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microstructure and tribological properties

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#### ABSTRACT

Fibre laser nitriding of titanium and its alloy in open atmosphere for

orthopaedic implant applications: Investigations on surface quality,

Laser nitriding is known to be an effective method to improve the surface hardness and wear resistance of titanium and its alloys. However, the process requires a gas chamber and this greatly limits the practicability for treating orthopaedic implants which involve complex-shaped parts or curved surfaces, such as the tapered surface in a femoral stem or the ball-shaped surface in a femoral head. To tackle this problem, a direct laser nitriding process in open atmosphere was performed on commercially pure titanium (grade 2, TiG2) and Ti6Al4V alloy (grade 5, TiG5) using a continuous-wave (CW) fibre laser. The effects of varying process parameters, for instance laser power and nitrogen pressure on the surface quality, namely discolouration were quantified using ImageJ analysis. The optimised process parameters to produce the gold-coloured nitride surfaces were also identified: 40 W (laser power), 25 mm/s (scanning speed), 1.5 mm (standoff distance) and 5 bar (N<sub>2</sub> pressure). Particularly, N<sub>2</sub> pressure at 5 bar was found to be the threshold above which significant discolouration will occur. The surface morphology, composition, microstructure, micro-hardness, and tribological properties, particularly hydrodynamic size distribution of wear debris, were carefully characterized and compared. The experimental results showed that TiG2 and TiG5 reacted differently with the laser radiation at 1.06 µm wavelength in laser nitriding as evidenced by substantial differences in the microstructure, and surface colour and morphology. Furthermore, both friction and wear properties were strongly affected by the hardness and microstructure of titanium samples and direct laser nitriding led to substantial improvements in their wear resistant properties. Between the two types of titanium samples, bare TiG2 showed higher friction forces and wear rates, but this trend was reversed after laser nitriding treatments.

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#### 1. Introduction

Titanium and its alloys have become the workhorse of orthopaedic applications in the past few decades because of their desirable material properties, such as excellent corrosion resistance and biocompatibility, high strength to weight ratio as well as high toughness. The major problems limiting the performance of titanium-based orthopaedic implants are their poor resistance to wear and the generation of debris when the implants are damaged or fractured in service. It is known that implant debris can cause inflammation and osteolysis [1,2]. Surface modification of titanium by laser nitriding is an efficient method to improve the surface hardness and wear properties [3–8]. The improvement of surface properties by laser nitriding comes from the formation of a titanium nitride (TiN) layer. Laser-formed TiN layers offer competitive advantages over the TiN layers created by other conventional methods, such as PVD, CVD, ion implantation, etc. The key advantages are high layer thickness (>50  $\mu$ m) and no issue of delamination, i.e. layers are metallurgically bonded to the substrate. On top of this, a laser is a highly flexible and accurate tool which can perform nitriding on selected areas without causing undesired heating of the substrate.

Briefly, the laser nitriding process starts by scanning the laser beam across the substrate surface in a nitrogen-filled chamber. When the substrate surface is heated up by the laser beam above its melting point, the laser-irradiated area will melt and a plasma forms above the surface. The high temperature and pressure created above the surface, resulting from the laser-plasma-material interactions, cause ionization and dissociation of nitrogen [9]. The ionized and dissociated nitrogen, namely nitrogen ions and atoms, will be absorbed by the melted surface and the TiN layer will form after solidification [10–12]. The physical reactions involved in the laser nitriding process are detailed by Höche and Schaaf [13]. The sequence of how nitrogen is activated by laser energy and

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Fig. 1. Schematic diagram of the laser nitriding setup (left) and the scanning movement of the laser beam (right). High purity N<sub>2</sub> is delivered coaxially via the laser nozzle. High pressure N<sub>2</sub> zone is created between the substrate surface and nozzle tip to prevent O<sub>2</sub> intruding from surrounding air.

absorbed by the titanium substrate to form TiN is provided by Kloosterman and DeHossen [14].

Despite laser nitriding possessing several attractive characteristics, the process requires a gas chamber and this greatly limits the practicability. For example, it is difficult to create a homogenous nitride layer on complex-shaped parts or curved surfaces, such as the tapered surface of a femoral stem or the ball-shaped surface of a femoral head. In other words, translation of the results from literature (i.e. laser nitriding in nitrogen-filled chamber) to industrial applications is unrealistic given that the nitride properties are not only controlled by the process parameters but also by the design of the gas chamber which determines the gas dynamic factors. To overcome this drawback, direct laser nitriding in open atmosphere (or without gas chamber) has been proposed, i.e. nitrogen is directly delivered to the laser-irradiated area via a coaxial nozzle in the laser head.

A brief summary of the literature on direct laser nitriding in open atmosphere is provided below. Chen et al. [15] used a specially developed nozzle to perform direct nitriding on Ti6Al4V using pulsed Nd:YAG laser, and investigated how the gas dynamic factors affect the quality of the nitride layer. Yu et al. [16] developed a hybrid laser-plasma nitriding method to obtain an oxide-free TiN layer on pure titanium. They performed a direct nitriding process using CW CO<sub>2</sub> laser coupled with a plasma gun. Nasser et al. [17] investigated the effect of laser-induced plasma in direct nitriding using CW CO<sub>2</sub> laser. They identified a process window for the formation of a near-stoichiometric, oxide-free TiN layer. Kamat et al. [18] produced the TiN layers on pure titanium by direct nitriding using CW CO<sub>2</sub> laser. Two different gas conditions, namely pure nitrogen and mixed nitrogen-argon were used in their nitriding experiments, and the effect of process parameters on the microstructure of the nitride layers were studied. May et al. [19] employed a custom coaxial nozzle for direct nitriding using pulsed fibre laser. They reported that the topography and wetting behaviour of the nitride layer can be modified by varying laser repetition rates.

Existing results on the direct laser nitriding process provide insights on the microstructure and some surface characteristics namely topography and wettability of the nitride layers. The success of the direct nitriding process relies heavily on the prevention of surface oxidation during the nitriding process, i.e. preventing the titanium substrate reacting



(a) Optical micrograph of TiG2 nitrided surface before ImageJ analysis

(b) Optical micrograph of TiG2 nitrided surface after ImageJ analysis

**Fig. 2.** (a–b) Example of ImageJ analysis showing the measurement of discoloured areas (i.e. blue-coloured area) in the TiG2 nitrided surface. The discoloured areas predominantly appeared in the crests of the nitride tracks. The shaded area in (b) corresponds to the blue-coloured area in (a). The laser processing parameters: laser power, scanning speed, stand-off distance and N<sub>2</sub> pressure were 40 W, 25 mm/s, 1.5 mm and 6 bar respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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