Color evaluation of a dielectric mirror coating using porcine tissue and prosthetic gingival material: a comparison of two models

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Abstract

The aim of this study was to firstly evaluate the esthetics of a dielectric multilayer coating on titanium below porcine tissue (in vitro porcine model). Secondly, a polymer model was used to investigate the same samples to compare the models to each other and discuss their validity for optical assessment of esthetic coatings for implant applications.

Reference


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Color evaluation of a dielectric mirror coating using porcine tissue and prosthetic gingival material: a comparison of two models

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Abstract
Aim: The aim of this study was to firstly evaluate the esthetics of a dielectric multilayer coating on titanium below porcine tissue (in vitro porcine model). Secondly, a polymer model was used to investigate the same samples to compare the models to each other and discuss their validity for optical assessment of esthetic coatings for implant applications.

Materials & Methods: A dielectric mirror coating was deposited on titanium substrates (Ti-Bragg) and tested below porcine tissue and polymer platelets of three test thicknesses (1 mm, 2 mm and 3 mm). Titanium without coating (Ti) was used as a negative control. Furthermore, the substrates were subjected to three different surface treatments (polished, machined and sand-blasted). The color difference values \( \Delta L, \Delta a, \Delta b \) and \( \Delta E \) were calculated for each sample. In total, six samples were tested in both models. Statistical analysis of the data (one sample Wilcoxon test, Kruskal-Wallis with Bonferroni-Holm corrected Mann-Whitney post hoc tests for multiple testing) was conducted for each sample in both evaluation methods.

Results: In the in vitro porcine model, sand-blasted Ti-Bragg and Ti samples showed \( \Delta E \) values significantly above the threshold value of 3.70, indicating a dark appearance of the 1 mm thick covering tissue. As the test thickness increased, polished and machined Ti-Bragg samples were significantly invisible (\( \Delta E < 3.70 \) with \( P < 0.05 \)). Excessive brightening effects from Ti-Bragg samples were not observed from the in vitro porcine model, but below polymer platelets the samples exhibited significantly high \( \Delta L \) values, which also resulted in a significant and visible color change (\( \Delta E > 3.70 \) with \( P < 0.05 \)).

Conclusion: Ti-Bragg was suggested to be an appropriate coating system for dental implants to improve the soft tissue esthetics. The design of this coating system can be adjusted by varying different parameters to satisfy the requirements of an esthetic coating. The polymer model is valid for test thicknesses of 2 and 3 mm, however, one might have to increase the thickness to 1.5 mm or alter the composition of the resin for 1 mm thick platelets to increase the opacity and therefore adapt to the soft tissue situation.

In the past few decades, titanium [Ti] implants and abutments have been used successfully in reconstructive dentistry, accommodating the physical as well as biocompatible requirements in this field (Bränemark et al. 1977; Scheller et al. 1998; Buser et al. 2012). However, other reconstruction materials are taken into consideration when it comes to the esthetic outcomes in the peri-implant region. Studies showed that Ti can cause dark discoloration effects of the soft tissue, whereas ceramics, such as zirconia \( \text{(ZrO}_2 \)\), achieve a better and natural appearance of the gingiva (Heydecke et al. 2005, Jung et al. 2007, Watkin & Kerstein 2008). Nevertheless, insufficient mechanical properties decrease the reliability of this dental ceramic during service (Wenz et al. 2008; Depprich et al. 2014). In Pecnik et al. (2014), the authors created dental coatings by combining both metallic and ceramic materials and showed that soft tissue esthetics were improved significantly, compared to the substrate material Ti. In vitro tests with porcine tissue displayed that Ti platelets coated with \( \text{ZrO}_2 \) thin films and intermediate metallic layers [Al or Ag] were nearly invisible below 1 mm thin test tissue. However, light brightening effects were still observed for the coating system Ti-Ag-ZrO\textsubscript{2}, which could still be visible and compromise the patient’s appraisal of the treatment. Furthermore, if the mechanical integrity of the \( \text{ZrO}_2 \) layer would be compromised, the coating could...
potentially corrode as Al and Ag dissolve in saliva environment (de Mele & Dauro 2002).

