Center: Consortium for Materials Development in Space
The University of Alabama in Hunstville (UAH)

Task Name: **"Powdered Metal Sintering and Infiltration"**

Industrial Participant: Teledyne Brown Engineering
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The University of Alabama in Huntsville
James E. Smith, Jr.

Annual Report: September 15, 1986 to September 14, 1987

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Center: Consortium for Materials Development in Space
The University of Alabama in Hunstville (UAH)

N88-11870

Task Name: **"Cast Iron Freezing Mechanisms"**

Industrial Participant: Deere and Company
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Annual Report: September 15, 1986 to September 14, 1987

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Introduction

These two tasks are being worked by the same investigator in his metals laboratory using essentially the same equipment. Consequently, they are being documented in one report rather than in separate reports.

This task has focused on liquid phase sintering and infiltration studies of refra(ory metals and metal composites. Particular emphasis is being placed on those powdered metal compacts which produce liquid alloys on sintering. For this class of materials, heating to a two phase region causes the constituent

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components to react, forming an alloy liquid which must wet the solid phase. Densification is initially driven by the free energy effects which cause rapid rearrangement. Further densification occurs by evaporation and condensation, surface diffusion, bulk flow, and volume diffusion.

In unit gravity, sedimentation causes stratification within the sample that results in a non-uniform coarsening of microstructure. Sintering, in a reduced gravity environment, should produce a smaller grain size, a more uniform microstructure possibly with less voids or defects, and improved mechanical properties.

Many of the most interesting liquid phase sintered materials generally include a refractory metal or ceramic which is sintered or infiltrated at temperatures in the range of 1000 - 1800°C. this requires a furnace capable of melting such materials while maintaining an inert or slightly reducing atmosphere during the sintering or infiltration operation.

Potential uses for liquid phase sintered products include bearings, magnetic materials, electrical brushes and contact points, cutting tools, irregular shaped mechanical parts for high stress environments, and possibly new and improved catalysts for chemical production. Space processing in the areas of uniform cutting tools, magnetic materials, and supported inter-metallic alloy catalysts offer the greatest research and development potential.

Furnace Completion

The construction and retrofit of a TEM-PRES corporation high temperature electric furnace has been completed. Only minor technical problems, as will be described below, remain unresolved. This furnace along with numerical modeling of both isothermal and directional solidification modes, has increased the overall understanding of furnace design, application, control, and integration for space processing systems.

Updated Furnace Control Program

An updated control program was developed for the high temperature sintering furnace. This program was developed to provide complete interactive or automatic control of the furnace as needed. Since this furnace works at the upper margin of electrical furnace performance, an adaptive computer control scheme was required for rapid heating during initial stages, with more critical control as the furnace achieves its final operating temperature. The program writes a complete experimental history of the furnace operating parameters and processing temperatures to computer disk.

Initial Furnace Performance Testing

The furnace o-ring seals, failure of which was discussed in a previous report, were replaced and tested, achieving the desired performance. The furnace was heated to 250°C to test the control program and power systems. To date, we have limited the testing to operating temperatures below 300°C. To operate the furnace at temperatures above 300°C, a hydrogen reducing environment must be used or the molybdenum heating coils will oxidize, becoming unusable. For safe operation approximately 50 cc/min of 5% H_2-95% N_2 or 5% H_2-95% He gas mixture must flow over the coils. Attempts were made to find rotameters with small capacity to mix pure H2 and inert carrier gases. The only commercially available flowmeters found capable of providing the hydrogen rates were mass flowmeters. Due to the high cost of these flowmeters, a decision was made to use premixed gases. This required a slight modification of the gas regulation system to permit the use of premixed gases. With this modification now complete, tests will be conducted near the furnace limits.

Several hardware and software modifications were accomplished to improve both furnace temperature control and positioning. A linear bearing guide was designed, constructed, and installed to improve crucible alignment within the furnace, increase the maximum furnace translation rate, and eliminate potential damage to the crucible during furnace translation. Several moderate temperature experiments were conducted at temperatures less than 800°C to test the validity of the temperature control routines. One sintering experiment was attempted on a precompacted $A\ell$ sample. It was determined after this experiment that additional data will be required on both positioning and temperature profiles. To expand the number of temperature measurements, preliminary testing of an additional data acquisition board and signal conditioner was performed. The data acquisition board was tested at a communication distance of 26' which is 16' beyond the communication distance recommended by the manufacturer. The test was successful and will permit the installation of up to 16 additional thermocouplers. The temperature measurement, furnace control, and positioning programs are being combined into a system program.

