Feasibility of Fabricating Metal Parts from 17-4PH Stainless Steel Powder

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Abstract

17-4 PH stainless steel is known to provide an attractive combination of high strength and corrosion resistance. In this research, the feasibility of SFF fabrication of high density parts using 17-4 PH powder is examined. A part can be fabricated using both indirect and direct methods. The indirect method includes making a negative RTV mold, making compounded material using ECG binder and stearic acid with the metal powder, and pouring the compounded material to get a green part. This is followed by binder burn out(BBO) and sintering cycles. The direct method uses Fused Deposition of Metals(FDMet). In FDMet, the 17-4PH powder is compounded with a FD binder and extruded into filaments, followed by part building, BBO and sintering. The initial results of the indirect method of fabrication produced 91% theoretical density of 17-4 PH parts with Vickers hardness of 223.

Keywords: **Rapid Prototyping RTV mold 17-4PH FDMet Binder Burning Out(BBO) Sintering**

Introduction

Solid Freeform Fabrication emerged about ten years ago, due to the advantage of making complex parts with low cost and high speed. It is becoming one of the most important new techniques in the manufacturing area. Although there are many advantages, there exists some drawbacks. For example, in many cases, a part can be used as a physical prototype, but can not withstand high service stresses and temperature. Various efforts have been focused on developing new process to make high strength, low cost parts with SFF, such as DTM Rapid Tooling[1], MIT 3DP[2], and Fused Deposition of Ceramic[3].

The objective of this study is to develop an efficient method to fabricate metal parts quickly with low cost. Two methods are adopted in the research, one is the direct method or Fused Deposition of Metal(FDMet), and the other is RTV Molding method. Building a metal part by FDMet is our final goal, the indirect method is used as an initial step to determine the right material composition. In this paper, full details of this RTV Molding technique are reported.

Experiment Procedure

Two different approaches are under investigation for manufacturing functional metal components referred to as the "direct" and the "indirect" techniques as Fig.1. The direct method (Fused deposition of metals, FDMet) uses metal particle loaded thermoplastic filament as the material feedstock, fed via counter rotating rollers into a heated extruder, referred to as a liquefier. The filament acts as the piston to extrude the molten metal loaded polymer material out of a 250 micron to 635 micron diameter nozzle onto a z-stage platform, where the material cools rapidly and bonds to adjacent layers. The material deposition rate and liquefier x-y position, etc., are controlled by the computer. For powder metal parts, the binder is subsequently removed in a thermally driven process called binder burn out (BBO), then sintered to full density. In the indirect method or RTV Molding method, a STL file is generated from the desired part, the file is delivered to the SLA or FDM machine which builds a positive prototype. The RTV mold is made by pouring a molding material around the prototype. This is done by mixing the metal powder with the ECG2 and stearic acid, pushing the mixture into the RTV mold. The binder removal and sintering process of the indirect method are the same as those of the FDMet.

The development of the direct and indirect SFM processes for hard tooling are going on simultaneously. Several steps in each of these processes have been accomplished. The processing details and the preliminary results are described below.

