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Innovative surface modification of Ti6Al4V alloy by electron beam technique for biomedical application

Claudia Ramskogler [a,*], Fernando Warchomicka [a], Sepideh Mostofi [b], Annelie Weinberg [b], Christof Sommitsch [a]

[a] Institute of Materials Science, Joining and Forming, Graz University of Technology, Kopernikusgasse 24, 8010 Graz, Austria
[b] Department of Orthopaedics, Medical University Graz, Auenbruggerplatz 5, 8036 Graz, Austria

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ABSTRACT

The low elastic modulus, high corrosion resistance and excellent biological response allow titanium alloys to be used for permanent orthopaedic devices. Furthermore, the design of specific multi scale surface topographies on titanium alloys can provide a fast osseointegration. This work highlights the use of electron beam as a promising technique to produce a designed surface topography and improve the tribological behaviour of Ti6Al4V alloy. The produced surface topography due to the transport of molten material is influenced by the deflection figure, the physical properties of the material and the energy input. The analysis of the surface roughness shows an increment of the area up to 26% and a canal shape in a range from 1.3 μm up to 9 μm depth and from 68.6 μm up to 119.7 μm width. The high solidification rate reached during the process affects the microstructure, provoking the formation of martensite and thus the improvement of hardness. In vitro studies with pre-osteoblastic MC3T3-E1 cells performed for several cultivation times show the cells with a polygonal shape and built connections through elongated filopodia. A notable increase of cell spreading area on surface structure with a finer canal shape is found after 48 h cultivation time.

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1. Introduction

The continuous increment in the life expectancy and the technological improvement to fix complex fractures drive the need for enhanced orthopaedic devices with a prolonged lifetime, excellent mechanical and surface properties. Metals with biocompatible conditions are suitable mainly for permanent orthopaedic devices such as hip replacement, dental implants etc. and for internal plate system for fracture fixation.

Titanium and its alloys are widely used as permanent implant materials not only due to their good biological response but also their lower elastic modulus and excellent corrosion resistance compared to stainless steel and Co—Cr based alloys [1,2]. However, their area of application is somewhat limited by their low wear resistance and high friction coefficient in comparison with other metals [3,4,5]. To overcome this problem, several surface modification techniques have been proposed, such as shot peening [6], electric discharge machining (EDM) [7], thermal oxidation [8], among others. However, nucleation of voids and propagation of cracks in the subsurface region as well as additional treatment necessary to remove weakly attached particles limit the application of such techniques. Furthermore, prolonged implant lifetime can be achieved by cementless fixation technique. This technique allows the direct contact between the metallic implant and the receiving living tissue and permits better osseointegration. The use of EDM with additional chemical milling on Ti6Al4V improves the proliferation and metabolic activity of osteoblastic cells [7], while, chemical milling of pure titanium with HNO_3/NF roughs the surface to $R_{max} = 19.5$ μm and shows after an incubation time of 14 days a 1.5 times higher cell growth rate compared to standard samples [9]. Further, laser scanned topographies of a beta titanium alloy with micro grooves of 50 μm in width permit a better cell proliferation and the best cell differentiation compared to sandblasted, machined tooled and polished surfaces [10].

Another challenge in the field of biomaterials design is the biomechanical interlocking capacity [11,12], where a rigid fixation between implants and bony bed is required. Hansson et al. [13] conclude to an increase of interfacial shear strength between bone and implant by rough surfaces with an optimized micro geometry. Furthermore, previous in vitro studies on E-glass fibre-reinforce polymethyl methacrylate (PMMA)-based composite (FRC) show a higher incorporation of artificial implant with surrounding bone in surface modified specimens than in control specimen without modification [14].

A promising technique of controlled surface modification is the use of a highly energetic electron beam (EB). The principle of beam heat generation is similar to a conventional scanning electron microscope. The impact of high-speed focused electron beam onto the material causes heating, melting, local evaporation and formation of the so called “keyhole”: a hole with a high vapour pressure in the middle that presses

* Corresponding author.
E-mail address: claudia.ramskogler@tugraz.at (C. Ramskogler).

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the molten metal against the side walls. By moving the electron beam over the surface of the workpiece, a material transport occurs in the opposite direction of welding direction: the molten material is moved behind the beam and solidifies at the backside, resulting in a protrusion at the beginning and an intrusion at the end of the weldment [15]. The surface structuring is based on the effect of creating protrusion patterns by repeating the beam movement on the same path. Depending on the repetition numbers, the protrusion height and intrusion depth can be raised. Moreover, the nearly mass less electron beam can be deflected very fast and very precisely by an electromagnetic field. The deflection of the EB can be programmed in a specific way to generate corresponding figure arrays on metallic materials under vacuum. This process principle was firstly developed by Dance et al. [16] as *Surfi-Sculpt®* technology and it was applied successfully for pinning aluminium sheet for automotive industry [17], among other applications.

The aim of this work is to create a defined surface topography on Ti6Al4V for biomedical application by using the promising EB technology. EB structuring is carried out by varying machine and deflection parameters to show the potential of this technique to create multi scale topography. The influence of EB process on the changes in the mechanical properties as well as on the surface topography is evaluated. Furthermore, the behaviour of pre-osteoblastic cells on different surface structures is determined by in vitro cultivation.

