

SLS MATERIALS DEVELOPMENT METHOD FOR RAPID MANUFACTURING

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Abstract

As soon as SFF technology development began to make Rapid Prototyping possible the interest in Rapid Manufacturing (RM) began to grow. The advantages in terms of functional integration, elimination of tooling and fixtures and mass customization make a compelling case for RM, leading some in the field to call it the next industrial revolution. Yet without the materials properties necessary to provide the function and variety currently available from mass production methods, the application of RM will remain limited. Developing new materials for the SLS process, one immediate step toward a larger portfolio of RM materials, is very challenging. The formation of high quality SLS parts relies on appropriate powder characteristics, thermal cycles and sintering behavior. Based on a brief examination of the key factors in SLS processing and a research project to develop a new binder material for Silicon Carbide composites, a systematic materials development method is proposed in this paper. The method provides guidance for introducing new SLS materials, support for educating new SLS users and researchers and direction for several future research projects.

Introduction

What is most important to RM depends upon the person answering the question, but tends to follow three main themes. Many authors have described the lack of materials and material processing knowledge. Others talk actively about improving the capability of the machines used. While a third caucus places the adoption of rapid manufacturing into engineering practice as the primary driver for future success. Without engaging too deeply in this ongoing debate, each of these is actually critical and it may be more beneficial to consider them as an undivided system of necessary elements rather than separate tasks. It is with this overarching concept that the development of materials is discussed in this paper.

Selective laser sintering, as it is discussed in this paper, is about spreading out a thin layer of powder, fusing certain regions of that layer of powder via a polymeric sintering process driven by the heat of a scanning IR laser and simultaneously fusing scanned regions of the current layer to fused sections of previous layers. The process is repeated until a final part shape is built in thin layers of fused powder. This process might include a single polymer component which is processed into a higher-density solid or the polymer as a binder bonding inert particles together into a stable mass. In either case, the idea is to get an undistorted uniform part from a process that heats and cools the part differently at each point. Doing this requires consideration of the framework defined by the behavior of powdered materials, heat transfer and the details of polymer sintering.

Each of these areas has been the subject of research for many years and some work has been done to assemble prototyping models such as the virtual prototyping model proposed by Choi and Samavedam, 2002. Despite incorporating over thirty variables, the model did not have the capability to represent shrinkage and the associated warping of parts (which come from the sintering process). In addition, the model was focused on the shape of final parts and did not include material properties. Hague et al., 2003, argue, “there is a dearth of information about even the limited number of materials that are available today – materials research is one of the key areas at present.” In terms of pursuing rapid manufacturing this materials research need is even greater. This is a sentiment reinforced by Hopkinson and Dickens, 2003, who postulate, “if the material properties for RP parts were known in detail... then functional parts could be designed to be manufactured by RP processes.” What is certain is that material behavior and properties within the SLS process should be better understood and that the lack of materials currently available is a very significant limitation in terms of any future rapid manufacturing using SLS technology. Still, even the current economics show potential for RM. While Pham and Dimov, 2003, echo the traditional industry perspective that SLS prototyping is cost effective when less than 10 parts are produced, the widely cited work of Hopkinson and Dickens, 2003, indicates something significantly different. They found that for a certain test part current SLS technology is capable of breaking even with injection molding (in cost per part) at nearly 14,000 units. The costs and most critical operational variables of Rapid Manufacturing are different from Rapid Prototyping.

Essentially, there is a need for more commercially available SLS materials and there is also a need for greater understanding about the link between the process and material properties associated with the SLS process. Capturing the full complexity of the SLS

process is beyond the scope of this paper and significant work remains to understand it completely, but many new materials can be developed now. With some specific research projects a more general materials development process can be developed that will evolve with improvements in machine development and wider adoption of RM in product manufacturing.

Development Cost, Sintering, Heat Transfer and Powder Processing

Developing new materials for SLS machines is costly. To fill an SLS machine with the current industry-standard Duraform powder costs several thousand dollars. Polymer companies typically produce test quantities either below 10lb. (too little to test in an SLS machine) or above 1 ton (a large amount before the SLS processing traits are understood). Few polymers are produced directly as powders and must be ground and classified to appropriate particle size distributions. Long-term changes in material behavior and machine operating conditions must be characterized if a material is to be used commercially.

