Compensation of Non-Linear Shrinkage of Polymer Materials in Selective Laser Sintering

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Abstract

Inaccuracies in the selective laser sintering (SLS) process using polymer materials are typically caused by inhomogeneous shrinkage due to inhomogeneous temperature distribution in the powder bed of the SLS machine. These shrinking effects lead to stress in the sintered parts, causing the part to distort. The inhomogeneous shrinkage of benchmark parts has been compensated empirically in a former work. The results cannot been transferred to all geometries, because each geometry requires a specific temperature for laser sintering and, thus, has its own related shrinkage distribution. In a new theoretical approach, shrinkage behavior is to be integrated in a thermal simulation of the SLS process and the thermal shrinkage calculated prior to the building process. In the following, experimental data of the temperature- and pressure-dependent shrinkage of laser-sintered powder samples is presented. Possible theories for a physical model of thermal shrinkage are discussed. In particular, these models have to consider granular characteristics such as internal friction, particle sliding, and powder compaction.

Introduction

Today rapid prototyping, rapid tooling and rapid manufacturing processes are counted as established means of accelerating the product development cycle in the automobile and aeronautics industries. Thus we see more and more prototype parts, which in the past were traditionally produced using high-cost, time-consuming milling and joining techniques, being replaced by investment cast parts. And the master forms deployed in investment casting are now typically plastic forms produced by means of selective laser sintering (SLS). In addition SLS processes are used to generate plastic functional prototypes for both the automobile and aeronautics industries. Yet the above applications alone are not what makes SLS techniques so interesting for industry: the growing demand for customized automotive products and the resulting small lots needed for low-volume production has caused a commensurate demand for the fabrication of volume-produced parts using generative processes. As a key manufacturer of premium cars, DaimlerChrysler is driving research in these direct-to-product technologies to accelerate product development and reduce costs. Thus, an essential part of our work focuses on the very strict requirements for accuracy in the SLS process to achieve the desired high quality of the parts produced.

Typically inaccuracies in the selective laser sintering process using polymer materials occur due to inhomogeneous shrinkage during the building and cooling processes, which causes stresses and results in distortion of the laser-sintered part. Temperature measurements [1] performed within an SLS machine showed a strong temperature gradient in the x-y plane of a layer, thus making the shrinkage of a geometry dependent on its x and y positions in the build room. Yet part shrinkage does not only depend on the temperature at which the part is subjected to laser sintering, it is also impacted by cooling, i.e. the length of time the powder bed retains heat, and by the thickness of the powder layer (causing pressure on the part). Experiments previously performed on sample geometries consisting of hollow rectangular parallelepipeds with equidistant measuring points placed at various x-y locations in the build room showed non-linear shrinkage of the height. We thus follow that a linear scaling of a geometry is not sufficient. A subsequent real geometry scaled non-linear in accordance with the empirical data generated resulted in a greatly improved accuracy of the laser sintering process

The temperature distribution within a build room and the changes in temperature during the building and cooling processes differ with the geometry of each part to be produced. The reason for this lies in the variation in the energy applied by the laser. The energy generated by the laser heats the powder and causes the sintering effect, finally dispersing into the powder bed. And due to the very low thermal conductivity of the polymer powder deployed, the build room retains heat over a relatively long period of time. Moreover, shrinkage in geometries which take up a large part of the build area is very different from that occurring in single, smaller parts since shrinkage in large geometries tends to be retarded owing to internal stresses. Hence, the related non-linear scaling function needs to be determined separately for each part to be laser sintered. So, the process has to be run twice: the geometry is built, shrinkage ascertained, the related scaling data generated, and the upscaled geometry compensating shrinkage then built. To save build time and the related costs, it would therefore seem sensible to calculate shrinkage before laser sintering and update the data model, thus doing away with the need to replicate the building process.

The Procedure

Experiments performed with sample geometries have shown that shrinkage occurring in the SLS machine is dependent on time, temperature, and pressure, i.e. the weight of the powder in which the part is embedded. Hence, to be able to calculate and compensate shrinkage, we must first ascertain the values of these three factors. The basis for any workable shrinkage compensation must therefore be an SLS process simulation that allows calculation of thermal distribution within the build room during the building and cooling processes.

