Title: Direct Selective Laser Sintering and Melting of Ceramics: A review

Authors:

Swee Leong Sing [a, b], (S.L. Sing)

Wai Yee Yeong [a, b], (W.Y. Yeong) wyyeong@ntu.edu.sg +65 6790 4334 (corresponding author)

Florencia Edith Wiria [a, c] (F.E. Wiria)

Bee Yen Tay [a, c] (B.Y. Tay)

Ziqiang Zhao [d], (Z. Zhao)

Lin Zhao [d], (L. Zhao)

Zhiling Tian [d], (Z. Tian)

Shoufeng Yang [e], (S. Yang)

Affiliations:

[a] SIMTech-NTU Joint Laboratory (3D Additive Manufacturing), Nanyang Technological University, Singapore

[b] Singapore Centre for 3D Printing, School of Mechanical & Aerospace Engineering, Nanyang Technological University, Singapore

[c] Singapore Institute of Manufacturing Technology, Singapore

[d] China Iron & Steel Research Institute Group, Beijing, China

[e] Faculty of Engineering and the Environment, University of Southampton, Southampton, United Kingdom.

Abstract:

Purpose

This paper provides a review on the process of additive manufacturing of ceramic materials, focusing on partial and full melting of ceramic powder by a high energy laser beam without the use of binders.

Design/methodology/approach

Selective laser sintering or melting (SLS/SLM) techniques are first introduced, followed by analysis of results from silica (SiO_2), zirconia (ZrO_2) and ceramic reinforced metal matrix composites processed by direct laser sintering and melting.

Findings

At the current state of technology, it is still a challenge to fabricate dense ceramic components directly using SLS/SLM. Critical challenges encountered during direct laser melting of ceramic will be discussed, including deposition of ceramic powder layer, interaction between laser and powder particles, dynamic melting and consolidation mechanism of the process, and the presence of residual stresses in ceramics processed via SLS/SLM.

Originality/value

Despite the challenges, SLS/SLM still has the potential in fabrication of ceramics. Additional research is needed to understand and establish the optimal interaction between the laser beam and ceramic powder bed for full density part fabrication. Looking into the future, other melting-based techniques for ceramic and composites are presented, along with their potential applications.

Keywords:

Additive manufacturing, 3D printing, Ceramic, Laser, Melting, Sintering

1. Introduction

Ceramics, as a class of inorganic and non-metallic materials, has expanded the range of applications in modern industries such as the automotive, aerospace, defence and machining sectors (Dutta 2001, Liang and Dutta 2001, Ralph et al. 2001), due to their high mechanical strength, low thermal conductivity, high wear resistance and outstanding corrosion resistance. Moreover, the biocompatibility of some ceramics in the medical field is a focus of research activities worldwide, which allows them to be used for body prostheses, dental and tissue engineering scaffolds (Doremus 1992, Vaezi and Yang 2015).

The starting point for manufacturing ceramic parts is usually a powder material mixed with binders and stabilizers. Figure 1 shows the classification of processing techniques for ceramic materials (Yeong et al. 2013).

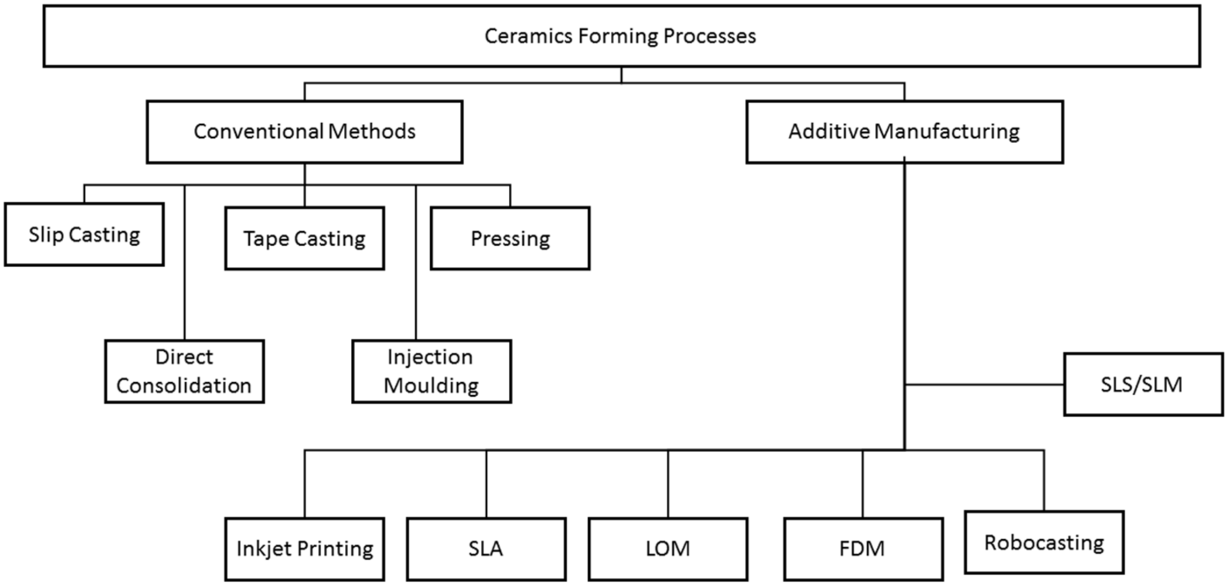

Figure 1 Classification of processing techniques for ceramic materials

Ceramics components are conventionally shaped from powder to form a green compact followed by steps of machining and sintering to achieve functionality with high densification (Denry and Holloway 2010). Examples of conventional ceramic forming technologies include slip casting, tape casting, pressing, direct consolidation and injection moulding (Fanelli et al. 1989, Yu et al. 2010). Limitations of these conventional techniques include high wear rates for the machining tool, high labour and cost intensities. There is also need for a tight process control to avoid undesirable shrinkages from sintering of green parts. In order to overcome the challenges above, additive manufacturing (AM) techniques have been used to manufacture ceramic parts with complex shapes (Mitteramskogler et al. 2014). AM is a process of making objects by adding materials according to the geometry specified by a three-dimensional (3D) model data, usually layer by layer (Levy et al. 2003, Sing et al. 2013, Sing et al. 2016, Sing et al. 2016). AM techniques reduce customization costs and lead time for individualized parts such as implant and prostheses

(Sing et al. 2016). Other advantages of AM include design freedom and the ability to allow precise control over the shapes and sizes of complex ceramic structures.

AM technologies that have been used for ceramic manufacturing are inkjet printing (Ebert et al. 2009), laminated object manufacturing (LOM) (Klosterman et al. 1998), fused deposition modelling (FDM) (Kalita et al. 2003, Francis and Jain 2016), stereolithography (SLA) (Tian et al. 2012) and selective laser sintering/melting (SLS/SLM) (Shahzad et al. 2013, Vaezi et al. 2013, Deckers et al. 2014). There are several review articles published on general AM techniques for ceramic forming (Deckers et al. 2014, Travitzky et al. 2014, Zocca et al. 2015). However, these reviews serve as concise studies and provide limited insights to allow further research into attempts of direct ceramic additive manufacturing using SLS/SLM.

