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Additive manufacturing based on laser cladding of cp-Ti for dental implants

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Abstract

Humans have attempted to replace missing teeth with root form implants for more than 4000 years using bamboo, ivory, shells or precious metals. Nowadays, a typical dental implant consists of a screw made of commercially pure titanium, cp-Ti, with a roughened surface to improve the osteointegration. Titanium and its alloys are successful metallic biomaterials for dental implants because of their biocompatibility, corrosion resistance, fatigue strength and a relatively low elastic modulus to minimize “stress shielding” and osteopenia.

Dental implants are manufactured by conventional subtractive methods, which show a set of inconveniences: titanium has low machinability; loss of high cost material during machining; and the geometrical limitation for the shapes of the dental implants achieved by subtractive methods. Nevertheless, Rapid Prototyping based on Laser Cladding (RPLC) can become a solution to manufacture advanced pure titanium dental implants tailored to the patient, with new geometries to improve the osteointegration and with enhanced microstructures/mechanical properties for a better performance.

This study is an initial analysis to produce pure titanium parts by RPLC as dental implants. RPLC is employed to generate simple 3D geometries using cp-Ti powder as precursor material. The parts generated are studied to determine the properties related to a good fixation of the implant and the osteointegration: elastic modulus is analyzed by nanoindentation; surface roughness is measured by optical interferometry; and wettability by means of contact angle technique.

Keywords: Macro Processing; Additive manufacturing; Laser Cladding; Titanium; Dental Implants.

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1. Introduction

In the biomedical field, dental implants have the function of restoring one or more teeth and they are extremely important to recover the health of the mouth and to improve the quality of patient life (Oshida et al., 2010). Dental implants are subject to highly demanding working conditions: they have to bear the pressures of chewing; must have high hardness, wear resistance and fatigue strength. They have to be biocompatible and must have a high resistance to corrosion in an environment with wide variations of acidity and temperature. Moreover, a good osseointegration is critical for implant stability and for long-term clinical success of the dental implant (Shalabi et al., 2006).

Humans have attempted to replace missing teeth with root form implants for more than 4000 years using bamboo, ivory, shells or precious metals. Nowadays, a typical dental implant consists of a screw made of commercially pure titanium, cp-Ti, with a roughened surface to improve the osteointegration (Shalabi et al., 2006, Le Guéhennec et al., 2007, Oshida et al., 2010, Rosales-Leal et al., 2010, Shibata et al., 2015a). Titanium and its alloys are successful metallic biomaterials for dental implants because of their biocompatibility, corrosion resistance, fatigue strength and a relatively low elastic modulus to minimize stress shielding and osteopenia (Shibata et al. 2015b).

Dental implants are manufactured by conventional subtractive methods, which show a set of inconveniences: titanium has low machinability; loss of high cost material during machining; and the geometrical limitation for the shapes of the dental implants achieved by subtractive methods, Mangano et al., 2014. Nevertheless, Rapid Prototyping based on Laser Cladding (RPLC) can become a solution to manufacture advanced pure titanium dental implants tailored to the patient, with new geometries to improve the osteointegration and with enhanced microstructures/mechanical properties for a better performance (Krishna Balla et al., 2007, Naveed Ahsan et al., 2011).

Rapid Prototyping based on Laser Cladding (RPLC) is an additive manufacturing process based on the overlap of layers of material, one above the others, to produce metallic or ceramic functional components. This manufacturing process can be applied to any material which can be melted and it can allow the production of tailored parts for multiple purposes. The last years, the interest in Laser Cladding has increased and is progressively considered for research in the biomaterials field: the surface treatment approach conducted to calcium phosphate and bioactive glass coatings on Ti6Al4V alloy (Lusquiños et al., 2001, 2003 and 2005, Comesaña et al., 2010). It is also possible to find works of Rapid Prototyping based on Laser Cladding for processing three-dimensional bioceramics, calcium phosphate parts and bioactive glass (Lusquiños et al., 2015, Comesaña et al., 2011a and 2011b) and for processing metallic biomaterials, like CoCrMo (España et al. 2010); titanium alloy (Ti6Al4V) (Dinda et al., 2010, Naveed Ahsan et al. 2011); or cp-Ti, (Arias-González et al., 2013 and 2014, Meacock et al. 2007, Krishna Balla et al., 2007).

