

UNIVERSITÄTSKLINIKUM JENA

POLIKLINIK FÜR ZAHNÄRZTLICHE PROTHETIK UND WERKSTOFFKUNDE Direktor: Univ.-Prof. Dr. H. Küpper Bereich Werkstoffkunde und Technologie

(University of Jena, Germany Department of Dental Prosthetics and Materials Science Director: Univ.-Prof. Dr. H. Küpper Materials Science and Technology Division)

In-Vitro Investigation of the Bond Strengths of "Hybrid-Ceramic" Blocks to Resin Cements

By Dr. A. Rzanny and Dr. R. Göbel Test report for Shofu Dental GmbH 11 pages, incl. 5 tables and 5 illustrations Jena, Germany, 28 March 2017

1. Objective

The objective of this investigation was to test the bond strengths of hybrid-ceramic blocks made by four manufacturers to different resin cements. The influence of the use of different primers and surface conditioning methods was also tested.

2. Materials, Methods and Test Conditions

2.1 Materials

The hybrid-ceramic blocks, resin cements, primers, and instruments or systems for surface roughening used in this study are listed in Table 1, along with their batch numbers.

Table 1

HC Blocks						
	Batch No.	Manufacturer				
Lava Ultimate	N676654	3M ESPE				
CeraSmart	1601251	GC				
Block HC	111501	Shofu Dental				
Vita Enamic	65220	VITA				
Resin Cements						
ResiCem	091549	Shofu				
BeautiCem	011630	Shofu				
Primers						
Cera Resin Bond	121519	Shofu				
HC Primer	101502	Shofu				
visio.link	154126	bredent				
Instruments for Surface Roughening						
Dura-Green Stones	0116003	Shofu				

Diamonds	Q37607	Meisinger			
Composite Material					
Ceramage	011640	Shofu			

2.2 Methods and Test Conditions

Compressive Shear Strength

The bond strengths were evaluated by compressive shear strength tests with the aid of a Z005 universal testing machine (Zwick/Roell).

The four longitudinal sides of each hybrid-ceramic block were used for testing.

In the first part of the compressive shear strength tests, these surfaces were initially roughened with Dura-Green Stones ("initial situation"). For comparison, one bond series each was tested after sandblasting with corundum and after roughening with diamonds. For all subsequent test series, initial surface roughening with diamonds was selected for reasons of clinical relevance.

First, different primers and resin cements were tested in combination with Block HC hybrid-ceramic blocks. After surface conditioning with three primers – Cera Resin Bond (only Bond 2), HC Primer or visio.link – following the manufacturers' instructions, two resin cements – ResiCem or BeautiCem – were applied.

Based on the results of these tests, and in consultation with Shofu, the primers HC Primer and visio.link and the cement ResiCem were selected for the comparative evaluation of four different hybrid ceramics. All blocks were initially roughened with diamonds.

To measure the hybrid-ceramic/resin-cement bond strength, the resin cement was applied to the primed surface in a thin layer, approx. 50-100 μ m in thickness. After 10 min of chemical curing, the cement was light-cured for 90 s in a Dentacolor XS unit. Then the composite material Ceramage was built up in the form of a cylinder with the help of a metal ring (diameter: 5 mm; height: 2 mm) and light-cured for 90 s in a Dentacolor XS unit. Subsequently, the ring was removed, and the composite was light-cured for 90 s.

The specimens were subjected to two different aging processes:

- 1. Initial value (base value): 24 h storage in water at 37°C
- 2. Artificial aging by thermocycling: 25,000 thermocycles (TC) between 5 and 55°C

The compressive shear strength tests were performed using a Z005 universal testing machine (Zwick). The crosshead speed was 1 mm/min. Five specimens were made for each series.

The following material combinations were tested:

Initial situation: *Dura-Green Stones* Block HC with HC Primer and ResiCem Block HC with HC Primer and BeautiCem Block HC with Cera Resin Bond and ResiCem Block HC with Cera Resin Bond and BeautiCem Block HC with visio.link and ResiCem Block HC with visio.link and BeautiCem

Initial situation: *Dura-Green Stones* Block HC with HC Primer and ResiCem Initial situation: Sandblasting with corundum (110 μm; 3 bar) Block HC with HC Primer and ResiCem Initial situation: *Red diamond* Block HC with HC Primer and ResiCem

Initial situation: Diamond

Block HC with HC Primer and ResiCem Block HC with visio.link and ResiCem Lava Ultimate with HC Primer and ResiCem Lava Ultimate with visio.link and ResiCem CeraSmart with HC Primer and ResiCem Vita Enamic with HC Primer and ResiCem Vita Enamic with visio.link and ResiCem

3. Results

Tables 2 to 5 show the bond strengths measured after storage of the specimens for 1 day and after 25,000 thermocycles (TC). The results are also compared graphically in Figures 1 to 5.

