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APPLICATION OF RESONANT ULTRASOUND SPECTROSCOPY TO DETERMINE THE ELASTIC PROPERTIES OF MACROSCOPIC ROCK SAMPLES

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ABSTRACT

Resonant Ultrasound Spectroscopy (RUS) is a method whereby the elastic tensor of a sample is extracted from measured resonance frequencies. Typically, a rectangular parallelepiped sample is placed between two piezoelectric transducers, a source and a detector. The sample is driven at constant voltage as the frequency is swept through multiple resonances. The measured resonance frequencies are the input to an iterative inversion algorithm that finds the best match between the data and a set of resonances generated from a model. RUS has been used successfully to determine the elastic properties of single crystals of minerals found in the earth's mantle (e.g., [Oda et al., 1992]). We are extending the applicability of RUS to macroscopic samples of rock. Rocks are potentially difficult samples on which to apply RUS, because of their high acoustic attenuation (low Q), inhomogeneity, anisotropy, and the difficulty preparing suitable samples. In preliminary measurements, we have analyzed a variety of rock types to determine optimal sample sizes and aspect ratios, the minimum number of resonances necessary for obtaining an accurate inversion, the sensitivity of RUS to anisotropy in rock samples, and the precision of measurements on samples of varying sizes and aspect ratios.

Assuming isotropy, we have found that RUS provides reliable results for relatively high Q materials such as basalt, primarily because a large number of resonance frequencies can be accurately determined. For example, application of RUS to a sample of basalt with an aspect ratio of 4 yields an RMS error of 0.31% in the fit between the predicted and measured resonance frequencies, and the elastic constants c_{11} and c_{44} change by 0.54% and 0.12% respectively with a chi-squared increase of 2%. Application of RUS to low Q materials such as sedimentary rock is considerably more difficult, and it remains unclear if RUS is viable for such materials. (Oda, H., O. L. Anderson, D. G. Isaak, and I. Suzuki, Measurement of elastic properties of single-crystal CaO up to 1200K, *Phys. Chem. Miner.*, 19, 96, 1992.)

TRANSCRIPT

DR. MCCALL: I brought my samples with me so that everybody could see them. Please pass them around.

[Transparency 1]

We chose the alliterative title: "RUS and ROCKS."

The work has had a lot of contributors, but most of it was done by TJ Ulrich, who is the graduate student who, of course, does everything I tell him to do in hopes that he will graduate.

[Transparency 2]

I am going to start out by telling you a little bit about the history of my involvement in this project. I was trained as a condensed matter theorist; now I am sort of doing experiments. Sometimes I wonder how I got here.

Then I will tell you about the experimental system we are using, some of the numerical modeling that we use to explore important issues that come up for the samples that I am passing around, a few of our experimental results, and where we want to go.

The history of the project was given to you in the last talk.

[Transparency 3]

I went to Los Alamos in 1992. One of the groups in the division I joined as a postdoc had an enormous bar of Berea sandstone, 6 cm in diameter and 2 m long. It was just beautiful; I loved it. Of all the places that I applied to or interviewed at, Los Alamos was my top choice as a place to work, because people at Los Alamos were doing experiments on this rock.

At the time they were just starting to look at the nonlinear elastic properties of rocks. As you heard in the last talk, we find that if we measure stress versus strain, as shown in the figure, we see nonlinearity, hysteresis, and discrete memory. The inset shows the load versus time. As Robert showed you, this curve was not taken by just going straight up in stress, it was taken by coming up part way, stopping, turning around, going back, going up a little farther, going around, and so forth. If we do a resonance measurement --- the figure shows just a single peak of the frequency versus acceleration --- we see this nice bending here, indicating a nonlinear response.

We started thinking about theoretical models, and I assume that Robert will tell you more about that in the next talk. The idea is that the modulus is nonlinear. The modulus relating stress to strain in this equation here does strange things as a function of strain. We can do an analytic

calculation by assuming we have a base-line modulus, with a perturbation that is proportional to strain, and perhaps a second perturbation that is proportional to strain squared, etc. To account for hysteresis we might add something that looks like this, where α is a measure of the strength of the hysteresis, and the multiplicative factor is dependent on the absolute magnitude of the strain, and the sign of the strain.

