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# The relationship of surface roughness and wettability of 316L stainless steel implants with plastic deformation mechanisms

S. Cicek<sup>a</sup>, A. Karaca<sup>a</sup>, I. Torun<sup>b</sup>, M. S. Onses<sup>b</sup>, B. Uzer<sup>a,\*</sup>

<sup>a</sup>Department of Mechanical Engineering, Abdullah Gül University, 38080, Kayseri, Turkey

<sup>b</sup>Department of Materials Science and Engineering, Nanotechnology Research Center (ERNAM), Erciyes University, 38039, Kayseri, Turkey

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## Abstract

The wettability of the implant plays significant role in successful tissue-implant integration and shows strong dependence on the surface topography of the material. Recent studies showed that the plastic deformation mechanisms can improve cell response, and increase surface roughness and energy. In order to understand the effect of these mechanisms on wettability, 316L stainless steel samples were subjected to tensile test and deformed up to 15% to 35% of strain levels. Atomic force microscopy (AFM) presented approximately 22-fold greater average surface roughness on the 35% deformed sample compared to undeformed one. On the other hand, sessile drop test showed contact angle decrease from 82° to 52° as the deformation increased. This finding is significant since much higher contact angle value at similar surface roughness was presented in the literature. This demonstrates that the plastic deformation mechanisms can play significant role in enhancing the surface wettability without a need for a surface treatment technique. Hence, through the activation of these mechanisms, wettability and surface energy of the implant materials could be further increased which would result with enhanced cell response and lessened post-surgical complications.

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*Keywords:* 316L stainless steel; implant; roughness; wettability; Wenzel equation; plastic deformation mechanisms

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\* Corresponding author. Tel.: +90-352-224-8800 (7231)  
E-mail address: [benay.uzer@agu.edu.tr](mailto:benay.uzer@agu.edu.tr)

## Introduction

Metallic materials particularly stainless steel, Co-Cr alloys and titanium (Ti) are extensively used as implant materials in biomedical applications such as artificial knees, heart pacemaker and dental implants [1,2]. As the implants placed in the body they interact with cells and this initial interaction constitutes utmost importance [3,4]. Specifically, relatively high cell adhesion and the non-existence of fibrous tissue interface are significantly important in order to accomplish a successful treatment, [5,6]. Furthermore, any bacterial infection and inflammatory response must be averted in order to obtain an ideal cell attachment on the implant [7].

Researches validate that implant success is dependent on material selection, and physicochemical properties such as wettability, surface free energy, topography and also the roughness of the material [8,9,10]. Studies show that creating surface features in micro- and nano-scale with the techniques such as acid etching or polishing increase the surface roughness and enhance the surface wettability [3,5,10]. This leads implant surface to be more hydrophilic which consequently improves cell attachment and eventually provides a better tissue-implant integration [5,11]. Although the roughness and wettability are two important parameters affecting the cell response on implant significantly, their relationship and joint effect on cell response has not yet been thoroughly analyzed.

Additionally, current studies have addressed that plastic deformation mechanisms (i.e. slip and twinning) increase surface energy and modify the surface properties in such a way that cell response is promoted significantly [6,9]. This recent study on the response of the osteoblast cells on 316L stainless steel showed that cell attachment on the implant surface improved significantly due to the activated plastic deformation mechanisms and increased surface roughness. Especially, significant collagen formation was observed on the sample deformed up to 25% of the strain, which could be due to the ideal match of the surface roughness and collagen molecules [6]. Although these studies have presented the significant effect of the plastic deformation mechanisms on the surface roughness, they lack in defining its relation with the surface wettability, which constitutes importance in cell response. Thus, the current study was carried out with the motivation to elucidate the effect of plastic deformation mechanisms on the surface wettability and its relation with the roughness.

Hence, 316L stainless steels were subjected to tensile test up to different plastic strains in order to activate microstructural mechanisms in different volumetric fractions. Moreover, sessile drop test was performed to analyze the wettability of the sample surfaces. Atomic force microscopy (AFM) was used to investigate the surface topography of the specimens and to measure average surface roughness. The results of this study showed that surface wettability could be increased significantly by plastic deformation, which might be due to the greater energy provided by the slip and twinning mechanisms. In this way implants promoting a better cell adhesion and tissue integration could be manufactured.

## Experiments and Methods

Austenitic stainless-steel with the grade of 316L were used in the current study since it has high mechanical strength, good corrosion resistance and biocompatibility, and low cost [9,12,13]. Initially, the specimens were cut in the dog bone shape by laser cutting in order to use for tensile testing (Figure 1). The samples were obtained mirror polished from the company, thus there was no need further polishing. Uniaxial tensile tests were carried out with the strain rate of  $10^{-4} \text{ s}^{-1}$  and the tests were interrupted at two plastic strains: 15 and 35%, which activated the microstructural mechanisms (slip and twinning) in increasing volumetric fractions. The static water contact angle of the surfaces was measured using Attension Theta Lite system. Prior to the measurements, the samples were washed in acetone followed by ethanol under sonication for 5 min each and then dried with nitrogen. The volume of the droplets was 2  $\mu\text{L}$ . The reported contact angles represent the arithmetic average of 5 measurements taken at different locations of the samples.

