Materials Letters 345 (2023) 134454

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

A novel method to fabricate micro-nano hierarchical topographies on zirconia dental implant using femtosecond laser

Wenjin Li [a], Qian Ding [a], Xianzhi Wang [b], Zhaohua Wang [b,c,d], Zhiyi Wei [b,c,d], Haizheng Xu [e], Lei Zhang [a,*]

[a] Department of Prosthodontics, Peking University School and Hospital of Stomatology & National Center of Stomatology & National Clinical Research Center for Oral Diseases & National Engineering Laboratory for Digital and Material Technology of Stomatology, Beijing 100081, China

[b] Beijing National Laboratory for Condensed Matter Physics, Institute of Physics, Chinese Academy of Sciences, Beijing 100190, China

[c] University of Chinese Academy of Sciences, Beijing 100049, China

[d] Songshan Lake Materials Laboratory, Dongguan 523808, China

[e] Department of Precesion Instrument, Tsinghua University, 100084 Beijing, China

ARTICLE INFO

Keywords:
Laser processing
Microstructure
Bioceramics

ABSTRACT

Zirconia implant has complex structure that is difficult to realize selective femtosecond laser surface micro-structuring with high precision. An acrylic resin guide rail was designed and 3D printed to facilitate femtosecond laser surface microstructuring on zirconia dental implant, and obtained two surface topographies: microgrooves and nanoparticles. The fracture strength of control, microgrooved, and nanoparticles implants were 907.8 ± 59.0 N, 793.7 ± 83.3 N, and 755.6 ± 144.5 N, respectively. Using femtosecond laser with the advocated guide rail is an effective method to fabricate micro-nano hierarchical topographies on zirconia dental implants precisely.

1. Introduction

Zirconia is a viable alternative to titanium with excellent biological, mechanical, aesthetic properties for dental implant application [1]. Femtosecond laser direct writing is a promising surface treatment method for zirconia dental implant, which can form micro-nano hierarchical surface topographies [2]. Previous research reported that microgrooved zirconia surface produced by femtosecond laser could improve osteoblast proliferation and differentiation, regulate collagen fiber orientation through “contact guidance”, thus enhance osseointegration [3,4].

Although fabrication of microgrooves on flat surface is convenient, to achieve identical precision on dental implant is rather difficult that requires high demanding processing platform. The platform should comprise a rotary device and a linear rail, which are coaxial with the implant during processing. Eccentricity or disalignment of the rotary device would result in defocus of the laser and change the microgroove size. Besides, zirconia is a brittle material that is susceptible to fatigue fracture [5]. The edge of the implant threads played a role as stress riser [6]. Therefore, avoiding bringing defects on the area of thread may benefit the mechanical strength of zirconia implants. In summary, to guarantee the precision of the femtosecond laser surface treatment and prevent the implant threads from laser surface treatment is a rather challenging issue.

This research proposed a method to achieve selective femtosecond laser surface treatment on zirconia implants and form micro-nano hierarchical surface topographies precisely despite the inherent motion error of the device, and the fracture strength of zirconia implants was evaluated.

2. Materials and methods

One-piece cylindrical implants with a diameter of 4.1 mm and an intraosseous length of 12 mm were designed using CAD software (Catia V5R19, Dassault System, France; Geomagic Studio 12.0, Geomagic, USA). The spiral threads on the implant had a 1.2 mm pitch and 0.25 mm depth. Then, zirconia implants were CAD/CAM milled (Zenotec Mini, Wieland, Germany) from pre-sintered 3Y-TZP blocks (Zenostar T, Wieland, Germany) and sintered in a high-temperature furnace (Ceramill Therm 3, Amann Girrbach).

* Corresponding author at: Department of Prosthodontics, Peking University School and Hospital of Stomatology, No. 22 Zhongguancun Nandajie, Haidian District, Beijing 100081, China.

