Metallic part fabrication using selective inhibition sintering (SIS)

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Abstract

Purpose – The purpose of this paper is to investigate the fundamentals of the selective inhibition sintering (SIS) process for fabricating dense metallic parts.

Design/methodology/approach – A SIS-Metal process based on the microscopic mechanical inhibition is developed. In the process, salt solution is printed in the selected area of each powder layer; the salt re-crystallizes when water evaporates; salt crystals decompose and grow rapidly prior to sintering; the generated salt particles spread between metal powder particles and prevent the fusing of these particles together, hence inhibiting the sintering process in the affected regions.

Findings – The SIS-Metal process has numerous advantages. An inhibition of sintering mechanism is established for the future development of the technology. Through chemical and visual analysis using STM the mechanism for the inhibition phenomenon has been identified.

Research limitations/implications – Only bronze powder has been used in the research. Accordingly, the inhibition chemical has been engineered for this material choice. The approach should be feasible for other metals but a proper inhibitor would need to be found for each material choice.

Practical implications – The only limitation envisioned for the process may be the removal after sintering of inhibited sections in hard-to-reach areas using physical means such as scraping or vibration. Chemical removal of such sections should be possible, however.

Originality/value – The paper illustrates a new additive manufacturing technology for metallic parts fabrication.

Keywords Sintering, Metals, Advanced manufacturing technologies, Metallic parts fabrication, Powder sintering, Selective inhibition, Powder metallurgy

Paper type Research paper

1. Introduction

The selective inhibition sintering (SIS) process is an additive manufacturing (AM) technology which builds parts on a layer-by-layer basis. The principle idea of the SIS process is the prevention of selected segments of each powder layer from sintering. Therefore, the SIS process may be considered as an opposite approach to the selective laser sintering (SLS) process in which selected areas of powder are sintered by a fine laser beam. In previous work on the SIS for polymer powder (Khoshnevis et al., 2003; Asiabanpour et al., 2004, 2006) it was demonstrated that the SIS-Polymer process can fabricate high quality parts at a high speed using relatively low cost machines. In the research reported here the application of SIS to metal powder (denoted as SIS-Metal) is investigated. Due to the high temperature and oxygen-free environment required in the sintering of metallic powders (e.g. 800-1,300°C) the previously developed SIS-Polymer process in which each layer is sintered on the fabrication machine is not applicable to SIS-Metal. Hence, the SIS-Metal process requires a dedicated effort for identifying a new inhibition mechanism by considering the sintering properties of metal powder and a new sintering method, preferably using conventional sintering furnace. The experimental results have demonstrated that the SIS-Metal concept is feasible and that commercial quality metallic parts can be fabricated using the process.

The inhibition mechanism used in the SIS process plays the major role in successfully developing the process (Khoshnevis et al., 2003). There are four possible inhibition mechanisms in SIS:

1. macroscopic mechanical inhibition;
2. microscopic mechanical inhibition;
3. chemical inhibition; and
4. thermal inhibition.

Due to the high sintering temperature of metallic powders applying thermal inhibition to the SIS-Metal process is difficult. Consequently, the remaining three inhibition mechanisms have been tried by this team for metal powders. The inhibition methods investigated are:

• the use of a ceramic as a macroscopic mechanical inhibitor;
• application of lithium chloride and aluminum sulfate as microscopic mechanical inhibitor; and
• application of sulfuric acid and hydrogen peroxide as chemical inhibitors.

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Two prototype machines have been developed for compact and loose powder sintering and a set of experiments for copper and bronze powders has been performed. The experimental results based on macroscopic mechanical inhibition and chemical inhibition are briefly discussed as follows.

Figure 1 shows a coin with a part that is fabricated by compressing copper powder in a ceramic inhibitor shell. Note that the ceramic inhibitor used in fabricating the part is shaped by the tip of an end mill tool and not by a deposition system. Although some specialized print heads are claimed to be able to print dilute ceramic slurry and methods have been developed to extrude viscous ceramic slurry (such as the work done at Sandia National Laboratory (Stuecker et al., 2003)), the deposition systems are not commercially available. Our experimentation of the macroscopic mechanical inhibition approach has thus been limited to a few basic trials. However, our experiments support the feasibility and merit of the approach, which can be further developed if an accurate and versatile ceramic printing method becomes available.

