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The influence of electron beam irradiation on the shear bond strength of glass-reinforced frameworks and veneer composites

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Abstract The bond between glass fibre framework and veneer composite can be achieved by silane coupling agents or by monomers that penetrate into a polymer network. However, it has been clinically demonstrated that his bond can fail. This study investigated whether electron beam irradiation improved the bond strength of fibre-frameworks and veneer composite with and without additional coupling agents.

On Co Planar specimens of woven glass-fibre mats veneer composite cylinders were bonded on a restricted area of Ø5 mm with and without recommended coupling agents. The StickNet/Sinfony system with an interpenetrating polymer network and the Vectris Frame/Adoro system with a silane coupling agent were used. The shear bond strengths (SBS) were determined after 24 hrs, electron beam irradiation (100 KGy) and after irradiation (100 KGy) inclusive 12.000 cycles of thermal loading (5°/55°C).

The controls without coupling agents and irradiation had the lowest median SBS (Vectris: 6.9 MPa, StickNet: 8.7 MPa). After irradiation (no coupling aggent) the SBS increased significantly (Vectris 22.5 MPa, StickNet 13.7 MPa). Thermal cycling changed the SBS of irradiated specimens slightly (Vectris: 21.1 MPa, StickNet:15.9 MPa). With the application of a coupling agent, the SBS was significantly higher than the controls (Vectris: 15.1 MPa, StickNet: 15.7 MPa). Additional irradiation did not ameliorate significantly the median SBS of Vectris, but of StickNet (20.0 MPa). Thermal cycling decreased the SBS of StickNet significantly (13.7 MPa) while the SBS of Vectris remained unchanged.

Conclusion: Electron beam irradiation can improve the bond strength between fibre-framework and veneer composite. In some cases a silane coupling agent can be avoided.

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1. Introduction

One factor who determines the clinical success of fibre-reinforced composite (FRC) restorations is a reliable bond between the fibre framework and its facing material. This bond based on a stiff framework and can be realised by an as much as possible high fibre content. It was shown in the literature that higher framework fibre volume enhances the fracture load of FRC-restorations [1,2]. The limit of an increased fibre volume is given by the anatomical situation.

Functional (occlusal) and esthetic reasons restrict the space of the framework. In these cases, the chemical bond between the fibre framework and veneer material influences more and more the clinical success [3].

This investigation focus on the bond strength between veneer materials and glass fibre frameworks. The frameworks consist of pre-impregnated glass fibres (Pre-pegs). This means that the fibre is embedded in a resin matrix [4]. The frameworks are formed manually or in vacuum pressure devices. They are cured using light and/or heat. Examples are the Stick and the Vectris system. After curing the frameworks are fitted using diamond burs. Depending on the system used, different methods are recommended by the manufactured to bond the veneer material on the fitted framework [5–7].

It was stated that a well cross-linked polymer matrix with high degree of carbon double bond convertion is difficult to adhere to dimethacrylate monomer resins [5]. There are different approaches to solve this problem. The Stick system

uses the interdiffusion of monomers into the polymer network [5,6]. Low molecular weight monomers dissolve the linear PMMA phase on the Stick FRC polymer matrix of the framework and offer bonds between framework and veneer composite. In contrast, Vectris needs a silane coupling agent to improve adhesion to the glass fibre framework [7]. The oxygen inhibition layer of the matrix could contribute to the bond, but is mostly removed during the fitting process. Therefore, the application of a silane coupling agent is necessary [7]. Despite all manufacturing efforts using interpenetrating networks, functional monomers or silane coupling agents, it was shown clinically that the bond to composite could fail [3]. One reason may lay in the fact that the recommended procedures are comprehensive and difficult so that handling errors occur. This was for example one reason for the failure rate of the former Targis/Vectris system [3].

This study investigated whether electron beam irradiation [8,9] can enhance the bond between glass fibre frameworks and their veneering composites with and without coupling agents. Cross-linking between polymers and glass with radiation is a method, which is widely used in industry, but is up now poorly introduced in dentistry.

2. Material and methods

A shear bond strength test (SBS) was carried out following ISO 11405 [10]. Composite cylinders were fixed on rectangular glass fibre-reinforced composite panels, and then sheared off (Fig. 1).

