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**FIRST-STAGE DENSIFICATION OF CaF_2
UNDER PRESSURE AND TEMPERATURE**

Berkeley, California

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Yasou Hashimoto

(M. S. Thesis)

January 21, 1964

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FIRST-STAGE DENSIFICATION OF CaF_2 UNDER PRESSURE AND TEMPERATURE

Yasou Hashimoto

Department of Mineral Technology, and
Inorganic Materials Research Division
Lawrence Radiation Laboratory
University of California
Berkeley, California

January 21, 1964

ABSTRACT

The initial densification of spherical CaF_2 powders was determined by dilatometric and microscopic observations. The experiments consisted of heating the powder to a fixed temperature at a constant rate under a load and then releasing the load and quenching. Further experiments were performed by heating to a fixed temperature without load application, rapidly applying the load and holding for 40 sec. or 1 h, and releasing the load and quenching.

The initial stages of hot pressing were divided into three regions. Region I exhibited little densification. Region II showed densification by limited plastic flow and fracturing. Region III above a pressure dependent critical temperature, exhibited extensive densification due to plastic flow, fracturing of particles, and their subsequent rearrangement. After this initial densification, the densification rate was consistent with the equations proposed by Murray et al.⁸

I. INTRODUCTION

Hot pressing is widely used to achieve high density ceramic materials. Bodies with low porosity and a small grain size can be produced by hot pressing at lower temperatures and in shorter times than in pressureless sintering. From a theoretical point of view, the mechanism of mass transport during hot pressing is of great interest. In sintering two major mechanisms have been reported; volume diffusion^{1, 2, 3, 4} and plastic flow.^{5, 6, 7} Murray et al.⁸ have suggested that a plastic flow mechanism is the major densification mechanism in hot pressing because the volume diffusion mechanism is too slow to account for the rate of densification and can not explain the observed end point density phenomenon. Murray et al. also modified the MacKenzie-Shuttleworth model⁷ and developed a rate equation for densification under applied pressure at high temperatures. A number of investigations have supported this work.^{9, 10, 11, 12} These investigations, however, have concentrated on the later stages of densification where porosities are below 25 percent.

The initial stages of hot pressing have not been extensively investigated. Williams¹³ in studying the densification of metal powders considered the process to occur in two stages. In the first stage a high temperature creep mechanism was proposed which agreed with the experimental data. Felton¹⁴ hot pressed alumina powder at low temperatures to obtain data on the initial stages of densification. His results precluded plastic flow as the predominant mechanism responsible for the large initial density increases. Felton suggested the mechanism operative in the early stages was that of particle rearrangement by boundary sliding.

Because each stage contributes to the achievement of the final density this study was concentrated on the initial stages of densification. The system selected and equipment used allowed observations both of the microstructure produced by hot pressing and the densification rate during hot pressing.

II. EXPERIMENTAL PROCEDURE

A. Preparation of Spherical CaF_2 Powder

Natural crystals of Illinois Fluorite were selected because of their ease of spheroidization and relatively low melting point. Table I gives the spectrographic analysis of this material as determined by Allen.¹⁵ The crystals were slightly violet in color. X-ray analysis indicated a trace of CaO in the received crystals and approximately the same amount in the spheroidized material.

The crystals were milled in aqueous suspension in an alumina ball mill. Particles -270 to +400 mesh were separated by wet screening. After complete drying the particles were feed through a Thermal Dynamics DC Plasma Jet.¹⁶ Argon gas was used both for the primary arc and the carrier gas to prevent oxidation of the CaF_2 by oxygen or water vapor.¹⁷

The particles obtained from the plasma torch were either unspheroidized angular particles, hollow spheres, or solid spheres. A fraction of the material from the plasma torch that passed through a 325 mesh (44 micron) screen and that was retained on a 400 mesh (37 micron) screen was air elutriated with the elutriator adjusted to remove hollow spheres and retain solid spheres of the previous size range. In this operation a large fraction of the angular particles were also removed. Further separation of the nonspherical particles was made by passing the remaining batch through a series of 325 mesh screens. During this operation spheres passed through the screens faster than angular particles and further assisted in the separation.

The above process yielded approximately 15 weight percent of the original feed to the plasma torch as spherical particles. Photomicrographs of the particles including a cross section of a representative batch are shown in Fig. 1. As shown in Fig. 1b both single crystals and polycrystalline spheres were obtained. An occasional particle exhibited some porosity.

B. Hot Pressing

The hot pressing assembly is shown in Fig. 2. The graphite die was heated by radiation from the molybdenum coil. All runs were made under a vacuum less than 10^{-3} mm of Hg. The temperature was measured by a thermocouple in the graphite die as close as possible to the sample position. The temperature was controlled by an automatic temperature controller driving a saturable core reactor which in turn varied the power to the molybdenum coil. The die assembly was charged with 8 gms of spheroidized powder for each run. This sample charged would result in a disk one inch in diameter and approximately one quarter inch thick if pressed to theoretical density.

During hot pressing the change in sample thickness and dimensional changes in the die assembly and plungers due to thermal expansion were recorded by a linear variable differential transformer mounted as shown in Fig. 2. The position of the transformer was selected to keep it as far removed from high current AC lines carrying power to the heating coil.

After hot pressing, compact volumes were calculated by mercury displacement and dimensional measurement. An accuracy of about ± 3 percent was achieved in the final density calculations.

Two series of hot pressing runs were made. In the first, the pressure was applied at room temperature and kept constant to the maximum temperature when it was released to the minimum pressure of 125 psi. The die assembly was heated at 40°C per minute to the desired temperature and then rapidly cooled. The plunger travel was recorded continuously during this process. The low temperature slope of the recorded runs varied with applied pressure and the temperature at which displacement was first noted also was pressure dependent. It is thought that the compressibility of the graphite and associated parts is responsible for this pressure effect. The initial displacement temperature and low temperature slope were reproducible and once a linear slope was obtained any deviation was due to dimensional changes in the sample.

In the second series, a holding pressure of 125 psi was applied by the vacuum operating on the plunger. This pressure was maintained constant while the sample was heated to the desired temperature at 40°C per minute. After a short temperature stabilization period, the pressure was rapidly applied. The pressure was held for either 40 secs or 1 h then rapidly released to 125 psi and the sample cooled. During the 1 h soak at constant temperature, the continuous record of sample displacement and final measured density were used to calculate a density-time curve. All hot pressing runs showed good reproducibility and no chemical changes in the sample were detected by X-ray analyses.

C. Microscopic Examination

The low density hot pressed compacts were so brittle that a mounting technique was developed for their examination. The specimen was

cut on a diamond saw to give a flat plate with its width either perpendicular or parallel to the applied load during hot pressing. Both flat surfaces were then covered with paper cement.¹⁸ A sugar block was placed on the specimen and an organic casting resin was used to encase the specimen and sugar as shown in Fig. 3a. After the resin had hardened, the sugar was dissolved in warm water and the paper cement exposed was scratched away. The sample was then mounted on a vacuum flask as shown in Fig. 3b and impregnated with a dilute organic resin. The specimen surface was rough ground on emery paper and polished with submicron alumina powder on a cloth wheel.