The capability of producing dense 3D geometries of pure titanium by Rapid Prototyping Based on Laser Cladding, keeping all the process parameters constant, were studied in a previous work by our group (Arias-González et al., 2013 and 2014). The research work here presented is an initial analysis to produce pure titanium parts by RPLC as dental implants. RPLC is employed to generate simple 3D geometries using cp-Ti powder as precursor material. The parts generated are studied to measure some properties related to a good fixation of the implant and the osteointegration: elastic modulus (Shibata et al. 2015b) is analyzed by nanoindentation; surface roughness (Shalabi et al., 2006, Le Guéhennec et al., 2007, Rosales-Leal et al., 2010, Shibata et al., 2015a) is studied by optical interferometry; and wettability (Rupp et al. 2014, Gittens et al., 2014) is determined by the measured of the contact angle by means of the sessile drop technique.

2. Materials and methods

The laser cladding by powder flow technique was selected to produce pure titanium parts, see Fig. 1. A fiber laser is focused initially on the substrate surface by a lens of 120 mm of focal length. A power of 100 W is delivered, in continuous mode, to generate the molten pool.

The precursor material selected is cp-Ti grade 4 powder with a particle size between 75 and 180 μm (see Fig. 2), which is carried by inert gas (Argon) and blown to the molten pool at a fixed rate: 7.5 mg/s. The powder material is melted and distributed layer by layer over the partly built sample. This heated material solidifies in an inert atmosphere when the laser beam goes on sweeping a path leaving the interaction zone.

The metallic substrate moves at 5 mm/s with regard to the laser beam and particle injector, generating the first clad. When it reaches the end, the substrate is moved down one step (50 μm) and starts the movement in the opposite direction to create a new overlaid clad. This sequence of movements is performed continuously in a loop and a sample of several millimeters in height is built, layer by layer. The samples were generated by depositing 400 layers continuously with a step between layers of 50 μm.

The samples were cut, embedded in epoxy resin and polished to examine the cross-section of the built part. The microstructure and local elemental composition were studied via scanning electron microscopy (SEM, Philips XL-30) and an energy dispersive X-ray spectroscope coupled to the microscope (EDS, EDAX PV9760). The Young´s modulus was analyzed by nanoindentation (Nanoindenter NanoXP). The surface roughness was measured on the lateral of the built part using a non-contact optical profiling system (Wyko-NT1100). Static water contact angle measurements were carried out by a water contact meter (Pocket Goniometer PG2).

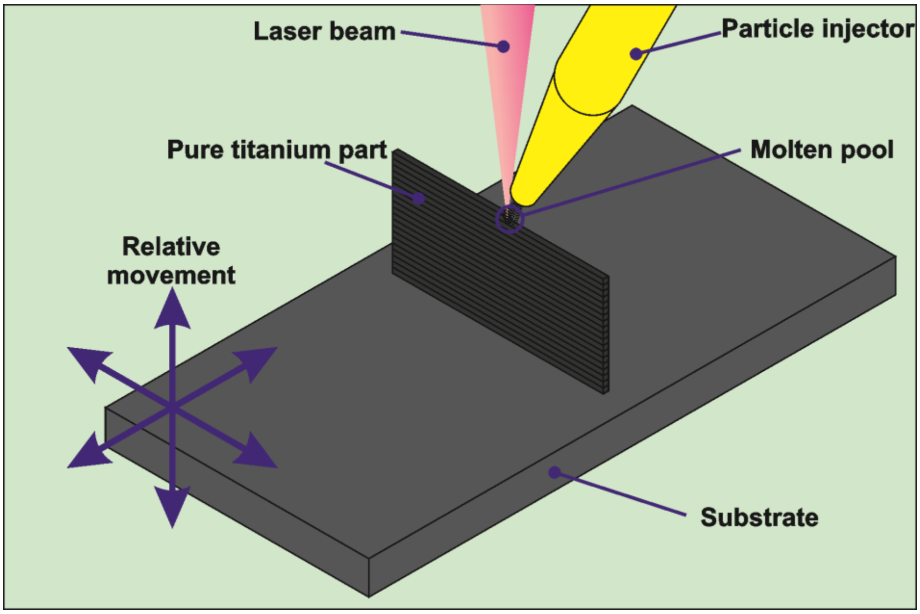


Fig. 1. Sketch of Rapid Prototyping based on Laser Cladding technique with side particle injection.