The focus of this paper is specifically on the direct laser sintering or melting of ceramics, which has the promise to fabricate functional ceramic parts directly without any binders or post-sintering steps. The results of two oxide ceramic materials processed using these techniques, silica (SiO_2) and zirconia (ZrO_2), as well as ceramic reinforced metal matrix composites will be presented. Critical challenges encountered during direct laser sintering and melting of ceramics will be discussed, including deposition of ceramic powder layers, interaction between laser and powder particles, dynamic melting and consolidation mechanisms of the process, optimization of process parameters and presence of residual stresses in ceramics.

2. Selective laser sintering and melting

Selective laser sintering/melting (SLS/SLM) falls under the category of powder bed fusion manufacturing technologies, with the use of a laser beam as an energy source to fuse powder particles together. In general, the binding mechanisms of laser powder bed fusion processes can be classified under (Kruth et al. 2005):

- Solid state sintering
- Liquid phase sintering (structural materials with distinct binders)
- Partial melting (structural materials without distinct binders)
- Full melting

The solid state sintering process involves neck formation between particles, which requires ~~a~~ long heating time during which no melting or phase change takes place. Rapid melting and cooling of ceramics in SLM will also negatively impact the grain size of the ceramic part produced. Conventionally, ceramics are sintered with solid state sintering methods in which slow diffusion of atoms are needed to achieve fine grain size. This low sintering temperature will also help to maintain the fine grain size, which is the key characteristic for high mechanical properties of high performance ceramics. However the laser will not be able to provide such capacity for slow diffusion (Ouyang et al. 2001). For liquid phase sintering, the structural material remains in the solid phase while a binder material melts to form liquid. This binder can be made of various materials, such as metal, polymer or glass etc. The partial melting mechanism happens when the input energy can only melt the shell of the ceramic particles, and this molten material acts as a binder between non-molten grains. Due to the low densities and weak binding strengths of the parts formed by partial melting, the potential for their application is limited. Full melting of powder particles has been used in metal processing to form net shape parts with almost 100 % density and excellent mechanical properties (Frazier 2014). It would be of interest to determine if it would yield similar results for ceramics.

SLS is a process that uses high power laser beams to sinter powder material, usually polymeric due to low power of the lasers, into the solid products layer by layer, which may need debinding and heat treatment (Leu et al. 2012). These additional steps are usually not required in SLM. There are several methods that are summarized under the SLS name. These methods are commonly described as solid or liquid phase sintering methods. The powder bed can compose of either only a single material or together with organic/inorganic binders. The SLS process is performed in vacuum or atmospheres such as air, argon and nitrogen. Non-equilibrium phases can also be formed during SLS just as in SLM, due to conditions being far from the thermodynamic equilibrium (Qian and Shen 2013, Deckers et al. 2014).

SLM is a powder bed fusion process initially designed for 3D freeform fabrication of metals (Kruth et al. 2004). The SLM process begins with the deposition of a thin powder layer onto the substrate. The powder particles are then selectively melted by a high energy laser beam (Gu and Zhang 2013) (usually without any binders) according to computer aided design (CAD) data. After the laser moves away, the melted region undergoes rapid solidification. The steps are repeated with another layer of powder deposited over the previous solidified region and subjected to further melting and solidification. As this process proceeds, a 3D pattern forms. Figure 2 shows a schematic of a typical SLS/SLM process. SLS/SLM is a form of direct laser processing of materials as the laser beam is used to melt and form the part directly.

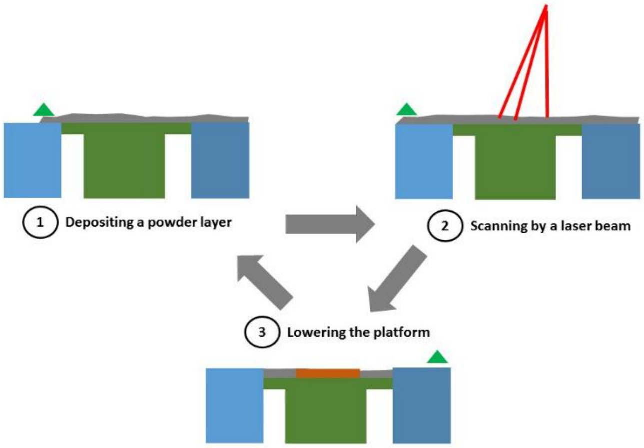

Figure 2 Schematic of a typical SLS/SLM process

SLM can produce highly dense parts with good mechanical properties and complex structures (Yeong et al. 2010). New material phases and microstructures may form during SLM processing since non-equilibrium conditions occur during laser melting with high cycling rates of heating and cooling (Murr et al. 2012). The SLM process has been researched with a variety of metals for fabricating net-shaped and high performance parts, such as stainless steel (Delgado et al. 2011, Khairallah and Anderson 2014), high speed steel (Liu et al. 2013, Holzweissig et al. 2015), aluminium (Louvis et al. 2011, Kempen et al. 2012, Loh et al. 2014, Lam et al. 2015, Loh et al. 2015), titanium (Chlebus et al. 2011, Vrancken et al. 2012, Do and Li 2016, Sing et al. 2016) and tungsten (Zhang et al. 2012). Recent developments in SLM has also made it possible to process multiple materials in a single build (Liu et al. 2014, Sing et al. 2015).

In recent years, direct laser processing of ceramic powders (which refers to the building of ceramic parts by laser partial or full melting) without distinct binders has garnered the interest of the research community. Recent research has demonstrated the possibility of using SLM to produce ceramic parts that are high in density and strength, and complex in shape (Yves-Christian et al. 2010).

2.1 Selective laser sintering of silica

Silica (SiO_2) is regarded as one of the most complex and abundant ceramic materials, and constitutes more than 60% of most glasses (Ojovan 2004). The refractory properties of silica make it a useful material in applications such as reusable thermal protection tiles (Steyer 2013) and casting moulds. Silica parts built by direct laser melting or sintering have been examined to fabricate complex tooling in sandcasting with reduced process time, as described further in the subsequent paragraphs.

Tang *et al.* (Tang et al. 2003) and Wang *et al.* (Wang et al. 2003) investigated direct laser sintering of silica sand for the casting industry. The build process using a CO_2 laser is shown in Figure 3.

