JOURNAL OF MATERIALS SCIENCE. MATERIALS IN MEDICINE 5 (1994) 434–440

Methods to characterize the surface roughness of metallic implants

A. UNGERSBÖCK, B. RAHN
AO Research Institute, CH-7270 Davos Platz, Switzerland

The surface roughness of surgical implants has a significant influence on the tissue reaction at the interface. The purpose of the present study is to describe methods which allow a detailed characterization of the surface roughness. Pure titanium plates with different surface treatments and electropolished stainless steel plates, were analysed. For the surface roughness measurement, a profilometer with a 4 μm tip was used to determine the following roughness parameters: R_a = arithmetic mean of the roughness height, S_m = arithmetic mean of the groove distance, R_{tm} = average of the roughness height. Furthermore the surface was observed under a scanning electron microscope (SEM) at a magnification of 1000× and 2000× so that even small pores which cannot be measured using the profilometer are detectable. Standard reflected light microscopy and interference contrast microscopy was used to optically measure the height of depressions and elevations, and to study in particular the surface colour, which is related to the thickness of the oxide film of anodized implants. It is concluded that, for the characterization of the surface roughness of metallic implants, the measurements using the profilometer and SEM are recommended. For anodized surface treatments, interference contrast microscopy seems to be a valuable aid to judgement of the homogeneity and, via colour, the thickness of the oxide layer.

1. Introduction

The thickness, structure and cellular composition of the soft tissue layer covering an implant depend on the biocompatibility of the material, and the roughness and chemistry of the implant surface [1–7]. This paper focuses on the detailed description of the surface roughness using adequate methods.

The roughness of the surface is a factor which can be expected to play a dominant role with regard to soft tissue adhesion [3, 8, 9]. Various strategies have been devised to improve the interfacial attachment. Ingrowth of tissue into porous material to achieve mechanical interlocking has been widely investigated [10]. Optimum pore sizes compatible with osteon formation, the type of pore interconnectivity needed, and appropriate porous coating thicknesses have been identified [11]. Microscopic surface inhomogeneities, such as grain boundaries or second phase particles, could create a situation where protein adsorption occurs nonuniformly, and heterogeneous adsorption of protein on metal has been reported [7].

In experimental animal studies to test different surface treatments it was observed that a detailed description of the surface roughness is a precondition. This description should give detailed, objective, measurable data of the surface roughness. This should allow a controlled change of one or more roughness parameters and correlation with the soft tissue reaction. For this purpose the profilometer technique was applied for roughness measurement. Furthermore, all implant surfaces were observed under scanning electron microscope to obtain three-dimensional information. Interference contrast microscopy was used to optically measure the height of depressions and elevations, and to study, in particular, the surface colour, which is related to the thickness of the oxide film of anodized implants.

2. Materials and methods

Five different surface treatments of specimens made from commercially pure titanium (cp Ti) and electropolished stainless steel (SS) plates were tested (Table I). The following surface conditions were used:

(a) Titanium plate anodized rough: these specimens were blasted and anodized according to a treatment used for fracture fixation plates in clinics* (interference colour, dark gold).
(b) Titanium plate anodized fine: the plates were tumbled and anodized according to the current protocol of anodization (interference colour gold).
(c) Titanium plate handground: the plates were handground, without further surface treatments, a semi-rough structure oriented in one direction resulted.
(d) Titanium plates Al_2O_3 blasted: these plates were blasted with Al_2O_3, a quite rough surface resulted.
(e) Titanium plates electropolished: these plates

*as commercially used by Stratec Medical, Waldenburg, Switzerland

0957–4530

TABLE I Classification of implant material and surface condition. The main steps concerning the surface preparation are described as well as a short-hand designation

Implant material	Surface condition	Short-hand designation
Titanium c.p.	Tumbled before anodization	Ti anod. fine
Titanium c.p.	Blasted before anodization	Ti anod. rough
Titanium c.p.	Tumbled	Ti tumbled
Titanium c.p.	Handground	Ti handground
Titanium c.p.	Al_2O_3 blasted	Ti blasted
Titanium c.p.	Electropolished	Ti electropol.
Implant steel	Electropolished	SS electropol.

underwent an electropolishing surface treatment, resulting in a clean and smooth surface.

(f) Stainless steel plates electropolished: these plates underwent a routine electropolishing surface treatment, resulting in a clean and smooth surface.