With these kinds of ideas in mind, we go back to the measurements and try to make predictions. What is β ? What is δ ? What is α ? Can we extract those numbers?

[Transparency 4]

That is where RUS came in. The hypothesis is that while we can start by trying to measure β , δ , and α , the results will be misleading, unless we have a really good handle on the base-line K_0 . What is the linear modulus of the material that we are studying? If you pick up some of the samples of rock coming around, you should be able to see that a number for one piece of basalt is not necessarily the number that is appropriate for a piece of basalt from someplace else. So, when we measure the nonlinear properties of rock samples, we also need to measure something that other people will understand characterizes the sample, the linear modulus.

The rock mechanics way of measuring moduli is to measure stress versus strain by pushing very hard on a piece of rock until it breaks, or reaches failure. If we do not look too closely at the stress-strain curve, we can easily draw a straight line fitting the low strain data (the rule of thumb is that you go to somewhere between 70 and 90% of failure), and claim that that the slope is the modulus. There is a problem with this, as far as I am concerned. First of all, the measurement is made at very high amplitude, and we are talking about something that is nonlinear at low amplitudes. I would rather have a low amplitude measurement. The second problem is that we can never look at the same sample again. It is gone; it is dead.

The moduli can also be derived from time-of-flight measurements. There are people making very nice, beautiful time-of-flight measurements. They take a lot of time, though, and the problem with inhomogeneous samples is that the result could very well depend on which path the acoustic wave traverses, that is, where we place the transducers.

I want something that is more of a bulk measurement, an average measurement, of modulus. So we are trying RUS.

[Transparency 5]

We have an experimental system that is on loan to us. It includes both the control hardware and the analysis software. We spend a lot of time playing with the analysis software, and I am very happy to say that no, we did not write the analysis software, we just play with it. The transducers are similar, perhaps identical, to the ones you saw this morning at the demo. We have two arms holding the transducers, and the transducers hold the rock sample. Nothing fancy.

There are several issues that we want to consider when we are looking at rocks. Rocks are kind of messy looking; they are not single crystals. First, because of the assumptions made in the analysis software, we need free boundaries. That is not too hard to get. Second, in the analysis we assume that the sample is homogeneous. Our samples are visually inhomogeneous, quite inhomogeneous. Third, we assume that the samples are perfect parallelepipeds. If you look at one of the samples going around, the dark, fat basalt, you can see that the sides are not parallel.

Then we want other conditions to be met in order to get really good results. First, we want the resonance peaks to be distinct. This is an issue because the Q is so low in our samples. We have very broad peaks and they may overlap each other. Also because the Q is so low, sometimes it is very hard to pick the peaks out of the background; we have to look very, very carefully. Second, we want to be able to use the fewest modes to get the most information. Again this is necessary in large part because of the low Q of our samples. If we want to find two moduli, for instance, we need to have a lot of C_{11} dependence in the first few modes. Otherwise we can't resolve both C_{44} and C_{11} . This is where we got to play with the software that we had around. It has been provided at various times by Tim Darling, DRS and Albert.

[Transparency 6]

Okay, so I said free boundaries. That was easy. Then we must think about the homogeneous sample issue. Well, we are looking for constants that people might use in seismic research. They are looking for a general number; they do not really care about the elastic properties of a quartz grain, but they do want to know what the elastic properties or the velocity of sound is where a seismic wave might propagate.

Making a simple little estimate, we start by saying that any experimentalist knows that the probe we use has to have a wavelength about the size of the thing we are trying measure. Conversely, the wavelength has to be much larger than the size of an inhomogeneity in order to average over it, or not see it. Our estimated one-dimensional resonances have wavelengths of

size $2L/n$. If ξ is the size of the inhomogeneity, then the length of the sample has to be greater than 5 times the inhomogeneity. I kept this little tiny sample up here because I am going to show you the results from it. The reason that the results are really bad is that the inhomogeneity is getting really big as compared to the sizes of the sides. $L > 5 \xi$ is one of the limits we are going to use as a general estimate.

