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# Isostatic Solvent Pressing

**It is not likely that this method will replace methods used with present materials in production. Its chief value lies in the increased flexibility given in the formulation of new materials**

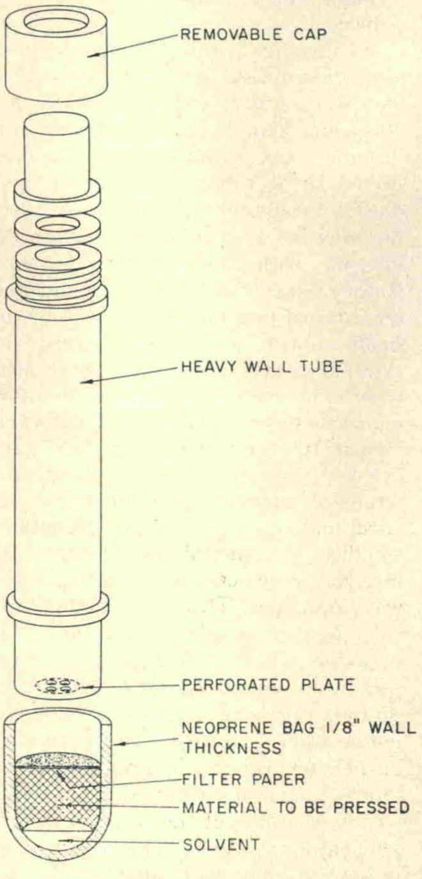
ISOSTATIC SOLVENT PRESSING is a method for forming solid, dense compacts of crystalline materials. It is a combination of two techniques: isostatic pressing and solvent pressing. Isostatic pressing consists of placing the material to be pressed into a rubber bag, evacuating it, and then subjecting the bag to isostatic pressure in a thick-walled pressure vessel capable of withstanding 20,000 to 30,000 p.s.i. The pressurizing fluid may be water or, if a pressing temperature higher than 100° C. is required, other fluids may be used. This process has been developed by the ceramic industry for the manufacture of certain types of insulators, and has been adapted to the pressing of high explosives by various agencies of the Department of Defense. Solvent pressing consists of placing a small amount of a suitable solvent in a die, adding the material to be pressed, and then applying mechanical pressure by means of a loose-fitting ram. The powder under pressure is believed to have a greater solubility at the points of contact of the individual grains, hence is dissolved at these points and redeposited in the interstices of the granular mass. This results in a compaction of the material, which forces the solvent upward where it finally escapes around a loose-fitting ram or through an orifice. The addition of a small amount of solvent to a powder or granular material prior to pressing is an old trick. Water, for example, is sometimes added to salt before it is pressed into salt blocks for cattle.

Isostatic pressing has economic advantages over mechanical pressing in cases where large pieces are to be made for later machining. The temperatures and pressures needed for the formation of dense compacts are approximately those required for ram and die pressing.

Solvent pressing has the advantage over other forms of mechanical pressing in that lower pressures and temperatures are required. In addition, it can be used to form dense pieces of materials and mixtures of materials which cannot be formed by any other method.

## Apparatus

The two techniques—isostatic pressing and solvent pressing—were combined at this laboratory in an attempt to utilize the special advantages of each system. The special apparatus for this purpose (below) consists of an open-end Neoprene bag into which is inserted a metal chamber (heavy wall tube) fitted across the mouth with a perforated metal plate. In operation, solvent is placed in the Neoprene bag, powder is poured in, and the chamber is inserted. The assembly is then subjected to isostatic pressure. Solution and recrystallization proceed as in mechanical solvent pressing. As compaction progresses, air and solvent from the powder are forced out into the chamber (called the solvent chamber).



