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Micromechanical Characterization of Earth Materials

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ABSTRACT

In this experiment, two different quartz samples were carefully prepared and tested using nanoindentation to better understand their material properties. Though this type of testing is common in materials science, these techniques are rather innovative in the field of Earth science and have never been applied to minerals and rocks. This unique approach allows for a different perspective in the micromechanical characterization of these Earth materials and provides the opportunity to learn new information about the material properties or confirm previous estimations. All samples were prepared through a series of sectioning, grinding, and polishing. Samples were then indented at a load of 8 mN in either a 7x7 or 5x5 array at various temperatures ranging from 23°C to 550°C. The data produced was then used to calculate and plot the hardness and reduced modulus values. The trends in these plots were observed and analyzed.

INTRODUCTION

The purpose of this research is to perform micromechanical testing of abundant minerals found in the crust and upper mantle of the earth to better understand the material properties of these minerals, with the hope that this knowledge will lead to an increased comprehension of mountain formation and plate tectonics. Large-scale deformation studies of rocks are commonly conducted at high temperatures and under large confining pressures, simulating the conditions found in the mantle, to prevent cracking and promote plastic deformation. However, such testing provides limited insight into the local deformation processes occurring at the atomic level, and repeated testing to achieve statistically significant results can be challenging. In this research, we apply nanoindentation methods to characterize the local plastic deformation behavior of rocks at low temperatures. By pressing a small pyramidal indenter into the rock surface at positions of interest, we can probe variations in properties over micron length scales, while the undeformed material surrounding the indent provides a natural constraint against crack formation and growth. This approach allows us to gather information about the hardness and yield strength of these minerals in a broader range of temperatures than what has previously been accomplished.

It is first important to properly prep the mineral samples for nanoindentation through a series of sectioning, grinding, and polishing processes. Sectioning a specimen is the process of removing a small piece of material from the original parent piece. This piece should be representative of the parent piece, meaning that its overall composition and treatment should be the same. During this process, one must be careful to not alter the microstructure in any way, otherwise the sample piece will yield different conclusions than the actual parent piece. It is important to use a cutting technique that is gentle to the microstructure of the specimen and minimizes or eliminates heat and deformation. Before the grinding process, the sample is usually mounted in some way to make it easier to handle. Grinding and polishing prepare the sample by creating a smooth, mirror like finish, exposing the microstructure. This is accomplished by using successively finer abrasive papers, pastes, and powders. Before proceeding from one operation to the next, the specimen is carefully rinsed, by use of a sonicator, to avoid cross contamination. All grinding is done on a wet or well lubricated grinder to avoid or minimize the creation and effect of heat on the specimen. The first stage of grinding results in a flat, parallel surface, with each successive stage further exposing the microstructure.

APPARATUS AND PROCEDURES

In this lab, two quartz samples were prepared and tested. One sample had a y-axis oriented crystal structure, with the other having a z-axis oriented crystal structure. The samples were sectioned from their respective parent material using a diamond blade precision saw. In order to do this, the material was bonded to a piece of metal using crystal bond. The metal piece was then screwed into the lever arm and then gently laid perpendicular to the blade. After ensuring that water was running and constantly wetting the blade, the saw was turned on and the sample was held on the blade by gravity to make the singular cute. This process was repeated for each cut on the sample until a 1x1 cm piece was produced.

To prepare the sample for grinding, instant adhesive was applied to one side of the piece, which was then placed onto the center of a small metal puck that locks into the Allied MultiStep™ Polishing system parallel polisher. The sample set to dry for at least 5 minutes. While waiting for the sample, the polisher disk was prepared. 400 grit silicon carbide paper was adhered to the disk, pressing while applying to avoid bubbles between the paper and disk. Once the polishing plate was placed into the polisher, the grit paper was wetted. The sample was then locked into place and lowered onto the plate until contact was made. Water was set to drip onto the polisher at a rate of about 1 drop per second. The sample was then polished for 30 minutes at 75 rpm. This process was repeated for 600, 800, and 1200 grit paper.

