Surface Morphology of a Catalytic Wall Microreactor Constructed by Direct Metal Laser Sintering Process

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Micro-reactors technology have attracted considerable interest in chemical processing due to their potential advantages regarding improvements on mass and heat transfer, smaller size, energy and cost saving and safety. Various types of micro-structured reactors have been developed in the last decade. Among them, the most promising concept considers stacked systems of channelled metallic platelets, coated with active catalysts. This systems are fabricated using any traditional tooling, but in this work a relatively new technology enabling that three-dimensional parts can be easily fabricated was used. It is based on DMLS (Direct Metal Laser Sintering), a process that uses a metal powder and a laser beam to produce directly metal parts. This process is one of a few Rapid Prototyping (RP) technologies which possess the capability to produce metal parts and prototype tools directly from powders with a high precision. In this paper a micro-channels plate was fabricated using DMLS process and micro-channels surface for catalyst deposition was evaluated. The results of SEM study reveal that a porous surface on the walls was formed because powder was not wholly sintered. For microchannels plate applied for gas-phase reactions this porous surface is especially interesting because the surface area is increased and the mass of catalysts impregnated on the walls can also be increased, which consequently improves the conversion of products. Thus, the DMLS not only can facilitates the rapid development of catalytic wall microreactors, but also permit the control of the structures formed during sintering which are extremely relevant for some processes in micro-reactors for gas and liquid phase.

1. Introduction

Microreactors technology have attracted considerable interest in chemical processing due to their superior advantages to improve mass and heat transfer, smaller size, energy and cost saving (Men et al, 2007), and additionally, safety. The benefits of micro process engineering are currently within the focus of the world research, and numerous reactors have been built up applying micro-technology (Kolb et al, 2007).

Microreactors offer high heat and mass transfer coefficients due to high surface to volume ratio, lower pressure drop compared to classical packed bed reactors, good structural and thermal stability and more precise control of the process conditions leading higher product yields. They are generally also referred to as microstructured reactors, microchannel reactors or microreactors (Cai et al, 2010). These devices contain open paths for fluids with dimensions in the submillimeter range and mostly of microreactors have multiple parallel channels ranging from 10 to several hundred micrometers (Kiwi-Minsker and Renken, 2005).

Generally, to generate micro channels systems several techniques may be applied such as micro milling, electro discharge machining (EDM), wet chemical etching, punching, embossing, laser micro machining (ablation) and sintering (Hessel et al, 2005). In this way, Direct Metal Laser Sintering (DMLS), as a typical Rapid Prototyping (RP) technique, is proposed to build-up microreactors. It enables the quick production of complex three-dimensional parts directly from metal powder. This process uses a laser that is directly exposed to the metal powder (Khaing et al., 2001) and creates parts by selective fusing and consolidating thin layers of loose powder with a scanning laser beam process. Additionally, due to its flexibility in materials, shapes and control of construction parameters, porous metallic component may also be produced (Gu and Shen, 2008).

Bearing this in mind the objective of this paper is propose the construction of micro-channels plate using DMLS system and evaluate the surface for catalyst deposition.

1.1 DMLS Principle

Figure 1 shows a schematic diagram for construction of any part by machine. First, the building and dispenser platform are lowered by one layer thickness for that the recoater blade can move without collision. When the recoater stands in the right position the dispensor platform rise to supply the amount of powder for the next layer. Then, the recoater moves from the right to the left position; in this way the metal powder is spread from dispenser to the building area and the excess metal powder falls into collector. Then, the head scan move the laser beam through two-dimensional cross section and is precisely switched on and off during exposure of designated areas. The absorption of energy by metal powder will generate the cure and sinter of the already solidified areas below. This process proceeds layer-by-layer until all parts in a job are completed (EOS, 2009).

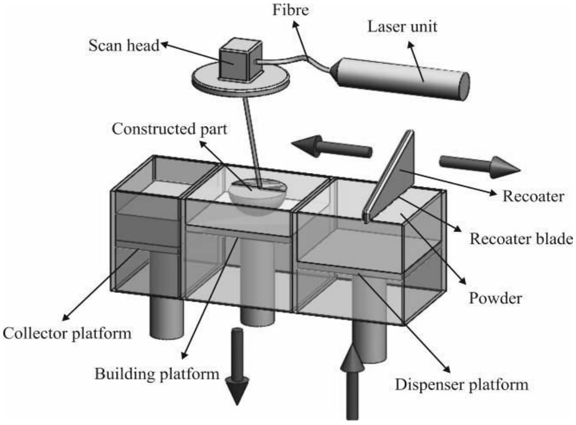

Figure 1. Schematic diagram of the DMLS system.