Another design strategy was developed which aims the properties of an esthetic dental coating [Pecnik et al. 2014]. Dielectric mirrors or Bragg reflectors, which are used extensively in optical systems such as in interferometers or in lasers as optical resonators, can reach very high reflectance values due to their multilayer structure of alternate thin films of high and low refractive index (Macleod 2001). If the optical thicknesses ($t$) are chosen appropriately, the reflected beams interfere constructively from all the interfaces. Titanium dioxide ($\text{TiO}_2$) and silicon dioxide ($\text{SiO}_2$) are suitable materials as the difference in refractive index ($n$) is large [$\Delta n = n_{\text{TiO}_2} - n_{\text{SiO}_2} = 2.60 - 1.54 = 1.06$] (Macleod 2001). This refractive index contrast and the number of layer pairs can be adjusted to receive a dielectric mirror with high reflectance over a defined optical bandwidth (wideband reflector). Therefore, the Bragg reflector classifies itself as a candidate for esthetic dental coating: It might assure a natural appearance of the peri-implant mucosa, without darkening or brightening the tissue. Moreover, the use of these oxides would promise a higher corrosion resistance, even if the coating would mechanically fail during the application. The color of this new coating system and its behavior below soft tissue should be evaluated according to previous in vitro studies made by Jung et al. (2007) and Pecnik et al. (2014). It is anticipated that the Bragg coating developed in this study might reach optimum color differences below the threshold of visual distinction between colors ($\Delta E < 3.70$) (Johnston & Kao 1989).

In vitro assessments based on porcine tissue are valid models and similar to the in vivo situation for human gingiva. Various measurements of porcine tissue, for example, permeability and composition of the tissue, were in agreement with the properties of human tissue (Lesch et al. 1989, Kuytz et al. 2001; van Eyk & van der Bijl 2004). However, for color evaluations, the implementation of in vitro models with pig maxillae is a time-consuming procedure, which includes careful selection and precise preparation of the pig maxillae. Furthermore, the condition of the porcine tissue changes with time, which decreases the repeated use of the same maxillae for in vitro measurements to a daily limit. Alternative models that would be applicable at any time would facilitate the evaluation of the color and overall esthetics of potential dental coatings. Prosthetic gingival materials would be suitable for this purpose as they are often used to reestablish the gingival esthetics, especially in cases where surgical intervention is unpredictable or impossible (Barzilay & Irene 2003; Coachman et al. 2009; Alani et al. 2011). Commonly, acrylic resins are picked to fabricate customized gingival prostheses as they are low-cost, durable, and removable (Lai et al. 2003; Ajita et al. 2012). Platelets made of acrylic polymer would adopt the task of porcine tissue in vitro color evaluations with the advantage that they maintain their shape, structure, and color. These parameters would then be constant for every spectrophotometric measurement and not depend on fluctuations of the test tissue. However, there are no reports on the application and validity of a polymer model for discoloration evaluations of the peri-implant mucosa.

This study investigated primarily the esthetic appearance of a dielectric mirror coating deposited on Ti substrates of different rough surfaces below porcine tissue. The potential of this coating for the future application was discussed afterward. The second color evaluation, where polymeric platelets were used instead of porcine tissue, was compared to the already established pig model and its applicability for color screenings discussed.

Material and methods

Sample preparation and coating technique

Ti (Grade 4, ThyssenKrupp, Essen, Germany) platelets were used as a substrate material. Uncoated Ti samples were used as a negative control. Each platelet exhibited a diameter of 15 mm and thickness of 0.5 mm. Three different surface modifications of the samples were performed: polished (p), machined (m), and sandblasted (s) surfaces. The average roughness values $S_a$ of the platelets were $S_a = 0.005 \mu m$ for polished substrates, $S_a = 0.099 \mu m$ for machined substrates, and $S_a = 0.247 \mu m$ for sandblasted substrates. The modification procedure was adopted from the previous study (Pecnik et al. 2014).

The design of the wideband reflector coating [Ti-Bragg] is illustrated in Fig. 1. The multilayer coating consisted of two stacks of alternating TiO$_2$ and SiO$_2$ layers with different thicknesses. Each stack is characterized by a certain design wavelength, which is defined as

$$\lambda_d = 4 \cdot \tau = 4 \cdot n \cdot d$$

with $\tau$ as the optical thickness, $n$ as the refractive index, and $d$ as the physical thickness of the film. The design wavelengths of the stacks were $\lambda_1 = 480 nm$ with four repeating units of TiO$_2$/SiO$_2$ layer pairs and $\lambda_2 = 650 nm$ with six repeating units of TiO$_2$/SiO$_2$ layer pairs. The coating was applied by reactive magnetron sputtering [PVD Products Inc., Wilmington (MA), USA] (George 1992) on Ti substrates. Si ($99.999\%$, MaTeck GmbH, Germany) and Ti ($99.9\%$, MaTeck GmbH, Germany) targets were used, from which Si and Ti atoms were emitted and later reacted with oxygen ($O_2$) to condensate as oxides on the substrates. The deposition was carried out at room temperature, and the base pressure in the deposition chamber was lower than $1.1 \times 10^{-6}$ torr. The thickness of each SiO$_2$ layer was 78 nm for the first stack and 106 nm for the second stack. The TiO$_2$ layers were deposited with a thickness of 48 nm in the first stack and 68.5 nm for the second stack. The total thickness of the coating was approximately 1.55 $\mu m$.

