Bending strength of ceramic compounds bonded with silicate-based glass solder

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Article Information

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In the field of dental technology, the length of ceramic pontics is limited to avoid mechanical failure. To reduce thermal-induced residual stress within the ceramic, using smaller subcomponents and subsequent bonding with silicate-based glass solder may be a favorable approach. Thus, the bending strength of zirconia compounds bonded with different silicate-based glass solders was investigated. For this purpose, rectangular specimens made of zirconia were bonded by glass solder. Parameters such as the scarf angle (45° and 90°), two different glass solders, as well as the soldering process (pressure and surface treatment) were varied. All specimens were subjected to quasi-static four-point bending tests according to DIN EN ISO 843-1. Additionally, the quality of the glass solder connection was evaluated using µCT and fractography.
In the present study, zirconia compounds were sucessful bonded of zirconia compounds using silicate-based glass solder was. No significant differences in terms of bending strength were observed with respect to the different bonding parameters analyzed. The highest bending strength of 130.6 ± 50.5 MPa was achieved with a 90° scarf angle combined with ethanol treatment of the specimens before soldering and an additional application of a pressure of 2 bars in a dental pressure pot before subsequent soldering. Nevertheless, the bending strengths were highly decreased when compared to monolithic zirconia specimens (993.4 ± 125.5 MPa).

For dental applications, several oxide-ceramics have received great research interest over the past decade [1]. Superior physical, biological, aesthetic, and corrosion properties, compared to commonly used metals and their alloys, are leading to hopeful optimization of implants for dentistry [1, 2]. The major limitations faced while utilizing ceramics is their limited fracture toughness and susceptibility to notch-induced stress concentrations [2-4]. Specifically, high tensile stresses must be prevented. Therefore, the construction of complex parts must be done according to design guidelines [5, 6]. Furthermore, sintering of ceramic parts leads to thermal gradients over the cross section, which is enhanced with larger and more complex designs. Thermal residual stresses, which increase the fracture risk, are attributed to these thermal gradients [7, 8].

To avoid stress concentrations due to cross-sectional transitions and complex designs as well as residual stresses as a result of sintering or other technological operations, one useful approach can be the manufacturing and subsequent sufficient bonding of ceramic subcomponents [9].

Various approaches have been developed to bond ceramics with metals [10-12] and ceramics [11, 13–17]. Fernie et al. [2] divided these methods into mechanical and chemical, where the latter is sub-divided into solid-state and liquid-state bonding. Liquid-state bonding comprises brazing, adhesive gluing and glass-soldering. The latter method is characterized by high temperatures, where elements of the glass solder diffuse into the outer layer of the ceramic and constitute a material bond [10, 18].

Silicate-based glass solders are available to bond ceramics [10, 11, 19–21] and provide biocompatibility and high bonding strength [10, 22, 23]. In an earlier study, silicate-based glass solder showed higher bending strength (118 ± 33 MPa) than adhesive glue (30 ± 4 MPa) when alumina toughened zirconia was connected with titanium of grade 5 and tested in a four-point-bending test [10].

Börner et al. [11, 17] utilized laser soldering for connecting components of zirconia using different barium silicate-based glass solders. Bending strengths up to 227 ± 15 MPa were observed. They identified viscosity and wettability as crucial properties of the glass solder and ceramic materials respectively. Additionally, gap geometry, chemical composition of glass solder and ceramics, surface properties and ambient conditions must be considered to reach a firm bond [11, 20, 24–26].

Moreover, for practical application in the field of dentistry, a four-unit fixed dental prosthesis made of zirconia was manufactured using silicate-based glass solders. The stated hypothesis that soldering influences the fracture load and mode was not proven through this study [27].

Although this practical application has shown the feasibility of silicate-based glass solders to bond zirconia compounds, the literature lacks fundamental studies concerning the influence of surface treatments prior to soldering, ambient conditions during manufacturing and gap geometry. Thus, in the present experimental study, the bending strength of zirconia compounds bonded with different glass solders was investigated. Gap geometry in terms of scarf angle, and conditions of the manufacturing process during bonding were varied and their influence on bending strength was evaluated. Furthermore, µCT scans were used to evaluate the quality (gap thickness and homogeneity) of the glass solder bond. Additional microscopic analysis of the fracture surface was conducted after the four-point bending tests.

