

Electrochemical Polishing of 316L Stainless Steel Slotted Tube Coronary Stents: An Investigation of Material Removal and Surface Roughness

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Summary

Surface roughness is one of the properties that determines the performance of coronary stents. Therefore, in the production and application of stents, surface polishing is of paramount importance. In the present study, electrochemical polishing was performed on 316L stainless steel slotted tube coronary stents made by laser cutting. Additionally, both acid pickling and annealing, as the pretreatment of electrochemical polishing, were also conducted. Material removal of the stents (both weight loss and strut width change) in the process of both pickling and electrochemical polishing was investigated. Both qualitative and quantitative roughness measurements were made to evaluate stent surface quality. Furthermore, material characterization of the stents was determined by means of composition analysis, metallographic characterization, and hardness measurement. The removal of material during both pickling and electrochemical polishing was within an acceptable range. Pickling caused a decrease in roughness of the cutting zone. Annealing resulted in an increase in roughness of the stent surfaces. Electrochemical polishing caused a smooth stent surface, which is comparable to commercially available stents. Moreover, the annealing treatment caused the stents to change from a deformed microstructure to a homogeneous structure, which is the major determining factor for the expandability of balloon stents.

Key Words

Coronary stents, electrochemical polishing, surface roughness of stents

Introduction

Metallic coronary stents are medical devices that can provide endovascular scaffolding to relieve vascular obstructions. They exert a continuous radial pressure on the diseased coronary artery, resulting in a compression of atherosclerotic plaques, sealing of dissections, and expansion of the coronary vessel [1,2]. When used as an adjunct to conventional balloon angioplasty, they improve vessel patency [3,4].

The surface roughness of stents is an important determinant of their thrombogenicity and tissue reaction [5],

i.e., the nature of the metal surface is crucial to blood compatibility [6]. A smooth surface can help prevent the activation and aggregation of platelets, which is recognized as one component of the thrombosis process. Previous animal experiments [5,7-9] have shown that surface treatment using electrochemical polishing can improve the performance of stents. The results of implantation of the polished stainless steel coronary stents were compared with those of non-polished stents. The conclusion is that metallic surface

treatment with electrochemical polishing effectively results in a decreased thrombogenicity. The most important clinical problem after stent implantation is still neointimal hyperplasia within the stent, which results in a significant 16% – 30% narrowing of the stent [2,3,10-12]. Further efforts to improve the clinical results of coronary stents should focus on decreasing this neointimal hyperplasia. Previous studies with animal models [7,8] have shown that the metallic surface treatment using electrochemical polishing decreased not only thrombogenicity but also neointimal hyperplasia.

Electrochemical polishing is a process in which a metallic surface is smoothed by polarizing it anodically in a suitable electrolyte [13]. It is understood as two processes: anodic leveling and anodic brightening [14]. Anodic leveling results from a difference in the dissolution rate between the peaks and valleys on a rough metal surface, depending on the current distribution or mass-transport conditions. On the other hand, anodic brightening is associated with suppressing the influence of the metal microstructure on the dissolution rate. A smooth electrochemically polished surface, which appears bright to the naked eye, results from a combination of these two factors [14]. Optimization of electrochemical polishing of 316L stainless steel slotted tube coronary stents produced by laser cutting has been investigated [15]. The focus of the present study is on the investigation of material removal and the surface roughness of the 316L stainless steel slotted tube coronary stents during electrochemical polishing. The objective is to evaluate the effect of electrochemical polishing on the 316L stainless steel slotted tube coronary stents.

Materials and Methods

Materials

The original material used in this study was 316L stainless steel slotted tube coronary stents produced from a stainless steel tube (surgical grade stainless steel, ASTM F138) by laser cutting (Precision Cutting Systems nv, Belgium), which had a length of about 15.0 mm and a diameter of 1.6 mm. The wall thickness of the stents was 95.0 μm . Additionally, two commercial coronary stents, a Cook stent and an ACS stent, were used for the sake of comparison. The received samples were cleaned using distilled water and then ethanol in an ultrasonic agitation bath for at least 15 min, and dried by air blowing.

	HF (48%)	HNO ₃ (65%)	H ₂ O
Amount (ml)	3	9	88

Table 1. Composition of pickling solution.

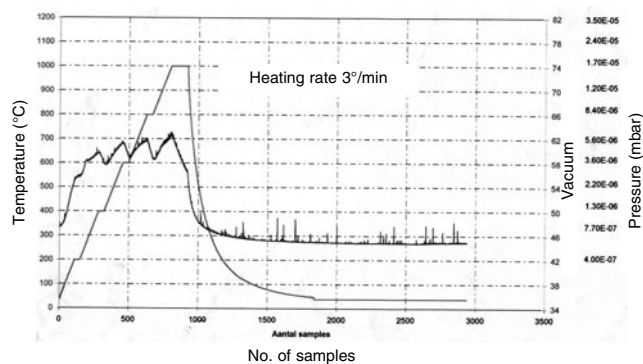


Figure 1. Condition for annealing. See text for detailed information.

Acid Pickling

Pickling was performed on the cut stents by immersing them in an acid solution (Table 1), consisting of HF, HNO₃, and H₂O, at 40 °C for 40 min. A thermostat was used to establish a constant temperature. After this pickling treatment, the pickled stents were cleaned ultrasonically using distilled water and then ethanol for at least 15 min, and were then dried by air blowing.

Annealing

Annealing was conducted on the pickled stents in a vacuum furnace (Leybold Heraeus PD 400, Germany). As shown in Figure 1, the stents were initially heated in a vacuum at a heating rate of 3 °C/min, from room temperature to the annealing temperature of 1000 °C, and were then kept at 1000 °C for 1 h. Finally, they were cooled in a vacuum at room temperature. After annealing, the annealed stents were cleaned by rinsing them ultrasonically in distilled water and then ethanol for at least 15 min, and dried by air blowing.