2. Materials and methods

The material used for this study was Ti6Al4V with a chemical composition of 6.51–6.57 Al, 4.1–4.32 V, 0.18–0.22 Fe, 0.18–0.19 O, 0.032–0.033C, 0.022–0.024 N, 0.0006–0.0007H, Y < 0.001 and bal. Ti, in wt% [18]. The material was forged in different steps and afterwards, annealed at 730 °C during 1 h and cooled in air. Sample plates of $15 \times 15 \times 2$ mm^3 were machined and the surface was mechanically polished with silicon carbide paper up to P4000 to induce the same surface and material condition.

Surface structuring experiments were performed using an electron beam welding machine (EBW) model Probeam EBG 45-150 K14. The chamber of the EBW was under vacuum during all experiments. The design of EB surface structuring is influenced by machine and deflection parameters. Each figure has to be described with coordinate points and arranged in a desired way. An explanation of deflection figure design is shown in Fig. 1. Furthermore, the beam travel direction changes the orientation of the figure: the travel direction to the centre produces an inward structure (wall) and out of the centre an outward structure (pin). For the preliminary phase the machine parameters were changed with respect to the welding process. Schulz [15] describes a higher beam stability condition and smaller beam diameter with an acceleration voltage of 150 kV. For this reason, the maximum voltage of the machine used in this work was 150 kV. Furthermore, the beam focus was kept constant at the surface of the workpiece, while the current, velocity and beam travel direction were changed. In the first step, a series of bead on plate were produced to define minimal parameters combination for structuring. Due to the fixed voltage, the minimum energy input (generally known as $E = \frac{U^*}{I}v$, where U represents the acceleration voltage, I the beam current and v denotes the deflection velocity) can only be modified by the beam current and velocity of the deflection. The minimal parameter for the beam current was set at 0.8 mA, due to the beam focus at the surface. The velocity is calculated as $= \frac{L}{s}$, where L is the total length of the deflection figure and s the total structuring time for each deflection figure. In the second step, multi scale topography surface with micro and macro roughness of defined figures were performed to show the variety of this technique.

Wennerberg et al. [19] analysed several works about the effect of titanium surface topography on bone integration and concluded that the comparison of roughness values from different investigations was very difficult due to the changing in the surface evaluation. For this reason, in this work, the surface was analysed by using Alicona Infinite Focus microscope. A special focus variation technique allowed to create a 3D profile of the studied surface. The EB structured Ti6Al4V samples were recorded with 10× and 50× objective lenses, with a maximal vertical resolution of 100 nm and 50 nm, respectively. The lateral resolution was 3 μm for lens of 10× and 2 μm for 50×. Fig. 2 illustrates examples of acquired surface profiles, the determined width Δw and depth/height Δz measurements and the position of measurements (position 1 at 200 μm and position 2 at 600 μm from the border) for designed figures.

Post structuring heat treatments were performed under vacuum with a heating rate of 300 K/min, up to 650 °C and 720 °C, then isothermally held for 8 h, followed by quenching in argon atmosphere to improve the mechanical behaviour of the melted zone.

Vickers micro-hardness measurement was carried out using a MHT4 (Anton Paar) with an indentation load of 0.03 kg and a dwell time of 15 s

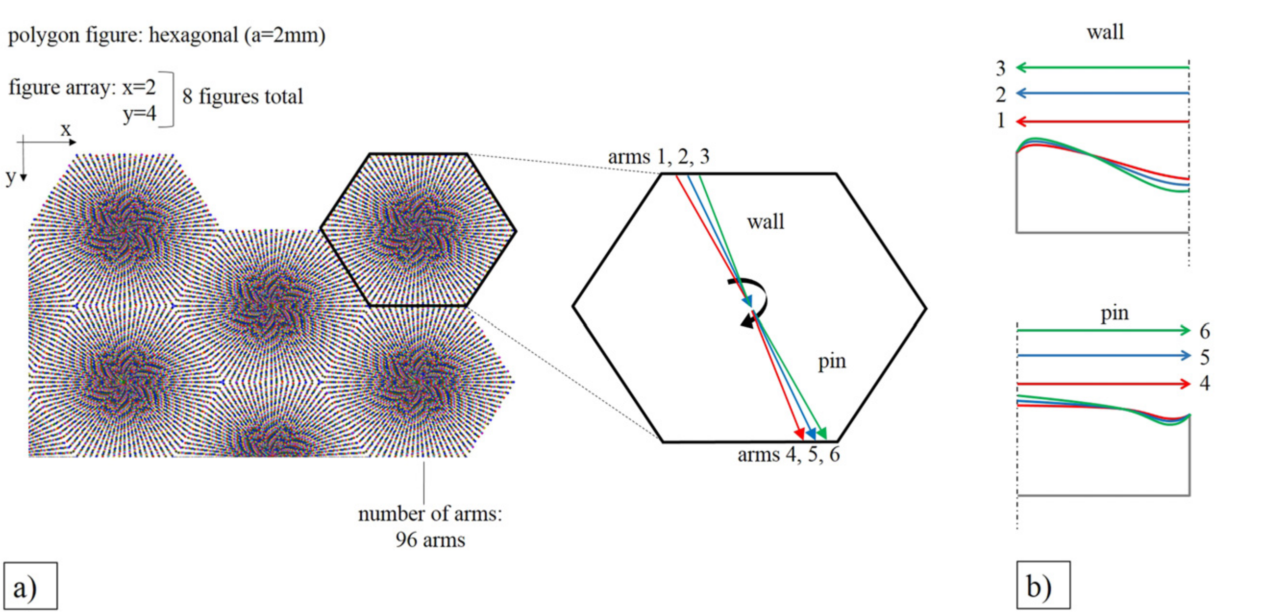

Fig. 1. a) Example of a designed deflection figure; b) the effect of the beam travel direction on the final shape.

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