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Predicting processing parameters in high temperature laser sintering (HT-LS) from powder properties

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ABSTRACT

New materials for laser sintering (LS) are usually developed using a trial and-error approach that consists of a series of builds within LS systems. This strategy is time consuming, costly and focuses only on the optimisation of the processing parameters, ignoring the powder properties of the materials under examination. Being able to predict processing parameters on the basis of the powder material properties would enable a faster development of new materials and new applications, while acknowledging a more in-depth understanding of the mechanisms involved in LS. This paper provides new results into the prediction of processing conditions from the material properties. It is here shown that high temperature polymers such as poly ether ether ketone (PEEK) and poly aryl ether ketone (PEK) can be successfully used in LS despite the lack of a super-cooling window. The evaluation of the stable sintering region of PEEK 450PF and the application of the energy melt ratio parameter in relation to the mechanical performance of laser sintered PEEK samples are also provided. Lastly, a new method for estimating the powder bed temperature is proposed.

1. Introduction

Laser sintering (LS) is a powder bed fusion additive manufacturing process where polymeric powders are consolidated layer upon layer by means of a CO_2 laser beam. In the process, polymer powders contained in the building chamber of a LS system are gradually heated up to a temperature (part bed temperature) where they can then be exposed to the laser. The latter will trigger the phase transition from solid to liquid phase using ideally the minimum input of laser energy. Once the laser exposure is finished, a new fresh layer of powder is spread and the laser exposure performed again. These operations are repeated until all desired components included in the build job are completed. At that point a long cooling phase will start.

When new materials are to be trialled in the LS process, the general approach for choosing the processing temperatures is based on qualitative criteria such as visual inspection of the powder bed for dark areas (hot spots) and cracks. If either occurs, approximate temperature adjustments such as changing the temperature by a few degrees within the LS system are applied. EOS [1], a top-leading manufacturer of LS equipment, provides a method for the evaluation of the optimum processing temperatures of PEK HP3 (EOS method). The steps of the EOS method for finding the optimum processing temperatures are the following: set the part bed processing temperature at 320 °C; deposit layers as per normal operation; pause build process, increase the part bed temperature by 5 °C, deposit one layer and wait for the heater power to drop below 40%; increase the part bed temperature by 5 °C to 330 °C, deposit one layer and wait for the heater power to drop below 50%; increase the part bed temperature in 2 °C increments, deposit one layer for each increment until the surface starts to discolour showing hotspots (melt of the material); reduce the temperature from this transition temperature, in 1–2 °C stages, deposit one layer for each increment until the hot spots stop to occur; set the exchangeable frame (temperature in the middle of the building chamber) and building platform (temperature at the bottom of a building chamber) temperatures at 20 and 25 °C below the part bed temperature. Although reliable, this procedure could be greatly improved with a more accurate methodology.

The optimisation of the processing parameters for a desired part performance is instead based on an iteration of trial and error builds, where one parameter is changed at a time and its effect on the laser sintered part properties is evaluated through experimental mechanical and thermal testing.

Both strategies – the one for choosing the processing temperature and the other for optimising the processing parameters for enhanced part properties – can be highly time consuming and extremely expensive depending on the cost per kg of material being examined, its ability to be recycled, the physical size of the LS equipment available and the failure build risk of the system.

Polyamide (PA) based polymers are the most used polymers in the LS process. These plastics offer a wide super-cooling window [2], acceptable properties for a wide range of applications and are

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exhaustively used in prototyping. Their high recycle rate has allowed to margin the cost due to the trial-and-error builds and also promoted further development towards PA composites.

Recently a new generation of high temperature (HT) polymers have entered the LS process. Examples are poly ether ketone (PEK) and poly ether ether ketone (PEEK).·These polymers provide superior mechanical, thermal and chemical performance than PA based polymers and therefore find application in high tech sectors such as defence, medical and oil and gas industries. Their unit cost is however significantly higher than PA based polymers and therefore it could be beneficial to formulate a method or a small set of experiments that allows prediction of theprocessing temperature of an unknown material and identify the optimal set of processing parameters for a certain application a priori, i.e. before physical testing of a powder within a HT-LS system.

A number of attempts to optimise the LS process are available in the literature. Ho et al. [3] evaluated the effect of the energy density (*ED*) parameter on polycarbonate structures, while a similar approach was adopted by Drummer et al. [4,5] and Caulfield et al. [6] for the analysis of PA structures. Most of them focussed on the influence of *ED* on the mechanical performance of laser sintered parts and the main outcome from these studies was that an increase of the *ED* caused the mechanical properties, especially tensile strength, to improve up to a value where they stabilise or slightly decrease. Franco et al. [7,8] modelled the LS process and quantified the correlation between *ED* and layer consolidation depth. Although some structures were laser sintered only for a feasibility purpose, rather than for mechanical characterisation, the authors offered a model of LS that includes processing parameters and fundamental material properties for the first time, unlike any of the previous models, which were based on the *ED* factor only.