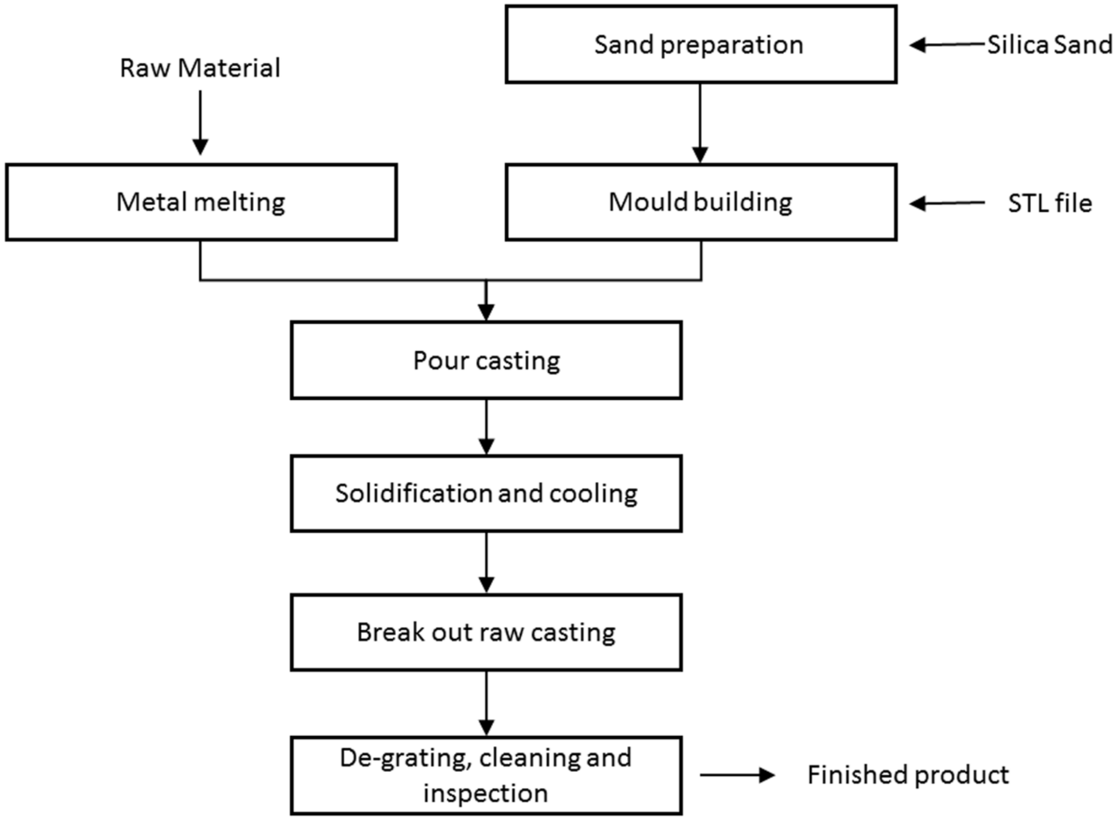

Figure 3 Adapted flowchart of casting process with direct laser sintering silica sand mould

The surface roughness, Ra, of ceramic parts were between 18 and 35 μm, and the compressive strengths were between 6 and 16 MPa. Compared with the compressive strength of dense silica, the strength of the sintered silica material is very poor and has almost no practical use. Low purity of sand casting silica was used in the study, which resulted in a reduced melting temperature, and poor mechanical strength. The high roughness and low strength attained can be attributed to the large powder size and high porosity formed in the material after partial melting. The microstructure of the sintered part was not reported in the article; hence it is not clear if there were cracks in the sintered samples. It was suggested that increasing laser power and scanning speed could minimize the dimensional errors.

Exner *et al.* (Exner et al. 2008) developed a Q-switched pulse laser system with 532 nm wavelength for laser micro sintering of silica-alumina (SiO_2/Al_2O_3) powder. In this process, a relatively fine powder with particle diameter less than 1μm was used. The produced components had high geometric resolution and good surface quality. The pores and micro-cracks decreased after thermal treatment and a tensile strength of 120 MPa was achieved, which limits its use in high strength components as the tensile strength achieved is still low.

In biomedical research, Liu (Liu 2012) used direct laser sintering on a hydroxyapatite (HA) and silica slurry mixture to produce bone scaffolds. Tian *et al.* (Tian et al. 2012) simulated the laser sintering ceramic process and found that the laser scan strategy would influence the residual stress distribution, as well as sintering temperature.

Besides being used in earthbound applications, research on creating ceramic and glass parts from silica powder and sand via SLM for manufacturing on extra-terrestrial planets have been launched, since silica can be easily obtained on some of these places such as the moon and Mars (Fateri et al. 2013, Fateri et al. 2014).

2.2 Selective laser melting of zirconia

Zirconia is an oxide ceramic material that possesses high flexural strength, high fracture toughness and good corrosion resistance, especially yttrium stabilized zirconia (YSZ). The fracture properties of YSZ can be enhanced via transformation toughening mechanism (Mamivand et al. 2014). In dental application, zirconia offers unsurpassed mechanical properties which can be used to develop an all-ceramic implant system (Denry and Holloway 2010). Zirconia and its powder mixtures have been explored in SLM process.

Shishkovsky *et al.* (Shishkovsky et al. 2007) researched on the direct laser sintering of mixture of YSZ and aluminium/alumina powder. The ceramic parts they developed were porous systems without full density. Bertrand *et al.* (Bertrand et al. 2007) built zirconia-yttria (ZrO_2/Y_2O_3) component with a density of 56 % by partially melting the ceramic powder via SLS/SLM.

Yves-Christian *et al.* reported SLM of Al_2O_3/ZrO_2 components with almost 100 % density and flexural strength above 500 MPa, based on complete laser melting of the ceramic powder with high temperature preheating of 1600 ^oC during the process (Yves-Christian et al. 2010). In their experiment, the SLM samples have a surface roughness of $Rz = 60$ μm and dimensional accuracy of 150 μm. The poor surface quality can be attributed to the large melt pool formed under high temperature preheating treatment and low viscosity of the melt that outflows into the surrounding powder bed. Better surface quality can be achieved without preheating, but the mechanical strength of those parts is limited (Wilkes et al. 2013).

2.3 Selective laser melting of ceramic reinforced metal matrix composites

SLM processing of ceramic reinforced metal matrix composites (MMC) has relevance in applications for automobile and aerospace industries. Ceramic reinforced MMCs, with an optimum combination of metal matrix and stiffer, stronger ceramic reinforcements, provide improvements in wear resistance, strength and high-temperature mechanical properties. In SLM, it can be manufactured by either mixing the required individual components that make up the composite or by in-situ chemical reactions that produce the required components (Gu et al. 2012). Carbides are the most commonly used ceramics for MMCs, such as SiC/Al (Simchi and Godlinski 2010), TiC/(Fe,Ni) (Gåård et al. 2006) and TiC/Ti (Gu et al. 2012). Oxide ceramics and others can also be used (Leong et al. 2002, Czelusniak et al. 2014). Special functionally graded parts built by SLM processing was reported via the addition of small amounts of zirconia within the nickel alloy, which can be used in high temperature applications (Mumtaz and Hopkinson 2007). The *in-situ* formation of ceramic composites exhibits more advantages compared to pre-added composites because of the better wetting between ceramic and metal, as well as fine and uniform distribution of compounds (Rajan et al. 1998, Gu et al. 2012). Examples of this process include the formation of TiB_2 and TiC reinforced MMCs with the raw powder of copper, titanium and B_4C (Leong et al. 2002).