[Transparency 7]

The importance of an imperfect parallelepiped is determined by just how anti-parallel the sample is. Fortunately, our samples are really big, so our $\Delta L/L$ could be the same size as a $\Delta L/L$ for a very small sample, even though we can see our sample is not parallel by visual inspection.

This is Robert Guyer's work. He did a first-order perturbation theory analysis of what to expect if we have a nick in our sample (for example, one of the samples going around has a nick in it). He asked, how does the frequency change when you put in an imperfection? He found these results. Suppose we take a nick out of the corner that is 1% of the mass. We find a frequency change in the first 20 resonances of about 0.15%. We guessed that this level of error would not matter too much and, empirically, it does not. The sample that is going around that has the nick in it was measured before it had the nick and measured again after it had the nick. The nick did not make much difference in the results that we got, which was really nice.

Robert obtained similar results when he considered the kinds of distortions we have in our samples. For example, we have parallelepipeds with non-parallel sides. We call those distortions symmetric distortions, in that we can draw an average size and make the sample parallel. Even when the symmetric distortion is about 5% of the mass, to first order in perturbation theory we find a 0% frequency change. This is pretty easy to explain, because first-order perturbation theory averages over symmetries. For asymmetric distortions we start to find some pretty big errors, although they are all about 1.5% or less. I am going to claim that the problems we have with our samples are mostly one and two, that is, the nick at the corner and the symmetric distortions.

[Transparency 8]

We want our resonance peaks to be distinct. This is a Q issue. We have a very low Q, in the hundreds mostly, 150 to 250 for most of the samples that we are looking at, so we need to use sample aspect ratio to separate our resonance peaks.

What we did here was run a model over and over again. We calculated the resonance frequencies for various aspect ratios. In the figure, a and b are held at a fixed ratio of 1.2, while the ratio of c to a is increased. The ratio of c to a starts out at 1.4 and is increased up to 8. We are using the standard book-value elastic coefficients for basalt. That is, we looked in a book of rock elastic constants under basalt and used the tabulated C_{11} and C_{44} in the program. Then we calculated the first lots of resonance frequencies.

In the figure, the first 3 frequencies are nicely spread. Then we start seeing triplets and doublets, indicating to us that it is going to be really hard to pick out enough resonance frequencies in a sample that is made almost cubically. Because we already have a peak overlap problem due to our low Q, we look to higher aspect ratios. In the calculations, the volume of the sample was kept fixed as the aspect ratio was changed. The fixed volume keeps the resonant frequencies in the same range over the figure. We decided that an aspect ratio of 4 -- actually, 6 is a really nice place, too -- would allow us to pick 10 or more frequencies that are nicely separated. It turns out that cutting our samples at an aspect ratio of 4 was reasonable, so that is what we did. We are going to claim our best results come from samples of aspect ratio 4.

[Transparency 9]

We ran the simulation again to look at the C_{11} dependence of the first 8 modes. Here we have exactly the same situation, b/a is 1.2, while the ratio of c to a is increasing. We are looking at the dependence on C_{11} of each of the first 8 modes. Because we have 8 symmetries in the resonances of the sample, we assume that the next 8 modes have essentially the same dependence on C_{11} .

The first mode dependence on C_{11} is 0 (it is hard to see in the figure) and another mode rapidly decreases to 0. However, you can see that as we increase the aspect ratio, we get a lot more of overall dependence on the C_{11} constant versus the C_{44} constant. At an aspect ratio of 4, where we are going to claim our best samples are, we have 25 to 35% dependence on C_{11} .

[Transparency 10]

Okay, so what do the results look like? Here is a nice picture of a spectrum. This is basalt with an aspect ratio of 4. This measurement was made on one of the samples that is going around. The analyzed data has a percent in the fit of 0.31. The fit and the experimental frequencies start at the far left of the figure. This is our first peak, that is the second one, and so forth. We have a huge gain problem here, and must be very careful as we pick out the peaks.