1.2 Exposure strategies

Prescribed Figure (2) shows the laser scanning the top surface of a thin powder layer to form the area enclosed by cross-sections of the sliced object. Initially, all contour of the layer structure is exposed with a laser power (L_{pw}) and contour speed (C_{sp}). As the diameter of the sintered zone is usually larger than the laser diameter, effective laser diameter (Senthilkumaran et al, 2009), it is necessary to compensate the dimensional error and the laser beam must be shifted by half the curing width from the contour to the inside, to make sure that the contour of the later part will correspond exactly to the original dimensions. This correction of the position is called Beam Offset (BO).

During hatching, the laser beam moves line after line several times to assure that the sintering process can unroll completely, because it maintains the temperature for a long period. The distance between the lines is called hatch spacing (Hs) and is set about one quarter of the laser beam. Here the Beam Offset value is again defined with respect to the edge of the boundary, and if this value is higher or less than the correct value, the particles of the irradiated region may be either not sintered or over-sintered.

Another important parameter is the layer thickness. Regarding this parameter if the value is too high, no optimal adhesion between the single layers can be achieved because the curing depth is not high enough; furthermore, mechanical tension can be generated through this layer which can lead to detachment of the layer below. If the selected value is smaller, a tearing-off of a structure can happen during the recoating process, since the sintered particles get struck between it and recoater blade (EOS, 2009).

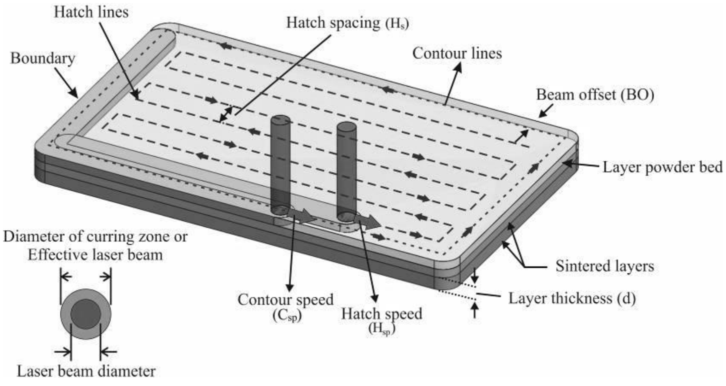

Figure 2. Exposure strategies and process parameters.

2. Manufacturing methodology

In general, to reproduce a virtual object (designed in CAD software) to a physical object in Rapid Prototyping (RP) process, there are two stages, that include the data preparation stage and the object construction procedure. First a digital model is created assisted by a CAD system. After that, it can be exported to STL (Stereolithography format) from the model file; then the object is sliced in many layers, where the parameters for components construction are also selected. Finally the part is constructed using RP machinery (Jardini et al., 2008).

In this work, DMLS machine (EOSINT M270®) was used for microchannels plate fabrication. The EOS Aluminium powder (AlSi10Mg) with particles ranging from 25μm to 45μm was used. The aluminium alloy have good thermal conductivity properties, light weight and high strength. The dimensions of microchannels plate is 50 x 30 mm and is composed by 30 parallel channels of 0.5 mm width x 0.25 mm height. These structures can be multiplied and arranged to form a stack.

2.1 DMLS parameters

Maximum power available with Ytterbium laser in the DMLS machine is 200 W. In this work 58.5% (117 W) and 29.25% (58.5 W) of the maximum power was used. Scan speed from 225 to 400 mm/s was chosen for a shorter construction time. The laser beam diameter and energy density used are 40μm and $2.328 \times 10^{10} W/m^2$, respectively. The process parameters used for contour exposure are lower laser power and higher scan speed compared to hatching exposure in order to achieve a porous surface finish. The process parameters used in the present study are given in Table 1.

Table 1. DMLS process parameters for the components construction.

