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# The impact of argon/oxygen low-pressure plasma on shear bond strength between a veneering composite and different PEEK materials

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## ABSTRACT

**Objective.** The aim of the study was to evaluate the impact of low-pressure argon/oxygen plasma with and without previous sandblasting on the shear bond strength (SBS) between dental PEEK compounds and a veneering composite.

**Methods.** Of one type of unfilled PEEK and two pigment powder filled PEEK compounds, forty rectangular plates each were prepared and polished up to 4000 grit. The samples were randomly assigned to four surface pre-treatment groups, each consisting of ten specimens (1. Untreated; 2. Plasma treatment; 3. Sandblasting; 4. Sandblasting + plasma treatment). Plasma treatment was performed for 35 min using a low-pressure plasma system with a 1:1 mixture of the process gases argon and oxygen. Surface roughness and water contact angles were recorded. An adhesive (Visio.link, Bredent GmbH & Co KG, Senden, Germany) was applied onto the specimen surfaces and light cured. A mold was used to shape the veneering composite (Vita VM LC, Vita Zahnfabrik, Bad Säckingen, Germany) into a cylindrical form on the sample surface before light curing. SBS was measured after 24 h incubation at 37 °C in distilled water using a universal testing machine.

**Results.** The samples pre-treated according to group 4 (sandblasting and plasma treatment) showed the highest SBS overall, whereas the unfilled PEEK showed the highest SBS ( $19.8 \pm 2.46$  MPa) compared to the other PEEK materials ( $15.86 \pm 4.39$  MPa and  $9.06 \pm 3.1$  MPa). **Significance.** Sandblasting and surface activation with low-pressure argon/oxygen plasma in combination with an adhesive causes a favorable increase in shear bond strength, especially on unfilled PEEK material.

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## 1. Introduction

Due to its outstanding properties, the use of the thermoplastic high-performance material polyetheretherketone (PEEK) is

becoming more and more commonplace in the field of dentistry. Among those unique and beneficial properties are not only its attested biocompatibility but also its bone-like rigidity (modulus of elasticity: 3–4 GPa) combined with a low density of  $1.32 \text{ g/cm}^3$  [1]. Furthermore, the use of PEEK meets the growing patient demand for metal-free dental reconstructions [2] as framework material for removable and fixed prostheses [3–6].

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Because of its innate opacity, PEEK must be covered with veneering composites when used as a framework for dental prostheses if an acceptable esthetic outcome is to be attained [7]. Its base color, which is grey in an unfilled material, can be adjusted through the addition of appropriate pigments.

PEEK exhibits low surface energy and is chemically inert. Consequently, a chemical alteration of the PEEK surface is essential in achieving sufficient bond strength to a veneering material. This is generally considered a challenge in the processing of PEEK [8,9].

Accordingly, different methods of modifying the PEEK surface prior to veneering have been investigated. The simplest of these procedures is sandblasting [10–15], which results in an increase of surface roughness, in turn allowing for the micromechanical anchoring of the veneering composite [16]. More complex methods range from the Rocatec procedure [10,11,14,17] to surface etching with concentrated sulfuric acid [10,11,13–15,18], Piranha solution (peroxymonosulfuric acid, a mixture of hydrogen peroxide and sulfuric acid) [14,15,18,19] or hydrofluoric acid [13]. In addition to the surface roughening effect of these more advanced techniques, they generate reactive groups for a direct chemical connection to the adhesive system [20]. In general, the use of an adhesive system seems to increase bond strength [12,15,17,18].

In contrast to the risky application of the aforementioned aggressive acidic solutions a chemical surface modification with plasma is a non-hazardous procedure. In generating plasma a process gas is ionized in a high voltage electric field. The thereby produced charged particles induce certain effects on the treated surface, which are [20]:

- Removal of organic residues.
- Microetching.
- Cross-linking.
- Surface activation.

The treatment of PEEK surfaces with atmospheric plasma prior to veneering does not affect shear bond strength (SBS) [7], but SBS was markedly improved in a polyether imide/nano-SiO<sub>2</sub>/PEEK compound through the treatment with argon low-pressure plasma [13].