Materials and methods

Preparation of the test specimens. For the mechanical investigation in terms of four-point bending tests, twenty-eight compound specimens made of yttria stabilized tetragonal zirconia (Metoxit GmbH, Thayngen, Switzerland) were prepared according to the specifications of the standard DIN EN ISO 843-1 [28]. Measured dimensions of the specimens were 3.09 ± 0.26 mm in height and 4.13 ± 0.02 mm in width, respectively and showed no significant differences over all groups.

Two different processing methods characterized by specific surface treatments and ambient conditions during soldering were investigated. In addition, sub-groups were characterized by their gap geometry, in terms of scarf angle of 90° and 45° (see Figure 1), respectively, and the glass solder used. The different test groups and descriptions of their test specimens are summarized in Table 1.

Group I of soldered specimens were fabricated as follows: The glass solder powder was blended with Duceram Plus SMH-Liquid (DeguDent GmbH, Hanau, Germany) to create a paste. Both components were mounted on a custom-made device made of firing pillow with a gap of 0.1 mm. The gap was filled with the glass solder and the set-up was put in the furnace, which was heated up with 60 °K × min^{-1} to the maximum temperature of 1030 °C (glass solder 1) and 950 °C (glass solder 2), respectively. The maximum temperature was held for three minutes before cooling the specimens down to room temperature. This method of fabrication is referred to as simple manufacturing. The manufacturing process in group II was modified: Both components were cleaned in an ultrasonic bath using ethanol and then stored in ethanol for 30 minutes to improve the wettability of the ceramic abutting surface. Moreover, the test specimens were submitted to a pressure of 2 bar in a dental pressure pot after filling up the gap with the glass solder to reduce air voids in the bond.

The coefficient of thermal expansion (CTE) and chemical constitution of the two glass solders and the zirconia ceramic are summarized in Table 2.

Monolithic zirconia specimens (wide: 3.15 ± 0.01 mm, height: 4.14 ± 0.01 mm) were used as a reference and also heated at 950 °C (n = 3) and 1030 °C (n = 3), respectively.

Mechanical testing. The specimens were tested in a four-point bending test in accordance with the standard DIN EN ISO 843-1 [28] using a universal testing machine (Zwick Roell Z050-50 kN, Zwick Roell, Ulm, Germany). The support and the loading span were set to 40 ± 0.5 mm and 20 ± 0.2 mm, respectively. Axial pre-load (5 N) was applied and the ceramic specimens were subsequently loaded until fracture with a crosshead velocity of 1 mm × min^{-1}. For comparability with results from the literature, the nominal bending strength was evaluated.

Group	Gap geometry	Glass solder	Process	Specimen size
Ia-GS1	90°	Glass solder 1	Simple	3
Ib-GS1	45°	Glass solder 1	Simple	3
IIa-GS1	90°	Glass solder 1	Modified	4
IIb-GS1	45°	Glass solder 1	Modified	4
Ia-GS2	90°	Glass solder 2	Simple	3
Ib-GS2	45°	Glass solder 2	Simple	3
IIa-GS2	90°	Glass solder 2	Modified	4
IIb-GS2	45°	Glass solder 2	Modified	4
Simple process: No surface treatment and soldering under atmospheric pressure				
Modified process: Ethanol treatment of the surface and soldering under 2 bars pressure				

Table 1: Different test groups and their specifications

Materials	CTE [K^{-1}]	Chemical constitution
Glass solder 1	9.5 × 10^{-6}	SiO_2, Al_2O_3, K_2O, Na_2O, SrO, ZnO, CeO_2, B_2O_3
Glass solder 2	9.5 × 10^{-6}	SiO_2, Al_2O_3, K_2O, Na_2O, SrO, ZnO, SnO_2, CeO_2, La_2O_3
Zirconia	10.5 × 10^{-6}	ZrO_2 (additional oxides: HfO_2, Y_2O_3, Al_2O_3)

Table 2: Summary of the coefficients of thermal expansion (CTE) and chemical constitution of the glass solders and the used zirconia

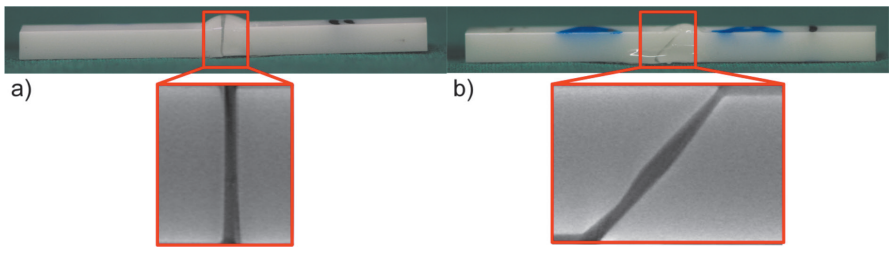

Figure 1: Bonded specimens with a scarf angle of, a) 90°, b) 45° and a µCT image of the bond