Several successful experiments have also been performed using chemical inhibition. However, it was concluded that this method of inhibition is limited because the related chemical reactions are often slow and many desirable metals and alloys (e.g. super alloy powders) are resistant to chemical reaction. Furthermore, many chemicals that are used to etch or oxidize metals also corrode the metallic components of the printing mechanisms and other machine components. They are also irritating and/or harmful to living organisms and hence present safety and environmental concerns.

Based on the above observations it has been decided that for SIS-Metal process the most promising inhibition method is microscopic mechanical inhibition, which is the focus in the remainder of this article. Through this experimental research a microscopic mechanical inhibition theory has been formed which encompasses the selection of inhibitors and the inhibition behavior in the presence of metal powders prior and during the sintering process.

2. SIS-Metal process based on microscopic mechanical inhibition

The SIS-Metal process based on microscopic mechanical inhibition is shown in Figure 2. Compared to the layer-by-layer sintering approach used in the SIS-Polymer process, a bulk sintering approach is adopted for SIS-Metal. This is mainly due to a longer cycle time that is required in heating and cooling metal powder compared to plastic powder. In addition, the layer-by-layer sintering of metal powder would require the fabrication machine to operate in an oxygen-free environment. Hence, the required hardware would be cost prohibitive. Finally, undesirable shrinkage effect among sintered layers may be generated in the layer-by-layer sintering approach, which would create undesired internal stresses.

The SIS-Metal process based on the bulk sintering approach has five main steps as follows:
1. Printing sintering inhibitor. A deposition nozzle with a fine orifice, or an inkjet print head, is used to deliver a sintering inhibitor to the selected areas (i.e. layer profile) of the powder layer. If aqueous solutions are used, the layer is dried before continuing with the subsequent layer.
2. Laying a thin powder layer. Metal powder is spread as a thin layer over the build tank using a blade or a roller.
3. Creation of a boundary to contain part. A consolidating liquid is deposited on the powder bed at the periphery of the part profile. The profile of this deposition may be a simple shape such as square or circle. When all the layers are completed, such depositions will create a solid container around the area that includes the 3D part. Using this container the completed green part can then be removed from the machine and transferred to the sintering furnace. Interestingly, the salt solution used as the printed inhibitor can hold the metal powder particles together after it is dried. The consolidating liquid for creating the container can be different from the inhibitor solution, however.
4. Heating and compression. A heater is used to evaporate water and other liquid additives (such as alcohol which may be needed for breaking surface tension) in the printed inhibition sections. An optional motorized press may be used to compact the metal powder layer after drying the inhibitor solution in order to create a powder bed with increased density. Although layer compression can improve part density, it may also result in part deformation. Successful implementation of compression requires careful study of the deformation process so that the effect may be properly incorporated in the CAD model. The layer compression was not performed in the study presented in this paper.
5 Bulk sintering in an oven. After all the layers have been completed, a metal powder block is extracted from the build tank and placed in a conventional sintering oven. A high temperature ceramic base plate, initially placed on the build tank piston may be used for picking up the unsintered loose powder block. After sintering and cooling, the sintered metal block is removed from the oven. Finally, the fabricated part can be extracted by removing the regions that are isolated by the inhibited powder areas.

2.1 Advantages of SIS-Metal

Before presenting the experimental procedure and related results let us first compare the SIS-Metal process with other metal fabrication processes in order to fully understand its unique properties and related advantages.

Metallic parts fabrication based on metal casting and powder metallurgy (P/M) (Kalpakjian and Schmid, 2006) requires tooling construction. However, the construction of such tooling can be expensive and time consuming, especially for small quantities of parts or parts with complex geometry. In comparison, AM processes can be much faster and less expensive for such small lot or complex part fabrication.

