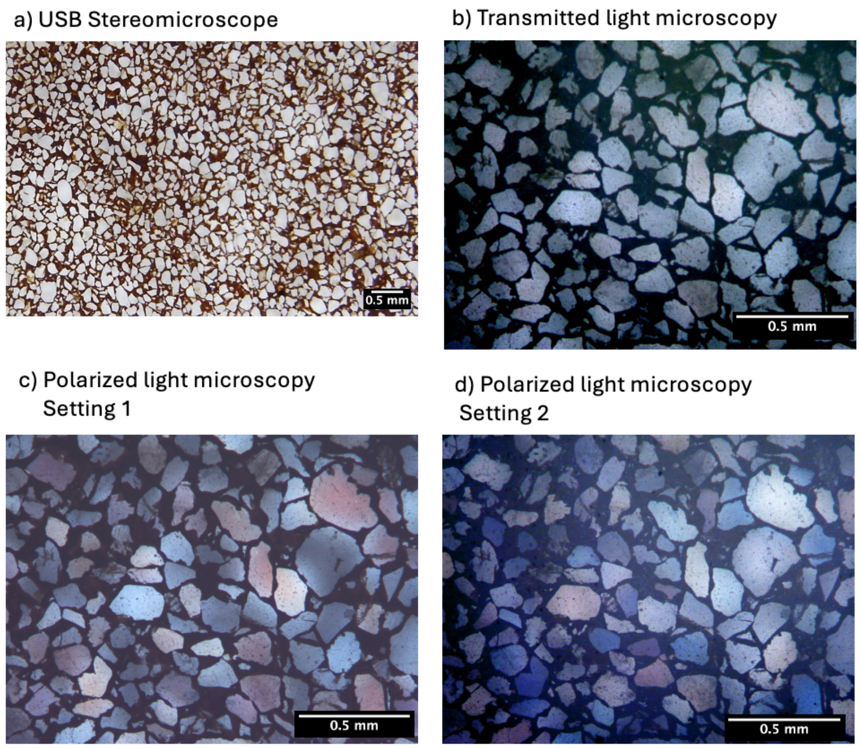

Figure 3. Ironstone sample as seen under different imaging modes: (a) low-cost USB stereomicroscope, (b) transmitted light optical microscopy, and (c, d) polarized light microscopy using different rotations of the polarizers.

Three other geologic specimens were also evaluated. Volcanic scoria (Figure 4) was easy to visualize under the USB stereomicroscope, as shown in Figures 4a and b. The dark rock and light porosity were easy to visualize, and secondary particles, such as the one in Figure 4b, were also observed. However, viewing the scoria in the optical microscope (Figure 4b) was more challenging, as some features were quite large, even under the lowest magnification. The sedimentary particles in aeolian sandstone (Figure 5) could be visualized using optical microscopy, but this was aided by carefully adjusting the lamp power, aperture, and diaphragm to improve the optics. The individual grains and boundaries were apparent using standard optical microscopy (Figure 5a), with polarized light highlighting some grains due to their orientation (Figure 5b and 5c). The USB stereomicroscope did not sufficiently visualize the grains, so this was not included. Finally, oolitic limestone (Figure 6) was the most difficult to observe. With moderate adjustments to the microscope, the layered structure of the elliptical oolites was apparent. However, it was difficult to adjust the microscope under either dark field reflected microscopy (Figure 6a) or transmitted light microscopy (Figure 6b); it is worrisome whether students with teaching assistants could consistently obtain useable petrographs for quantitative analysis.

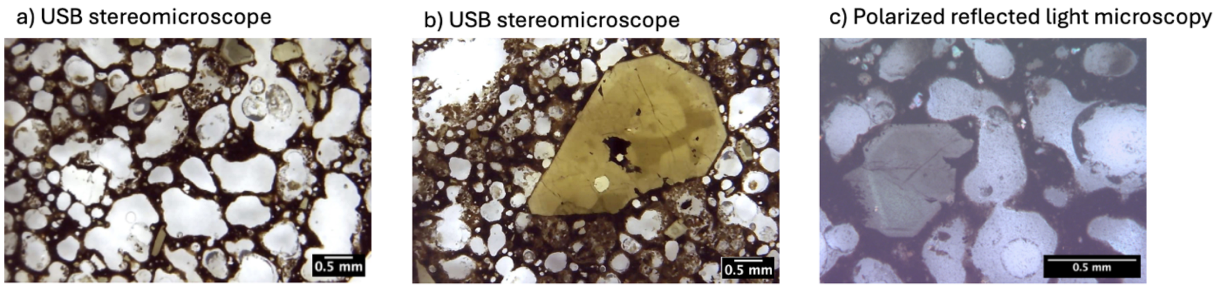

Figure 4. Volcanic scoria as seen under a USB stereomicroscope (a, b) and polarized reflected light microscopy (c).

