



## NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

WASHINGTON, D.C. 20546

REPLY TO  
ATTN OF: GP

October 14, 1970

TO: USI/Scientific & Technical Information Division  
Attention: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General  
Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned  
U.S. Patents in STAR

In accordance with the procedures contained in the Code GP to Code USI memorandum on this subject, dated June 8, 1970, the attached NASA-owned U.S. patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No. : 2,944,316

Corporate Source : Lewis Research Center

Supplementary  
Corporate Source : \_\_\_\_\_

NASA Patent Case No.: XLE-00106

Gayle Parker

Enclosure:  
Copy of Patent

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W. A. MAXWELL ET AL

2,944,316

PROCESS OF CASTING HEAVY SLIPS

Filed Dec. 20, 1956

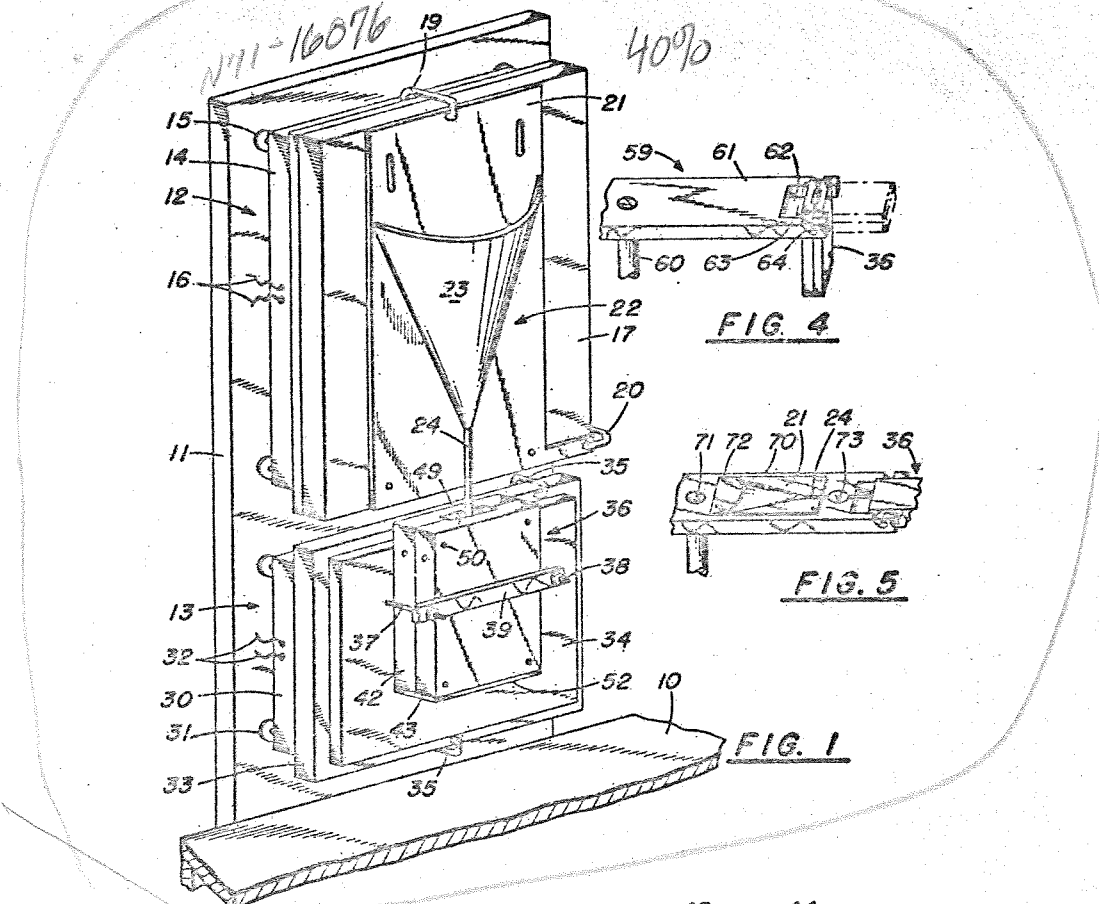


FIG. 4

FIG. 5

FIG. 1

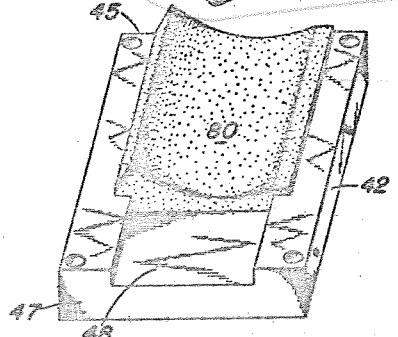


FIG. 2

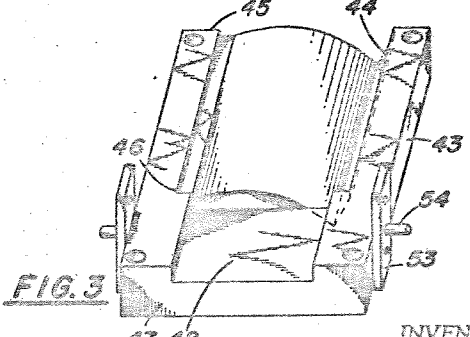


FIG. 3

INVENTORS  
WILLIAM A. MAXWELL  
JOSEPH DOUGLASS

BY

R. J. Tompkins  
ATTORNEYS

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2,944,316

## PROCESS OF CASTING HEAVY SLIPS

William A. Maxwell, Towson, Md., and Joseph Douglas, Parma, Ohio, assignors, by mesne assignments, to the United States of America as represented by the Administrator of the National Aeronautics and Space Administration

Filed Dec. 20, 1956, Ser. No. 629,759

4 Claims. (Cl. 25-156)

(Granted under Title 35, U.S. Code (1952), sec. 266)

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates to the casting of a slip by use of freezing procedures.

In the copending case of William A. Maxwell et al., Serial No. 402,828, filed January 7, 1954, now Patent No. 2,893,102, granted July 7, 1959, a process of casting metal ceramic and refractory compound powders is described wherein the powders are formed into a plastic slip and forced into a mold by a plunger, the resulting casting being frozen and vacuum dried, to eliminate a liquid phase. While this method is satisfactory for many uses there are limitations to the size of casting possible and the procedure does not lend itself to producing castings of high solidity.