**Experimental isostatic-solvent-pressing apparatus**

**The removable cap is for cleaning**

Although isostatic solvent pressing can be done at high pressures like conventional isostatic pressing, a principal advantage of the system is that a low pressure of 2000 p.s.i. may also be used. The press time in isostatic solvent pressing is normally much longer than for conventional mechanical or isostatic pressing. Even though most of the compaction occurs in the first few hours, in many cases press times of 18 hours or longer have been used. Pressures of 2000 p.s.i. can be obtained from bottled compressed gas, and in these investigations this source of pressure was used rather than a mechanical pump.

## Parameters Affecting Density of an Isostatic-Solvent-Pressed Explosive

In addition to the geometry and size of the piece to be pressed, other parameters of significance are press time, pressure, temperature, particle size and size distribution, and solvent.

**Press Time, Pressure, and Temperature.** The effects of time, pressure, and temperature are shown in Table I for an experimental explosive. (To achieve a density of 1.85 grams per cc. in conventional isostatic pressing, this particular material requires a temperature of 125° C. and a pressure of 20,000 p.s.i.) Table II shows the results for shorter press time at a high pressure.

**Particle Size and Size Distribution.** Particle size, apparently, should be as small as practical—preferably below 10

Reprinted from **INDUSTRIAL AND ENGINEERING CHEMISTRY**, Vol. 53, Page 737, September 1961

microns as an average particle size—with the distribution normally found in ground materials. In one instance an explosive having a particle size of 100 microns could not be formed into a cohesive piece, but when ball-milled to an average particle size of 6 microns it was pressed to 100% theoretical density.

**Solvent.** The amount of solvent used varies from 2 to 10%. This would not be critical, except that too little solvent means that only a small portion of the material is exposed to solvent action at one time and the press time is prolonged. An excess of solvent results in a slurry which gives a deformed part or allows material to be extruded through the perforated plate into the solvent chamber.

In selecting a suitable solvent, several factors were considered. Solvents that would dissolve between 5 and 25% of the material being pressed were considered to have a satisfactory solvent action. Consideration was also given to the ease of removal of residual solvent and to possible reactions between the solvent and any of the components of the material to be pressed. The boiling point of the solvent can and should be fairly low. During pressing, the system is completely enclosed, and no evaporation can take place. It was found to be quite feasible to perform the pressing operation well above the normal boiling point of the solvent provided the system is cooled below the solvent's boiling point before releasing the pressure on the press. Another alternative is to use a venting system from the solvent chamber to the atmosphere. Acetone, by virtue of its general good solvency and ease of removal, has been in the solvent most used in these experiments.

### Table I. The Effect of Certain Variables on the Density of an Isostatic-Solvent-Pressed Explosive

Theoretical maximum density = 1.89 g./cc.
Solvent: acetone, 5% by weight
Sample size: 10 grams

| Variable | Time, Hr. | Pressure, P.S.I. | Temp., ° C. | Density, G./Cc. |
|---|---|---|---|---|
| **Time** | 1 | 1700 | 25 | 1.83 |
| | 2 | 1700 | 25 | 1.84 |
| | 4 | 1700 | 25 | 1.88 |
| | 18 | 1700 | 25 | 1.89 |
| **Pressure** | 18 | 270 | 25 | 1.80 |
| | 18 | 500 | 25 | 1.85 |
| | 18 | 1000 | 25 | 1.86 |
| | 18 | 1700 | 25 | 1.89 |
| **Temperature** | 1 | 2000 | 100 | 1.87 |
| | 18 | 1000 | 25 | 1.88 |
| | | | 3 | 1.85 |

### Table II. The Effect of Press Time on the Density of an Explosive Isostatically Solvent Pressed at 20,000 P.S.I.

Sample material: same as in Table I
Solvent: acetone, 5% by weight
Sample size: 10 grams

| Time, Hr. | Temp., ° C. | Density, G./Cc. |
|---|---|---|
| 0.5 | 25 | 1.85 |
| 1 | 25 | 1.86 |
| 2 | 25 | 1.87 |

The apparatus of all tables (page 737) was used.

