Lasers in Manufacturing Conference 2023

Laser Directed Energy Deposition of biocompatible beta type Ti alloys: Development of an intense crystallographic texture to achieve a very low elastic modulus

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Abstract

The elastic modulus of the cortical bone is below 30 GPa, whereas biomedical titanium implants exhibit an elastic modulus above 100 GPa. This mismatch in the elastic modulus can lead to bone resorption caused by the stress-shielding effect and poor osseointegration of the implant. This study aimed to determine whether the intense <100> fiber texture developed in Laser Directed Energy Deposition (also known as Laser Metal Deposition) of beta-type Ti alloy ingots, results in a significant reduction in the elastic modulus. We demonstrated that laser-deposited beta-type Ti-42Nb (wt%) alloy ingots exhibit anisotropic mechanical properties. A low elastic modulus (below 50 GPa) and a high yield strength (above 700 MPa) were obtained in the building direction because of the intense <100> fiber texture. The novel laser-deposited Ti-42Nb alloy also shows excellent biological performance in vitro, which suggests its suitability for biomedical applications.

Keywords: Laser Directed Energy Deposition; titanium alloys; microstructure; Young's modulus; cytocompatibility

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1. Introduction

Titanium and its alloys are highly regarded as the most suitable metallic materials for biomedical applications due to their exceptional comprehensive properties (Niinomi et al., 2012). Commercially pure titanium (cp-Ti) and Ti-6Al-4V have established themselves as the predominant choices in orthopedic and dental applications, thanks to their impressive attributes including toughness, excellent biocompatibility, strong corrosion resistance, and a relatively low elastic modulus compared to other metallic materials (Szczęsny et al., 2022). However, cp-Ti cannot meet the requirements for high-strength applications, while Ti-6Al-4V can release cytotoxic ions, such as Al and V (Li et al., 2014; Biesiekierski et al., 2012). Moreover, the higher elastic modulus of these materials compared to cortical bone can lead to stress shielding, hindering bone regeneration and potentially causing complications like bone fractures and implant loosening (Li et al., 2014; Brizuela et al., 2019).

Ongoing research focuses on developing new biomedical titanium alloys, particularly β-type titanium alloys, using elements with low toxicity like Nb, Ta, Zr, Sn, and Hf, which offer a favorable combination of low elastic modulus, high strength, corrosion resistance, and biocompatibility (Biesiekierski et al., 2012; Kim et al., 2020). Binary Ti-Nb alloys have gained attention as implant materials due to their non-toxicity, biocompatibility, and corrosion resistance (Godley et al., 2006; Bai et al., 2016). A critical concentration of 36 wt% Nb is needed to retain the β-phase at room temperature, and the elastic modulus reaches a minimum (~62 GPa) at 40-45 wt% Nb (Hanada et al., 2005; Ozaki et al., 2004). Although lower than cp-Ti and Ti-6Al-4V alloy, the elastic modulus remains higher than cortical bone, emphasizing the desire for further reduction.

A promising approach to reduce the elastic modulus of biocompatible β-type Ti alloy implants involves controlling the crystallographic texture to influence the orientation-dependent elastic modulus (Hermann et al., 2012). It has been shown that β-type Ti alloys exhibit anisotropic elastic modulus, with the highest value along the <111> orientation and the lowest value along the <100> orientation. By manipulating the crystallographic texture, the elastic modulus of β-type Ti alloys can be effectively lowered. However, the challenge lies in developing new manufacturing processes to achieve controlled crystallographic texture in biomedical implants. Recent research has demonstrated successful texture control in β-type Ti alloys using selective laser melting (Ishimoto et al., 2017) and cold groove rolling techniques (Shinohara et al., 2018), resulting in low elastic modulus values that potentially can minimize stress shielding and improve implant performance.