The resultant properties of SLM/SLS processed ceramics have been summarised in Table 1.

Table 1 Mechanical properties of laser processed ceramics

Material	Relative Density (%)	Strength (MPa)	Roughness (μm)	Reference
SiO_2		6 – 16 (compressive)	18 - 35	(Tang et al. 2003)
SiO_2			25 - 36	(Wang et al. 2003)
Al_2O_3	94			(Balla et al. 2008)
SiO_2/Al_2O_3		120 (tensile)	5	(Exner et al. 2008)
ZrO_2/Y_2O_3	56			(Bertrand et al. 2007)
Al_2O3/ZrO_2	100	Above 500 (flexural)	60	(Yves-Christian et al. 2010)

2.4 Other technologies used in direct additive manufacturing of ceramics

Variants of SLM techniques such as laser engineered net shaping (LENS^{TM}) have been shown to be able to process ceramics. In LENS^{TM}, powder is supplied coaxially to the focus of a high power laser beam, where a molten pool is then formed subsequently on the substrate (Lewis and Schlienger 2000).

Das *et al.* (Das et al. 2011) used LENS^{TM} to create a Ti-SiC composite coating on a titanium substrate, with the aim of improving the wear resistance of the substrate. After laser deposition, the coating contains TiC, $TiSi_2$, Ti_5Si_3 and SiC. Bernard *et al.* (Bernard et al. 2010) deposited lead zirconate titanate (PZT) structures directly on a metal substrate with a similar process, obtaining dielectric properties without any post heating treatment. Crack-free and dense PZT samples were reported using 150 W laser power, 5 mm/s scanning speed and 1.3 g/min powder flow rate. However, the best sample was porous with few cracks. The true density of the samples was not reported. The relative permittivity was measured as 430 (at 100 Hz) and decreased dramatically at higher frequencies.

The same group fabricated dense net-shaped structures of α-Al_2O_3 parts by laser deposition (Balla et al. 2008). Those Al_2O_3 parts had low fracture toughness of 2.1 MPa and relatively low density of 94 %. Cracks developed along the columnar gain boundaries when the tensile stresses were loaded. Su *et al.* (Su et al. 2011) also studied the rapid oxide eutectic growth of Al_2O_3/Y_2O_3 mixture by using high energy laser direct forming method, which differs from selective laser melting in that a powder layer is not deposited prior to melting, but is deposited onto the laser beam spot as the beam moves instead.

3. Challenges and potential in laser processing of ceramics

This section presents technical issues during laser processing of ceramics, including power layer deposition, laser-powder interaction, melting and consolidation mechanism and residual stress analysis, where further research is needed.

3.1 Powder layer deposition

The SLM system uses a blade to spread powder onto the substrate platform or the preceding part layer. Good flowability of powder particles is necessary to form a thin and even powder layer (Marcu et al. 2012). Powder bed density also has a direct influence on the final part quality. Powder parameters such as particle size and distribution, particle shape and flow characteristics are important and have to be well-controlled.

Powder size and distribution are the most influential factors for powder deposition in SLM. Smaller particle size is favourable for higher powder bed density and solid density. It was suggested that ceramic powders with particle sizes 10 times smaller than the layer thickness would be fit for the laser process (Shishkovsky et al. 2007). However, small powder particles pose a critical challenge in terms of flowability due to the tendency for powder agglomeration.

The small powder particles tend to agglomerate and adhere to the surface of the coater and printing platform due to electrostatic charges. This created a challenge in achieving a homogeneous powder layer deposition. Figure 4 shows the comparison of titanium powder and silica powder deposition by blade spreading process.

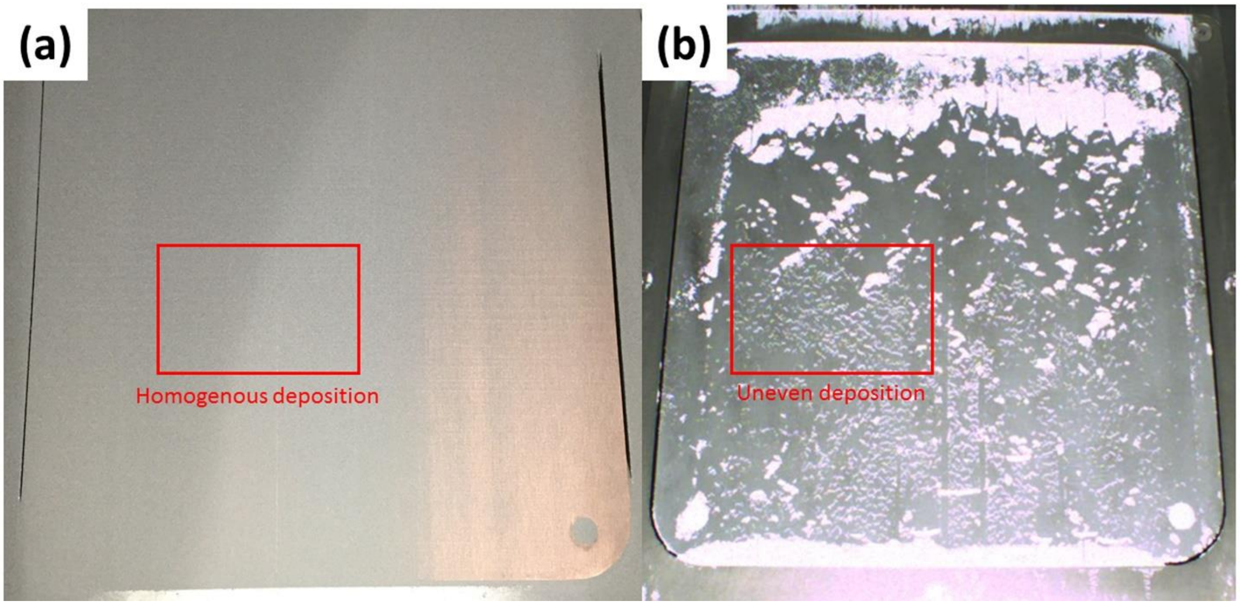

Figure 4 Powder deposition of (a) titanium and (b) silica on metallic substrate plate

The silica powder layer was uneven on the substrate due to particle agglomeration. Removing smaller particles from the powder feedstock may improve the flowability but will still negatively impact the density of the powder bed and subsequently the density of the fabricated part (Bourell et al. 2011). Spierings and Levy (Spierings and Levy 2009) suggested a range of metal powder size distribution selection for the SLM process. Similar research on ceramic powder size distribution is thus needed.