We laid the experimental foundation for the quantification of shrinkage by carrying out dilatometer measurements as a function of time, temperature, and pressure. We measured the changes in height of laser-sintered, cylinder-shaped PMMA samples at temperature and pressure conditions identical to those in the laser-sintering equipment over a period of time of 10 hours. The measured curves were then mathematically set out as fit functions. However, transfer of idealized dilatometer measurements to the real conditions in the SLS machine, e.g. under consideration of temperature-dependent friction between the part and the bulk powder, will necessitate a more exact examination of the granular characteristics of the material used. Here,

we provide a possible theoretical description of particle sliding under consideration of temperature-dependent cohesion, friction, and sintering.

Thermal Simulation as a Basis for Shrinkage Compensation

We decided to compensate shrinkage by means of an FEM simulation which replicates the thermal conditions prevalent within the build room during the building and cooling processes. The heat-conductivity model used for determining heat distribution is described in a previously published work [2]. As shrinkage is a long-term effect, the highly dynamic effects of sintering may be neglected when calculating the heat balance. Exposure of the part and the sintering process itself are instantaneous, i.e. time is not a factor in the transition from the initial density of the powder bed to the final density of the part. This simplification allows us to select suitably large values for both the distance between nodes for the FEM simulation and the time intervals for the FEM calculation, thus greatly minimizing computation time.

Initially, we were only able to compute very simple geometries; yet creating an STL interface to the simulation seemed promising for calculation of parts whose geometries are more complex. As the exposure time for each layer and, thus, the resulting overall time needed for the building process are greatly dependent on the geometry of the part to be sintered, the interface to STL allows specification of the time each layer is subjected to a certain temperature. And the pressure to which each layer is subjected due to the weight of the bulk powder lying on top may easily be ascertained via the current z position of the layer.

Figure 1 depicts isothermal distribution inside the build room using the example of a hollow rectangular parallelepiped. For reasons of symmetry, only one-fourth of the build room need be displayed in the figure.

Of course, the results of the shrinkage compensation are only as accurate as the thermal simulation itself, i.e. the actual temperature conditions in the build room must be simulated as closely as possible. For this reason we compared the results of the simulation with empirical data. However, as we were not able to measure the exact temperature within a part, the temperatures within the powder bed near a sintered geometry were compared.

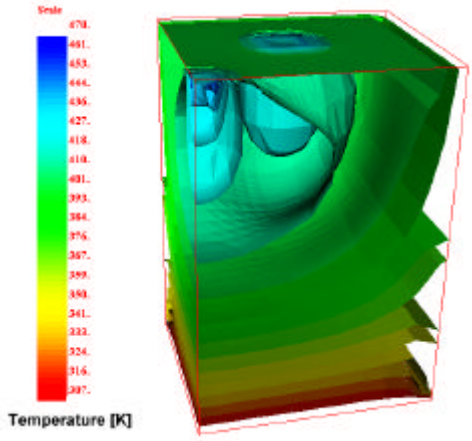

Figure 1: Isothermal distribution within the build room for a hollow rectangular parallelepiped.