### Table III. Isostatic Solvent Pressing of Propellant Formulations$^a$

| Oxidizer | % | Fuel | % | Solvent$^a$ | Pressure, P.S.I. | Time, Hr. | Temp., ° C. | Density, G./Cc. |
|---|---|---|---|---|---|---|---|---|
| **$NH_4NO_3$** | 75 | Al | 25 | $NH_4NO_3$ Saturated acetone | 2000 | 18 | 25 | 1.93 |
| **$NH_4ClO_4$** | 75 | Al<br>KelF | 22.5<br>2.5 | $NH_4ClO_4$ Saturated acetone | 2000 | 18 | 25 | 1.97 |

$^a$ 10%.

**Size and Shape of Piece to Be Pressed.** The initial experiments were conducted with 10-gram samples, which were subsequently increased in several cases to 300 grams and then to 35 pounds. The densities achieved under the same operating conditions were as high for the large samples as for the 10-gram samples. In larger billets, it seemed advisable to consider the transmission of pressure within the piece. The ratio of volume to area would increase, which might mean that the pressure at the center of a large billet would be considerably reduced.

To investigate this problem of pressure transmission, a rubber bulb filled with water was placed in the center of a 300-gram sample in a conventional isostatic press. This bulb was connected by a tube to a pressure gage outside the isostatic pressure vessel. In this way it was possible to compare the internal with the isostatic pressure. Rather unexpectedly, it was found that the internal pressure as measured by the small rubber bulb could exceed the external pressure by as much as 40% with some materials, while with other materials there would be no measurable internal pressure even with 2000 p.s.i. external pressure. About a dozen materials at random were tested for this effect, but no method for predetermining whether a material would show an increase or decrease in internal pressure was apparent. This effect is undoubtedly related to the "pressability" of materials under nonsolvent isostatic pressing conditions, as well as to solvent isostatic pressing. This pressure intensification effect is being studied further for other applications. If an "ideal" material exists, that is, one which would transmit all the force externally applied, a means of producing ultrahigh pressures would be obtainable.

In addition to size, which the above experiment would seem to indicate is not a problem, the shape of the piece for this type of pressing is important. This is determined by the shape of the rubber container, how the material is loaded—i.e., how tightly it is packed—the bulking value of the material, and probably many other factors as yet not even known.

**Geometry.** Although, in the laboratory experiments, the solvent chamber was placed with the rubber bag in the isostatic vessel, when the time came to scale up the technique it was decided to place the solvent chamber outside the pressure vessel. This was done by using a conventional isostatic rubber bag with a rubber cover. A metal tube was inserted into the rubber cover and was passed through a seal in the isostatic vessel. The solvent chamber, no longer having to withstand external pressure, could be replaced by a glass bottle into which the solvent flowed as the pressing proceeded.

## Pressing of Propellant Formulations

In addition to pressing pure crystalline compounds, it is possible to press mixtures where one or both materials are soluble in the solvent. To maintain the original concentration of the two materials, it is necessary to use a solvent that has been saturated with the two materials. For example, a mixture of 75% ammonium nitrate and 25% aluminum powder was pressed to over 99% theoretical maximum density using acetone saturated with ammonium nitrate (Table III). The addition of a polymer to the material being pressed is possible in certain instances where the polymer is not soluble in the solvent. In this way it was found possible to combine the components in a high-energy propellant system which could not be cast or extruded and which could be pressed only under very high pressures. Ammonium perchlorate (75%), aluminum powder (22.5%), and a fluorinated hydrocarbon (2.5%) were combined in this way into a billet having 96% theoretical density (Table III). Unfortunately, such a material does not have the rubberlike qualities favored in solid propellants.

RECEIVED for review December 27, 1960
ACCEPTED March 30, 1961

Work supported by U. S. Atomic Energy Commission under Contract W-7405-eng-48.

PRINTED IN U. S. A.