Electrochemical Polishing

Electrochemical polishing was conducted on the annealed stents. The device used for electrochemical polishing was a self-construction, as illustrated in Figure 2. A 400 ml glass beaker was used as a cell. A DC rectifier (Polipower, Struers, Denmark) was used as a power supply (30 V maximum). The stents were

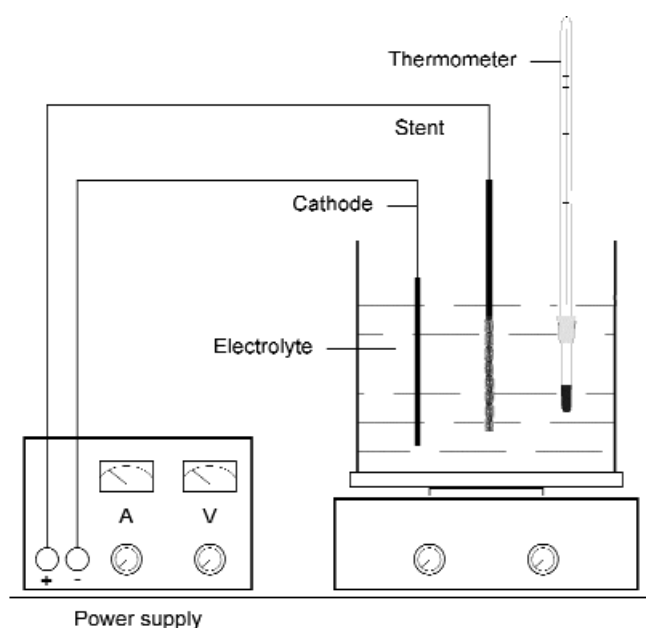


Figure 2. Set-up for electrochemical polishing.

	H ₃ PO ₄ (85wt%)	Glycerol	H ₂ O
Amount (wt%)	42	47	11

Table 2. Electrolyte for electrochemical polishing. wt% = weight percentage.

Voltage (V)	Current (A)	Time (min)	Temperature (°C)
10 – 12	1.2	1	90 – 95

Table 3. Condition for electrochemical polishing.

used as an anode, and the cathode was a 316L stainless steel sheet (15 cm long, 4 cm wide, and 0.2 cm thick). The polishing temperature was controlled with a thermostat. The electrolyte is summarized in Table 2. It consists of H₃PO₄ (85wt%, weight percentage), glycerol, and H₂O. The conditions for electrochemical polishing, which were determined by experiment, are presented in Table 3. After electrochemical polishing, the polished stents were cleaned ultrasonically using distilled water and then ethanol for at least 15 min, and were then dried by air blowing.

Measurements of Weights and Dimensions of Stents

Measurement of the weights of a cut stent, a pickled stent, and an electrochemically polished (ECP) stent was carried out with an electronic analytical balance

(Mettler AE 100, Switzerland). The widths of the stent struts (as a cut stent, a pickled stent, and an ECP stent) were measured using a micrometer (Filar Micrometer Eyepiece, American Optical 426C, USA) in conjunction with a light optical microscope (Metalloplan, Leitz, Germany). Five measuring sites of the stent struts were arbitrarily chosen to conduct the width measurement, and a mean value was calculated. Weight loss and strut width reduction of the stents during the pickling and electrochemical polishing process were calculated as follows:

$$\text{weight loss} = (\text{weight}_{\text{before}} - \text{weight}_{\text{after}}) / \text{weight}_{\text{before}}$$

$$\text{reduction} = (\text{width}_{\text{before}} - \text{width}_{\text{after}}) / \text{width}_{\text{before}}$$

Roughness Measurements of Stents

Roughness measurements were performed qualitatively and quantitatively to the stents, consisting of the cut, pickled, annealed, ECP, ACS, and Cook stents. Evaluation of the surface morphologies (qualitative roughness measurement) was performed by means of scanning electron microscopy (SEM) (Philips 515 SEM, The Netherlands). Pictures were taken with the SEM. The quantitative roughness was measured by means of profilometry (WYKO NT 3300 Profiling system, Veeco, The Netherlands). The long cutoff wavelength (sampling) and the short cutoff wavelength were 25 μm and 0.2 μm , respectively. The evaluation length was 125 μm (five sampling lengths). Approximately 150 measuring lines were used, and thus the mean roughness values could be obtained.

Material Characterization of Stents

Composition analysis of the cut stent was performed using energy dispersive spectrometry (EDS) in conjunction with SEM (Philips 515 SEM, the Netherlands). Metallographic characterization was performed on the transversal section of the cut and ECP stents. First, grinding was done with waterproof abrasive paper (1200, 4000). Mechanical polishing was then performed gradually with 3 μm and 1 μm diamond pastes and finally with a slurry of 0.05 μm SiO₂ suspension. After this mechanical polishing, the samples were rinsed ultrasonically using distilled water and were then dried by air blowing. Etching was conducted on the mechanically polished samples using an etchant (Glyceregia) consisting of 10 ml HNO₃ (65%), 30 ml HCl (36% – 38%), and 30 ml glycerol. Light optical microscopy (LOM) (Polyvar met, Reichert-

Jung, Germany) was then performed to observe the microstructures. Pictures were taken with the LOM. Vickers hardness was measured with a Micro-Vickers tester (Leitz) on the cross section of the cut and ECP stents with a load of 50 g.

Results

Removal of Stent Material

Table 4 presents the weights, the mean strut widths, and the calculated weight loss and width reduction of the cut, pickled, and ECP stents. As shown, the cut stent had a weight of about 13.0 mg and a strut width of approximately 138.5 μm . After pickling, there was a weight loss of about 7.7%, and the strut width decreased by 5.4% to about 131.0 μm . Using these measured values together with the original wall thickness of the stent (95.0 μm), the strut thickness could be calculated from the relationship between the weight and volume, assuming that the strut length remained constant before and after pickling. By this calculation, a strut thickness decrease of approximately 2.4% was obtained, i.e., after pickling, the strut thickness was about 92.7 μm . Due to electrochemical polishing, the weight and strut width of the stent decreased by 16.7% and 6.0%, respectively. The calculated result showed that the strut thickness had decreased by 11.4%, i.e., the strut thickness was approximately 82.1 μm . Therefore, after these two chemical processes, the final dimensions of the stent strut were approximately 123.2 μm wide and 82.1 μm thick.

Evaluation of Surface Morphology

Figure 3 shows SEM pictures of the morphologies of the cutting zone and outer surface of the cut, pickled, annealed, and ECP stents. As can be seen, due to the slag in the cutting zone, considerable roughness of the cut stent resulted in an unacceptable surface quality (Figure 3a). By effectively removing the slag after the acid pickling process, a relatively smoother surface was thus obtained (Figure 3b), as compared to the cut stent. Moreover, pickling could also remove the products covering the outer surface of the stent formed while producing the tubing, thus resulting in a rougher outer surface. The grain boundary grooves were observed in the SEM picture of the annealed stent (Figure 3c), which could probably result in an increase in the surface roughness. Clearly, after electrochemical polishing, the grain boundary grooves disappeared,

Stent	Weight (mg)	Width (μm)	Weight loss (%)	Width reduction (%)
As cut	~13.0	138.5 \pm 1.5	–	–
Pickled	~12.0	131.0 \pm 1.3	~7.7	5.4
ECP	~10.0	123.2 \pm 1.5	~16.7	6.0

Table 4. Weights, strut widths, weight losses, and strut width changes of the stents.

