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Laser surface treatment of sintered alumina

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Abstract

Sintered alumina ceramics are used as refractory materials for industrial aluminum furnaces. In this environment the ceramic surface is in permanent contact with molten aluminum resulting in deposition of oxidic material on its surface. Consequently, a lower volume capacity as well as thermal efficiency of the furnaces follows. To reduce oxidic adherence of the ceramic material, two laser-based surface treatment processes were investigated: a powder-based single-step laser cladding and a laser surface remelting. Main objective is to achieve an improved surface quality of the ceramic material considering the industrial requirements as a high process speed.

Keywords: rapid manufacturing; laser sintering; laser cladding; ceramic; alumina; aluminum oxide; remelting; surface treatment

1. Introduction

In industrially used aluminum furnaces the formation of corundum is a well-known problem. Corundum is the non-scientific name of α-Al_2O_3 which is one of the crystalline modifications of aluminum oxide. It forms out of the amorphous modification γ-Al_2O_3 of aluminum oxide as a result of temperatures above 700 °C in long-term periods [1]. Corundum is formed frequently on the refractory lining at the transition zone between the surface of the aluminum melt and the oxygen containing atmosphere. It is a result of oxidation of the molten aluminum as well as a reduction reaction of molten aluminum and the refractory material of the lining [2]. The molten aluminum chemically reduces the silica based refractory material as well as other reducible oxides. The formation of so called internal corundum results in differences in strain coefficients inside the refractory lining material that can cause cracks and failure. In the case of corundum growth from the inner lining, the effective volume capacity will decrease until the furnace needs to be shut down [2]. The process of corundum-formation in aluminum melt is a function of time and directly of temperature. Although the melting temperature of

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high purity aluminum is at approximately 660 °C overheated zones occur with the refractory lining inside a furnace. Particularly heating systems in the form of gas burners lead to areas heated up significantly above 1000 °C that temporarily stimulates the corundum formation.

To prevent the refractory lining of corundum formation, various techniques exist. It is recommended to use low silica refractory material to reduce its tendency of reduction. Using low cement castables and refractories containing non-wetting additives, the adherence of molten aluminum to the lining is decreased. The composition of the refractory lining material as well as a surface treatment, e.g. with a phosphoric acid, play an import role in avoiding penetration of melt into the lining material. An overheating of the melt or direct flame impingement of the lining promotes the corundum formation too [3].

In the present work, two laser-based surface treatment methods, one powder-based and one non-powder-based, were investigated. Priority was to achieve a significantly improved surface quality of the investigated sintered ceramic refractory materials. The modified surface should have a positive impact on the wetting behavior of the aluminum melt i.e. primarily less corundum formation and adherence due to a smooth, even and defect free refractory material surface.

Laser-based surface treatment of surfaces is particularly established for metallic materials. The single-step laser cladding is a powder-based process. The materials surface is heated up above the melting point by a laser radiation. Synchronously powder is fed coaxially or laterally via a nozzle to the molten substrate zone. The melting pool and the added powder lead to a high formation of material layer which has a solid metallurgical bond to the substrate material. Single-step laser cladding can be used as Rapid Manufacturing process but is most commonly used to increase the wear resistance of functional surfaces [4] or to maintain high pressure turbine blades of aircraft engines [5]. Krishna et al. investigated the single-step laser cladding successfully by building up various shapes of alumina ceramics [6]. Laser processes with ceramic powder material is of industrial significance, particularly when applied to two-step Rapid Prototyping processes and also known as selective laser melting. It is used to produce all-ceramic frameworks for dental restoration applications like bridges [7].

In comparison to laser cladding, the laser remelting of surfaces by laser radiation is a simplified process without any additional feeding of powder material into the process zone. After the substrates surface is temporary warmed up above its melting point, the material cools down rapidly and solidifies. The material’s microstructure changes to finer grains resulting in an increased wear resistance behavior [8]. In case of gray cast iron metastable phase structures like ledeburite might occur [9]. The laser remelting of ceramic coatings may result in a change of the materials properties concerning the material’s microstructure, its density and as well the hardness [10].

2. Experimental work

The experiments were carried out with cylindrical shaped white fused alumina substrates of a diameter of 50 mm and a varying height between 10 and 20 mm as shown in figure 1 d. They are made of powder material with a grain size distribution from 0 to 6 mm of an alumina content of 93 % in addition to oxides containing Mg, Na and Ca, including an organic binder of 3,5 %. Under high molding pressure of around 2000 kg/cm^2 a square brick is shaped and machined afterwards to obtain the cylindrical substrates. During the subsequent two days firing process at a temperature of 1700 °C, the binder disappears. The smallest granulate particles create a physical ceramic bond.