In the present invention an important object is to provide a method of freeze casting in which use may be made of slips of high viscosity in forming the casting. An object also is to provide a freeze casting method which lends itself to centrifugal pressure procedure for compacting the forming powder slip. Still another object is to provide drying means for the casting which will dispense with vacuum applications while at the same time maintain the casting shape after the freezing step without development of a liquid phase. Another object of importance is to provide a drying step for the frozen casting which will tend to maintain the casting shape throughout the thawing thereof up to the time of sintering.

Other objects and many of the attendant advantages of this invention will be readily appreciated as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawings wherein:

Fig. 1 is a view in perspective of the mold and the mold filling equipment;

Fig. 2 is a perspective view of one mold section with the casting therein;

Fig. 3 is a perspective view of the other mold section showing the pin brackets for support in the centrifuge;

Fig. 4 is a perspective view of a fragment of the centrifuge for compressing the slip powder; and,

Fig. 5 is a fragmentary view of a centrifuge unit showing the rotating arm and the slip receptacle mounted for feeding while the unit is rotating.

Referring to Fig. 1, there is shown a horizontal work surface, such as the table 10 to which a vertical back-plate 11 is attached. To this back-plate on the face adjacent the table, two units, 12 and 13, are attached, unit 12 forming the vibrator-feed unit and unit 13 the vibrator-mold unit. The vibrator-feed unit is in the form of a rectangular casing 14 supported free of the back-plate 11 by projecting rod elements 15 and containing the usual coil and vibrator armature operated by a 60-cycle current from power lines 16. The vibrator armature is attached to the vibrator support 17 and to this support

2

the slip container 18 is attached, as by clamps 19 and 20. The container 18 takes the form of a baseboard 21 and a receptacle 22 including a funnel cavity 23 tapering to a connecting outlet pipe 24, which may be flexible, for conveyance of the slip to the mold unit 13.