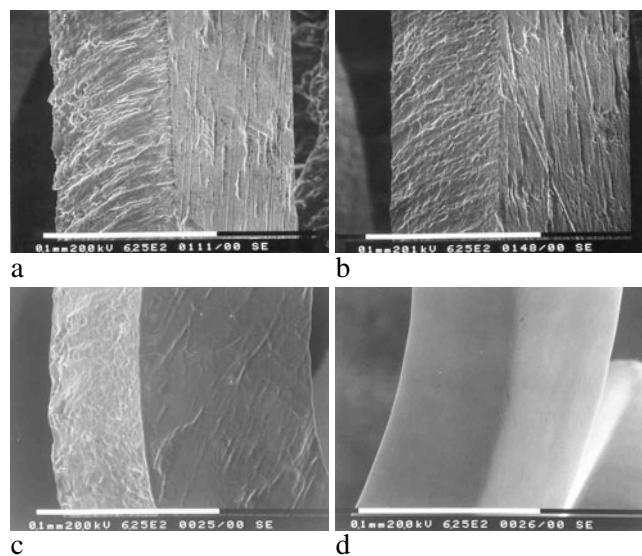


Figure 3. Scanning electron microscopic pictures of the cutting zone and outer surface of four stents: panel a) cut, panel b) pickled, panel c) annealed, and panel d) electrochemically polished.

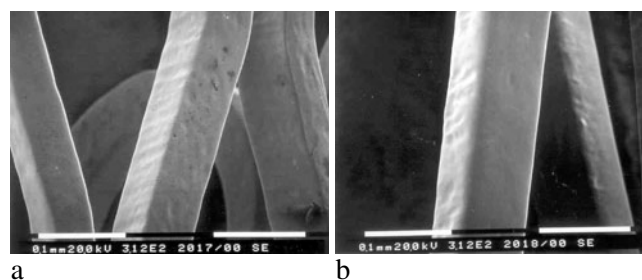


Figure 4. Scanning electron microscopic pictures of a Cook stent (panel a) and an ACS stent (panel b).

and a considerably smooth surface was revealed (Figure 3d). The surface quality was largely improved compared to both the pickled stent and the annealed stent. Figure 4 provides SEM pictures of the surface morphologies of the two commercial stents, the Cook stent (Cook Group, USA), and the ACS stent (Guidant, USA). As can be seen, although the cutting zone revealed relatively less smoothness, these two com-

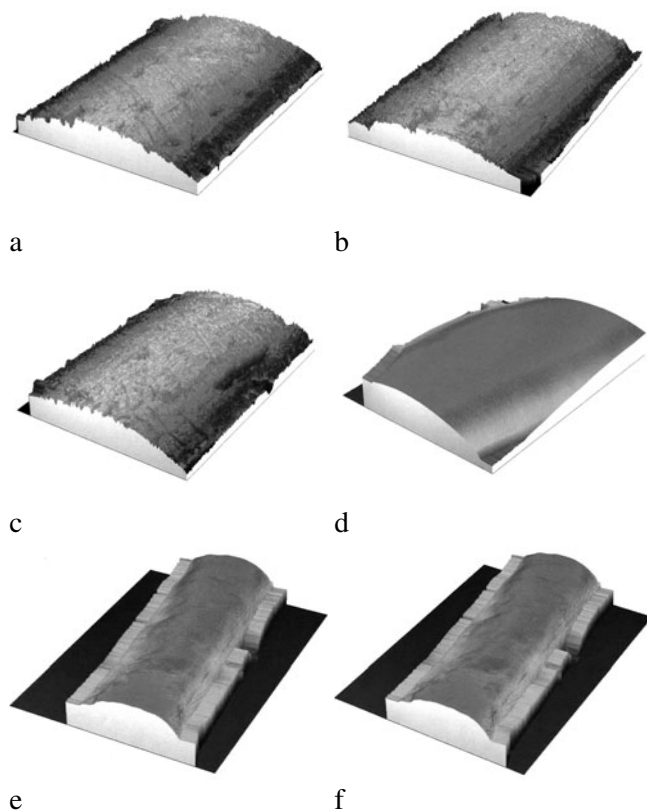


Figure 5. Three-dimensional display of the outer surface of the six stents: panel a) cut, panel b) pickled, panel c) annealed, panel d) electrochemically polished, panel e) Cook, and panel f) ACS.

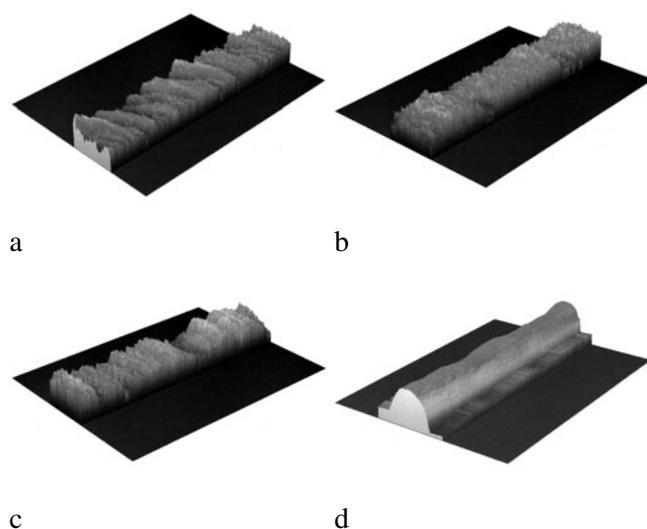


Figure 6. Three-dimensional display of the cutting zone of the four stents: panel a) cut, panel b) pickled, panel c) annealed, and panel d) electrochemically polished.

mercial stents had a smooth surface. Comparing these two surface morphologies with the ECP stent (Figure 3d), it can be concluded that the surface quality of the ECP stent was acceptable.

Figure 5 shows the three-dimensional surface status, obtained by profilometry, of the outer surface of the cut stent, pickled stent, annealed stent, ECP stent, Cook stent, and ACS stent. These were the areas on which the quantitative roughness was measured. It is apparent that the ECP stent (Figure 5d) had a smoother outer surface, compared to both the Cook stent (Figure 5e) and the ACS stent (Figure 5f). The polished stents, consisting of the ECP stent, the Cook stent, and the ACS stent, had a much smoother outer surface than the non-polished stents, i.e., the cut, pickled, and annealed stents. Figure 6 shows the three-dimensional surface status of the cutting zone of the cut, pickled, annealed, and ECP stents. Clearly, the ECP stent had a much smoother surface in the cutting zone than the non-polished stents, consisting of the cut, pickled, and annealed stents. These results further demonstrated the effectiveness of electrochemical polishing.