Afterwards, a slightly different process was used for polishing with diamond paste. Instead of grit paper, a polishing pad that had been lubricated was adhered to the polishing disk. A rice size drop of 3µ diamond paste was applied to the surface of the quartz sample, and in figure eight motions it was mixed with the lubricant. Once thoroughly mixed, the plate and sample were locked into place in the polisher and set to polish for 2 intervals of 20 minutes at 25 rpm. During polishing, lubricant had to be reapplied to the plate every 2-3 minutes. This was repeated for 1µ, 0.5µ, and 0.1µ diamond paste.

For the final polishing step, a cloth plate was put into the polisher and colloidal silica mixed with water was set to drip onto the plate. The polisher was set at 45 rpm for 1 hour. Once completed, the sample was thoroughly cleansed with an organic soap solution and removed from the metal puck by being soaked in acetone. In between each step of polishing, the sample was sonicated for at least 5 minutes.

During the polishing process, the samples were checked for scratches on their surfaces to ensure as many as possible were removed. After polishing with the diamond paste, seeing these scratches became more difficult. However, they were still checked to ensure no overwhelmingly large scratches remained. Polishing quartz does not produce a surface completely free of impurities. As shown in Fig. 1, even after the entire polishing process, spots of impurities remain. This is due to the composition of the quartz mineral and is unavoidable. However, the quartz was smoothed enough to allow for testable locations for nanoindentation.

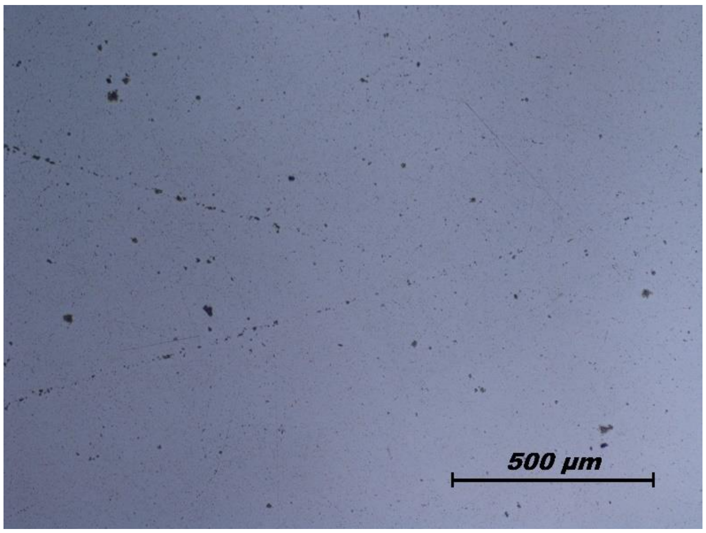

Fig. 1 Image of quartz sample after completing polishing process.

The prepped samples were tested in the Hysitron nanoindentor at a load of 8 mN. 49 indentations in a 7x7 array were performed for both samples at 23˚C. All testing done afterwards were performed in 5x5 arrays. Figure 2 shows the shape and approximated depth of a sample indentation. Both samples were also tested at 50˚C, 100˚C, 150˚C, and 200˚C. Additional tests were performed on the z-axis-oriented quartz at temperatures of 250˚C, 300˚C, 350˚C, 400˚C, 450 ˚C, 500˚C, and 550˚C.

