ELASTIC PROPERTIES OF SINGLE-CRYSTAL NaCl UNDER HIGH PRESSURES TO 80 kbar

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Adiabatic elastic moduli for an NaCl single-crystal under high pressures up to 80 kbar at room temperature were determined through ultrasonic sound velocity measurements in a cubic anvil-type high pressure apparatus with a solid pressure transmitter. Ultrasonic measurements for both the longitudinal and transverse waves were carried out simultaneously by a pulse-echo-overlap technique. Pressures were determined by X-ray measurements of NaCl based upon Decker's equation of state. The adiabatic bulk modulus K_s calculated from the Decker's formulae as well as various pressures was combined with the ultrasonic sound velocity to provide pressure dependence of elastic moduli C_{ij} for NaCl.

Results of measurements yield zero-pressure values of $C_{11}=5.16 \times 10^{11}$, $C_{12}=1.22 \times 10^{11}$ and $C_{44}=1.36 \times 10^{11}$ dynes/cm². The plots of C_{ij} versus pressure are fit by a least square method with straight lines for pressure range zero to 80 kbar, to give the following slopes: $\partial C_{11}/\partial p=8.72$, $\partial C_{12}/\partial p=1.22$ and $\partial C_{44}/\partial p=0.31$. In the overlapping pressure range, $\partial C_{ij}/\partial p$ values obtained by this work are slightly smaller than those obtained by others.

1. Introduction

Elastic properties of single-crystals under high pressures and high temperatures have been studied in various ranges of pressure and temperature (BARTELS and SCHUELE, 1965; SOGA, 1967; SPETZLER, 1970; SPETZLER *et al.*, 1972; WHITFIELD *et al.*, 1976 etc.). Most experiments available to date have been carried out in a hydrostatic pressure condition in order to avoid the plastic deformation of a specimen. Pressure and temperature ranges of these measurements are limited either to below 35 kbar or 500°C as far as experiments on a single-crystal of NaCl is concerned. We have concentrated our effort on developing a technique of measuring an ultrasonic wave velocity for various minerals of geophysical importance under high pressures and high temperatures.

The frame of our study is threefold. First, we have built up electronics

circuits and the related apparatus in order to provide sharp reflection signals from a small specimen through a non-metallic acoustic buffer (FUJISAWA *et al.*, 1974). Second, we established a technique of ultrasonic measurements with a ceramic transducer attached on an outer surface of a press anvil exposed to normal atmospheric pressure and room temperature. A sound signal sent from outside reaches a specimen and then is reflected back to an electronic receiver to afford an ultrasonic sound velocity of a specimen within a high pressure cell. Third, we achieved a pressure and temperature range of up to 80 kbar and 1,000°C under which we could observe ultrasonic sound signal of both the longitudinal (L) and transverse (T) waves simultaneously. The X-ray measurements on NaCl powder was employed to determine the absolute values of pressure exerted onto a specimen.

Our measurements were carried out only in a solid pressure transmitting medium (baked pyrophyllite) so as to make a good contact between ultrasonic sound sources and the specimen. Care was taken to eliminate plastic deformation of the specimen. High temperature experiments do not yet provide reliable data due to poor reproducibility of the measurements.

2. Sample Arrangement

The electronic circuit prepared for the present work is essentially the same as those described elsewhere (FUJISAWA *et al.*, 1974). Some modifications were made to the original circuit so that it meets the requirements of high pressure vessel geometry. A tone burst wave signal of a 10 MHz (or 7 MHz) carrier frequency is generated every few tens of microseconds and is fed to a ceramic PZT transducer of L (or T) mode. Ten MHz and 7 MHz signals are generated in two independent circuits and are fed to transducers glued on an outer surface of two opposite anvils (Fig. 1).

High pressure apparatus of a cubic symmetry is found to be the best for our purpose. Other types of multistage anvil systems were also tested and were rejected for various reasons. In a cubic anvil system, six anvils with flat square shaped surfaces move synchronously toward the center of the high pressure space to squeeze a specimen placed at the center of a solid pressure transmitting medium. Both the front and tail ends of an anvil are cut parallel to each other with an accuracy of ± 0.01 degree and then polished until optically flat. The ceramic transducers of both the L and T modes are also polished as close as possible to the optical quality flatness and then cut to 5 mm by 5 mm square plates. They are tightly glued to the polished surface of the tail end of the anvil by epoxy resin.