The white fused alumina powder particles used in the laser cladding process are non-spherically shaped, with a grain size range of 45 to 100 microns and can be fed using a disk type powder feeder.

The laser cladding tests were performed with a fiber-coupled diode-pumped laser source with a maximum output power of 680 W, as shown in figure 1 a. In the focal plane the beam diameter is about 900 μm. The welding head contains the beam shaping optical elements as well as the coaxial powder leading nozzle which is positioned by a 6-axes linear stage machine base. Two different laser scanning systems were used to remelt the ceramic surfaces: a 1000 W solid state system as shown in figure 1 b and a 2250 W CO_2 system.

To study the laser processes, a range of relevant parameters were tested. Concerning the laser powder cladding process, the following key parameters were identified: laser power, feed rate, powder feed rate and the preheating temperature. Initially, the dependency of the powder feed rate on the rotation speed of the feeder disk was determined. Test structures of single laser cladding tracks with a length of approximately 15 mm were generated on the substrate. The thickness as well as the height of the structures was analyzed in connection to the different process parameters. To improve the bond strength between substrate and built up structures, preheating tests by laser radiation were performed. To confirm the parameter setup, complete substrates were surface treated as well.

Regarding the laser remelting powder free process as shown in figure 2 c, the following parameters were investigated: laser power, scanning speed, scanning strategy and preheating temperature. Similar to the cladding tests, linear test structures were generated to analyze the influence of the parameters on the results. To evaluate the parameters the complete substrates were treated subsequently as shown in figure 1 c. Additionally, to avoid the formation of cracks within the remolten surface, a strategy of preheating using an industrial oven was implemented as shown in figure 1 d.

For both laser process variants, the focal positions were chosen based on the highest intensity and biggest melting pool occurrence.

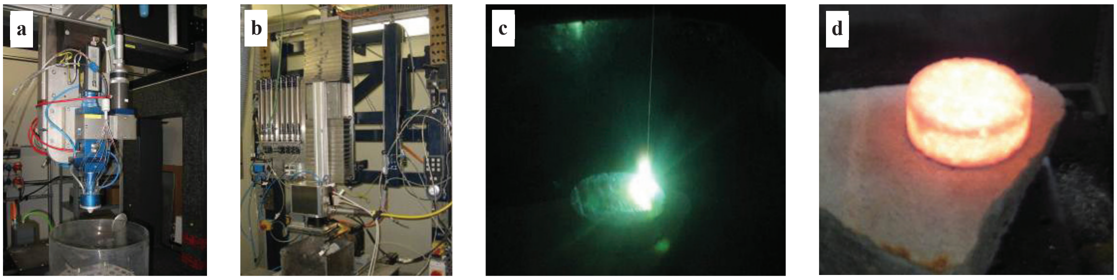

Fig. 1. (a) laser cladding system; (b) laser scanning system; (c) remelting process by laser scanning system; (d) preheated substrate

3. Results and Discussion

3.1. Laser powder cladding

To reduce the influence of process parameters of laser cladding the tests were simplified to single cladding tracks as shown in figure 2a. In order to achieve a satisfactory cladding height the powder feed rates had to be adjusted to relatively high values from 0.1 to approximately 1 g/min. At lower feed rates there was no cladding material on the surface, only the molten track of the substrate material. The influence of the laser power on the cladding tracks was exemplarily for a range of laser power from 30 to 180 W. Up to a laser power of approximately 120 W, the width as well as the height of the cladding tracks increased continuously. From 120 W to higher power the cladding height decreases as the melting pool becomes larger and deeper as a result of the higher energy input. Depending on the feed rate, there is a maximum achievable cladding height. Another limitation is the high temperature of much more than 2000 °C in the process zone, followed by the extreme lightning effects which may affect the powder nozzle due to the short distance of 10 mm to the surface. Figure 2 b shows the correlation of increasing sample weight and increasing laser power. More powder particles can be melted into a new layer with a wider and deeper track melt pool.

Due to the lack of mechanical stability of the single track layers, a preheating treatment of the surface by defocused laser radiation was performed. However, an improved bonding of the built up layers, as well as a significantly increased build up of material could not be determined. It is to be assumed that surface preheating by laser radiation delivers too little energy to achieve effective surface temperatures. Investigations from Nagel in the field of laser-based ceramic welding recommended a preheating up to 1500 °C to guarantee crack free results [11]. Consequently, an industrial furnace was used for extended preheating treatment strategies.