Quantitative Roughness Measurement

Figure 7 exhibits the profiles of the outer surface of the cut stent, pickled stent, annealed stent, ECP stent, Cook stent, and ACS stent. Figure 8 shows the profiles of the cutting zone of the cut, pickled, annealed, and ECP stents. Roughness can be determined from these profiles. Table 5 presents the average roughness R_a values of the cut, pickled, annealed, and ECP stents. Figure 9 shows how this average roughness value changed using the different treatments: pickling, annealing, and electrochemical polishing. As can be seen, in the original cut stent, the average roughness R_a of the cutting zone was four times greater than the outer surface. It was the largest roughness among all the measurements. By removing the slag, the roughness of the cutting zone of the pickled stent appeared to be two times smaller compared to that of the cut stent. Meanwhile, the roughness of the outer surface increased somewhat, as the product covering the outer surface during the tubing production was removed. As seen from the surface morphology of the annealed stent (Figure 3c), after annealing, grain boundary grooves appeared on the stent surface. This resulted in an increase in roughness, as compared to the pickled stent. By this quantitative roughness measurement, it is clearly demonstrated that electrochemical polishing

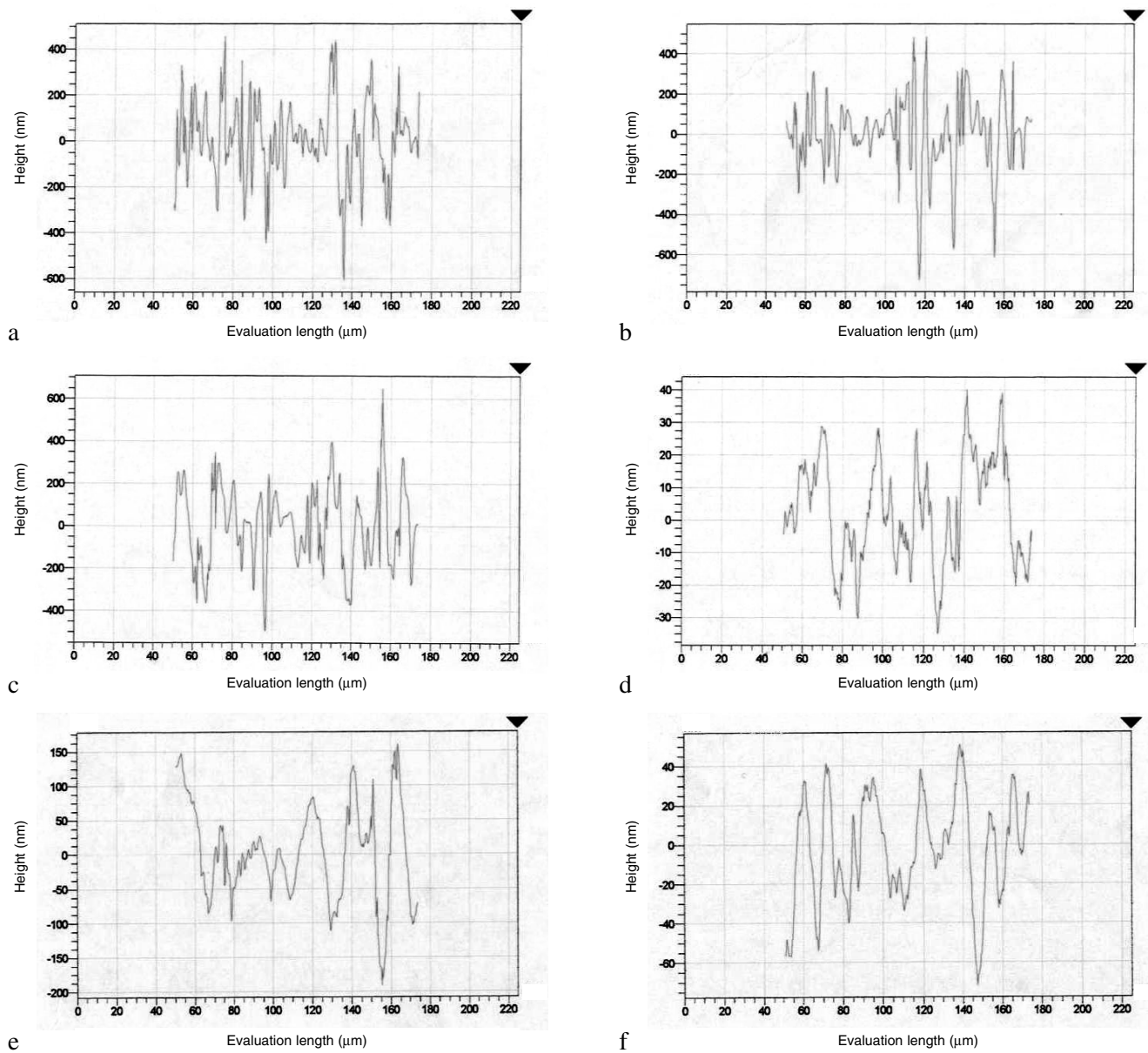


Figure 7. Profiles of the outer surface of the six stents: panel a) cut, panel b) pickled, panel c) annealed, panel d) electro-chemically polished, panel e) Cook, and panel f) ACS.

provided a large decrease in stent roughness. Meanwhile, the cutting zone of the ECP stent had almost the same roughness as the outer surface, i.e., it had a uniform surface status.

Figure 10 shows that the average roughness R_a of the outer surface of the ECP stent and the ACS stent was approximately three times smaller than the Cook stent. As seen from the SEM images in Figure 4, the cutting zone of both the ACS stent and the Cook stent was vis-

Sample	Outer surface (nm) mean \pm SD	Laser-cut zone (nm) mean \pm SD
As cut stent	120.52 \pm 25.65	491.26 \pm 52.46
Pickled stent	126.07 \pm 37.13	268.67 \pm 27.7
Annealed stent	142.71 \pm 26.20	302.90 \pm 23.33
ECP stent	13.13 \pm 1.56	15.01 \pm 1.79

Table 5. Roughness R_a of the stents determined by profilometry. SD = standard deviation.