Parameter	Value	
	Hatching	Contouring
Laser power (W)	117	58,5
Scan speed (mm/s)	225	400
Hatching spacing (mm)	0.18	Not applied
Layer thickness (μm)	30	
Beam offset (μm)	20	
Laser beam diameter (μm)	40	
Effective laser beam (μm)	200	
Scanning pattern	Stripes (alternating 45º)	

2.2 Morphology analysis

The Surface morphologies of laser sintered samples were characterized using a Leica LEO 440i scanning electron microscope (SEM), operated at an accelerating voltage of 20 kV.

3. Results and discussion

The results of SEM study, Figure 3, shows the morphology of sintered microchannels plate without any post-treatment. The surface can be considered as a porous surface because powder was not wholly sintered, which can be observed by continuous ball-like agglomerates of metallic powder.

Gu and Shen (2008) carried out study where the porous structure was obtained, which were tested different process parameters in the preparation of multi-layer components using 316L stainless steel. A fully dense sintered surface was achieved using higher laser power and lower scan speed by means of continuous scans tracks and an excellent bonding between them. However, at a higher scan speed, laser sintering at the same laser power produced discontinuous scan tracks consisting of significantly coarsened individual agglomerates in a spherical shape. The best porous structure obtained was processed at a lower laser power and a scan speed, which was characterized by open pores formed in sample surface.

In this work, lower laser power was used to sinter contour paths, and a higher power to hatch lines, which resulted in a porous structure on the walls. For microchannels plate applied for gas-phase reactions this porous surface becomes especially interesting because the surface area is increased and the mass of catalysts impregnated on the walls can also be increased, which consequently improve the conversion of products. In fact, for catalytic processes the superficial area is an important issue to be considered.

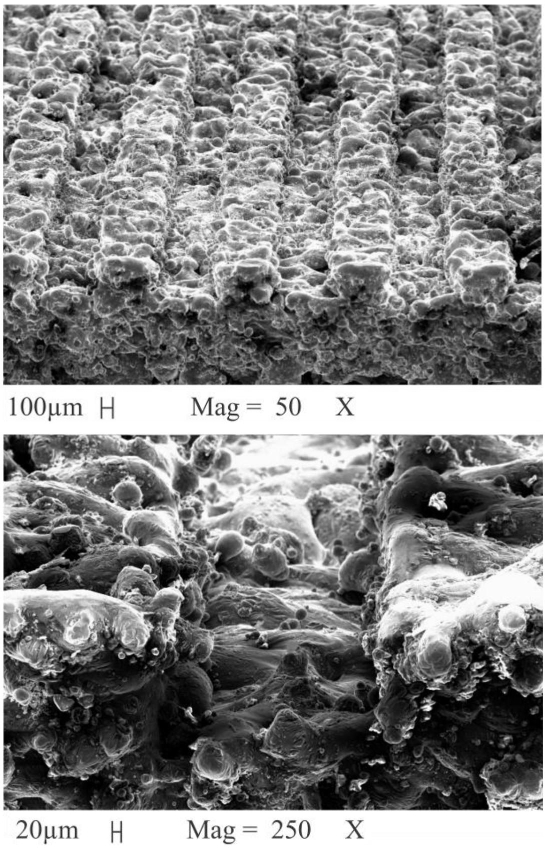

Figure 3. SEM images showing the surface morphology of microchannels plate.

Thus, different processing parameters lead the material to achieve a fully dense or porous structure and choosing correct parameters the DMLS not only can facilitates the rapid development of catalytic wall microreactors, but also permit the control of the structures formed during sintering which are extremely relevant for some processes in microreactors for gas and liquid phase.

4. Conclusion

In this paper, the construction of microchannels plate was proposed by using DMLS system (EOSINT M270). This system achieved a high precision and can be used to construct microreactors in aluminium-alloy, but can be extended using different materials such as cobalt-chrome, nickel-alloy, stainless-steel and titanium.

Optimal processing parameters, especially laser power and scan speed, are required in order to realize a feasible mechanism by means of sintering of the powder to obtain a porous structure or a dense structure on the parts, which is interesting for a rapid development and study of microreactors.

A porous surface increase the surface area which can enhance the mass of catalysts deposited on the walls, which consequently improve the conversion of chemical reactions.

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