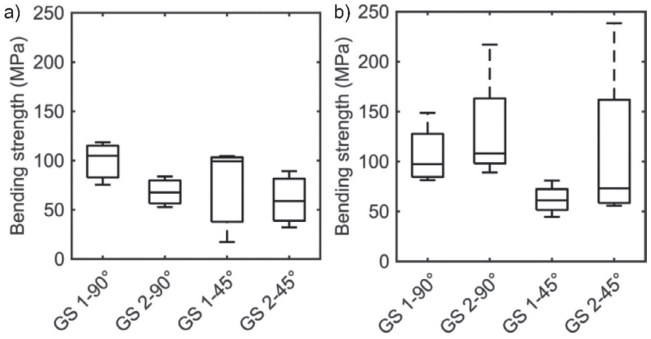

Figure 2: Measured bending strength (MPa) of, a) group I with the standard manufacturing method, b) group II with ethanol treatment of the surface and soldering under defined air pressure conditions of 2 bars and their sub-groups, which are defined by the glass solder (GS 1, GS 2) and gap geometry (45°, 90°)

Group	Gap thickness	Air voids	Adhesive failure area
	mm	Absolute quantity	$mm^2 \times mm^{-2}$
Ia-GS1	0.33 ± 0.05	6.3 ± 1.3	0.12 ± 0.02
Ib-GS1	0.19 ± 0.03	–	0.12 ± 0.04
IIa-GS1	0.52 ± 0.17	4.3 ± 3.6	0.09 ± 0.03
IIb-GS1	0.16 ± 0.04	–	0.33 ± 0.23
Ia-GS2	0.25 ± 0.06	6.0 ± 2.0	0.09 ± 0.02
Ib-GS2	0.17 ± 0.05	–	0.34 ± 0.09
IIa-GS2	0.37 ± 0.10	0.8 ± 0.8	0.12 ± 0.06

Table 3: Mean values and standard deviation of the gap thickness (mm), the relative adhesive failure area ($mm^2 \times mm^{-2}$) and the quantity of air voids divided by different groups which are characterized by the used glass solder, joint geometry and manufacturing method as described above

µCT and fractographic analysis. Prior to mechanical testing, all bonded test specimens were scanned using a µCT scanner (Skyscan1076, Bruker Corp., Billerica, MA, USA). Voltage and current were set to 95 kV and 104 µA, respectively. The pixel size was set to 9.2 µm. Afterwards, the gap thickness between the two components was evaluated in Amira (Amira 5.4.1, Thermo Fisher Scientific, Waltham, MA, USA) and the expression and occurrence of air voids were examined. Due to x-ray artifacts within the specimens with a 45° scarf angle, air voids in the bond could not be assessed. For the specimens with a 90° scarf angle, air voids with a diameter less than 0.1 mm could not be determined with sufficient reliability. Hence, 0.1 mm was defined as the critical resolution limit and the occurrence of air voids with diameters larger than 0.1 mm were examined.

After the four-point-bending test, the fractured areas were inspected using a digital microscope (VHX-6000, Keyence, Osaka, Japan) with the 3D-stitch technology. Fracture analysis included the distinction between adhesive failure, cohesive failure and a mixture of both in the cross-sectional area using ImageJ 1.52u (public domain).

Statistical analysis. Statistical analysis was performed in SPSS Statistics (v25, IBM Corp., Armonk, NY, USA). Bending strength, measured gap thickness, percentage of adhesive failure area and quantity of air voids of the soldered specimens were examined for significance using the Kruskal-Wallis test (level of significance $p = 0.05$). Additionally, the Pearson correlation factor was calculated for bending strength dependent on the adhesive failure area and gap thickness.

Results

Mechanical Testing. Mean values of the bending strength of bonded zirconia specimens (91.0 ± 47.7 MPa) compared to monolithic specimens (993.4 ± 125.5 MPa) were significantly reduced ($p < 0.001$). No significant difference was observed for the bending strength of the monolithic specimens in dependency of the heating temperature (950 °C: 997.0 ± 147.1 MPa, 1030 °C: 989.9 ± 99.3 MPa). Data of the measured bending strengths for the bonded zirconia specimens and corresponding sub-groups are presented in Figure 2. No differences were observed in dependency of the manufacturing method of the bonded specimens, the glass solders used, and the scarf angle.