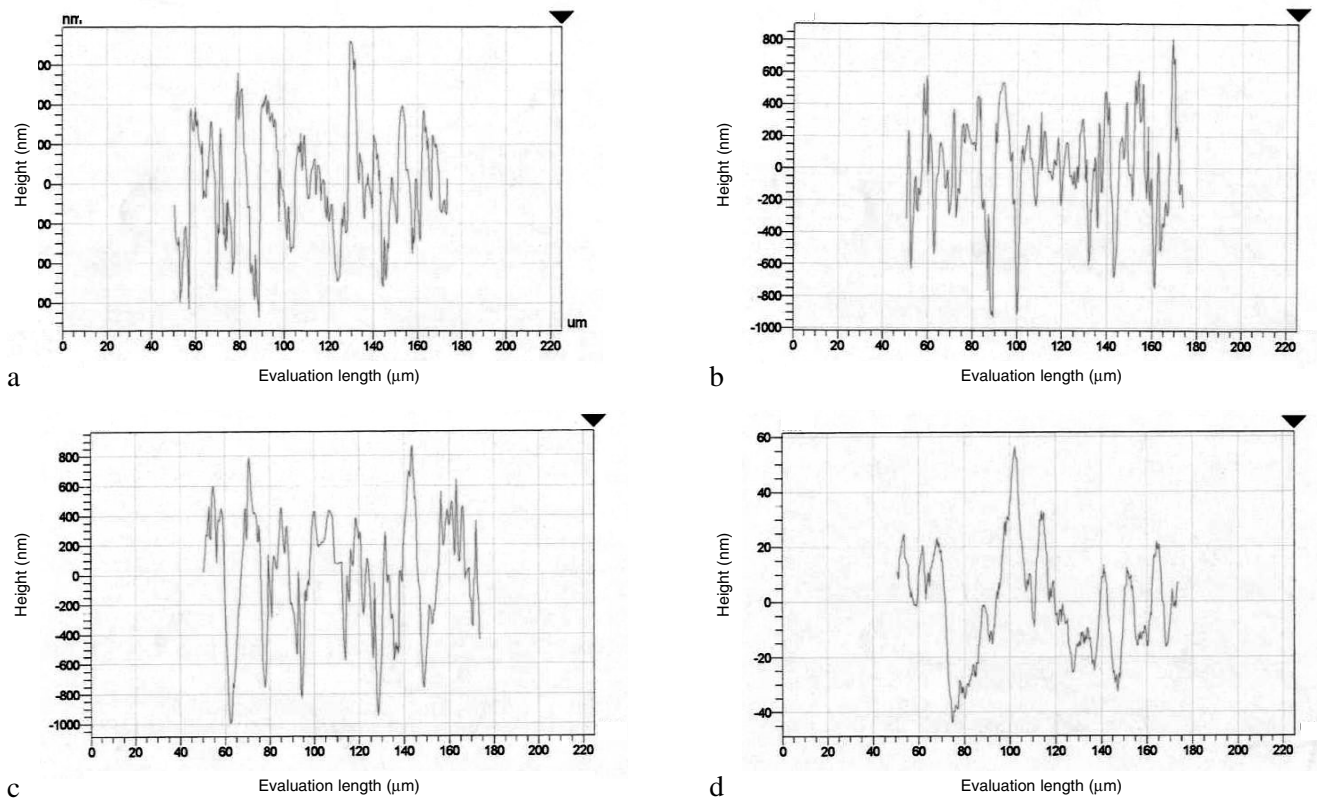


Figure 8. Profiles of the cutting zone of the six stents: panel a) cut, panel b) pickled, panel c) annealed, and panel d) electrochemically polished.

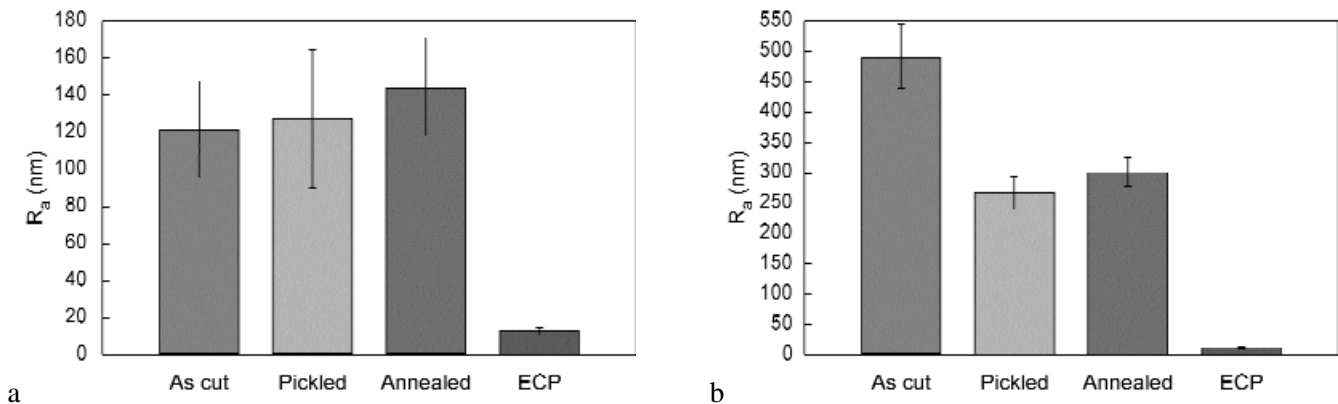


Figure 9. Comparison of roughness between the different treated stents. Panel a) outer surface, panel b) cutting zone. ECP = electrochemical polished.

ibly rougher than their outer surfaces. Therefore, it can be demonstrated that in respect to smoothness the surface quality of the ECP stents obtained by applying electrochemical polishing was completely acceptable. Topography is the geometrical surface structure, including roughness, form errors, waviness, and veining. According to the British Standard, roughness is

defined as "The irregularities in the surface texture which are inherent in the production process but excluding waviness and errors of form" [16]. The roughness measurement technologies are divided into qualitative measurement and quantitative measurement. SEM gives a good qualitative image of surface roughness. Features magnified to about 10 nm can be

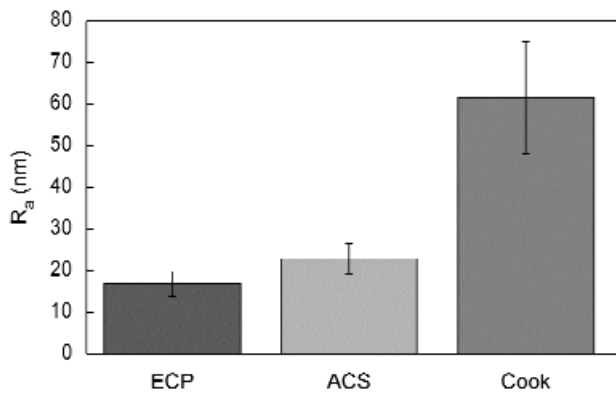


Figure 10. Comparison of the outer surface roughness between the electrochemical polished (ECP) stent and two commercial stents: ACS stent and Cook stent.