Currently, many AM processes have been developed for building metallic parts, such as SLS, 3D printing (3DP), fused deposition modeling (FDM), direct metal laser sintering (DMLS), laser engineered net shaping (LENS), and electric beam melting (EBM). These processes can be classified based on the usage of binders and the sintering approach as shown in Figure 3:

- **Usage of binders.** Some AM processes such as SLS, 3DP, and FDM utilize certain binders in the fabrication of green parts. For example, the SLS process uses polymer-coated metal powder (Wohlert et al., 1996; Pease, 1998); the 3DP process deposits droplets of liquid acrylic copolymer binder onto a bed of metal powder (Sachs et al., 2000; Pease, 1998); and the FDM process uses filaments that are made by a mixture of binder and metal powder (Greul et al., 1995). The binders in the fabricated green parts can then be removed in a thermal debinding step. In comparison, AM processes such as DMLS, LENS, and EBM can directly sinter metal powders layer-by-layer using high-power energy sources. For example, the DMLS process can directly sinter metal powder without using polymer coating (Simchi et al., 2001; Kotila et al., 2001; Behrendt and Shellabear, 1995). The LENS process directly produces metal parts from metal powder injected by a powder delivery nozzle (Atwood et al., 1998). The EBM process can also directly melt metal powder in a controlled atmosphere chamber using an electron beam.

- **Sintering approach.** Based on how metal powders are sintered into finished metallic parts, AM processes can also be classified into bulk sintering and layer sintering. In the AM processes based on bulk sintering of metal powders (e.g. SLS, 3DP, and FDM), green parts are fabricated first and are then transferred to a sintering oven. In comparison, in the AM processes based on layer sintering of metal powders (e.g. DMLS, LENS, and EBM), powders are sintered into the predefined geometry layer-by-layer. As discussed before, bulk sintering can have advantages such as lower hardware cost, reduced deformation, and higher building speed.

Based on such a classification the SIS-Metal process is uniquely positioned among all the metallic part fabrication processes because it is a layered fabrication process which is based on the bulk sintering approach without using any binder. As shown in Figure 3, the SIS-Metal process is classified in the same category as the traditional powder metallurgy process. However, as no tools are required, the SIS-Metal process may be regarded as a moldless powder metallurgy process.

The SIS-Metal process has the following advantages:

- The hardware of the SIS-Metal process can be inexpensive. A green part can be fabricated using a print head to deposit the inhibitor solution to powder layers; the green parts can then be sintered in a conventional sintering oven, which is widely available in a typical powder metallurgy manufacturing facility.
- The SIS-Metal process is potentially fast. The inhibitor can be deposited using a multi-jet print head, which has proven performance and impressive speed in other applications such as two-dimensional (2D) printing. The printing time can be further reduced using a vector printer, because in most cases the inhibitor needs to be deposited as a thin line only along the boundary of each layer profile.
- The SIS-Metal process can have less shrinkage and deformation than no de-binding is involved.
- Unlike LENS or DMLS, no complex supports are required in the SIS-Metal process since overhang features are supported by powder volumes underneath.

Consequently, the development of the SIS-Metal process is important for future metallic part fabrication.

### 3. Experimental procedure

A three-axis prototype machine has been developed as shown in Figure 4. In the machine a single print nozzle with an orifice size of 0.005" (0.127 mm) is used. The large orifice size of the nozzle allows the trial of different inhibitors at different viscosity levels. The printing nozzle used has an electromagnetic solenoid valve for droplet printing. The deposited...
droplets from the nozzle are 300 nl and are dispensed with a frequency of 1,500 HZ. The nozzle moves in the X- and Y-axes by stepper motors with a continuous speed of 31 mm/s. A heater has been incorporated for heating up every layer after printing the inhibitor. The heater can heat up every layer up to 170°C to evaporate the liquid in the inhibitor solution.

Metal powder
Bronze powder has been the choice of metal powder in this research because of its relative ease of sintering and because of the popularity of bronze parts. Mixed, partially alloyed and fully alloyed bronze powders have been investigated for use in this research project. Sintered parts with fully alloyed bronze powders showed the best mechanical properties and the least shrinkage. In addition to copper (about 90 percent), the bronze powder used in our experiments has the following chemical compositions:
- Tin: 10 percent.
- Lead: 0.025 percent.
- Zinc: 0.04 percent.
- Iron: 0.058 percent.
- Phosphorous: 0.085 percent.