Development of a New Die and Pre-Sintering Environmental Chamber

A pre-sintering environmental chamber was designed and built to permit powdered metal compact preparations under vacuum or a reducing atmosphere at temperatures higher than the commercially available system. The white iron powder studies to be conducted for John Deere, Inc., along with several other materials currently being considered, require either vacuum or a reducing environment to reduce oxidation during preparation. The pre-sintering environmental chamber, shown in Figure 1, encloses the test cylinder in a controlled environment. A high temperature resistance heater, capable of temperatures to approximately 1000°C, surrounds the test cylinder body. To reduce the heat loss in the axial directions, cartridge heaters were installed in both the base of the chamber and plunger extension as shown in Figure 1. The upper operating temperature of this prototype configuration will be limited to approximately 600°C since the chamber base was constructed from aluminum. To extended the upper temperature range, a base made of stainless steel may be needed. Temperature measurement and control techniques along with operating procedures are currently being developed for this unit.

A new test cylinder, with a 0.25 inch diameter bore, was constructed to provide samples for testing in the Rapid Melt/Quench Furnace, currently under development, aboard the KC-135. An initial plunger made of 316 stainless steel was tested. This plunger would not tolerate the stress created at 5000 lbs applied pressure. A modified plunger made of case-hardened steel corrected the problem. With the development of the Rapid Melt/Quench Furnace, the test cylinder, and pre-sintering chamber, we now have the necessary elements to pre-screen samples in a low gravity environment.

The 0.25 inch bore test cylinder along with the 2 cm bore and 1.25 inch bore test cylinders permits the preparation of 0.25 inch, 2 cm, and 1.25 inch diameter samples up to 7 cm long. These test cylinders can be used in the environmental chamber to prepare powder metal samples at sintering pressures exceeding 25 tons, temperatures up to 600°C, and vacuum or reducing atmospheres, for use in the high temperature furnace.

Further, during the next reporting period, we hope to complete the sample pre-sintering experimental apparatus, making refactory sintering experiments possible.

Pre-Sintered Sample Preparation

Powder metal sample preparation procedures have been developed to insure sample uniformity. The powder metal is first sieved to separate a specific particle size range. The test cylinder body, plunger, and base are then cleaned using a caustic solution and rinsed with distilled water. The parts are then hot coated with a sodium sterate and water solution to help prevent galling during the pressing operation. The sterate solution was prepared by mixing 18 gm of sodium sterate with 500 ml water while heating to 85°C to dissolve the sterate. The press parts are immersed in the solution and cured in air prior to use. When cured, the pressing parts have a thin film of sodium sterate which melts at high temperatures to lubricate the die.

For each material studied the pre-sintering temperature, applied pressure, and pressing time must be determined experimentally. The low gravity reactive sintering studies will a require porous refractory metal skeleton of tungsten or tungsten-carbide, which will be coated with and in-situ formed intermetallic alloy. The uniformity of the pre-sintered skeletal material is determined by measuring the material's void fraction as a function of position. To determine the void fraction distribution of the pre-sintered compact a Reichert metallurgical microscope has been integrated with New Brunswick Scientific Co., Inc. Biotran III imaging system, as shown in the photograph in Figure 2.

The Biotran III is a precision instrument designed to automate particle counting and area measurements in an image plane. A high resolution vidicon TV camera system is coupled to a 4-digit numerical display to count particles or any distinct separately bounded group of positive or negative image areas in a field of view. The Biotran III then calculates the ·rcentage ratio of either positive to negative or negative to positive areas in the field of view. To fully utilize the Biotran III to experimentally measure the void fractional area of a powder metal compact, the contrast between the voids and polished metal surface must be enhanced. This is accomplished by blacking the voids to enhance contrast.