In this study, Laser Directed Energy Deposition (LDED) was used to develop a <100> fiber texture in β-type Ti-42Nb alloy ingots, aiming to achieve anisotropic mechanical properties and a very low elastic modulus. LDED is an additive manufacturing process that utilizes a laser beam to selectively deposit melted material onto a surface (Saboori et al. 2017; Arias-González et al. 2018; Barro et al. 2020; Barro et al. 2021; Arias-González et al., 2021a; Arias-González et al. 2021b; Arias-González et al., 2022). This process has been successfully applied to produce various titanium and titanium alloy components. In previous studies, LDED-generated β-type Ti alloys exhibited a retained metastable β-phase and a distinctive elongated columnar β grain structure with a prominent <100> fiber texture (Banerjee et al., 2006). Building upon this background, we aimed to manufacture biocompatible β-type Ti-42Nb alloy ingots with a strong <100> fiber texture using LDED and evaluate their crystallographic texture, mechanical properties, and cytocompatibility. This study provides insights into the elastic modulus of laser-deposited biocompatible β-type Ti alloy ingots and their potential applications in orthopedic and dental fields.

2. Materials and methods

The Laser Directed Energy Deposition (LDED) technique was used to create Ti-42Nb specimens on flat cp-Ti grade 2 substrates. The LDED set-up included a Rofin-Dilas High Power Diode Laser (HPDL) with a wavelength of 940 nm and a maximum power of 1600 W, a pneumatic powder feeder, a coaxial injection powder system, and a CNC controlled three-axis positioning system. The processing zone was enclosed in an inert chamber using argon gas with oxygen levels below 50 ppm. A lens focused the laser beam onto the substrate, creating a circular spot with a diameter of around 3 mm. Process conditions were a laser irradiance of 14 x10^3 W/cm^2, processing speed of 6 mm/s, and Ti-42Nb powder flow rate of 2.2 g/min. The scanning path followed

a back-and-forth pattern with a 90º rotation between layers. Spherical pre-alloyed Ti-42Nb powder with particle sizes of 63-105 µm was used as the precursor material.

The Ti-42Nb samples were prepared for examination by cutting them, embedding them in resin, and polishing them. The crystallographic texture of the samples was then analyzed using Electron Backscatter Diffraction (EBSD) with the help of ATEX software (Beausir and Fundenberger, 2017). To evaluate their mechanical properties, uniaxial tensile tests were conducted following the ISO 6892 1:2020 (ISO, 2019) standard, using a Zwick Z100 universal machine. The tests measured the values of elastic modulus (E) and yield strength (Y).

Human osteoblast-like cells SaOS-2 were used to assess the in vitro cytocompatibility of the laser-deposited Ti-42Nb alloy compared to the cp-Ti grade 2 control. A cellular density of 20 000 cells/well was cultured on both types of samples in Falcon 12-well plates. Adhesion studies were conducted for 4 hours, while proliferation studies were performed for 3, 7, and 14 days. The cells were examined using a field emission scanning electron microscope (FESEM). For quantitative analysis, ten random images were taken at 300x magnification from the surface of four samples.

3. Results and discussion

Through the precise adjustment of process parameters, we achieved successful fabrication of solid β-type Ti-42Nb alloy ingots using Laser Direct Energy Deposition (LDED) technique. To examine the crystallographic texture of the laser-deposited Ti-42Nb, we conducted an analysis using Electron Backscatter Diffraction (EBSD). Our findings revealed a notable <100> fiber texture in the laser-deposited Ti-42Nb samples (Fig. 1), with a considerable number of β-phase grains aligning one of their <100> axes nearly parallel to the building direction (Z-axis).

The intense <100> fiber texture present in these laser-deposited ingots gives rise to anisotropic mechanical properties. In Fig. 2, we provide a comparison of the elastic modulus (E) and yield strength (Y) of the laser-deposited Ti-42Nb alloy (X-axis and Z-axis) with selected biomedical titanium alloys: cp-Ti grade 2, cp-Ti grade 4, Ti-6Al-4V and Ti-6Al-4V ELI (Niinomi, 1998). In the scanning direction (X-axis), the laser-deposited Ti-42Nb exhibits a relatively low modulus of elasticity (E_x = 59.4 ± 3.0 GPa), and reasonably high yield strength (Y_x = 735 ± 22 MPa). On the other hand, the elastic modulus of the laser-deposited Ti-42Nb in the building direction (Z-axis) is even lower (E_z = 47.9 ± 3.9 GPa), while maintaining comparable yield strength (Y_z = 715 ± 41 MPa). Notably, the elastic modulus along the Z-axis is very low compared to the commercially available biomedical titanium alloys (cp-Ti grade 2, cp-Ti grade 4, Ti-6Al-4V and Ti-6Al-4V ELI).