The process of powder sintering has been researched for several decades and laser sintering for a period approaching its second decade. There has been significant work related to powder metallurgy and reactive ceramic formation. More recently, powder coating processes and interest in rotomolding has driven some work in polymer sintering and several models of the densification kinetics of polymeric compacts have been proposed in literature. Models have followed a bulk densification approach on one hand and a more micro-scale approach focusing on the details of coalescing adjacent particles. From these detailed models, numerical methods have been used to explore the behavior of compacts having ideal geometry. As a more practical example, Kontopoulou and Vlachopoulou, 2001, identify two main phases of polymer sintering; the development of interfaces and bridges between particles and the subsequent shrinking of voids with further densification. Their paper reviews three separate densification models for polymer sintering.

A simpler and more widely used continuous media approach is discussed by Kolossov et al., 2004 in terms of sintering under a scanning laser. In this approach the concept of a 'sintering potential' is substituted for the complexity of the formation of interfaces and bridges as well as the division of the sintering process into separately modeled phases. The sintering potential, $\phi(\bar{x}, t)$, of loose powder is '0', while that of a fully dense solid is

'1'. It is well known that sintering processes are driven by surface tension, $\gamma(T)$, and resisted by viscosity, $\mu(T)$. By using a linear model for the surface tension of the form, $C_\gamma T + \gamma_0$, and a step function for the viscosity across the melt temperature of a polymer, as suggested by the work of Wouters and de Ruiter, 2003, it is possible to modify the model proposed by Kolossov, et al., 2004 into the form shown as Equation (1). In this expression the linear model of surface tension is integrated over the 'i' time increments spent above the melt temperature of the polymer, ' t_{mi} ' as a parameter in a ' $(1 - e^{-x})$ ' s-curve form. Since lasers are often scanned across the surface with subsequent passes within the beam width a point in the powder is often heated by 5 or more passes, each with different and possibly overlapping thermal cycles. While the internal stresses and warping associated with actual parts is not a part of this model, the dependence of density upon cumulative melt history is important. It also provides an explanation for lower material density at edges parallel to the scanning direction since fewer passes heat the powder at these edges. Sintering potential is increased at edges perpendicular to the laser scanning direction since the process of the laser arriving, moving along the edge and then returning in the opposite direction exposes these edges to a longer, more intense heating cycle. Further, scanning a rectangular region in different directions yields a different layer density. In summary, there is a reasonable understanding of how heat cycles contribute to density, but there are not models currently available that capture the details of material properties that could shed light on shrinkage or strength. The next section describes heat transfer models for exploring the temperature cycles, $T(\bar{x}, t)$, seen within the powder.

$$(1) \quad \phi(\bar{x}, t) = 1 - \exp\left(-\frac{1}{\mu_{liq}} \sum_{t_{mi}} \int C_\gamma T(\bar{x}, s) + \gamma_0 ds\right)$$

Ion, et al, 1992, describe a model of bulk metal temperature under a scanning laser that is useful for the present discussion. It is shown as equation (2), where 'A' is absorptance 'a' is thermal diffusivity, 'q' is beam power, 'v' is beam speed and 'k' is thermal conductivity while 't₀' and 'z₀' are reference parameters of characteristic time and length respectively used to capture actual data. Equation (3) indicates that 't₀' is related to the beam radius and the thermal diffusivity of the substrate material. 'z₀' on the other hand is to be set by matching the peak temperature of the equation to the observed temperature at

the *actual* surface of the powder. Kai and Shaw, 2004 offer a means of extending this temperature model to powdered substrates, shown in Equations (4) and (5). Their expression for the effective thermal conductivity for a powder bed, ‘ k_{eff} ’ relates the respective conductivities of the solid powder ‘ k_s ’ and interstitial fluid ‘ k_f ’ as well as the radiation from one particle to another within the powder bed ‘ k_r ’. ϕ is the fractional porosity and x_r is the average particle radius.

