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AN INVESTIGATION OF THE SLIP-CASTING
MECHANISM AS APPLIED TO STAINLESS
STEEL POWDER

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February 23, 1956

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ABSTRACT

The slip-casting technique, which is successfully employed in the ceramic industry, was utilized to produce sintered stainless steel components experimentally. The procedure used is described, along with an evaluation of the physical and mechanical properties of the samples produced. Specimens were made with properties comparable to those of wrought stainless steel and of cold pressed and sintered steel powder.

I. INTRODUCTION

Suspensions of solids in a liquid media have been used since the first pottery pieces were produced early in our civilization. The ceramics industry today still uses these basic materials in the production of various components. The oldest method of working clay and water suspensions consisted of forming plastic clay by hand to form a brick or other simple shape; the potters wheel was used to form hollow shapes, such as vases. More recent developments utilize a plastic clay, thicker than that which can be worked by hand, and which instead is forced through a die to form a continuous block or tube. These pieces are cut to desired length and fired at elevated temperatures to drive off the water and to consolidate or densify the product.

Clay and water mixture of a thin consistency, which cannot be formed in the manner described above, can be fabricated into various types of hollow or solid pieces by a casting process. This method consists essentially of casting the water and clay mixture, which is known as a slip, into a plaster mold, permitting the slip to dry or set as the water is absorbed by the porous mold, then followed by high-temperature firing to impart the final physical and mechanical properties of the material.

In powder metallurgy as well as in ceramic processes, solid components can be produced from pulverized materials which are usually compacted with no liquid present, although in some instances, a solid type of binder or lubricant may be used. After pressing,

samples are then sintered at elevated temperatures to densify the part and to improve its properties.

The use of steel dies for compacting make it difficult to produce pieces which are hollow or have projections or other characteristics which would make it difficult to eject from the die. A method of slip casting for fabricating intricately shaped objects from refractory powders is described in reference 1. Slips with a high solid content were used in the investigation of reference 1, and the feasibility of the process was demonstrated.

The purpose of this present report is to present the results of an investigation to determine the feasibility of applying slip casting techniques, using low metal content slips to powder metallurgy materials. Solid samples were prepared from stainless steel powders, in order to permit comparison of properties of this product with that produced by compacting and sintering.

II. APPARATUS AND PROCEDURE

A. Molds

The prime requisite of the mold in this application, as in any casting process, is to provide the desired contour to the cast material while in the plastic state and maintain support until the cast material has sufficient strength to retain the induced shape. Slip casting of metal powders differs from conventional casting in that the prepared slip contains a considerable amount of moisture which must be removed before stripping. Removal of this moisture can be accomplished by evaporation or by mold

absorption. Also, the slip is prepared and cast at room temperature. Consequently, the requirements for a satisfactory mold material were: sufficient mold strength for providing support and shape, ability to absorb moisture, ease of preparation, ease to release compact after drying, and inexpensiveness.

Plaster of Paris appeared the most logical material for this application. Investigations were conducted on the preparation of plaster molds, with considerable attention devoted to the effects of such variables as water content, mixing time, and mixing speed on mold appearance. The schedule which appears below produced a mold of extremely sharp edges, and also one that was void of air bubbles on the surface of the cavity.

Mold Schedule

<u>%</u>	<u>Constituent</u>
55	Red Top White Gauging Plaster
45	Tap Water (25°C)
Mixing time 3 minutes - Mixing speed 450 rpm	

Other gypsums, such as Fisher Scientific Company's calcium sulphate, U. S. Gypsum Company's Hydrocal B-1 were also evaluated. The molds produced from these materials had properties similar to those of the white gauging plaster.

Additives of 0.5% silicon dioxide and 0.5% graphite were also tried in an attempt to enhance the plaster mold release qualities.

No particular improvement occurred as a result of these additions. Additives in quantities greater than this tended to provide particles which served as nuclei for air bubbles which were undesirable.

The actual mold preparation technique consisted of coating the wooden shell and metal insert shown in Fig. 1 with a soap solution which was then allowed to air-dry. A coating of this type was found to be extremely effective with respect to mold release. The metal insert was then placed between the shells and the prepared plaster was poured. Thirty-minutes after pouring, the plaster mold was removed. The finished mold was allowed to air-dry at room temperature for forty-eight hours. Heat applications, of course, would reduce the mold drying time considerably.

To further improve the release characteristics of the plaster mold itself, a coating of soap solution was applied. Clear lacquer was also tried and found to be equally as good as the soap solution with respect to release and somewhat more substantial with respect to endurance.

B. Slip Blending

The essential components of the slip investigated in this study were stainless steel powder (302-B), ammonium alginate for bonding the powder particles, DuPont's Elvanol Grade 72-60 (polyvinyl alcohol) for elasticity, and water for fluidity.