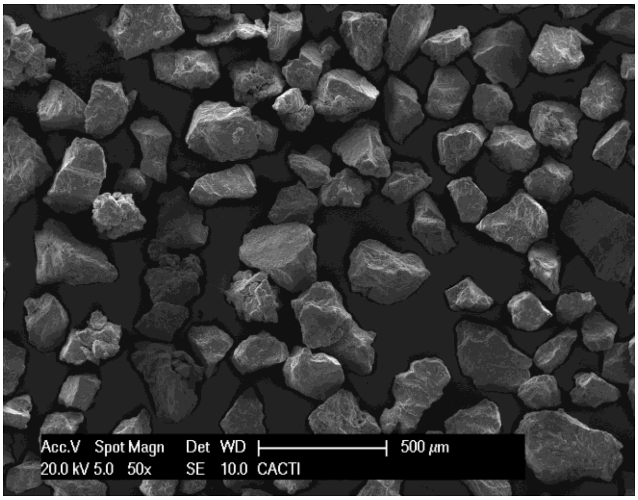


Fig. 2. Particles of cp-Ti grade 4 with a size between 75 and 180 µm.

3. Results and discussion

3.1. Microstructure and elemental composition

The samples generated are dense thin parts with transverse dimensions below 1mm, see Fig. 3. To generate a part using Rapid Prototyping Based on Laser Cladding technique, it is crucial a fine control of the layer vertical dimensions. If the vertical relative movement between the substrate and the laser system is too much, the focus point of the laser beam will separate from the surface of the part, layer by layer. The result is a laser beam unfocused on the surface of the part and a misalignment between the flow of powder and the molten pool. The vertical step between layers (50 µm) is the optimum for the selected process parameters.

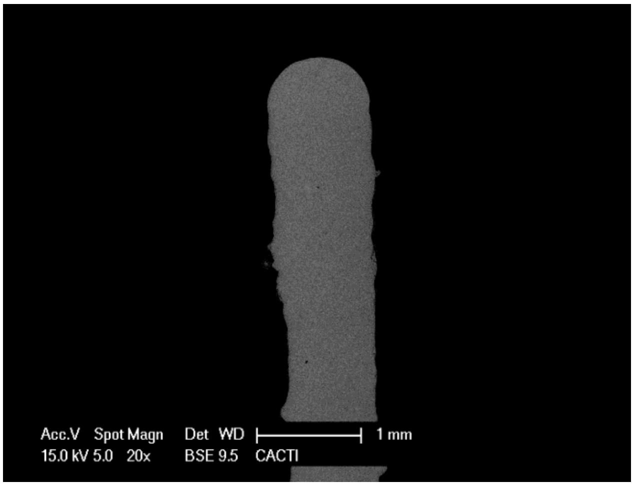


Fig. 3. SEM micrograph of the cross-section of the sample generated by RPLC.

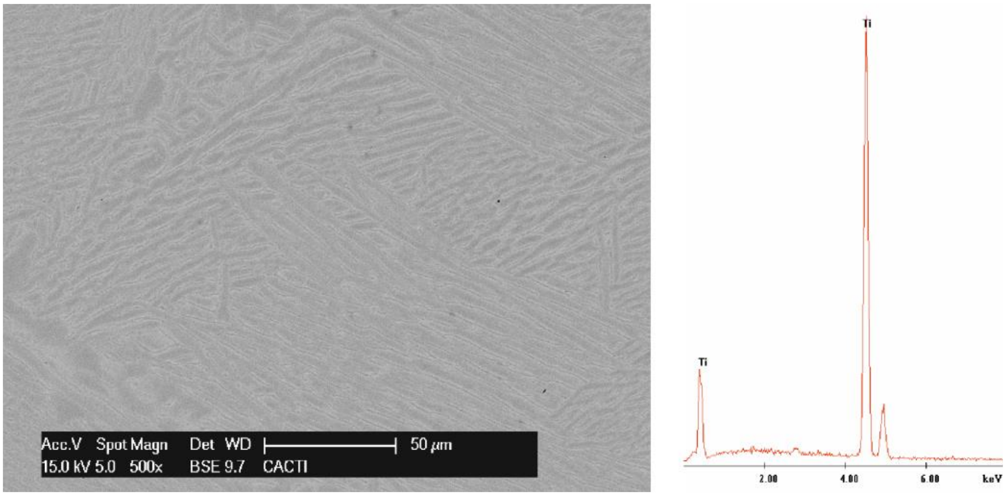


Fig. 4. Left, SEM micrograph showing the microstructure in the cross-section of the samples. Right, EDS study to determine the elemental composition of the samples.