A pyrophyllite cube of an 8 mm edge length, which was baked at 700°C for 20 minutes in air prior to experiments, was mounted between the six anvils.

Each square flat surface of the pyrophyllite cube was aligned exactly parallel to the front surface of the corresponding anvil. This alignment was automatically attained when all the edges of the pyrophyllite cube were trimmed and a specially disgned preformed gasket made of the same pyrophyllite was glued close to the rim of the anvil. The gasket was so designed as to prevent any appreciable deformation of the pyrophyllite pressure transmitter at the initial stage of loading.

However, some plastic deformation of the specimen was observed after unloading. After we pressurized a disk shaped NaCl single-crystal (2.5 mm in diameter and 1.5 mm in thickness) up to 80 kbar in this manner, we found the maximum distortion of the disk diameter to be about 3%; $\{(\text{maximum diameter} - \text{minimum diameter}) / (\text{mean diameter of the disk})\} \times 100\%$. It was found through X-ray study that even though there is a small plastic deformation of NaCl crystal, an original single-crystal structure of both the central part and the peripheral part of the specimen remained undisturbed as a whole.

A disk shaped NaCl single-crystal is cut out and placed at the center of the high pressure space for the ultrasonic measurements. The disk surface

Fig. 1. A schematic lateral view of a high pressure cell used for ultrasonic sound velocity measurements. The anvil height is shortened for convenience in drawing. At the bottom of the figure, a trace showing where the sound echoes are generated is drawn schematically. Abbreviations are as follows: TR, a ceramic transducer for longitudinal (L) and transverse (T) mode; G, preformed gasket to confine pressure and protect anvils; P, a pyrophyllite pressure transmitter; S, a specimen; R, a metallic reflector; INC, an incident sonic wave; R_n , a reflection at anvil surface; R_s^1 , a first signal reflected from a specimen surface; R_s^2 , a second signal reflected from the other surface of a specimen.

was parallel either to (0 0 1) or (1 1 0) plane of the cubic system (we call them (0 0 1) specimen and (1 1 0) specimen hereafter). The [0 1 0] crystallo-graphic axis of a (0 0 1) specimen and $[1 \bar{1} 0]$ of a (1 1 0) specimen are aligned parallel to the direction of displacement of the T mode sound wave.

The specimen was sandwiched between a couple of thin metallic foils of 10 to 20 micron thickness and inserted through a cylindrical hole into the pressure transmitter. The hole was then plugged with a couple of pyrophyllite cylinders so that the specimen was firmly held. Two thin metallic films act as reflectors for sound wave travelling from both sides of the specimen disk. The thickness of the reflectors was determined empirically and a 10 to 20 micron thick platinum, gold, molybdenum or nickel foil was found to be most favorable for our experiments. A schematic lateral view of the sample space is shown in Fig. 1.

The geometrical and acoustic characteristics of the system are as follows;

Edge length of anvil face	6 mm
Initial edge length of pyrophyllite cube	8 mm
Initial thickness of preformed gasket	1.5 mm
Diameter of specimen disk	2.5 mm
Thickness of specimen disk	1.5 to 1.8 mm
Thickness of reflector	10 to 20 μ m
Resonant frequency of L mode transducer	10 MHz
T mode	7 MHz

Acoustic impedances (zero pressure) for L mode

Tungsten carbide (anvil)	105×10^9 kg/m ² .sec
NaCl, (0 0 1) specimen	11
Pyrophyllite	(close to NaCl)
Polycrystalline platinum	69
Polycrystalline gold	63
Polycrystalline molybdenum	64
Polycrystalline nickel	53

3. *Metallic Reflectors and Pulse-Echo-Overlap Method*

An ultrasonic sound velocity of a specimen under high pressure was measured by the pulse-echo-overlap method (PAPADAKIS, 1968). A tone burst sound signal generated on the tail end of the anvil travels through the anvil to reach the front end. A part of the signal is reflected and travels back in the opposite direction, whereas the rest of the signal breaks through the boundary between anvil and pyrophyllite to approach one end of the specimen disk. At the boundary between pyrophyllite and the specimen there arises a second

reflected wave which is again sent back along the way it emerged. In this same manner a third reflected signal is generated at the other end of the specimen disk. A schematic trace of where the reflected signals are generated is represented at the bottom of Fig. 1. All these signals are converted into an electric output signal through the same transducer which generates the original excitation signal and are displayed on an oscilloscope screen. An example of a signal display is shown in Fig. 2.