HCP^{17, 19} (10 gm sulfamic acid in 500 cc of water) was used to etch the specimens. Adequate etching was achieved in 10 to 15 minutes at room temperature. The etched surfaces were plated with a thin gold layer and examined with a metallographic microscope.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Densification Under Constant Applied Pressure

The displacement-temperature curves obtained by heating at a constant rate under a constant applied pressure are shown in Fig. 4. Each curve shows the same general features. First, a linear slope due to the thermal expansion with little indicated densification (Region I). Second, a departure from the linear slope and a region in which the sample densification matches the expansion characteristics of the die assembly (Region II). Lastly, a region in which the sample densification proceeds at a very rapid rate and results in shrinkage in the overall assembly's length (Region III). This is well illustrated by the curve for 1000 psi in Fig. 4. The temperatures which divide these regions are shown to be pressure dependent by the two dotted curves (S and P) in Fig. 4. Table II summarizes the final density achieved by heating samples at a constant rate to various temperatures under various pressures. As shown under 2000 psi pressing to 700°C resulted in a small (2.9 percent) change in density where as the same pressure when applied till 1000°C results in a 19.6 percent increase in density.

The effect of pressure on increasing the density of a specimen heated to one temperature and quenched is shown in Fig. 5 for temperatures of 700 to 1000°C. The density increase over the room temperature value attained by heating below the transition temperature from Region I to II is relatively small. Heating above the Region II and III transition temperature causes a marked density increase. The displacement curves and densities after heating to 700, 850, and 1000°C were used to construct the density-temperature curve shown in Fig. 6.

The three regions are again evident. The rate of densification for the three regions were calculated to be 0.18, 0.65 and 5.0 percent of theoretical density per minute for Regions I, II, and III respectively. However, the rates are difficult to interpret since the samples were being heated continuously.

The microstructures of specimens hot pressed to 700 and 1000°C at 40°C per minute under 1000 psi are shown in Fig. 7. Figure 7a shows essentially the initial packing of the spheres. Figure 7b has the appearance of a sintered compact with porosity occurring between grains and evidence of bonding between particles.

Figure 8 better illustrates the changes in microstructure occurring during the hot pressing sequence. Figure 8a shows spheres in random packing with little evidence of deformation. After hot pressing to the end of Region II (Fig. 4), the microstructure shows fracture of particles (top right Fig. 8b) and rearrangement to give a denser structure. Except for fractures noted in a few particles, the spherical shape is being maintained. On heating into Region III (Fig. 8c), extensive deformation and fracture of the particles has occurred.

In Fig. 9, the effect of the uniaxial loading during hot pressing is shown. When the surface normal to the direction of load application (Fig. 9b) is compared to the surface parallel to the direction of load application (Fig. 9c), it appears as if a less dense structure is formed. This anisotropy would occur if particles were deforming and forming oblate spheroids with the minor axis parallel to the load. This would also account for the apparent grain size difference in the two photomicrographs. Figure 9a well illustrates the extensive particle

deformation and fracturing occurring when pressing above the transition temperature from Region II to III.

The above observations indicate that the initial stages of hot pressing CaF_2 spheres can be divided into three stages. Stage I, the spherical particles rearrange themselves by boundary sliding to give a few percent increase in density. Stage II, a few particles fracture and some deformation of individual particles is observed. In Stage III, particles deform significantly and fracturing accompanies this deformation. The deformation and fracturing is pressure sensitive and allows particle rearrangement to give significant densification. Fracturing appears the predominate reason for densification because unfractured particles retain their spherical shape.

B. Densification Under Rapidly Applied Pressure at Temperature

Four runs were made by heating to a fixed temperature and rapidly applying the desired pressure. In the first two runs, the CaF_2 was heated to 850°C at 40°C per minute under 125 psi. A pressure of 2000 psi was rapidly applied after the temperature was stabilized. The pressure was released after 40 sec and the sample rapidly quenched. The above procedure was also followed except that the pressure was held for 1 h. The displacement-temperature and displacement-time results are shown in Fig. 10. The microstructure after applying pressure for 40 sec and 1 h is shown in Fig. 12a and 12b, respectively. The temperature of 850°C is below the Region I to II transition temperature observed in Fig. 4 for 125 psi and above this transition temperature for 2000 psi applied load.

In the second two runs, the sample was heated to 920°C at 40°C per minute under 125 psi. After a short temperature stabilization, a

pressure of 3000 psi was applied. The pressure was held for 40 sec or 1 h, rapidly released and the sample quenched. The record of these runs is shown in Fig. 11. The microstructures after 40 sec and 1 h under pressure are shown in Fig. 12c and 12d respectively. In this case, the initial heating was maintained below the Region I and II transition temperature and the densification under pressure above the Region II to III transition temperature as given in Fig. 4.

The density increase achieved in applying pressure for 40 sec or 1 h below the Region II to III transition temperature was 9.2 and 14.9 percent respectively for 2000 psi applied load. Above the Region II to III transition temperature, the density increases were 17.7 and 33.8 percent for 40 sec and 1 h respectively. These results are summarized in Table II. The microstructures for the 850°C runs (Fig. 12a and 12b) indicate that in the first 40 sec particle rearrangement is primarily responsible for the densification and that this continues when held under pressure for longer periods. Little particle deformation or fracturing is observed.

If the pressure is applied above the Region II to III transition temperature particle deformation and fracturing occur immediately (Fig. 12c) and continue for longer times leading to structures of decreased individual particle size due to fracturing and deformation of the spherical particles that do not fracture (Fig. 12d).

These results are compatible with the other series of runs where the pressure was continuously applied while heating at a constant rate. The sudden application of pressure does result in slightly increased

densities probably due to the lack of bridges formed between particles by rearrangement when heated under pressure.

C. Plastic Deformation of CaF_2

Parker et al.²⁰ attempted to plastically deform single crystals of CaF_2 at room temperature by bending. No ductility was observed. Burn and Murray²¹ found considerable ductility in CaF_2 single crystals at 175°C in bending tests performed with low loading rates. Phillips¹⁷ observed considerable ductility about 400°C in compression tests. He proposed that the critical resolved shear stress in CaF_2 could be fitted to an equation:

$$\tau_y = \tau_o \exp \frac{K}{T} \quad (1)$$

where τ_y = the critical resolved shear stress for flow,
 τ_o = a stress constant,
 K = a constant, and
 T = the absolute temperature.