Spectrophotometric measurements

In this study, a spectrophotometer CM 2600-d [Konica Minolta, Dietikon, Switzerland] was used for the reflectivity and color measurements. The resulting reflectance and laboratory color values were examined with the manufacturer’s software SpectraMagic NX [Konica Minolta]. The observer angle was set to 2°, and the D65 standard light source with 100% UV with specular reflection included (SCI) was used. For the measurement of diffused illumination, the device uses an integrating sphere with apertures of 3 mm in diameter [SAV – small area view]. The spectrophotometer was zero-calibrated with a black box (CM-A 32) and then calibrated with a white plate (CM-A 142). Three consecutive measurements of each specimen were averaged automatically during the spectrophotometric measurement for the reflectance and color values. First, the samples were measured without covering tissue respectively polymeric platelet using the spectrophotometer to determine the reflectivity properties of the coating and substrate material. Then, the samples were evaluated using the two models explained in the following.

In vitro color evaluation with porcine tissue

The specimens were measured spectrophotometrically below porcine tissue according to the in vitro model based on Jung et al. (2007) and Pecnik et al. (2014). Six pig maxillae, which were cut from pigs raised for food production only, were used for the in vitro measurements. The mucosal flaps prepared in the palatal region of the maxillae exhibited a thickness of 1 mm ($0.98 \pm 0.12$ mm). Tissue
Color evaluation with prosthetic gingival resin

A self-curing acrylic denture polymer (Aesthetic Basismaterial, Candulor AG, Switzerland) was used in this study. The color system was no. 34, which corresponded to a dark pink and opaque material with veined structure. The polymer was dosed and mixed according to the pouring technique suggested by Candulor AG. After the mixing, the resin was poured between two glass plates of defined distances, where it polymerized. Finally, the polymer resin was high-gloss polished with pumice on a goat hairbrush.

The dimensions of the polymer platelets were 2 × 2.5 mm, whereas the thickness varied for each test platelet [1, 2, and 3 mm]. Five new pig maxillae were used for the baseline measurement to achieve an equivalent background compared to the in vitro porcine model. In the palatal region, the mucosa was removed and the three test platelets were placed on the bone surface and measured spectrophotometrically. The sample measurement was conducted with the samples and polymeric test platelets only. Water droplets were applied between the samples and the polymeric test platelets to simulate the conditions in the in vitro porcine model. The \( \Delta L_{\text{polymer}}, \Delta a_{\text{polymer}}, \) and \( \Delta b_{\text{polymer}} \) values were determined by the following equation:

\[
\Delta x_{\text{polymer}} = x_{\text{polymer-on-bone}} - x_{\text{sample-below-polymer}}
\]

The color change \( \Delta E_{\text{polymer}} \) value was then calculated by the following equation:

\[
\Delta E_{\text{polymer}} = [(\Delta L_{\text{polymer}})^2 + (\Delta a_{\text{polymer}})^2 + (\Delta b_{\text{polymer}})^2]^{1/2}
\]

Analogously, the critical threshold value \( \Delta E = 3.70 \) was used for the color evaluation with polymeric material [Ruyter et al. 1987; Johnston & Kao 1989]. In total, six specimens were tested below the polymer platelets.

Statistical analysis

Coding of the measured and calculated data was performed in Microsoft Excel and further analyzed with SPSS version 22 (IBM, Armonk, NY, USA). Mean, standard deviation (SD), median (MD), and interquartile range (IQR) were calculated. Statistical significance in both evaluation models was determined for \( \Delta E, \Delta L, \Delta a, \) and \( \Delta b \) by the one sample Wilcoxon test at 1 mm, 2 mm, and 3 mm thick porcine tissue, respectively, polymer platelet. It was tested whether the median \( \Delta E \) value is significantly different from the cutoff value 3.70 [Ruyter et al. 1987; Johnston & Kao 1989]. Likewise, the median values \( \Delta L, \Delta a, \) and \( \Delta b \) were tested against 0.00. The results would be judged to be statistically significant showing a P-value < 0.05.