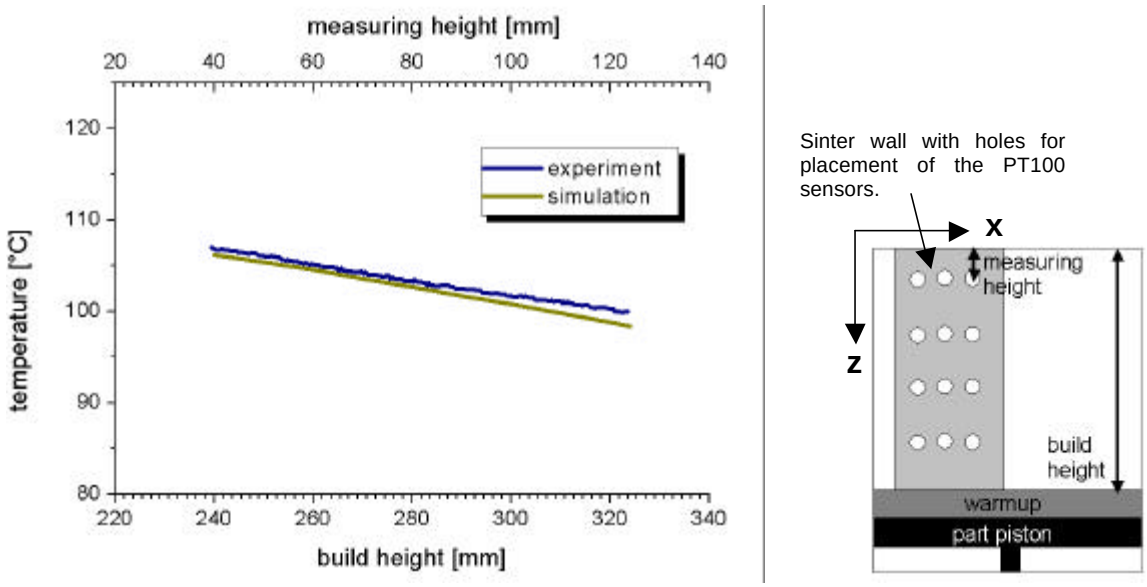

Figure 2: Comparison of the temperatures measured during experiments and the results of the thermal simulation.

Figure 2 compares the measured temperatures with data from the thermal simulation. In the experiment, we inserted PT100 temperature sensors into holes in a sinter wall and measured the temperature in the powder bed. The temperature curve illustrates the cooling of the powder effected by lowering the build platform. Comparison with simulation data showed excellent consistency.

Ascertaining Experimental Data for the Shrinkage Model

To quantify shrinkage of PMMA parts during the selective laser sintering process, we performed dilatometer measurements of the change in height of the samples as a function of time, temperature, and pressure. The resulting data served as the foundation for a physical shrinkage model.

Samples and measuring conditions: The samples used in the experiment were cylindrical in shape with a height of 5mm and a diameter of 7mm. At isothermal conditions, the height of the samples was measured over a period of time of 10 hours: temperatures under T_G (T_G = 123° C) and over T_G (T_{max} = 160° C) were applied. Pressure on the samples that would occur from the weight of the powder in the SLS machine was simulated during measuring by the application of various weightloads. A stamp was used to ensure that pressure was distributed equally over the entire surface of the sample. Measurements were performed at four different loads and at no fewer than five different temperatures for each load applied. A very slight bulging of the samples was registered at the highest temperature and the greatest pressure but, as this was negligible, it was disregarded. For all samples, heat-up time was 4 K/min, so that we were able to assume even heating of the small samples. The samples were placed into the dilatometer under two different conditions:

1.) Friction-free shrinkage: the samples did not come into contact with the quartz walls of the device.

2.) Retarded shrinkage: to be able to estimate the impact of friction between the sample and the powder bed on shrinkage behavior. As we were unable to achieve selective heating between the powder bed and the sample, polyamide powder was scattered between the sample and the quartz walls of the dilatometer to simulate the different flow and cohesive properties of the PMMA powder and the sample at the different temperatures.

Qualitative description of the shrinking curves: *Figure 3* illustrates the shrinking curve of a PMMA sample over a period of time of 10h at a temperature of T=140°C and a pressure of p=18.5g/cm^2, which corresponds to the weight of a powder bed that is approx. 30cm deep. During the heat-up phase, the sample initially expands due to thermal expansion. Yet, when a certain temperature is reached, shrinkage begins. As thermal expansion is not yet fully complete at this point, the sample is in a mixed state between expansion and shrinking. To more exactly define this state, we subtracted the thermal expansion of the sample from the curve which shows the change in height (red line): this curve thus shows greater shrinkage then the measured curve where thermal expansion is considered.

The largest degree of shrinkage (>50%) occurred for all samples within the first 100 minutes with a sharp decrease in the height of the samples registered. After this length of time, shrinking seems to change over to a state of saturation. This is best explained if we see shrinkage as the effect of particles or particle clusters sliding from their original positions to fill empty spaces. This happens when sinter contacts are thermally activated by the temperature applied, which reduces the friction between the individual particles, causing them to slide. The particles which are the first to slide into vacancies or vacancy clusters are those whose sliding planes are tilted in the direction of the space to be filled out (*Figure 4*), as this is the path of the least resistance. This explains why the degree of shrinkage is greatest at the beginning of isothermal densification. The directions of the sliding planes in the samples are distributed statistically and, over time with more and more particles filling vacancies, the concentration of those particles able to slide into vacancy clusters decreases so that shrinkage approaches a saturation value for longer periods of time.

Temperature- and pressure-dependency of the final values of shrinkage: As expected, the height of the cylinder-shaped samples after shrinkage can be expressed as a function of the temperature and the pressure applied to the samples. When the final values of shrinkage and the temperature applied are expressed in the form of an Arrehnius plot, it becomes evident that the two values are linear dependent. Thus, the dependency between temperature T and shrinkage s_{fin} may be expressed as follows:

$$s_{fin} \propto e^{-\frac{E}{cT}}, \qquad \textbf{(Eq. 1)}$$

with c=const.

