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COMPACTION OF LITHIUM ALUMINATE AND LITHIUM SILICATE POWDERS BY HIGH ENERGY RATE COMPACTION AND HOT PRESSING

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PRELIMINARY REPORT

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SUMMARY

Lithium aluminate and lithium metasilicate powders compacted to 82% of theoretical density by "hot" pressing at 119,000 psi and at either 500°C or 800°C were well bonded. The same powders pressed at room temperature at 119,000 psi had densities of 75 to 77%, and were not bonded. Lithium aluminate powders pneumatically impacted at 800 and 1200°C under pressures from 115,000 psi to 350,000 psi were well bonded. Approximately 97% of theoretical density was attained at 345,000 psi and 1200°C. Relationships between compacted density and punch pressure, or energy, are plotted.

INTRODUCTION

This study was initiated in order to 1) determine the densities achievable for lithium aluminate and lithium silicate by hot pressing or high energy rate compaction, 2) provide samples of varying high densities for small scale irradiation testing, and 3) provide densified materials for vibratory compaction studies.

The starting powders used were those commercially available from Lithium Corporation of America. Both had average particle sizes near 100 microns. Both powders were incompletely calcined, having a small percentage of unreacted alumina or silica remaining. Crystallographically,

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the lithium silicate was 100% lithium meta silicate, Li_2SiO_3, and the lithium aluminate was composed of approximately 50% α-$LiAlO_2$ and 50% γ-$LiAlO_2$. The powders are more thoroughly characterized in BNWL-CC-464.(1) The total lithium content of the lithium aluminate powder was 8.3 wt%. This is substantially lower than the stoichiometric content of 10.5 wt% Li. The theoretical density for stoichiometric lithium aluminate, 2.62 gms/cc, was used in calculating compacted densities as a per cent of theoretical, rather than making some arbitrary adjustment for non-stoichiometric lithium content.

Pressures used in this study were limited by the safe operating conditions of the available tooling. The optimum temperatures for hot pressing are near the sintering temperature of the materials being pressed. For lithium aluminate the sintering temperature is around 1300°C and for lithium silicate it is approximately 1000°C. In order to avoid excessive reaction between the powder and the can, hot pressing temperatures of 500 and 800°C were chosen along with a maximum pressure of 119,000 psi. The powders were loaded in mild steel cans and pressed in a tool steel container after a two hour preheat.

Maximum pressure for pneumatic impaction was limited by the strength of the carbide punches to 400,000 psi. A preheat temperature of 1200°C was chosen because it is just below the sintering temperature of lithium aluminate, and the results of the combined temperature and pressure would not be influenced by any significant amount of sintering during preheat. The powders for pneumatic impaction were loaded in stainless steel cans and evacuated during preheat. Two cans of lithium aluminate were impacted at 800°C for comparison with that hot pressed at 800°C.

The data presented in this report for hot pressing can, and should be, extended to temperatures in the sintering ranges of the respective powders using the same techniques applied here. Such studies may disclose the possibility of forming high density (roughly 95% dense), high strength, finished compacts of low cost by hot pressing methods. Some work should also be done with the somewhat classical hot pressing techniques using graphite dies and temperatures nearer the sintering temperatures of lithium aluminate and lithium silicate.

Generally, hot pressing and pneumatic impaction have several advantages over conventional cold compaction followed by sintering in the preparation of finished compacts(2):

1. Higher density
2. Better thermal schock resistance due to reduced porosity
3. Elimination of some powder preparation steps such as calcination, grinding, grading, or binder addition.

Disadvantages of the processes for producing finished compacts are:

1. Much slower press throughput
2. Increased die costs
3. Generally increased unit cost.

EXPERIMENTAL DETAILS

Hot Pressing

Powders were loaded into mild steel cans 2.98 inches OD by 2.715 inches ID by 3.0 inches high. Packing density achieved was a "tap" density. The cans were vented at the top end to allow escape of moisture or other volatiles generated during the two hour air preheat. The preheated cans were then pressed in a cold 3.050 inches diameter die at 420 tons (119,000 psi) with aquadag lubricant. The ram pressure was allowed to build up until 420 tons was reached and then released. The can was then ejected from the die and allowed to air cool.

Bulk density of the compacted powder was calculated from the bulk density of the hot pressed can of powder and the original can weight.