Furthermore, the Kruskal–Wallis test was applied with Bonferroni-Holm-corrected Mann–Whitney post hoc tests with respect to substrate treatment for \( \Delta E \) at 1, 2, and 3 mm for both evaluation models, separately split for coated (Ti-Bragg) and uncoated (Ti) samples. The Bonferroni–Holm procedure was applied for multiple comparisons and is defined as follows (Holm 1979):
1. Ordered \( P \)-values: \( P_1 \leq P_2 \leq \ldots \leq P_k \)
2. Adjusted \( P \)-values:
   \[ P_{adj}^i = \max_j \{ (k-j+1) P_j \} \]
3. Where \( |x_i| = \min(|x_i|, 1) \) and \( i = 1, 2, \ldots \)

The smallest \( P \)-value is multiplied by the number of individual tests \( k \), the second smallest by \( k-1 \), and so on (with the restrictions that the initial order of \( P \)-values is kept and all \( P \)-values < 1), and evaluation at the initial level \( \alpha \) (typically \( \alpha = 5\% \)).

Results

Reflectance measurements of Ti-Bragg and Ti samples

The detailed results of all tests are displayed in Tables 1a–d, 2 and 3, and in Figures 2–6. Table 1a and Figure 2 show the reflectivity behavior of Ti-Bragg and Ti samples in the visible spectrum. In general, Bragg-coated samples showed higher reflectance values compared to the substrate material. High and constant reflectance (stop band) was observed for Bragg coatings on polished samples between 525 and 600 nm. A small minimum in reflectance was observed at 580 nm, where the two high-reflectance bands of the two stacks overlap. On either side of the stop band, the reflectance decreased in an oscillating way. The level of reflectance decreased slightly by 20% for machined Ti-Bragg samples, but the stop band was kept in the same wavelength range. Sandblasted Ti-Bragg samples showed the lowest reflectance values, and the stop band became narrower with a shift toward smaller wavelengths. Comparably, the reflectivity of polished and machined Ti samples behaved similarly between 40% and 60% reflectance, whereas the sandblasted samples exhibited the lowest reflectance values (below 20%).

Evaluation of Ti-Bragg and Ti samples with the in vitro porcine model

The color changes \( \Delta E_{\text{tissue}} \) from the in vitro porcine tests are shown in Fig. 3. Below 1 mm thick soft tissue, all samples show a mean \( \Delta E_{\text{tissue}} \) value above 3.70, significantly for sandblasted Ti and Ti-Bragg samples, which indicated a dark appearance of the soft tissue (Fig. 3a). There was a significant difference in \( \Delta E_{\text{tissue}} \) for polished and sandblasted Ti, as well as for sandblasted Ti-Bragg samples at 1 mm thick soft tissue. With increasing soft tissue thickness, the \( \Delta E_{\text{tissue}} \) values decreased as shown in Fig. 3b,c. At 3 mm thick soft tissue, all mean values were below the critical threshold. The \( \Delta E_{\text{tissue}} \) values of polished Ti and polished and machined Ti-Bragg samples were significantly below the threshold of 3.70. A significantly green discoloration (negative \( \Delta a_{\text{tissue}} \)) could be observed for all coated and uncoated samples tested below 1 and 2 mm thick soft tissue, except for machined Ti samples (Table 1c). The \( \Delta E_{\text{tissue}} \) values increase for 3 mm thick soft tissue with statistical significance for machined and sandblasted Ti-Bragg samples. In Table 1d, significantly negative \( \Delta b_{\text{tissue}} \) values were particularly noted for sandblasted Ti-Bragg samples tested below 1 and 2 mm thick mucosa.

Evaluation of Ti-Bragg and Ti samples with the polymer model

The same samples were tested with the polymer platelets made of acrylic denture base material. In Fig. 4a, significant and high \( \Delta E_{\text{polymer}} \) values were observed for Ti-Bragg for...
Table 1. Mean values, standard deviation (SD), median (MD), and interquartile range (IQR) of \( \Delta E \) (1a), \( \Delta L \) (1b), \( \Delta a \) (1c), and \( \Delta b \) (1d) values for both evaluation models. The investigated samples, Ti and Ti-Bragg, exhibited different substrate treatments and were tested either below porcine tissue or below polymer plates of different thicknesses. \( \Delta E \) values marked with * were found to be significantly above respectively below the threshold value of 3.70 (one sample Wilcoxon test). Correspondingly, \( \Delta L \), \( \Delta a \), and \( \Delta b \) values marked with * were significantly positive respectively negative with respect to the test value of 0.00 (one sample Wilcoxon test). Brightening respectively darkening were expressed by positive respectively negative \( \Delta L \) value; positive respectively negative \( \Delta a \) values corresponded to red respectively green color shifts; a positive respectively negative \( \Delta b \) values corresponded to yellow respectively blue color shifts.