For a velocity measurement we have to measure the time interval between the second and third reflected signals denoted as R_s^1 and R_s^2 in Figs. 1 and 2. A special timing circuit has been built up for this purpose. A continuous sinusoidal wave of a certain frequency is fed to a Schmidt trigger to generate a continuous pulse train. One of the pulse peaks is picked up every few tens of microseconds and is fed to a tone burst signal generator to excite the transducer (INC in Figs. 1 and 2) while other pulse signals are cut off. About 10 to 20 microseconds later (manually adjustable) after input of this

Fig. 2. (Top) A train of reflected L mode waves on an oscilloscope display along with the pair of triggering pulses. (Bottom) A closer look at the amplified L mode reflection.

particular pulse signal, two successive pulse peaks out of the original pulse train are sent through a timed digital gate to the triggering terminal of an oscilloscope. After some necessary adjustment of the electronics circuit and timing dials on a front panel of the oscilloscope, we are able to detect R_s^1 and R_s^2 at the same time on the oscilloscope display. The reflected waves R_s^1 , R_s^2 and the successive two triggering pulses are all shown in Fig. 2. If the time intervals both between reflected signals and the two successive triggering pulses coincide, R_s^1 and R_s^2 overlap completely and the travel time of a round trip of the ultrasonic sound wave is equivalent to time interval between two triggering pulses.

In order to make a correct overlapping of R_s^1 and R_s^2 , we have tried to sharpen them by inserting a pair of thin metallic films one on each side of the specimen disk. Several combinations of metals were tested and we found that platinum, gold, molybdenum and nickel thin films give a sharp signal when both L and T mode sound waves are measured simultaneously. Thickness and combination of metals showed a substantial effect not only on the sharpness of reflected waves but on an amplitude ratio between them. If it is desired to enhance reflections of only L mode, a platinum-platinum couple is suggested. In case an enhancement in T mode is necessary, a platinum film closer to L mode transducer and a gold film closer to T mode transducer would give a good result.

Special care must be taken about a phase angle of reflected waves. In any combination of metals, their acoustic impedances (ρV_L or ρV_T) at an atmospheric pressure and room temperature are larger than those of pyrophyllite or NaCl single-crystals. Therefore, reflected signals R_s^1 and R_s^2 are to be overlapped in-phase. It seems likely that this relation holds for pressure range from zero to 80 kbar inferred from pressure dependence of density and sonic velocity of metals, sodium chloride and soft rocks such as limestone (BECHMANN and HEARMON, 1966; BIRCH, 1966 and WHITFIELD *et al.*, 1976).

4. Pressure Calibration

Pressure calibration was carried out in two way, in order to see internal consistency of our measurements. Firstly, phase changes of bismuth and barium were used. Secondly, the X-ray measurement of the lattice parameter of NaCl at high pressure was used to give precise and continual data about pressure through Decker's equation of state for NaCl (DECKER, 1971). For the latter, an energy-dispersive X-ray technique combined with a solid state detector was used (AKIMOTO *et al.*, 1977). Four diffraction peaks, (2 2 2), (4 0 0), (4 2 0), and (4 2 2) of NaCl were selected as they are closely spaced. All the measurements of ultrasonic velocity, X-ray and phase transitions were

Fig. 3. Pressure calibration obtained by NaCl pressure scale versus load cell type pressure gauge output (unit: strain). Five different calibration runs (denoted 1 to 5) are plotted in the figure. One to 4 represent results from X-ray measurements and 5 is obtained by phase transitions.

provided by independent runs due to difficulties in geometrical requirements.