$$(2) \quad T - T_0 = \frac{Aq}{2\pi k\nu\sqrt{t(t+t_0)}} \exp\left[\frac{-(z+z_0)^2}{4at}\right]$$

$$(3) \quad t_0 = \frac{r_B^2}{4a}$$

$$(4) \quad \frac{k_{eff}}{k_f} = (1 - \sqrt{1 - \phi}) \left(1 + \frac{\phi k_r}{k_f}\right) + \sqrt{1 - \phi} \left(\frac{2}{1 - \frac{k_f}{k_s}} \left(\frac{1}{1 - \frac{k_f}{k_s}} \ln\left(\frac{k_s}{k_f}\right) - 1 \right) + \frac{k_r}{k_f} \right)$$

$$(5) \quad k_r = \frac{4}{3} \sigma T^3 x_r$$

For the binder development research for silicon carbide composites described in Evans, 2005 and Evans et. al, 2005, the heat transfer model described above was used to assess the expected thermal cycles. The effective thermal conductivity of 320 grit silicon carbide powder (mixed with 10% phenolic binder) whose bulk conductivity is 110 W/m-°C was found to be 0.29 W/m-°C. It is interesting to note that the thermal conductivity of bulk nylon is 0.3 W/m-°C while the conductivity of Duraform powder is estimated by the above model as 0.07 W/m-°C. Powder conductivity is dominated by the interstitial fluid. By putting a 49.5 in/sec beam speed, 10W power, a 450 μ beam width and an estimate of the peak temperature into the temperature model in Equation (2) the graph shown in Figure 1 was created. What is clear is that a layer of 0.004” would be heated enough to have at least some interlayer adhesion. For a purely polymeric powder it is important that an appropriate viscosity be reached within perhaps 40-50°C of the melt point, depending of the surface tension of the polymer, so that significant densification can occur within the brief thermal cycle. It is also clear that there is significant work remaining to completely capture the behavior of a three-dimensional polymeric heating process. Changing specific heat, density, transmissivity, and conductivity greatly complicate the standard heat equations.

Similarly, the intimate details of powder behavior in terms of relating temperature dependent surface interactions with the properties of a powder bed present challenges for the future. The basic behavior of powders and powder mixtures has been well characterized by German, 1989. This includes relationships between size, distribution and shape to density and other powder characteristics. In terms of testing powder for use in the SLS machine, small cylindrical glass jars can be easily used to examine the tap density and basic flow behavior of powders. In addition, current SLS materials can serve a benchmarks for this examination. Basically, polymeric powders work best as low surface area (medium to high sphericity) powders with few particles either above 80μ or below 10μ . Binders, on the other hand are more efficient as smaller particles, even 1μ size or less. The ultimate key is in terms of actual bulk powder behavior.