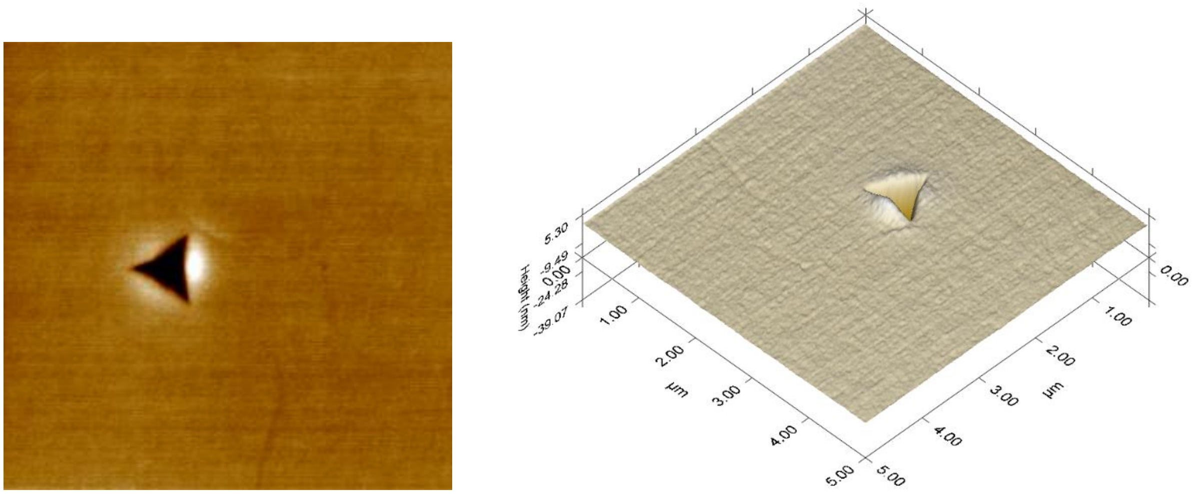

Fig. 2 Pyramidal shape of nanoindentation on quartz surface and depth of the indention.

RESULTS AND ANALYSIS

Each indentation performed for the quartz samples produced a text file of information. The data obtained was run in a Matlab script created to calculate the resulting hardness and reduced modulus of each sample. Figures 3 and 4 graphically display the hardness found at each temperature for the y-axis-oriented and z-axis-oriented quartz respectively. Figures 5 and 6 show the yield strength calculated for each indentation for each sample at the different testing temperatures.

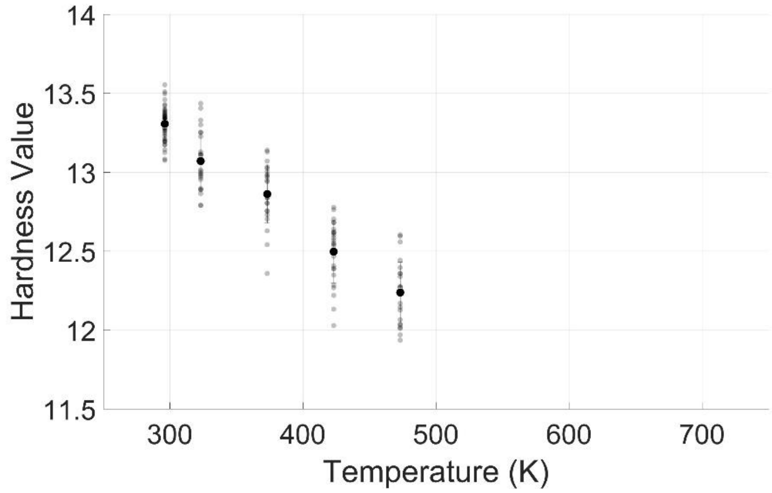

Fig. 3 Hardness values for y-axis-oriented quartz at various temperatures.

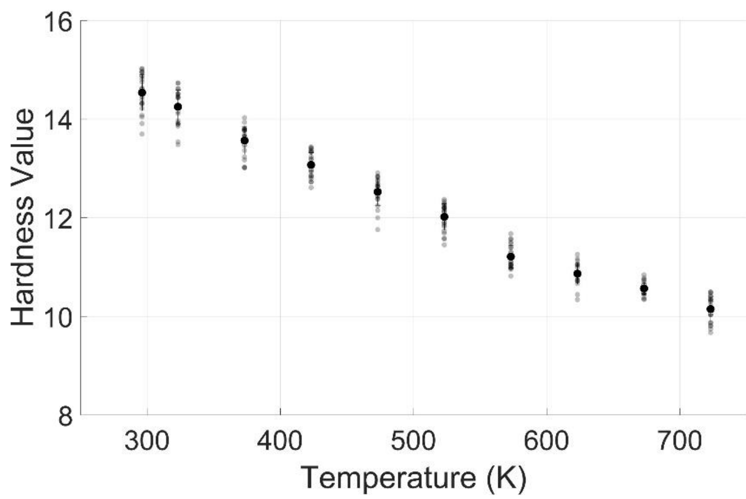

Fig. 4 Hardness values for z-axis-oriented quartz at various temperatures.