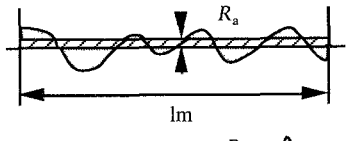
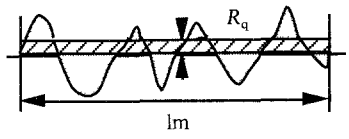
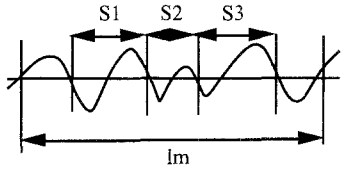
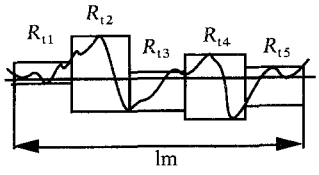
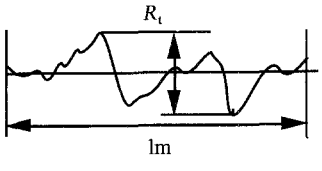
The specimens had the following dimension: length 35 mm, width 5 mm, thickness 1 mm. The roughness measurement was made on a profilometer type Taylor Hobson with a four-sided pyramidic tip of 4 μm diameter. The implants were examined using a scanning electron microscope (SEM) type Cambridge 604. The interference contrast microscopy was performed on a Zeiss Axiomat using reflected light.

Three randomly chosen original packed plates of each group were measured on the surface which is *in situ* in contact with the soft tissue. During the whole measurement cotton gloves were used to prevent the surface from scratches and direct skin contact. No additional cleaning was performed to avoid any surface changes. Five measurements perpendicular to the long axis [12] were made. Dependent on the implant dimension and the types of surface treatments, a sampling length of 1.3 mm and a cut-off of 0.25 mm was chosen [12]. The roughness parameters, their definition and a graphic illustration are shown in Table II. To guarantee the measurement of a representative part of the surface, the five measurements were equally distributed along the long axis, since no heterogeneity could be observed in the previous screening under the light microscope. The 15 measurements (three plates, five measurements per plate) for each type of surface was sufficient since the measurement data were quite uniform and no change of the standard deviation could be observed after 10 measurements.

For the scanning electron microscopy (SEM) the samples were oriented at 45° to the long axis, and with the transverse axis parallel to the beam. Under these standard conditions three samples of each group were examined. During the SEM examination attention was paid to avoid any surface contamination.

The interference contrast microscopy was carried out using a 50 × Epiplanapo lens. Using a circular

TABLE II Summary of the definition, the international standards and a graphic illustration of the roughness parameters applied in this study using the profilometer measurement

Roughness parameter	Standard	Definition	Graphic illustration
R_a	DIN 4768/1 ISO 4287/1	Arithmetic mean of the roughness height	
R_q	DIN 4762/1 ISO 4287/1	Root mean square of the roughness height	
S_m	DIN 4762/1 ISO 4287/1	Arithmetic mean of the groove distance	
R_{tm}	DIN 4768/1	Average of five consecutive values of roughness height	
R_t	DIN 4762/1 ISO 4287/1	Maximum roughness height between a peak and valley for sampling length (lm)	

grid a distance measurement was possible, whereby the smallest circle corresponded to the diameter of the profilometer tip (Fig. 3a). The use of the focusing micrometer screw allows a depth/height measurement, thanks to the very shallow depth of field of the interference contrast microscopy technique.

Using the Newman–Keuls multiple range test (variance analysis), the significance of the roughness parameters ($p \leqslant 0.05$) was calculated.

3. Results

In general it can be stated that the roughness is relatively low for all the plates except the blasted surfaces. The three techniques used for the assessment of surface roughness produced comparable results, and each technique presented certain advantages and thus helped in interpretation of the findings obtained by the other techniques. In particular, the profilometer technique profited from the parallel use of morphologic techniques.

The profilometrically measured surface roughnesses gave characteristic patterns for the different surface treatments (Table III, Fig. 1a–c, Fig. 3c and Fig. 5c). The results of the five measurements on each plate and the comparison of the three plates of the same group show very uniform and constant results, indicated by a low standard deviation. The S_m values for "Ti tumbled" and "handground" are significantly lower than for the other surface treatments. The difference in the S_m results between "Ti anodized fine" and "Ti anodized rough" is also significant. The S_m results for "Ti blasted", "Ti electropolished" and "SS electropolished" are not significantly different. Concerning the R_{tm} results; the very low standard deviation is obvious. All surface treatment differences, except "Ti anodized fine" versus "handground", are statistically significant for the R_{tm} values when compared to each other. The R_a values are low for all types of surface treatments, except "Ti rough" and "Ti blasted". The results for "Ti tumbled", "Ti electropolished" and "SS electropolished" are not significantly different, whereas all other surface treatments show significant results when compared.

Scanning electron microscopy (Figs 2b and 5b) was of special value in depicting rough surfaces, especially deep and narrow indentations. Very small pores and structure inhomogeneities immediately below the surface could not be easily detected with the optical microscope or the profilometer. For softer surface contours, however, the SEM image revealed a topography which was compatible with profilometric analysis.