The microstructure of the samples is fine acicular, see Fig. 4 (left), obtained as a result of the rapid cooling of titanium from a temperature above the β-transus temperature (882 ºC). The study via EDS, see Fig. 4 (right), shows the elemental composition of the part is pure titanium.

3.2. Young´s modulus

The Young´s modulus of the part was analyzed by nanoindentation, see Fig. 5. A mean value of 113 ± 6 GPa was measured. The literature reports the Young´s modulus of cp-Ti grade 4 to be about 100-120 GPa (Leyens and Manfred, 2003). The value obtained in the parts generated by RPLC is similar to the elastic modulus of cp-Ti grade 4 and it is relatively low compared to other metallic biomaterials.

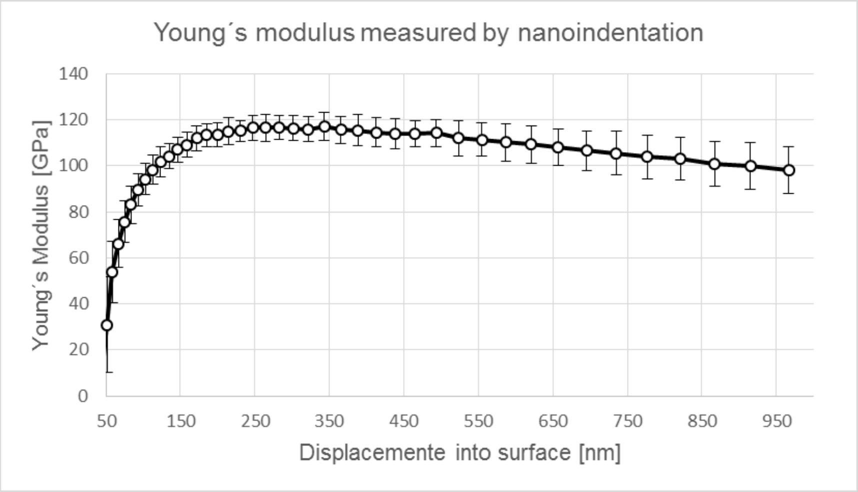


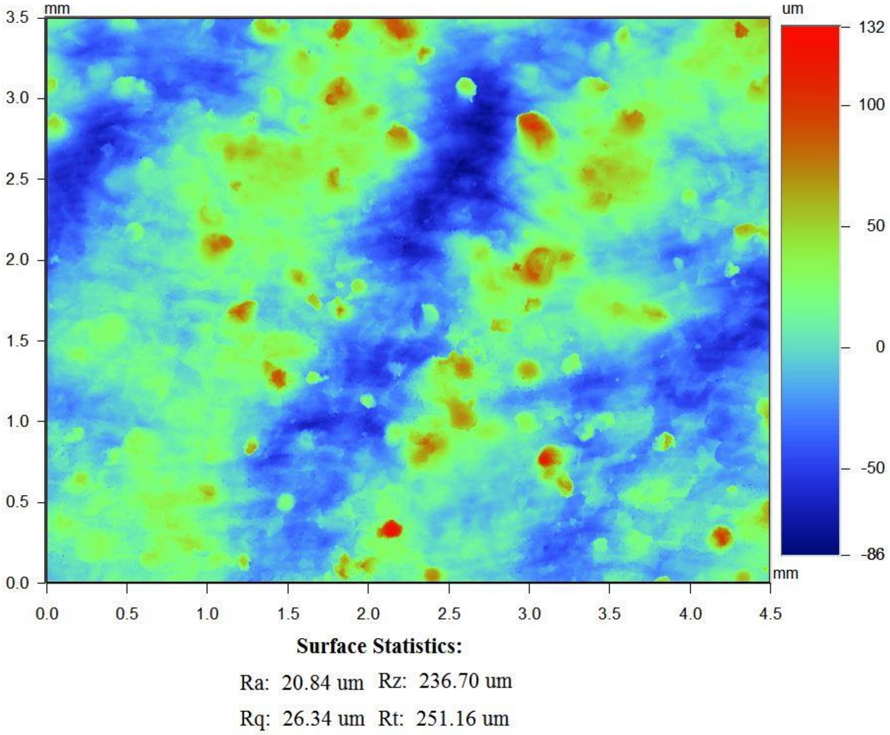
Fig. 5. Young´s modulus values at different depth obtained by nanoindentation in a polished cross-section of a part.