The columns are the experimentally picked frequencies and the fit frequencies obtained from the analysis routine. We are very happy with these results.

[Transparency 11]

Here are comparisons of different aspect ratio samples. We will look at the top table first, the basalt. The green numbers are book values for the elastic moduli, the values we found in the handbook of rock constants. C_{11} is about 87 GPa and C_{44} is about 32 GPa. The results are ordered by aspect ratio, from 1.5 to 4. TJ found ξ , the measure of inhomogeneity, by looking at each sample and measuring the size of the biggest surface blob. The biggest blob was 0.61 cm on the smallest aspect ratio sample, and the fit C_{11} and C_{44} are given here. None of the fit moduli for these samples is far from the book values. However, the percent error changed by a factor of 3. That is quite a bit, actually. As advertised, we found the smallest error for an aspect ratio of 4. The average Q seems to increase with aspect ratio, as well. We feel these are reasonable results.

The second table shows the Sierra white granite results. Here we are in a little more trouble. We start out with book values for the elastic constants of 43 and 19 GPa. The aspect ratios of the granite samples are approximately the same as for the basalt samples. However, the aspect ratio 3.3 sample is this tiny sample I am holding. The fit to the resonances for this sample has a huge error. We claim that the large error is due to inhomogeneity. The biggest blob on the surface of the sample was half the size of one of the sides of the sample. The inhomogeneity is starting to make an average measurement impossible on this sample. Note that the average measured Q's are all similar for these samples, but the C_{11} 's and C_{44} 's are quite spread. I would say that the best measurement here results in moduli quite a bit different from what the book values. One thing that TJ is quite concerned about is that the Poisson ratio is pretty extreme for these samples.

[Transparency 12]

Remember that we started out wanting to know if we can make baseline linear measurements on samples whose nonlinear properties are of interest. Can we stick a core of sandstone into a RUS apparatus and measure the linear properties of the core before making the nonlinear measurements? Actually, sandstone turns out to be a problem sample type. The Q is so low on sandstones that it is very hard to measure enough resonance frequencies to successfully use RUS.

However, most geophysical samples are cores, or cylinders. Thus, that is the first place we want to go with this work. Now that TJ has done the groundwork with parallelepiped samples, a talented undergraduate, Izabela Santos, is going to work on core samples. First she will play with the calculation, forward modeling to study the aspect ratio dependence and the C_{11} dependence. We wish to find out whether we can actually make those measurements on long cores of sandstone.

TJ wants to look at saturation and temperature control. He will attempt to build an apparatus to allow temperature and saturation control of his samples. Unfortunately, building these kinds of cells is something that I do not know very much about.

Thank you very much.

DR. SACHSE: The stress-strain curve that you showed for your rock was very different from the stress-strain curve shown by the previous author. Yours was what I would call a typical stress-strain curve for a metal, with softening, because you had stress and strain on different axes than he did. I'm referring to your schematic drawing where the curve went up and bent over like this. That is the conventional way it is drawn, but his curves always had load on the horizontal axes.

DR. MCCALL: Right.

[return to Transparency 3]

Like this? Physicists like to put load on the horizontal axis, because that is usually what the experimentalist is controlling.

DR. SACHSE: It depends on which kind of machine you are using.

DR. MCCALL: That is also true. For the measurements that we like to use, stress is the control.

DR. SACHSE: The reason I mention this is that the curve on the right shows acceleration versus frequency. I would think that in one case you might get bending one way and in another case you might get bending in the other direction. You can have softening and hardening effects.

DR. MCCALL: Right. You certainly get hardening if you are just pushing down on a sample. You can think of yourself as pushing the grains closer and closer together, making the material stiffer. When you resonate a sample, it gets softer. You have to remember you have free boundaries, so the sample can expand if it wants to.

DR. ANDERSON: I noticed you list Q values. Presumably they are Q's of the individual modes. How do you measure the Q? What is the definition of Q? Is it the half-width?

DR. MCCALL: Those measurements are the result of the program given to us by Tim Darling, so you can ask him how they were made, but I believe it is half-width at half-maximum, isn't it?