Reflected light microscopy (Figs 4a and 5a) included colour as an important carrier of information. Depending on the thickness of the oxide layer, titanium surfaces appear in different colours. Different anodizing protocols may influence thickness, density and homogeneity of the oxide layer and thus the colour of the surface. In the anodized specimens such inhomogeneities could be detected. While the general aspect of these samples was a metallic yellow, areas 20–50 μm wide could be seen in which the metallic grey of titanium was visible (Fig. 4a). Usually these areas were delimited by metal grain boundaries; occasionally single grains showed a fine yellow-grey striation. No correlation of these aspects was found in the other two evaluation techniques. The shallow depth of field of the interference contrast technique, on the one hand, has limitations in visualizing rough surfaces, since it is not possible to focus all the structures at the same time (Fig. 5a). On the other hand, this shallow depth of fields allowed for optical measurement of elevations and depressions (R_t). The results of these measurements correspond to the data obtained by profilometry. By means of the grid in the eyepiece the average groove distance (S_m) of the profilometric analyses could be confirmed.

4. Discussion

A detailed description of the surface characteristics seems to be a precondition for the interpretation of biocompatibility studies of implants. Roughness of the surface is one of the factors which can be expected to play a dominant role in the soft tissue reaction at the interface. The use of profilometric surface characterization, reflected light interference contrast microscopy, and scanning electron microscopy helps to characterize implant surface topography and its dimensional relationship to interfacing cells. While the general findings for all three techniques go in parallel, each of the techniques has strengths and weaknesses. Thus, the supplementing information obtained from all three techniques is important for the interpretation of results.

The techniques of surface roughness measurement can be divided into two main groups, contacting (stylus) methods [12, 13] and non-contacting methods [14–16]. The latter are the more recent, but support by international standards is still pending and so far they are not widely used. We chose the more common

TABLE III Results (mean value ± standard deviation) of all roughness parameters for each type of surface treatment

Types of plates	$R_a \pm SD$	$R_q \pm SD$	$S_m \pm SD$	$R_{tm} \pm SD$	$R_t \pm SD$
Ti anodized fine	0.33 ± 0.06	0.41 ± 0.07	43 ± 8.2	1.7 ± 0.2	2.3 ± 0.3
Ti anodized rough	0.75 ± 0.19	1.01 ± 0.09	61 ± 16	4.2 ± 0.42	5.6 ± 0.7
Ti tumbled	0.15 ± 0.01	0.19 ± 0.03	19 ± 2.2	1.1 ± 0.1	1.4 ± 0.2
Ti handground	0.23 ± 0.01	0.30 ± 0.02	19 ± 1.3	1.7 ± 0.17	2.3 ± 0.5
Ti Al_2O_3 blasted	1.50 ± 0.2	1.90 ± 0.30	42 ± 7.6	8.1 ± 1.3	11 ± 1.9
Ti electropolished	0.16 ± 0.02	0.22 ± 0.04	52 ± 6.7	0.88 ± 0.08	1.3 ± 0.3
SS electropolished	0.08 ± 0.02	0.11 ± 0.05	48 ± 11	0.52 ± 0.15	1.04 ± 0.6