<table>
<thead>
<tr>
<th>Coating</th>
<th>Substrate treatment</th>
<th>1 mm</th>
<th>2 mm</th>
<th>3 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \Delta E_{\text{tissue}} )</td>
<td>( \Delta E_{\text{polymer}} )</td>
<td>( \Delta E_{\text{tissue}} )</td>
<td>( \Delta E_{\text{polymer}} )</td>
</tr>
<tr>
<td>Ti</td>
<td>Polished</td>
<td>3.88 ± 0.88</td>
<td>3.70 ± 0.93</td>
<td>3.17 ± 0.30</td>
</tr>
<tr>
<td></td>
<td>Machined</td>
<td>5.29 ± 1.89</td>
<td>4.85 ± 1.57</td>
<td>4.11 ± 0.30</td>
</tr>
<tr>
<td>Sandblasted</td>
<td>Polished</td>
<td>6.30 ± 2.34</td>
<td>9.89 ± 1.52</td>
<td>9.21 ± 2.81</td>
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<td>Machined</td>
<td>4.63 ± 1.63</td>
<td>9.09 ± 0.99</td>
<td>9.29 ± 2.17</td>
</tr>
<tr>
<td>Ti-Bragg</td>
<td>Polished</td>
<td>4.40 ± 2.30</td>
<td>8.70 ± 0.93</td>
<td>8.65 ± 0.50</td>
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<tr>
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<td>Machined</td>
<td>7.99 ± 2.44</td>
<td>8.54 ± 1.43</td>
<td>7.89 ± 2.70</td>
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<th>2 mm</th>
<th>3 mm</th>
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<tr>
<td></td>
<td>( \Delta a_{\text{tissue}} )</td>
<td>( \Delta a_{\text{polymer}} )</td>
<td>( \Delta a_{\text{tissue}} )</td>
<td>( \Delta a_{\text{polymer}} )</td>
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<tr>
<td>Ti</td>
<td>Polished</td>
<td>0.11 ± 0.26</td>
<td>0.59 ± 0.31</td>
<td>0.97 ± 0.47</td>
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<td></td>
<td>Machined</td>
<td>2.55 ± 0.72</td>
<td>1.18 ± 0.50</td>
<td>2.95 ± 1.31</td>
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<td>Sandblasted</td>
<td>Polished</td>
<td>0.44 ± 0.28</td>
<td>0.42 ± 0.62</td>
<td>0.67 ± 0.89</td>
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<td>Machined</td>
<td>2.69 ± 0.92</td>
<td>2.95 ± 1.36</td>
<td>3.37 ± 0.93</td>
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<tr>
<td>Ti-Bragg</td>
<td>Polished</td>
<td>2.63 ± 0.92</td>
<td>0.42 ± 0.62</td>
<td>0.67 ± 0.89</td>
</tr>
<tr>
<td></td>
<td>Machined</td>
<td>0.15 ± 0.15</td>
<td>0.42 ± 0.62</td>
<td>0.67 ± 0.89</td>
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<tr>
<th>Coating</th>
<th>Substrate treatment</th>
<th>1 mm</th>
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<th>3 mm</th>
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<tbody>
<tr>
<td></td>
<td>( \Delta b_{\text{tissue}} )</td>
<td>( \Delta b_{\text{polymer}} )</td>
<td>( \Delta b_{\text{tissue}} )</td>
<td>( \Delta b_{\text{polymer}} )</td>
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<tr>
<td>Ti</td>
<td>Polished</td>
<td>0.16 ± 0.08</td>
<td>0.67 ± 0.42</td>
<td>1.16 ± 0.10</td>
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<td></td>
<td>Machined</td>
<td>0.15 ± 0.08</td>
<td>0.67 ± 0.42</td>
<td>1.16 ± 0.10</td>
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<tr>
<td>Sandblasted</td>
<td>Polished</td>
<td>0.16 ± 0.08</td>
<td>0.67 ± 0.42</td>
<td>1.16 ± 0.10</td>
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<tr>
<td></td>
<td>Machined</td>
<td>0.15 ± 0.08</td>
<td>0.67 ± 0.42</td>
<td>1.16 ± 0.10</td>
</tr>
</tbody>
</table>

