131. Pressure Generation Using New Transmitting Media

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(Comm. by Tei-ichi Ito, M. J. A., Sept. 12, 1975)

Fine MgO particles have been semi-sintered at 1,300°C for about 10 hours till its density increases up to 2.3. The quenched block contains still so many voids uniformly distributed that an ordinary machine tool can work well upon it. A new pressure transmitting medium is made of this material as shown in Fig. 1. Besides, a pyrophyllite medium with similar shape and dimension is carefully prepared. In the center of each medium a pressure calibrant ZnS is embedded. The two specimens are compressed at room temperature within a spherical pressure vessel designed by Kawai et al. (split sphere with eight cubic anvils).

The metallic transition of the calibrant is observed under an external oil pressure of 100 kg/cm² in pyrophyllite, whereas it occurs under only one half of pressure in the new medium.

Next a similar comparison has been carried out using GaP as calibrant. The metallic transition occurs at 60 kg/cm², in contrast with 460 kg/cm² required in pyrophyllite.

To reconfirm the high efficiency a well known electrical change of ZnTe has been tested in the same way as before.

ZnTe has three phase transitions under increasing pressure at room temperature, as shown by sharp kinks a, b and c in resistance (Fig. 2). It first drops at 80 kbar, jumps up at 110 kbar, and finally drops again abruptly at 130 kbar. The curve obtained within the new medium resembles to that obtained within pyrophyllite before

Fig. 1. Pressure transmitting media octahedral in shape. Pyrophyllite (left) and MgO (right).
the load reaches 60 kg/cm². On loading further, however, the first kink foregoes slightly, but the second one goes further in the MgO medium. The final drop is completed even before it starts in pyrophyllite. To understand the quick pressure rise in MgO, one must assume an existence of an inward pressure rise whose gradient is gentle in the beginning, but becomes progressively steep.

The voids within the shrinking MgO medium have been examined under the ordinary microscope. They are diminished and soon turn out to be invisible. Some part of the medium becomes semi-transparent, too. The void reduction is not taking place uniformly in the medium, it initiates in the proximity to the anvils and then goes farther away. A solid octahedral shell is, therefore, formed and soon thickens till each calibrant is completely surrounded (Fig. 3). When outer part of the MgO octahedron extrudes out into the interfaces between anvils, air filling in the nearby voids comes out together. However, the remaining air, especially that trapped in closed voids is liquefied and eventually solidified at pressure above 30 kbar and

![Fig. 2. Curves showing electrical resistance change of ZnTe.](image)
stays therein. A completely non-void block is made before the highest internal pressure is attained. MgO single crystal possesses six planes of cleavage, each being equivalent to (001). Therefore, the crystal is quite crisp being, pulverized by a small shearing stress. In the outer part of compressed medium white thin layers are recognizable with the naked eye. They are lying on three sheared planes (mutually perpendicular and parallel to the anvil faces). When an outer shell of the MgO octahedron extrudes, pulverized particles move along the planes, although the amount becomes smaller very quickly. In consequence of this flow, the octahedron looks as though split up into eight quasi-tetrahedrons whose each center makes a fine contact with the calibrant. Since the center of each tetrahedron has a very limited area in case the calibrant is small, each tetrahedron plays the leading role in both the transmission and magnification of external oil pressure. It is nothing but a growing anvil with a top progressively sharpened, and three shoulder surfaces supporting mutually. By using a relatively large MgO medium we can prevent the rupture of anvil made of expensive tungsten carbide without sacrificing the pressure-volume attainability.
There remains a serious problem to be solved as yet namely whether the elevated pressure around the calibrant is hydrostatic, or not. Air in the voids is quite ample, since the semi-sintered MgO shrinks till it has the true density of 3.6. A great deal of air leaks out of the medium in the early stage. But still sufficient air remains inside, some being solidified and the other moved to encounter the calibrant. The calibrant is to be lapped up with a solidified layer, however thinner it may be. The thin layer is made up of such solids as O₂, N₂ and others which are softer than the growing MgO anvil. The pressure must, therefore, be for the most part hydrostatic, although generated in the solid medium.

A particular explosion has been clearly noticed in the course of decreasing load. It occurs when the internal pressure drops to about 30 kbar. It is quite likely that the solidified air is released and the gas is emitted instantaneously.

As other media alternative, we have used aggregated hard powders of diamond, boron, cubic BN, BeO, TiO₂, ZrO₂, WC, TiC, B₄C, etc., differing widely in hardness and cleavage. Their behaviors are the same as with MgO.

On the other hand, the new media show almost similar effect in such pressure vessels as the classical piston-cylinder or any new sliding multi-anvils.

Our heartfelt thanks are first to Prof. Ito who kindly looked through a series of our manuscripts and then to the Ministry of Education whose financial support enabled our experiment.

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