Powder shape has an influence on the powder spreading and layer density of the powder bed (Sordelet and Akinc 1988). Wilkes *et al.* (Wilkes et al. 2013) informed that the particle shape has a strong influence on the density of ceramic object produced by SLM. The improvement in density of SLM parts was thought to be related to the spherical powder shape. As there is no ceramic powder products specially designed for SLM process, it may not be easy to select the optimal powder for SLM. Different powder types such as crushed and atomized powder are commercially available. Ceramic powder produced by crushing method has an angular shape and relatively high bulk density, which is widely used in the industry. However, spherical particles atomized by spray drying technology with size ranging from fine primary ceramic particles of less than 1μm to tens of microns, have better flowability compared to crushed products. Nonetheless, density of spray dried powders are relatively low due to their porous characteristics (Naglieri et al. 2013). High temperature plasma technology can be used to fabricate spherical and densified micron-size ceramic and metal powders. In this process, the raw particles are fed into a plasma flame, where the particles melt and form into spherical shape due to their high surface area to volume ratio during the solidification (Kumar et al. 2006, Garcia et al. 2009). Flame treated ceramic powder may be good candidate for SLM process. Figure 5 shows the SEM images of the zirconia powder produced by spray drying and plasma spheroidization.

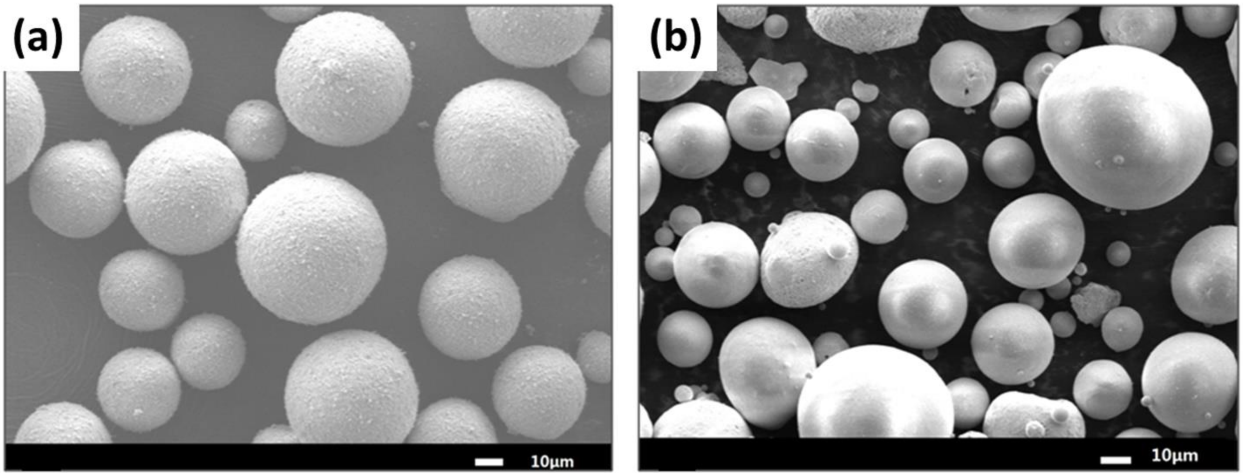

Figure 5 Zirconia powder particles prepared by (a) spray drying and (b) plasma treatment

Powder flowability measurement is necessary in SLM processes for quantitative prediction of the powder deposition behaviour. Common powder characteristics include angle of repose, bulk density, tapped density, Carr’s compressibility index and Hausner ratios, which are mainly for specific processes such as compaction, storage and volume filling (Rastogi and Klingzing 1994, Shah et al. 2008).Thus, it is desirable to measure flowability at flow conditions that are representative of SLM processing conditions (Mapar et al. 2013). There is not much information on powder performance index tailored to SLM of ceramics, hence, further research in this area is needed.

3.2 Laser-powder interaction

The SLM process uses Nd:YAG laser or fibre laser with wavelengths of approximately 1.06 µm as a heat source. A major challenge in the processing of oxide ceramics with near infrared wavelength lasers is related to their low and temperature dependent absorbance of the laser wavelength, as described by Fermi function (Zhang and Modest 1998, Regenfuss et al. 2007). The experimental absorbance measurement of ceramic powder is shown in Table 2 (Tolochko et al. 2000).

Table 2 Absorbance of ceramic powders under Nd:YAG and CO_2 lasers (Tolochko, Khlopkov et al. 2000)

Material	Absorbance (%)	
	Nd:YAG (λ = 1.06 µm)	CO_2 (λ = 10.6 µm)
Al_2O_3	3	96
SiO_2	4	96
SiC	78	66
WC	82	48

The absorption rate of oxide powders to laser with 1.06 µm wavelength is less than 10 %, which causes high laser energy loss and reduces production rate. Absorbance of ceramic powders for CO_2 laser with wavelengths of approximately 10.6 µm (Tolochko et al. 2000) is much higher, making the CO_2 laser more suitable to be used for direct ceramic processing, provided that the laser can provide the same energy output.

A Q-switched laser system has been developed for micro sintering of ceramics (Exner et al. 2003, Regenfuss et al. 2007). The Q-switched pulse laser would be more suitable for ceramic processing as the high energy laser pulses could increase the frequency of multi-photon excitation of electrons in the valence band and result in higher absorption rate. Highly dense ceramic parts several millimetres high, as well as metal parts, have been made by the pulsed laser system (Fischer et al. 2002, Exner et al. 2008, Mumtaz et al. 2008). However, the recoil effect of heated vapour induced by higher energy pulse would influence the stability of the powder bed and melt pool, as well as part qualities.

Optical interaction between the laser and powder could also impact the laser beam propagation profile in the powder bed. The powder bed in SLM machine has a high porosity as powder is spread freely without compression or heat treatment on the platform. The input laser beam undergoes multiple scattering or reflections in the bulk powder layer before it is being absorbed to generate the corresponding powder density and layer thickness (Gusarov and Smurov 2010, Kovaleva et al. 2014). Liu *et al.* (Liu et al. 2012) modelled the laser beam propagation of fibre laser through silica powder. In their model, it was noticed that the laser beam was further focused into the powder bed by the spherical geometry of the silica powder. Energy absorbed was not uniform with the maximum absorption areas occurring near to the third layer of the powder stacking. Streek *et al.* (Streek et al. 2013) used a ray tracing algorithm to describe the laser energy absorption and conversion during the laser sintering process, which could be regarded as a function of grain size, grain density, laser beam intensity and material properties. It was reported that powder particles with small sizes also require increased laser energy penetration to form melting pool due to their high surface-to-volume ratio.

3.3 Melting and consolidation mechanism

In the partial melting process, a laser beam melts the grain surfaces or particles with low melting point. The molten material act as a binder to form neck connections between particles and subsequently form a 3D porous structure after laser processing (Gusarov et al. 2003, Kruth et al. 2007). The temperature effects, gravity and capillary forces are the main driving forces for consolidation of molten material during partial melting (Kruth et al. 2007). For full melting, a moving molten pool is formed in response to a moving laser energy input. The interaction between the laser and powder induces high temperature gradients and significant temperature differences inside the small molten pool as the melt pool experienced high cooling rates at the passing of the laser beam (Vilaro et al. 2011, Van Belle et al. 2013). The temperature gradients can cause rapid motion, also known as Marangoni flow. This effect becomes increasingly significant for ceramics as ceramics have higher melting point and lower thermal conductivity compared to metals. Similar phenomenon can be seen in other laser-based processing of ceramics such as laser welding of aluminium and alumina (Hirsch et al. 1998, ThomazinL et al. 2014).