A data summary is given in Table I. For the sake of comparison, several pellets of each powder were pressed at 119,000 psi at room temperature. The powder had no prior preparation, and only the die walls were lubricated with lithium stearate.

TABLE I

Hot Pressing Summary

Material	Preheat Temp.	Tapped Density-% of 2.62 gms/cc	Pressed Density
$LiAlO_2$	20°C	---	77.3%
$LiAlO_2$	500°C	51.5%	81.3%
$LiAlO_2$	800°C	52.3%	81.7%
Li_2SiO_3	20°C	---	75.1%
Li_2SiO_3	500°C	51.6%	82.6%
Li_2SiO_3	800°C	53.2%	82.6%

High Energy Rate Compaction of Lithium Aluminate

Lithium aluminate powders were "tap"loaded into stainless steel cans 2.50 inches OD by 2-3/8 inches ID by 4 inches high. The top, containing means for evacuating the powder mass through a stainless steel filter, was welded on. Each can load of lithium aluminate was then preheated for 45 to 315 minutes at either 800°C or 1200°C while being evacuated to 30 to 80 microns. After preheat the evacuation tube was sealed and snipped off. Immediately thereafter the can was charged into the press container and compacted. The compacted can of powder was then ejected from the die container and water quenched.

A Dynapak Model 1220F, tooled with carbide punches, was utilized for this work. Punch pressure was controlled roughly by the fire pressure of the machine which is proportional to the energy imparted to the canned powder and can. The maximum allowable punch pressure was limited by the carbide punches to 400,000 psi. (Tool steel punches can be used to 300,000 psi.) Punch pressure was measured by a thin wire resistance strain gage mounted on the side of the punch. The signal from the strain gage was fed to a recording oscilloscope with circuitry that changed strain to stress.

Table II contains a data summary for the eight cans of powder impacted. Figure 1 displays compacted density as a function of punch pressure. The straight lines indicate that at least 98% of theoretical density is obtainable at 400,000 psi with a 1200°C preheat. When the preheat is lowered to 800°C, the highest density obtainable at 400,000 psi is approximately 93%. If punch pressures are limited to 300,000 psi by tooling, densities of 93% and 87% are obtainable at 1200°C and 800°C respectively. Further increases in preheat temperature may result in extensive chemical reaction between the stainless steel and the lithium aluminate. At 1200°C the reaction layer was in the order of 1 to 10 mils thick.

The energy imparted to the canned powder during compaction is directly proportional to the fire pressure of the machine. Figure 2 is of interest because it illustrates the fact that the compacted density is a function of the energy imparted to the canned powder and is independent of preheat temperature. Simply stated, one may directly control the compacted density simply by choosing the correct fire pressure. This last statement is based on the assumption that the green density of the powder and the powder to can mass ratio are held constant. Any green density increase will naturally result in a higher compacted density. Since punch

TABLE II

High Energy Rate Compaction Data Summary

Run No.	Fire Pressure Psi	Punch Pressure Psi	Preheat Temperature & Time	Evacuation Pressure Microns-Hg	Green Density*	Compacted Density* Bulk	Hg Poros.	Krylon Coat
A	150	115,000	1200°C - 105 min.	30	47.0	78.3	77.8	78.3
B	200	150,000	1200°C - 60 min.	30	46.4	78.3	80.8	77.5
C	300	182,000	1200°C - 60 min.	30	47.0	84.0	85.6	85.2
D	450	285,000	1200°C - 45 min.	30	46.4	88.2	91.7	89.9
E	500	292,000	1200°C - 90 min.	50	47.3	92.8	92.3	92.8
F	550	345,000	1200°C - 315 min.	60	48.1	95.4	98.4	97.1
G	300	235,000	800°C - 85 min.	80	48.1	84.1	83.6	82.5
H	450	350,000	800°C - 95 min.	80	47.7	88.5	91.3	89.7