all the three substrate treatments. In Table 1b, the lightness values for polished and machined Ti-Bragg samples indicated a strong and significant brightening below 1 mm thick polymer. However, sandblasted Ti samples showed a significant color change and significantly negative ΔE_p value in the 1 mm thick polymer platelet, which indicated a darkening. Bragg coatings on polished and machined Ti samples did not induce a visible color change for polymer thicknesses of 2 and 3 mm as seen in Fig. 4b,c. Significant darkening was still present for sandblasted Ti and Ti-Bragg samples below 2 mm thick polymer (Fig. 4b and Table 1b), but the ΔE_p values decreased below or close to the threshold of 3.70 with increasing test thickness [Fig. 4c].

Nearly, all Δa_p values of the samples were significantly negative for all thicknesses, demonstrating a strong green discoloration through the polymeric test platelets [Table 1c]. Only polished Ti-Bragg showed a positive Δa_p value at 3 mm thick polymer platelet, which provides redness to the overall color. In Table 1d, positive Δb_p values contributed to yellowness of the covering polymer, which were only observed for polished and machined Ti-Bragg samples. All Ti samples and sandblasted Ti-Bragg samples showed a shift in to the blue region of the spectrum, partially significant deviations in Δb_p.

The tissue grafts and polymer platelets were measured on bone, and their color values are compared in Table 2. The lightness of porcine tissue on bone increased with increasing thickness, whereas the lightness values of the polymer platelets decreased. The tissues showed higher L values compared to the polymer. The fraction of redness was smaller for tissue grafts than for polymer platelets. Both platelets had positive b values in a similar range and induced yellowness to the overall appearance on bone.

The results in Table 3 for ΔE_tissue and ΔE_polymer demonstrated that several interactions were significant, generally with sandblasted samples. However, only Ti-Bragg samples below 2-mm- and 3-mm-thick tissue showed two significant interactions for ΔE_tissue values [both machined and sandblasted with P_{BH} < 0.05], whereas Ti samples did not demonstrate significant influence on ΔE_tissue. For the polymer model, significant interactions were observed for Ti samples for all the three test thicknesses (P_{BH} = 0.027 for the interactions polished-sandblasted and machined-sandblasted). Only two interactions significantly influenced ΔE_polymer for the coating system Ti-Bragg at 2 mm test thickness (P_{BH} = 0.027 for the interactions

![Fig. 4. Mean value of ΔE_polymer (±SD) of Ti and Ti-Bragg samples tested below different thicknesses of polymer platelets: (a) 1 mm, (b) 2 mm, and (c) 3 mm thick acrylic polymer. Ti served as a negative control group. The critical threshold value of 3.70 for intraoral distinction by the naked eye is illustrated by the dotted line [Johnston & Kao 1989]. Bars marked with * were significant on the cutoff value of 3.70.](image-url)
Table 3. (a) Results of the Kruskal–Wallis test: probability values $P$ for $\Delta E_{\text{tissue}}$ and $\Delta E_{\text{polymer}}$ values of Ti-Bragg and Ti samples below the three different test thicknesses (significant statistical differences for $P < 0.05$ are marked with *). (b) Results of the Mann–Whitney test: probability values $P$ and corrected values $P_{\text{BH}}$ with the Bonferroni–Holm procedure for multiple testing. The $P$ respectively $P_{\text{BH}}$ values were considered to be statistically significant at the 0.05 significance level and were marked with *.

