

# Laser processing of bioactive tricalcium phosphate coating on titanium for load-bearing implants

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## Abstract

Laser-engineered net shaping (LENS™), a commercial rapid prototyping (RP) process, was used to coat titanium with tricalcium phosphate (TCP) ceramics to improve bone cell–materials interactions. During LENS™ coating process, the Nd:YAG laser melts the top surface of Ti substrate in which calcium phosphate powder is fed to create a TCP–Ti composite layer. It was found that an increase in laser power and/or powder feed rate increases the thickness of the coating. However, coating thickness decreased with increasing laser scan speed. TCP coating showed columnar titanium grains at the substrate side of the coating and transitioned to equiaxed titanium grains at the outside. When the scan speed was reduced from 15 to 10 mm s<sup>-1</sup>, coating hardness increased from 882 ± 67 to 1049 ± 112 Hv due to an increase in the volume fraction of TCP in the coating. Coated surfaces showed uniformly distributed TCP particles and X-ray diffraction data confirmed the absence of any undesirable phases, while maintaining a high level of crystallinity. The effect of TCP coating on cell–material interaction was examined by culturing osteoprecursor cells (OPC1) on coated surfaces. The results indicated that TCP coating had good biocompatibility where OPC1 cells attached and proliferated on the coating surface. The coating also initiated cell differentiation, ECM formation and biomineralization.

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## 1. Introduction

Bioactive calcium phosphate-based ceramics, especially hydroxyapatite (HAp) and tricalcium phosphate (TCP), have received a great deal of attention for their use as bone graft substitutes due to their chemical and crystallographic similarity to natural bone [1–7]. Both *in vitro* and *in vivo* studies have shown that these ceramics are biocompatible and osteoconductive [2–7]. TCP find its applicability in bone reconstruction and remodeling due to its bioresorbable property [2–4]. Its bioresorbable property encourages bone growth and facilitates integration with bone tissue as it resorbs. A major advantage of the use of resorbable bioceramics (TCPs) over autologous bone grafts (HAp) is

a ready supply, a controlled variation in size and the elimination of a potential second surgical procedure [2]. These ceramic materials are brittle in nature and can only be used as a coating or bone fillers. Among these applications, the coating of metallic implants improves the tissue integration of the coated implants by providing a bioactive surface [8] on otherwise bioinert material, and this can improve healing time.

The usefulness of a coated implant depends on the stability of the coating which is governed by its physical and mechanical properties. The most important point that needs to be addressed for any coated implant is the long term adherence of the coating with the substrate. A coating which separates from the implant *in vivo* would provide no advantage over an uncoated implant and less desirable due to problems with debris materials within the body. A variety of different techniques have been used to coat metallic

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implants with calcium phosphate-based ceramics, such as dip coating [9], sol–gel [10], electrophoretic deposition [11], biomimetic coating [12], simultaneous vapor deposition [13], pulsed laser deposition [14] and plasma spraying [15–17]. The success of these coating processes depends on achieving a high crystallinity in the coatings, good adherence between ceramic and metal, control over coating thickness and the ability to coat porous and complex shapes. Of these, the electrophoretic deposition process is a good method to coat porous and complex-shaped implants. However, high temperature sintering of such electrophoretically deposited coatings can often lead to cracking at the substrate–coating interface. Dip coating and sol–gel processes are good methods for producing a thin coating on implants, but achieving a thicker coating is often very difficult. Plasma spraying became the most popular coating process for commercial application in biomedical devices due to certain advantages, like simplicity, high deposition rates, low substrate temperature and economic viability [15]. Regardless of it being a successful commercial coating process, there are still drawbacks related to plasma-sprayed coatings. Plasma spray calcium phosphate-based ceramic coating suffers from low crystallinity and poor interfacial bonding [16,17]. In addition, a high cooling rate can introduce cracks in the coatings which can reduce the adhesion strength between the substrate and the coated ceramic. However, some improvement in crystallinity and interfacial strength is possible for plasma-sprayed coatings using post-deposition heat treatment [18].