Table 2: Compressive shear strengths of the bonds between Block HC, two different resin cements and three different primers after storage for 1 day and after 25,000 TC

Initial situation: DuraGreen Stones

		Compressive Shear Strength [MPa]			
	Primer	1 day		day 25,000 TC	
		ResiCem	BeautiCem	ResiCem	BeautiCem
		SD()	SD ()	SD()	SD ()
Block HC	Cera Resin Bond	31.9 (2.0)	32.0 (1.2)	31.6 (1.5)	31.1 (1.2)
Block HC	HC Primer	35.0 (2.0)	35.2 (1.0)	34.5 (1.3)	34.0 (0.8)
Block HC	visio.link	35.4 (1.6)	34.3 (1.8)	34.5 (0.7)	33.5 (0.7)

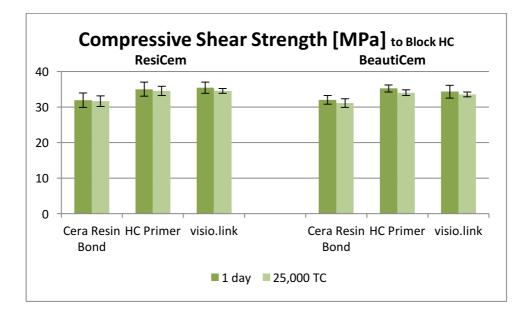


Fig. 1: Compressive shear strengths of the bonds between Block HC and ResiCem or BeautiCem, in combination with three different primers, after storage for 1 day and after 25,000 TC

Table 3: Compressive shear strengths of the bonds between Block HC and ResiCem

 resin cement following different surface pretreatments

				Compressive Shear Strength [MPa]	
Hybrid Ceramic	Primer	Resin Cement	Pretreatment	ResiCem SD ()	ResiCem SD ()
				1 day	25,000 TC
Block HC	HC Primer	ResiCem	Sandblasting with corundum	35.3 (1.0)	33.7 (1.3)
Block HC	HC Primer	ResiCem	Dura-Green	35.0 (2.0)	35.2 (1.0)
Block HC	HC Primer	ResiCem	Diamond	32.1 (1.9)	31.7 (2.8)

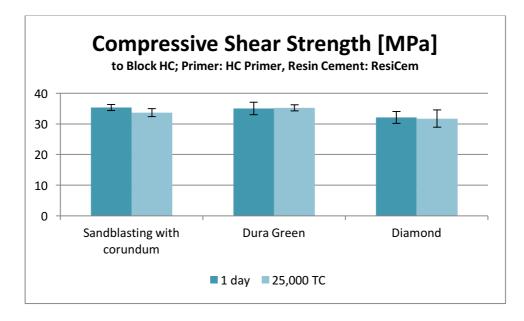


Fig. 2: Compressive shear strengths of the bonds between Block HC and ResiCem following three different surface pretreatments – sandblasting with corundum, roughening with Dura-Green or diamonds – and the use of HC Primer, after 1 day and after 25,000 TC.

Table 4: Compressive shear strengths of the bonds between different hybrid ceramics

 and ResiCem resin cement following the use of different primers, after storage for 1 day

		Compressive Shear Strength [MPa]			
	Resin Cement	1 day			
		HC Primer	Cera Resin Bond	visio.link	
		SD ()	SD ()	SD ()	
Block HC	ResiCem	32.1 (1.9)	32.1 (1.4)	32.7 (2.2)	
Lava Ultimate	ResiCem	34.0 (2.0)		34.0 (2.0)	
CeraSmart	ResiCem	36.3 (1.6)		37.3 (2.3)	
VITA Enamic	ResiCem	26.0 (2.0)		25.3 (2.4)	

Initial situation: Diamond

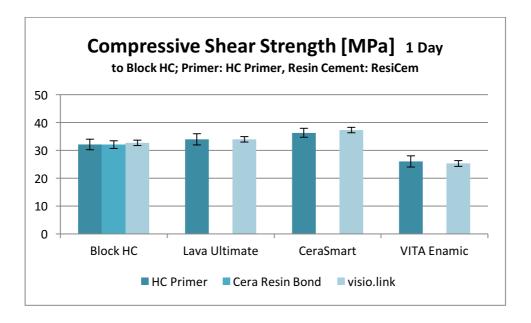


Fig. 3: Compressive shear strengths of the bonds between four different hybrid ceramics – Block HC, Lava Ultimate, CeraSmart, VITA Enamic – and ResiCem following the use of different primers, after 1 day

