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Permeability of Stemming Materials for Prompt Gas Sampling

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Manuscript Date: January 1982

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Abstract

The permeability and porosity of a suite of man-made granular aggregates and stemming materials currently in use at NTS was measured in 1-D loading as a function of stress. In all cases, the gas permeability was measured at 22 MPa after cycling up and down from 100 to 1200 MPa. Depending on stress and material, permeability decreased up to three orders of magnitude, porosity up to 63% and the sample compacted by as much as 35%. Steel ball bearings were found to retain the highest permeability of all the materials tested. The enhancement of prompt gas sampling through alternate stemming material in the column above the nuclear device is discussed.

Introduction

One way to determine nuclear device performance after underground nuclear detonations is to sample the composition of the radioactive cavity gases. Prompt gas sampling system was used to recover samples soon after detonation. The downhole portion of this system is primarily a hose put in before stemming (filling) the emplacement hole. The hose ends above the anticipated cavity. Gas from the cavity must flow to the hose through stemming material compacted by the detonation.

Optimal use of stemming material and sampling hose placement requires knowledge of the *in situ* stress field and the resulting changes in stemming material permeability and porosity immediately after a detonation. The determination

of laboratory permeability porosity stress data when taken together with stress calculations could be used to significantly enhance prompt gas sampling success.

Eight different materials were tested. Five of the materials are currently in use as stemming materials at NTS: Chevreux quartzite gravel, Overton quartzite sand, HNL coarse fill from NT5 (tuff fragments), HNL stemming mix (NTS fines and flyash), and magnetite (Table I). The remaining three materials were selected for their hardness, toughness, cost, and availability in quantities appropriate for prompt gas sampling. These three materials were impure alumina (fired bauxite), pure alumina balls, and steel ball bearings.

Table I. Tested materials.

Material	Specification No. description	Size	Initial porosity
Chevreux quartzite gravel	MEI 79-001486-A	2.36 to 12.5 mm	0.43
HNL coarse fill	MEI 76-001400-A	2 to 12.5 mm	0.49
Overton (quartzite) sand	MEI 76-001292-A	75 μ m to 2 mm	0.35
HNL stemming mix (H) with flyash	MEI 75-001282-A	.15 to 12.5 mm	0.31
Magnetite	MEI 76-001300-A	150 μ m to 4.72 mm	0.31
Alumina, impure	Porous, irregular shaped balls	5.08 to 6.35 mm	0.43
Alumina, pure	Spheres (3.4 specific gravity)	6.35 mm	0.44
Steel ball bearings	Spheres, chrome alloy, composition I, grade 50	5.56 mm	0.42

Experimental Method

For each material type listed in Table 1, we measured sample permeability and porosity as a function of axial stress. The samples were progressively cycled to 1200 MPa in 100-MPa increments. After each cycle, axial stress was released to 22 MPa and permeability and porosity were measured. This cycling simulated the increase/release of stress after a nuclear detonation. The chosen 22-MPa stress value maximized the ease of die alignment and press operation. Although a lower value would better approximate the 10- to 20-MPa

residual stress field forming a containment cage around the detonation cavity (Terhune, 1978),¹ we believe that measurements made at 22 MPa would not be significantly different from those determined at 10 to 20 MPa.

Figure 1 shows a schematic diagram of the die assembly containing the test sample. The sample is 50.8 mm in diameter and has an initial

¹ R. W. Terhune, *Tracers in High Temperature and Pressure*, Lawrence Livermore National Laboratory, Livermore, CA, UCRL 52395 (1978).

Figure 1. Schematic diagram of pressure die and sample.

length of 45.7 mm. The die is terminated at each end with 5.55 mm steel ball bearings in a dimpled, drilled plate and a slotted piston that has a central orifice (Fig. 2). Axial stress is applied to the top of the sample by a 500-ton capacity hydraulic press. Figure 3 is a schematic diagram of the plumbing and instrumentation.

We introduced a flow of nitrogen to the sample, maintaining the pressure drop across the sample by adjusting the vent valve. The mass flow rate, upstream pressure, and average pressure were monitored with mass flow and pressure transducers. All measurements were performed at about 20 °C. To assure reproducibility of the data, we measured at least two samples of each of the different materials (except the steel ball bearings).

Measurements at several different flow rates were recorded after each stress increment to assure that the measurements were performed in the Darcy flow regime. The permeability of the sample to gas is expressed by:

$$K = \frac{Q\mu L}{A\Delta P}$$

where,

K is permeability;

Q is nitrogen flow rate divided by average density;

μ is nitrogen viscosity;

L and A are length and cross sectional area of the sample respectively, and

Figure 2. Schematic diagram of piston and dimpled, drilled plate.