2.1. Vectris Frame group

Rectangular specimens (n = 60) made of the woven glass fibre mat Vectris Frame with a length of 20 mm, a width of 10 mm and a thickness of 3 mm were constructed. The Vectris Frame specimens were deep drawn in a vacuum pressure process and light-cured using the VS-1 device (Ivoclar-Vivadent, Schaan, FL). Care was taken that the surface was plan parallel.

2.1.1. Vectris Frame control groups

Thirty Vectris Frame plates were randomly chosen. They were steam cleaned and shortly dried using pressed air. Without any pre-treatment, composite cylinders were made of the veneer material Adoro (Ivoclar-Vivadent, Schaan, FL) The cylinders had a diameter of 5 mm and a height of 3 mm They were directly light-cured onto the plates using the Targis Quick device (Ivodar-Vivadent, Schaan, FL) and finally cured using light and heat in the Targis Power upgrade device (25 min).

2.1.2. Vectris Frame groups with silane coupling agent

Thirty Vectris Frame plates were covered with a punched tape so that a restricted area of Ø5 mm was created. This area was sandblasted with 50 μm grain size Al_2O_3 for 10 s and 2 bar. The chosen parameters cleaned the surface and avoided damages of the glass fibres. Residual Al_2O_3 grains were removed using an ultra sonic device (Sonorex; AD Jensen, Zwolle, NL). The restricted area was coated with the silane-coupling agent Wetting agent (Ivoclar-Vivadent, Schaan, FL) and dried for 1 min. The veneer material Adoro was directly placed onto the pre-treated surface using a steel mould (Fig. 2). A cylinder of Ø5 mm and a height of 3 mm were constructed. The curing process was carried out using firstly the Targis Quick device (10 s light-curing per layer) and finally the Targis Power upgrade device.

2.1.3. StickNet control groups

In order to diffuse a monomer into the polymer network, the StickNet fibre woven mats were wetted with Heliobond (Ivoclar-Vivadent, Schaan, FL) resin for 10 min. Every

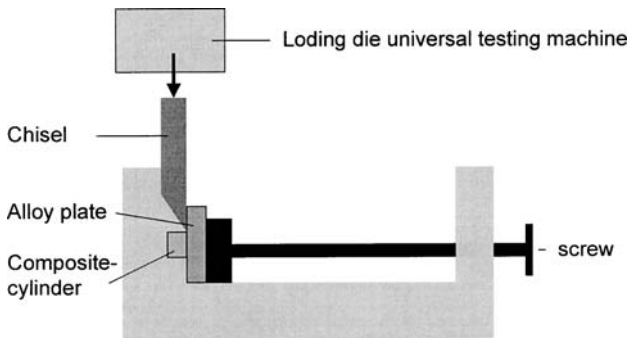

Fig. 1 Assembly for shear bond testing.

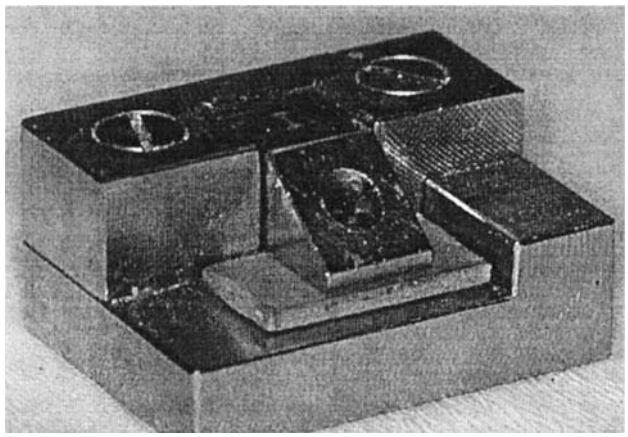

Fig. 2 Steel mould to form composite cylinders.

light was excluded during the wetting procedure, which is recommended by the manufacturer [5]. The low molecular weight monomers dissolved the linear PMMA phase on the Stick FRC polymer matrix of the frameworks and offer bonds to other fibre mat layers or veneer materials [5]. The wetted fibre mats were placed in a silicon mould and plates with a length of 20 mm, a width of 10 mm and a thickness of 3 mm were constructed. The plates were light-cured for 60 sec using Elipar Trilight (3M-Espe, Seefeld, G).

Thirty StickNet plates were randomly chosen. Without any pre-treatment composite cylinders made of the veneer material Sinfony (3M-Espe, Seefeld, G) were constructed. The cylinders had a diameter of 5 mm and a height of 3 mm They were directly light-cured onto the plates using the light-curing device Espe Visio Alpha (3M-Espe, Seefeld, G) and finally cured under vacuum in the Espe Visio Beta device (15 min).

