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Energy input effect on morphology and microstructure of selective laser melting single track from metallic powder

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ABSTRACT

Process parameters of selective laser melting affect the response of a powder–substrate system and, therefore, the geometry and microstructure of the manufactured parts. The experiments were carried out at fixed values of laser power (50 W), spot diameter (70 μ m) and powder layer thickness (80 μ m). In this research, influence of the energy input parameters (80–900 °C preheating temperature and 0.08–0.28 m/s laser scanning speed) on microstructure and geometry of single tracks fabricated of stainless steel grade 316L powder was analysed. Both factors were found statistically significant with regard to their influence on the remelted depth and the primary cell spacing in the colonies observed in the tracks cross-sections. More specifically, the contact angle and track height were controlled by the preheating temperature, and track width and contact zone characteristics were governed by the laser scanning speed. Because of the threshold behaviour of these two factors, values starting with 700 °C and 0.24 m/s were found not optimal and causing instability and balling effect. Conclusions regarding the selection of process parameters for the formation of tracks with the desired geometry and microstructure were formulated based on statistical analysis of the experimental data.

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1. Introduction

In recent years selective laser melting (SLM) shows considerable progress in manufacturing customized parts with full density, complex form and structures (Wohlers, 2010). The essential operation in SLM is the line-by-line melting of powder material to form successive cross-sectional layers of a final 3D part. A necessary condition for obtaining a quality 3D object is stability of the geometrical characteristics of each single track and a good adhesion (cohesion) to the previously remelted layer (substrate).

A laser beam/powder/substrate system involves phenomena such as absorption, reflection, radiation and heat transfer, phase transformations, fluid flow driven by surface tension gradients and mass transfer within the molten pool, and chemical reactions. Modification of process parameters can cause various undesirable effects as geometrical irregularity of the sintered track (Niu and Chang, 1999) or balling effect (Tolochko et al., 2004). This explains the need for developing a comprehensive scientific understanding of SLM process phenomena in the whole and responses of powder–substrate system to changes of process parameters. Laser power density is the most influencing process parameter in SLM (Yadroitsev, 2009). Studies on selective laser sintering/melting of powder report that preheating powder and substrate can reduce requirements to laser power (Leu et al., 2010), improve radiation absorptivity of the powder and wettability of the substrate (Campanelli et al., 2010). Theoretically, the effect of preheating can be roughly estimated from the expression (Steen, 2003):

$$Q = \rho c_p (T_m - T_0) + \rho L_f$$

where Q is the energy necessary to melt a unit volume of material, ρ is the density of bulk material, c_p is the specific heat, T_m is the melting point, T_0 is the initial temperature, and L_f is the latent heat of fusion. For example, energy required for melting of a certain volume of stainless steel grade 316L ($\rho_{bulk} = 7800 \text{ kg/m}^3$, $c_p = 515 \text{ J/kg K}$, $T_m = 1673 \text{ K}$, $L_f = 275 \text{ kJ/K}$) preheated to 900 °C (1173 K) is 43% less than for the same material at 80 °C. In additive manufacturing, traditionally used preheating systems are infrared heaters or resistive heaters placed near/around the building platform to maintain high temperature around the workpiece or feed cartridges to preheat the powder before spreading over the build area (Campanelli et al., 2010).

The formation of single tracks from metal powders by SLM has a threshold character: for each powder material, there exist sets

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of process parameters that yield stable and unstable tracks. The mechanism of distortion, irregularities and drops formation may be associated with thermophysical properties of the material, granulomorphometric characteristics of the powder, peculiarities of its deposition and spreading, layer thickness, energy parameters of laser radiation, laser scanning speed, and melt hydrodynamics (Yadroitsev et al., 2010).

The formation of satellites, remolten drops surrounding and having metallurgical contact with the track, could eventually provoke the formation of pores in 3D parts and increase surface roughness (Mumtaz and Hopkinson, 2010a). This effect is significant for the internal structures of the parts, especially for thin-walled and fine channel structures. Satellites may be formed by two main mechanisms: melt spattering and partial powder melting in peripheral zone of laser spot.

Thermal transport in liquid and solid phases governs solidification processes in the track and, therefore, impacts the mechanical properties of SLM parts (Dai and Shaw, 2002). Multipass remelting influences microstructure of the previously synthesized tracks and layers. The last scan not only re-melts parts of previous tracks but affects regions beneath, causing thermally activated diffusional processes like phase transformation, grain growth, precipitations etc. As a result, a multilayered object with non-uniform microstructure, grain size and shapes, non-equilibrium phases is formed. Certain phases may deteriorate properties of the final component manufactured by SLM. It has been shown that temperature gradient and local heat transfer conditions determine a cooling rate and grain growth (Kruth et al., 2010), therefore changing the process parameters and scanning strategy would affect the final microstructure and properties.

The energy input strongly influences the response of a powder–substrate system and therefore has to be carefully controlled for SLM part quality. In this study, role of two main influencing energy input factors at fixed laser power, preheating temperature and laser scanning speed, on a single track formation was investigated. Microstructural (grain size, texture, morphology) and geometrical (remelted depth, width, contact angle, etc.) characteristics were investigated in correlation to changes in these parameters. To quantify statistical significance of these factors on track geometry and microstructure a statistical analysis of variance was done.