To test the integrated imaging system, several 2 cm diameter aluminum compacts were pre-sintered using the environmental chamber at room temperature. Three 16 gm Al samples were pre-sintered at 10,000 lbs applied pressure for 30 minutes, 1 hour, and 2 hours, respectively. The 30 minute and 1 hour cylindrical samples were cut in half lengthwise using a metallurgical saw. The samples were polished using standard metallurgical techniques, coated with a fine particle black paint, and repolished to retain the paint within the pores. The Reichert metallurgical microscope and Biotran III imaging system was then used to determine the porosity of the sample as a function of position. A photograph of the aluminum sample as imaged by the above system is shown in Figure 3. Darken areas, outlined in white by the image processor are voids. The Biotran III ratios the two image fields as discussed above. This ratio was then used to determine the void fraction. A representative plot of porosity as a function of position for the 30 minute pre-sintered aluminum sample is shown in Figure 4. Samples were found to be more compact near the plunger end of the sample with this difference diminishing as the sintering time was increased. A study to determine optimal pre-sintering conditions is currently underway.

Future Plans

With furnace design, interfacing, and operational verification virtually completed, initial testing on lower melting sintered materials such as Al-Fe and Cu-Si will continue. Once the controls and measurement systems on the environmental chamber are completed, the John Deere materials along with the reactive sintering of Fe-Ti in a refractory matrix of tungsten and tungsten-carbide will be studied.

In coming months, an axial and radial profile of the furnace will be obtained at several operating temperatures. These profiles will be measured in all three zones, namely the isothermal zone and the cold over hot and hot over cold gradient zones. Once mapped, a better understanding of the sample operating conditions, gradients, etc. will permit more rapid sample analysis. We will continue our test on AL until we can confirm these operating conditions.