E-mail address: drzhanglei@yeah.net (L. Zhang).

https://doi.org/10.1016/j.matlet.2023.134454
Received 1 December 2022; Received in revised form 3 April 2023; Accepted 26 April 2023
Available online 29 April 2023
0167-577X/

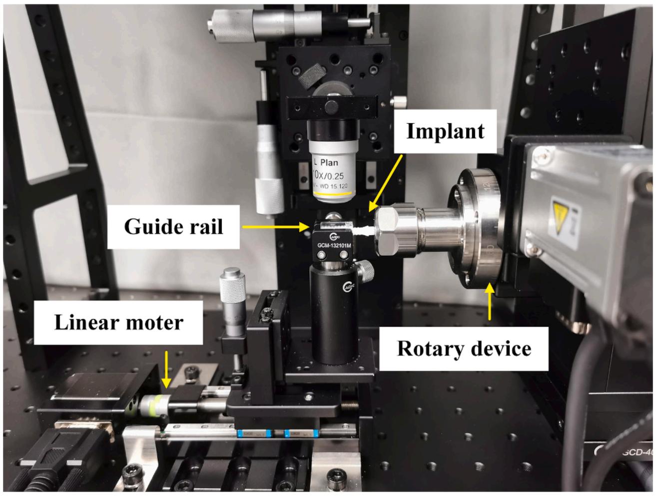

Fig. 1. The femtosecond laser surface treatment platform.

The femtosecond laser surface treatment platform was shown in Fig. 1. Implant was clamped on a rotary device (ZX110-60, Lyseiki, China). To restrict the processing direction of the implant, a guide rail (Fig. 2) was designed in software (UG NX12.0, Siemens, Germany) according to the intraosseous geometry of the implant and presented as a negative mold of the intraosseous part, fabricated with acrylic resin using a 3D printer (Object 30 pro, Stratasys, Israel). The guide rail would maintain restricting and guiding the implant during rotary and processing movement, so that the radial error of the rotary device could be eliminated. The guide rail was held on a linear motor (GCD-101050 M, Daheng, China) with a 5 mm × 4 mm window (Fig. 2b) preserved for laser treatment. The laser scan lines were produced radially by rotating the implant in the guide rail. The fabrication procedure was as follow: Start the rotary device, a laser scan line starts from the neck of the implant and ends at the apical part of the implant, thus forms a microgroove that is parallel with the implant threads. The laser was switched off, then the reverse rotation starts, and makes the implant move back to the start point. The linear motor controls the implant to move horizontally with a distance consistent with the pitch. Afterwards, the laser was switched on, and a new cycle starts to fabricate the next microgroove. A CCD (MER-130-30UM, Daheng, China) was placed on the platform for real-time observation of the femtosecond laser treatment.

Implants were divided into 3 groups according to different surface topographies:

(1) Control (CTRL): no surface treatment after sintering.
(2) Microgrooved (MG): treated by Ti: sapphire femtosecond laser (Spectra physics, USA) with 800 nm wave length, 1KHz repetitive rate, 38 fs pulse width, and 70 μJ pulse energy to fabricate microgrooves with 30 μm width, 7 μm depth, and 60 μm pitch.

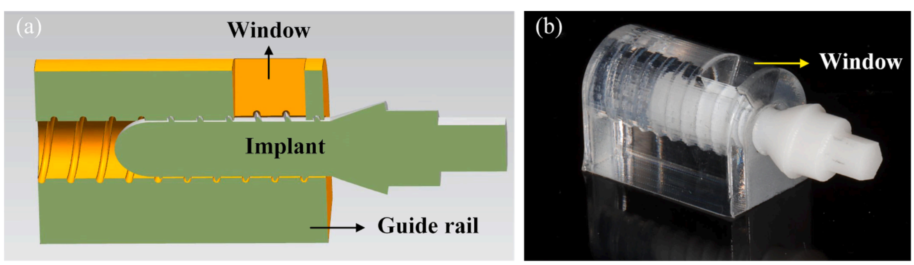

Fig. 2. Cross section diagram (a) and picture (b) of the guide rail and zirconia implant.