Table 5: Compressive shear strengths of the bonds between different hybrid ceramics

 and ResiCem resin cement following the use of different primers, after 25,000 TC

		Compressive Shear Strength [MPa]			
	Resin Cement	25,000 TC			
		HC Primer	Cera Resin Bond	visio.link	
		SD ()	SD ()	SD()	
Block HC	ResiCem	31.7 (2.8)	32.5 (2.6)	32.5 (1.5)	
Lava Ultimate	ResiCem	33.5 (1.2)		33.4 (1.4)	
CeraSmart	ResiCem	34.7 (1.2)		34.6 (2.0)	
VITA Enamic	ResiCem	20.8 (0.7)		19.3 (1.0)	

Initial situation: Diamond

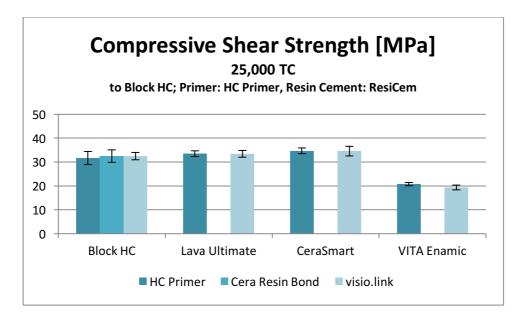


Fig. 4: Compressive shear strengths of the bonds between four different hybrid ceramics – Block HC, Lava Ultimate, CeraSmart, VITA Enamic – and ResiCem following the use of different primers, after 25,000 TC

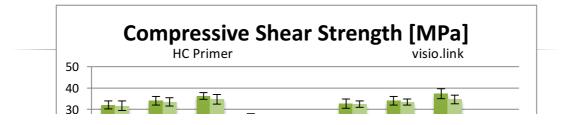


Fig. 5: Compressive shear strengths of the bonds between four different hybrid ceramics – Block HC, Lava Ultimate, CeraSmart, VITA Enamic – and ResiCem following the use of HC Primer and visio.link, after 1 day and after 25,000 TC

4. Additional Remark

For VITA Enamic, the manufacturer has specified a different conditioning method. VITA Enamic should be pretreated by hydrofluoric acid etching, followed by silanisation. Then the resin cement can be applied as described above. After this pretreatment, bond strengths of approx. 30 MPa were measured.

5. Summary

The first tests with Block HC did not show any differences in bond strength between the two different resin cements, ResiCem and BeautiCem. The bond strengths of all material combinations remained stable after 25,000 thermocycles (TC). The bonds achieved after priming with HC Primer and visio.link were equally good, whereas the values achieved using Cera Resin Bond were slightly lower.

The comparison of different surface pretreatments – sandblasting with corundum, roughening with Dura-Green Stones or diamonds – showed that compressive shear strengths were 2 to 3 MPa lower after the use of diamonds, as compared to sandblast-ing or Dura-Green. Nevertheless, roughening with diamonds was used as the standard pretreatment in the subsequent tests, since this method is most clinically relevant.

Based on the results of these tests, and in consultation with Shofu, the primers HC Primer and visio.link and the cement ResiCem were selected for the ensuing comparative evaluations.

Following the surface conditioning procedure used in this investigation, VITA Enamic showed the lowest bond strengths of all hybrid ceramics compared, and failures were adhesive, in contrast to the three other hybrid ceramics, which exhibited cohesive failures after testing. This is why VITA Enamic should be conditioned only as recommended by the manufacturer. The reason is its composition. VITA Enamic consists of a sintered ceramic network infiltrated with a polymer material; as a result, the inorganic ceramic content is 86 % by weight, and the organic polymer content is only 14 % by weight. The three other hybrid ceramics are essentially CAD/CAM composite materials, with ceramic particles embedded in a very hard polymer matrix. So these three products can be effectively cemented after the use of resin primers.

Regarding the bond strength of Block HC hybrid ceramic to ResiCem resin cement, there were no significant differences between the three different primers, neither after one day nor after 25,000 TC. The bond strengths of Lava Ultimate and CeraSmart to ResiCem did not show any differences between HC Primer and visio.link either. Minor differences of up to approx. 4 MPa between the three composite materials with embedded ceramic particles are probably due to different compositions.

6. Conclusion

Basically, both HC Primer and visio.link can be recommended as primers for Block HC, Lava Ultimate and CeraSmart hybrid ceramics. All combinations showed very good bond strengths, ranging from 32 to 37 MPa. Compressive shear strength testing of these three products resulted in cohesive failures, recognisable by the fact that polymer material was torn out of the blocks.

VITA Enamic, by contrast, needs to be pretreated like a ceramic material before cementation, i.e. by hydrofluoric acid etching and silanisation, since it mainly consists of a sintered ceramic network.