R_a	R_t	λ_c (mm)	S/E length (mm)
$R_a < 0.2 \mu\text{m}$	$R_t < 0.1 \mu\text{m}$	0.08	0.08/0.4
$0.02 \mu\text{m} < R_a \leq 0.1 \mu\text{m}$	$0.1 \mu\text{m} < R_t \leq 0.5 \mu\text{m}$	0.25	0.25/1.25
$0.1 \mu\text{m} < R_a \leq 2 \mu\text{m}$	$0.5 \mu\text{m} < R_t \leq 10 \mu\text{m}$	0.8	0.8/4
$2 \mu\text{m} < R_a \leq 10 \mu\text{m}$	$10 \mu\text{m} < R_t \leq 50 \mu\text{m}$	2.5	2.5/12.5
$10 \mu\text{m} < R_a \leq 80 \mu\text{m}$	$50 \mu\text{m} < R_t \leq 200 \mu\text{m}$	8	8/40

Table 6. Relationship between standard roughness and evaluation length. S/E length = sample/evaluation length.

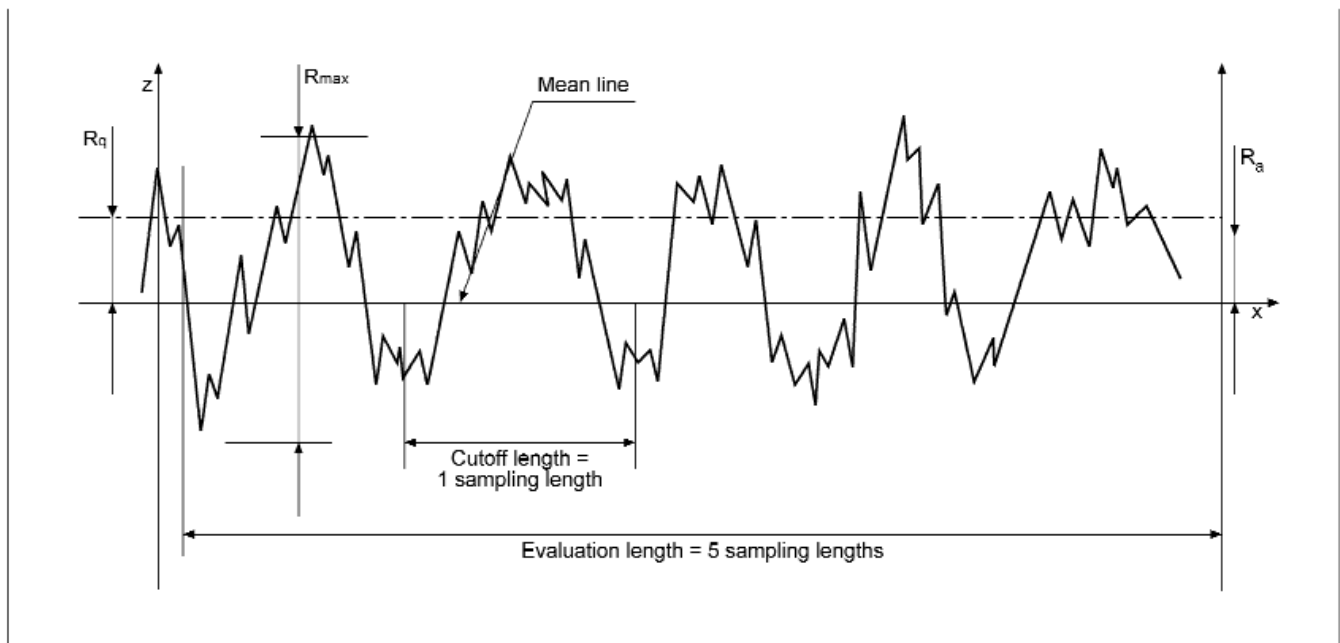


Figure 11. Illustration of definitions of roughness. z = outer surface; x = cutting zone.

observed [17]. Quantifying the degrees of roughness can be very complex, although various methods have been used to provide a quantitative description of the roughness, such as contact profilometry and optical non-contact profilometry.

Various roughness values have been defined in the International Standard [18], which are used to quantitatively describe the roughness of a material surface, some of which are shown in Figure 11, including average roughness R_a , root mean square roughness R_q , and maximum peak-to-valley height R_{max} . In the International Standard [19], some surface profiling

parameters are defined. Profile filters separate profiles into long-wave and short-wave components. The λ_c profile filter separates the roughness profile from long-wave components (e.g., waviness). The cutoff wavelength λ_c of a profile filter determines which wavelengths belong to roughness and which ones to waviness. The sampling length is the reference for roughness evaluation. Its length is equal to the cutoff wavelength λ_c . The evaluation length is the part of the length over which the values of surface roughness are determined. The standard roughness evaluation length comprises five consecutive sampling lengths. Different

roughness is related to the selection of cutoff λ_c of a profile filter. Table 6 shows the standard measurements defined in the International Standard [20]. In this study, the roughness values of these stents were beyond the ranges of the International Standard in the evaluation length due to the very small strut dimensions. They can also be valuable for a comparison of the surface roughness between the different stent surface states, but they must be obtained in the same evaluation length.

Material Characterization

Table 7 shows the result of the composition analysis obtained using EDS. These values could provide a preliminary expression of the stent composition. As can be seen, no carbon was detected. This is due to the shortcomings of the employed analysis method. The element with an element number below 8 could not be detected using EDS. Moreover, the expected concentration of carbon is too low to be detected by EDS. Figure 12 shows two LOM pictures of the microstructure of the cross section of a cut stent. Figure 13 shows the microstructure of the cross section of an ECP stent. Figure 14 shows the Vickers hardness of the cut stent and the ECP stent. As can be seen, the cut stent had a "deformation" microstructure, which was formed while producing the tube. Due to the annealing treatment prior to electrochemical polishing, the ECP stent revealed homogeneous grains. The annealing treatment provided a decrease in the Vickers hardness from 363.69 ± 10.80 to 159.26 ± 5.76 . As can be observed in Figure 12b, the microstructure at the cutting edge was the same as in the bulk. It is known that the thermal effect of laser cutting can cause a heat-affected zone (HAZ) formation at the cutting edge showing a different microstructure from the bulk. Therefore, it might be assumed that the HAZ resulting from the employed laser cutting was very small. Obviously, as can be seen in Figure 13b, with such a small initial HAZ, the ECP stent revealed no HAZ after electrochemical polishing.

	Fe	Cr	Ni	Mo	Si
Fraction	63.13	21.31	13.57	1.43	0.56
Fraction	63.14	19.84	14.27	2.46	0.28

Table 7. Composition of the 316L stainless steel coronary stent determined by energy-dispersive spectrometry. at% = atom percentage, wt% = weight percentage.