2.1.4. StickNet groups with Sinfony activator

On thirty StickNet plates were restricted areas of Ø5 mm diameter constructed using a punched tape. The area was sandblasted for 10 s with 50 μm grain size Al_2O_3 and steam cleaned. Sinfony Activator was applied and dried for 60 s.

Cylinders made of the veneer material Sinfony were cured onto the plates as described above.

2.2. Post-curing with irradiation and aging

All groups of Vectris and StickNet with and without pre-treatment were randomly assigned in subgroups of 10 specimens each. Subgroup one was stored for 24 hrs. in distilled water at 37°C and then sheared off. This was the control. The second group was electron beam irradiated in five steps of 20 kGy (2 s) resulting in a total irradiation of 100 kGy (10 MeV) using a Rhodotron electron beam accelerator (BGS beta gamma service, Saal a.D., G). The third subgroup was irradiated with 100 kGy and then 12.000 times thermally cycled at 5°C/55°C in distilled water.

2.3. Shear bond strength test

The shear bond strength (SBS) was determined following ISO 11405. The plates with the composite cylinders were fixed in a shear device so that the shear chisel hit vertically (Fig. 1) the cylinder. Care was taken that the space between chisel and alloy panel was as close as possible to avoid a cantilever effect on the adhesive surface. The chisel was moved down using a universal testing machine Zwick 1446 (Zwick, Ulm, G) with a cross-head speed of the 1 mm/min. The SBS was calculated using the formula: $\sigma_{shear} = F_{max}/A$ [MPa].

2.4. Analysis of the fractured area

The type of fractured area (cohesive, adhesive, mixed) was analysed using a reflected-light microscope Stereoscan (Zeiss, Jena, G). A fracture was defined to be adhesive if more than 75% of the fibre framework (of the restricted area) was visible. A cohesive fractured area showed more than 75% of the surface covered with veneer composite. As mixed fractured area all cases were defined which could not be distributed to adhesive or cohesive fracture mode.

2.5. Statistics

Median and 25%/75% percentiles were calculated. One-way ANOVA was used to detect statistical significant differences. The level of significance was set at $= 0.05$.

3. Results

3.1. Vectris Frame/Adoro SBS

The lowest SBS was found for Vectris Frame/Adoro specimens without any pre-treatment or irradiation (6.9 MPa) (Fig. 3). Significantly higher SBS (15.1 MPa) had specimens, which were pre-treated using a silane-coupling agent (Wetting agent). However, additional irradiation (19.0 MPa) or thermal cycling (17.8 MPa) did not change significantly the SBS of silaneted specimens. It is remarkable that the highest SBS was measured for specimens who were not silaneted but irradiated with electron beams (22.5 MPa). Even thermal cycling reduced slightly the values of the irradiated specimens to 21.1 MPa.

3.2. StickNet/Sinfony SBS

As expected, the SBS was found to be lowest for specimens without any pre-treatment or irradiation (8.7 MPa) (Fig. 4). Significantly higher SBS values were found, if the specimens were pre-treated with Sinfony Activator (15.9 MPa) or irradiated (13.7 MPa). The highest value was found for combination pre-treatment and irradiation (20.0 MPa), but significantly lowered after thermal cycling (15.2 MPa). The irradiated specimens (no pre-treatment) showed slightly increased SBS (15.1 MPa).

3.3. Analysis of the fractured area

All types of fracture modes occur (Fig. 5, Fig. 6). No cohesive fractured area was found with not pre-treated groups after 24 hrs and after thermal cycling. There was no difference between Vectris/Adoro or StickNet/Sinfony.