To evaluate the in vitro cytocompatibility of the laser-deposited Ti-42Nb alloy compared to cp-Ti grade 2 control, human osteoblast-like cells SaOS-2 were employed (Fig. 3). The cell count analysis revealed significant differences in cell adhesion after 4 hours of incubation (Fig. 4), with the Ti-42Nb substrate exhibiting more favorable results. Over a period of 14 days, cell proliferation was observed on both substrates; however, the laser-deposited Ti-42Nb surface promoted a higher degree of proliferation. Overall, the laser-deposited Ti-42Nb alloy demonstrated enhanced osteoinductive effects by facilitating the adhesion and proliferation of SaOS-2 cells.

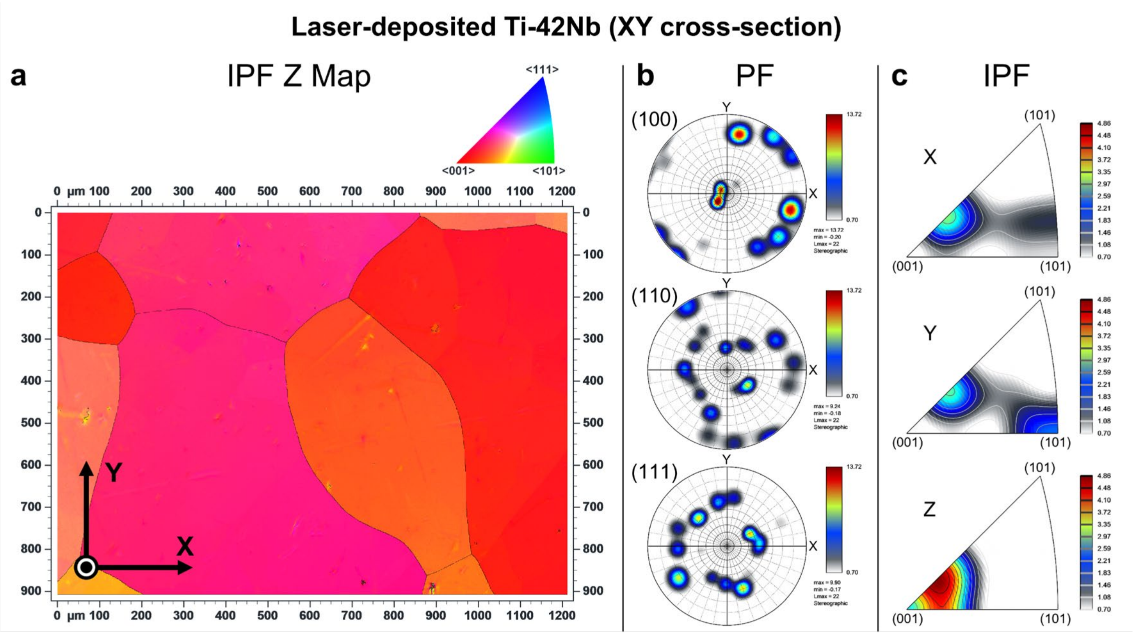

Fig. 1. Electron backscatter diffraction (EBSD) results at XY cross-section (normal to Z-axis): (a) inverse pole figure Z map (IPF Z Map); (b) pole figures (PF); (c) inverse pole figures (IPF).

