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Influence of different low-pressure plasma process parameters on shear bond strength between veneering composites and PEEK materials

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ABSTRACT

Objective. The aim of this study was to evaluate the impact of oxygen and argon/oxygen low-pressure plasma on the shear bond strength (SBS) between dental PEEK compounds and veneering composites as a function of plasma process time.

Methods. Of an unfilled PEEK ("Juvora") and two pigment powder filled PEEK compounds ("DC4420", "DC4450"), 273 rectangular plates were prepared and polished up to 1200 grit. Afterwards the samples were sandblasted and randomly assigned to five different surface pre-treatment groups (1. No plasma (control); 2. O₂ plasma for 3 min; 3. O₂ plasma for 35 min; 4. Ar/O₂ plasma for 3 min; 5. Ar/O₂ plasma for 35 min). Surface roughness and water contact angles were recorded using three samples of each PEEK compound for each of the plasma treatment groups. 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 three different veneering composites (a) Vita VM LC, "Vita" (Vita Zahnfabrik, Bad Säckingen, Germany); (b) GC GRADIA, "Gradia" (GC Europe, Leuven, Belgium); (c) GC GRADIA DIRECT Flo, "Gradia Flo" (GC Europe, Leuven, Belgium)) into a cylindrical form on the sample surface before light curing. SBS was measured using a universal testing machine after 24h of incubation in distilled water at 37 °C.

Results. The two pigment filled PEEK compounds treated with O_2 plasma and veneered with Gradia Flo showed the highest SBS values ($34.92\pm6.55\,\text{MPa}$ and $34.2\pm1.87\,\text{MPa}$) followed by the combination of the unfilled PEEK material with Gradia Flo ($29.57\pm3.71\,\text{MPa}$). The SBS values of the samples veneered with Gradia were lower, but not significantly so. The SBS values of the specimens with Vita were for the most part associated with significantly lower results.

Significance. A low-pressure plasma process using oxygen plasma for a duration of 35 min, preceded by sandblasting, seems to be the most effective in increasing shear bond strength between veneering composites and PEEK materials.

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1. Introduction

The high-performance plastic polyether ether ketone (PEEK) is polyaromatic thermoplasic resin from the family of polyaryil ether ketones [1]. Apart from its potential applications in the medical field [2] it is increasingly gaining interest for dental purposes [3-5], which is in large part due to its exceptional physical and chemical properties as high-performance polymer. First among these the modulus of elasticity stands out, which at 3-4 GPa is much closer to that of bone than that of metal alloys or ceramics [6,7]. This matching elasticity is assumed to be of benefit to the oral maxillofacial system with its particular mechanical loads [5,8]. Furthermore, PEEK impresses with a very low density of 1.265 g/cm³ as well as excellent values for tensile strength, flexural strength and resistance to abrasion [1], indicating high wearing comfort and durability. PEEK is biologically inert and as such constitutes a hypoallergenic metal-free alternative to conventional prosthetic materials such as dental alloys [9,10]. The chromatic appearance of PEEK is preferable to the metallic sheen of conventional alloys [9] and can be adjusted within certain limits with additives such as pigment powder. High accuracy of fit can be achieved using CAD-CAM milling systems [11]. Based on these positive characteristics PEEK could soon become a dependable, esthetically satisfying alternative to metal based materials for dental restorations, possibly even as alternative to ceramic dental implants [12]. In scientific literature, a number of potential applications in the field of dentistry have been described. For example, research on the use of PEEK as a framework material for fixed [4,9] and removable [5,13] prostheses as well as material for implant abutments or implant bodies [5,14] has been conducted. Due to its optically opaque characteristics, PEEK has to be veneered with appropriate composites if it is to be used as a framework material for esthetically satisfying restorations [9,15]. Achieving the pursued chemical bond between PEEK and veneering composites continues to represent a challenge, which is due to the low surface energy of PEEK [16]. In the past, a number of different methods of surface modification have been investigated, that primarily rely on the mechanical alteration of the surface. Accordingly, research has been conducted employing sandblasting [9,17-21] and acid etching techniques [9,17-19,21,22] for the creation of a surface topography on PEEK that facilitates the micromechanical interlocking of the veneering composite. Other studies evaluated the utility of the Rocatec procedure [9,17,18,23]. Additionally, it has been concluded that coupling agents exert a positive influence on bonding strength [17-19,22]. Another method of surface modification is the application of plasma, the possibilities of which are a topic of research in many areas of medicine [14,24,25]. Plasma is considered the "third physical state". It consists of ionized gas particles. Apart from electrically charged particles, a plasma produces ultraviolet and thermal radiation, visible light as well as reactive molecules [24], the composition of which can vary according to the plasma parameters. The main surface alterations resulting from plasma treatment are increased wettability of the surface [26], changes in surface roughness [27] and the creation of radicals through ultraviolet radiation [28].