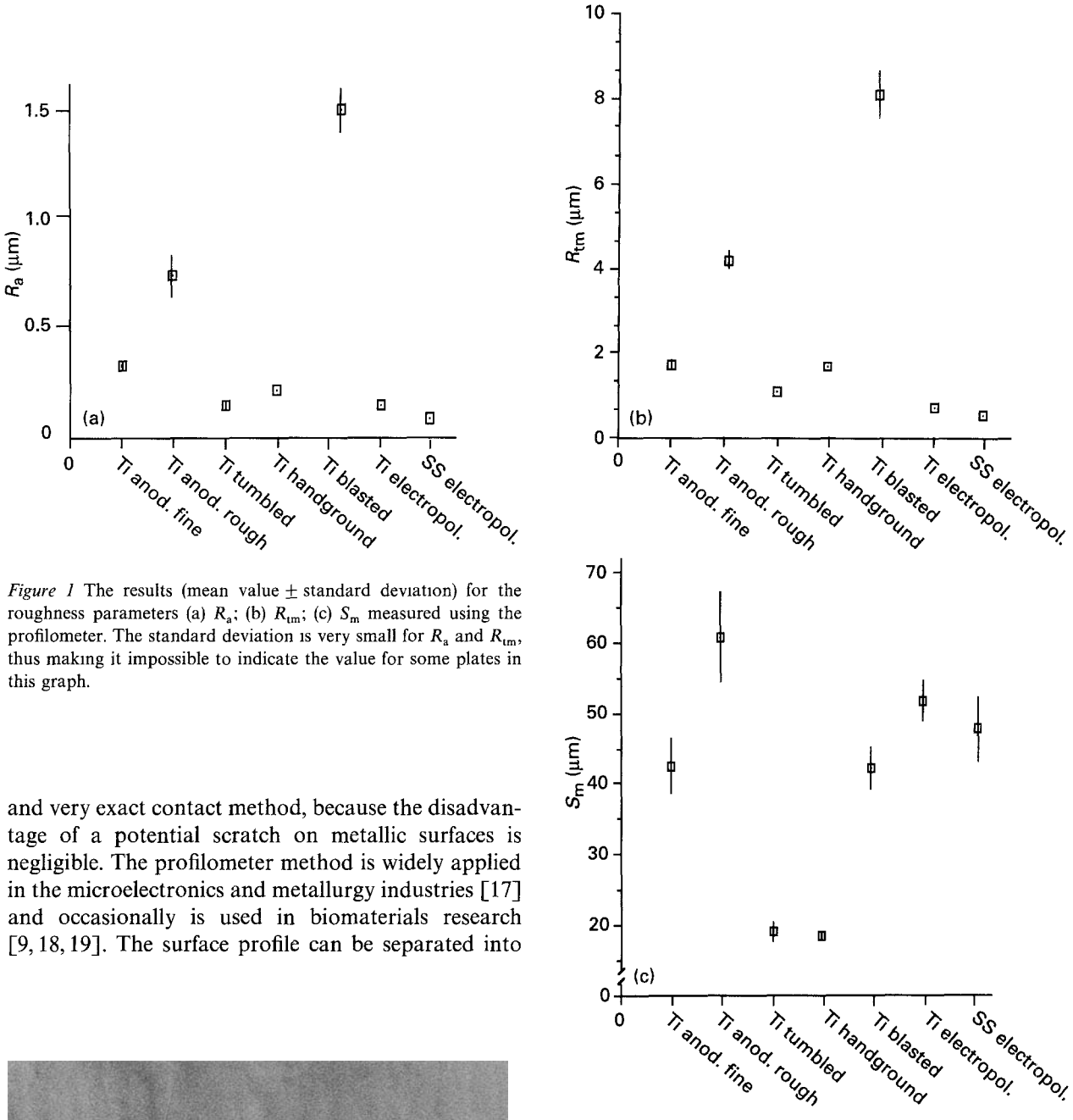


Figure 1 The results (mean value ± standard deviation) for the roughness parameters (a) R_a; (b) R_{tm}; (c) S_m measured using the profilometer. The standard deviation is very small for R_a and R_{tm}, thus making it impossible to indicate the value for some plates in this graph.

and very exact contact method, because the disadvantage of a potential scratch on metallic surfaces is negligible. The profilometer method is widely applied in the microelectronics and metallurgy industries [17] and occasionally is used in biomaterials research [9, 18, 19]. The surface profile can be separated into