DR. DARLING: I do not think I gave TJ any programs that calculate Q. I did not give him the Lorentzian fitting program.

DR. MIGLIORI: One component of the phase, one component of the response. If they adjust the phase so that it looks like it is in phase with the resonance, there is a symmetric peak. Then the Q is the full-width at half-maximum of the real part.

DR. DARLING: It is coming out of the DRS program.

DR. MCCALL: Yes, and TJ did do the calculation by hand as well. He measured the peak heights and widths and divided.

DR. LEISURE: You mentioned that the scale of the inhomogeneities should be less than the wavelength. That seems like a weak condition. I would have guessed you need a factor of 10 or so.

DR. MCCALL: Yes, it is a very weak condition.

DR. LEISURE: Is that sufficient, just slightly under the wavelength?

DR. MCCALL: It seems to be. Our results are totally empirical. I said the wavelength should be 5 times the size of the inhomogeneity, at least. 10 times would be a nice rule of thumb, because scientists like factors of 10, right? However, we have samples that are very definitely visually inhomogeneous, for which we find very low fitting.

PARTICIPANT: [Question inaudible]

DR. MCCALL: That is about 10. That will do it.

DR. SACHSE: On these plots of acceleration versus frequency, the frequency is the driving frequency, is it not?

DR. MCCALL: Yes.

DR. SACHSE: Have you ever made measurements where you plot the response frequency?

DR. MCCALL: No, what we are doing is measuring the response at the driving frequency with a lock-in amplifier. We drive the sample at one frequency and lock in at that frequency at the detector, so we measure the amplitude at the drive frequency.

DR. SACHSE: I know that if you are studying the nonlinear vibrations of plates and you measure the response of the plate at the horizontal axis, you will find, not a curve that goes like this, but a jump that goes back and forth. The frequency oscillates in a very narrow band, and it is not exactly the center frequency. You get these results only if you are at the top, stop the electronics, and then watch the sample go down the free vibration curve, which is the backbone of this curve.

[Simultaneous discussion]

DR. SMITH: The question is, is the receiver selective?

DR. MCCALL: Yes, the receiver is fixed.

DR. MIGLIORI: -- was under 100 Hz.

DR. MCCALL: I am not sure I understand your question, but can I talk to you afterward?

DR. MIGLIORI: I do not, either. (Laughter)

DR. SACHSE: If you are driving a system at large amplitude, with large excitations, you find that when you go through one of the peaks, you go up the curve like this and then you get a sudden discontinuous jump. The jump is seen only if you are applying the excitation frequency to the horizontal axis. If you take the output sensor, let's say the accelerometer, and you plot its frequency output, you will find that the response frequency jumps back and forth between the boundaries of the width of the excitation frequency peak.

DR. MIGLIORI: Wait a minute. If you drive at a given frequency, and the oscillator is anharmonic and you are doing a steady-state measurement, then the response of the object is highly nonlinear, and you can get only a certain collection of --

DR. SACHSE: You are sweeping the frequency of the excitation. You are sweeping through resonance, and you find out that the curve goes something like this and then goes down.

DR. MIGLIORI: That happens only if you are sweeping so fast --

DR. GUYER: It is almost happening at the top of the figure. It is almost happening, not quite.

DR. SACHSE: I think you will see it.

DR. GUYER: Well, if it were not for the slow dynamics, we would have seen it.

DR. MAYNARD: Has anyone done any measurements of these nonlinear effects as a function of humidity?

DR. MCCALL: Not deliberately, but we have done them accidentally. Humidity makes a huge difference in where the resonance is. Fortunately, most of the experiments are done in Los Alamos and it is pretty dry most of the time, but during monsoon season, July and August, you can watch the resonances shift as the storms move through, and then they come back.

PARTICIPANT: Does the pattern change, does the evolutionary pattern change?

DR. MCCALL: No, I do not think so.

DR. HARGROVE: The Q usually goes down because you get into an evaporation-condensation condition, adding another --

DR. MCCALL: You would have to put a lot of energy into it in order to get that during the experiment, but sure, yes. Anything else?