E may be understood as a thermal energy of activation: the higher the temperature, the greater the number of particles contacts which are thermally activated with the result that a greater number of particles become active, sliding to fill the empty spaces. This increases shrinkage and subsequently the final density.

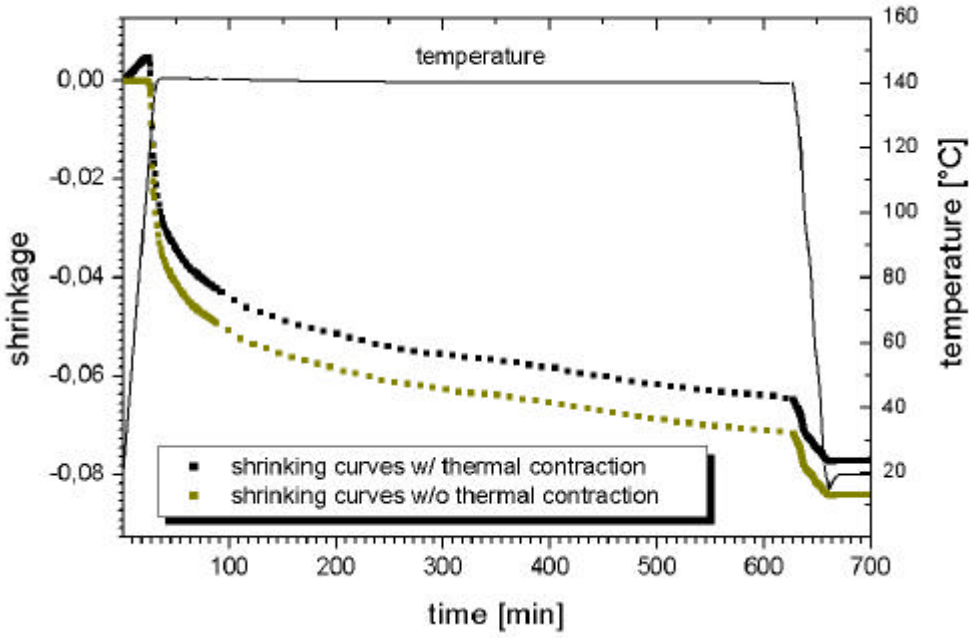

Figure 3: Shrinking curves of a PMMA sample sintered at T=140°C and p=18.5g/cm^2. Note that, in the red curve, thermal expansion of the sample during the heating process has been subtracted. The temperature curve for the overall process is included.

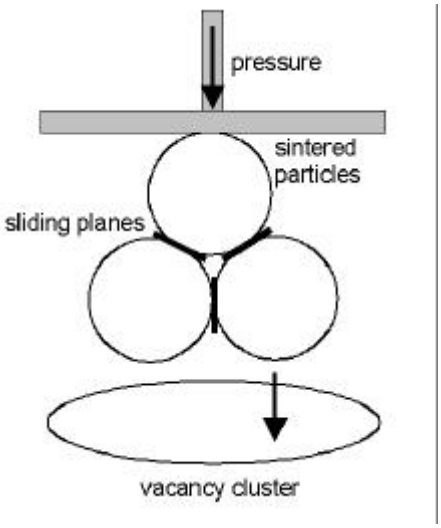

Figure 4: Schematic sketch illustrating the shrinkage mechanism for laser-sintered polymer samples.

