23rd International Conference
ENGINEERING MECHANICS 2017
Svratka, Czech Republic, 15 – 18 May 2017

TESTING THE STRENGTH OF THE ADHESIVE CONNECTION IN SPECIMEN - MONOTONIC TENSILE AND SHEAR WITH UNDER VARIABLE LOAD

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Abstract: *Ceramic implants can create original terms of occlusion in mouth. The results obtained for zirconium dioxide as the ceramic material used in implantology. Describes the technology of the blank (trabecular) and research glued connection of two trabecular zirconia 3M ESPE LAVA with batch number:508150. Performed with the help of two different dental adhesive properties glue named: 3M ESPE RelyX and Kerr Maxcem Elite. Monotonic tensile tests were carried out for two groups of 15 samples and the shear tests were adopted one group of 11 samples. The results obtained are characterized by high reproducibility in the sample geometry made of zirconia, as confirmed by the p Shapiro-Wilk test (p = 0.06). It showed no statistical differences in an attempt to monotonic tensile Student's t test (p = 0.608).*

Keywords: Zirconium dioxide, Static stretching tests, Adhesive, Dentistry.

1. Introduction

Zirconium dioxide has versatile applications. It is used as a heavy-duty material in engineering but also in dentistry: for production of implants designed to restore the original occlusion of teeth after a major damage to, or loss of, the tooth crown. Featuring very good mechanical strength and hardness, this zirconium dioxide continues to replace conventional materials providing similar performance, such as gold alloys (Rizkalla, 2004; Guazzato, 2004; Yilmaz, 2007 and Manicone, 2007). Both these materials have good biocompatibility but the advantage of zirconium dioxide over other materials is its natural bony colour. The present production of very sophisticated dentures, such as 6-point full-ceramic bridges, is possible only due to excellent strength of the materials involved: up to 1200 MPa. (Raigrodski, 2006; Edelhoff, 2008). Today, suppliers of dental materials offer hybrid (multi-component) self-adhesive cements featuring better strength than unary adhesives (Watanabe, 1994).

The purpose of the work was to compare two types of adhesives featuring the same adhesive properties, one much more expensive than the other. An additional aim was to show differences between bonds and between denture materials in terms of their mechanical properties.

2. Methods

2.1. Zirconium dioxide

The ceramic material we tested was 3M Zirconium Lava commonly used for production of crowns and bridges (designed using CAD/CAM software). The advantages of the material include good strength, very natural appearance, translucency, biocompatibility and absence of metals in its structure.

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We cut the material supplied by the manufacturer nominal dimensions 25 mm x 16 mm x 90 mm and batch number: 508150. He was cutted with a circular saw (Isomet 5000 from Buehler, Poland) into 25 x 16 x 1.87 mm blocks. Cutting material was cooled with distilled water. Then, we cut each block into 9 blanks sized 1.87 x 1.87 x 15 mm with a laser cutter (Alfalas WS, Poland). The beam power settings protected the material against overheating. See Fig. 1 for the blank cutting design before sintering.

Next step was sending the blanks (trabeculae) to a manufacturer-certified laboratory for sintering. This process was performed on a device called The Lava Furnace 200 [POLAND]. The samples were sintered at 1400 °C for 8 hours, which caused material volume shrinkage by 20 %, on average (according to the material manufacturer). After sintering, the material is snow-white, much harder and more resistant to 3-point bending. See Fig. 1b for the geometry of the blanks after sintering, now measuring 1.5 x 1.5 x 12 mm.

a) b)

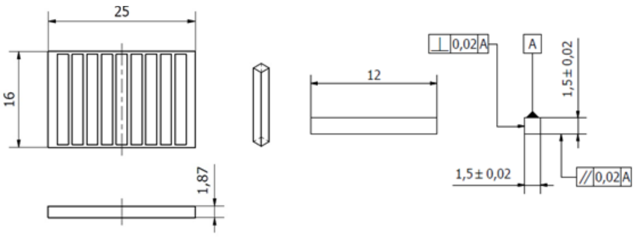


Fig. 1: a) Blank cutting; b) Blank geometry and dimensions.