5. Derivation of Elastic Moduli

Elastic moduli C_{ij} 's for a single-crystal of the cubic symmetry will readily be determined if, at least, three independent velocity values are given, i.e. L and T waves propagating along $[001]$ with the T mode polarization along $[010]$ and L and T waves propagating along $[110]$ with the T mode polarization along $[1\bar{1}0]$. For calculating wave velocity values at a given pressure, we will have to measure the time interval between reflected waves R_s^1 and R_s^2 (refer to section 3) and the absolute value of sample thickness. If there is no plastic deformation at all upon compression of a specimen, we are able to assume that the length of the compressed specimen is directly proportional to the lattice parameter of the specimen. We have found, however, that there is a small amount (maximum 3%) of plastic deformation of a NaCl single-crystal after compression to 80 kbar. Therefore we have to find a solution that is not affected by a small amount of plastic deformation.

As to the ultrasonic velocity measurements, this condition can only be satisfied by a velocity (or inversely, time interval) ratio of L mode wave (V_L)

to T mode wave (V_T) through simultaneous measurements of both modes. This method was applied by MORRIS *et al.* (1976) to determine Poisson's ratio of NaCl at high pressures up to ca 100 kbar. As a result, from ultrasonic velocity measurements we get the following independent data.

Quantity of data	Equivalence	Sound waves	
		Direction	Polarization (T)
$(V_L/V_T)^2$	C_{11}/C_{44}	[001]	[010]
$(V_L/V_T)^2$	$\frac{C_{11}+2C_{44}+C_{12}}{C_{11}-C_{12}}$	[110]	[110]

The adiabatic incompressibility K_s for a cubic crystal is given by

$$K_s = (C_{11} + 2C_{12})/3.$$

Therefore, if K_s is given at a certain pressure at which V_L/V_T values are obtainable, all C_{ij} 's for a cubic crystal can unambiguously be calculated. In the first place, the isothermal incompressibility K_T for NaCl is directly derived from Decker's equation of state for NaCl (DECKER, 1971). And then correction between K_s and K_T is made through the same equation combined with the Debye formula for the heat capacitance and Decker's assumption on the Grüneisen parameter (DECKER, 1971). It is noted that the uncertainty in the absolute value of Decker's NaCl pressure scale is about 1.6% for a pressure range from zero to 80 kbar.

6. Results

It is of primary importance to check whether the reproducibility of the pressure generated in the pyrophyllite pressure transmitter is sufficiently good. Because we were not able to measure the absolute pressure while the ultrasonic measurements were carried out on a single-crystal of NaCl, we had to rely upon a load cell type pressure gauge installed in a oil-operated hydraulic ram. There is evidently a hysteretic behaviour in a displacement of press anvils between loading and subsequent unloading cycles. It was found that the reproducibility of both the pressure calibration and the ultrasonic measurements during a loading process is far better than in an unloading process. Therefore, we carried out all the measurements only on a loading cycle. The relation between pressure gauge readings (arbitrary unit in strain) and real pressure values within a pyrophyllite pressure transmitter is represented in Fig. 3. Pressures calibrated by different runs and independent samplings compared to the pressure gauge readings lie along a monotonic curve indicating good reproducibility of pressure generation and reliability of sampling procedures.

Fig. 4. Ratios of longitudinal wave velocity (V_L) to transverse wave velocity (V_T) versus pressure. Plots *a* represent data for (001) specimen (refer to text) and *b* for (110) specimen. Waves propagate along [001] with T mode displacement along [010] in a (001) specimen. In a (110) specimen waves propagate along [110] with T mode displacement along [1 $\bar{1}$ 0]. Results of two different runs are mixed for plots *b*.

Fig. 5. Elastic moduli are plotted as a function of pressure. Open symbols represent C_{ij} calculated from present measurements combined with K_s derived from Decker's equation of state. Solid lines through plots are a tentative fit by a least square calculations for pressure range, zero to 80 kbar. Solid symbols and dotted lines are C_{ij} and calculated K_s reproduced from the data of WHITFIELD *et al.* (1976).