µCT and fracture analysis. In Table 3, the results of µCT and fracture analysis are shown. Additionally, in Figure 3 the fracture surfaces and the corresponding µCT sectional view of specimens with minimum and maximum bending strengths are illustrated.

No significant differences between manufactured groups and sub-groups were identified concerning the gap thickness. The high variance was attributed to the manual manufacturing of the soldered joints. Moreover, µCT analysis has shown an improvement concerning air voids within the glass solder using the modified manufacturing, see Figure 3. These air voids are identifiable as regions with low gray values in the µCT scans. Differences found between the groups were not statistically significant.

Fractography revealed that a mixed fracture of adhesive and cohesive failure occurred in all groups. In each group, the adhesive failure area was increased non-significantly by modifying the scarf angle from 90° to 45°. Apart from that, no tendency could be proven as to in which way the different glass solders and the simple or modified manufacturing method favors adhesive or cohesive failure, respectively.

Microscopic fractography in combination with µCT analysis revealed differences between specimens manufactured with simple or modified conditions. Specimens of group Ia-GS2 indicated adhesive failure areas over the whole cross section in rather small proportions. Furthermore, air voids smaller in size but higher in quantity were found. In contrast, specimens in group IIa-GS2 revealed adhesive failure areas at their edges, predominantly. Air voids in these specimens were bigger in size but fewer in quantity.

Furthermore, the correlation between bending strength and measured gap thickness, as well as between bending strength and adhesive failure area, were investigated (see Figure 4). No correlation ($r = 0.07$) was found between bending strength and gap thickness. A correlation coefficient of $r = -0.33$ was calculated for the bending strength in dependency of the adhesive failure area. The specimen with the highest bending strength (238.5 MPa) was characterized by a gap thickness of 0.07 mm and an adhesion failure area of 0.05 $mm^2 mm^{-2}$ with respect to the total area

Discussion

Due to outstanding mechanical properties combined with chemical inertness and optical properties allowing excellent aesthetics, ceramics were introduced in dentistry decades ago [1]. Nevertheless, fracture toughness and susceptibility to notch-induced stress concentrations are restricting the use of ceramics [2-4]. Additionally, sintering of ceramic parts leads to thermal

gradients causing residual stresses that increase the risk of fracture [7, 8]. One promising approach to avoid residual stresses is the manufacturing of smaller sub-compounds that are subsequently connected by glass soldering [2, 9, 18].