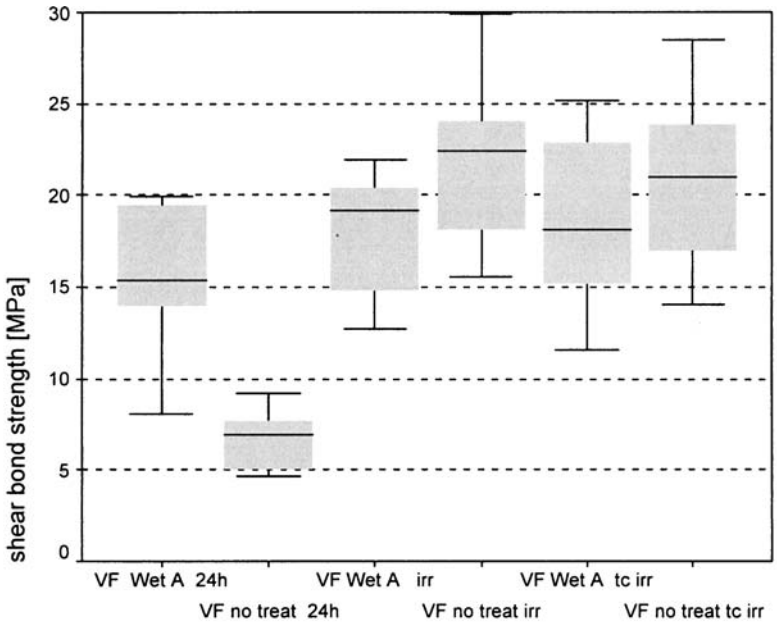

Fig. 3 Shear strength test: Vectris Frame/Adoro and different pre-treatment methods. VF = Vectris Frame; Wet A = Wetting agent; 24 h = test after 24 hrs water storage; no treat = non pre-treatment; irr = test after electron beam irradiation; tc irr = test after irradiation and thermal cycling.

	Wet A 24h	No Treat 24h	Wet A irr	No treat irr	Wet A irr tc
No treat 24h	0.000				
Wet A irr	n.s.	0.000			
No treat irr	0.003	0.000	n.s.		
Wet A irr tc	n.s.	0.000	n.s.	n.s.	
No treat irr tc	0.022	0.000	n.s.	n.s.	n.s.

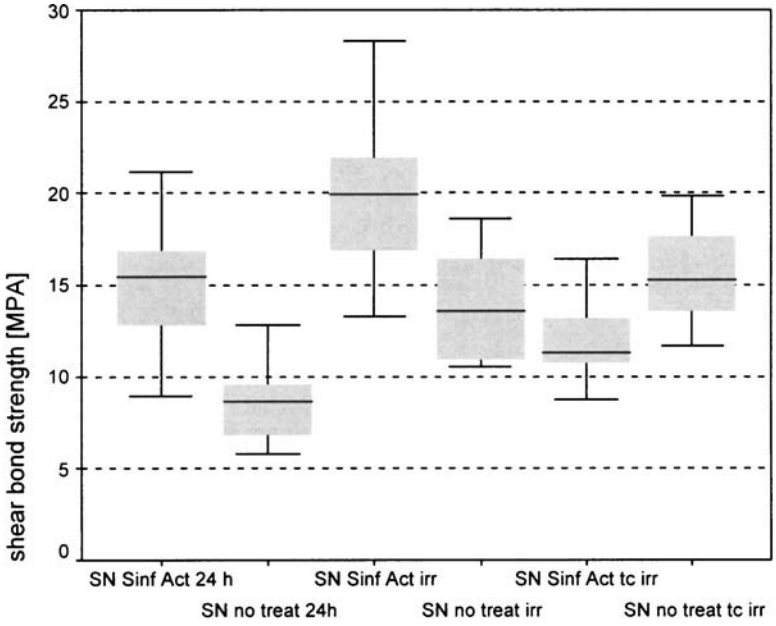

Fig. 4 Shear strength test: StickNet/Sinfony and different pre-treatment methods. SN = StickNet; Sinf Act = Sinfony Activator; 24 h = test after 24 hrs water storage; no treat = non pre-treatment; irr = test after electron beam irradiation; tc irr = test after irradiation and thermal cycling.

	Wet A 24h	No Treat 24h	Wet A irr	No treat irr	Wet A irr tc
No treat 24h	0.000				
Wet A irr	n.s.	0.000			
No treat irr	0.003	0.000	n.s.		
Wet A irr tc	n.s.	0.000	n.s.	n.s.	
No treat irr tc	0.022	0.000	n.s.	n.s.	n.s.