The mold unit 13 similarly, includes a rectangular casing 30 supported outwardly from back-plate 11 by attachment rods 31 and having a vibrator device supplied with 60-cycle electrical power by power lines 32.

The vibrator plates 33, 34, which are placed over the outer side of casing 30 and attached thereto by clamps 35, are connected to and actuated by the vibrator coil armature, as in the unit 12, and plate 34 supports the mold 36 by means of spaced outwardly extending bolts 37 and 38 joined over the front mold section by bolt clam 39. The mold 36 includes the two mating dies 42 and 43 formed of conventional alloy metals, preferably with low temperature coefficients of expansion. Except for the mold areas, the dies are identical in size and shape, each die being in the shape of a flat rectangular block having a mold recess 44 on one side extending from the die end 45 to a point 46 short of the end 47 and having a recess 48 extending from the mold cavity 44 to the die end 47. When assembled, as by bolts 50 with the mold cavities in registration, the opposed recesses 47 form a passageway 49 for insertion of the powder slip, as shown in Fig. 1, and when assembled as shown in this figure, the slip receptacle outlet pipe 24 projects into this passageway. The mold includes, also, the base edge plate 52 (Fig. 1) which seals the mold cavity at end 45 of the die plates. Also, as shown in Fig. 3, pivot straps 53, consisting of flat plates perforated for screw attachment to both dies and having a centrally disposed pivot pin 54, are provided for centrifuge use.

Fig. 4 illustrates diagrammatically a form of centrifuge 59 usable in the compacting step of the process. This unit includes a vertically positioned power shaft 60, rotatable by any conventional power means, to which an arm 61 is fixed. In form, this arm is a horizontally flat strip having at its outer end a rectangular recess 62 forming symmetrically positioned terminal support side members 63. These side members are partially slotted, downwardly from the top edge and outwardly to form outwardly inclined slots for reception of the mold pivot pins 54. Normally, when the mold is placed in the slots 64 with shaft rotation at zero, it is vertically pendant, but on shaft rotation the mold swings outwardly and if the slip is present this material is compressed to a degree varying with the speed of shaft rotation.

Having indicated the structural elements involved, the details of the process may now be set forth.

The metal is first granulated to powder form with an average diameter of about nine microns or less and then formed into a slip by mixture of sufficient water to form a thick non-flowing slip. While not essential, it is preferable to include in the mix a binder so that not only may the amount of water needed and requiring later removal be further reduced but, also, the final mixed product has a consistency and coherence such that, under its own weight, the mix does not slump after prolonged standing but at the same time permits flow of the powder constituent under conditions of vibration or centrifugal pressure of the mixed mass. It is, of course, apparent that the percentage of liquid in the mix varies with the type and particle size of powder, but it may be generally stated that all slips are usable for the described method which contain up to 50 percent less moisture than would be normally required by conventional procedures in powder metallurgy.

For example, in the case of molybdenum disilicide powder, 3.55 grams of water is added to 20.0 grams of the powder.



While the binder used may be selected from numerous flow inhibiting substance, the use of a rubber-base binder has been found particularly successful, the binder including, with rubber latex, an emulsifying agent and water. Slips prepared with powders having 3 to 5 microns average particle size are effective with 16.5 percent binder. Otherwise stated, 1 to 2 percent of the powder weight in dry rubber makes an adequate binder.

After preparation of the slip it is placed in receptacle 22 and the vibrators in both the receptacle and mold units energized. At once a flow of slip moves into the mold where it is again vibrated to compact and solidify the mass in mold form. The mold is then removed from the vibrator assembly, suspended in the centrifuge 59, and subjected to the centrifugal action of this unit, whereby the slip is closely compacted in the dies. Obviously, high speeds of centrifuge rotation are desirable, the higher the speed the drier and sounder the resulting castings secured.