Laser surface treatment is becoming popular for modifying the surface properties of metals and their alloys [19–24]. Lasers have been used to produce ceramic coating on metallic structures for biomedical applications. The simple ways of coating is by pre-placing ceramic powders on top of a substrate with the help of binder, and then use a laser to locally melt the top layer of the substrate while entrapping the powder [19–22]. However, a better approach is to directly feed ceramic powders into the pool of molten metal with the help of a carrier gas used concentrically with the laser beam [23,24]. The critical issues that need to be addressed in laser coating are controlling phase transformation of ceramics during deposition, producing a crack free metal–ceramic interface and avoiding cracks in the coating. Previous works have shown a thick bioceramic coating on top of a metal with negligible dilution between the metal substrate and the coating [23,24]. Such little dilution of ceramic into metal forms a sharp substrate–coating interface, which then becomes the weakest zone and can lead to premature failure of the coating *in vivo*. An ideal coating should have an intermediate metal–ceramic region with a compositionally gradient interface. This type of coating design has the potential to reduce interfacial problems and enhance *in vivo* lifetime.

In this study, TCP coatings were prepared on commercially pure Ti (cp-Ti) substrate using LENS<sup>TM</sup>. TCP was used as a representative material from the calcium phos-

phate family of bioceramics for which 45–150  $\mu\text{m}$  size powders, required for LENS<sup>TM</sup>, are easily obtainable. However, this process can easily be extended to calcium phosphate materials other than TCP. Laser beams, owing to their high coherence and directionality, has the ability to locally melt the surface. A 0.5 kW continuous wave neodymium–doped yttrium aluminium garnet (Nd:YAG) laser beam was used to coat TCP particles on cp-Ti. Influence of laser processing parameters on coating microstructure and properties are discussed here. The simplicity of coating process and flexibility in selecting process parameters with a high degree of accuracy for desired physical and mechanical properties are some of the advantages of LENS<sup>TM</sup>. Although LENS<sup>TM</sup> is a line-of-sight process it can create designed coatings on a flat or a curved surface. This coating technique can also be used to repair damaged coating due to its selectivity and precise treatment.

## 2. Materials and experimental procedure

A schematic representation of the LENS<sup>TM</sup> process is shown in Fig. 1. The process uses Nd:YAG laser power focused onto a metal substrate to create a molten metal pool on the substrate. Metal powder is then injected into the metal pool, which melts and solidifies. The substrate is then scanned relative to the deposition head to write a line of the metal with a finite width and thickness. Rastering of the part back and forth to create a pattern and fill material in the desired area allows a layer of material to be deposited. This procedure is then repeated many times until the entire object represented in the three-dimensional CAD model is produced on the substrate, which is a solid or tailored porosity object. The concept of LENS<sup>TM</sup> can also be used for surface treatments where ceramic powder can be used to feed into the molten metal pool to form a metal–ceramic composite.

Commercial-grade calcium phosphate powder, with mainly HAP as the primary phase, having a particle size ranging from 45 to 150  $\mu\text{m}$ , based on sieve analysis, was used to coat 0.89-mm-thick Ti substrate (Alfa Aesar) of 99.7% purity. Ti substrate was cleaned with acetone to remove organic materials from the surface prior to coating. LENS<sup>TM</sup> 750 (Optomec, Albuquerque, NM, USA) unit with 0.5 kW continuous wave Nd:YAG laser was used to coat Ti substrate. During laser fabrication, the top surface of Ti metal substrate is melted and TCP powder is fed to the molten metal region with the help of a carrier gas (Ar). The molten Ti and TCP solidify rapidly as the laser head moves on. This is a controlled environment laser cladding of TCP and Ti. This process was carried using a commercial LENS<sup>TM</sup> system. Powdered material is melted using laser in order to coat a substrate. To reduce the oxidation of Ti, the coatings were fabricated in a controlled atmosphere with total O<sub>2</sub> content less than 10 ppm in the chamber. The laser power, scan speed and powder feed rate were varied for these coatings. LENS<sup>TM</sup> parameters used in the present work are shown in Table 1. Ti substrate was coated

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