The high viscosity of the ceramic melt has strong influence on the final density of the part fabricated by SLM. There are two aspects that the viscosity can affect during the laser melting and solidification process. One aspect is the flow and deformation of the melted particle to join into a bigger melt as droplets merge, and another aspect is the speed of air bubbles escaping from the melt, if the entrapped gas cannot diffuse out. Apparently, when the viscosity is very high, the melt might not be able to flow even when it is fully melted, for example many melted glasses exhibit non-Newtonian fluid behavior, like honey or lava, rather than liquid. Although there are very few data on viscosity of ceramic, one of the most well studied is SiO_2, which has viscosity of approximately 10,000 Pa.s at 2400 K (Nordine et al. 2009). This temperature is well above the melting temperature of SiO_2, which is 1986 K. The viscosity of molten optical glasses is in the range of a few hundreds Pa.s to tens of thousands Pa.s, depending on composition and temperature (Shartsis and Spinner 1951). In comparison, most metals have low viscosity at melting point, normally lower than 10 mPa.s, which is about six order of magnitude lower than ceramics. For example pure iron has viscosity of 4.7 - 6.2 mPa.s at 1808 K and Ti has viscosity 2.2 - 5.2 mPa.s at 1998 K(Kaptay 2005). This can be compared with the viscosity of water and peanut butter at room temperature which is 0.89 mPa.s, and 150 - 250 Pa.s, respectively.

Apparently the low viscosity of metal melt facilitates flow and merging of melted particles into full density as droplets merge. However the high viscosity of ceramic melt could dramatically hinder such flow and merging. Low surface tension corresponds to low sintering speed. The low surface tension of ceramic melt causes the low driving force for droplets to merge. The merged melted particles have lower specific surface area, hence, lower free energy. Consequently, the sintering speed is inhibited. High surface tension favours droplet merging as it helps reduce the free surface energy. It is difficult to achieve high density of ceramic by SLM without sufficient flow and droplet merging.

The low viscosity of metal melt also facilities air bubbles escape from the melt consolidation and during the SLM of metals. Oxygen can diffuse out through volume diffusion of vacant lattice sites after 6 – 12 hours of sintering if oxygen is trapped in isolated pores during the liquid phase sintering of oxide ceramic. Such case is demonstrated when MgO-$CaMgSiO_4$ undergoes liquid phase sintering (Kim et al. 1987). However diffusion will not happen for trapped nitrogen gas as there is no nitrogen vacant lattice site in oxide ceramic. Thus diffusion is not the main mechanism when nitrogen gas is trapped in melt. Instead, bubbles rising directly from the liquid can be the main driving force of densification.

Terminal velocity of the bubble rising in a liquid can be determined as (Yersel 1991)

$$v = \frac{2(\rho_l - \rho_g) g r^2}{9\eta} \qquad (1)$$

Where v is the velocity of the bubble when it reaches the surface, ρ_l and ρ_g are the density of liquid and bubble respectively, g is gravity, r is radius of the bubble, and η is viscosity of the liquid. It can be seen from the equation above that the velocity is inversely proportional to the viscosity. As time is inversely proportional to velocity, the time needed for the bubble to escape from the melt is approximated to be proportional to the viscosity. For a 20 µm diameter bubble in iron to travel in 100 µm distance, supposing the viscosity of iron is 5 mPa.s and speed is uniform along the rising direction, the time required is estimated to be 294 ms. However the viscosity of Silica is about 10^6 times higher than that of metals and the density is lower (2648 kg/m^3). It can be calculated that the same size 20 µm air bubble requires 1.7x10^6 s (or 20 days) to travel 100 µm distance in melted silica under 2400 K. So neither diffusion nor bubble rising could happen during the SLM of ceramic when the viscosity is high. Eutectic ceramics have been selected for SLM to reduce viscosity. At eutectic point, a material composition has the lowest melting point, hence, this would help in reducing the temperature that the SLM has to reach during direct ceramic processing. However, the requirement to use eutectic ceramics limited the range of ceramic which can be processed.

Surface tension can cause opposite effects to the SLM process. Surface tension is the driving force of sintering (Mackenzie and Shuttleworth 1949). The sintering speed is proportional to $\gamma/2\eta$, where γ is surface tension and η is viscosity. Hence, higher surface tension gives faster sintering speed. However, low surface tension favours the elimination of gas bubbles (Singer and Singer 1963). The surface tension of metal melt is in the range of 1000 mN/m but ceramic is one order of magnitude lower, which typically is in range of 100 mN/m. The Bond number is given by

$$Bo = \frac{\Delta\rho g L^2}{\sigma} \quad (2)$$

Bond number is usually used to compares gravitational and surface tension effect (Gu et al. 2011), with $\Delta\rho$ the difference in mass density between the two fluids, g the gravity acceleration, L a characteristic length scale, and σ the interfacial tension,. The characteristic length, L, in additive manufacturing is small so the $Bo \ll 1$, which signifies that gravity effect is much smaller than surface tension effect. Therefore, it is unlikely that gravity has major effect on ceramic densification by SLM.

Therefore, it is contradicting that high surface tension favours the sintering speed of ceramic while low surface tension favours the bubble elimination. Unfortunately the research in this area is lacking. From the research of the flow of glazes during firing, glass workers have pointed out that low surface tension favours elimination of gas bubbles during glass firing, while high surface tension favours reabsorption of the bubbles during the cooling of the glass (Singer and Singer 1963). Hence, it is delicate to maintain the balance on the required surface tension to optimise SLM of ceramics.

During the SLS/SLM process, three types of process behaviour can be observed, namely: formation of irregular and unstable melt tracks, continuous and stable melt tracks (Yadroitsev et al. 2010), and presence of balling effect (Tolochko et al. 2004). These behaviours are influenced by powder characteristics and process parameters, such as laser power, scanning speed and layer thickness (Klocke et al. 2003, Kruth et al. 2007, Yadroitsev and Smurov 2010). Balling effect occurs due to poor wetting between the molten pool and underlying solid, causing the molten pool to breakup into small ceramic spheres (Gu and Shen 2009). As for laser processing of ceramics, the poor wetting behaviour of the liquid phase often leads to formation of loose droplets and inhomogeneous layers. Figure 6 shows the balling effect during SLM process of zirconia/alumina mixture done in-house. A commercially available Alumina (Al_2O_3) with purity 99.8 % and particle size distribution < 5 μm with average particle size of 3 μm from Industrial Powder Company, USA, and 3 mol. % Y_2O_3 stabilized Zirconia (ZrO_2) nano-powder (30 - 60 nm), 99.95 % purity from Inframat Advanced Material, LLC was used. The powders were mixed together in the eutectic ratio and then spray dried before SLM. The balling phenomenon needs to be characterized further with experiments.