After the centrifuge treatment the mold is subjected in appropriate chambers to freezing temperatures, a temperature of  $-15^{\circ}$  F. being found adequate. The casting in the form shown in Fig. 2 at 30, may now be readily handled and, on removal from the mold, it is placed in a container under normal room conditions of temperature and pressure and covered with a drying agent, such as silica gel, activated alumina, fuller's earth or bentonite. The mass is then allowed to come to room temperature. As the casting temperature rises, the released moisture is absorbed by the drying agent so that any deformation of the casting, such as would occur on passage through a semi-liquid phase, is avoided. It is notable that the drying agent supports the casting during the drying step by completely enclosing the casting, thus avoiding any local stresses which might distort the original mold configuration.

After drying, the casting is sintered at appropriate sintering temperatures, values varying for different metals. For example, for the refractory metal compound molybdenum disilicide a sintering temperature of about  $2300^{\circ}$  F. is used. If desired, the dried mass may be infiltrated with liquid metals or ceramics.

While the process has been described as applicable to special materials the various process steps are applicable to conventional powder techniques of both ceramics and metals. Also, it should be stated that although spinning the casting in a centrifuge is desirable in obtaining sound products, under some conditions this step may be omitted. It is noted further that, if desired, the mold may be filled during rotation on the centrifuge, using the so-called "splash-filling" or other method, the container 12 being aligned on arm 70 (Fig. 5) between the mold unit 36 and the arm pivot 71, with conventional retaining means, as hooks 72 holding the receptacle baseboard 21 in place. The tube 24, being flexible, accommodates itself to the varying position of the pivoted mold, and on rotation a measured or valved mass of the slip is forced into the mold, a valve 73 being indicated.

The factors of paramount importance in the process, as described, include the use of combined gravitational and vibrational forces to fill the mold with a low-liquid content slip, whereby the particled mass is solidly packed without being unduly forced as by extrusion methods. Additionally, the rubber-based binder, as employed, imparts a texture to the slip which is most effective in insuring adequate flow under vibrational treatment. Other important factors include the firm compaction of the slip in centrifuge treatment and the successful maintenance of casting form during drying by use of a combined packing and drying agent.

Obviously many modifications and variations of the

present invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

What is claimed is:

1. A process of making articles from powdered materials which comprises mixing the powder with a liquid in such amount as to form a thick slip which flows only under pressure, applying pressure to said slip to obtain a free slip flow into a mold to form a mold mass, subjecting the mold mass to centrifugal forces to compact the mass powder in the mold, freezing the mold mass to form a casting, drying the casting by heating it to standard room temperature at normal atmospheric pressure and by application of a solid moisture absorbing agent to the casting surface, and sintering said casting.

2. A process of making articles from powdered materials which comprises mixing the powder with a liquid in such amount as to form a thick slip which flows only under pressure applying pressure to said slip to obtain a free slip flow into a mold under both gravitational and vibrational forces to form a mold mass, subjecting the mold mass to centrifugal forces to compact the mass powder in the mold, freezing the mold mass to form a casting, drying the casting by heating it to standard room temperature at normal atmospheric pressure and by application of a solid moisture absorbing agent to the casting surface, and sintering said casting.

3. A process of making articles from powdered materials which comprises mixing the powder with a liquid in such amount as to form a thick, non-flowing slip, applying pressure to force said slip into a mold to form a mold mass, vibrating said mold mass to solidify the same, subjecting the mold mass to centrifugal forces to compact the mass powder in the mold, freezing the mold mass to form a casting, drying the casting by heating it to standard room temperature at normal atmospheric pressure and by application of a solid moisture absorbing agent to the casting surface, and sintering said casting.

4. A process of making articles from powdered materials which comprises mixing the powder with water in amounts to form a thick, non-flowing slip mixing a viscous binder with said slip, applying pressure to said slip to obtain a free slip flow into a mold to form a mold mass, subjecting the mold mass to centrifugal forces to compact the mass powder in the mold, freezing the mold mass to form a casting, drying the casting by heating it to standard room temperature at normal atmospheric pressure and by application of a solid moisture absorbing agent to the casting surface, and sintering said casting.

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