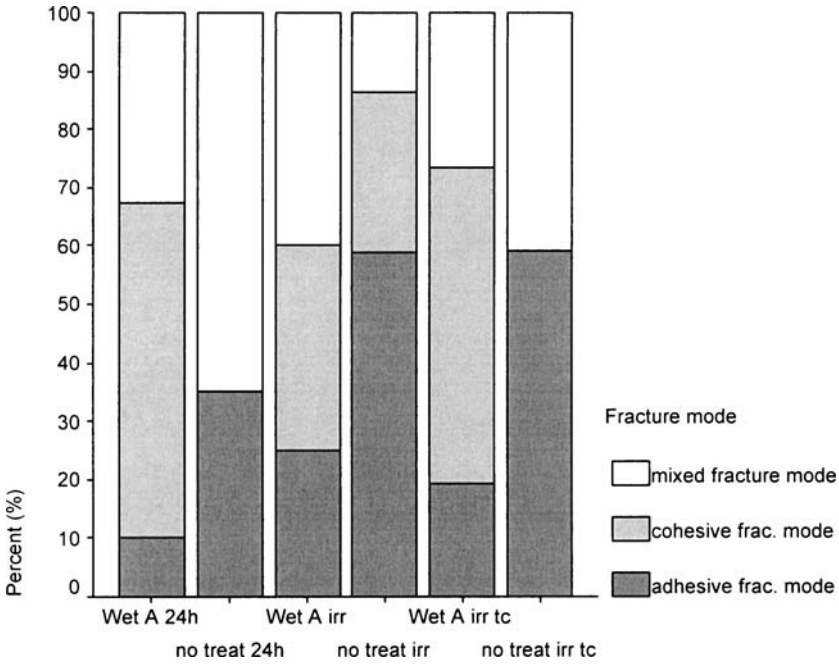

Fig. 5 Fracture mode of the shear strength test: Vectris Frame/Adoro and different pre-treatment methods. VF = Vectris Frame; Wet A = Wetting agent; 24 h = test after 24 hrs water storage; no treat = non pre-treatment; irr = test after electron beam irradiation; irr tc = test after irradiation and thermal cycling.

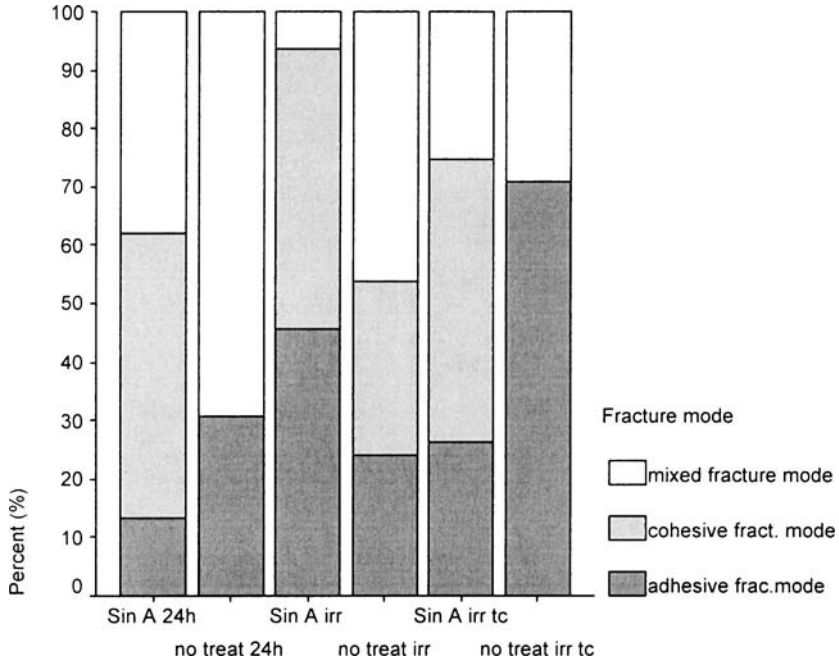

Fig. 6 Fracture mode of shear strength test: StickNet/Sinfony and different pre-treatement methods. SN = StickNet; Sinf Act = Sinfony Activator; 24 h = test after 24 hrs water storage; no treat = non pre-treatment; irr = test after electron beam irradiation; irr tc = test after irradiation and thermal cycling.