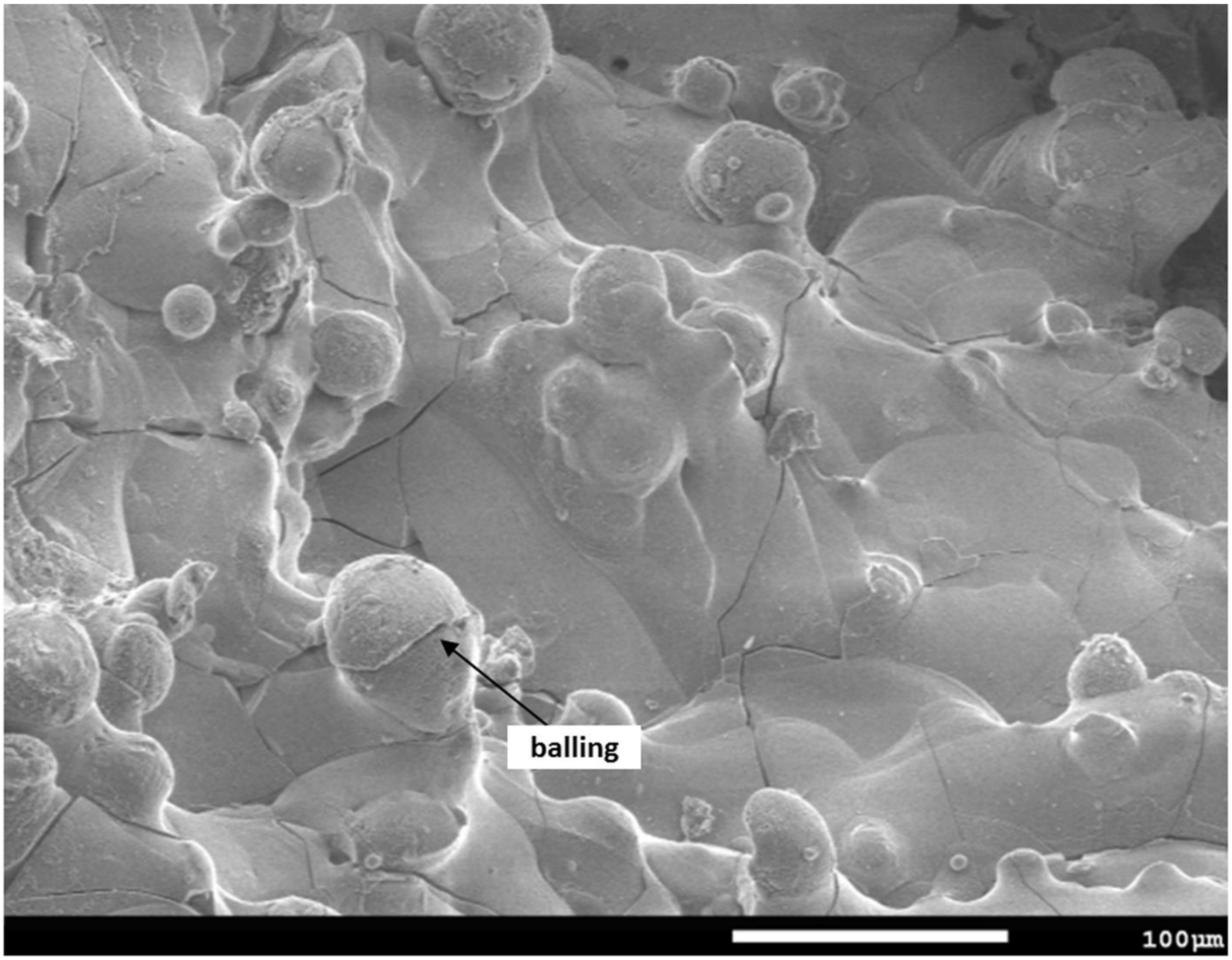

Figure 6 Balling effect in SLM process of ceramic powder

Modelling work on temperature and stress profiles of ceramics materials using various parameters during laser processing has been reported. Li *et al.* (Li et al. 2004, Li et al. 2004) developed a volumetric heating source model for laser surface melting of ceramics. However, fluid flow should be added to their models for large molten pools. Tian (Tian et al. 2012) investigated the effect of different laser scan patterns in laser direct sintering of ceramics. Further studies on simulating SLM processing of ceramics are required for parameter selection and optimization.

3.4 Thermal and residual stress analysis

The presence of thermal effects in full melting of powder layer via laser processing typically generates significant residual stresses and strains during rapid solidification. In the case of partial melting without infiltration, residual stresses were also reported (Mercelis and Kruth 2006). Residual stresses depend on temperature gradient and material physical properties. Ceramics generally allow only low plastic deformation at room temperature (Wakai 1991), with typical elongation at break at about 0.2 % to 0.6 % for oxide ceramics.

Rapid melting and cooling of ceramics in SLM will also negatively impact the grain size of the ceramic part produced. As SLS/SLM laser system is unale to provide the capability to reach high temperature that allows solid state sintering and slow diffusion, there is a risk of high temperature gradient occurring during ceramic processing. This undesired condition often results in cracks in the ceramic parts (Ouyang et al. 2001). Figure 7 shows the top surface SEM image of zirconia/alumina part built by SLM in-house with the same powder mixture described previously. A highly dense surface with cracks can be observed.