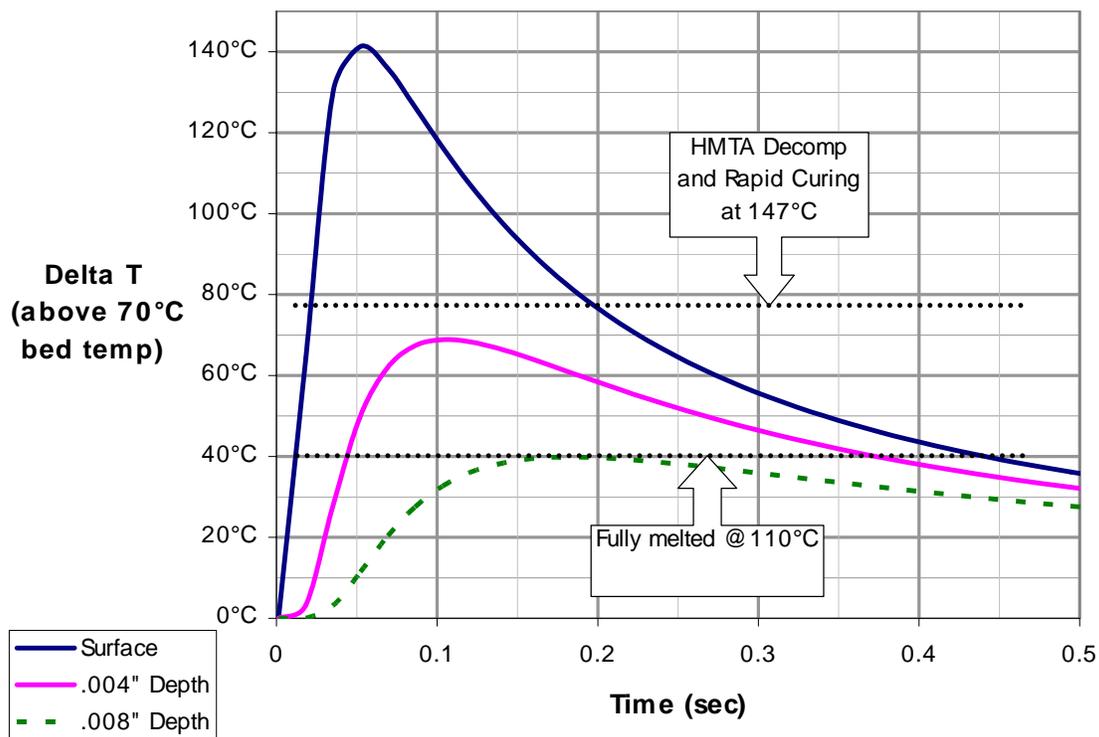


Figure 1: Example Temperature Cycle Graph (SiC powder)

There are basic models available with a range of complexity that describe polymer sintering. However, the details of material stresses and other final material properties cannot be readily related to processing characteristics. While a basic sense of thermal cycles can be assembled, a complete three-dimensional model is likely years away. Each of these will be needed to build an analytical representation of SLS. Until then, the

machine itself is its own most useful model. More bluntly, in the words of an SLS operator interviewed during the course of the binder research, “you don’t know much until you just throw it in the machine.” Still, with a few preliminary tests, it is possible to organize the development of new materials into a useful materials development method.

Current Materials Development Method

New SLS materials are often pursued because of interesting end properties. Examples of this include PEEK as a high performance engineering polymer or PLGA for its use as a bio-compatible scaffold for tissue engineering. It is appropriate to assume for the following discussion, that an interesting polymeric material has been identified and there is some desire to examine that material in terms of SLS processing, particularly if some basic cost guidelines can be met. Lets call it material X. The examination of material X,

then, may follow the six-stage diagram shown in Figure 2, to the left. Along the way key questions provide guidance to determine whether further work and expense is necessary or the material is simply not a good candidate for SLS processing.

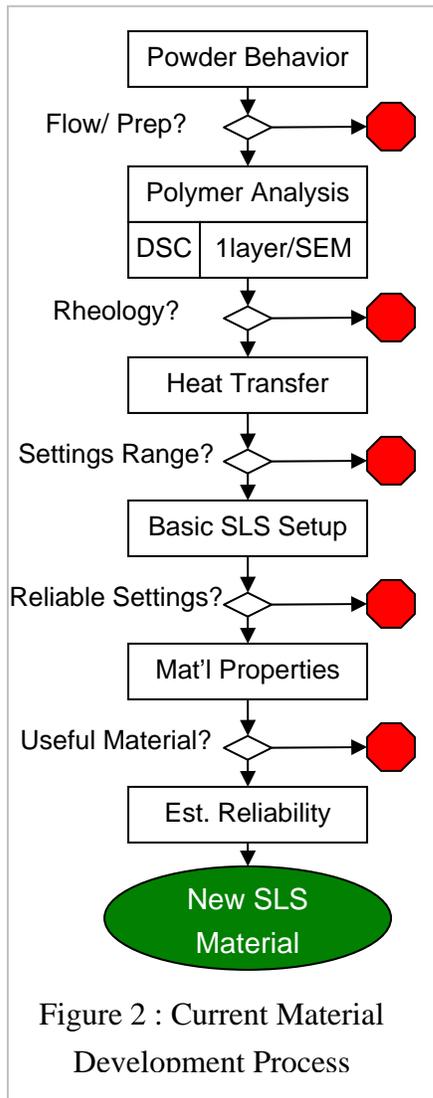


Figure 2 : Current Material Development Process

There are initial questions that can be answered simultaneously. Can material X be readily obtained in a powdered form? Does the melt or softening point of material X lie at 200°C or less (the maximum bed temperature of many SLS machines)? These fall into the first two stages respectively. Assuming that material X can be obtained in a powdered form it is important to assess the flow and tap density characteristics of the powder with respect to current SLS materials. Poor flow or low density will be difficult characteristics to overcome during the spreading of the powder and the densification associated with sintering. If the powder flows more like sand than baking flour and reaches a tap density of about 50%, the first stage is cleared. But, the final verdict will be rendered by the powder spreading mechanism in the actual machine.

