A Vacuum Furnace Process for DTM's RapidSteel 2.0 Material

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Abstract

The work described in this paper investigates the possibility to use a vacuum furnace in DTM's RapidTool LR process. This alternative process route is brought about to allow the usage of a more common type of furnace than the one recommended by DTM.

Based on the specified, well established H_2/N_2 furnace processing, a similar procedure, adapted to conventional cold wall vacuum furnaces has been developed and tested. The testing is described in detail, and the results so far are promising enough to justify the practical usage of this process, and more of in-depth analysis of all the various aspects of the procedure.

Introduction

The field of Rapid Prototyping (RP), or Solid Free Form Fabrication (SFF), has gone through quite a rapid development on its own during the last decade. From crude concept models made of brittle polymer material to more and more production like objects from a variety of materials including ceramics and metals. A major challenge has been to extend the application of RP-technology into the field of RT, -Rapid Tooling, thus a wide array of RT processes has been developed, all with their own limits and benefits. Among the most widely used of those is the Rapid Tool LR process from DTM Corp. Austin, TX.

The Rapid Tool LR process is an indirect SLS based process, where a powder material, RapidSteel 2.0, is processed in the Sinterstation system, followed by two subsequent furnace cycles. During the SLS stage a scanning laser melts the polymeric binder in each powder layer, thus gluing the metal powder together forming a fragile, but solid mass called the "green" part. The green part is placed in a crucible and sintered, at 1120°C in a 30/70 H_2/N_2 furnace atmosphere, forming a rigid but porous object called the "brown" part. A second furnace cycle at 1050°C in the same atmosphere infiltrates the part with bronze giving a final, ideally, fully dense part.

The specified furnace atmosphere with 30% H_2 requires a specialized furnace that in many cases has little other use. Vacuum furnaces, on the other hand, are often used in various heat treatments and brazing operations, and could in many cases, be available to use as an alternative. This paper describes the adaptation of the Rapid Tool LR process for processing in vacuum furnaces.

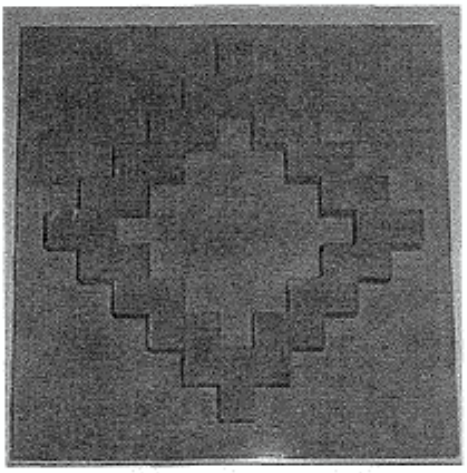

Fig. 1.
Standard sample part used to determine the scaling factors for the whole RapidTool LR process, from SLS processing to completed infiltration. The same geometry was used during the evolution of the vacuum furnace process. This picture shows the infiltrated sample 3.

Our Approach

Vacuum furnaces are used in conventional powder metallurgy to "maintain a clean reproducible, and controlled non reactive atmosphere" [4], especially for "corrosion resistant materials (stainless steels), vacuum is the most reliable atmosphere"[4]. The RapidSteel 2.0 powder metal is stainless steel, and our hypothesis was that it would process well in a vacuum furnace.

Most sintering practices in the powder metallurgy industry are set by trial and error techniques, but since there is a designated process at hand in this case, it is reasonable that a similar process could be deducted from the effect given by the H_2/N_2 atmosphere during the process and comparing that to the likely effect of vacuum.

Atmosphere: According to [1], the "hydrogen reduces any oxides in the surface of the iron powder and aids in infiltrating the bronze into the part". The nitrogen is simply inert, (and to a small extent dissolves into the metal, thus adding a hardening effect). Apart from chemical activity, the atmosphere gas is also an important medium for heat distribution by means of convection. That is obviously not the case with vacuum, and this difference should be accounted for in the settlement of times for the different process steps.

Temperatures: DTM has settled suitable temperatures for sintering (1120°C) and infiltration (1050°C). Ramp rates are mainly set by the equipment's (crucible and alumina plate) sensitivity to thermal shock, and a maximum ramp is given to 180°C/h. These values are determined by material properties and there are no reasons to make any changes at this stage.

Chemical activity: In DTM's standard furnace atmosphere, hydrogen reduces metal oxides,–and the better reduction, the better sintering and infiltration. A similar effect can be obtained in a vacuum furnace too; by lowering the partial pressure of oxygen in the equilibrium: $MeO \leftrightarrow Me + O$.