ΔP is the gas pressure difference between the ends of the sample.

Ball bearings distributed the gas over the entire sample end and prevented plugging of the holes in the plate with the crushed sample at high stresses. The dimpled tool steel plate supported the bearings and the drilled holes distributed the gas between the bearings. The other side of the plate was grooved to connect the gas path from the piston to the distribution holes. To eliminate the need to calibrate the pressure drop in the tubing, we monitored gas pressure with a pressure transducer at each sample end. The accuracy of the pressure transducers determined the maximum permeability that could be measured while the accuracy of the flow meters determined the

minimum permeability. Estimated accuracies for permeabilities of $2 \times 10^{-11} \text{ m}^2$ to $4 \times 10^{-16} \text{ m}^2$ (20 to 4×10^{-4} darcies) are $\pm 10\%$.

In addition to permeability, the initial length and initial porosity of each material were also measured. Initial porosity was determined from the weight difference between water saturated samples and dry samples. When the samples are water saturated, it is assumed that the water fills the intergranular pore space only. Porosity was determined from the initial porosity, initial sample length, and the change in length during the experiment. Initial sample porosity is expressed by:

$$\phi = \frac{a - b}{\rho V}$$

Figure 3. Schematic diagram of plumbing and instrumentation.

where,

ϕ is porosity,

a is mass of water saturated sample,

b is mass of the dry sample,

ρ is the density of water, and

V is the sample volume.

We used a film potentiometer as a displacement gauge to determine the change in length of the sample. The sample compaction was corrected

for the sample filling the voids around the ball bearings as the sample was stressed. Since the length measurements were made only at the top piston an error occurs after release of the stress because of the die "floating" on the bottom piston (Fig. 1). Estimated sample length and porosity errors are initially $\pm 1\%$ and $\pm 4\%$ respectively and range to $\pm 4\%$ and $\pm 6\%$ respectively after cycling to high stresses.

Results

We measured eight different materials for permeability and porosity. Figure 4 shows the permeability of the materials at 22 MPa axial stress versus the maximum stress to which they have been cycled. The five stemming materials

studied appeared to define two groups. In the first, magnetite, Chevreux quartzite gravel, and Overton sand have permeabilities that range from 10^{-12} to 10^{-13} m² for maximum stresses of 100 to 1200 MPa. The second group consists of H-Ni

Figure 4. Permeability at 22-MPa stress vs maximum stress.

Figure 5. Permeability at 22-MPa stress vs maximum stress and permeability at maximum stress.

course fill and LLNL stemming mix and are considerably lower in permeability. Second-group materials were not stressed to 1200 MPa since the flow rates were too small to accurately measure permeability. LLNL coarse fill permeability ranges from 10^{-12} to 10^{-10} m² for maximum stresses of 100 to 600 MPa and LLNL stemming mix permeability ranges from 10^{-13} to 10^{-15} m² for maximum stresses of 100 to 300 MPa. Several materials were examined as possible alternatives for prompt gas sampling application. These included alumina balls and steel ball bearings. Permeabilities for impure and pure alumina balls were about the same and range from 10^{-11} to 10^{-10} m² for maximum stresses of 100 to

1200 MPa. Permeabilities for steel ball bearings are substantially higher than all the other materials tested and range from 10^{-10} to 10^{-11} m² for maximum stresses of 100 to 1000 MPa. Comparison between duplicate tests on the same material indicates that the reproducibility of permeability data for each material at any stress is about $\pm 15\%$.

In Fig. 5, we compare the permeability behavior of several materials that are representative of the four groups shown in Fig. 4. The two curves for each material depict the effect on permeability of unloading from the highest stress. In each case, the upper curve, which is identical with the one in Fig. 4, shows the permeability-stress behavior

when measured at 22 MPa after unloading from the maximum stress. The lower curve shows permeability-stress before unloading. Unloading resulted in a 0.5 order of magnitude increase in permeability at the highest stresses. We believe that this increase is caused either by particle rearrangement or by fractures opening when the sample is unloaded. In addition, the data suggest that there is a greater difference in permeability for the less permeable materials.

Figure 6 depicts the sample compaction at 22 MPa stress as a function of the maximum stress to which the sample was cycled. The materials with the greatest initial porosity (see Table 1), except the steel ball bearings, decreased the most in length. For example, I.I.N.I. coarse fill decreased 38% in length for maximum stresses of 0 to 600 MPa. Alumina and quartzite gravel decreased 31% for maximum stresses of 0 to 1200 MPa. Overton sand decreased 29% and magnetite de-

creased 20% for maximum stresses of 0 to 1200 MPa. I.I.N.I. stemming mix decreased 14% for maximum stresses of 0 to 600 MPa. The steel ball bearings only decreased 18% for maximum stresses of 0 to 1000 MPa.