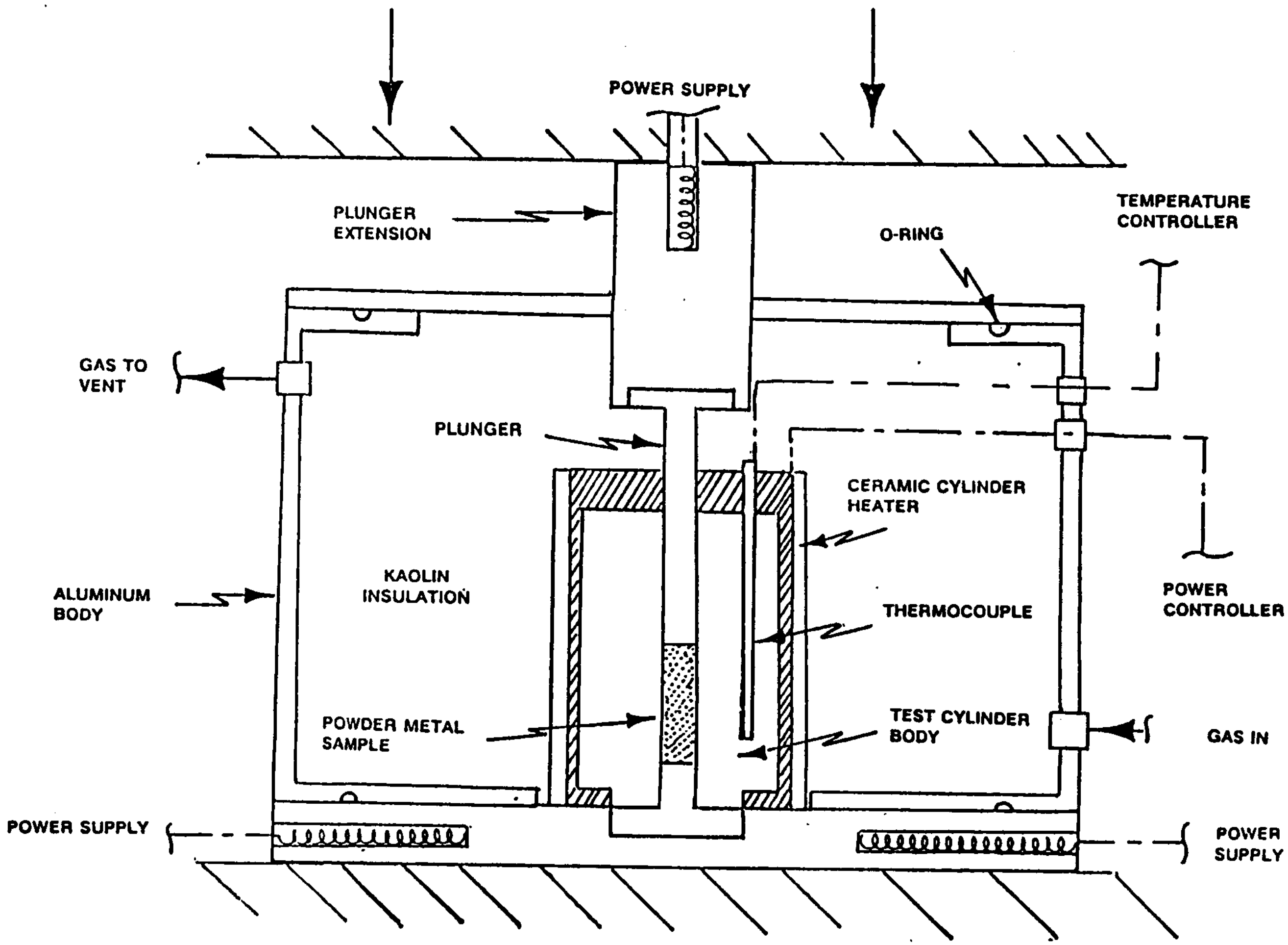

Figure 1. Detailed Drawing Showing the Various Components of the Pre-Sintering Environmental Chamber Currently Under Development.

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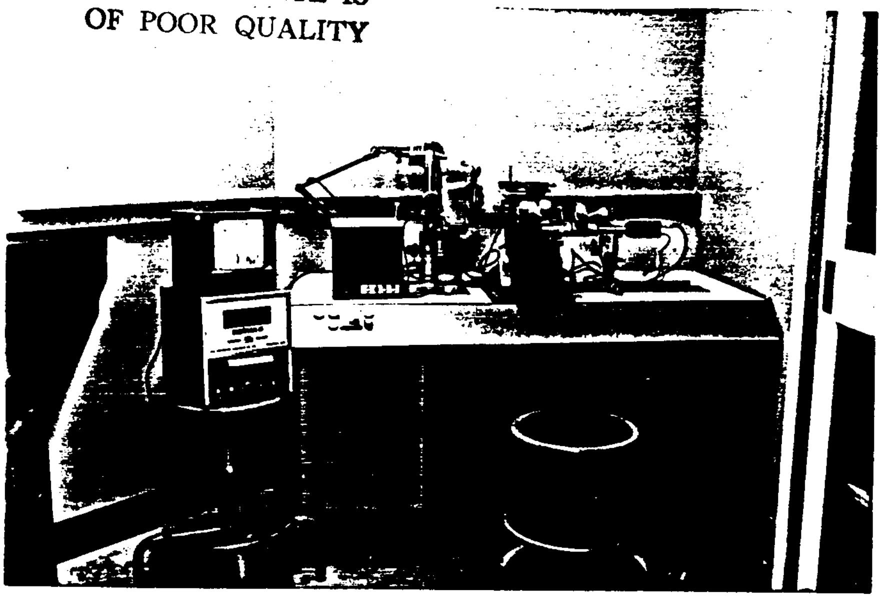

Figure 2. Photograph of the Reichert Metallurgical Microscope Integrated with the Biotran III Imaging System.

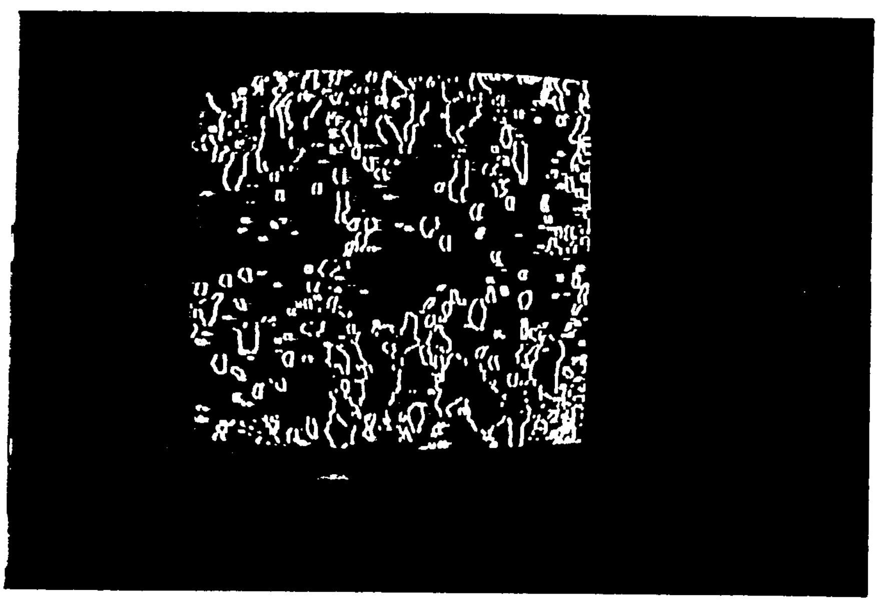

Figure 3 Photograph of the Biotran III Image of a Pre-Sintered Aluminum Sam 'e.