The exact stress transferred between particles in hot pressing is impossible to determine, but if the hot pressing pressure is plotted vs the reciprocal of the temperature between Regions II and III (dotted line P in Fig. 4) a relation:

$$P \propto \exp \frac{1}{T_c} \quad (2)$$

is obtained as shown in Fig. 13. This again indicates that plastic flow is a criterion for rapid densification. However, the microstructures observed also suggest that the fracturing of individual particles and subsequent rearrangement by boundary sliding accompanies plastic flow

and probably makes a major contribution to the density achieved in the early stages.

The rate equation for hot pressing developed by Murray et al.⁸ and based on the plastic flow model is

$$\frac{dD}{dt} = \frac{3p}{4\eta} (1-D) \quad (3)$$

where

D = the relative density,

t = the time,

p = the applied pressure, and

η = the viscosity of the rheological system.

This equation is valid where the temperature is above the critical temperature for viscous flow under the specific applied pressure and volume diffusion proceeds at a negligible rate. Integration of this equation gives

$$\ln (1-D) = \frac{3p}{4\eta} t + \ln (1-D_0) \quad (4)$$

where,

D_0 = the initial packed density.

In Fig. 14 $\ln (1-D)$ is plotted vs time for the two runs made at constant temperature and pressure. Both observed curves indicate a rapid initial densification followed by a linear portion agreeing with the behavior described by equation 4. The points on the figure refer to the microstructures given in Fig. 12.

Also, if the data from Table II is used for those samples heated under a constant pressure and at a fixed rate to either 700° or 1000°C and $\ln (1-D)$ is plotted vs pressure applied a straight line is obtained. This is shown in Fig. 15 and again agrees with equation 4. The time is

a constant fraction because at the fixed heating rate and quenched from the maximum temperature.

Therefore, the initial stages of hot pressing under constant pressure for CaF_2 spheres may be divided into three stages. The applied pressure determines the temperature of transition between stages. In the first stage a small amount of densification occurs through particle sliding and rearrangement. In the second stage plastic flow and some fracturing of particles occurs to give densification. The amount of plastic flow is limited probably by a creep mechanism and limits the amount of fracturing and subsequent densification. In the third stage, plastic flow becomes relatively easy and extensive fracturing accompanies the deformation to give a rapid densification.

When CaF_2 spheres are heated to a high temperature under low pressures and a high pressure rapidly applied, the densification rate is determined by the pressure. With the hot pressing in Stage II, an initial increase in density by limited plastic flow and fracturing occurs. The rate then decreases finally approaching a relationship as given in equation 3 and is probably determined by a creep mechanism. Hot pressing in Stage III results in a large initial densification due to extensive fracturing of the spherical particles and then a decreasing rate period over a large density range with continued plastic flow, fracturing, and rearrangement. The rate finally approaches the relationship as described in equation 3. Hot pressing in Stage III results in a much greater increase in density due to plastic flow and fracturing than is observed in Stage II hot pressing.

IV. CONCLUSIONS

Hot pressing of spherical CaF_2 powders result in initial densification by plastic flow accompanied by fracturing and rearrangement of the particles. A critical temperature for extensive fracturing due to plastic deformation exists and is pressure dependent. After densification by fracturing and rearrangement, the rate of densification approaches that proposed by Murray et al.⁸ The density at which Murray's equation applies depends on the temperature and pressure and how the pressure is applied to the system. Maximum initial densification is achieved by applying the pressure rapidly at a temperature above the critical temperature for that pressure.

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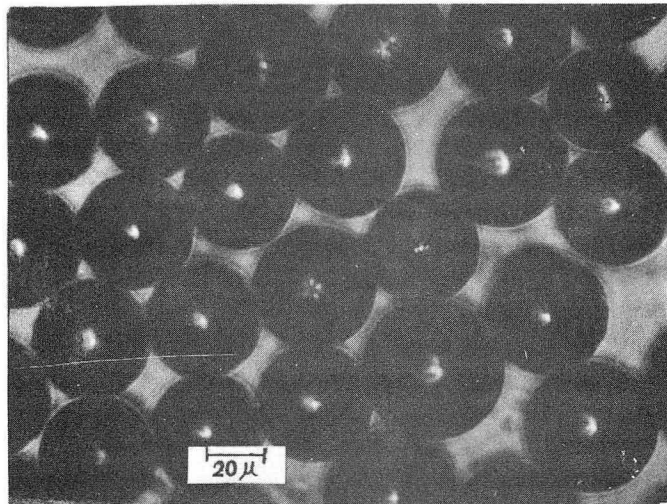
Table I. Spectroscopic Analysis of Minor
Elements in Illinois Fluorite¹⁵

Element	Limits (weight percent)
Mg	0.0005 - 0.005
Al	0.0005 - 0.005
Mn	0.0005 - 0.005
Sr	. 0.5 - 0.5
Y	< 0.0005
Rb	> 0.0005

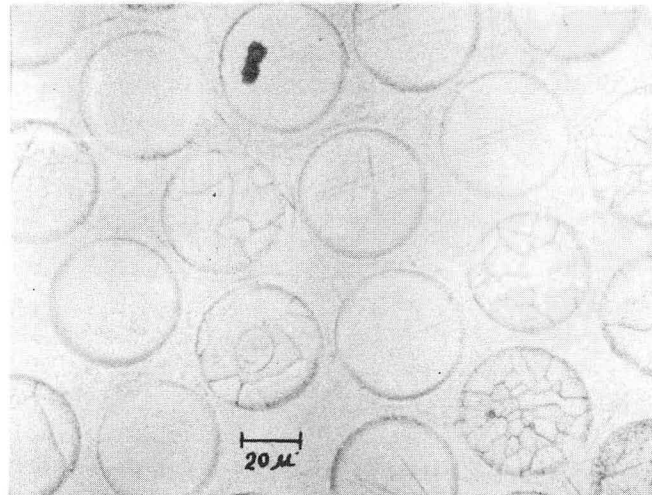
Table II. Density of Hot-Pressed Calcium Fluoride
Spherical Powder (3.18 gm/cm^3 , theoretical
density)

Specimen No.	Pressure (psi)	Max Temp. (° C)	Soak time (min.)	Density(% theoretical) Compact* Hot-Pressed	
1	125	1000	0	55.6	60.6
2	500	1000	0	57.8	65.0
3	1000	1000	0	58.4	71.2
4	2000	1000	0	60.1	79.7
5	3000	1000	0	60.9	86.6
6	500	700	0	58.0	57.8
7	1000	700	0	58.3	59.3
8	2000	700	0	60.0	62.9
9	1000	850	0	58.2	61.9
10	2000	900	0	59.7	67.1
11	125 → 2000 (850)	850	2/3	55.8	65.0
12	125 → 2000 (850)	850	60	55.5	70.7
13	125 → 3000 (920)	920	2/3	56.0	73.3
14	125 → 3000 (920)	920	60	55.6	89.4

*Initial compact density measured dimensionally before heating.