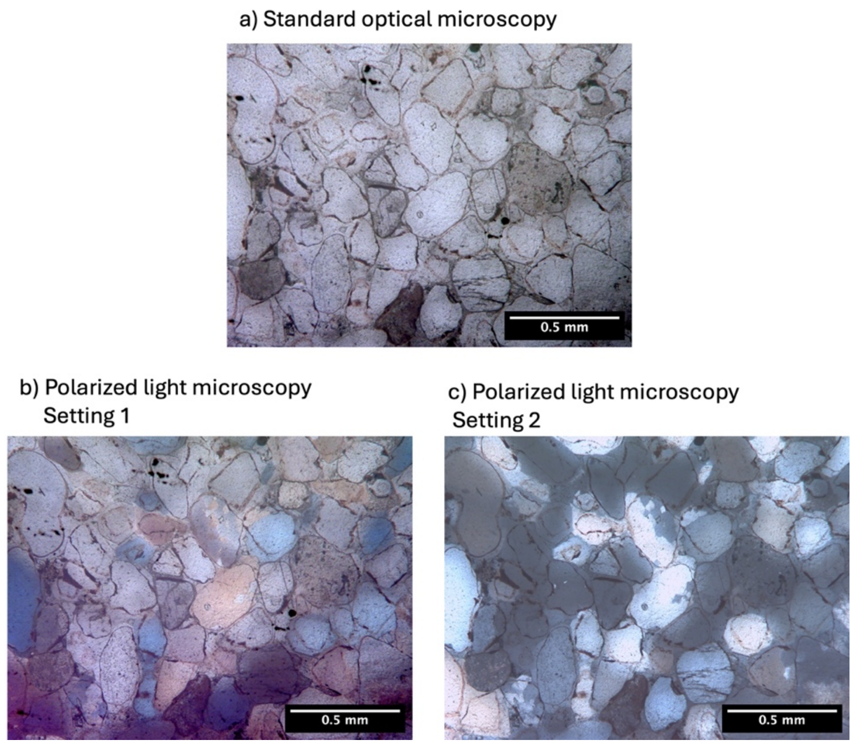

Figure 5. Optical microscopy of aeolian sandstone using reflected light. The same region is shown using standard bright field optical microscopy (a) and polarized light microscopy with the polarizers rotated to different settings (b and c).

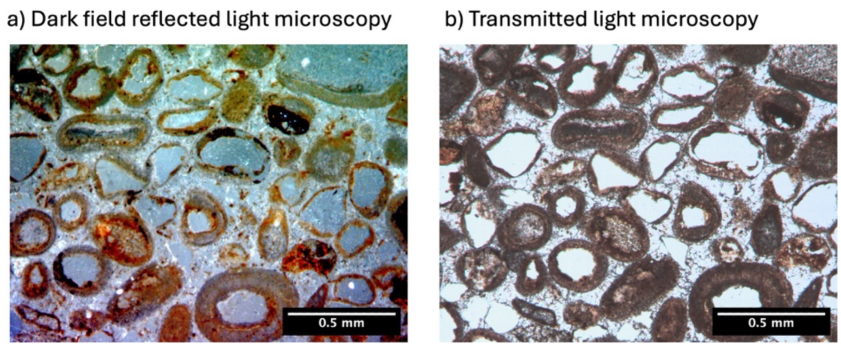

Figure 6. Oolitic limestone as seen under (a) dark field reflected light microscopy and (b) transmitted light microscopy.

Discussion

This new lab improved the alignment of course activities and learning objectives in the course's first module. Previously, students calculated density and conducted error propagation calculations using measurements of the masses and volumes of materials. However, this activity was perceived as juvenile by the students in the class, as it repeated activities from prior courses. Additionally, it reinforced the perception that standard deviations must be due to sources of measurement error rather than variability between samples. This new lab aimed to reduce that viewpoint by emphasizing the ranges of sizes in a sample.