After considerable investigation as to the proportions of each of the constituents, mixing speed, and mixing time, a schedule was evolved which rendered a slip which was fluid enough to

Fig. 1 Wooden Shell, Brass Insert and Plaster Mold.

Plate No. 432-2

pour easily and yet firm enough to retain its shape after stripping from the mold. The composition and mixing conditions arrived at are as follows:

74.55% Stainless steel powder

24.85% Tap water (room temp.)

0.35% Ammonium alginate

0.25% Elvanol

Mixing speed 1100 rpm

Mixing time 2 hours

In order to improve slip properties of fluidity and stripped-slip strength a "hot-slip" was also evaluated. This was prepared using the above constituents in the proportions given, except that the slip was heated to 60°C while mixing and then poured. Upon stripping, a relatively hard slip was obtained which was considerably more cohesive than the "cold slip" but consisted of several broken sections rather than one complete section. Repeated trials yielded similar results and it was not possible to cast a good, sound "hot-slip" section in this manner.

The use of commercial corn starch as a replacement for the binder, ammonium alginate, was also investigated. An equivalent weight of corn starch was initially substituted, but a very stiff slip, which would not pour was obtained. The fluidity of the slip was appreciably affected by decreasing the starch content.

C. Method of Casting

The methods of casting investigated were: drain casting, which, as the name implies, consists of filling a mold and allowing the slip to drain into the mold cavity; vibration, in which the mold is filled and vibrated; and centrifugal casting. Comparison of the three methods was accomplished by filling three molds to excess and subjecting the molds to the conditions of each process. The levels were then qualitatively compared. Centrifugal casting appeared to fill the mold with the greatest amount of slip. This was, therefore, used throughout the casting procedure. The centrifuge with pouring tube is shown in Fig. 2.

After casting, the molds were allowed to settle for forty-eight hours, during which time some of the inherent moisture in the slip disappeared. Stripping was then accomplished by gently hand rolling the as-cast sample out of its mold.

D. Evaluation

1. Density

Green density of the as stripped slip as determined by the usual Archimedes' technique was difficult to obtain primarily because of the fragility of the slip. Considerable fractures resulted when attempts were made to lacquer the slip, and hence density determinations were obtained from sections of the slip only. In no instance was it possible to obtain a density value for the entire slip. Densities from sections averaged 3.2 g/cc. Considerable variation was encountered, chiefly because of the inherent moisture in the slip.

Fig. 2 Centrifuge Used for Casting Showing Plaster Mold and Pouring Spout in Casting Position.

Plate No. 432-3

2. Sintering

The as-stripped sample was placed in a boat formed from a refractory brick. This assembly was then placed in the front portion of the hot zone so as to allow the sample to outgas. After several minutes had elapsed, the sample was moved into the hot zone. A temperature of 1320°C , which is approximately 65°C below the melting point, was found to be the optimum sintering temperature. A reducing atmosphere of hydrogen with a dew point of -72°C was used. Sintering for sixteen-hours at these conditions produced samples which were relatively clean and which were densified to 6.4 g/cc (80% theoretical). Fig. 3 shows "as-stripped" and sintered samples and indicates qualitatively the shrinkage which occurred during sintering.

III. RESULTS AND DISCUSSIONS

A. Use of Oxides

An investigation was conducted using chromium oxide (Cr_2O_3), nickel oxide (NiO) and iron oxide (Fe_2O_3) as slip components in quantities such that the oxides, after being chemically reduced, yielded a sintered product consisting of 18 percent chromium, 8 percent nickel, and the remainder iron. Initially the oxides were blended with tap water alone. However, this yielded a non-cohesive slip which did not provide a section suitable for sintering.

The usual binders ammonium alginate and elvanol were then added to the oxides and water mixture. A partially cohesive

Fig. 3 Plaster Mold Containing As-Cast Slip. Comparison of Sintered Sample to Unsintered Sample.

Plate No. 432-1

slip was obtained which was sintered at 1320°C in hydrogen. Physical measurements of the piece before and after sintering indicated a 90% volume reduction. The combination of electrolytic iron powder plus chromium oxide and nickel oxide was then tried in an attempt to combat this excessive shrinkage. A segregated slip was obtained with this mixture of powders which could not be used to obtain significant results. The segregation was probably caused by the difference in densities of the powders which were used.

B. Drying

Results from the initial sintering experiments yielded samples containing a considerable number of air holes, which were attributed to the entrapped moisture which generated steam during the heating cycle. An attempt to dry the as-stripped slip at approximately 100°C for one-hour prior to sintering resulted in an extremely dry slip, which could not be handled. In some instances, the drying effect was severe enough to transform the slip into a small mound of powder. Shorter drying times produced the same results. Vacuum drying at 800°C was also investigated. In anticipation that this elevated drying temperature might also serve as a presintering temperature. A powdery product, which could not be handled without fracturing was produced by this treatment. Consequently, the drying technique was omitted, and sintering was conducted immediately after stripping in order to eliminate any unnecessary handling operations.