Results of ultrasonic sound measurements are represented in Fig. 4 in the form of velocity ratios for two independent crystallographic orientations. Results of two different runs are plotted for curve *b*. A close spacing of values from both runs indicates a good quality of data.

Elastic moduli C_{11} , C_{12} , C_{44} and K_s were calculated from the relations given in section 5 and Decker's equation (DECKER, 1971). The results are plotted in Fig. 5. Pressure derivatives of C_{ij} 's are derived by a least square fit assuming tentatively that C_{ij} and pressure are linearly related in the pressure range, zero to 80 kbar. The results are listed as follows.

Zero pressure values (direct measurements of sound velocity)

$$C_{11} = 5.16 \times 10^{11} \text{ dynes/cm}^2 \quad (\text{with a } \pm 1.5\% \text{ error})$$

$$C_{12} = 1.22 \times 10^{11}$$

$$C_{44} = 1.36 \times 10^{11}.$$

Pressure derivatives (zero to 80 kbar)

$$\partial C_{11} / \partial p = 8.72$$

$$\partial C_{12} / \partial p = 1.22$$

$$\partial C_{44} / \partial p = 0.31.$$

7. Experimental Errors

There are several sources of experimental errors. The most significant of them is a signal to noise ratio (S/N) of ultrasonic waves. In the worst case S/N becomes down to about 5. Predominant part of background noise is induced by multiple reflections of sound at various parts of the high pressure system and, hence, have a frequency the same as the sound signal with various phase angles. If the typical travel time of a sound signal to complete a round trip through a 1.5 mm thick NaCl crystal is about 0.5 microseconds (L mode), S/N about 5 and a signal frequency 10 MHz, the maximum error in travel time would be 1.2%. This estimate also applies to the T mode measurements to give a similar error.

The second source of uncertainty is due to the pair of metallic reflectors. Suppose that the acoustic impedances of pyrophyllite, metallic reflectors and NaCl crystal remain constant throughout the pressure range from zero to 80 kbar, the pair of 10 micron platinum reflectors introduces a maximum relative phase shift between R_s^1 and R_s^2 of about 5 degrees (refer to the appendix). This would introduce an error of 0.8% for travel time measurements.

The third factor is an uncertainty in calculation of K_s . A discussion on the derivation of K_s has been given in section 5 and its maximum error is 4% including the uncertainty in the X-ray measurements. An additional

error induced by the correction factor between K_T and K_s is negligibly small compared to the error in K_T itself and, therefore, is not taken in account in the present study.

As a whole, a maximum error in calculation of C_{ij} is 12% assuming that all the error factors are additive. It is clear that the scatter of C_{ij} determined by the present work is well within the limit of the probable error estimated here.

8. Discussions

The pressure range of the ultrasonic sound velocity measurement of single crystals is greatly expanded, reaching 80 kbar by the present work. The technique can be applied to much higher pressures because ultrasonic sound sources are placed outside the high pressure cell. In this respect, the ultrasonic technique permits measurements in a pressure range similar to or beyond that of the Brillouin scattering method described by WHITFIELD *et al.* (1976). Figure 5 shows graphically the pressure dependence of the elastic moduli calculated from the present study and include the results from the Brillouin scattering measurements on NaCl in an overlapping pressure range. The data given by the latter were taken under hydrostatic conditions, on the other hand the present study was carried out using a solid pressure transmitter. There must be some difference in pressure conditions between the present measurement and other measurements that might have caused the discrepancy in the slope of C_{ij} with respect to pressure. The present values for $\partial C_{ij}/\partial p$ are systematically smaller than those obtained by the Brillouin scattering method. It is still not obvious that the discrepancy was really induced by a difference in pressure transmitting media. Taking a look at the K_s values versus pressure reproduced from WHITFIELD *et al.* (1976), we find a deviation from the values calculated from Decker's equation of state, which we had to rely on as a standard pressure scale. However, the agreement between present values and the Brillouin scattering data for C_{ij} 's is good if we take into account the difference in the experimental principles, the independence of experimental runs and the experimental error represented as a scatter in C_{ij} values as a function of pressure. Other data on $\partial C_{ij}/\partial p$ which have been obtained in pressure ranges lower than 10 kbar (e.g., BARTELS and SCHUELE, 1965; SPETZLER *et al.*, 1972) are systematically large compared to those given by the present work.