Figure 7 illustrates porosity at 22 MPa stress as a function of the maximum stress to which the sample was cycled. Porosities for impure alumina range from 0.43 to 0.18 and quartzite gravel from 0.43 to 0.13 for maximum stresses of 0 to 1200 MPa. Overton sand has a porosity ranging from 0.35 to 0.16, pure alumina 0.44 to 0.18, and magnetite 0.31 to 0.12 for maximum stresses of 0 to 1200 MPa. Steel ball bearings has a porosity ranging from 0.42 to 0.29 for maximum stresses of 0 to 1000 MPa. Porosities of I.I.N.I. stemming mix range from 0.31 to 0.17 and I.I.N.I. coarse fill range from 0.49 to 0.18 for maximum stresses of 0 to 600 MPa. Each material showed good reproducibility in porosity.

Figure 7. Porosity at 22-MPa stress vs maximum stress.

Discussion

Before it is subjected to high stress, Chevreux quartzite gravel has a permeability greater than that of Overton sand. We have shown that Overton sand and quartzite gravel have essentially the same permeability once subjected to stress exceeding 100 MPa. Therefore, the *less expensive* Overton sand can be used in the place of quartzite gravel as a stemming material. LLNL coarse fill and LLNL stemming mix have significantly lower permeabilities than the other materials tested. This is not unexpected since they are composed of a mixture of volcanic and sedimentary pebbles. The weaker sedimentary pebbles crushed sooner than the volcanic pebbles and filled the pore spaces around the volcanics (Fig. 8). This resulted in a greater decrease in permeability than if all the pebbles were of uniform composition. The phenomenon of material being crushed and flowing around intact pebbles or balls and providing support for the remaining intact material is clearly shown with alumina balls (Fig. 9). This phenomenon is not as obvious when the initial grain size is small and/or the sample is not cohesive after stress is applied.

Steel ball bearings retained the highest permeability of all the materials tested. Alumina is harder and stronger than the steel ball bearings. However, the bearings have a higher ductility and thus allow plastic deformation at the contact points (Fig. 10) resulting in lower stress concentrations. The steel ball bearings were thus able to support higher loads without crushing, and therefore maintain a higher permeability with stress.

From the tests that were conducted, we expect that the best prompt gas sampling material is both hard and ductile. Alumina and steel ball bearings are best suited for this use since they retain high permeabilities at high axial stresses. Overton sand and quartzite gravel are three orders of magnitude less permeable at the same conditions. The best stemming materials for gas containment are easily crushed and as a result are

low in permeability such as the LLNL stemming mix.

Figure 8. LLNL coarse fill (top) and LLNL stemming mix (bottom) after loading to 600 MPa.

Figure 9. Impure alumina balls (top) and pure alumina balls (bottom) after loading to 1200 MPa.

Figure 10. Steel ball bearings after loading to 1000 MPa. Arrow points to flattened surface.

Summary and Conclusion

Permeability, compaction, and porosity measurements were made on eight different materials for stresses to 1200 MPa. Stress was increased in 100 MPa increments and released to 22 MPa to simulate the increase/release of stress after a nuclear detonation *in situ*. Permeability of NTS stemming materials ranged from 10^{-14} m^2 to 10^{-15} m^2 at 600 MPa stress (Fig. 4). LLNL stemming mix showed the lowest permeability. Possible prompt gas sampling materials include alumina balls and steel ball bearings. Permeability of alumina balls and steel ball bearings ranged from 10^{-7} m^2 to 10^{-5} m^2 at 600 MPa at maximum stresses (Fig. 4). Steel ball bearings are one to four orders of magnitude more permeable than the other materials tested at the expected *in situ* stresses. Therefore, prompt gas sampling should

be much enhanced by the use of steel ball bearings in the critical stress region.

There is a general trend in average porosities ranging from 0.17 to 0.23 at 600 MPa with the exception of the steel ball bearings. The high permeability materials tend to have high average porosities; while the low permeability materials have low average porosities with the exception of magnetite. Whether this trend is meaningful is hard to determine since there is not a large difference in final porosities.

We believe that the permeability data in this report will allow engineers to better design prompt gas sampling systems. In addition, the data will have general applicability to the test program in the design of stemming plans for containment.

Acknowledgments

We thank M. Towler and E. Arnold for help in building the apparatus and H. Heard for helpful suggestions of alternate stemming materials.