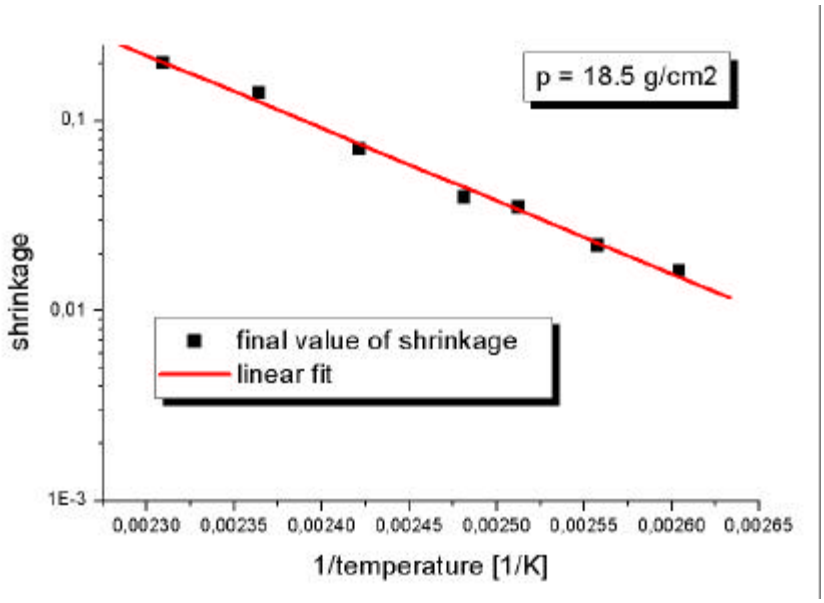

Figure 5: Temperature-dependency of the final values ascertained for shrinkage.

The dependency between the final values of shrinkage and the pressure applied is also linear *(Figure 6)*. However, increasing mass, i.e. the weight applied, does not have as great an impact on the degree of shrinkage as increasing the temperature. The linear dependency between these two values – shrinkage and pressure – may best be explained as follows. When the contact surfaces between the particles are thermally activated, cohesive strength is reduced. Similar to Hooke's Law for elastic bodies, pressure causes the particles to be compressed like a spring. Yet unlike a spring, where the compression of the coils is instantaneous, compression of the particles takes much longer. The internal stresses are relieved when the powder particles slide along their planes to fill any available empty spaces. Therefore, after pressure is removed, the samples – again unlike a spring - do not regain their original shape. This process is, in contrast, a relaxation process.

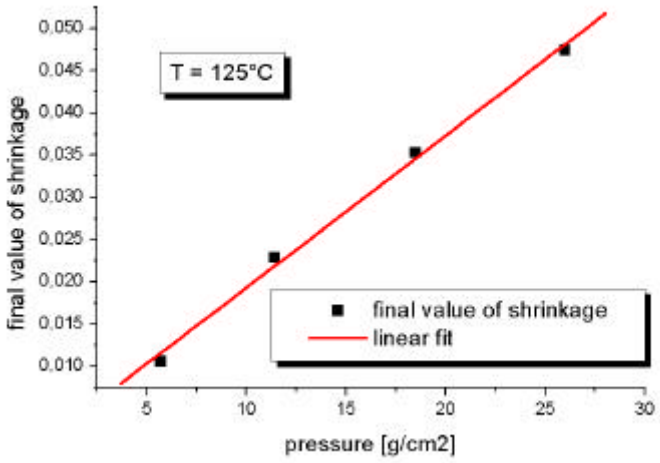

Figure 6: Pressure-dependency of the final values ascertained for shrinkage.

Impact of particle friction on shrinkage: To estimate the impact of friction between the bulk powder and the sinter geometry, we carried out the above-described shrinkage experiments using PA powder. *Figure 7* depicts shrinkage measured at three different temperatures: first without friction and then retarded through friction due to particle contact. For all three temperatures, retarded shrinkage is less than friction-free shrinkage by the same absolute value (1%). This behavior would indicate that, within the temperature range considered, contact friction between the bulk PA powder and the PMMA sample is not temperature dependent. At the temperatures selected here, the PA powder is neither cohesive nor flowable enough to significantly increase friction at the contact surfaces. It naturally remains to be examined in future work whether this behavior holds true for PMMA samples laser-sintered in a PMMA powder bed.

Integration of shrinking curves in the simulation: To be able to incorporate shrinkage in the thermal simulation, we must first express the change in height of the samples in mathematical form as a function of time, temperature, and pressure. When the shrinking curves are shown on the log-log scale, it becomes apparent that the curve, for most of its run, follows a line (*Figure 8).* Hence, according to a power law, the change in height and, thus, the density of the sample, may be expressed as a function of time for longer time periods. Adding the linear dependency of shrinkage s to pressure p renders the following equation:

$$s \propto t^{a(T)} \cdot cp\,, \qquad \textbf{(Eq. 2)}$$

with c=const.

The exponents of the exponential function and the temperature are linear dependent *(Figure 9):*

$$a(T) = c_1 T + c_2\,, \qquad \textbf{(Eq. 3)}$$

with c_1, c_2 = const.