Although rheometers are available that would permit the examination of the viscosity near the melt point, the capability has not existed at the University of Texas. SLS materials are routinely tested for melt flow which has been empirically correlated to sintering and sintered powder characteristics. But this method requires the polymers to be at a viscosity low enough to flow readily through an orifice under the pressure of a weighted piston – and thus away from the transition nearer the melt point. This second stage instead uses the SLS machine in conjunction with more readily available electron microscopy to assess sintering behavior. Basically a thin (8"OD, 0.100" depth) circular tray was built in an SLS machine, filled with leveled powder and scanned within the SLS machine. In this way the atmosphere and laser scanning parameters can be directly related to relative strength and observed changes in powder microstructure for a single layer of scanned powder. The question is whether the rheology and recrystallization of the powder is appropriate for high density, high accuracy, low internal stress parts. This stage requires some iteration and knowledge of running current materials. It is possible, as an example, with the right choice of laser scanning parameters and part bed temperature to have a layer of Duraform curl up like a potato chip. The idea here is that with a couple of pounds of powder some initial feel for the material's behavior in the SLS machine and the density and strength of final parts can be assessed.

The third stage of this method, since it is based on currently available models, is simply an assessment of interlayer heating. As shown in Figure 1, the phenolic binder would be taken above its melt point at least one 0.004" layer into the powder. Given the laser parameters explored using the single layer scanning tests of the previous stage, is it likely that strong interlayer adhesion will take place?

And then, the real work begins. The first three stages serve as a low-cost filter for materials in the SLS machine. Going forward the more challenging work of actually building multiple layer parts can begin. Completing this stage might require 50 to 100lb. of material or as little as 5lb. The second stage provided information about the range of part bed temperatures and scanning strategies appropriate for making an accurate, dense single layer. For this stage these serve as a starting point. During the supporting research, several different diagnostic parts were designed that helped to assess the breakout ability, directional strength, part growth and feature detail. This was basically achieved by orienting different walls and grooves along X, Y and 45° directions. Builds during this stage are typically less than one inch in total depth to preserve powder usage.

Again there is iteration through builds and observation of dimensional characteristics and strength. The question being answered by this stage is whether a combination of parameters in the SLS machine can produce reasonable parts. One indicator of this is whether the operator of the machine can run the build without constant surveillance.

With confidence that reasonable parts can be made it is possible to procure a larger amount of powder for longer builds and more challenging part fabrication. The focus of this stage is to improve the part characteristics, look at the longer time scale temperature transients in the machine and increase the build speed of the machine to get a sense of the economics associated with running the particular material. The ultimate question is about material properties. Can parts with desirable properties be reliably made without tending to the machine during the build?

The final stage of the process is the most costly in both time and resources for several reasons. Different SLS platforms operate differently. Some have higher maximum scan speeds that can be used others have different maximum part bed temperatures. The different materials and dimensions of machines alter their thermal transient behavior. A set of parameters that runs well in one machine of the same platform may not run well in another. That set of parameters may not yield high quality parts in the same machine a few days or a week later. In addition very few materials can be processed near their melt points without some changes in their properties. Duraform undergoes solid-state molecular chain growth within 20°C or perhaps more of its melt point. This slowly changes the sintering behavior of the resin and is in part the reason why there is a refresh rate used to maintain part quality in SLS service bureaus. The behavior of material X, if it makes it to this late stage of the game, must be understood in terms of all of the longer-term variability of the machines. In addition, indicators and solutions to the various problems must be established, particularly from the viewpoint of a company that will be supporting either its own staff or other users in the use of the powder for actual customer orders.