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* % of 2.62 gm/cc

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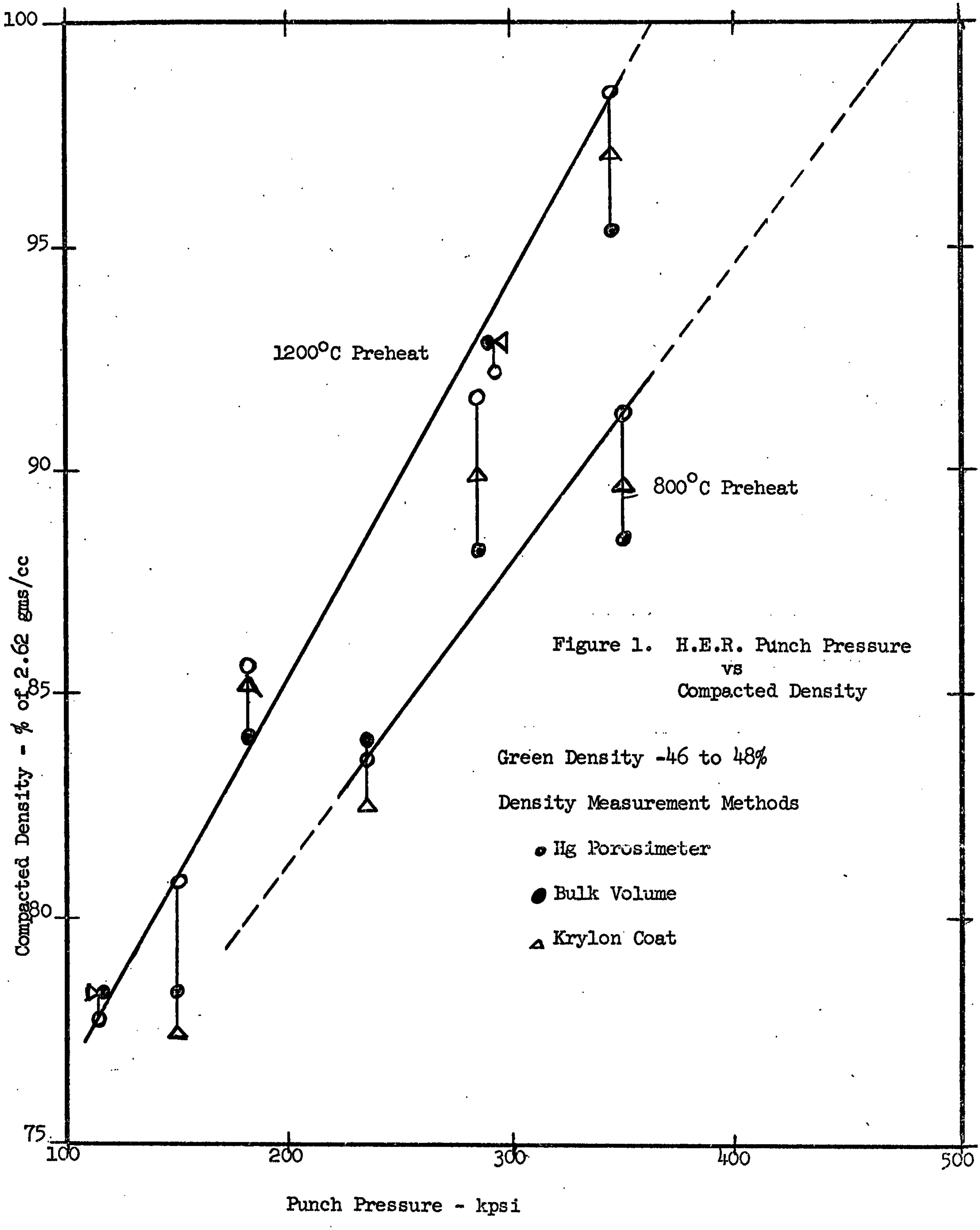

Figure 1. H.E.R. Punch Pressure vs Compacted Density

pressure increases with decreasing preheat temperature (at constant compacted density and energy)(see Figure 1), one must also choose a preheat temperature sufficiently high to limit punch pressures to safe levels. Carrying this reasoning further, compacted density may be increased when punch pressure is the limiting factor by increasing preheat temperature.

The bulk density of the compacted powders was measured by three methods:

1. Bulk Volume - Calculated from bulk density of compacted, canned powder and original can weight.
2. Mercury Porosimeter (Aminco-Winslow) - Volume of mercury displaced by a preweighed chunk of the powder.
3. Krylon Coat - A preweighed chunk of the compacted powder, coated with Krylon to prevent water absorption, weighed in water.