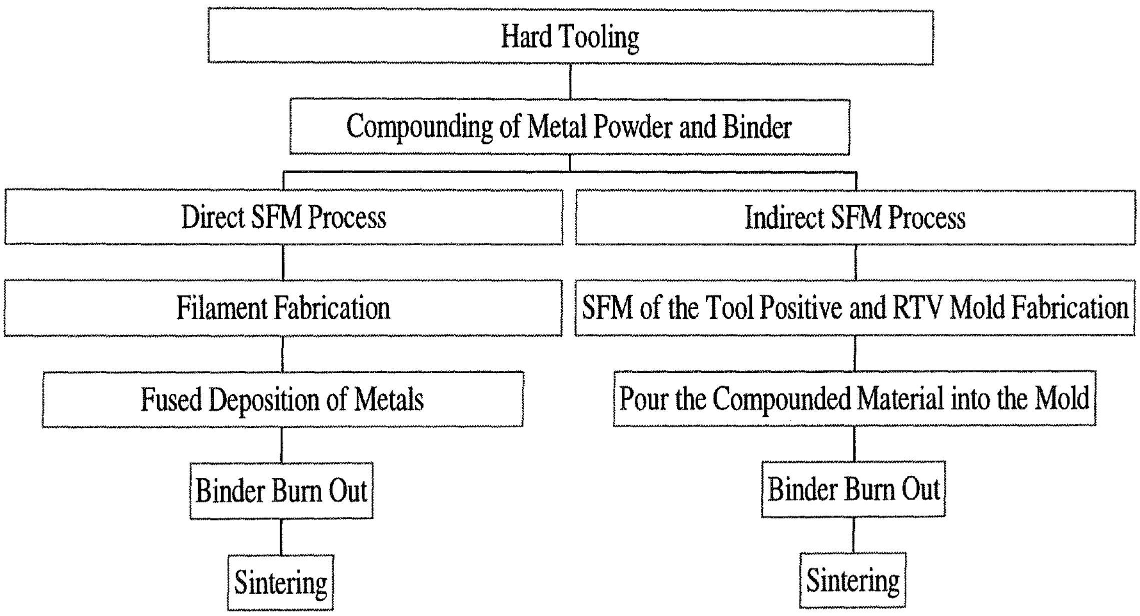


Fig1. Flow chat of FDMet and RTV mold methods

Materials

The material used in this research was 60% volume 17-4 PH stainless steel(-10 microns water atomized), provided by Ametek company in PA of U.S.A. The powder particles were irregular in shape, the chemical composition is Fe-0.031C-4.39Ni-15.82Cr-0.12Si-0.21Mn-

3.78Cu-0.014S-0.22Cb-0.61O-0.02P-0.09N, and its particle size distribution (as provided by the manufacturer) is given in the Table 1.

Table 1. The size distribution of the -10 microns 17-4PH SS powder

Microns	Percent(less than)
62	100.0
44	100.0
31	96.2
22	81.7
16	59.6
11	37.9
5.5	10.3
3.9	3.0

The binder used in this research was ECG2(Electro Ceramics Group binder #2),which was developed by the Ceramic and Materials Engineering Department of Rutgers University[4].

Stearic acid is a common surfactant for metal powder, although there are some other surfactants that could be used such as zinc stearate or magnesium stearate.

Feedstock preparation

In order to get full dispersion of the stearic acid, ball milling was used. First some grinding media and a certain volume percent of metal powder were added into a bottle, with some stearic acid added to acetone. The mixture was full dissolved and then added to the bottle, after the mixture was ball milled about 20 hrs, it was then passed through two sieves, one to remove the grinding media and one to sieve the powder, followed by drying.

The binder and coated powder are mixed using a HAAKE torque rheometer at a temperature of 165°C. First, all of the ECG binder is added to the hot mixer and melted. Next, the stearic acid coated metal powder is added into the mixer in four sub-steps (40%, 30%, 20% and 10% of the total amount of metal powder) at 15 minute intervals. After all of the metal powder is added, and when the torque in the mixer is constant, i.e. reaches a steady state value, the temperature was lowered to 130°C, after another hour a uniform compound is achieved. The whole process usually takes three to four hours. After the compounding is completed, the compounded material is removed from the mixer, and grounded into small pieces.

This granulated, compounded material is now ready either for RTV molding of the part in the indirect method, or for fabrication of filament (by extrusion) for direct part fabrication by FDMet.

RTV mold making

RTV tooling is a process which creates a soft rubber mold from an original positive prototype structure. In this work, the desired positive tool is made by stereolithography. The silicon material of the soft mold can withstand the 100-200°C temperature of the metal filled binder formulation that will be cast into the mold. In addition, the fabrication process is fast, accurate and inexpensive. The RTV mold process used in this project is as follows:

(1) Prepare the base, curing agent and a container.
(2) Stick wax on the bottom of the original positive part so that the part can be attached to the box, and prevent creation of air bubbles.
(3) Record the weight of the base mold material (approximately 40 g), use the curing agent (10% by weight of the base), stir the base and agent, and then put them into the vacuum oven to remove any air, pour the material around the part, making sure that the part is completely covered.
(4) Put the container (which now holds the uncured mold material and part) into the vacuum oven again. This is performed to remove any air trapped in the mold material. Continue this process until all of the air bubbles are removed.
(5) The mold material is fully cured by keeping it at room temperature and 1 atm pressure for 24 hrs, then the positive part is removed from the cured mold material.

After the RTV mold is ready, the compounded material is poured into the mold to form a green part.

De-binding and Sintering

Before sintering, the green part must be processed through a treatment to extract the organic binder. Based on earlier work, a heat treatment approach was developed which has successfully removed the binder from the green 17-4PH metal part. The development of an appropriate heat treatment to burn out the binder requires that we have a good understanding of weight loss and shrinkage characteristics of the material system during heating. Stresses can develop in the parts during heating if the binder decomposition gases evolve in the interior of the component without a sufficiently easy diffusion path to the surface. During the burn out process, the green part was buried in a very high surface area carbon powder to aid in the removal of the binder through wicking (via capillary suction) at low temperature. An optimized binder burnout schedule will be determined by measuring the shrinkage and weight loss after a finite hold at various temperatures, followed by furnace cool to room temperature.