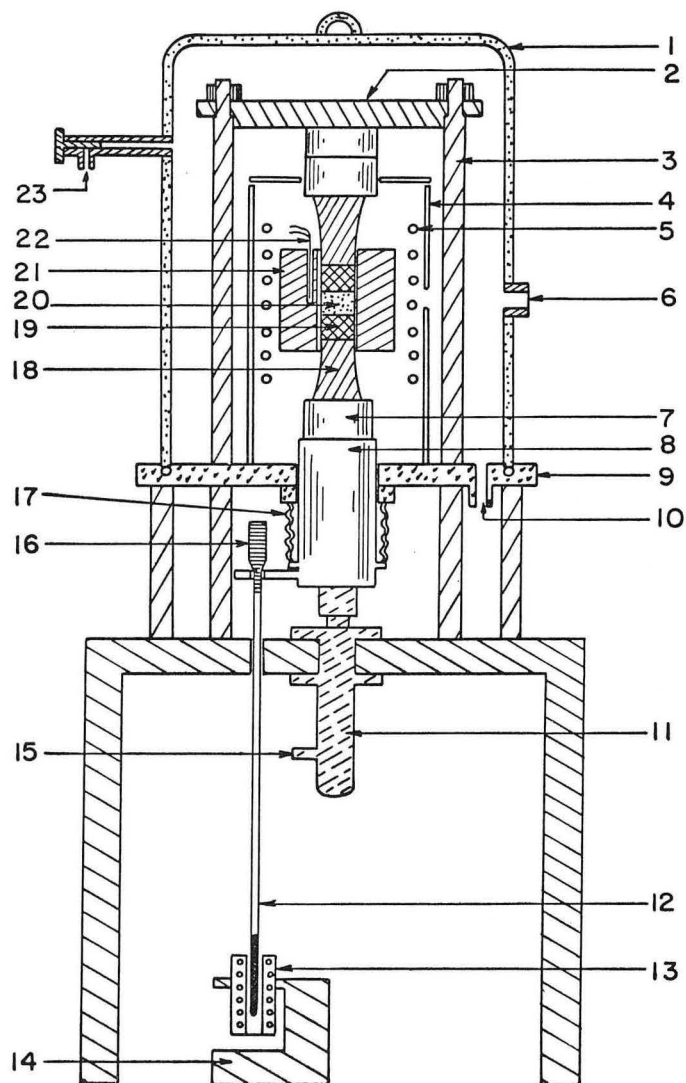
A



B

ZN-4175

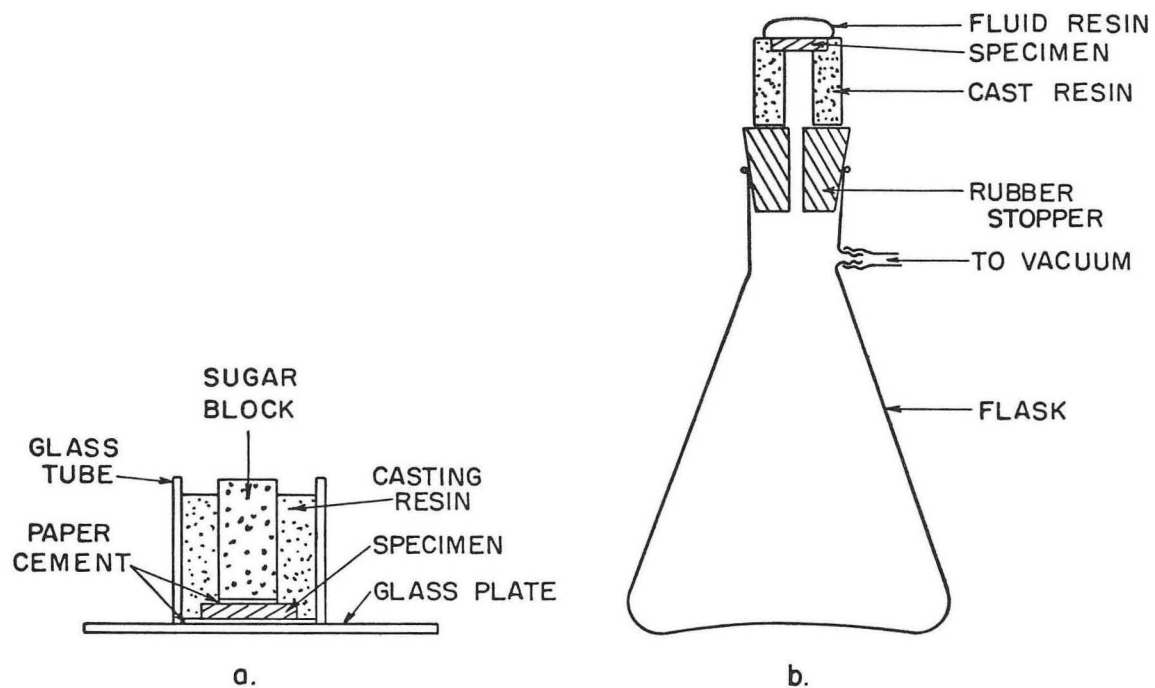
Fig. 1. Spheroidized CaF_2 particles (37 to 44 microns in diameter). (a) Dispersed on a glass plate, and (b) etched cross section (Black spot in the upper portion is a pore).



MU-33512

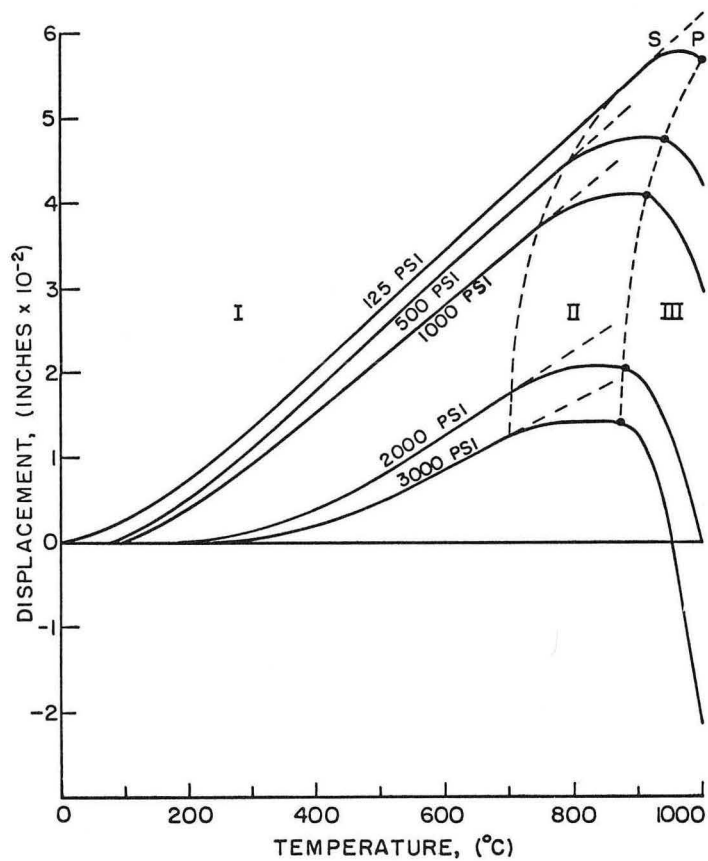
Fig. 2. Vacuum hot pressing assembly.