Metal powders with different mesh sizes and shapes have also been examined. The finer the grain size, the better the sintering process will be. However, if the grain size is too small it would be difficult to spread the powder as a uniform layer. The most suitable grain size was observed to be 325 meshes or 44 μm. The shape of metal powder is another important factor. The density of green parts is directly related to the packing density of the powder bed. The packing density of spherical powder is better than other shapes. Therefore, spherical fully alloyed bronze powder with 44 μm grain size was used in this study. The powder particle size has the following distribution:
- 325 Mesh (44 μm): 98.7 percent.
- 200/325 Mesh (44/75 μm): 1.3 percent.

Inhibitor selection
In our study, salts have been chosen as the best inhibitor candidates, since salts are usually inexpensive and safe for both human and environment. Salt crystals can be easily and quickly formed from the printable salt solution state. The following important factors need to be considered in choosing an appropriate salt candidate:
- the melting temperature of salt crystals must be higher than the sintering temperature of metallic powder;
- the initial salt crystal size must be significantly smaller than the size that is yielded under early sintering temperature of about 170–200°C;
- molecular mass of the salt solution and its surface tension should be small such that it can have sufficient penetration in metal powder; and
- the salt solution should have a high solubility for nucleating a higher amount of crystals from a droplet.

Accordingly, the best inhibitor was identified to be aluminum sulfate. The properties of aluminum sulfate are shown in Table I. The inhibitor is achieved by solving 32 g of aluminum sulfate anhydrous in 130 mL of water at room temperature with 30 mL of isopropyl alcohol. Isopropyl alcohol is added to the inhibitor in order to break the surface tension of the solution so that the inhibitor can easily penetrate the printed sections.

Green part fabrication procedure
The 3D CAD model of the part to be built is first sliced in a set of layers with equal thickness. Depending on the part geometry, the layer thickness in our tests varied from 0.25 to 1 mm. For the parts shown in Figures 7 and 8 the layer thickness is 0.5 mm. For every layer the platform of the SIS prototype machine goes down a layer thickness to accommodate the powder for the next layer to be built. After the metal powder is spread, the print nozzle which can move along both X- and Y-axes prints the inhibitor on the sections to be prevented from bonding by sintering. The inhibitor is also printed on the boundary of the part to create solid container around the area that contains the 3D part. After inhibitor printing is completed the heater heats up the layer to dry up the inhibitor solution. As shown in Figure 2, the process then repeats for other layers. After all layers are built the fabricated green part is transferred to a sintering furnace. The sintering path in the oven is discussed as follows.

Sintering path
The sintering happens in an inert environment with argon gas. Various sintering paths have been tested. A sintering path that yields reasonably small shrinkage is shown in Figure 4 where the part is first sintered at 600°C for 20 min;

<table>
<thead>
<tr>
<th>Molecular formula</th>
<th>( \text{Al}_2(\text{SO}_4)_3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molar mass</td>
<td>342.15 g/mol (anhydrous)</td>
</tr>
<tr>
<td>Melting point</td>
<td>770°C</td>
</tr>
<tr>
<td>Solubility in water</td>
<td>31.2 g/100 mL (0°C)</td>
</tr>
<tr>
<td></td>
<td>36.4 g/100 mL (20°C)</td>
</tr>
<tr>
<td></td>
<td>89.0 g/100 mL (100°C)</td>
</tr>
<tr>
<td>Solubility in alcohol and acids</td>
<td>Slightly soluble in alcohol and dilute mineral acids</td>
</tr>
</tbody>
</table>
the temperature is then increased to 825°C to sinter the part for 30 min; finally the part is slowly cooled down. A two-stage sintering process may also be used in which case the part would be removed from the oven before being fully sintered; after separating the inhibited area the part can then be put back to the oven for being fully sintered. Note that during the sintering process there would be shrinkage distortion due to the different shrinkage rate of the printed and un-printed sections. However, such difference would be small due to the fact that in most instances only the boundary of the part’s cross-section is treated with the inhibitor, not the whole volume of the unwanted sections (Figure 5).