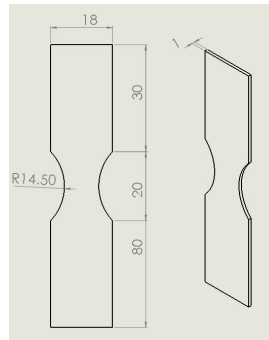


Figure 1. Technical drawing of the test specimens (all dimensions are in mm.)

Atomic force microscopy (AFM) analysis was carried out in order to investigate the surface topographies of the samples at a tapping mode in air utilizing a phosphorus doped silicon cantilever with a rotated tip with an 8 nm radius. The linear scanning rate was set to 1 Hz (1 line/s) and the scanning area of  $50\mu\text{m} \times 50\mu\text{m}$ .

## Results and Discussion

Surface topographies of the specimens deformed up to different strain levels were analyzed via AFM. 3D images of the specimens show that the surface topographies get distorted as the plastic strain increases (Figure 2).

Enhanced slip-twin interaction in parallel with the increasing plastic deformation was also observed on the sample deformed up to 15% of strain. These interactions constitute importance for the cell response on the implant surface since they increase surface energy and catalyze the deposition of proteins necessary for cell adhesion on the implant surface [9]. In addition, the activation of these mechanisms create grooves in micro- and nano-scale, which significantly provides cells to form focal contacts through which they attach, spread and proliferate on the surface [9]. Thus, these structures created by the plastic deformation mechanisms could enhance the tissue-implant integration and successively decrease the rehabilitation period after the surgery.

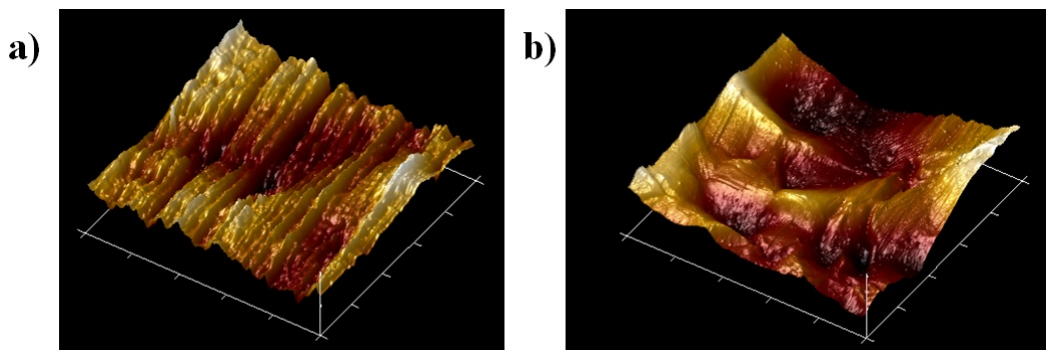


Figure 2. Surface topographies of the (a) undeformed and (b) 15% deformed sample (scanned area:  $50\mu\text{m} \times 50\mu\text{m}$ ).

This distortion increases roughness such that, average surface roughness ( $R_a$ ) data showed a 22-fold increase on the 35% deformed sample compared with the undeformed one (Table 1).

Table 1. Surface roughness and contact angle data of the test specimens. Values are the mean  $\pm$  SD

Strain value (%)	Roughness	Contact Angle
	R <sub>a</sub> (nm)	$\theta^{\text{water}}$ (deg)
0	25.41 $\pm$ 2.47	81.867 $\pm$ 0.27
15	296.11 $\pm$ 5.04	64.886 $\pm$ 1.04
35	569.85 $\pm$ 5.23	51.601 $\pm$ 0.09

Wettability of the samples was investigated by sessile drop test and the contact angle of the droplets on each specimen was recorded. While this angle was measured as 82° on the undeformed sample, showing a similar value with the literature [14], it decreased to 52° on the 35% deformed sample. The decrease of contact angle meaning a greater hydrophilicity and wettability might be stemming from the increased surface roughness and greater surface energy provided by the slip and twinning mechanisms [14]. The decrease of the contact angle following plastic deformation can be explained with the Wenzel equation ( $\cos\theta^* = r\cos\theta$ ) [15]. Based on the apparent contact angle ( $\theta^*$ ) of 52° and Young contact angle ( $\theta$ ) of 82°, the Wenzel equation predicts the roughness ratio ( $r$ ) of 4.4, which is the ratio between the actual and projected surface area. The value of  $r$  is greater than unity for rough surfaces and the Wenzel equation suggests increase of the wettability (i.e. decrease of contact angle) with increasing roughness for  $\theta < 90^\circ$ . The decrease of the contact angle together with the increase of the roughness following the plastic deformation, therefore is in good qualitative agreement with the Wenzel state.