<table>
<thead>
<tr>
<th>Coating system</th>
<th>$\Delta E_{\text{tissue}}$</th>
<th>$\Delta E_{\text{polymer}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<tr>
<td>(a) Kruskal–Wallis test</td>
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<td></td>
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<tr>
<td>Ti</td>
<td>0.113</td>
<td>0.071</td>
</tr>
<tr>
<td>Ti-Bragg</td>
<td>0.031*</td>
<td>0.039*</td>
</tr>
<tr>
<td>(b) Mann–Whitney test with Bonferroni–Holm correction</td>
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<td></td>
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<tr>
<td>Ti (1 mm)</td>
<td>Polished–machined</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>Polished–sandblasted</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>Machined–sandblasted</td>
<td>–</td>
</tr>
<tr>
<td>Ti (2 mm)</td>
<td>Polished–machined</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>Polished–sandblasted</td>
<td>0.009*</td>
</tr>
<tr>
<td></td>
<td>Machined–sandblasted</td>
<td>0.047*</td>
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<tr>
<td>Ti (3 mm)</td>
<td>Polished–machined</td>
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</tr>
<tr>
<td></td>
<td>Polished–sandblasted</td>
<td>–</td>
</tr>
<tr>
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<td>Machined–sandblasted</td>
<td>0.347</td>
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<td>Ti-Bragg (1 mm)</td>
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<td>Polished–sandblasted</td>
<td>0.037*</td>
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<td>Machined–sandblasted</td>
<td>0.150</td>
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<td>Ti-Bragg (2 mm)</td>
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<td>Polished–sandblasted</td>
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<tr>
<td>Ti-Bragg (3 mm)</td>
<td>Polished–machined</td>
<td>0.025*</td>
</tr>
<tr>
<td></td>
<td>Polished–sandblasted</td>
<td>0.749</td>
</tr>
<tr>
<td></td>
<td>Machined–sandblasted</td>
<td>0.016*</td>
</tr>
</tbody>
</table>

Discussion

This study investigated the optical properties of a dielectric mirror coating using two different evaluation methods: [a] The performance of the Bragg coating is being discussed with regard to its potential as an aesthetic dental coating for implants and prosthetic appliances, [b] in view of a straightforward and invariant evaluation procedure for dental materials, the polymer model is validated and compared to the established in vitro model using porcine tissue.

Fig. 5. Mean value of $\Delta E_{\text{polymer}}$ [±SD] of Ti and Ti-Bragg samples tested below 1 mm thick polymer platelet. Values marked with * were significantly positive respectively negative with respect to the test value of 0.00.

Polished–sandblasted and machined–sandblasted.

Polished Ti-Bragg samples showed partial delamination at the edges of the platelet during the in vitro porcine tests.

In vitro evaluation of the optical appearance of Ti-Bragg

Experiments to assess and improve the esthetics of peri-implant mucosa were made in previous studies [Jung et al. 2007; Pecnik et al. 2014]. Best results were achieved with zirconia veneers- or zirconia-based coatings such as Ti-Al-ZrO2 and Ti-Ag-ZrO2 systems. However, both coating systems on polished and machined substrates showed a light brightening due to significantly positive lightness values. This could still cause an esthetic impairment in regions near the gingival margin, where the soft tissue is thin and prone to discoloration. Surprisingly, polished and machined Ti-Bragg samples showed similar $\Delta E_{\text{tissue}}$ values when tested below 1 mm thick soft tissue, but their $\Delta E_{\text{tissue}}$ values were slightly negative without causing a significant darkening. The optical properties of the Ti substrate, which caused a visible darkening of 1 mm thick soft tissue, were still improved by the Bragg coating as observable in the reflectance curves. However, significantly negative $\Delta E_{\text{tissue}}$ values let these $\Delta E_{\text{tissue}}$ values increase above the threshold, as the Bragg coating reflected the green part of the visible spectrum to a high extend in the stop band. In this case, the design of the Bragg coating can be adjusted to optimize the final color below soft tissue, as the human gingival color differs strongly for each patient due to health condition and oral pigmentation [Dummett & Barens 1966, Powers et al. 1977, Dummet et al. 1980]. The lightness respectively the reflectance $R$ is correlated with the number of layers $N$ of each stack for a certain design wavelength $\lambda_d$ with $n_0$ as the originating medium (Sheppard 1995; Macleod 2001):

$$R = \left[ n_0 \cdot (n_{\text{TiO}_2})^{2N} - n_0 \cdot (n_{\text{SiO}_2})^{2N} \right] / \left[ n_0 \cdot (n_{\text{TiO}_2})^{2N} + n_0 \cdot (n_{\text{SiO}_2})^{2N} \right]$$

For porcine tissue ($n_0 = n_{\text{tissue}} > 1$), one might have to further increase the number of layers $N$ to reach enough light propagation from the coating through the tissue for an esthetic appearance of the gingiva.