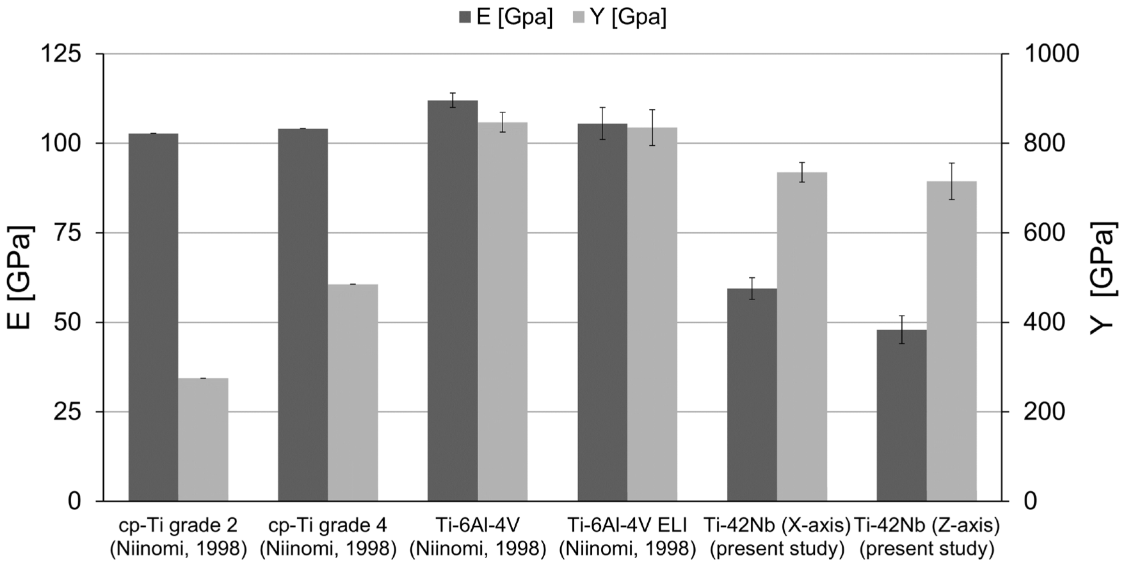

Fig. 2. Mechanical properties of selected biomedical titanium alloys determined in tensile tests: elastic modulus (E), yield strength (Y).

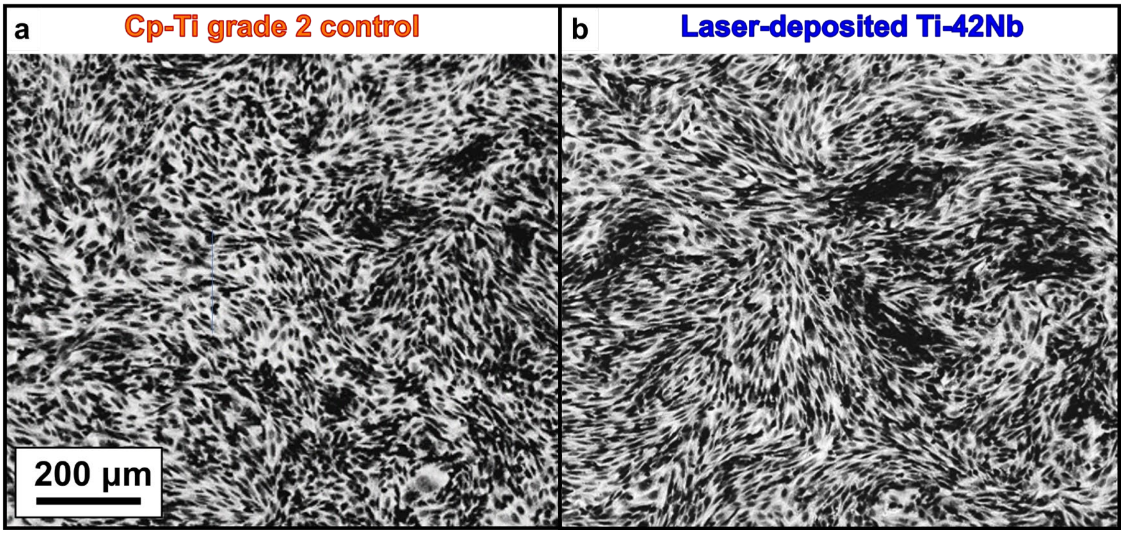

Fig. 3. Micrographs showing cell adhesion and proliferation after 14 days: (a) cp-Ti grade 2; (b) laser-deposited Ti-42Nb.