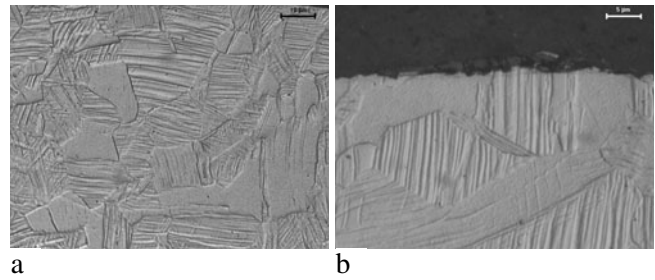


Figure 12. Microstructures of a cut stent. Scale: panel a) 10 μm , panel b) 5 μm .

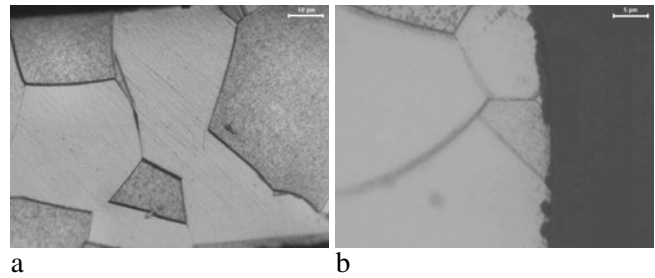


Figure 13. Microstructures of an electrochemically polished stent. Scale: panel a) 10 μm , panel b) 5 μm .

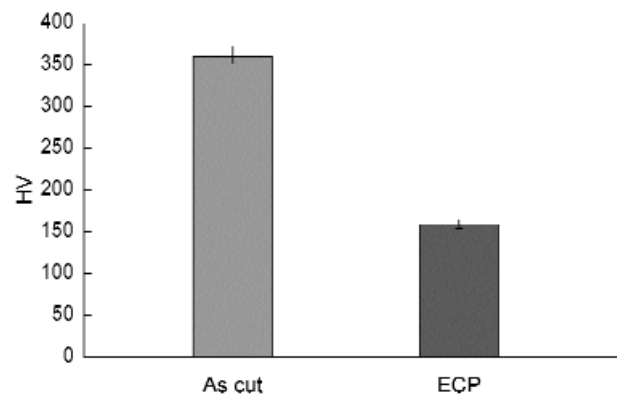


Figure 14. Vickers hardness (HV) of an cut stent and an electrochemically polished stent (ECP).

Discussion

Acid Pickling

The laser cutting process causes slag (burrs and depositions) to appear on the surface of the stents, resulting in a rough surface. Before electrochemical polishing of the stents, as was earlier discussed for electrochemical polishing of NiTi alloy and Ta stents [21], the slag must be removed. The surface quality of the polished stents without a pre-treatment to remove the slag was even worse than that of non-polished stents. The slag

adhered to the surface of the stents after polishing, resulting in a worse surface quality. In the literature [22], it is also mentioned that the burrs and the depositions must be removed after laser cutting in the next production steps. Acid pickling is an effective method for the chemical removal of the surface oxides and other contaminants from metallic materials by immersion in an aqueous acid solution [23]. In this study, acid pickling was employed as a pre-treatment of electrochemical polishing of the 316L stainless steel slotted tube coronary stents. The slag was effectively removed, and thus a clean stent surface was prepared for electrochemical polishing.

Annealing

The original cut stents are made from the stainless steel tubing, which is too rigid to be balloon-expandable. Therefore, the cut stents must be made balloon-expandable before implantation. Annealing is an effective method for softening the stent material. In this study, an annealing treatment was accomplished under vacuum conditions at 1000 °C for 1 h, resulting in balloon-expandable stents, which were verified in a balloon expansion trial. The purpose of using a vacuum environment is to reduce the occurrence of oxidation during the heat treatment. Since the carbon content (0.03% max) of low-carbon austenitic stainless steel, including 316L, is low enough to reduce precipitation of chromium carbides that markedly decrease the resistance to intergranular corrosion, it does not require a quenching treatment [24]. Therefore, in this study, annealing was performed and furnace cooling was applied. In addition, annealing results in the appearance of grain boundary grooves on the stent surface, which increases the surface roughness of the stents compared to the non-annealed (pickled) stent.

Electrochemical Polishing

Electrochemical polishing is a method of brightening and smoothing the surface of metals [14,25] by immersing the parts in an electrolyte and applying positive direct current to the sample. The main electrical parameters for electrochemical polishing are the anodic potential, the anodic current density, and the applied voltage. As the nature and rate of any electrochemical reaction are both determined by the electrode potential, the electrochemical polishing process should also be controlled on the basis of the anodic potential. In practice, the electrochemical polishing process is con-

trolled on the basis of the anodic current density and, in some cases, on the basis of the applied voltage [26]. In this study, applied voltage and anodic current were used as the controlling parameters during the electrochemical polishing process. All the parameters were determined experimentally. As discussed in some literature, electrochemical polishing generally occurs at the limiting current density (a current maximum or plateau in the current voltage curve) [26-29]. The rate of dissolution at the limiting current is controlled by the transport of cationic reaction products from the anode into the electrolyte [29]. Within the limiting-current plateau region, the applied voltage also plays an important role in the resulting surface finishing [28]. The temperature is also a critical parameter for electrochemical polishing. Polishing time is a parameter that influences the removal of material from the stents in case other parameters are fixed.

Material Removal

The strut dimensions of stents can directly determine their mechanical strength after deployment in the vessels, and in turn influence the final healing effects. Therefore, during electrochemical polishing of stents, it is important to control material removal, i.e., in the case of achieving a satisfactory polishing effect, material removal should be as limited as possible. Weight loss and dimension decreases are two parameters for the removal of material. Obviously, the final dimensions of the polished stents are determined from their original dimensions. Therefore, during pickling, a pre-treatment of polishing, overpickling should be avoided. Overpickling, underpickling, and pitting usually are the direct results of lack of control over process variables in the pickling of stainless steel [23]. After selecting a pickling solution, the pickling time, which can be determined experimentally, is a critical parameter for controlling material removal [30]. Additionally, the pickling temperature can have a pronounced influence on the pickling rate. An increase in temperature may cause an increase in the pickling rate [23]. During electrochemical polishing, the current density is not only a determinant of the final effect, but also a measurement of the polishing rate [26-29], which in turn affects the removal of material.

Surface Roughness Measurements

The roughness measurements not only create an image of stent surface status, but also provide a quantitative

surface roughness of the stents. The initial surface state, together with the factors of current density and polishing time, determines the final surface smoothness [26]. Therefore, determining the initial surface roughness demonstrates its importance. Most importantly, the roughness measurements clearly exhibit the final surface texture of a polished stent, which is a measurement that furthers medical and other investigations, i.e., provides an opportunity to investigate quantitatively the influence of roughness on stent biocompatibility.