C. Comparison of Properties of Wrought, Cold Pressed, Sintered and Slip Cast Stainless Steel Products

Samples prepared in this investigation were cast in the shape of tensile samples in order to facilitate evaluation of physical properties. The results obtained in the evaluation of these tensile samples were not reproducible in many cases. For example, samples prepared from slips made under the same conditions showed variations in sintered density from 5.2 to 6.4 g/cc.

The best strength properties were obtained with samples produced from a slip which consisted of:

-325 stainless steel powder (302B)	18.8%
-100 stainless steel powder (302B)	18.8%
-200 stainless steel powder (302B)	18.8%
-100+160 stainless steel powder (302B)	18.8%
elvanol	0.25%
ammonium alginate	0.35%
water	24.2%

The sample cast with this slip was sintered at 1320°C for eight hours and had the following properties:

density	6.62 gm/cc
ultimate tensile strength	70,500 psi
yield strength (0.2% O.S.)	40,200 psi
elongation	20%

A slip was prepared which had a higher percentage of fine particles. The sintered density of this sample was 7.08 g/cc which was the highest density obtained in this investigation.

Composition of the slip was:

-325 stainless steel powder (302B)	25%
-200 stainless steel powder (302B)	25%
-170+200 stainless steel powder (302B)	25%
elvanol	0.25%
ammonium alginate	0.35%
water	24.4%

Fig. 4 a and b shows that the low density of the sintered pieces was a result of porosity in the sample. A comparison of the two photographs indicates that increased sintering time decreases the porosity resulting in increased density. In addition, an increase in grain size was also evident with extended sintering time.

A comparison of the properties of a slip cast sample with cold pressed and sintered stainless steel and wrought material is shown in the table below:

Property	Wrought (a)	Cold Pressed 70 tsi (b) Sintered 1 hr. 1275°C	Cold Pressed 50 tsi (b) Sintered 1/2 hr., 1210°C	Slip Cast
Density, g/cc	8.03	7.33	6.51	6.41
Yield Strength, psi	30,000	-	-	40,400
Ult. Strength, psi	80,000	93,357	24,400	57,500
Elongation %	40	37	6.6	9.8
Hardness	90R _B	83R _B	69R _F	60R _F

- (a) Data from reference 2
(b) Data from reference 3

(a) Sintered 8 Hours - Density, 6.1 g/cc

Plate No. 13260

(b) Sintered 16 Hours - Density, 6.95 g/cc

Plate No. 13464

Fig. 4 Photographs of Structure of Slip Cast Stainless Steel (302B) Powder Etched. 250X

The data for the slip cast sample was selected in order to be representative of average material which was evaluated. As mentioned earlier, samples were produced with higher density as well as strength, but these data were not used in the comparison with other materials, because at this stage of the development it was not considered to be typical of samples tested. The higher strength and density values which were reported above, however, do indicate the feasibility of producing material by slip casting which has desirable properties.

A comparison of the data in the above table shows that generally the density of slip cast material is lower than the wrought and the cold pressed and sintered material. The strength of the cold pressed and sintered sample which has a density of 6.51 g/cc is considerably lower than the strength of the slip cast material which has a slightly lower density. These results may be an indication of the sintering action obtained in each process. As indicated earlier, the density of the slip cast sample before sintering was approximately 3.2 g/cc, and during sintering, as shown in Fig. 3, there is considerable shrinkage. The increase in density during sintering is an indication of considerable diffusion between particles to form strong metallurgical bonds. In the case of the cold pressed and sintered pieces however, the density increased from 6.47 to 6.51 g/cc during sintering. This slight increase in density indicates slight diffusion or bonding between particles has occurred and therefore

the strength results primarily from the mechanical bonds established during cold pressing. Increased strength is obtained for cold pressed samples when the density is high, as shown by sample having a density of 7.33 g/cc.

A photograph of the fracture of a slip cast tensile sample is shown in Fig. 5. The fracture is typical of that obtained for all samples tested. Little reduction in area or necking down was measured at the fracture, which is an indication of the brittleness of samples. The presence of voids and the low density of the material are probably the cause of the brittle fracture as well as the low percent elongation.

IV. SUMMARY

The investigation described was conducted in order to determine the feasibility of preparing solid material by slip casting of metal powders. A method is described which produced slip cast samples of relatively good physical properties. Some of the variables were not completely evaluated and therefore the results were not always reproducible. Additional development work on this process should provide the answers required in order to produce consistently good material.

Fig. 5 Photomicrograph of Tensile Sample Showing Fracture.

Plate No. 13667

14X

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