Thus, the higher vacuum, the better reduction, and the better sintering and infiltration. But high vacuum at high temperatures would not only evaporate the binder, but also risks to evaporate some alloying elements in the metal powder. In relation to that, it can be justified to assume that the organic binder would evaporate at a far lower temperature and pressure than the alloy components. In the H_2/N_2 atmosphere, burnout temperatures are between 450-650°C. Therefore, for the most likely success; -find the highest vacuum that, with reasonable security, does not evaporate the alloying elements.

The activity of any chemical reaction that takes place in the crucible is dependent of temperature and chemical composition. Since the proportions of the alloying elements are given within rather wide ranges, all calculations will be coarse and all resulting values must include large safety margins. The alloying component with the highest gas pressure at process temperatures gives the lowest possible gas pressure in the vacuum furnace, i.e. the highest vacuum acceptable.

Calculation

The Rapidsteel 2.0 powder is an iron based steel powder. According to [2] the alloying elements are ;

Chromium:12 - 30%

Molybdenum: 0 - 7%

Nickel:..... 0 - 35%

Manganese:... 1 - 4%

Given that

p: gas pressure; atm

ΔH_m: enthalpy for the phase transformation (s) $\rightarrow$ (g); cal

T: temperature; K

R: constant = 1.987 cal $deg^{-1}mol^{-1}$

$dlnp/dT = \Delta H_m/(RT^2) \Rightarrow dlnP = \Delta H_m/R \cdot dT \cdot T^{-2} \Rightarrow \int dlnp = \Delta H_m/R \cdot \int T^{-2}\, dT \Rightarrow$

$lnp_1 - lnp_2 = -\Delta H_m/R \cdot (T_1 - T_2)$, where index $_{(1)}$ is referring to the triple point equilibrium, and index $_{(2)}$ is referring to the process conditions.

For an ideal gas solution, partial pressure of a component = $p_2 \cdot$ (fraction of element) = p_p.

For Cr, in the sintering stage, (fractions according to worst case scenario):

$p_1 = 1\text{atm} \Rightarrow \ln p_1 = 0$ $\Delta H_m = 81.7$ kcal/mol

$T_1 = 2893$ K $T_2 = 1393$ K

$-\ln p_2 = -81.7 \cdot 10^3/1.987 \cdot (2893^{-1} - 1393^{-1}) = -15.3 \Rightarrow p_2 = 2.26 \cdot 10^{-7}$ atm

fraction of Cr; $X_{Cr} = 0.32$; $\Rightarrow p_{p,Cr} = 2.26 \cdot 10^{-7} \cdot 0.32 = 7.8 \cdot 10^{-8}$ atm

In a similar manner (fractions picked according to worst case scenario):

$p_{p,Mn} = 3.98 \cdot 10^{-4} \cdot 0.019 = 7.57 \cdot 10^{-6}$ atm

$p_{p,Mo} = 1.37 \cdot 10^{-16} \cdot 0.0234 = 3.2 \cdot 10^{-16}$atm

$p_{p,Ni} = 1.8 \cdot 10^{-8} \cdot 0.1632 = 2.89 \cdot 10^{-9}$ atm

With a calculated partial gas pressure of $7.57 \cdot 10^{-6}$ atm, manganese is the critical component..

A reasonable safety margin gives $\Rightarrow$ set point value $49.3 \cdot 10^{-6}$ atm, or in a common scale among vacuum furnaces; $50 \cdot 10^{-3}$ mbar.

Experimental

The experimental furnace cycles were run in a conventional cold wall vacuum furnace at Järfälla Härdverkstad AB, Järfälla, Sweden. Green bodies were manufactured following recommendations according to DTM's "Guide to Materials: The Rapid Tool LR Process Using RapidSteel 2.0"[1] and "RapidSteel 2.0 Mold Inserts for Plastic Injection Molding"[3]. Crucible setup was likewise according to recommendations from the same sources, but with one small exception: for increased safety during transportation, the fine grain alumina powder (240 grit) was used for both the infiltration and sintering cycles.

Sample 1. *Sintering:* The standard setup, ramp rate (180°C/h) and sintering temperature (1120°C) according to [1] was used. Since no other mechanism of heat transfer than radiation is active in vacuum, the dwell time at sintering temperature was prolonged from 3 hrs [1] to 3.5 hrs. For practical reason was the furnace allowed to cool down in its own pace from 500°C. Furnace temperature and pressure was monitored during the whole cycle.

The sintering result was quite satisfactory regarding strength, shape accuracy and sharpness of edges. However due to the fact that the equipment (crucible, bricks etc.) had not previously been subjected to a vacuum furnace cycle, some unaccounted gas was released during the furnace cycle, and it was not possible to draw any conclusions from the furnace atmosphere pressure.

Sample 1. Infiltration: The standard setup for solid bar bronze was used. The temperature (1050°C) and ramp rate (180°C/h) was set according to [1]. For reasons mentioned earlier the dwell time at 1050°C was prolonged from 2 hrs [1] to 2.5 hrs.