The Young´s modulus of the sample is still higher than the Young´s modulus of the bone, that is between 0.5 GPa and 20 GPa (Shibata et al., 2015b). However, the RPLC technique can be used to produce roughened or porous implants and increase the contact area between newly formed bone and the titanium implant. This is a solution to minimize stress shielding and osteopenia produced by the higher Young´s modulus of cp-Ti.

3.3. Surface roughness

The surface roughness was measured on the lateral of the sample, see Fig.6. The mean surface roughness (Ra) is 20.84 µm. The surface roughness observed is caused by three factors. Firstly, partially molten particles; secondly, a waviness with an oblique direction and a period in the order of millimeters, which is related with the angle of the particle injector; finally, it appears a waviness parallel to the horizontal and a period in the order of the step size, which is produced by the layer overlapping.

Different methods have been proposed to create a rough surface on dental implant: plasma-spraying, sand-blasting, acid etching, etc. (Le Guéhennec et al., 2005, Rosales-Leal et al., 2010). However, the surface roughness of the titanium sample manufactured by RPLC is higher than the obtained by the other proposed methods. A rougher implant has more available surface area, which leads to an increase in the cell adhesion (Rosales-Leal et al., 2010). It also has been reported a positive relationship between bone-to-implant contact and surface roughness (Shalabi et al., 2006).



Surface Statistics:

Ra: 20.84 um Rz: 236.70 um

Rq: 26.34 um Rt: 251.16 um

Fig. 6. Topography in the lateral of the sample measured by means of optical profilometry.

3.4. Wettability

The wettability was studied by depositing a drop of MiliQ water on the lateral of the sample and the contact angle was measured, see Fig.7. The mean contact angle determined by independent measurements is 48°±1°. It is known that surface roughness has a relevant effect on the contact angle; a greater surface area ratio increases the wettability for hydrophilic materials. The conducted analysis demonstrates that the surface is hydrophilic, which is adequate for dental implants. Hydrophilicity stimulates the integration of tissue with the implant, favors wound healing and early osseointegration (Rupp et al., 2014, Gittens et al., 2014).

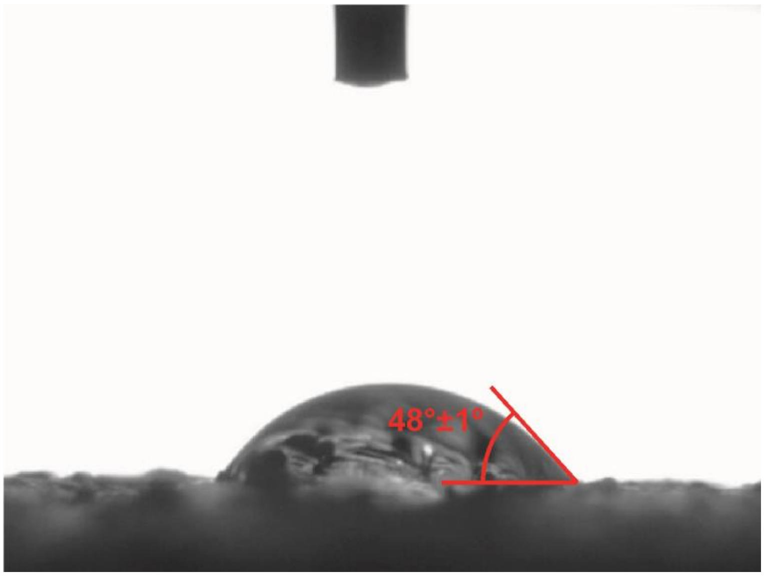


Fig. 7. Contact angle measured by sessile drop technique in the lateral of the sample.

4. Conclusions

The conclusions of this work are summarized as follows:

- The application of Rapid Prototyping based on Laser Cladding to generate dense pure titanium samples is a promising technique to create a new generation of cp-Ti dental implants.
- Young´s modulus of the dense pure titanium parts generated is similar to cp-Ti grade 4 (the most widely used material in dental implants). Despite it is higher than the Young´s modulus of the bone, RPLC is not limited to the generation of dense samples. The technique can be used to produce a new generation roughened or porous implants, increasing the contact area between newly formed bone and the titanium implant to reduce stress shielding.
- The mean surface roughness of the RPLC samples is relatively high, which is desirable to increase the available surface area and the cell adhesion. The mean surface roughness surpass the obtained by the present post-processing methods employed to produce rougher dental implants.
- The lateral surface of the samples is hydrophilic and adequate for dental implants to favor wound healing and early osseointegration.

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