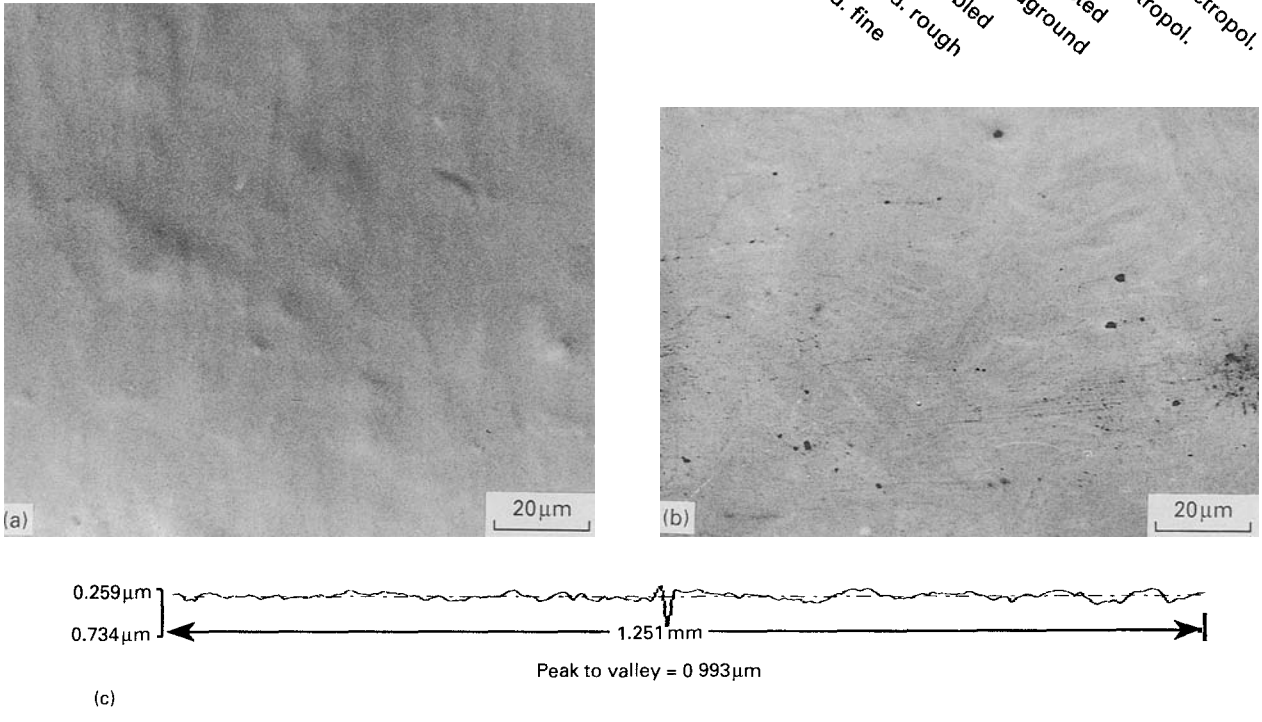


Figure 2 Stainless steel, electropolished: (a) reflected light interference contrast microscopy; (b) SEM; (c) profilometer. These steel specimens present the smoothest of all the investigated surfaces.

roughness (high frequency component) and waviness and form (low frequency components). The surface parameters measured in this study describe only the roughness component. The difference between waviness and roughness is essentially one of scale. The lower frequency components can be eliminated using the correct cut-off length which should be chosen according to the surface dimensions, shape and surface treatment [12].

The chosen roughness parameters describe different details of the surface profile. R_a (arithmetic mean of

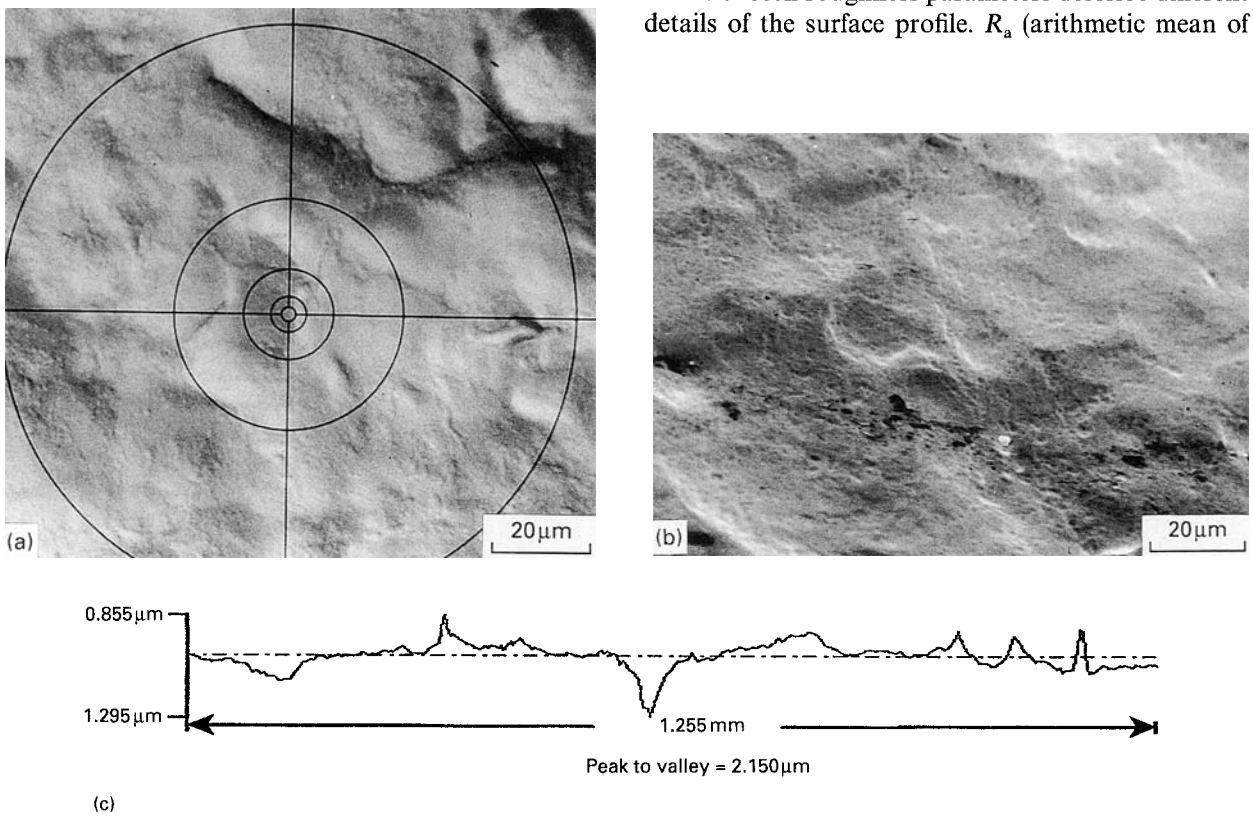


Figure 3 Titanium, electropolished: (a) reflected light interference contrast microscopy and (b) SEM give similar representations of the surface. The profile (c) shows the wavy aspect of the surface. The size of the smallest, innermost circle in Fig. 3a corresponds to the size of the tip of the profilometer.