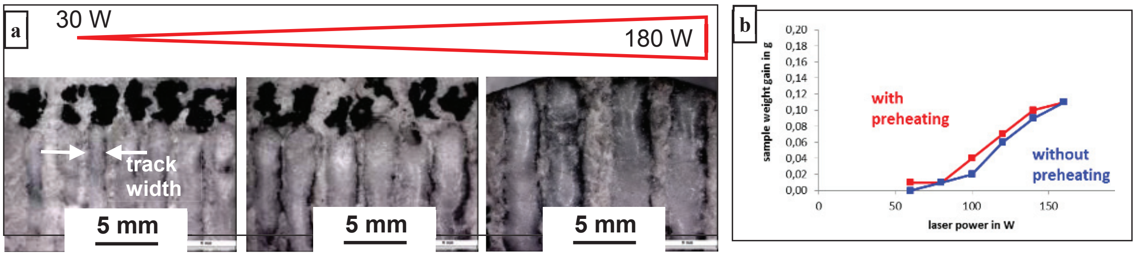

Fig. 2. (a) track width of test specimens produced with increasing laser power from 30 to 180 W, 96 mm/min, 0,6 g/min; (b) increasing weight gain of samples produced with increasing laser power

Similar effects of weight increase while increasing the feed rate is evident in figure 3 a. It increases asymptotically to a certain feed rate value. Exceeding this value resulted in no significant increase of material weight. From a certain point, the melting pool is saturated of new powder particles and the material build-up stagnates. To validate the investigated parameters substrates were cladded on a large area as shown in figure 3 b. The experiments of laser cladding resulted in successfully laminar treated surfaces, having an average ceramic cladding height of 300 microns. The distance of the meander hatching pattern was adjusted to obtain a totally closed and gap-free cladded surface. Therefore the single cladding tracks have to overlap sufficiently. Choosing 1.5 mm as hatching distance fulfilled these requirements. Nevertheless, the low bonding forces of the cladding layer remained. Besides the mechanical stress resulting from low ductility of the solidified ceramic, the main reason for gap formation (figure 3 c, d) is that the built up layers are not connected by a real ceramic bond to the base material. Additionally the molten and solidified layers have similar properties to glass, in particular a much higher density. The process speed is highly limited by the maximum usable laser power due to the plasma appearance at higher power levels and limited as well by the reduced maximum cladding rate. On account of these negative circumstances the research focus was changed to the powder-free process of laser remelting which is discussed in the following chapter 2.2.

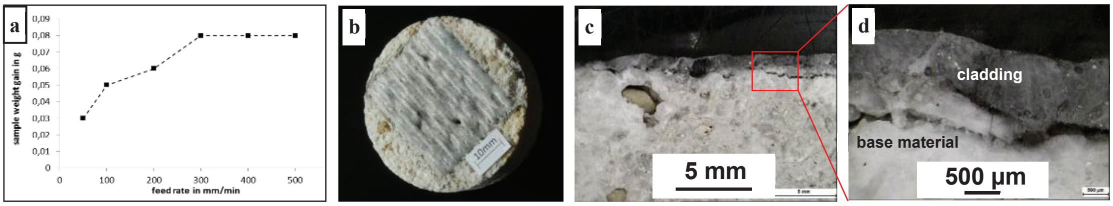

Fig. 3. (a) increasing feed rate from 48 to 480 mm/min: laser power 100 W, 0.6 g/min; (b) laser cladded substrate: 150 W, 96 mm/min, hatching distance 1,5 mm; (c,d) gap formation between base material and cladding

3.2. Laser remelting

The following diagram and associated cross sections of laser remolten specimens in figure 4 show the dependency of the melting depth on the laser feed rate. A 900 W solid state continuous wave scanning system was used. The dark grey colored melting layers can be divided in three subzones (picture number 2 in figure 4): the zone of volume shrinkage at the top (A), the glass like zone in the middle (B) and the transition zone (C) with the melting peaks which penetrate deep into the material. The total depth is plotted in the diagram in figure 4 on the left. It decreases continuously with increasing laser feed rate. The physical bonding between the molten layer and base material is significantly higher compared to the laser cladding process (see chapter 2.1). Between layer and base material, no gap formation is observable. The mechanical adherence of the layer is much stronger than the powder based claddings as there is a homogenous transition from cladding to base material. The color of the solidified layer is highly dependent on the surrounding process atmosphere. With an oxygen atmosphere it becomes bright white color, with an argon atmosphere it turns dark grey or black.