The infiltration could be termed "half successful", meaning that only half the bronze was infiltrated. Two infiltration plates had at some time lost contact with the sample and no infiltration was possible from those plates.

Sample1. Conclusion: The sintering cycle seemed good enough, so no further modifications were necessary at this stage. Instead securing the contact between the sample and the plates was the primary aim for the next sample.

Sample 2. *Sintering:* The setup, ramp rate, sintering temperature and dwell time was identical to sample1. To secure the best possible contact between infiltration plates and sample, they were sintered in direct contact with each other. Identical, still quite satisfactory results as for the previous sample1. The plates had bonded to the sample thus forming one large object with the plates stuck like wings to the sides.

Sample 2. Infiltration: Standard setup, and the same procedure as previous sample. All infiltrant bars were infiltrated, but still the result could only be termed a partial success. Each bar left a "foam" of nickel rich residue on the plates.

The sample was measured and scaling factors were calculated in the standard data sheet provided with the RapidSteel powder from DTM. See table1.

Sample 2. Conclusion: Connection between plates and sample secured infiltration, however since the brown specimen could not be weighed separately infiltration success could not be calculated. Still the nickel rich foam suggests that a modification of the infiltration procedure would be necessary to achieve a completely successful infiltration. It is also possible that the prolonged sintering time in combination with the extremely low gas pressure in the furnace has allowed the sample to sinter, and shrink a little bit more than expected.

Sample 3. *Sintering:* The sintering of sample 3 followed the same standard set up as previous samples and the result is also as good as previous samples. From the Time/Temperature/Vacuum-diagram (Fig.2) we find that the Time/Temperature relationship follows the expected program with almost perfect precision. The vacuum likewise behaves as might be expected: after it had reached the set point, it stayed fairly constant with a sharp dip during the binder's evaporation (burnout).

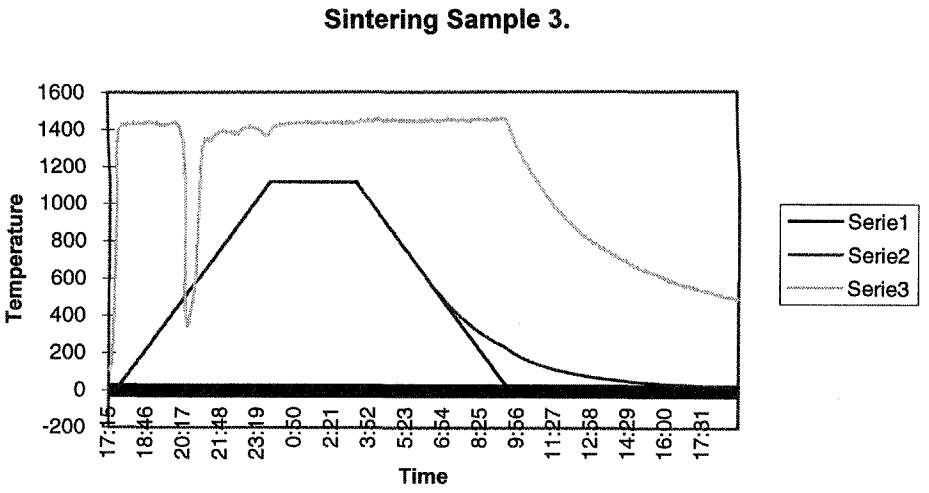

Fig.2.
Sintering of sample3. Temperatures in Celsius, "serie1" gives the temperature set point, "serie2" gives the actual temperature and although no scale values are given in the figure "serie3" indicates the vacuum. Notice the dip in the vacuum between 443°C and 586°C. Apparently, that is the interval where the binder evaporates under these circumstances. The fact that there no other dip is indicated verifies that no alloying elements are being evaporated.

Sample 3. Infiltration: To avoid the same problems as were experienced with the previous samples, some modifications of the procedure was necessary. The joints between the infiltration plates and the sample were powdered with some loose powder. If any vibration would separate the plates from the sample, the void would still be filled with RapidSteel powder thus ensuring free flow of the infiltrant. Since this powder was not weighed together with the plates and sample when the mass of infiltrant was calculated, and in order to assure that there would be enough bronze, 50g extra bronze was added to the calculated amount. To ensure that the nickel rich phases would melt and infiltrate with the rest of the infiltrant, a recommendation from [3] was followed and the temperature was ramped up at a rate of 120°C/h. The furnace was kept at infiltration temperature (1050°C) for 3hrs and lowered, as the previous samples, at a rate of 180°C/h.