- | | |
|-------------------------|--|
| 1. Vacuum cover | 13. Linear variable differential transformer |
| 2. Top yoke | 14. Support block |
| 3. Load bearing column | 15. Pressure inlet |
| 4. Radiation shield | 16. Micrometer |
| 5. Heating coil | 17. Bellows |
| 6. Sight window | 18. Graphite plunger |
| 7. Alumina block | 19. Graphite plug |
| 8. Copper plunger | 20. Specimen |
| 9. Bottom support plate | 21. Graphite die |
| 10. Vacuum port | 22. Thermocouple |
| 11. Hydraulic ram | 23. Air inlet |
| 12. Glass rod | |



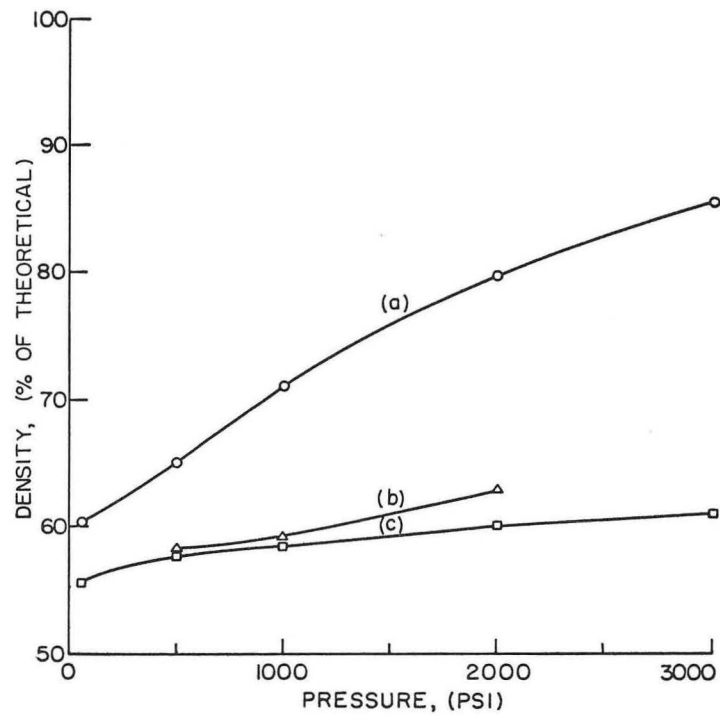
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Fig. 3. Mounting of porous specimens for microscopic examination. (a) Mounting the specimen for impregnation. (b) Impregnation technique.



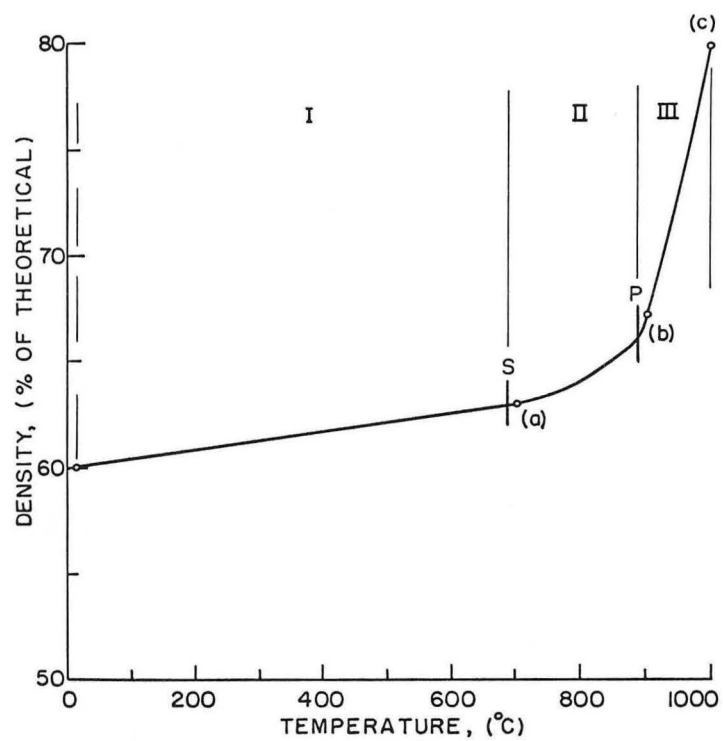
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Fig. 4. Displacement-temperature for CaF_2 heated to 1000°C under various pressures (heating rate 40°C per minute).



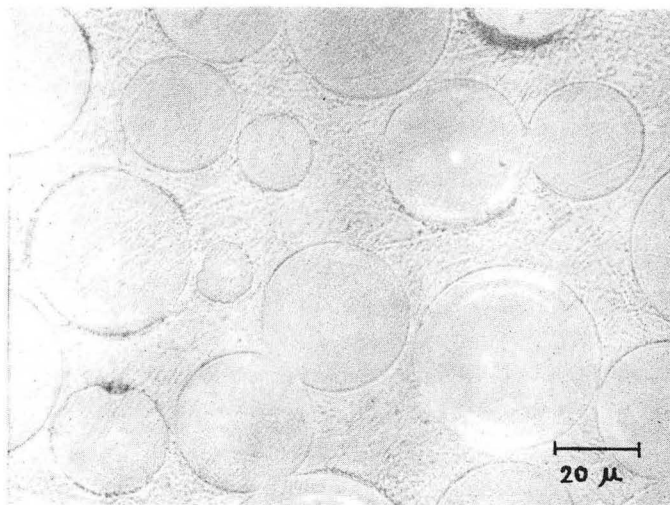
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Fig. 5. Effect of pressure on the final density. (a) Heated to 1000°C. (b) Heated to 700°C. (c) Compact density at room temperature.

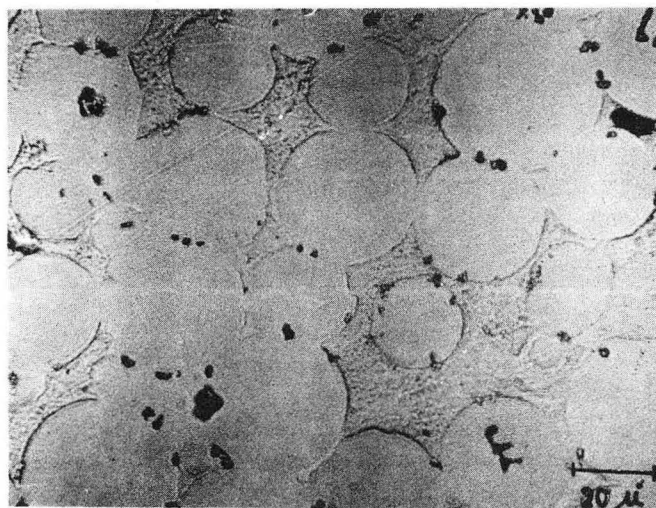


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Fig. 6. Density-temperature curve for heating at a constant rate of 40°C per minute under 2000 psi applied load.