4. Experimental results

An inhibition test using aluminum sulfate is shown in Figure 6. The solution of aluminum sulfate is first printed into the powder bed (Figure 6(a)). After sintering the sections with printed inhibitors are very brittle and easily be broken away, while other regions that do not contain salt are sintered properly with good strength (Figure 6(b)). Two more examples of fully sintered bronze parts made are shown in Figures 7 and 8. Note that the print nozzle used in our testbed has a low resolution (~0.13 mm). Accordingly the lines on the parts are relatively coarse. As illustrated by other commercial processes such as 3DP, improving the nozzle resolution can lead to drastically better surface quality.

The properties of the bronze part shown in Figure 7 have been determined and listed tested in Table II.

Figure 5 A sintering path used in the experiments

Figure 6 An inhibition test using aluminum sulfate

Notes: (a) Before sintering; (b) after sintering

The remainder of this section is devoted to the discussion of the underlying sintering inhibition mechanism in the SIS-Metal process. To the best of our knowledge, most researchers in the field of powder metallurgy focus on methods to “enhance” the sintering process through such methods as development of sintering additives. No research has been found that focuses on the ways of effectively inhibiting metal sintering. In German (1994) it is briefly mentioned that powder oxidation, adsorbed organic films, and the presence of surface coating like silica have undesirable impact on sintering. However, methods of imposing such effects have not been presented. In the field of ceramics Smith and Yanina (2002) reported on inhibition of sintering of aluminum oxide powder in the heat intensive process of producing this powder. These researchers demonstrated that the use of surface dopants/modifiers (magnesium, phosphorus, and iron) can inhibit low-temperature sintering in nanocrystalline $\alpha$-Al$_2$O$_3$, hence yielding fine aluminum oxide powder (instead of the unwanted sintered chunks of this ceramic).

Various tests have been performed in this research in order to understand the inhibition mechanism in the presence of printed salt in metal powder. A scanning electron microscope (SEM) is used to examine the interactions between metal powder particles and salt crystals in the SIS-Metal process. The SEM pictures of the bronze powder with and without salt, and also before and after sintering are shown in Figure 9. It should be noted that the sample without salt was not fully sintered so that the related SEM image could be produced. Based on these SEM pictures it can be observed that the
formation of salt crystals plays a big role in preventing metal particles from being properly fused in the course of sintering.

To explain the inhibition phenomenon as shown in our experimental results, we present two inhibition mechanisms as follows. We believe both of them contribute to the results as shown in Figure 7.

(1) The separation of metal powders due to the expansion of salt crystals into decomposed particles

During the SIS-Metal process the salt solution is first printed in the selected areas of metal powder. In the presence of moderate heat the water content of the salt solution evaporates; hence, salt crystals form between the metal powder particles in the printed area (Figure 9(c)). When the green part is moved to the sintering furnace in the early stages of sintering, the inorganic crystal salt, which has high melting temperature, rapidly expands in size when heated. The expanded crystal salt then starts to decompose at a temperature around 500°C (Tagawa, 1984; Apte et al., 1988; Pelovski et al., 1992). The decomposition of aluminum sulfate during the sintering process is shown as follows:

\[
\text{Al}_2(\text{SO}_4)_3 \rightarrow \text{Al}_2\text{O}_3 + 3\text{SO}_3
\]
\[
2\text{SO}_3 \rightarrow 2\text{SO}_2 + \text{O}_2
\]

The decomposed salt particles create a gap in the boundary due to their volumetric growth in the transformation process. Therefore, the affected metal powder particles in the printed sections can be prevented from proper sintering at the sintering temperature of metal powder. To observe the extent of expansion of aluminum sulfate (our choice of salt) an experiment was performed in isolation from metal powder. In our experiment with aluminum sulfate it was noticed that salt crystals decompose and grow rapidly during the heating process. As shown in Figure 8, the volumetric size of the salt sample is almost quadrupled based on the measured dimensions.

We believe the rapid crystal expansion causes internal stresses in the decomposed salt crystals. The stresses eventually fracture the crystals into fine ceramic particles. In the process of rapid expansion, the fine ceramic particles could then attach to the metal powder particles in the neighboring areas. Obviously, the quantity of these crystals is an important factor for the effectiveness of sintering inhibition. Depositing a higher amount of salt solution means putting more ceramic particles between metal powder particles; hence better separation can be achieved. However, too much inhibitor deposition can negatively impact the resolution of the fabrication process.