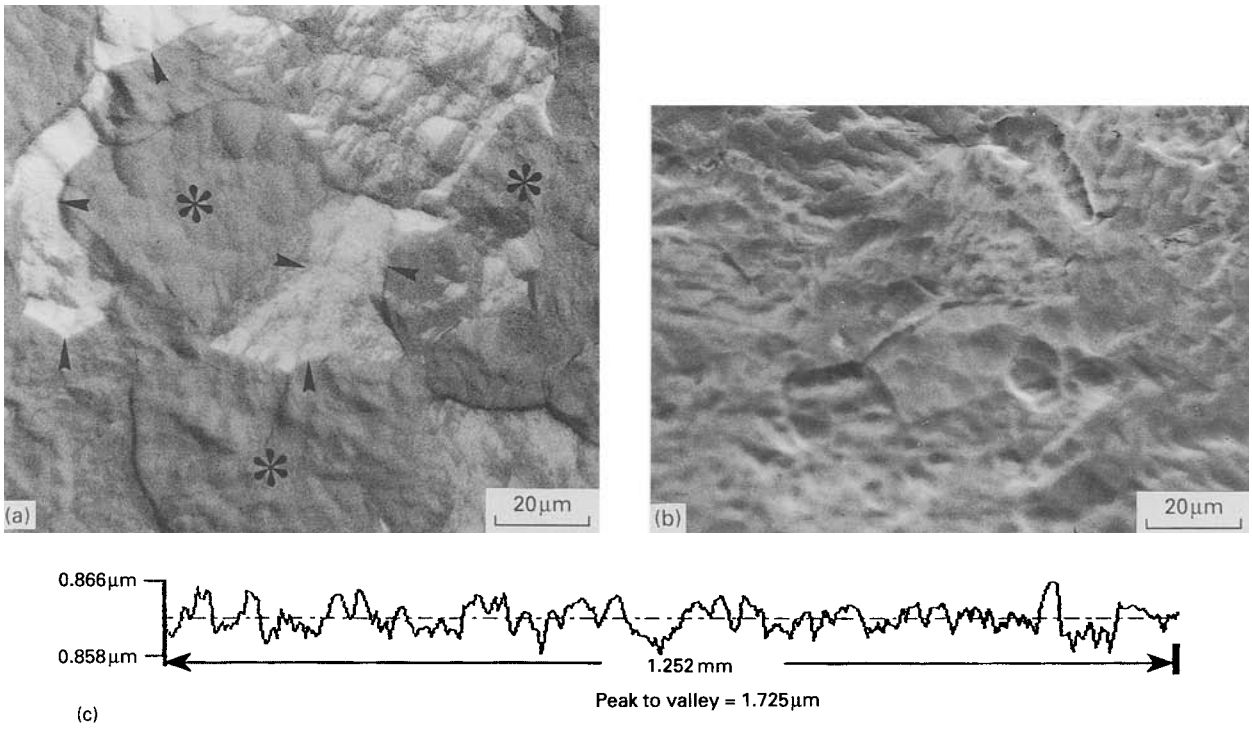


Figure 4 Titanium, anodized fine: (a) reflected light interference microscopy; (b) SEM; (c) profilometry. Reflected light interference contrast microscopy allows for the assessment of colour differences. In this case most of the area is covered by an oxide layer showing the interference colour of yellow (asterisks, darker area in the micrograph), while in other areas, usually delimited by grain boundaries, the oxide layer shows an interference colour of grey (arrow heads).