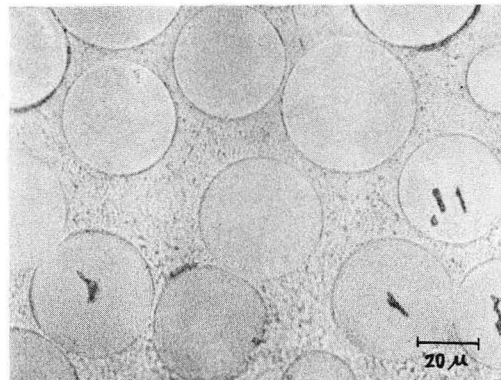
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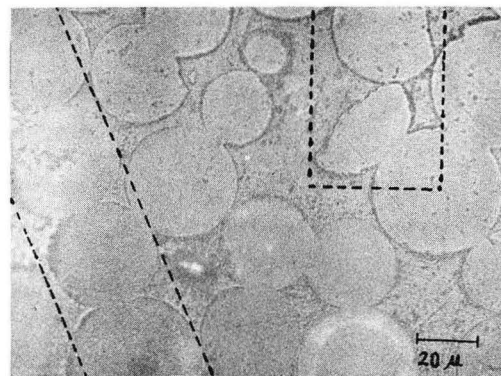
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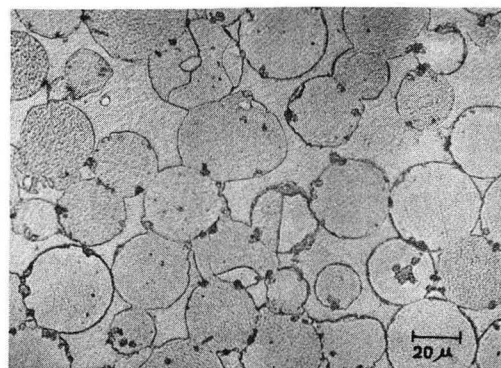
Fig. 7. CaF_2 spherical powders pressed at 1000 psi.
(a) Heated to 700°C . (b) Heated to 1000°C .
Specimens not etched and load applied parallel to
the sections.



A



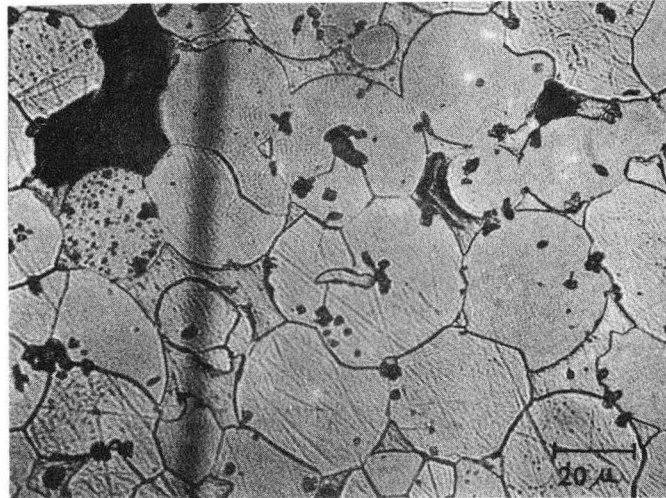
B



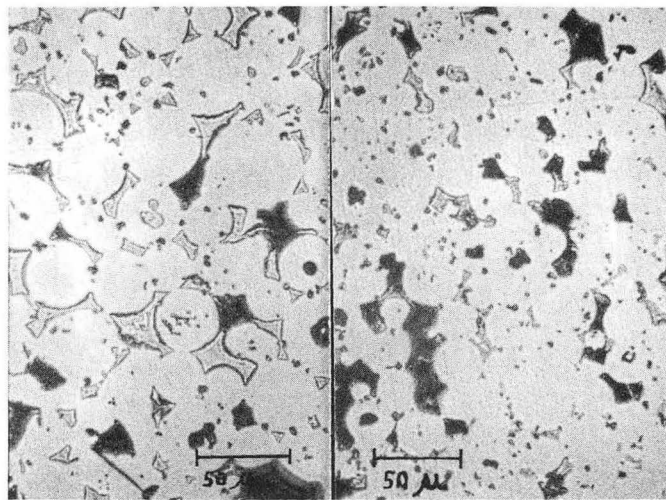
C

ZN-4177

Fig. 8. CaF_2 spherical powder hot pressed under a constant pressure of 2000 psi. (a) Heated to 700°C and not etched. (b) Heated to 890°C and not etched. (c) Heated to 1000°C and etched. Load applied parallel to the sections.



