

Fig. 1. Sample assembly. All dimensions given in inches.

point considerably, (2) be readily formable into a good-quality glass, (3) be readily devitrifiable, and (4) be well understood.

A system which easily meets all these criteria is the system lithia-alumina-silica. A great deal of information is available in the literature on this system.¹ It is readily formed as a glass and is as readily devitrified. For these reasons, the following composition was chosen for the study: Li_2O 20, Al_2O_3 10, and SiO_2 70% (wt).

The equilibrium crystalline phases are lithium metasilicate and β -spodumene. When crystallized from the glass, however, these phases appear only after treatment at elevated temperatures (600° to 900°C) for several (~16) hours, through recrystallization of a previously crystallized mixture of lithium metasilicate and β -quartz solid solution (i.e., silica "O").

II. Apparatus

The superpressure apparatus at the U. S. Army Signal Corps Laboratories was used for these experiments. This device, which has already been described,² consists, essentially, of two sets of symmetrically opposed stepped cemented carbide pistons and a cylinder assembly consisting of a double-bore cemented carbide member surrounded by a series of hardened-steel bands.

¹ (a) S. D. Stookey, "Method of Making Ceramics and Product Thereof," U. S. Pat. 2,920,971, January 12, 1960; *Ceram. Abstr.*, 1960, June, p. 142a.

(b) Rustom Roy and E. F. Osborn, "The System Lithium Metasilicate-Spodumene-Silica," *J. Am. Chem. Soc.*, **71** [6] 2086-95 (1949); *Ceram. Abstr.*, 1950, May, p. 111d.

(c) M. K. Murthy and F. A. Hummel, "Phase Equilibria in the System Lithium Metasilicate- β -Eucryptite," *J. Am. Ceram. Soc.*, **37** [1] 14-17 (1954).

(d) R. A. Hatch, "Phase Equilibrium in the System $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2$," *Am. Mineralogist*, **28**, 471-96 (1943); *Ceram. Abstr.*, **23** [1] 26 (1944).

(e) Rustom Roy, D. M. Roy, and E. F. Osborn, "Compositional and Stability Relationships Among the Lithium Aluminosilicates: Eucryptite, Spodumene, and Petalite," *J. Am. Ceram. Soc.*, **33** [5] 152-59 (1950).

² A. A. Giardini, J. E. Tydings, and S. B. Levin, "Very High Pressure-High Temperature Research Apparatus and Synthesis of Diamond," *Am. Mineralogist*, **45** [1-2] 217-21 (1960); *Ceram. Abstr.*, 1961, May, p. 132b.

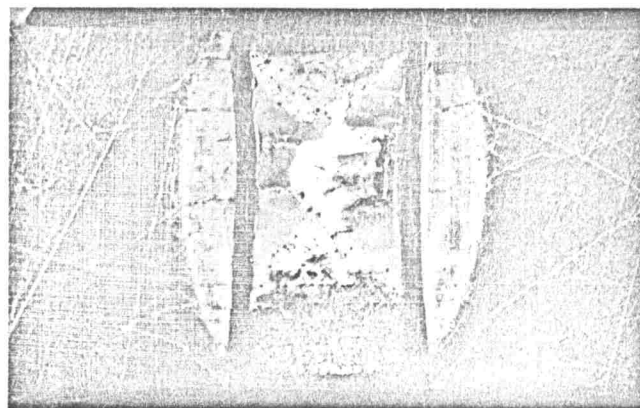


Fig. 2. Melting of a lithium aluminosilicate powder batch to form a glass.

The sample area is shown in Fig. 1. The sample itself is enclosed in a Pt20Rh tube with platinum caps at both ends. This in turn is placed into a pyrophyllite cylinder and capped at both ends with a thin nickel disk. The assembly is placed between the pistons of the superpressure apparatus for the experimental run.

Pressures were determined by extrapolation from the first two pressure-induced bismuth electrical resistance discontinuities³; the limits of error are approximately $\pm 5\%$ for the range of pressures used in this study. Temperatures were obtained by linear extrapolation of input power versus platinum melting point⁴ at the particular pressure employed; the accuracy is within $\pm 50^\circ\text{C}$ at 500°C and $\pm 100^\circ\text{C}$ at 2000°C for a given position in the sample capsule.

III. Experiments and Results

(1) Experiments on Glass Synthesis

A series of experiments was made to study the effect of pressure on the glass-melting process. The raw materials were Harshaw Chemical Company 325-mesh silica, Norton Company 38900 alumina, and Foote Mineral Company technical-grade lithium carbonate. The materials were carefully weighed out, intimately mixed, and compacted into the platinum heating tube. On completion of the experiment, the sample assembly was embedded in lucite and cross-sectioned to permit visual observation (see Fig. 2).

It was found that melting of the powder and subsequent formation of the glass was seriously impeded. The powder remained after subjection to temperatures well in excess of the normal melting point of the mixture at 900°C . The melting point appeared to increase with increasing pressure (see Fig. 3).

The alteration of the powder before melting was also investigated. As the temperature was increased the white powder turned to gray (see Fig. 4). During further heating the material became partly white again just before melting. Subsequent heating of the darkened powder at 600°C in air reversed the darkening. A similar darkening has been ob-

³ (a) P. W. Bridgman, "Resistance of Nineteen Metals to 30,000 Kg./Sq. Cm.," *Proc. Am. Acad. Arts Sci.*, **72**, 157-205 (1938).

(b) P. W. Bridgman, "Pressure-Volume Relations for 17 Elements to 100,000 Kg./Sq. Cm.," *Proc. Am. Acad. Arts Sci.*, **74** [13] 425-40 (1942).

(c) P. W. Bridgman, *Physics of High Pressure*. G. Bell & Sons, Ltd., London, 1949. 445 pp.

(d) F. P. Bundy, "Phase Diagram of Bismuth to 130,000 Kg./Sq. Cm., 500°C ," *Phys. Rev.*, **110** [2] 314-18 (1958).

⁴ H. M. Strong and F. P. Bundy, "Fusion Curves of Four Group VIII Metals to 100,000 Atmospheres," *Phys. Rev.*, **115** [2] 278-84 (1959).