Earlier studies carried out to analyze the relationship between the surface roughness and wettability utilized mechanical, physical or chemical treatments, which were confined with the modifications on the surface [15,16]. For example, surface mechanical attrition treatment (SMAT) utilized to improve the mechanical properties of the 316L steel exhibited the increase of surface roughness by approximately 20 fold, whereas decrease of the contact angle of the droplet from 88.6° to 74.4° [14]. Compared to the current study's results SMAT technique provides a lower wettability at a similar surface roughness. This shows that the wettability of the implants could be improved much further by tailoring its microstructure through plastic deformation mechanisms instead of solely modifying the surface properties. It should be noted that the surface roughness analysis is strongly dependent on the measurement technique and the area scanned. Therefore, in order to understand the effect of plastic deformation mechanisms on the surface roughness and consequently the wettability and cell response extensive research is required.

## Conclusion

The relationship of surface roughness and wettability of the implants with the plastic deformation mechanisms was analyzed on 316L stainless steel samples deformed up to 15 and 35% of strains with tensile test. AFM results showed highly distorted surface and 22 fold greater R<sub>a</sub> value on the 35% deformed specimen compared to the undeformed one. Sessile drop test presented a decrease of the contact angle from 82° to 52° as the deformation increased suggesting a greater wettability compared with the earlier study. These results show that by virtue of plastic deformation mechanisms, which lead to higher surface roughness and energy, surface wettability could be increased significantly without a need for a surface treatment technique. In this way implants leading to a better tissue-implant integration could be manufactured.

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## References

- [1] M. Niinomi, *Metall. Mater. Trans. A* 33 (2002) 477–486.
- [2] M.T.M. and Z.A.K. and A.N. Siddiquee, *Int. J. Chem. Mol. Nucl. Mater. Metall. Eng.* 8 (2014) 821–827.
- [3] Q. Chen, G.A. Thouas, *Mater. Sci. Eng. R Reports* 87 (2015) 1–57.
- [4] A. Zareidoost, M. Yousefpour, B. Ghaseme, A. Amanzadeh, *J. Mater. Sci. Mater. Med.* 23 (2013) 1479–1488.
- [5] K. Anselme, *Biomaterials* 21 (2000) 667–681.
- [6] B. Uzer, F. Monte, K.R. Awad, P.B. Aswath, V.G. Varanasi, D. Canadinç, in: *TMS Annu. Meet. Exhib.*, Springer, 2018, pp. 295–301.
- [7] P. Linez-Bataillon, F. Monchau, M. Bigerelle, H.F. Hildebrand, in: *Biomol. Eng.*, 2002, pp. 133–141.
- [8] Y. Iwaya, M. Machigashira, K. Kanbara, M. Miyamoto, K. Noguchi, Y. Izumi, S. Ban, *Dent. Mater. J.* 27 (2008) 415–21.
- [9] B. Uzer, S.M. Toker, A. Cingoz, T. Bagci-Onder, G. Gerstein, H.J. Maier, D. Canadinc, *J. Mech. Behav. Biomed. Mater.* 60 (2016) 177–186.
- [10] J.I. Rosales-Leal, M.A. Rodríguez-Valverde, G. Mazzaglia, P.J. Ramón-Torregrosa, L. Díaz-Rodríguez, O. García-Martínez, M. Vallecillo-Capilla, C. Ruiz, M.A. Cabrerizo-Vilchez, *Colloids Surfaces A Physicochem. Eng. Asp.* 365 (2010) 222–229.
- [11] K. Webb, V. Hlady, P.A. Tresco, *J. Biomed. Mater. Res.* 41 (1998) 422–430.
- [12] A. Latifi, M. Imani, M.T. Khorasani, M.D. Joupari, *Surf. Coatings Technol.* 221 (2013) 1–12.
- [13] B. Al-Mangour, R. Mongrain, E. Irissou, S. Yue, *Surf. Coatings Technol.* 216 (2013) 297–307.
- [14] B. Arifvianto, Suyitno, M. Mahardika, P. Dewo, P.T. Iswanto, U.A. Salim, *Mater. Chem. Phys.* 125 (2011) 418–426.
- [15] J. Drelich, J.D. Miller, *J. Colloid Interface Sci.* 164 (1994) 252–259.
- [16] L. Ponsoynet, K. Reybier, N. Jaffrezic, V. Comte, C. Lagneau, M. Lissac, C. Martelet, *Mater. Sci. Eng. C* 23 (2003) 551–560.