A

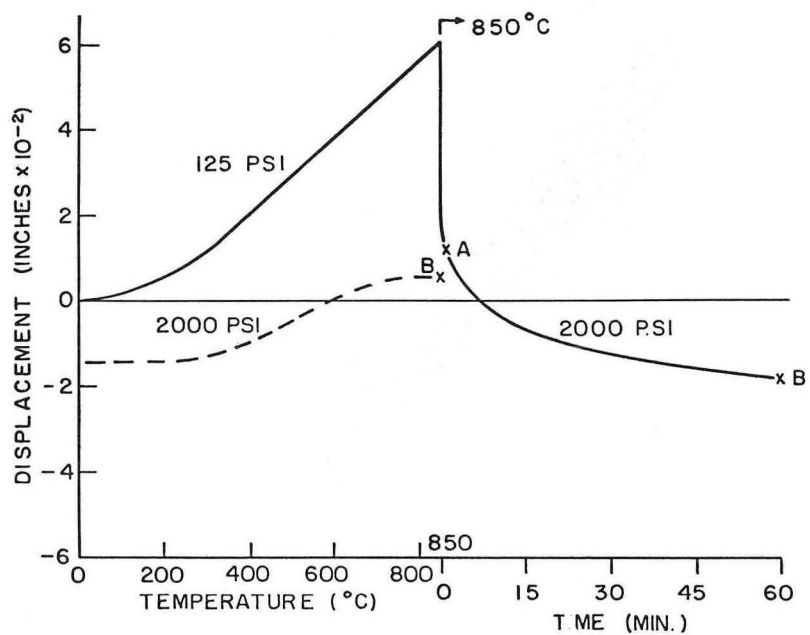


B

C

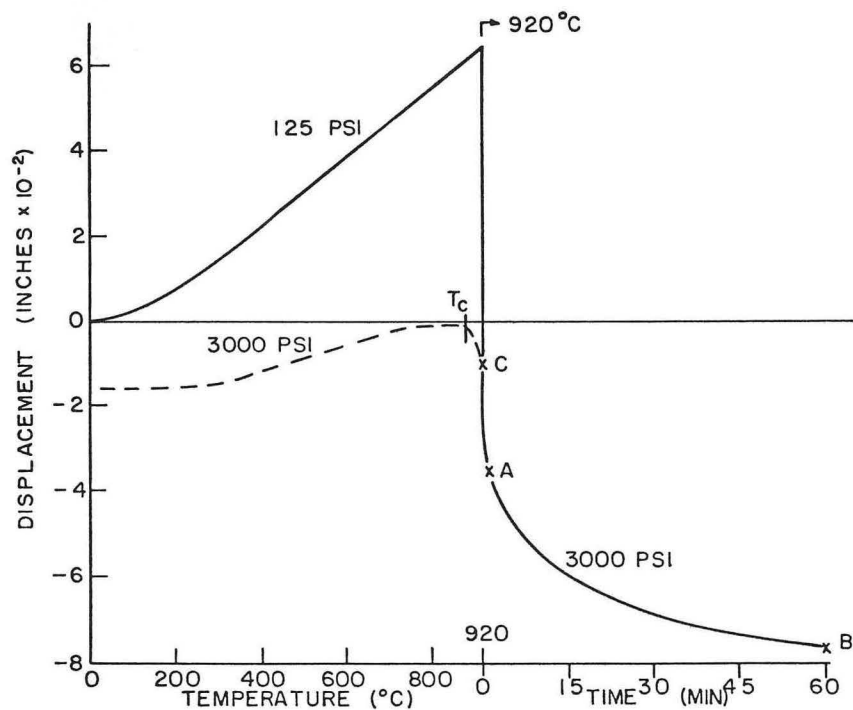
ZN-4178

Fig. 9. CaF_2 spherical powders hot pressed under a constant load of 3000 psi and heated to 1000°C . (a) Load applied parallel to the section. (b) Load applied normal to the section. (c) Load applied parallel to the section.



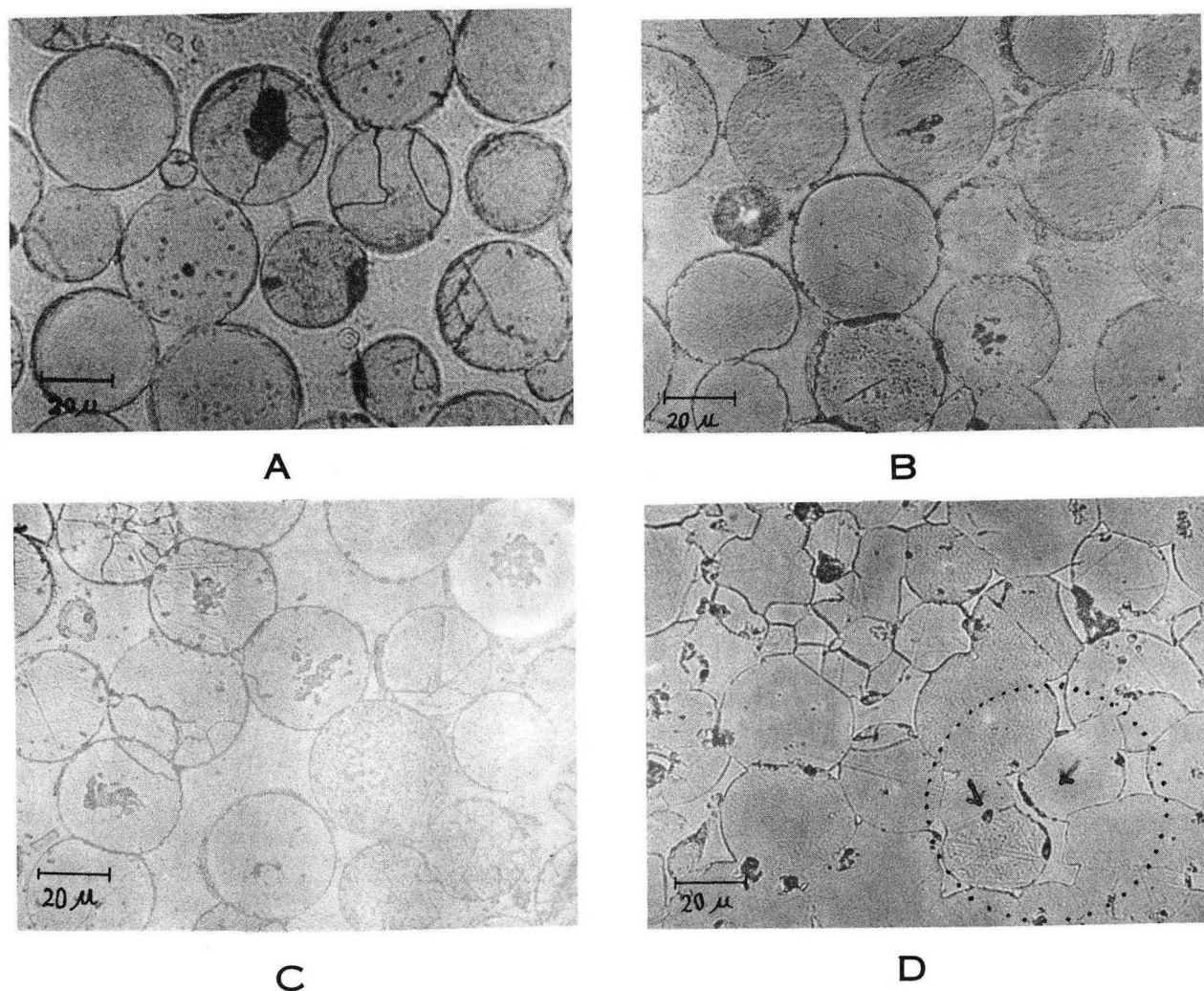
MU-33517

Fig. 10. Displacement-temperature and displacement-time curves for CaF_2 hot pressed at 850°C under 2000 psi applied load.