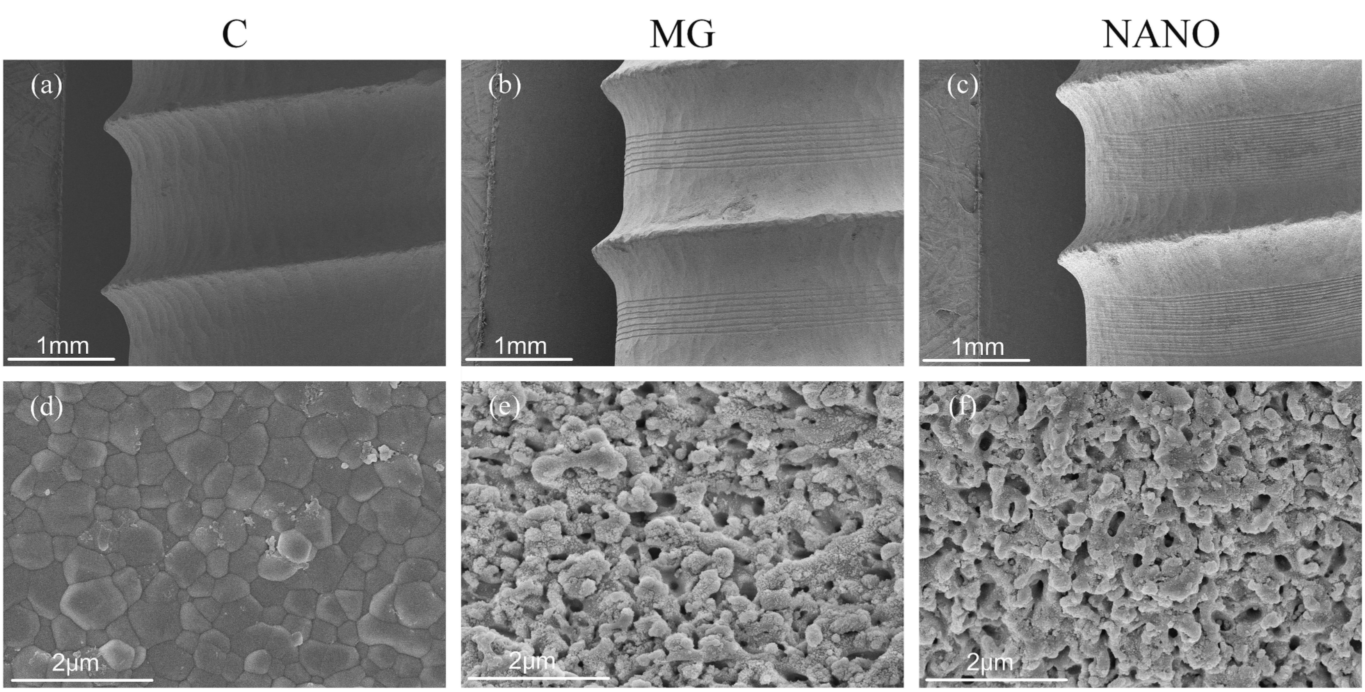

Fig. 3. SEM images of the zirconia implant surface with magnification.