There is currently no quick way to examine new materials for SLS processing. However the first three stages of the method described above do provide a relatively rapid and low cost filter for materials before the more intensive work begins within the SLS machine. Each new project does begin to establish a more comprehensive library of potential SLS materials which will better inform future development projects.

Next Steps in SLS Materials Development

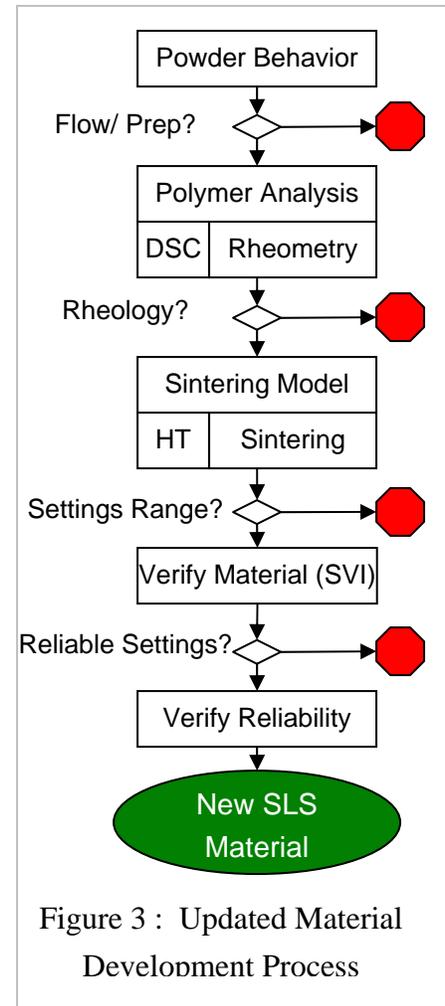
Looking at the challenges associated with the method reviewed above several next steps are clear which facilitate the discussion of a more capable future materials development method. Imagining that this future development method would be built at the University of Texas, the first step would be to obtain the capability to test the thermo-rheology of polymer samples. In this way the appropriate transitional properties of materials could be established and then used for further development.

The next step is related to the development of more sophisticated SLS process models. This involves both heat transfer and the sintering process itself. These two elements would eventually be coupled, but in the interim, thermal cycles within the powder might be used to subsequently estimate the sintering behavior of the powder. As mentioned above these models will be very challenging to prepare and it will likely take many years for them to be developed to a degree where single layer scanning and more empirical materials development steps would be replaced.

The third element going forward has actually been developed before. With an insert in each of the three beds of a 3D Systems SLS machine it is possible to run multiple layer builds using a small fraction of the powder needed to charge the whole machine. This also makes use of the thermal and powder delivery controls of the current machine. One such insert, called the Small Volume Insert, or SVI was designed for a Sinterstation 2000 machine during the binder development research, but was not qualified in terms of its heat transfer characteristics. Additional thermal control or perhaps cooling might be necessary to have a 3" OD stainless steel chamber exhibit similar thermal behavior compared to the entire part bed. It will at least be necessary to establish some type of relationship between the processing characteristics using the insert to those for the regular machine. The driver behind the SVI is twofold. First, some materials are rather expensive. A full machine charge of tungsten carbide powder, as an extreme example, would be many tens of thousands of dollars. Second, as mentioned in the introduction, it is easy to get 10-20lb. of powder, but difficult to get much more as an experimental sample from polymer manufacturers. The use of the SVI matches the initial SLS development to this industry sample limitation.

If all of these developments were applied to materials development creating new SLS materials would be significantly different than the description above at least until it was necessary to verify the longer-term reliability of the powder. It could be arranged into a similar method compared to that described above having 5 stages as shown in Figure 3, to the right.

The initial stage remains largely unchanged, but the second involves a more in depth assessment of the melt and recrystallization behavior of the polymer. If this behavior was appropriate, then it would feed a more comprehensive sintering model coupling laser heating, sintering and the formation of stresses and material properties. The output of this third stage would be appropriate settings for SVI builds within the machine. With verification that the material works under actual SLS processing, the assessment of the longer term effects could then take place. Of course, the greater understanding of the SLS process and its evolution could make the final verification process very short. It may be possible to prepare a new SLS material using perhaps 2 liters of powder.



