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5. A New Method of the Measurement of Ultrasonic Wave Velocity of Solids under High Pressures up to 40 Kilobars (Part I).

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Introduction

During the past twenty years a great many measurements on ultrasonic wave velocities of solids have been carried out under high pressures. In most of those measurements a liquid or gas type high pressure apparatus has been used. Because of technical difficulties which exist in this kind of apparatus the maximum pressure of each measurement has mostly been limited to less than 15 kilobars, although there are some examples of the measurements under an extended pressure range. (e.g. CHRISTENSEN (1974), FRENKEL et al. (1976), HEYNDEMANN and HOUCK (1971), ITO et al. (1977), KINOSHITA et al. (1979), KONDO et al. (1975), MORRIS et al. (1976)).

The result obtained from these experiments have greatly contributed to formulating the "equation of state" in which a mean atomic weight of a compound is a key parameter. Through the application of this equation of state to solid geophysics our knowledge about the materials in the mantle has in some degree been made clear. For the application of this equation to solve problems concerning the earth's deeper interior, however, it is deeply regretted that the maximum pressure of those measurements did not exceed 15 kilobars.

Purpose of this study is to develop a method of measuring the ultrasonic wave velocities of materials of geophysical interest under pressures as high as possible, more than 20 kilobars at the minimum, without sacrificing the accuracy which is common in the measurement under ambient conditions, and, if possible, under hydrostatic pressures.

Conditions necessary for the accurate measurement of ultrasonic velocity of solid are as follows; the size of a sample should be large enough (preferably a cm-size at the smallest), and the shape should be defined exactly.

For the measurement under very high pressures much more severe conditions are imposed. First of all let's consider the maximum available size of the sample. In generating high pressure, the higher the pressure to be generated the smaller the effective volume in which pressure is generated. Actually, it is almost impossible to use a cm-size sample in the velocity measurement under the high pressures on the order of tens of kilobars. Therefore, it becomes very important to develop a technique to measure sound velocities of a millimeter size sample without sacrificing accuracy. The authors have already developed the electronic system (pulse-echo-overlap method) for measuring sound velocity of a millimeter-size solid sample (FUJISAWA et al. (1974), KINOSHITA et al. (1979)), so this will not be discussed in this papper.

Another important point is the nature of pressure. Types of a high pressure apparatus which are able to generate pressures up to 40 kilobars or more in a volume large enough for the velocity measurement are very limited. A piston-cylinder type apparatus and a multi-anvil press might be only available types for the present purpose.

For the high pressure generation in the present measurements a piston-cylinder type apparatus has been used. In an experiment with this kind of apparatus a solid pressure medium is usually used. In this case pressures generated in a solid medium is not hydrostatic. Under nonhydrostatic conditions it is almost impossible to keep from changing the shape of the sample, and it is very difficult to keep the transducer from being destructed.

In this study the authors developed a new sample assemblage which is not only convenient for the sound velocity measurement but also has a liquid (hydrostatic) pressure cell. With this assemblage the authors succeeded in measuring ultrasonic wave velocities of very small solid samples (a few millimeters in length) under hydrostatic pressures up to 40 kilobars.

Sample Assemblage

The sample assemblage newly developed in this study is show in Fig. 1. This is an example for a 16 millimeter inner diameter cylinder. Lengths, diameters, or thickness of each part of this assemblage in Fig. 1 can be varied in accordance with requirements to each specific experiment, or with a different diameter of a cylinder. The simplest method for measuring velocity under high pressures is to put a sample bonded with transducer directly into a pressure medium. In this method, however, protection of the transducer might be a problem, because it is directly subjected to high pressures. Another difficulty which might arise from this method is the unequall amount of compression between a transducer, bound, and sample because of the difference of compressibilities of these components. This might destroy a good contact between a transducer and a sample, which is definitely important for the effective and noiseless transmission of ultrasonic waves.

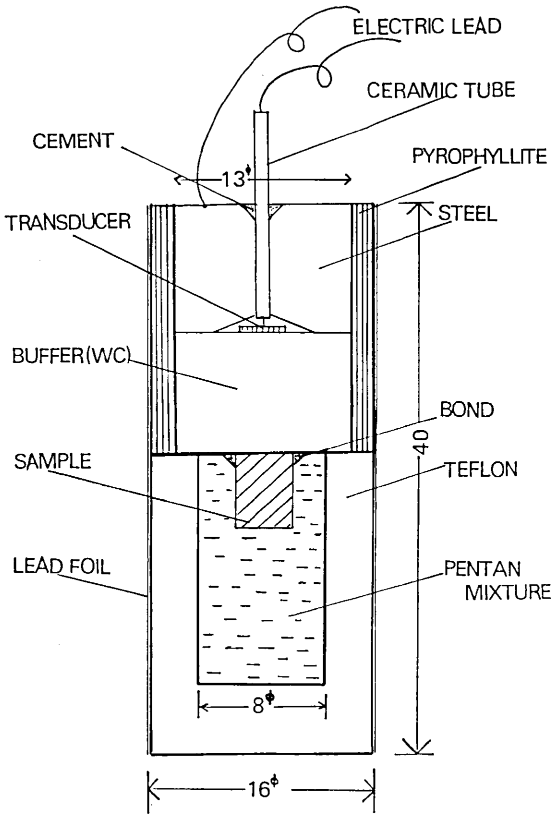

Fig. 1. The sample assemblage newly developed in this study. Dimensions in millimeter.