A recently published study demonstrated that treatment of PEEK with an Ar/O_2 low-pressure plasma prior to veneering has a beneficial influence on the shear bond strength (SBS) between the PEEK surface and a veneering composite [29]. In the study, sandblasting alone had a greater influence on SBS than the subsequent plasma treatment, which has been ascribed to a certain surface smoothing effect due to the etching effect of the plasma. The duration of the plasma processing in that study was 35 min. Consequently, it can reasonably be hypothesised that a shorter plasma process time will affect the surface roughness to a lesser degree, while retaining the positive impact of plasma treatment on SBS, thus improving overall SBS.

Accordingly, the aim of the present study was the evaluation of the influence of different plasma process durations and process gases of a low-pressure plasma on the shear bond strength between combinations of different veneering composites and PEEK compounds.

2. Materials and methods

As base material three different types of PEEK were employed:

- Juvora Dental Disk (Invibio Ltd., Lancashire, UK); unfilled, with gray color.
- DC 4420 (Evonik Industries AG, Essen, Germany); filled with 20% TiO₂ powder, white color.
- iii. DC 4450 (Evonik Industries, Essen, Germany), filled with 20% TiO₂ powder and 1% pigment, beige color.

272 samples with the dimensions of $10 \times 15 \times 2.5 \, \text{mm}$ were prepared from these three PEEK grades using a watercooled slow speed precision saw (IsoMet1000 Precision Cutter, Buehler, Lake Bluff, USA). The samples were polished with the aid of a water-cooled polishing machine (Exakt 400 CS, EXAKT Advanced Technologies GmbH, Norderstedt, Germany) using SiC sandpaper (Hermes Schleifmittel GmbH, Hamburg, Germany) of 320, 800 and 1200 grit. Subsequently, the samples were sandblasted with a pressure of 2.5 bar using Al₂O₃ blasting agent of 110 µm grain size (Korox 110, BEGO Bremen, Bremen, Germany) with a progression speed of 10 s/cm2 and from a distance of 10 mm perpendicularly to the surface. The samples were cleaned for 5 min with 96% ethanol in an ultrasonic bath and then air-dried. Of every PEEK grade, 12 samples were randomly selected for the measurement of surface roughness and water contact angle. The surface roughness was measured with the aid of the mechanical profilometer Perthometer S6P (Feinprüf Perthen GmbH, Germany). The measurement was carried out on three samples per PEEK type and per plasma pretreatment group, before as well as after plasma treatment. Roughness was evaluated on three different areas per sample and the average roughness value Ra per sample was calculated. The surface wettability was determined by means of the water contact angle. Hereby the sessile drop technique was used. A water drop with a volume of $10\,\mu l$ was placed on the sample surface. The angle measurement was conducted with the aid of a digital microscope (Keyence VHX-5000, Keyence GmbH, Neu-Isenburg, Germany), the optical axis of which was aligned in parallel to the hori-

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