After sintering, a discoloration of the metal surface is observed. This discoloration is due to metal oxidation. Since sintering happens in an inert environment, the oxygen must come from the decomposition of aluminum sulfate as shown in equation (1). This is also verified by the fact that metal surface is always adjacent to thin layers of aluminum oxide. Both aluminum oxide and the very shallow metal oxide can easily removed by sand blasting, after which the surface becomes quite shiny. This proves that only the part boundary sections near which aluminum sulfate has been printed are oxidized; the part core which is not affected by decomposition of aluminum sulfate never gets oxidized.

(2) Ceramic coating of metal particle

As noted before, aluminum sulfate starts decomposing at 500°C and fully decomposes at around 800°C. Another property of aluminum sulfate which becomes important here is that its melting point is around 760°C.
Therefore, when the furnace temperature is over the melting point of aluminum sulfate the remaining undecomposed salt crystals melt before they fully decompose to aluminum oxide. Hence, molten salt uniformly coats some adjacent metal particles. As the temperature rises this salt coating decomposes into aluminum oxide which gives a near-perfect ceramic coating to the affected metal particles. A sample particle which has been subject to such ceramic coating is shown in Figure 10. Note that the metal powder particles as shown in Figure 9(d) are covered with more randomly distributed aluminum oxide particles since those ceramic particles are generated by aluminum sulfate before it reaches its melting point. Since the sintering temperature of aluminum oxide is much higher than that of bronze powders, the metal powder particles that are covered with ceramic particles do not fuse with neighboring particles during the sintering process (Figure 11).

Thermogravimetric test shows that after the sintering process the weight loss of the decomposed aluminum sulfate is nearly 80 percent of its original weight[1]. Energy dispersive X-ray spectroscopy (EDS) analysis has been performed to determine the existence of aluminum oxide on surfaces of metal powders. The analysis on other randomly selected particles dispersed on and between metal particles also showed other substances. For example, a test result on such substances is shown in Figure 12, which illustrates large peaks on magnesium (possibly present in small amount in the metal powder alloy) and sulfur (in the form of sulfur dioxide and sulfur trioxide (Stern, 2001)). These other substances may also contribute to the inhibition effect in unknown ways.

**Discussion**

Sintering is a physically complex phenomenon which results in numerous structural changes for improved
mechanical properties. German (1994) divided the powder sintering process into six stages:
1. inter-particle bonding;
2. neck growth;
3. pore channel closure;
4. pore rounding;
5. pore shrinking; and
6. pore coarsening.

Different stages of sintering depend on various mass transport mechanisms. The transport mechanisms include viscous flow, plastic flow, evaporation-condensation, lattice (volume) diffusion, grain boundary diffusion, and surface diffusion. The initial stage of sintering mainly depends on surface diffusion.

In the SIS-Metal process salt crystal expansion happens at a temperature that is much lower than the sintering temperature of metal powder. The expansion generates inhibitor particles that can also prevent point contacts between neighboring metal particles. Hence, several stages of sintering can be prevented from occurring in the SIS-Metal process. The identified inhibition mechanisms can provide some guidance for future developments of the SIS-Metal process.

5. Conclusion
The SIS-Metal process based on microscopic mechanical inhibition has been investigated. The SIS-Metal process can
provide benefits such as low cost and fast building speed for metallic parts fabrication. Our experimental results have demonstrated that the SIS-Metal process can fabricate metal parts with desirable properties. Based on the experimental results an inhibition mechanism for preventing metal particles from sintering has been identified and described. The inhibition mechanism is based on the formation of salt crystals, expansion and decomposition of salt and subsequent transition to ceramic particles throughout the sintering process. By establishing the basics of the process this paper guides future researchers toward process improvement through attainment of higher accuracy and fabrication speed, and investigation of applicability of the process to other metal powder materials.

Note

1 Note that equation (1) shows about 69 percent weight loss but the salt crystals used in this experiment originally had a significant water content.

References


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