Therefore the ideal method of the measurement is that in which a sample only is subjected to pressures. The sample assemblage shown in Fig. 1 is one solution for the above mentioned problems, and is very near to the ideal one. In this assemblage no bond is used on the contact surface between a buffer rod and a sample. Bond is used only around the sample to fix it to the rod.

Maximum usable length of the sample in the assemblage is about 5 millimeters. Because of highly compressible nature of liquid the bottom of a pressure cell would touch directly to the end of the sample under the pressures of about 30 kilobars if its length is longer than about 5 millimeters.

Pulse-echo-overlap method, phase comparison technique, and a simple wave transmission method are easily applicable to this buffer rod configuration, but it would be very difficult to use pulse-superposition method in this sample-transducer assemblage.

Example of Result of the Measurement

An example of the result of the measurement of P-wave velocity of polycrystalline aluminum obtained by this assemblage with pulse-echo-overlap method is shown in Fig. 2. Length correction of the sample

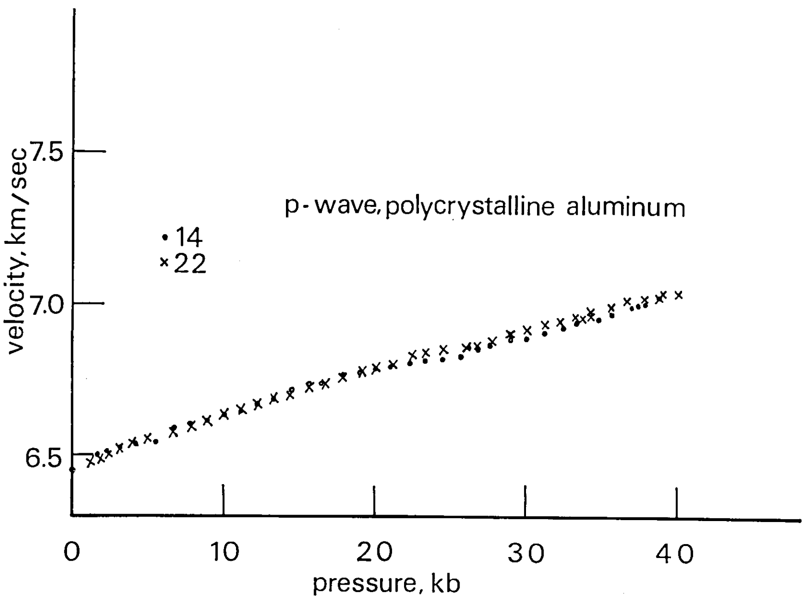

Fig. 2. Pressure dependence of P-wave velocity of polycrystallne aluminum. (Run No. 14 and 22).

under high pressures was made using recent lattice compression data of alminum (SENOO et al. (1976)).

Pressure calibration was made separately with a manganin wire or Bi. For the pressure calibration similar assemblage was used, but instead of a sample a caliblant (manganin wire, or Bi) was placed in a pressure cell. Pressure was estimated using the electrical resistance change of manganin wire based on recent experimental data (YAMAMOTO (1972a, 1972b)) and the Bi I-II transition defined by New NBC scale (HALL (1971)).

Sensitivity of the electronic system (pulse-echo-overlap method) to the change of velocity is more than 10^{-4}, and enough to follow the change of velocity with increasing or decreasing pressure. So scatter of data seen in Fig. 2 is originated mostly from error in estimating pressure in each run. This is a typical weekpoint in the separate calibration.

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5. 4万気圧までの高圧下で，固体の弾性波速度を測定する新しい方法について (第一報)

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今までの「高圧下での弾性波速度の測定」においては，ポンプ直結型高圧装置 (液圧または気体圧) が用いられるのが普通であった．そして，この種装置の制約から，その最高圧力が1万5千気圧を超える事はまれであった．それらの測定から得られた結果は，いわゆる「平均原子量一状態方程式」等の議論を通じて，地球物理学的にも，数多くの興味ある知見をもたらした．しかし，測定時の最高圧力が1万5千気圧程度という事は，地球の内部構造の研究という見地からすると，はなはだ不満足なものである．本研究では，測定の精度を犠牲にする事なく，4万気圧までの静水圧下で，固体の弾性波速度の測定を行う方法を開発したので報告する．高圧発生には，ピストン・シリンダー型装置を用いる。高圧シリンダー内で静水圧を発生させるためのテフロン液圧セル，振動子，中間導体，および試料間の構成法に，本方法の特徴がある．