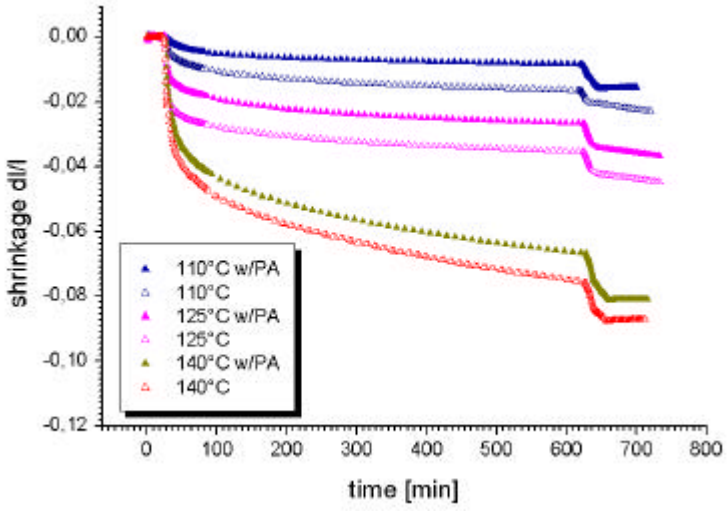

Figure 7: Comparison of shrinkage of pre-sintered PMMA samples tested first friction-free and then using PA powder to enhance cohesive strength.

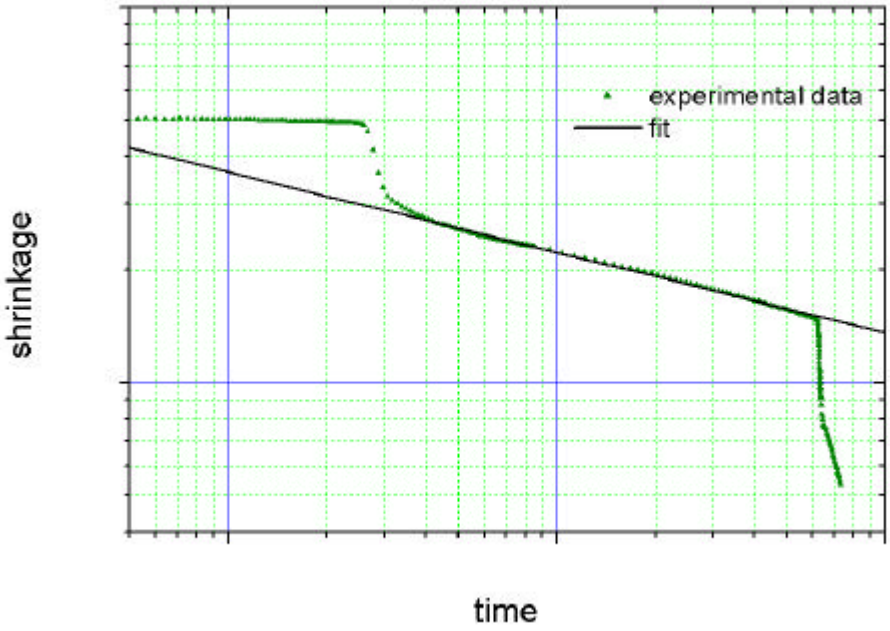

Figure 8: Time-dependency of shrinkage on a log-log scale.

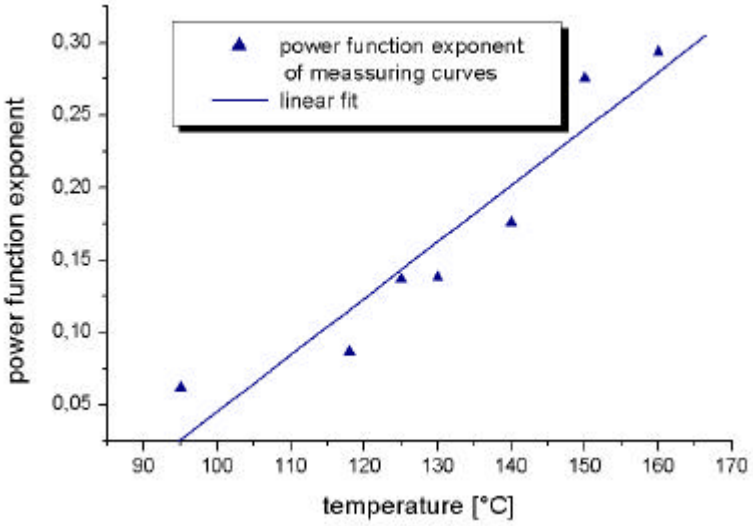

Figure 9: Temperature-dependency of the power function exponents.