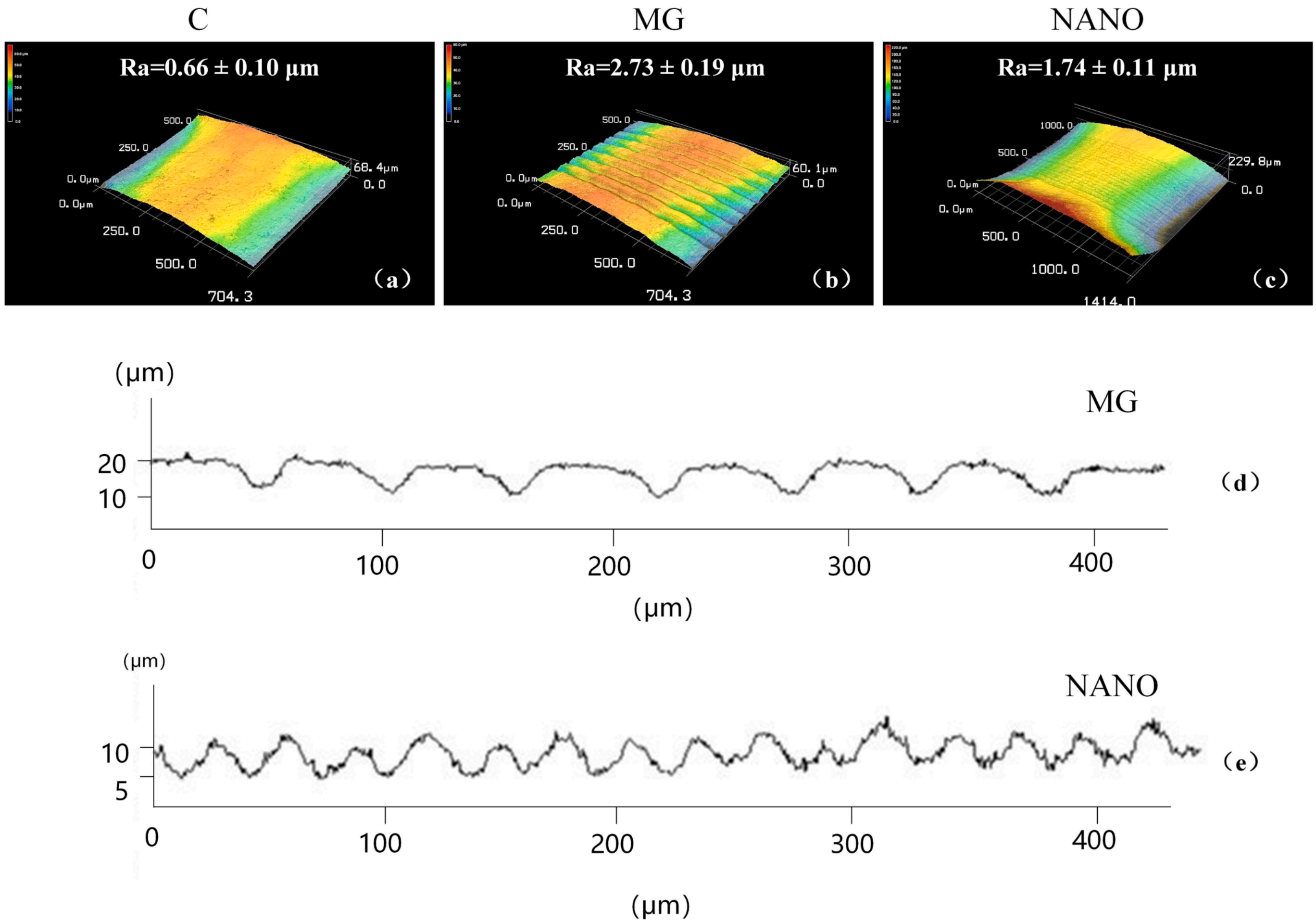

Fig. 4. Surface roughness of zirconia implants (a) (b) (c) and line profile of femtosecond laser treated surface (d) (e).

(3) Nanoparticles (NANO): the same processing parameters as MG and treated with 30 μm pitch (the distance between the center of the two adjacent laser scans).

Surface roughness and size of the microgrooves between the threads were measured by a 3D laser microscope (VK-9700 K, Keyence, Japan). The measurement area was 20 μm × 20 μm, which was entirely in inside of the microgrooves in MG. Three implants from each group were tested (n = 3). The surface topographies of the implants were observed using SEM (SU8010, HITACHI, Japan).

Implants (n = 3) were loaded on a universal testing machine (AGS-X, SHIMADZU, Japan). According to the ISO 14801: 2014 Standard, a static load was applied to the angulated implant under a crosshead speed of 1 mm/min at an angle of 120° to the horizontal plane until fracture. Fracture strength (static strength) was measured at the static load (N) at which the fracture occurred.