More recently, Vasquez et al. [9] reported on the investigation of a parameter called energy melt ratio (*EMR*), where material characteristics obtained by thermal analysis, gel permeation chromatography and thermo-gravimetrical analyses are used to predict the ideal LS processing parameters. “Ideal parameters” indicated those building settings that prevented material degradation during the laser consolidation while reaching the highest mechanical properties in the parts. Indeed, the authors built and mechanically tested samples at different *ED*s, calculated the corresponding values of *EMR* and then correlated these *EMR*s with mechanical and thermo-gravimetrical data. The *EMR* factor predicted well the parameters for which the material degradation started to occur during the LS process, especially for the PA 12 grade. Not surprisingly, the mechanical performance of the laser sintered samples under test improved with increasing values of *ED* and *EMR*, until it reached a peak value. Interestingly, the *EMR* value predicting material degradation was found to be relatively close to the highest limit value of the tensile strength. These findings also allowed the authors to identify a new temperature region for LS based on material properties called a “stable sintering region”. This temperature interval is based on the melt and degradation behaviour of the powders and should guide the choice of the processing temperature and laser parameters during a LS building operation. The *EMR* combined with the evaluation of the stable sintering region can help to reduce significantly the number of preliminary builds for a new material development and optimisation. However, a small number of build trials for a specific material will still have to be carried out.

The aim of this paper is to predict for HT polymers the processing temperature such as the part bed temperature and the laser exposure parameters from the powder properties. At present, a study into the development and optimisation for predicting processing parameters of the PEEK material within the only HT-LS commercial system EOSINT P 800 is still missing and therefore it constitutes the main investigation of this work. HT-LS can provide PEEK components for high demanding engineering applications in a wide range of industrial areas such as aerospace, automotive and medical industries. HT-LS of PEEK also relies on a lower processing temperature window than PEK, the current commercial HT-LS material, due to a 30 °C lower melting temperature between PEK and PEEK. Experimental studies on laser sintering of PEEK are not the main topic of this paper and can be found elsewhere [10–12].

2. Theory

2.1. Super-cooling window

The super-cooling window is the LS processing window defined based on the differential scanning calorimetry (DSC) thermoscan of the powder under test [13], largely used in the LS community [14–17]. This window is defined as the temperature gap between the onset of the melting event and the onset of the crystallisation event. This window essentially constitutes a wide range of temperature values that can be used as a processing temperature inside a LS system for successful LS manufacturing. This spectrum should ensure that the powder lying in the powder bed of a generic LS system does not melt before exposure to the laser and does not crystallise during laser exposure.

2.2. Stable sintering region

The stable sintering region is a concept introduced by Vasquez et al. [9] to study established and new potential LS materials in the region above their melting points and below their degradation temperatures. It aims to predict the temperature interval that leads to thermal degradation of the material in order to avoid it, during a LS building operation, especially during the laser exposure. The stable sintering region is bounded by values found with DSC, hot stage microscopy and thermo-gravimetrical analysis (TGA) of polymeric powders. The lower limit is the temperature at which the material is fully molten and is found with the combination of DSC data (melting peak temperature) and hot stage testing (temperature at which the powder particles were fully melted). The upper limit is the temperature at which the 1% weight loss degradation occurs during a TGA experiment that is carried out from room temperature to HT at a heating rate of 10 °C × min^{-1}. This method clearly does not take into account the crystallisation effect present in semi-crystalline polymers such as PA based powders or PAEK materials and therefore according to Vasquez et al. [9] can be used for more types of polymers. The authors showed that PA 12 presents a stable sintering region at 120 °C, with the lower limit at 200 °C and the upper limit at 320 °C. The processing parameters of the laser exposure during LS – laser power, scan spacing, scan count and laser speed – should therefore increase the temperature of the powder particles in the powder bed above 200 °C in order to achieve complete melting, but remain below 320 °C in order to avoid their thermal degradation [18].

2.3. Energy melt ratio

During a LS building operation, a laser exposes defined areas in the powder bed, triggering the melting of the material particles interested. In this operation, the laser is believed to fully transfer an amount of energy into the powder bed that depends on the characteristics of the laser exposure. Moreover, in order to achieve a specific successful sintering in only the laser exposed areas of the powder bed (and not across all the powder bed) the powder bed is kept at a temperature that is just below the material melting temperature.

The characteristics of the laser exposure are generally grouped under the “energy density” (*ED*) parameter which is defined (Eq. (1)) by the laser power (*P*), the number of times the same region is exposed by the laser (“scan count”, *C*), the distance between two next segments of laser exposure (“scan spacing”, *S*) and the laser beam velocity or laser