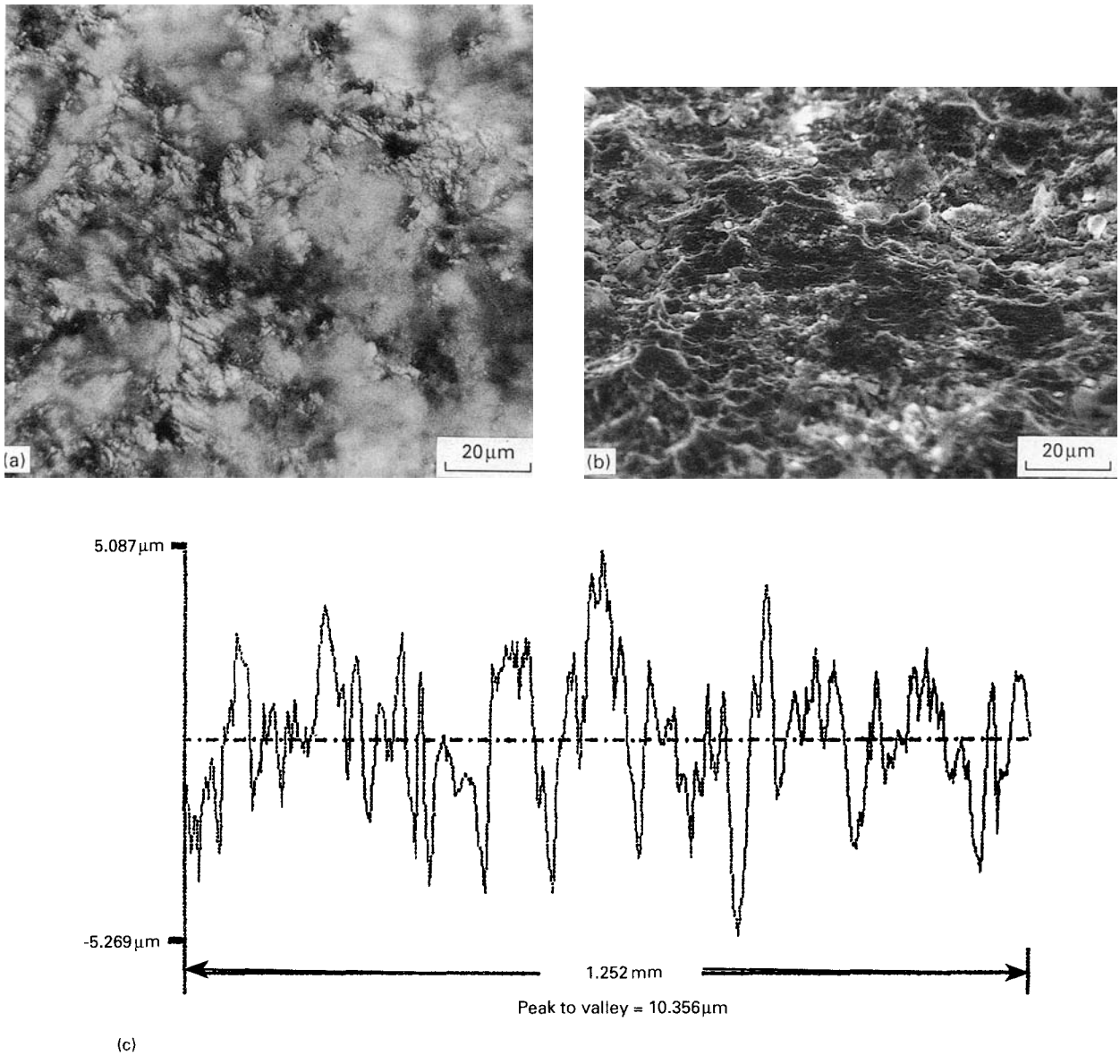


Figure 5 Titanium, blasted: (a) reflected light interference contrast microscopy; (b) SEM; and (c) profilometry. These specimens present the roughest of all the investigated surfaces.

the roughness height) summarizes the roughness height, it gives no information at all on the roughness profile. Different types of profile can result in the same R_a value. For this purpose, S_m (arithmetic mean of the groove distance) and R_{tm} (average of five consecutive values of roughness height) values should be measured. A low value for S_m and a high value for R_{tm} can result in the same R_a value and vice versa.

If we transfer the roughness measurements data to biological cell dimensions (fibroblasts, fibrocytes: longitudinal diameter 20–40 μm, transversal diameter 10–15 μm), we find S_m values in the range of two longitudinal cell diameters. The R_{tm} value is in the range of 20–50% of a transversal cell diameter. Clinical and experimental studies have shown that a thin connective tissue layer, mainly composed of fibroblasts and fibrocytes oriented parallel to the surface, covers the implant [20–22]. The biological effect of different values for S_m and R_{tm} needs to be studied.

Scanning electron microscopy (SEM) has to be used for resolution in the micrometre and submicrometre range. Samples for the SEM must be able to resist a vacuum environment and must be conductive. Both conditions are met by metallic implants; a modification of the surface by preparation techniques therefore is no issue. SEM is a valuable method to visualize different aspects of the surface structure and it allows stereo imaging techniques as well as image analysis.

Interference contrast microscopy requires no special preparation techniques. The additional colour information is of special interest for anodized surfaces. The observed colour inhomogeneities seem to be related to variations in oxide thickness. Possible reasons for such inhomogeneities could be minor variations in the anodizing protocol (e.g. time, voltage). At locations of interest a detailed examination of the composition and thickness of the oxide layer should be done using spectroscopic techniques [23, 24].