3. Results and discussion

SEM images showed that the control group had a flat surface (Fig. 3a) and clear grain boundaries (Fig. 3d). The selective laser treatment area started from 2.8 mm apically from the nominal bone level in MG and NANO groups and located between the threads. The microgrooves were parallel with the implant threads and evenly distributed with distinct boundaries (Fig. 3b). Inside of the microgrooves, the gap among the agglomerated nanoparticles formed a nanoscale porous surface. Nanoparticles with diameter of 200 nm ~ 1 μm were observed on the lateral walls of the microgrooves and the whole surface treated area of NANO, covered with nano-scale moss-like structure (Fig. 3e, f). Many studies have confirmed that micro-nano hierarchical surface topographies could accelerate osseointegration [7,8]. The advantages of the femtosecond laser used in this research are that it can regulate the topographies by changing laser parameter; and locate the surface treatment region selectively.

The size of the microgrooves was precise with 30.02 ± 0.66 μm width, 7.33 ± 0.36 μm depth, and 60.05 ± 0.79 μm pitch. The fabrication error was within 1 μm, which is a combination error of the femtosecond laser and fabrication platform. The surface roughness (Ra value) of CTRL, MG, and NANO groups were 0.66 ± 0.10 μm, 2.73 ± 0.19 μm, and 1.74 ± 0.11 μm respectively (Fig. 4). The Ra value of NANO was in accordance with most commercial dental implants that have moderately rough surfaces (Ra value between 1.0 and 2.0 μm), which has been proved to be favorable for osseointegration [9,10].

Rezaei et al. [3] fabricated microgrooves parallel with the long axis of the zirconia implants by laser scanning. The zirconia implants were smooth cylinder without threads, implanted into rat femurs and achieved higher osseointegration strength than machined surface zirconia implants. Taniguchi et al. [11] used fiber laser to fabricate microgrooves on cylindrical zirconia implants. The microgrooves were parallel with the long axis of the implant. The laser treated implants placed in rat tibia increased the bone-implant contact ratio and removal torque significantly, compared with machined surface. Calvo-Guirado et al. [12] used femtosecond laser to fabricate microgrooves on commercial zirconia implants, which obtained higher bone-to-implant contact percentage compared with titanium implants after implanted into the mandible of dogs. The microgrooves were vertical to the long axis of the implants and intersected the threads.

In this research, laser path that avoid the threads was achieved. The fracture strength of CTRL, MG, and NANO groups were 907.8 ± 59.0 N, 793.7 ± 83.3 N, and 755.6 ± 144.5 N, respectively. The fracture strength of zirconia implants from the three groups had exceeded the maximum bite force in posterior dentition (250–450 N) and anterior dentition (140–200 N) of natural dentition [13]. There was no implant fracture strength reported for laser surface treated implants. Based on our previous research, microgrooves with depth of 30 μm significantly reduced the flexural strength of zirconia specimen [14]. Therefore, the microgrooves depth was designed as 7 μm in this research, which has the potential to preserve the mechanical strength of the implant. The results proved that via rational design and precise implementation of the location and surface topography of the micro-nano structure, the implant fracture strength was minimally impaired and can be clinically acceptable.

4. Conclusion

The femtosecond laser surface treatment platform with the advocated guide rail provides a more flexible and precise solution for zirconia dental implant surface modification. The laser scanning path was parallel with the threads and achieved clinically acceptable fracture strength. Micro-nano hierarchical topographies were revealed by femtosecond laser on zirconia implant surface precisely.

CRediT authorship contribution statement

Wenjin Li: Writing – original draft, Conceptualization, Methodology, Investigation, Formal analysis. **Qian Ding:** Writing – review & editing, Conceptualization, Supervision. **Xianzhi Wang:** Methodology, Software. **Zhaohua Wang:** Supervision, Project administration. **Zhiyi Wei:** Supervision, Project administration. **Haizheng Xu:** Conceptualization, Methodology. **Lei Zhang:** Writing – review & editing, Supervision, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (No. 81671026) and the Capital Health Research and Development of Special (No. 2020-2-4104). Chen Jie is acknowledged for the help in guide rail design.