Physical Shrinkage Models

Modeling and simulation of shrinkage can be done on two scales: at a microscopic level which tracks an amount of distinct interacting particles, the so-called Distinct Element Model (DEM), and at a macroscopic level. The latter applies special laws for the plastic flow of cohesive granular matter. [3] With respect to the cohesive strength, relevant sintering laws [2] which may

be extended by a pressure dependence need to be found. This can be done, for instance, if the driving surface tension is enhanced by a value proportional to the local pressure and the particle diameter.

A simple version for the DEM system is given through an elastic contact model which consists of two different stiffnesses for loading and unloading in order to incorporate plasticity [3]. Also, cohesion strength has to be considered. The tangential motion of the particles is controlled by a Coulomb-type frictional law. Due to the history- and temperature-dependence of the sintering process, both the cohesion and the friction parameters need not be constant.

In principle, this microscopic simulation together with a certain averaging procedure leads to effective macroscopic dynamic laws. In practice, however, the number of particles is limited for computational reasons. Moreover, most current DEM simulations are restricted to two dimensions. Therefore, it is reasonable to try to directly find some macroscopic constitutive equations.

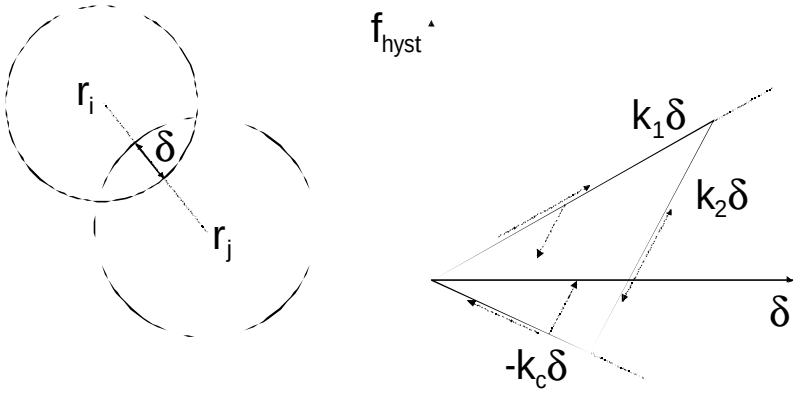

Figure 10: Two-particle contact with overlap d (left), elastic force law with hysteresis and cohesion (right).

Several authors have considered macroscopic stress-strain behavior in the context of soil mechanics. We may start here from a Drucker-Prager formulation of plasticity, which has to be generalized to include cohesion, for instance [3]. However, the resulting model still needs to be extended to make cohesion history- and temperature-dependent. This has not yet been done.

For low stresses, i.e. if no external pressing is carried out, a viscoplastic formulation of the constitutive equations would be more appropriate. The related viscosity coefficients will then depend on the density, leading to a non-linear model [4]. Finding the related sintering laws for the polymer powder remains an area for future work.

Summary and Future Work

Laser-sintered polymer parts shrink during the building and cooling processes. Shrinkage, which may be expressed as a function of time, temperature and pressure, is to be computed before sintering and compensated by upscaling the requisite datasets. The basis for shrinkage compensation as set out here is given through a thermal simulation of the SLS process, which replicates the temperature distribution in the build room very accurately. Data from dilatometer examinations of laser-sintered sample geometries serve as the foundation for a shrinkage model. In experiments, the samples demonstrated a change in height which, given longer periods of time, behaves in accordance with a power law with time. The exponents of the exponential function and the temperature proved to be linear dependent. In addition, the final values of shrinkage can be expressed in the form of an Arrehnius plot as a function of temperature and also show a linear dependency to the pressure applied. The former function results from the necessary thermal activation of the particle contacts, whereas the latter is explained as a stress-strain relationship similar to Hooke's Law, where internal stresses caused by applying pressure to the particles are relieved by the particles sliding into available vacancy clusters. If particle friction is generated through the addition of bulk powder, shrinkage is reduced significantly. Transfer of the experimental results achieved under the idealized measuring conditions prevalent in the dilatometer to the real conditions in SLS machines will require further work. However, this work offers various theoretical approaches for such transfer. While the resulting shrinkage model will need to consider friction and cohesion between the particles at the microscopic level, relevant macroscopic equations for shrinkage are required to significantly minimize computation time.

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