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Appendix Effect of Reflectors

The effect of reflectors on the phases of reflected waves is estimated simply based upon continuities of displacement and stress at the boundary of two different materials in tight contact. Suppose two different materials with acoustic impedances Z_1 and Z_2 are bounded by a flat surface S and the path of the incident plane wave is perpendicular to S as in Fig. 6. It is well established that reflection coefficient a_r (i.e., amplitude ratio of reflected wave to incident wave) and transmission coefficient a_t (i.e., amplitude ratio of transmitted wave to incident wave) are expressed as

$$a_r = \frac{Z_1 - Z_2}{Z_1 + Z_2} \quad \text{and} \quad a_t = \frac{2Z_1}{Z_1 + Z_2}.$$

It is also well established that if $Z_1 > Z_2$, the phase angle of the reflected wave and the incident wave at boundary S is equal to each other and if $Z_1 < Z_2$, the phase angle of the reflected wave differs by ± 180 degrees from that of the incident wave.

When a metallic reflector is inserted between a buffer and a specimen and the thickness D is smaller than the wave length of the incident acoustic wave, the amplitude of a reflected wave is given by adding reflected waves S_1 and S_2 as shown in Fig. 7. Suppose that the acoustic impedances of the buffer, the reflector and the specimen are Z_B , Z_R and Z_S , respectively, where $Z_R > Z_B$, $Z_R > Z_S$ and the incident angle of the wave is zero. Then the displacement of a reflected wave is given by

$$\phi_R = a \cos(\omega t + kx + \pi) + \sum_{n=1}^{\infty} b_n \cos\left\{\omega\left(t + \frac{2D}{V_R}\right) + kx\right\}$$

Fig. 6. A schematic lateral view of a flat boundary surface S between two different materials with acoustic impedances Z_1 and Z_2 .

Fig. 7. A thin metallic reflector sandwiched between different materials (buffer and specimen) with acoustic impedances Z_B and Z_S . Thickness and acoustic impedance of the reflector are shown by D and Z_R , respectively. S_1 and S_2 denote the flat boundary surfaces.

where

$$a = \frac{Z_R - Z_B}{Z_R + Z_B}$$

and

$$b_n = \frac{2Z_B}{Z_R + Z_B} \cdot \frac{2Z_R}{Z_R + Z_B} \cdot \frac{Z_R - Z_S}{Z_R + Z_S} \cdot \frac{Z_R - Z_B}{Z_R + Z_B} \cdot \left\{ \frac{Z_R - Z_B}{Z_R + Z_B} \cdot \frac{Z_R - Z_S}{Z_R + Z_S} \right\}^{n-1}.$$

The letter k denotes an arbitrary wave number, x denotes position and V_R denotes the wave velocity for a metallic reflector. The displacement ϕ_R can be expressed in a form

$$\phi_R = C \cdot \cos \{(\omega t + kx + \pi) + \Delta\varphi\},$$

where $\Delta\varphi$ is an angle of virtual phase shift caused by a reflector and C is a constant. In actual cases Z_R/Z_B and Z_R/Z_S are both close to 2 and, therefore,

$$\left\{ \frac{Z_R - Z_B}{Z_R + Z_B} \cdot \frac{Z_R - Z_S}{Z_R + Z_S} \right\}^{n-1} \doteq \{0.11\}^{n-1}$$

is negligibly small when n exceeds 2. Hence, the phase angle is approximately given in the following form;

$$\tan \Delta\varphi = \frac{b_1 \sin \frac{2D}{V_R} \omega}{a - b_1 \cos \frac{2D}{V_R} \omega}.$$

In the present work $Z_B \approx 20$, $Z_S \approx 20$, $Z_R \approx 60$ ($\times 10^6$ kg/m²·sec), $V_R \approx 3.3$ (km/sec), $D \approx 10$ (microns) and $\omega \approx 60$ (Mega rad/sec) and, therefore, $\tan \Delta\varphi$ is approximately equal to 0.1 which gives $\Delta\varphi$ approximately equal to 5.5 degrees.

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