The BBO schedule for hard tooling was derived from the current PZT project [4]. It is a slow process. Following the BBO process, the part is sintered in hydrogen. The sintering can be performed at temperatures as low as 1121°C, but higher temperatures will yield better mechanical properties. The cooling process can not be slower than what it would be in still air, and the part must be cooled to below 32°C prior to precipitation hardening heat treatment process[5].

Table 3. Sintering schedule for 17-4 PH metal parts

Temp(°C)	Rate	Time (Hrs)
20-1260	5°C/min.	4.1333
1260	hold 30min.	0.5
1260-20	Natural cooling	~ 5

Results

Two applications were investigated in this research, a wrench part and a lug fit part. The wrench part was first made, then the process was improved and a more complex lug fit part was made successfully.

Fabrication of the wrench part

The wrench part was used to demonstrate the process. A CAD file was generated in IDEAS-5 software. .STL file was exported to the Maestro software of Stereolithography machine(SLA-190). The generated data files were transferred to the control computer to run the machine and build the SLA wrench part.

The RTV wrench shown in Fig.2 mold was made after applying RTV molding process on the SLA wrench part as in Fig.3. The green wrench as Fig.4 was made by pouring the compounded material into the RTV wrench mold. The de-binding process took a long time and very fragile mechanical properties the BBO wrench part were found. The sintering process was done by putting BBO part into a Alumina tube at high temperature as Table 3. The reason the sintered wrench part in Fig.5 was broken is that the BBO part was so fragile as to be easily broken.

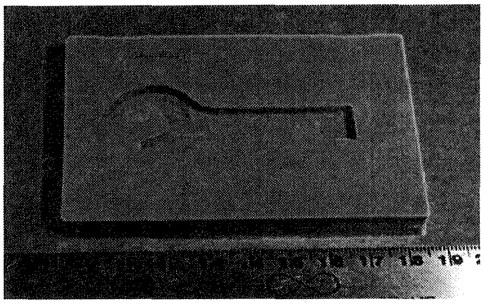


Fig.2 RTV wrench mold

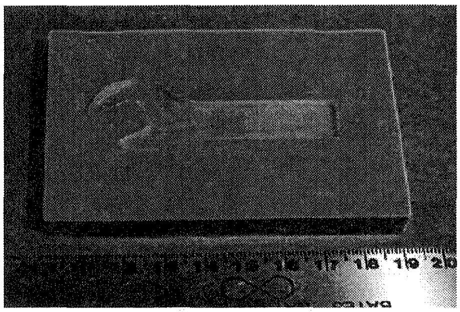


Fig.3 Demonstration of the RTV mold process

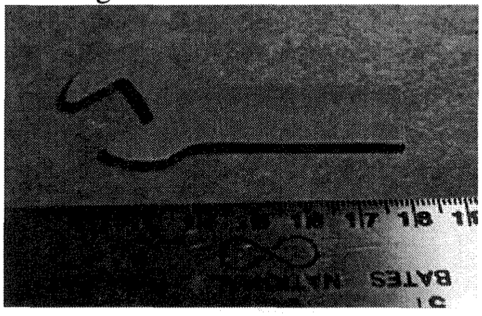
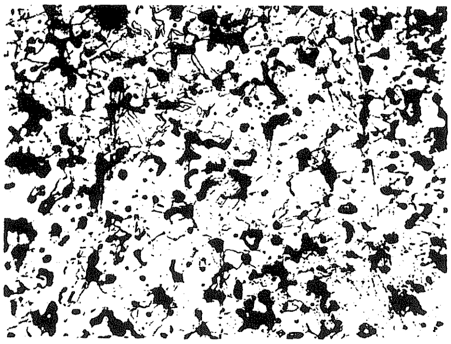


Fig.4 Green wrench part

Fig.5 Sintered Wrench part, broken during the BBO

Analyses of the sintered part were performed. The hardness of the sintered metal is B97 or Vickers hardness of 223. The microstructure consists of ferrite grains with ASTM grain size of 10-11 as Fig.6. It was also found that the sintered part was not full dense as Fig.7. The porosity is approximately 8%-9%. The distribution of the Pore Size is as Table 4. The Pore Size Range is 0.5-5.5 microns.



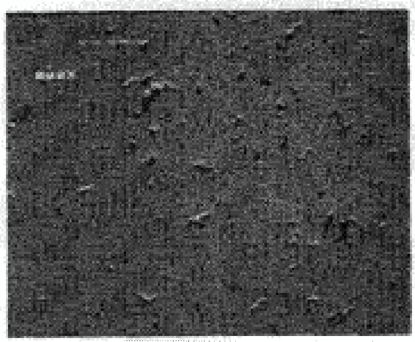


Fig.6 Microstructure of sintered part.