2.2. Tested adhesives

We tested the strength of bond provided by two adhesives: 3M ESPE RelyX U200 Automix with batch number: 56895 and Kerr Maxcem Elite with batch number: 34055. Adhesives used were characterized by similar: consistency of the product and similar aesthetic properties of adhesive connection, which formed. These adhesives are used in partner dental clinics. The former is a latest generation self-adhesive cement. The adhesives are hardened after application between two surfaces with 420 – 480 nm UV light. We used the Satelec Mini Led Black UV light with 1.250 mW/cm2 power. The exposure time was 10 seconds in each instance.

In Tab. 1 shown summary of the study provided the monotonic tensile strength, shear fatigue test and the shear strength.

Tab. 1: Summary of the research strength of the joint adhesive.

Tests name	Number of specimen	Group name	Dental adhesive	Glue data
Monotonic tensile strength testing	15	A1	3M ESPE RelyX	Made in Germany by 3M ESPE AG Seefeld
	15	A2	Kerr MaxCem Elite	Made in Italy by Kerr Italia S.r.l. Scafati

2.3. Strength of bond

We tested the tensile strength of the bond on the blanks prepared beforehand (zirconium dioxide trabeculae). Each blank was bonded to two custom-made stainless steel plates using a pre-made special device for axially bonding the zirconia samples.

First, the samples were subjected to a degreasing formulation Emulsol RN-1. Then glued 15 samples adhesive 3M ESPE RelyX U200 Automix (A1) and 15 samples of adhesive Kerr ^ maxc Elite (A2). Using a previously described UV lamp company Satelec Mini LED Black. The exposure process lasted 10 seconds. The last step was the mechanical removal of excess adhesive dental device Dremel 200 of the grinding wheel spindle alumina.

Before each test, we checked each sample for bond quality under a stereoscopic microscope (OPTA-Tech MN-800). The thickness of the bond was 0.05 ± 0.002 mm. See Fig. 3 for the design of the bonded sample.

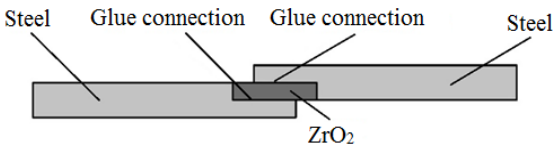


Fig. 2: Test sample cross-section.

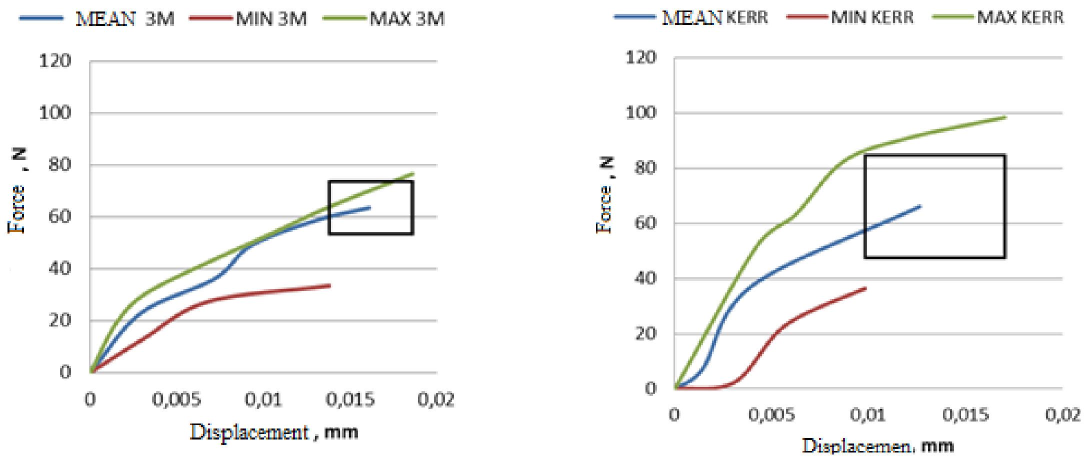
3. Results and analysis

Compared their pattern to the normal distribution with the Shapiro-Wilk test, compared the bond strengths using the t Student test for independent variables. The statistical comparisons relied on the critical value of $p < 0.05$ for checking whether the variation of the results was statistically significant. We measured the samples to ensure that they were uniform, i.e., to exclude bonding area variability as a possible influence on the bond strength. The distribution of the dimension values was statistically significantly similar to the normal distribution, which we verified using the Shapiro-Wilk test (Tab. 2).