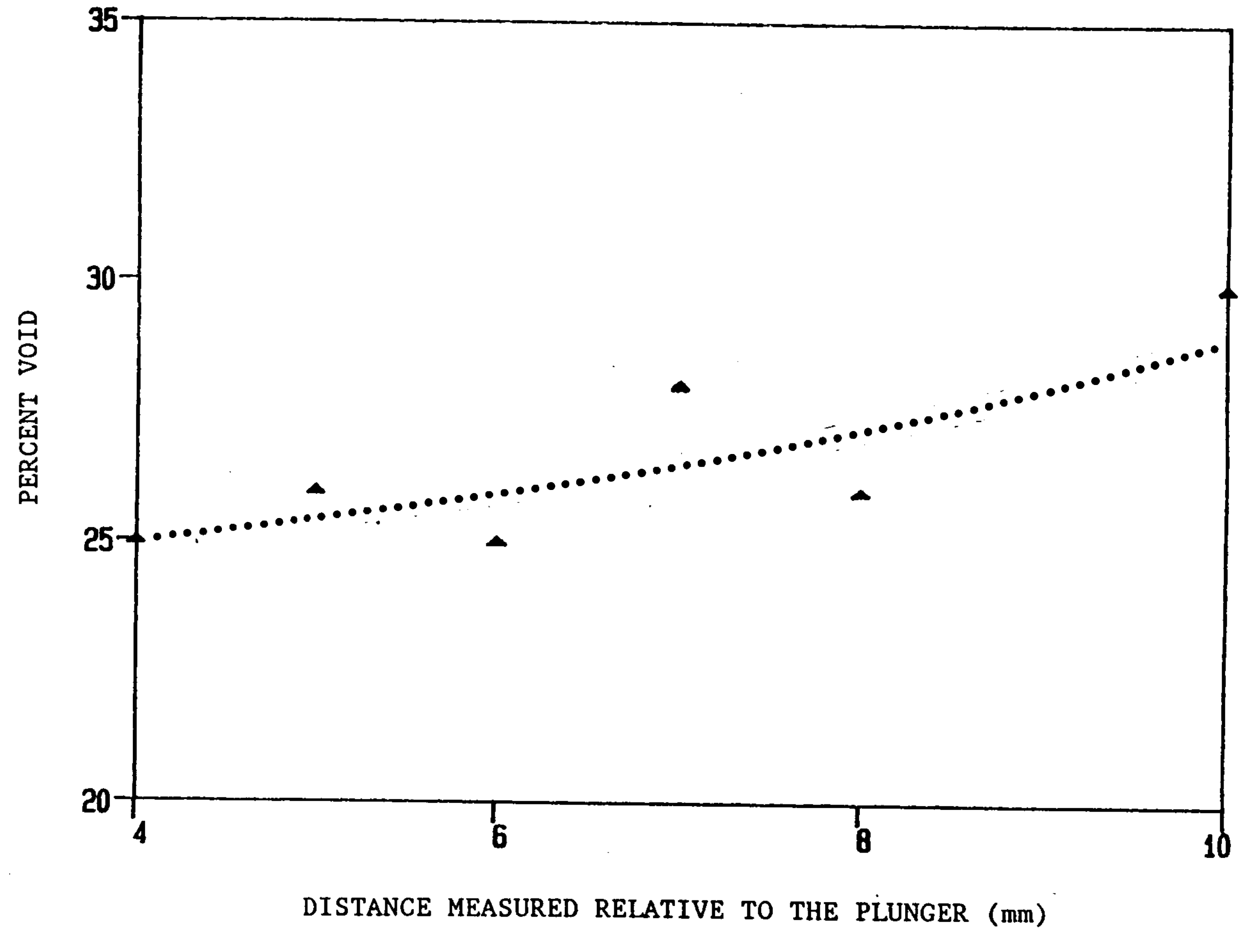

Figure 4. Void Fraction Distribution of a Pre-sintered Aluminum Sample.