A similar positive influence on SBS with the application of argon-oxygen low-pressure plasma is to be expected. Accordingly, the aim of the current study was to investigate the influence of argon-oxygen low-pressure plasma treatment on SBS between different types of PEEK and a veneering composite with and without previous sandblasting of the surface with corundum, assuming that argon-oxygen low-pressure plasma pre-treatment leads to higher SBS-values.

## 2. Materials and methods

Three different types of PEEK were used in this study: an unfilled PEEK type (Juvora Ltd., Lancastershire, UK), a compound containing 20% titanium oxide (TiO<sub>2</sub>) powder (Vestakeep DC4420, Evonik Industries AG, Essen, Germany) and a beige colored compound filled with 20% TiO<sub>2</sub> powder and about 1% of pigment powder (Vestakeep DC4450, Evonik Industries AG, Essen, Germany). The three bulk specimens

were cut into 40 rectangular slabs (total n=120) measuring 10 × 15 × 2.5 mm with a slow speed precision saw (IsoMet 1000 Precision Cutter, Buehler, Lake Bluff, USA) using water cooling. The specimens were subsequently polished using a polishing machine (Exakt 400 CS, EXAKT Advanced Technologies GmbH, Norderstedt, Germany) with 320, 800, 1200, 2500 and 4000 grit rotating silica carbide (SiC) papers (Hermes Schleifmittel GmbH, Hamburg, Germany) while rinsing with water. Prior to veneering, the specimens were divided into four groups, which were pre-treated as follows:

- No treatment.
- Only plasma treatment.
- Only sandblasting.
- Sandblasting and plasma treatment.

Sandblasting was performed with Al<sub>2</sub>O<sub>3</sub> particles with a grain size of 110 µm (Korox 110, BEGO Bremen, Bremen, Germany), applied perpendicularly to the surface of the specimens for 10 s/cm<sup>2</sup> at a pressure of 2.5 bar and at a distance of 10 mm. Afterwards, specimens were ultrasonically cleaned for 5 min in 96% ethanol and finally air-dried.

For the plasma treatment a low-pressure plasma system (Femto PCCE, Diener electronic GmbH & Co KG, Ebhausen, Germany) was employed. A 1:1 mixture of argon and oxygen was used as process gas. Plasma was applied to the specimens for 35 min at 70 °C and a pressure of 0.3 mbar using a HF generator with a frequency of 100 kHz and a power output of 200 W.

After pre-treatment, surface roughness (Ra) was measured using the Alicona infinite focus system (Alicona Imaging GmbH, Raaba/Graz, Austria). This system records a 3D picture of the surface at 20-fold magnification. The mean surface roughness (Ra) along two perpendicular lines, each with a length of 4 mm, was calculated based on the obtained 3D image.

Water contact angles were measured after the pre-treatment procedures with the sessile drop method using a H<sub>2</sub>O droplet of 10 µl on a representative spot outside of the veneering area. For the angle measurement a digital microscope (Keyence VHX-5000, Keyence GmbH, Neu-Isenburg, Germany) was used, whereby the optical axis was set horizontally and aligned parallel to the specimen surface. The measurement was taken at room temperature and 10 s after the initial contact between droplet and surface.

After measuring surface roughness and water contact angle, an adhesive consisting of MMA, PETIA and photoinitiators (Visio.link, Bredent GmbH & Co KG, Senden, Germany) was applied to the specimen surfaces using a small brush. The adhesive was light cured (Spektramat, Ivoclar Vivadent GmbH, Ellwangen, Germany) for 90 s following the manufacturer's recommendations. Afterwards, a veneering composite consisting of triethylene glycol dimethacrylate, 2-dimethylaminoethyl methacrylate and 41–52 wt% filler (SiO<sub>2</sub>) (Vita VM LC, Vita Zahnfabrik, Bad Säckingen, Germany) was bonded to the PEEK samples. For reproducibility and uniformity a cylindrically shaped Teflon mold with an inner diameter of 5 mm and a height of 2 mm was used in applying the composite. The mold was set on the specimen surface

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