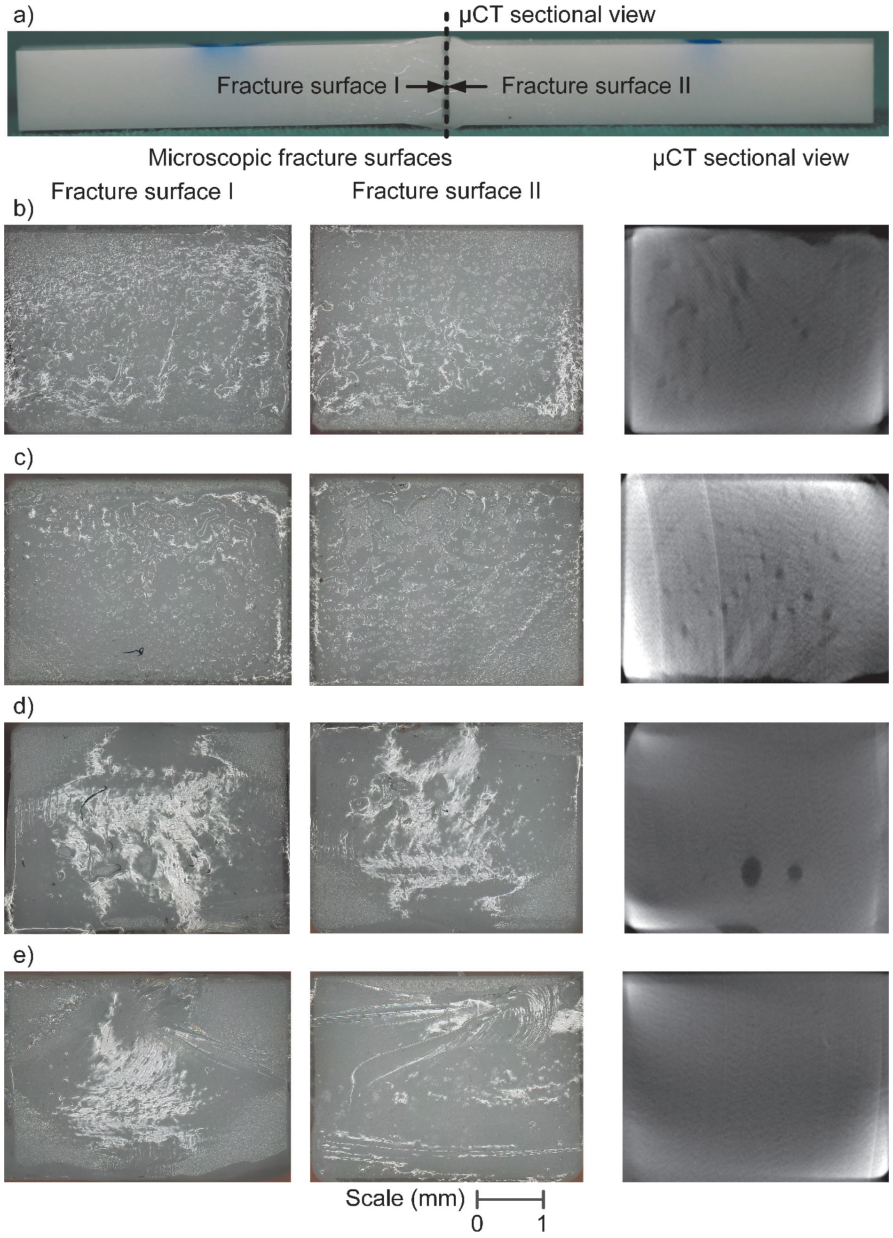

Figure 3: a) Microscopic overview of fracture surfaces I and II (200×) and position of the µCT sectional view (9.2 µm resolution) of, b) specimen in group Ia-GS2 with minimum bending strength of 52.8 MPa, c) specimen of group Ia-GS2 with maximum bending strength of 83.9 MPa, d) specimen of group IIa-GS2 with minimum bending strength of 89.1 MPa, d) specimen of group IIa-GS2 with maximum bending strength of 217.0 MPa

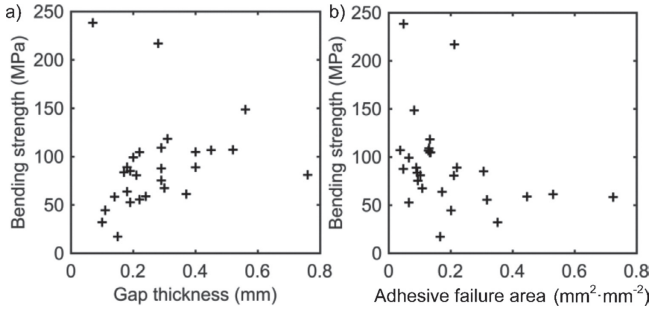

Figure 4: Measured bending strengths (MPa) in correlation with, a) gap thickness (mm), b) Adhesive failure area with respect to the total area (mm² × mm⁻²)

Although the feasibility of silicate-based glass solders to bond zirconia compounds has been shown previously, the literature lacks fundamental studies concerning the influence of surface treatments prior to sintering, ambient conditions during manufacturing, and gap geometry. Therefore, the aim of this experimental study was to evaluate the bending strength of zirconia compounds bonded with different glass solders and joint geometries. Additionally, the bonding process was varied.

According to DIN EN ISO 6872 [29] ceramic materials for dental use for pontics should reach a bending strength of 800 MPa in a four-point bending test, and fracture strengths of 886 MPa to 1526 MPa have been described for yttria stabilized zirconia [30]. This limit is ensured by monolithic specimens (993.4 ± 125.5 MPa). The soldered specimens could not reach bending strengths similar to those of the reference specimens (maximum: 130.6 ± 50.5 MPa (90° scarf angle; GS II)). This is attributed to unsmooth geometrical appearance at the glass solder bonding area, as well as notch stresses, as a consequence of material inhomogeneity and voids within the glass solder.