Conclusion

As concluded in a previous study [15], the sequence of pickling and annealing prior to electrochemical polishing is of importance and should be reversed. The surface quality of 316L stainless steel slotted tube coronary stents is largely improved by means of electrochemical polishing. The surface roughness of electrochemically polished stents is in an acceptable range, which is comparable to commercial stents. Using roughness measurements, combined with material removal measurement, an optimal condition can be determined, under which relatively small roughness and along with large strut dimensions can be obtained. More importantly, roughness measurements not only determine the parameters for electrochemical polishing, but also evaluate precisely the final effectiveness of the electrochemical polishing process. Due to the pretreatment of annealing, the polished stent has a homogeneous microstructure and uniform hardness, and thus has good balloon expandability.

Acknowledgments

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References

- [1] Puel J, Juilliere Y, Bertrand ME, et al. Early and late assessment of sterosis geometry after coronary arterial stenting. *Am J Cardiol.* 1988; 61: 546-553.
- [2] Serruys PW, Strauss BH, Beatt KJ, et al. Angiographic following-up after placement of a self-expanding coronary artery stent. *N Engl J Med.* 1991; 324: 13-17.
- [3] Serruys PW, de Jaegere P, Kiemeneij F, et al. A comparison of balloon-expandable stent implantation with balloon angioplasty in patients with coronary artery disease. *N Engl J Med.* 1994; 331: 489-495.
- [4] Fischman DL, Leon MB, Baim DS, et al. A randomised comparison of coronary stent placement and balloon angioplasty in treatment of coronary artery disease. *N Engl J Med.* 1994; 331: 496-501.
- [5] Wang K. Biocompatibilisation of Coronary Artery Stents (Acta Biomedica Lovaniensia , No 162). Leuven: Leuven University Press. 1997: 89-102.
- [6] Steinemann SG. Metal implants and surface reactions. *Injury.* 1996; 27(Suppl 3): SC16-SC21.
- [7] De Scheerder I, Verbeken E, van Humbeeck J. Metallic surface modification. *Semin Intervent Cardiol.* 1998; 3: 139-144.
- [8] De Scheerder I, Sohier J, Wang K, et al. Metallic surface treatment using electrochemical polishing decreases thrombogenicity and neointimal hyperplasia of coronary stents. *Int J Cardiol.* 2000; 13: 179-185.
- [9] De Scheerder I, Sohier J, Verbeken E, et al. Biocompatibility of coronary stent materials: Effect of electrochemical polishing. *Materialwiss Werkstofftech.* 2001; 32: 142-148.
- [10] De Scheerder I, Strauss BH, de Feyter PJ, et al. Stenting of venous bypass grafts: A new treatment modality for patients who are poor candidates for reintervention. *Am Heart J.* 1992; 123: 1046-1054.
- [11] De Scheerder I, Wang K, Kerdsinchai P. Clinical and angiographic experience with coronary stenting using the Freedom Stent. *J Invas Cardiol.* 1996; 8: 418-427.
- [12] Strauss B. H, Serruys PW, De Scheerder I, et al. Relative risk analysis of angiographic predictors of restenosis within the coronary Wallstent. *Circulation.* 1991; 84: 1636-1643.
- [13] Vidal R, West AC. Copper electropolishing in concentrated phosphoric acid. *J Electrochem Soc.* 1995; 142: 2682-2694.
- [14] Magaino S, Matlosz M, Landolt D. An impedance study of stainless steel electropolishing. *J Electrochem Soc.* 1993; 140: 1365-1373.
- [15] Zhao H, Van Humbeeck J, Sohier J, et al. Electrochemical polishing of 316L stainless steel slotted tube coronary stents. *J Material Science: Materials in Medicine.* 2002; 13: 911-916.
- [16] Assessment of Surface Texture, part 1, British standard 1134. London: British Standard Institute. 1972.
- [17] Baro AM, Garcia N, Miranda R, et al. Characterization of surface roughness in titanium dental implants measured with scanning tunnelling microscopy at atmospheric pressure. *Biomaterials.* 1986; 7: 463-466.
- [18] DIN EN ISO 4287, ASME B46.1. Berlin: Deutsches Institut für Normung.
- [19] DIN EN ISO 11562, A.B. Berlin: Deutsches Institut für Normung.

- [20] DIN EN ISO 4288, ASME B46.1. Berlin: Deutsches Institut für Normung.
- [21] Zhao H, Van Humbeeck J, De Scheerder I. Surface conditioning of NiTi and Ta stents. *Prog Biomed Res.* 2001; 6: 439-448.
- [22] Momma C, Knop U, Nolte S. Laser cutting of slotted tube coronary stents: State-of-the-art and future developments. *Prog Biomed Res.* 1999; 4: 39-44.
- [23] Hudson RM, Joniec RJ, Shatynski SR. Pickling of Iron and steel. In: Cubberly WH, et al. (editors). *Metals Handbook, Volume 5, 9th ed.* Metals Park, Ohio: American Society for Metals. 1982: 68-82.
- [24] Rapoport D, Byrnes ER, Crooks KL, et al. Heat treating of stainless steels. In: Alban LE, et al (editors). *Metals Handbook, Volume 4, 9th ed.* Metals Park, Ohio: American Society for Metals. 1981: 623-646.
- [25] Mills K, Davis TR, Destdfani JD, et al (editors). *Metals Handbook, Volume 9, 9th ed.* Metals Park, Ohio: American Society for Metal. 1985: 48.
- [26] Shigolev PV. *Electrolytic and Chemical Polishing of Metals, Revised 2nd ed.* Tel Aviv: Freund Publishing House. 1974: 22-36.
- [27] Mathieu JB, Mathieu HJ, Landolt D. Electropolishing of titanium in perchloric acid-acetic acid solution. *J Electrochem Soc.* 1978; 125: 1039-1043.
- [28] Piotrowski O, Madore C, Landolt D. Electropolishing of titanium and titanium alloys in perchloric-free electrolytes. *Plating and Surface Finishing.* 1998; 85: 115-119.
- [29] Piotrowski O, Madore C, Landolt D. Electropolishing of tantalum in sulfuric acid-methanol electrolytes. *Electrochimica Acta.* 1999; 44: 3389-3399.
- [30] Bauccio M L. Technical note 9: Descaling and special surface treatments. In: Boyer R, et al. (editors). *Materials Properties Handbook: Titanium Alloys.* Metals Park, Ohio: American Society for Metals, 1994: 1145.

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