Tab. 2: Descriptive statistics of sample dimensions and Shapiro-Wilk test results.

	Mean	SD	Relative SD [%]	Value p
Mean x [mm]	1.47	0.02	1.51	0.819
Mean y [mm]	1.47	0.02	1.54	0.304
Cross-section area A_0 [mm^2]	2.16	0.06	2.58	0.06

In Tab. 3 the tensile strengths of the bonds formed by the two adhesives. The mean destructive forces were 63.5 N for RelyX and 66.8 N for Maxcem, with the mean stress of approx. 7 MPa for the two adhesives. The results were widely scattered for the both adhesives: up to 15.9 % and 27.9 %, respectively. Results distribution destructive force as the minimum and maximum waveforms and standard deviation values obtained from the samples tested. Fig. 3 presented Values course force in three point bending test.



a) b)

Fig. 3: Values course force max, min, average, along with a distribution that for: a) 3M ESPE, b) Kerr Maxcem Elite.

Tab. 3: Test results for bonds formed by 3M ESPE RelyX U200 and Kerr Maxcem Elite.

Adhesive	Bonded area [mm^2]	Mean force [N]	Mean stress [MPa]	SD [N]	Relative SD [%]
3M ESPE RelyX U200	9	63.5	7.05	10.1	15.9
Kerr Maxcem Elite	9	66.8	7.42	18.6	27.9

We checked the results statistically, with the Shapiro-Wilk test, for similarity to the standard distribution, and the results indicated that the distribution was indeed standard (see Tab. 4). The t Student test of the mean results achieved during the test did not reveal any statistically significant differences between the tensile strengths of, or stresses in, the bond between the blanks. Statistics for bonds formed by 3M ESPE RelyX U200 and Kerr Maxcem Elite

Tab. 1: Results statistically, with the Shapiro-Wilk and t-Student tests.

Adhesive	Shapiro-Wilk test critical value	Shapiro-Wilk test p value	t Student test critical value	t Student test p Value
3M ESPE RelyX U200	0.910	0.248	-0.520	0.608
Kerr Maxcem Elite	0.939	0.518		

4. Discussion

Authors managed to obtain repeatable geometry of the blanks with the coefficient of variability of approx. 1.5 % for dimensions "x" and "y" (see Tab. 1). Next, our material processing method provided low variability of the bonding result and consistent bonding area. The typical methods of blank processing by researchers included cutting with diamond saw blade disks or machining. The mean monotonic tensile strengths of the two self-adhesive cements of the same generation that we tested were 63.5 ± 10.1 N for 3M ESPE RelyX U200 and 66.8 ± 18.6 N for Kerr Maxcem Elite. Our statistical review of the strengths did not reveal any significant differences between the adhesives ($p = 0.608$ for the t Student test). The strengths of the bonds had a 15.9 % scatter for the former adhesive and 27.9 % scatter for the latter one. Static stretch tests of the adhesive bond similar to ours, are described in paper (Poitevin, 2010). Its authors cut out trabeculae with transverse section area of 1 x 1 mm from natural teeth topped with all-ceramic crowns bonded with three adhesives. The bond strengths of the three tested adhesives were as follows: 51 ± 17.6 N for OptiBond FL, 43.7 ± 14.3 N for Clearfil SE and 28 ± 15.3 N for G-Bond. The coefficients of variability were 34.5 %, 32.7 % and 54.6 %, respectively. The scatter in these results is much wider than the scatter described in this paper.

5. Conclusion

The methods described in the references were strongly varied in terms of sample preparation, which is problematic from the point of view of comparability of the results. Another important factors that can possibly bear on the results are the size of adhesive squeeze-out (extra bonding area) and preparation of surface (which should be clean, degreased and free from foreign matter or oxidation). Our monotonic tensile strength test method has no matching equivalent in the existing sources. We recommend undertaking further research into the monotonic stretch of the adhesive bond to identify other possible factors affecting the denture lifetime and to minimize implant visual flaws and rework rates.

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