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Resin bonding to three types of polyaryletherketones (PAEKs)—Durability and influence of surface conditioning

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ABSTRACT

Objectives. The purpose of this in vitro study was to evaluate the bond strength and durability of adhesive bonding systems to amorphous and crystalline PEKK and fiber-reinforced PEEK using five types of surface conditioning methods.
Methods. One hundred and fifty specimens of each material were conditioned mechanically and chemically, bonded with Multilink Automix to Plexiglas tubes, filled with Multicore Flow, and stored in water at 37 °C for 3, 30 and 150 days. The long-term storage series were thermal cycled between 5 and 55 °C for 10,000 times (30 days) or for 37,500 times (150 days) prior to tensile bond strength test (TBS). Statistical analysis was performed using Kruskal–Wallis and Wilcoxon tests with a Bonferroni–Holm correction for multiple testing ($\alpha = 0.05$).
Results. Fiber-reinforced PEEK exhibited higher bond strengths in all five conditioning groups and at all three storage times than crystalline and amorphous PEKK, which showed lowest TBS. Highest TBS was achieved after conditioning with silica coating and priming (Rocatec Soft, Monobond Plus, Luxatemp Glaze & Bond; TBS up to 23.6 MPa).
Significance. The conditioning method has a significant influence to the bond strength of the bonding to the amorphous and crystalline PEKKs and fiber-reinforced PEEKs.

1. Introduction

Polyaryletherketones (PAEKs) are a group of high-performance semicrystalline thermoplastic resins, which family members differ according to their ratio of keto- and ether-groups (Fig. 1). With a higher ratio and sequence of keto groups, the rigidity of the polymer chain and the glass, as well as melting temperature are increasing [1,2]. Different PAEKs have similar high-quality characteristics such as good dimensional stability at high temperatures (melting temperature is over 300 °C), high chemical and mechanical resistance against wear, and high tensile, fatigue and flexural strengths ([2,3], see Table 1). According to the manufacturer (Cendres+Métaux, Switzerland) they are compatible with reinforcing materials such as glass and carbon fibers and can be sterilized with the current methods like the gamma- and steam-sterilization [2]. Their characteristics make PAEKs highly attractive for industrial usage. Therefore, their field of application extends from food, aircraft and automobile industry to medical products.

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Table 1 – Physical and chemical properties of amorphous and crystalline PEKK and fiber-reinforced PEEK (according to the information provided by the manufacturer and by www.matweb.com).

Physical and mechanical properties	PEKK (amorphous)	PEKK (crystalline)	PEEK (fiber)
Flexural strength (MPa)	140	200	312
Tensile strength (MPa)	89	117	215
Glass temperature (°C)	160	157	143
Softening temperature range or melting temperature (°C)	305–325	364	341

In medicine, PEEK, the best-known PAEK member, mainly serves as implantation material due to its mentioned features and good biocompatibility. It has been proven as an adequate alternative for the long-term proven titanium in orthopedic applications [4–6]. In dentistry, the usage of PEEKs is increasing mainly as temporary implant abutments [4,5,7]. First results are available as well for its use as dental clasps and frameworks for partial removable dental prostheses [8].

Due to its chemical and physical properties, the high-performance thermoplastic PEKK outclasses all the other PAEK materials [9]. The manufacturer (Cendres+Métaux) reports about an up to 80% higher compressive strength of PEKK as compared to the unreinforced PAEK material PEEK. A wider processing window of parameters makes PEKK also especially interesting for the fabrication of crowns and fixed dental prostheses (FDPs). The possibility of producing different rigidity also makes PEKK useful for different applications, e.g. crystalline PEKK for crowns and FDPs, amorphous PEKK for removable prosthesis. However, a clinically adequate bonding to PEKK is a prerequisite for intraoral usage of bonded PEKK restorations.

Up to date, only rare data on bonding to the class of PAEK materials exists. When testing the tensile bond strength (TBS) of differently conditioned PEEK no adequate bonding could be achieved using dental universal composite resin cement in contrast to using an adhesive composite system [10]. Another study [11] showed that the conditioning of PEEK with air-abrasion or silica-coating improved the adhesive properties of PEEK, because the micro-roughness enhances the contact surface with its functional groups between the PEEK and the adhesive. Another recent study [12] tested different primers on PEEK after artificial aging. Only when using a multifunctional methacrylates containing primer (Luxatemp Glaze & Bond), a durable resin bonding was achieved, which therefore was recommended for clinical use. For bonding to PEKK no data could be found in the dental literature.

In view of the limited data available on bonding to PAEK materials, the purpose of the current study was to evaluate different methods for bonding to amorphous and crystalline PEKKs and to fiber-reinforced PEEK. In addition the durability of the achieved bonding should be tested. The null hypothesis was that the bonding method of surface conditioning does not influence the bonding durability and the used methods.

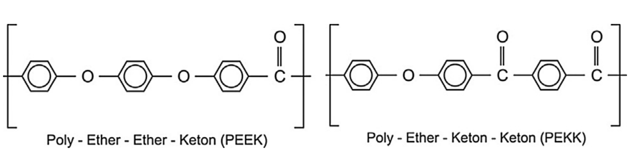

Fig. 1 – Chemical structures of PEEK and PEKK.

2. Materials and methods

In this study, the tensile bond strength and the durability of adhesive bonding of amorphous and crystalline PEKK and fiber-reinforced PEEK discs (150 specimens each) was tested using a luting resin (Multilink Automix, Ivoclar Vivadent AG, Schaan, Liechtenstein). The bonding surfaces of in total 450 discs, with a diameter of 8 mm and a thickness of at least 3 mm each, were polished with rotating silicon carbide paper (SiC Grinding Paper, Grit P600, Bühler GmbH, Düsseldorf, Germany) under water rinsing.

Specimens of each material (amorphous, crystalline, fiber) were divided into the following five groups with different surface conditioning with 30 specimens each.

Pre	For surface cleaning and activation air-abrasion with alumina particles (Rocatec Pre, 3M Espe, Seefeld, Germany) at 0.25 MPa for 15 s was used, then cleaning with compressed air for 15 s. Bonding with Multilink Automix without any adhesion promoter served as negative control group to reveal whether the application of adhesion promoters is effective
PreLu	Air-abrasion with Rocatec Pre at 0.25 MPa for 15 s was used, then cleaning with compressed air for 15 s. Application of the adhesive primer Luxatemp Glaze & Bond (DMG, Hamburg, Germany) for 20 s, light curing (Elipar™ 2500 Halogen Curing Light, 3M ESPE, Seefeld, Germany) for 20 s. Bonding with Multilink Automix. The use of the adhesive primer on the air-abraded surface served as positive control as it has been shown to be effective on PEEK in a previous study [12]
PreLu5	Air-abrasion with Rocatec Pre at 0.25 MPa for 15 s was used, then cleaning with compressed air for 15 s. Application of the adhesive primer Luxatemp Glaze & Bond with storage under a lightproof box for 5 min, light curing for 20 s. Bonding with Multilink Automix. It was assumed that with a longer residence time of the adhesive primer a better penetration into the surface of the substrates might occur resulting in improved bonding