4. Discussion

Bond strength tests can be performed as shear bond test (SBT) or tensile strength tests (TST) [11,12]. Some authors favourites the TST because this test investigates closer the adhesive properties of a bonding system than the SBT [11]. TST is difficult to manage. The specimens had to be fixed in grips or clamps, which are, connected which a universal joint [11,12]. It is necessary that the specimens can move freely during the load application in order to avoid bending- or shear stress on the bond. While a safe fixation is thinkable with little metal specimens, the brittle character of composites did not allow a use of clamps or grips. With increasing bond strength it can be expected that the inner strength of the specimen do not withstand the force necessary for fixation. Fracture occurs in the specimen and the measured value does

not reflect the adhesive bond strength. Therefore, a SBT was carried out, which is easier to handle for brittle small specimens. However, the SBT has some limitations. If the bond strength is very high during, the inner shear stress of all parts will increase. More and more the shear stress cannot be transferred to the border between adhesive and alloy [11]. This results in an increasing inner stress of the fibre framework and/or the composite cylinder. Now, not the adhesive properties than the inner strength of specimens are responsible for the breaking strength. It is therefore expected that cohesive fracture area will occur [11]. In this study these fracture types were observed in more than a third indicating that high inner stress of the composite was present.

Based on the estimation that the maximal oral load in the anterior area is about 250 N and that the bonding area of a composite veneer of a crown may be 25 mm^2 a bond strength of 10 MPA should be reached [13] ($\sigma = F_{250\,N}/A_{25\ mm^2}$ [MPa]. Without any pre-treatment a direct bonding of the tested veneer composites and the fibre frameworks did not reach this level. Clinically it must be expected that such a bond fail. The pre-treatment methods, which are recommended, improve the bond strength significantly. Vectris Frame benefits from a silane coupling agent and StickNet from the Sinfony Activator Monomer.

Meanwhile it was published that the Sinfony Activator liquid does not dissolve the linear PMMA phases of Stick the polymer matrix and that EBS Multilink should be used [5]. Despite this possible problem the adhesion between StickNet and the veneer composite Sinfony was not an affected in this study, what is demonstrated by the fact that un-pretreated control group and pre-treated group had significantly different shear strength values. The aim of this study was not to compare product A with B, but to investigate the influence of electron beam irradiation on the bond of different fibre framework/veneer composite systems. Electron beam irradiation is method described to change the mechanical properties of polymers [8, 9]. It is widely used with polymers like polyethylene, polycarbonate or polysulfone [8]. Generally, two types of reactions exist with electron beam irradiation, which compete during radiation: chain linkage or breakage [14,15]. Charlesby [8,14] stated in 1953 "the degree of cross-linking produced in these polymers is proportional to the radiation dose over a wide range of values". However, it was shown [15] that for low dose radiation Charlesby' law does not agree with the experimental results. Further investigations show that cross-linking of polymers with radiation does not follow an easy dose-reaction relationship. It depends on the structure of the polymer, functional groups, and temperature during the investigation and on the irradiation parameters like dose rate or accelerating of the electrons [15–18] Remarkable were the results of the irradiated un-pre-treated specimens. Their bond strength values were the highest for the Vectris Frame/Adoro system. Untreated StickNet/Sinfony-specimens reached after irradiation the level of pre-treated specimens. Even after 12.000 thermal cycles the bond strength stay on a high level and statistical significant differences were not observed for both materials.

The effect of electron irradiation depends on two processes. First, the inner strength of dental composites is enhanced [18]. The high applied energy allows increasing the conversion rate of the composite. The inner strength increases and withstands easier the shear stress during the SBT. On the other side it seems the irradiation enables bonds between the fiberglass surface and the composite resin. It could be assumed that the sandblasting process denudes the pre-impregnated glass fibers. The blank fibers come in contact with the composite where catalyst by the irradiation new bond was achieved. It is not clear why Vectris Fibers seems to bond better than the StickNet fibers. Different glass types and composite resins could be a reason. Another aspect is the PMMA content of the StickNet network. PMMA showed chain breakage at higher electron beam dose rates [19]. The used dose could be too high for the Stick network.

Attempts with a lower dose rate of e.g. 30 kGy should be performed [18,19].

This investigation demonstrated that electron beam irradiation could improve the bond strength between glass fiber frameworks and veneering composite. This method can only be used lab-side. Due to the great effort the irradiation makes no sense for a single crown. Only pre-fabricated FRC parts as for example posts or inlay fixed partial denture frameworks can be economic. Despite this limits, electron beam irradiation demonstrates that glass-fiber re-inforced composites can still become a valuable tool to treat patients successfully.

Conclusion

The bond strength between glass fiber surfaces and composites can be improved using electron beam irradiation. In some cases the silane-coupling agent can be avoided.

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