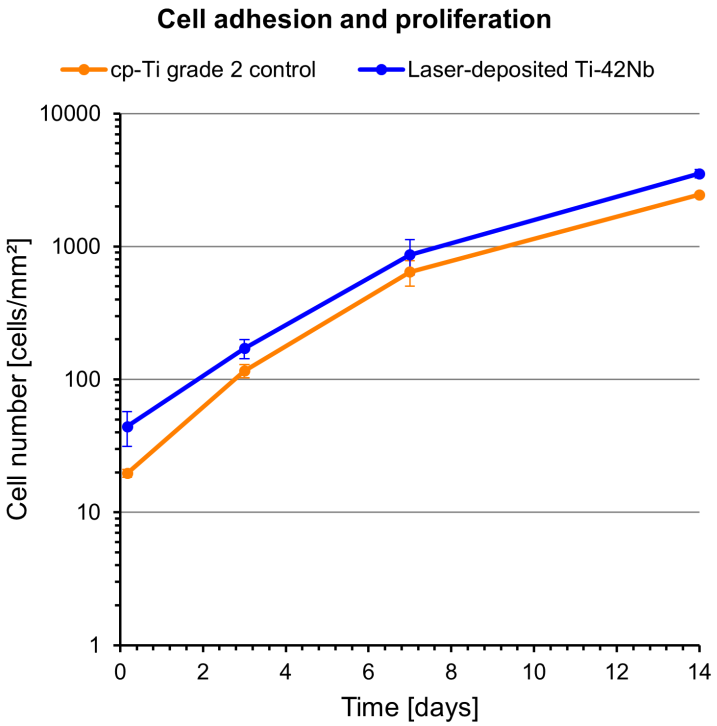

Fig. 4. Cell adhesion (4h) and proliferation (3, 7 and 14 days) on cp-Ti grade 2 and laser-deposited Ti-42Nb samples. Error bars represent standard deviation.

The findings from this study suggest that the laser-deposited β-type Ti-42Nb alloy holds significant potential for the development of innovative biomedical implants characterized by a remarkably low elastic modulus, which can help mitigate stress shielding effects. The achievement of such a low elastic modulus in the building direction (Z-axis) is attributed to the pronounced <100> fiber texture, wherein a substantial number of β-phase grains align one of their <100> axes parallel to the Z-axis. Previous studies by Tane et al., 2008 and Hermann et al., 2012 had already proposed that biocompatible β-type titanium alloy polycrystals featuring a texture aligning the crystallographic <100> directions or single crystals with the {100} direction oriented along the loading direction in human bones show promise as biomedical implant materials with an elastic modulus comparable to that of natural bones. However, a major challenge lies in establishing new processes for producing large single-crystals or textured polycrystal ingots and subsequently precisely machining the implants. This paper successfully demonstrates the feasibility of Laser Directed Energy Deposition for generating a β-type Ti-42Nb alloy textured polycrystal ingot with an exceptionally low modulus, opening doors for the development of novel biomedical implants.

4. Conclusions

Biocompatible β-type Ti-42Nb alloy ingots were successfully produced using Laser Directed Energy Deposition (LDED) technique. The laser-deposited Ti-42Nb samples exhibited a notable <100> fiber texture, with a significant number of β-phase grains aligning one of their <100> axes parallel to the building direction (Z-axis). This intense texture resulted in the specimens displaying anisotropic mechanical properties. Notably, the laser-deposited Ti-42Nb demonstrated a remarkably low elastic modulus in the building direction (E_z = 47.9 ± 3.9 GPa) and high yield strength (Yz = 715 ± 41 MPa). Furthermore, the laser-deposited Ti-42Nb alloy exhibited enhanced osteoinductive effects promoting the adhesion and proliferation of Saos-2 cells. These findings suggest that the laser-deposited β-type Ti-42Nb alloy is a promising candidate for the development of novel biomedical implants with a very low elastic modulus, capable of mitigating stress shielding effects.

Acknowledgements

The authors wish to thank the technical staff from Barcelona Research Center in Multiscale Science and Engineering (Universitat Politècnica de Catalunya) and Light Alloys and Surface Treatments Design Centre (Universitat Politècnica de Catalunya) for their technical assistance with the characterization techniques. Technical assistance of Mr. A. Abalde (Universidade de Vigo) is also acknowledged. This research was partially funded by SOADCO S.L. (Escaldes Engordany, Andorra), by the Spanish Government (RTI2018-098075-B-C21), by Xunta de Galicia (ED431C 2019/2; ED481B-2021-113), by Generalitat de Catalunya (2017 SGR-1165) and by the EU through the European Regional Development Funds (MINECO-FEDER, EU). This publication is part of the TED2021-131721B-I00 project, funded by MCIN/AEI/10.13039/501100011033 and European Union “NextGenerationEU”/PRTR

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