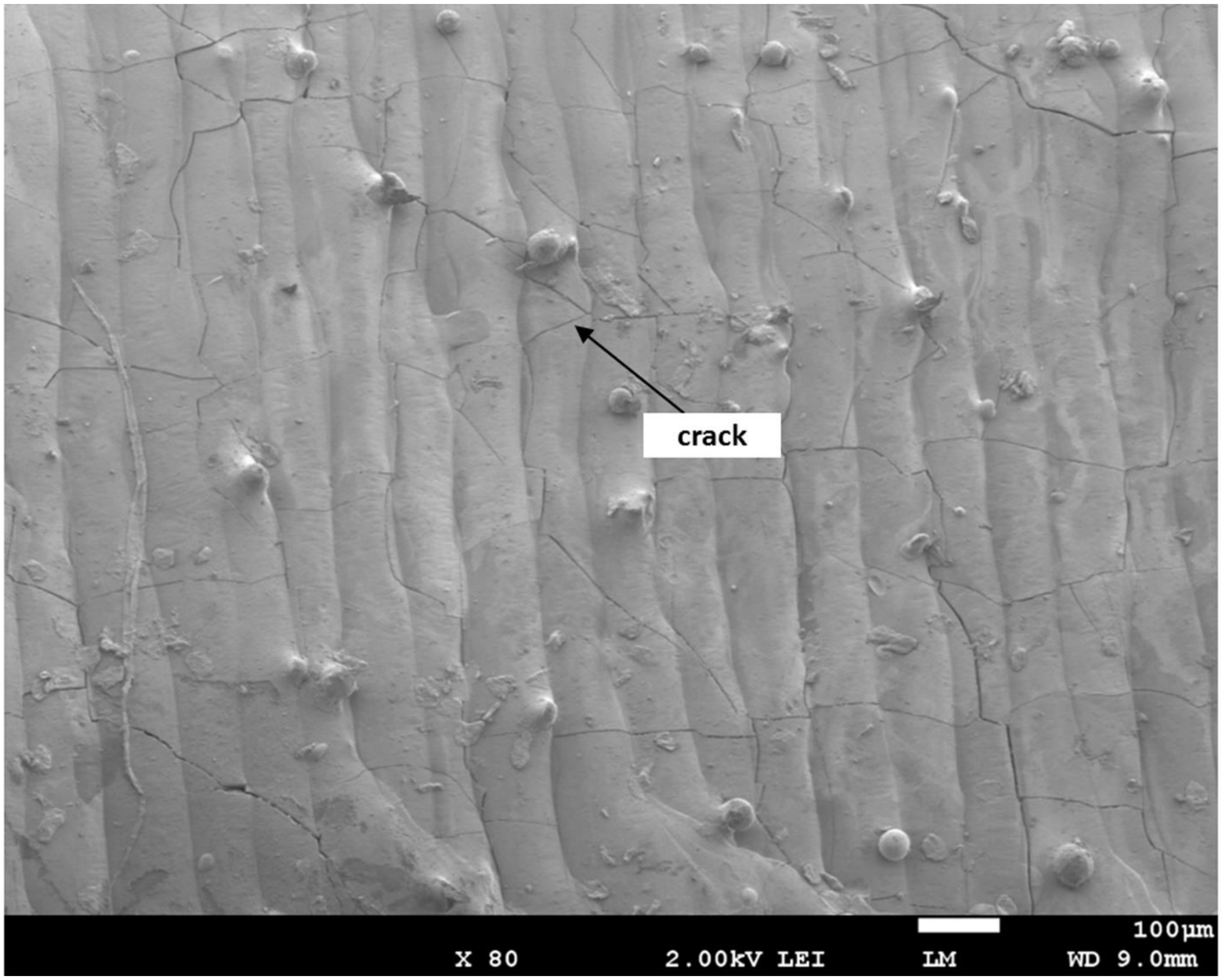

Figure 7 SEM image of ceramic part surface built by SLM

Li *et al.* (Li et al. 2004) found that in the laser-ceramic interaction process, the whole laser melted track is in tension, and cracks occur in the region of highest tensile stress throughout the solidified tracks. It is well known that ceramics typically have much higher compressive strengths than tensile strengths. Furthermore, ceramics have much lower fracture toughness than metals. For example the fracture toughness of 316L stainless steel is in the range of 100-200 MPa.m$^{1/2}$. However the fracture toughness of silica is 2 order of magnitude smaller, which is about 0.6 - 0.7 MPa.m$^{1/2}$. Although some technical ceramics have higher fracture toughness, such as fracture toughness of alumina and zirconia is about 4 MPa.m$^{1/2}$ and 6 - 7 MPa.m$^{1/2}$, respectively. However this fracture toughness value is still much lower as compared to metals. A higher heating temperature of base platform even the whole chamber will be needed for ceramics compared to metals to reduce the temperature gradients as well as thermal stresses. Hagedorn *et al.* (Yves-Christian et al. 2010) used preheating setup during the process, reaching temperature above 1400 °C to build highly dense and crack-free parts. Challenges with high temperature preheating include formation of coarse grain structure which will lead to poor mechanical strength, and rough surfaces and imprecise contours due to the large melt pools generated and sintering of the powders close to the solidified tracks. Other approaches to reduce the residual stress focus on the investigation of factors such as sample height, exposure strategy and the use of pulse lasers (Fischer et al. 2004, Mercelis and Kruth 2006, Yves-Christian et al. 2010).

3.5 Potential applications of laser processed ceramics

Direct laser sintering/melting of ceramic composites has much potential, especially for applications in the medical field. During laser process of silica based ceramic powder mixture, such as Al_2O_3/SiO_2 and $Al_2O_3/ZrO_2/SiO_2$, silica was melt to form a liquid glass phase and bridge other particles together to build a porous structure (Yang et al. 2003, Wang et al. 2013). Similar process in SLS of glass-ceramics, which consists of at least one glass phase and one crystal phase, can be used for medical implants. The density and microstructure of the part depend on the contents of the ceramic composites and interaction between components of the powder mixture, as well as the rate of heating and cooling (Grüner and Shen 2011). Glass systems such as $SiO_2/Al_2O_3/P_2O_5/CaO/CaF_2$ (Goodridge et al. 2007) and $SiO_2/CaO/Na_2O/P_2O_5$ (Liu et al. 2013) have been studied. Apatite and mullite phases can be produced with good fracture toughness and mechanical strength from the glass-ceramic system $SiO_2/Al_2O_3/P_2O_5/CaO/CaF_2$ (Goodridge et al. 2007). Bioactive porous scaffolds were made by SLS from amorphous glass powders of

$SiO_2/CaO/Na_2O/P_2O_5$ to form parts with high density, large degree of crystallinity and superior fracture toughness (Liu et al. 2013).

4. Conclusions

Selective laser melting has shown its potential in manufacturing metal parts with complex shapes directly with minimal processing steps. However, many challenges remain in fabricating fully dense ceramic parts using direct melting or sintering. Successful integration of ceramics into selective laser sintering/melting will spur significant developments in the field of technical ceramics.

Further research on the technical issues and challenges of the SLM processing of ceramic powders is required, especially in resolving challenges such as:

Powder layer deposition

There is need for investigations of powder size distribution optimal for laser sintering and melting of ceramics, to improve flowability and reduce powder agglomeration. These will lead to thin and even powder layer deposition which is critical for the processes.

Laser-powder interaction

Oxide ceramics have low temperature dependent absorbance of certain wavelengths. There is need to develop laser sources that can provide the energy output needed to sinter or melt the ceramics, while keeping their wavelength suitable for absorption by the ceramics.

Melting and consolidation mechanism

Melted ceramics have high viscosity that can hinder the flow and air bubbles escaping from the melt pool. These results in porosity formed in sintered or melted ceramic parts. Simulations on flow characteristics of ceramics should be carried out to study the mechanisms involved in SLS/SLM processing of these materials.

Thermal and residual stress analysis

Ceramic parts are prone to cracking under high temperature gradients which are apparent in SLS/SLM. There is need for studies on thermal and residual stress so as to control these effects on the processed parts.

When these challenges are resolved, the future of direct laser sintering or melting of ceramics will be disruptive to various industries, such as the aerospace and biomedical sectors.

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