References

[1] K. Sivaraman, A. Chopra, A. Narayan, D. Balakrishnan, Is zirconia a viable alternative to titanium for oral implant? A critical review, J. Prosthodont. Res. 62 (2) (2018) 121–133.

[2] A. Carvalho, L. Cangueiro, V. Oliveira, R. Vilar, M.H. Fernandes, F.J. Monteiro, Femtosecond laser microstructured Alumina toughened Zirconia: A new strategy to improve osteogenic differentiation of hMSCs, Appl. Surf. Sci. 435 (2018) 1237–1245.

[3] N.M. Rezaei, M. Hasegawa, M. Ishijima, K. Nakhaei, T. Okubo, T. Taniyama, A. Ghassemi, T. Tahsili, W. Park, M. Hirota, T. Ogawa, Biological and osseointegration capabilities of hierarchically (meso-/micro-/nano-scale) roughened zirconia, Int. J. Nanomed. 13 (2018) 3381–3395.

[4] R.A. Delgado-Ruiz, M. Abboud, G. Romanos, A. Aguilar-Salvatierra, G. Gomez-Moreno, J.L. Calvo-Guirado, Peri-implant bone organization surrounding zirconia-microgrooved surfaces circularly polarized light and confocal laser scanning microscopy study, Clin. Oral Implant Res. 26 (11) (2015) 1328–1337.

[5] Y. Zhang, I. Sailer, B.R. Lawn, Fatigue of dental ceramics, J. Dent. 41 (12) (2013) 1135–1147.

[6] R.K. Schenk, D. Buser, Osseointegration: a reality, Periodontol 2000 (17) (1998) 22–35.

[7] Q. Wang, P. Zhou, S. Liu, S. Attarilar, R.-L.-W. Ma, Y. Zhong, L. Wang, Multi-Scale Surface Treatments of Titanium Implants for Rapid Osseointegration: A Review, Nanomaterials-Basel 10 (6) (2020) 1244.

[8] I.-S.-L. Yeo, Modifications of Dental Implant Surfaces at the Micro- and Nano-Level for Enhanced Osseointegration, Materials 13 (1) (2019) 89.

[9] T. Albrektsson, A. Wennerberg, Oral implant surfaces: Part 1 - Review focusing on topographic and chemical properties of different surfaces and in vivo responses to them, Int. J. Prosthodont. 17 (5) (2004) 536–543.

[10] A.I. Nicolas-Silvente, E. Velasco-Ortega, I. Ortiz-Garcia, L. Monsalve-Guil, J. Gil, A. Jimenez-Guerra, Influence of the Titanium Implant Surface Treatment on the Surface Roughness and Chemical Composition, Materials 13 (2) (2020) 314.

[11] Y. Taniguchi, K. Kakura, K. Yamamoto, H. Kido, J. Yamazaki, Accelerated Osteogenic Differentiation and Bone Formation on Zirconia with Surface Grooves Created with Fiber Laser Irradiation, Clin. Implant Dent. Relat. Res. 18 (5) (2016) 883–894.

[12] J.L. Calvo-Guirado, A. Aguilar-Salvatierra, R.A. Delgado-Ruiz, B. Negri, M.P. R. Fernandez, J.E.M.S. de Val, G. Gomez-Moreno, G.E. Romanos, Histological and Histomorphometric Evaluation of Zirconia Dental Implants Modified by Femtosecond Laser versus Titanium Implants: An Experimental Study in Fox Hound Dogs, Clin. Implant Dent R 17 (3) (2013) 525–532.

[13] F.A. Fontijn-Tekamp, A.P. Slagter, A. Van Der Bilt, M.A. Van 'T Hof, D.J. Witter, W. Kalk, J.A. Jansen, Biting and chewing in overdentures, full dentures, and natural dentitions, J. Dent. Res. 79 (7) (2000) 1519–1524.

[14] W.J. Li, Q. Ding, F.S. Yuan, F.B. Sun, J.Q. Zheng, R. Bao, L. Zhang, Effects of femtosecond laser treatment on surface characteristics and flexural strength of zirconia, Beijing Da Xue Xue Bao 53 (4) (2021) 770–775.