There was no close agreement between the three density measurement methods. The values from the mercury porosimeter appear to be more consistent, therefore, the lines drawn through the data in Figures 1 and 2 were drawn using data from this method only.

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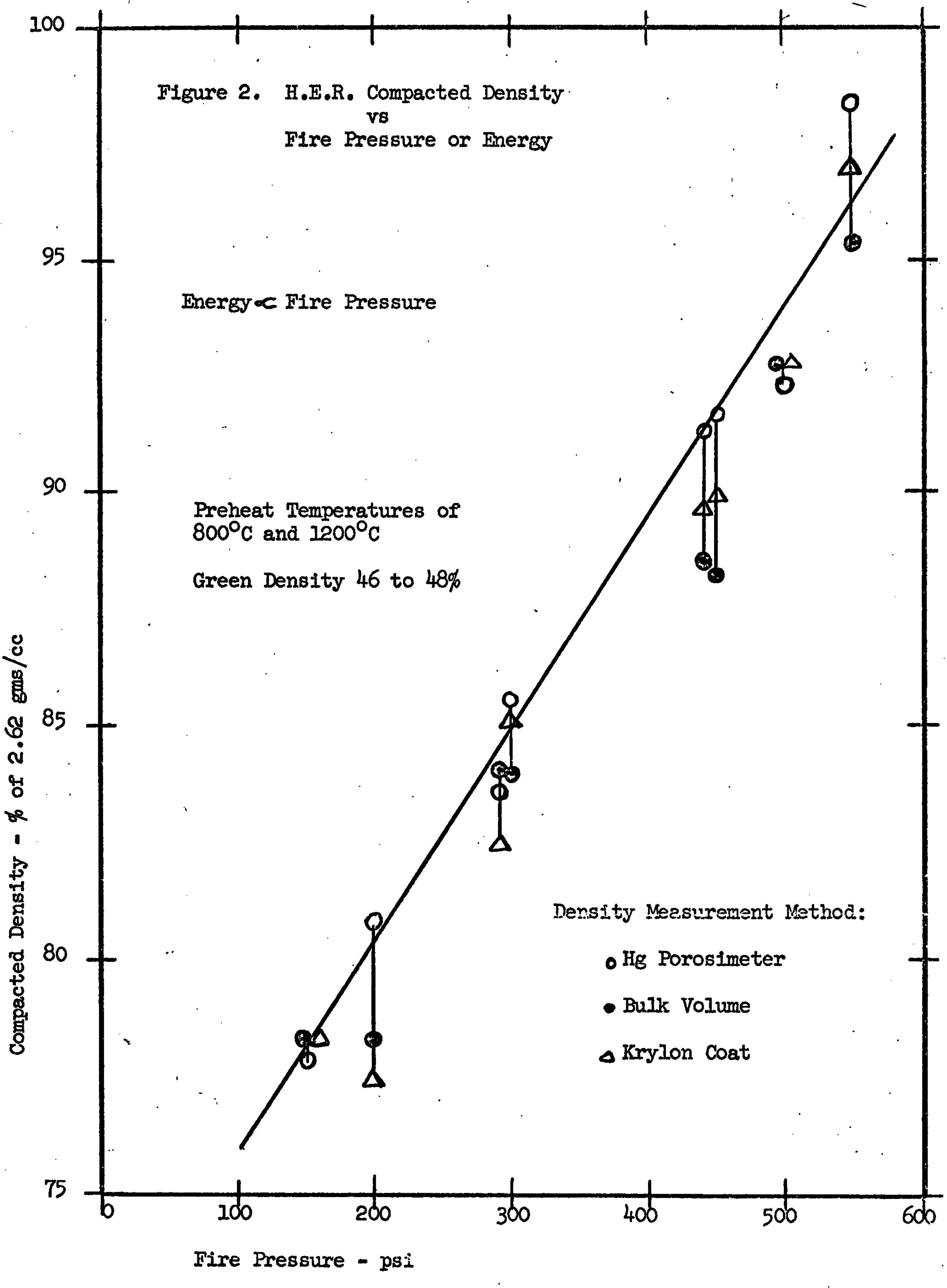

Figure 2. H.E.R. Compacted Density vs Fire Pressure or Energy

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