Fig.7 Pore size graph of the sintered part.

Table 4 Pore Size Distribution of the Sintered Wrench Part

Size, Microns	Distribution -#/Inch^2
Less than 1.0	3270
1.0-5.0	1048
More than 5.0	635

Fabrication of the lug fit part

Since the wrench was not a complex geometry, further efforts were made to fabricate a more complex part, the lug fit part which was required by the research program.

Since the lug fit part is hollow inside, care must be taken during the RTV mold making process. Since the process was done in a vacuum, if the SLA part was not attached to the bottom of the container firmly, the SLA part would rise in the RTV material, and RTV material would fill inside the lug fit part. A de-molding agent was also preferred because it was very hard to remove the SLA lug fit part from the mold. The mold was cut in half, and the SLA lug fit was removed. A piston RTV positive mold was also made to push the compounded material into the mold. The SLA lug fit, Green part, BBO part and sintered part can be seen in Figures 8 to 11. The dimension of the part in various stages are shown in Table 5.

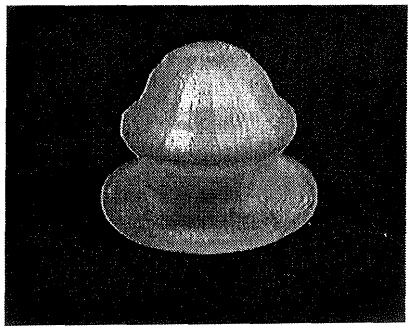


Fig.8 SLA lug fit part

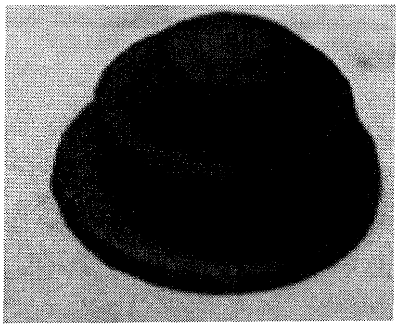


Fig.9 Green lug fit part

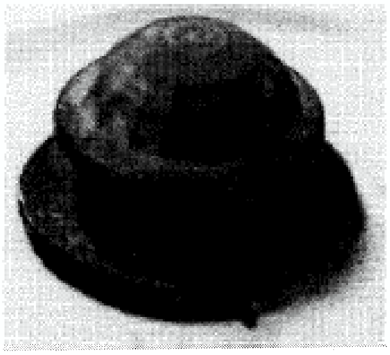


Fig.10 BBO lug fit part

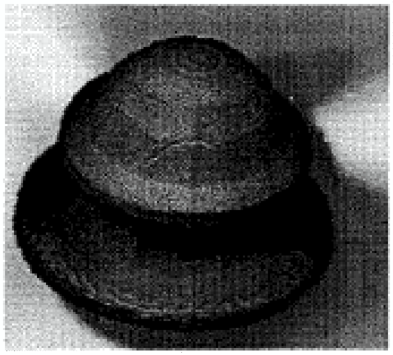


Fig.11 Sintered lug fit part

Since the lug fit part was hollow inside, additional supports were added when it was put into the sintering oven. Initially, no additional support was used and the part collapsed during the sintering, after correcting the problem, a successful Lug fit part was fabricated as shown in Fig.11 .

Shrinkage of the green to sintered part was measured. The results are shown in Table 5. The maximum shrinkage of 8.64% in diameter in the sintered part was found. The shrinkages in all three dimension were similar. The shrinkage effect on the final dimension of the part can now readily be incorporated into the whole design process to gain higher accuracy.

Table 5 Shrinkage Measurement Results

	SLA	GREEN PART *	SINTERED PART*
Inner Diameter(in)	0.466	0.471(+1.07%)	0.430(-7.7%)
Middle Outside Diameter(in.)	1.053	1.059(+0.57%)	0.962(-8.64%)
Bottom Outside Diameter(in.)	1.353	1.368(+1.1%)	1.245(-7.98)
Height(in.)	0.935	0.974(+4.17%)	0.880(-5.8%)

* The data in the '()' is the increase/decrease percent with respect to the SLA PART.

Conclusions

The present experiment work showed that using 17-4PH metal powder to fabricate a part directly and indirectly is very promising. From the results, the following conclusions can be drawn as follows:

(1) Materials for the hard tooling formulation were investigated, SS 17-4PH with ECG2 binder and stearic acid as a surfactant.
(2) RTV mold development for 17-4PH SS powder has been demonstrated. Several prototype hard tool structures have been fabricated and are being evaluated.
(3) Compounding of SS 17-4PH with ECG2 has been performed and the investigation on the filament fabrication is underway.

Acknowledgments

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