Ironstone's broad particle size distribution complicates its analysis. The number-average grain size is straightforward to calculate but is skewed by the large number of small particles. A person would visually estimate the grain size to be hundreds of microns, but a computer algorithm can measure the many micron-sized particles. Students found it challenging to perform area-weighted calculations in Microsoft Excel or similar programs, but this was necessary to obtain meaningful results. Most anecdotal feedback about the lab was related to the level of support for this task, so the lab materials have been modified to provide additional support.

Conclusion

This lab aims to teach students about the analysis of microstructures through natural materials, expanding the instruction of what Prof. James Shackelford terms the "menu of materials." The lab module engages students in MSE topics early in the quarter, but does not rely on covering specific course topics. The unit's learning goals focused on highlighting the underlying variability that occurs in materials, such as a distribution of particle sizes, and that reproducibility of measurements can be attributed to sources other than "measurement error" or "user error." Data on students' performance in the lab or their perceptions of geology-based labs were not collected.

Finally, using geological materials for an introductory class requires selecting materials and imaging modes that match the students' skill levels, the laboratory's technological capabilities, and the desired learning goals. Microscopists can optimize the imaging by adjusting the optics, such as the field and aperture diaphragms. However, this is rarely performed by novices or graduate students who teach the labs. Unpolarized, reflected light is commonly used for metallographic microstructures, which are also covered in the course, so this technique was selected for the lab. Ironstone was chosen for the lab assignment due to its ease of imaging and the parallels in particle size analysis that are relevant to ironstone and engineering materials. Overall, the samples have held up well after use in 8 laboratory sections; one sample was broken due to improper microscope use, but the other samples remain in good condition. This is equal to or better than the durability of metallographic samples that are frequently scratched and require re-polishing.

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[5] Kemet. “Rock Thin Sections (Petrographic Thin Section Preparation).” https://www.kemet.co.uk/blog/petrography/rock-thin-sections (retrieved January 8, 2025).

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[9] Microtrac Particle Characterization “Particle Size Distribution.” https://www.microtrac.com/knowledge/particle-size-distribution/ (retrieved September 15, 2024).

Appendix A: Lab Prompts for Submission

The following information was provided to the students to guide their final submission.

Students should individually complete the data analysis and answer the questions below. You should answer each question as given below. These should be typed using word processing software, although a full lab report is not needed. *References should be indicated where appropriate, including literature values. Answers should be given in full sentences in paragraph format unless otherwise specified (i.e., a table).*

1. Figure manipulation must be reported when publishing scientific figures. In paragraph form, describe how the image was adjusted to conduct the particle size analysis, referring to specific functions or steps. Provide images of one region, before and after the manipulation. These images should include a scale bar.
2. Single Particle: Provide the image of the single particle that was used for the analysis; this image should include a scale bar and one line showing the maximum Feret diameter. Provide the five Feret diameters determined from your analysis including the average value and standard deviation. INCLUDE UNITS! *Appropriate significant figures should be used when reporting all laboratory results, even if the question does not specify it.*
3. Discuss the error in the measurement, addressing the magnitude of the standard deviation relative to the average value. Your discussion should include possible sources of uncertainty or variability in the measurement. *The discussion of error should be 2-4 sentences.*
4. Particle Size Analysis:
    a. Provide the image that was used for the particle size analysis. Again, this image should include a scale bar.
    b. Calculate the average particle size as weighted by the area.
    c. Plot the cumulative size distribution, with cumulative area fraction on the y-axis and Feret's diameter on the x-axis. Use a logarithmic scale for the x-axis.
    d. Provide the d_{10}, d_{50}, and d_{90} values.
5. Compare the average particle size to the d_{50} value. Discuss the sources of error and variability for either/both measurements (*Your answer should be about 2 paragraphs)*.
6. If you used any references, provide the references that were used to answer the above questions. These should be formatted as a "Reference" section at the end of the postlab writeup that uses a standard numbered citation format such as IEEE [1, 2]. (Note: I will provide an exception that references do not need to be placed in order of appearance.). You may find it helpful to use a reference management software, such as Zotero [3], taking care to verify that citations are correct when inserted into the document.

References

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