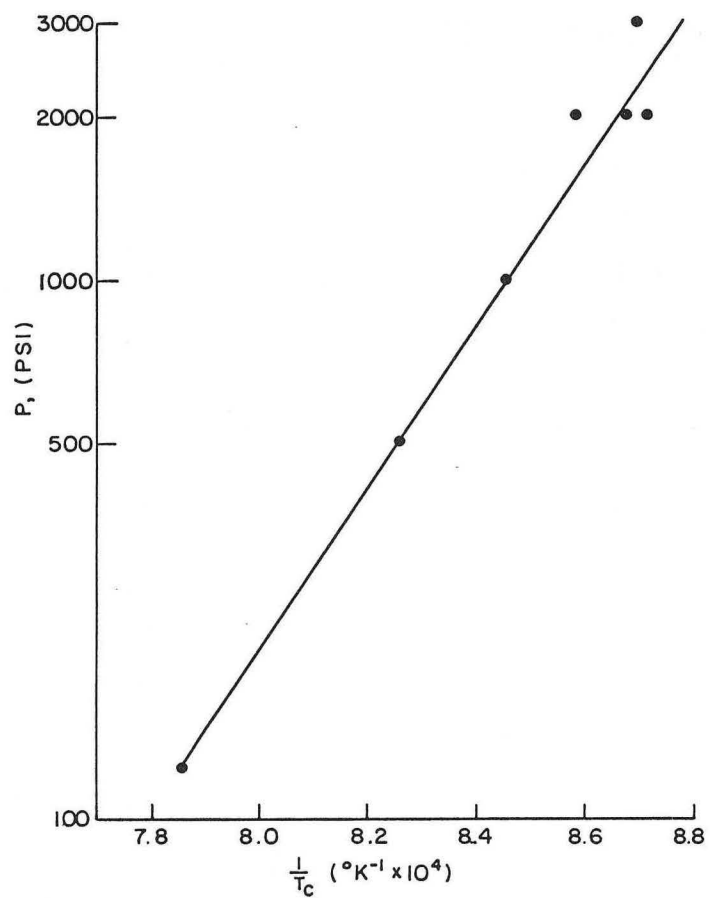
MU-33518

Fig. 11. Displacement-temperature and displacement-time curves for CaF_2 hot pressed at 920°C under 3000 psi applied load.



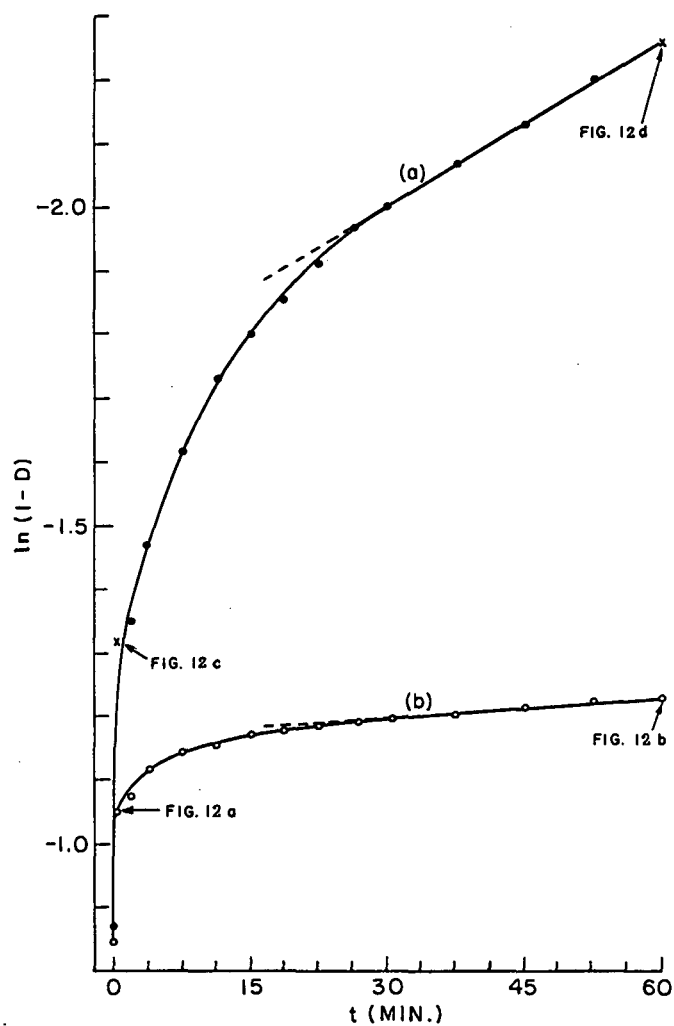
ZN-4179

Fig. 12. CaF₂ spherical powders hot pressed under constant temperature with a rapidly applied load at temperature. (a) Load-2000 psi; temperature-850°C; load applied 40 sec. (b) Load-2000 psi; temperature 850°C; load applied 1 h. (c) Load-3000 psi; temperature-920°C; load applied 40 sec. (d) Load-3000 psi; temperature-920°C; load applied 1 h. All sections parallel to the load and etched.



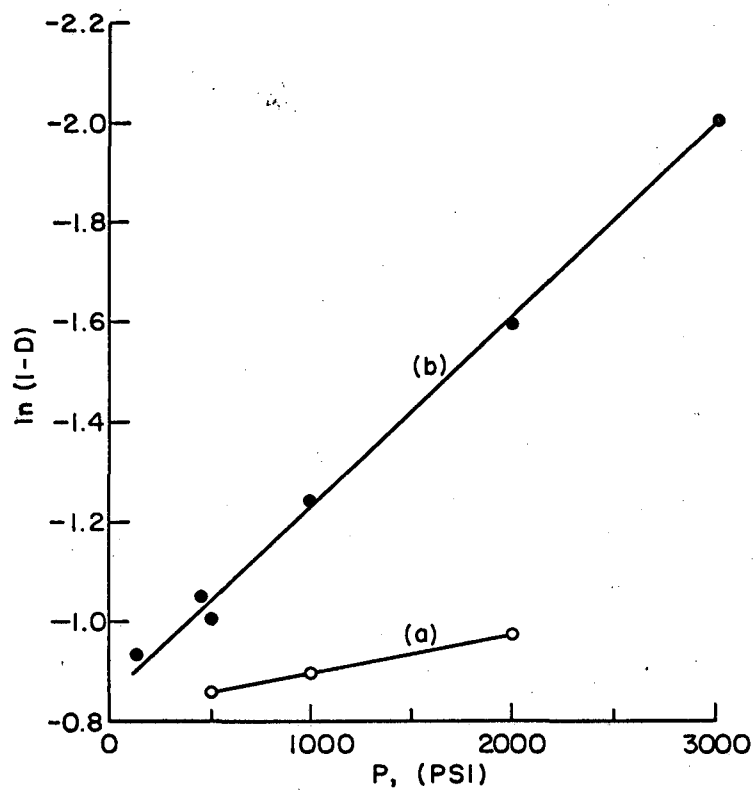
MU-33519

Fig. 13. Relationship between the applied load in hot pressing and the temperature for transition between Regions II and III (Fig. 4).



MU-33520

Fig. 14. Density-time curves for hot pressing with loads rapidly applied at temperature. (a) 920°C and 3000 psi applied load. (b) 850°C and 2000 psi applied load.



MU-33521

Fig. 15. Effect of hot pressing pressure on final density for specimens heated at a constant rate of 40°C per minute to the maximum temperature and quenched. (a) Heated to 700°C . (b) Heated to 1000°C .

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