2. Materials and methods

Stainless steel grade 316L powder, Fig. 1(a), was selected for this study because of the simplicity and availability of this single-phase metallic alloy. The 316L steel is austenitic, non-magnetic steel nowadays widely used in chemical, pharmaceutical, textile, energy, marine, pollution control and biomedical implant industries. Due to extra-low carbon content, this grade demonstrated minimized welding carbide precipitation, higher resistance to intergranular corrosion, high creep properties and strength at elevated temperatures. The nominal chemical composition of the employed powder was as follows (weight %): Fe - balance, Ni - 10-14%, Cr - 16-18%, Mo - 2-3%, Si - 0.75% max, Mn - 2% max, C - 0.03% max, P - 0.045% max, S – 0.03% max. The powder was supplied from Sandvik Osprey Ltd. The powder was dried by heating up to 80 °C during 12 h, and then cooled down to room temperature in a cooling chamber. Granulomorphological analysis of the powder was carried out by an optical granulomorphometer ALPAGA 500 NANO. The 10th, 50th and 90th percentiles of equivalent diameter (weight by volume) of 316L powder were d_{10} = 3.0 µm, d_{50} = 7.1 µm and d_{90} = 27.5 µm, respectively.

Substrate was 304L stainless steel with chemical composition Fe – balance, Ni – 8–12%, Cr – 18–20%, Si – 1% max, Mn – 2% max, C – 0.03% max, P – 0.045% max, S – 0.03% max (in weight %). The substrate and the powder material were similar in chemical composition, but 316L contained 2-3% of molybdenum.

SLM experiments were carried out using single-mode continuous-wave Ytterbium fiber laser operating at 1075 nm wavelength (IPG Photonics Corp.). The laser beam has a TEM₀₀ Gaussian profile, 70 μ m spot size, and 50 W maximum power. An infrared heater placed inside the working chamber filled with nitrogen as a protective gas was used for preheating. Preheating temperature was controlled by a thermocouple.

The height of the track is determined mainly by powder layer thickness. For small layer thicknesses (less than doubled d_{90}), the track height becomes too small to study geometrical characteristics and microstructure (Yadroitsev et al., 2012). Therefore, powder layer thickness of 80 µm was chosen for this study. Single tracks were formed by P = 50 W laser power at scanning speeds (V) ranging from 0.08 to 0.28 m/s with a step of 0.04 m/s. Operational temperatures of the process chamber (preheating temperature, T) were chosen to be 80, 300, 500, 700, and 900 °C. Process-parameters were selected so that regimes causing pronounced balling effect were avoided. The length of the scan lines (tracks) was 20 mm for all the experiments. Six individual tracks were produced for each set of parameters, forming statistically groups, and geometrical characteristics of their cross-sections were analyzed (Fig. 1(b)): width and height of the tracks (w_1, h_1) , width of the contact zone (w_2) , remelted depth (h_2) , and average value of the contact angle $(\alpha = (\alpha_1 + \alpha_2)/2)$. Statistics were calculated for a total of 180 single tracks.

Samples were subjected to a standard metallographic procedure: grinding with 320, 800 and 1200 papers, and then polishing by diamond suspensions (9, 3, 1 µm size). Scanning electron microscopy was carried out with LEO 1350 FEG-SEM and Tescan Vega 3 at 20 kV accelerating voltage and 5-15 mm working distance. EDX analysis was realized by Oxford Instruments INCAxsight EDX detector, energy spectra were calibrated to a Cobalt standard. Electron channeling contrast (ECCI) study was performed using the backscatter mode in an analytical SEM, Hitachi SU70 operating at 20 kV. Orientation imaging microscopy was performed using an electron back-scattering diffraction (EBSD) system from HKL Technology. No etching was performed on the samples for EBSD investigations and the contrast was achieved from differences in composition and crystallographic orientation. Other specimens for SEM investigations were finish polished with 1 µm diamond paste and then electrolytically etched in 10% aqueous oxalic acid at 15V during 60s for 1 cm² area. Primary cell spacing, a distance between centers of neighboring cells measures along the growth direction, was measured directly in the SEM; at least 30 measurements were done in each investigated region.

Analysis of variance (ANOVA) was applied to investigate SLM parameters with significant effects on track characteristics using statistical software Statistica 6.0 (StatSoft, Inc.). ANOVA uses Fstatistic - a ratio {found variation of the group averages}/{expected variation of the group averages} - to test the null hypothesis that the means of all the groups are equal. If the calculated value of F is greater than the tabulated criterion, the null hypothesis is rejected. It implies that the mean values over all the groups are not equal, and the analyzed parameter is a factor with a significant effect. For parametric ANOVA analysis, tests for normality of all the variables were executed (Shapiro-Wilk's test); all the studied parameters were normally distributed. To verify the hypothesis of equality of variances of the studied quantitative values in the groups defined by the factors Levene's test was realized (Hill and Lewicki, 2007). It showed that the difference in variances is available and to analyze the influence of the factors the significance level less than 0.001 was chosen.

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