Conclusion

Work to develop a new thermosetting binder for the SLS process has been generalized into a more general materials development method. The current method can be used to guide materials development as well as support the education of new users in the various parameters and behaviors of the SLS machine. New materials can be developed for the SLS materials portfolio now. However the challenges of the current SLS process, which is focused on prototyping, make this a difficult and time consuming process. By performing a few initial material assessments it is possible to make materials development much easier and less costly. A method organizing these processes has been discussed in this paper.

New research to prepare a model of the SLS process will greatly improve the ability of materials development work to prepare a much wider variety of materials which is needed to support RM capability using SLS technology. It is likely that this development work will advance in stages each becoming a more comprehensive and useful tool supporting SLS technology.

As described at the outset, there are three main pillars for the realization of rapid manufacturing, materials, machine technology and integration of RM into engineering practice. It may be that the central element for all three of these is a greater understanding and modeling of the SLS process. As described above, it will certainly have a significant effect on the development of new materials. It will also inform the development of new machine technologies and may be a medium for greater support of RM in general engineering practice as well. These discussions are left to subsequent papers and additional research.

References

- Choi, S.H., Chan, A.M.M., (2003), "A Virtual Prototyping System for Rapid Product Development", *Journal of Computer Aided Design*, June 25, 2003
- Choi, S.H., Samavedam, S. (2002), "Modelling and Optimization of Rapid Prototyping", *Computers in Industry*, Vol. 47, pp.39-53.
- Dai, K., Shaw, L. (2004), "Thermal and Mechanical Finite Element Modeling of Laser Forming from Metal and Ceramic Powders", *Acta Materialia*, Vol. 52, pp.69-80.
- Evans, R.S., (2005), "Indirect Rapid Manufacturing of Silicon Carbide Composites", *Doctoral Dissertation*, University of Texas at Austin, May 2005
- Evans, R.S., Bourell, D.L., Beaman, J.J., Campbell, M.I., (2005), "Rapid Manufacturing of Silicon Carbide Composites", *Rapid Prototyping Journal*, Vol. 11, No. 1, pp.37-40
- German, R. (1989), *Particle Packing Characteristics*, Metal Powder Industries Federation, Princeton, NJ.
- Hague, R., Campbell, I., Dickens, P. (2003), "Implications on Design of Rapid Manufacturing", *Journal of Mechanical Engineering Science*, Vol. 217, pp. 25-30.
- Hopkinson, N., Dickens, P. (2003), "Analysis of Rapid Manufacturing – Using Layer Manufacturing Processes for Production", *Journal of Engineering Science*, Vol. 217, pp.31-9.

- Ion, J.C., Shercliff, H.R., Ashby, M.F. (1992), "Diagrams for Laser Materials Processing", *Acta Metallica Materialia*, Vol. 40, No. 7, pp.1539-51.
- Kai, K., Shaw, L. (2004), "Thermal and Mechanical Finite Element Modeling of Laser Forming from Metal and Ceramic Powders", *Acta Materialia*, Vol.52, pp.69-80.
- King, D., Tansey, T. (2002), "Alternative Materials for Rapid Tooling", *Journal of Materials Processing Technology*, Vol. 121, pp.313-7.
- Kolossov, S., Boillat, E., Glardon, R., Fischer, P., Locher, M. (2004), "3D FE Simulation for Temperature Evolution in the Selective Laser Sintering Process", *International Journal of Machine Tools & Manufacture*, Vol. 44, pp.117-23.
- Kontopoulou, M., Vlachopoulos, J. (2001), "Melting and Densification of Thermoplastic Powders", *Polymer Engineering and Science*, February 2001, Vol. 41, No. 2, pp. 155-69.
- Pham, D.T., Dimov, S. S. (2003), "Rapid Prototyping and Rapid Tooling – The Key Enablers for Rapid Manufacturing", *Journal of Mechanical Engineering Science*, Vol. 217, pp.1-23.
- Wouters, M., de Ruiter, B. (2003), "Contact Angle Development of Polymer Melts", *Progress in Organic Coatings*, Vol. 48, pp.207-13.