This time infiltration was quite satisfactory apart from the fact that the color of the sample was not completely even, and that there still was small amounts of infiltrant remaining on the plates. Still, according to the calculation of the infiltration in [1] and [3], the amount of bronze infiltrated should equal 85% of the mass of the brown part. Since the mass of this brown part was 1360g, $\Rightarrow 1360 \cdot 85\% = 1156$g, and, 1360 + 1156 = 2516g. Hence 2516g is the theoretical mass of the full density object. The actual mass of the infiltrated sample 3 is 2513g, that gives an infiltration success of 99.8%, which we consider satisfactory.

Similar to the previous sample, the offset and scaling factors were calculated. See table 1.

The Time/Temperature/Vacuum-diagram (Fig 3,) shows a stable behavior, where the temperature follows time according to program, and vacuum keeps around the set point.

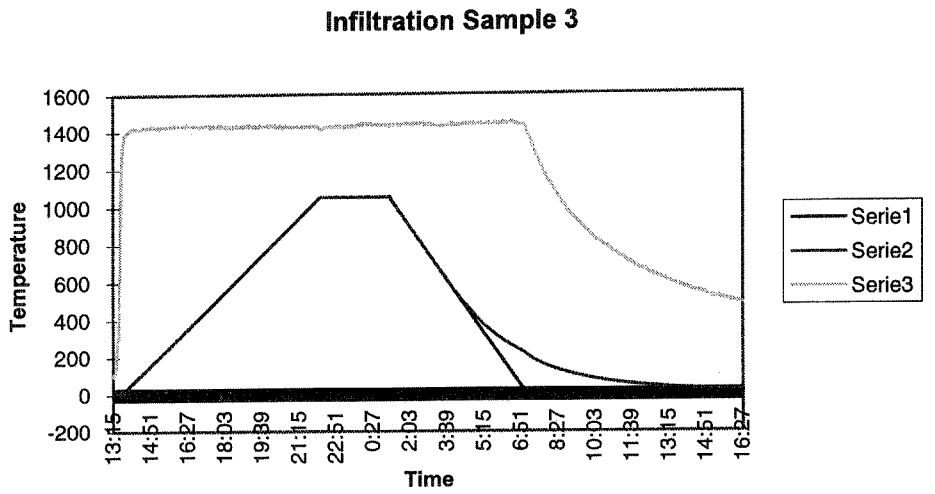

Fig.3. *Infiltration of sample 3. Temperatures in Celsius, "serie1" gives the temperature set point, "serie2" gives the actual temperature and "serie3" gives the vacuum all identical to Fig.2. The decreased ramp rate has given a longer warm up time. The vacuum stays at the set point during the whole cycle, which verifies that there is no evaporation of any alloying components (or anything else).*

Sample 3. Conclusion: The process used for sample 3 gives satisfactory results for DTM's RapidSteel 2.0 material and could be useful to manufacture functional molds.

Table 1.

	X offset	X Scale	Y offset	Y scale
Sample2	0.0070	1.019	0.0086	1.0133
Sample3	0.0041	1.0173	0.0070	1.0130
H2/N2*	0.1825	1.0072	0.1603	1.0001

**) Scaling and offset values for the conventional H_2/N_2 furnaces have been supplied by courtesy from DTM GmbH, Germany. All individual machines have different scale and offset values, especially between Sinterstation 2000 and 2500(+) there are big differences. In addition to that, comes the difference between individual furnaces. In this case however, all objects, Swedish and German, have been SLS processed on Sinterstation 2000s, and even with the individual differences accounted for, there still remains a significant difference in scaling factors. This must be referred to the difference in furnace processing. Apparently the shrinkage is larger with the used vacuum procedure. Among other things this means that the value of infiltration success in reality is higher than the calculated value of 99.8%.*

Conclusion and Further Study

This investigation has showed that it is possible to process DTM's RapidSteel 2.0 material to satisfactory objects in a vacuum furnace process. Little experience of this process has been accumulated so far, and more work is required to fine tune every aspect of a large number of variables. For example: it is likely that the prolonged dwell time at sintering temperature introduced in the sintering cycle for sample 1, in combination with low the gas pressure in the pores, (vacuum) could have led to higher shrinkage, higher sintering density and more closed pores which could not be infiltrated, which in turn led to the unevenness in color and the comparably large scaling factors in the infiltrated sample 3.

This effect could possible be turned into something very positive. By making use of the enhanced sintering of liquid phase sintering it could be possible to sinter full density objects in a single furnace cycle. During sintering the "liquid phase provides a high diffusivity pathway for atomic motion, and a strong capillary force that induces particle compression at point contacts" [4]. This phenomena continues until the connections between the pores are closed and the remaining pores are filled with furnace atmosphere, which is exerting a gas pressure and thus locking further pore closure. Since vacuum furnace atmosphere means virtually no atmosphere, nothing would fill the pores and nothing could lock the pore closure, thus it would be possible to reach full density.

Acknowledgments

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