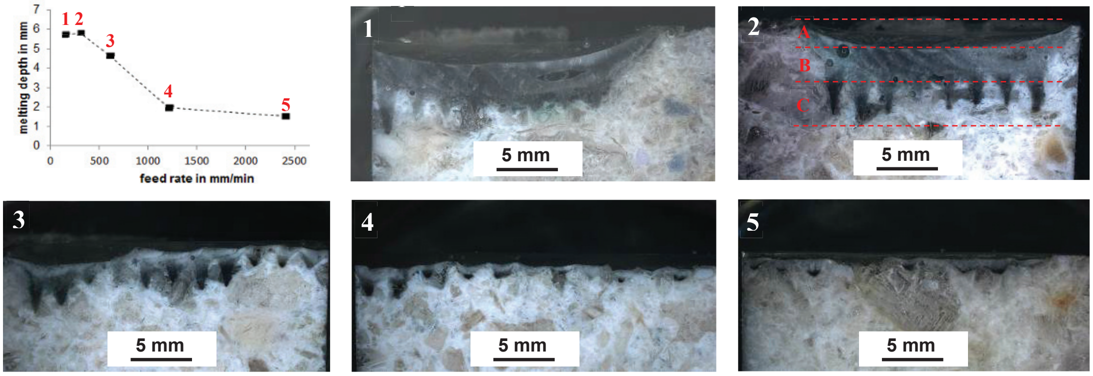

Fig. 4. cross sections of laser remolten ceramic surfaces (number 1 to 5): melting depth as function of feed rate at 900 W laser power

Figure 5 a shows the result of laser remelting investigations with a large area treatment and included preheating. The whole substrate surface was treated using a laser scanner. The chosen laser parameters were 900 W laser power and 600 mm/min feed rate. Since cracks also occurred during the cooling down phase, the surface was preheated by defocused laser radiation. A surface temperature of 600 °C measured by an infrared temperature gun during the preheating process was maximal. Referring to the investigations of Nagel [11], a preheating treatment needs significantly more than 1000 °C of temperature. The first preheating tests up to 1100 °C in an industrial furnace showed a significantly decreased tendency of crack formation within the remelted surfaces. This improved performance was confirmed in additional surface treatment test using a preheating of 1100 °C. The measured surface quality could be improved by about 60 % from R_a = 14.6 µm to R_a = 5.71 µm. Figure 5 b shows a SEM micrograph of a remelted surface. The dendritic structures as result of the crystalline solidification are remarkable. The figures 5 c and 5 d show the crystalline structures in detail. A typical crystalline, periodically repeating pattern with orthogonal angles can be seen. In figure 5 e, a SEM micrograph of transition area between a partially remelted material area and an original sintered formation is depicted. In the upper part of the micrograph the sintered particles are visible which results in a rough surface. The lower part reflects the crystallized remolten area that results in an obviously lower surface roughness.

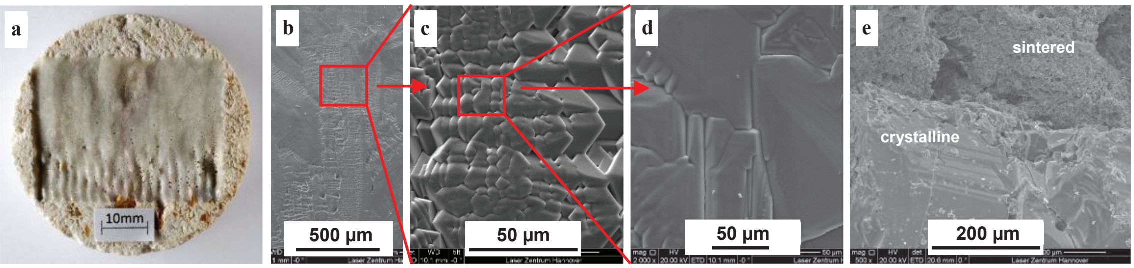

Fig. 5. (a) large area surface treated substrate; SEM micrographs: (b) remolten surface; (c) detail of figure b; (d) detail of figure c; (e) transition zone of crystalized and sintered basic material

4. Conclusion and Outlook

The previous investigations have shown the feasibility of using laser surface treatment processes for ceramic refractory materials. At an industrial scale, the requirements of speed and the economic feasibility of the process are of major importance. The initially tested laser cladding process is limited by process speed, mechanical properties of the cladding layer and as well, the resulting surface quality. Thus the research lines were modified from laser powder cladding to laser surface remelting. Using a 1000 W laser scanning system, the process time and the surface roughness could be reduced significantly. Additional experiments using a high power CO_2 scanning system permitted further increase in process speed due to the high absorption coefficient of the 10 µm wavelength for ceramic materials and the maximum laser power of 2250 W. Initial investigations on the corrosion and wetting behavior of the treated surfaces showed a reduced penetration of the molten aluminum into the material since surface defects were closed by laser treatment. Concerning the performance during corundum formation, long term experiments have to be carried out for quantification.

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