Furthermore, the width of the stop band $\Delta \lambda$ depends on the design wavelength $\lambda_d$ as described in the following equation (Macleod 2001):

$$\Delta \lambda = 4 \cdot \lambda_d / \pi \cdot \arcsin(\Delta n / (n_{\text{TiO}_2} + n_{\text{SiO}_2}))$$

As there are two stacks of TiO2/SiO2 layers in the present coating, the design wavelengths might be designed separately. The design wavelength of one stack can be...
controlled through the thickness of TiO₂ and SiO₂ layers (Heavens & Liddell 1966). To reduce the green shade in 1 mm thick tissue, one might increase the thicknesses of the oxide layers in the second stack to increase λ₂ and therefore reach the red regime of the spectrum. However, properties such as adhesion or strength could suffer from an excessive increase in coating thickness (Volinsky et al. 2003; Kraft et al. 2010). Additionally, adhesion problems can be avoided by the right choice of the substrate roughness. As proposed in Pecnik et al. (2014), machined substrates could provide a mechanical interlocking between coating and substrate to reach an appropriate adhesion. In this study, coatings on polished substrates showed high reflectance values, but also partial delamination from the edges of the samples. Bragg coatings on machined substrates still featured high enough reflectance values without showing any undesired discolorations or delamination. In contrast, sandblasted surfaces exhibited a poor reflectance behavior. Furthermore, significant differences in ΔΕₜissue were mostly observed for interactions with sandblasted Ti-Bragg samples. Consequently, Bragg coatings on machined Ti might be an even more effective and esthetic solution than the suggested coatings in Pecnik et al. (2014).

**Validation of the polymer model for color assessments**

Efforts to regenerate the oral esthetics can be undermined by gingival recession (Kassab & Cohen 2003; Joss-Vassalli et al. 2010). Alternatively, patients, who do not respond to surgical procedures due to poor oral hygiene and systematic diseases, receive gingival replacements made of silicone, porcelain, composite, or acrylic resins (Barzilay & Irene 2003; Coachman et al. 2009). Acrylics possess beneficial properties such as color stability and stain resistance, as confirmed in studies and clinical cases on acrylic gingival prostheses (Lai et al. 2003; Alani et al. 2011; Ajita et al. 2012). Up to now, only color changes in resins immersed in staining solutions were studied in detail. Literature on shade guides for this polymer is limited (Godoy et al. 1992). Instead, the color of human mucosa was evaluated frequently in a subjective as well as objective way (Heydecke et al. 2005; Park et al. 2007; Huang et al. 2011; Paniz et al. 2014). In the present study, the color values of resin on porcine bone were similar to the measurements of human mucosa (Park et al. 2007; Huang et al. 2011), whereas porcine tissue demonstrated even lighter values. Moreover, λₜissue-on-bone values increased with increasing porcine tissue thickness, contrary to the behavior of human mucosa observed in Park et al. (2007). This implies that the acrylic polymer used in this study might also be an appropriate model for the evaluation of peri-implant discolorations. Nevertheless, excessive brightening was observed for Ti-Bragg samples below 1 mm thick polymer platelet (Fig. 5), which did not match with the results obtained in the in vitro porcine evaluation. The reflectance curves in Fig. 6a,b revealed that samples below the polymer material contributed to a lighter appearance as the reflectance was higher than its baseline measurement, in contrast to the in vitro model, which showed nearly matching reflectance curves up to a wavelength of 600 nm. For porcine tissue, the absorption peaks at 420, 550, and 580 nm were caused from oxygenated hemoglobin (Altshuler & Yaroslavsky 2004), which strongly absorbed the light to and from the underlying Bragg coating through the tissue. Apart from the oxygenated hemoglobin, biological tissue possesses scattering centers such as fiber structures, vessel walls, and cell

**Fig. 6.** The reflectance curves for |a| polished, |b| machined, and |c| sandblasted Ti-Bragg samples were measured without and below 1 mm thick porcine tissue and 1 mm thick polymer platelets.
organelles, which let the light decay while traveling through it (Tuchin 2007). Therefore, it is suggested that the in vitro porcine model transmitted less light compared to the polymer model at a thin thickness. However, the samples behaved similarly in both models with increasing test thickness. It is presumed that the polymer platelets with thicknesses of 2 and 3 mm can be used to simulate the accordingly thick porcine tissue flaps. Tests with thin respectively 1 mm thick polymer, one might have to increase the thickness, for instance to 1.5 mm, or increase the amount of filler material, which simulated the veined structure, in the polymer to establish the same situation for 1 mm thick gingival tissue. More studies are needed to further analyze this new simplified model.

Conclusions

This study investigated the following two aspects:

- Esthetic behavior of Ti-Bragg below porcine tissue
- Qualification of the polymer model for color evaluations

The polymer model showed similar color values compared to human gingival color from other studies. However, for the 1 mm case, the results deviated strongly from the approved in vitro porcine model. It is suggested to either increase the thickness or change the composition of the polymer accordingly.

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References