In addition, interference contrast microscopy allows to perform dimensional measurements of the height of peaks and depth of valleys as well as the groove distances, values which are comparable to profil-

ometer measurements (R_t, S_m). The results obtained with both techniques are almost identical.

5. Conclusions

In conclusion, it is suggested that a profilometer be used as a routine measuring technique giving reproducible quantitative data. This technique, however, should not be used as a complete standalone method, since other aspects invisible to the probe of the profilometer could influence the mechanical interface behaviour of a certain surface roughness. It is clear that surface morphology, chemical composition, surface charge, and other characteristics, are some of the parameters influencing the interface behaviour. The correlation of visual aspects to the more abstract profilometric data seems to be a useful supplement which helps in interpretation of reactions at interfaces.

Acknowledgements

The authors would like to express their gratitude to Professor Adolf Wirtz and Mr Rolf Businger of the Ingenieurschule Neutechnikum Buchs, department of Feinmesstechnik. This work was supported by the AO/ASIF foundation.

References

1. T P RUEDI, *Hefte Unfallheilkunde* **123** (1975) 1.
2. P. G. LAING, in "Tissue reaction to biomaterials" (National Bureau of Standards, Special Publication, Washington DC 1977) p. 31.
3. B. A. RAHN, V. GERET, C. CAPAUL, M. LARDI and B. SOLOTHURNMANN, in "Clinical Applications of Biomaterials", edited by A. J. C. Lee, T. Albrektson, P. J. Branemark (Wiley, Chichester, 1982) p. 263.
4. T N. SALTHOUSE and B F. MATLAGA, in "Biomaterials in reconstructive surgery", edited by L. R. Rubin (Mosby, St. Lous, 1983) p. 40.
5. T. ALBREKTSSON, *CRB Crit. Rev. Biocomp.* **1** (1985) 53.
6. S. M. PERREN and O. POHLER, *AO/ASIF Dialogue* **1** (1987) 11.
7. R. L. WILLIAMS, D F. WILLIAMS, *J. Biomed. Mater. Res.* **23** (1989) 339.
8. E. J ANDREWS, P. W. TODD and N. E. KUKULINSKI, *ibid.* **13** (1979) 173.
9. B. A. RAHN, H. W. GERBER, J. SIMPSON, F. STRAUMANN and S. M. PERREN, in "Biomaterials", edited by G. D. Winter, D. F. Gibbons, H. Plenk, Jr (Wiley, Chichester, 1980) p. 39.
10. J. D. BOBYN, C. A. ENGTH and R. M. PILLIAR, in "Quantitative Characterization and Performance of Porous Implants for Hard Tissue Applications", edited by J. E. Lemons (ASTM STP 953, Phil., 1987) p. 185.
11. J. C. KELLER and F. A. YOUNG, in "Quantitative Characterization and Performance of Porous Implants for Hard Tissue Applications", edited by J. E. Lemons (ASTM, STP 953, Phil. 1987) p. 219.
12. K. J STOUT, *Mater. Engng.* **2** (1981) 260.
13. B. D. RATNER, A. B. JOHNSTON and T. J. LENK, *J. Biomed. Mater. Res.* **21** (1987) 59.
14. J. M. BENNET, *Appl. Opt.* **15** (1976) 2705.
15. M. RASIGNI, G. RASIGNI, J. P. PALMARI and A. LLEBARIA, *J. Opt. Soc. Amer.* **71** (1981) 1124.
16. J. A. SCHMID and J. BLACK, *Biomaterials* **13** (1992) 483.
17. D J. WHITEHOUSE, in "Stylus techniques, characterization of solid surfaces", edited by P. F. Kane and G. R. Larrabee (Plenum Press, New York, 1974) p. 49.
18. B. D RATNER, *J. Biomed. Mater. Res.* **14** (1980) 665.
19. J. F. HECKER and R. O EDWARDS, *ibid.* **15** (1981) 1.
20. J. A. JANSEN, J. P. C. M. VAN DER WAERDEN and K DE GROOT, *J. Mater. Sci.* (1990) 192.
21. C. B. JOHANSSON, T. ALBREKTSSON, L. E. ERICSON and P THOMSEN, *ibid.* **3** (1992) 126.
22. A. UNGERSBOECK, V. GERET, M. SCHÜTZ, O. POHLER and S M. PERREN, in Proceedings of the Fourth World Biomaterials Congress, Berlin, 1992.
23. J. E. SUNDGREN, P. BODOE and I. LUNDSTROEM, *J. Colloid Interface Sci.* **110** (1986) 9.
24. J. LAUSMAA, Thesis for Physics University of Göteborg (1991).