Comparable studies concerning soldering of different ceramic compounds using various glass solders have described average bending strengths between 150 MPa and 227 MPa [11, 13, 17] and shear strengths of 112 MPa [19]. Therefore, no study reached the described limit of 800 MPa. Consequently, in practical applications, the bond should not be applied at small cross sections or in locations with high loads, to avoid high stresses at the bonded region. Furthermore, the functional parameters of joint geometry and gap thickness as well as the manufacturing method must be chosen carefully to gain a bond with high strength.

Our experimental study is restricted by certain limitations. Firstly, the ceramic compounds were manually bonded before soldering. This method led to high deviations in the relative position of the bonded compounds, gap thicknesses and hence in the calculated bending strengths. Moreover, rather small sample sizes were investigated for each group.

Despite these limitations and lower bending strengths, as compared to other studies [11, 13, 17], tendencies regarding the joint geometry, manufacturing method, and gap thickness were derived within our experimental study.

The bonding thickness crucially influences the strength of the bond [24, 31, 32].

Alam et al. [31] investigated the shear strength of soldered bonds using metal-based solders. They concluded that a thicker bond leads to higher stress intensity factors and thereby reduces the strength of the compound. Similar results were obtained in a numerical study of adhesively bonded materials [24]. Lautenschlager et al. [32] stated that bonds with a larger mismatch between mechanical properties of the used solder and parent material tend to be strengthened by a thinner bonding. However, in the present work, no correlation between gap thickness and bending strength was determined. This is attributed to the multifactorial dependence of the bending strength and the scatter due to the manual manufacturing.

Different joint geometries were examined with the hypothesis that, due to stress transformation at the diagonal surface (45° scarf angle), tensile stresses are decreased and superimposed by shear stresses. Nevertheless, a negative impact on bending strength was observed. Numerical investigations of adhesively bonded materials have shown that bond strength increases between 45° and 90°, reaching a maximum at about 60° [24]. Thus, the difference between 45° and 90° is rather small. Therefore, the reduction in average bending strength from 90° to 45° could be explained by an increase of thermal residual stresses, which are highest at the edges [33].

Nevertheless, the manufacturing method showed the strongest influence on bending strength, which defines the homogeneity and wetting of the bond. Ethanol treatment of the surface with subsequent soldering under defined pressure conditions (group II) led to bending strengths up to 130.6 ± 50.5 MPa (90° scarf angle; GS II). Sufficient wetting of the ceramic surface is required to produce uniform bonds [11, 26]. Thus, wetting is dependent on the surface tension of zirconia and the glass solder. Consequently, wetting can be improved by surface treatments of the zirconia, customization of the chemical constitution of the glass solder, and by varying the bonding temperature [11, 16, 17, 26]. Based on the fracture surfaces, a superior wetting after ethanol treatment was derived as the fracture surfaces of the untreated specimens have always shown free ceramic surfaces in rather small proportions over the whole cross section. We assumed that these areas were not entirely wetted by the glass solder, and that the improved bonding due to ethanol treatment is a result of degreasing the surfaces of the zirconia parts.

Therefore, a homogeneous bond is necessary to ensure high strength. Since air voids form geometrical notches and lead to stress concentration, thereby reducing the strength of the bond, they must be prevented. Although the utilized method of manufacturing the bond under defined pressure conditions did not significantly reduce the quantity of air voids, a positive tendency was shown in the investigations.

Sun et al. [21] examined the mechanical properties of bonds between ceramic specimens using glass solder in tensile tests. They observed that the bond within the glass solder is more likely to fail once the amount of air voids is high. Conversely, the failure occurs within the interface between glass solder and ceramic for specimens of less air. This is in line with the observations within our present study. Lis et al. [34] have shown, that a numerical approach is suitable to investigate the tolerance of soldered joints to artificial flaws in detail.

We manually soldered zirconia sub-compounds by using silicate-based glass solders and evaluated the bending strength in a four-point bending test. Due to the high standard deviations found, further research should be carried out to enable smaller variations between soldered specimens. Moreover, an advanced manufacturing method is required to transfer the bonding technology to practical applications in the field of dentistry. Moreover, investigations are necessary to define optimal parameters concerning the gap thickness and the scarf angle. Numerical simulations, assuming perfectly bonded and homogenous bonds may be suitable for defining these parameters.

Conclusions

Silicate-based glass solders appear to be feasible to manually bond zirconia compounds for practical applications in the field of dentistry. Further optimization and standardization of the soldering process is suggested to enhance mechanical bending strength.

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