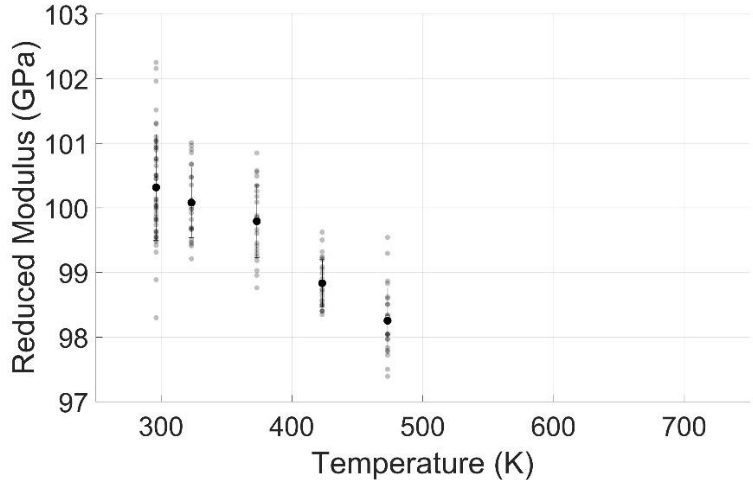

Fig. 5 Reduced modulus values for y-axis-oriented quartz at different temperatures.

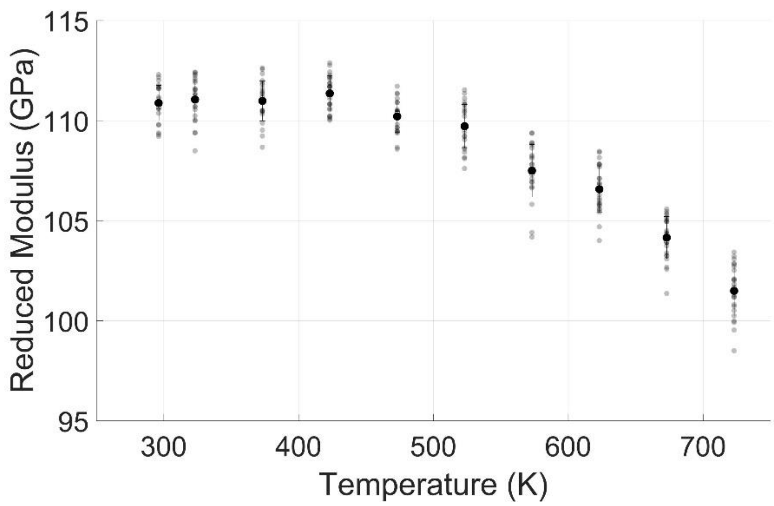

Fig. 6 Reduced modulus values for z-axis-oriented quartz at different temperatures.

It was expected to see values for hardness and reduced modulus decrease with an increase in temperature, which was shown in the trends for all the data obtained through experimentation. However, the decrease in reduce modulus for the z-axis-oriented quartz decreased sharply. It was thought that this decrease would occur gradually.

FUTURE WORK

The data obtained from this experiment is a start to understand the relationship between different mechanical properties of quartz at a range of low temperatures. With continued experimentation, this data will be compared with theorized predictions previously made in the field of Earth science. Repeated experiments will also help to confirm data that was found and allow for more conclusive assumptions to be made. Going forward, the y-axis-oriented quartz will also be tested at temperatures of 250°C, 300°C, 350°C, 400°C, 450°C, 500°C, and 550°C. The test performed on the z-axis-oriented quartz will be repeated to further understand that trend shown in the decrease of its reduced modulus. Additional quartz samples will be tested, along with other materials such as granite.