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(Director: Prof. Dr. Maximiliane Schlenz-Helmke)
at the Universitätsklinikum Schleswig-Holstein, Campus Kiel
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**Effects of sterilization, conditioning, and thermal aging on the retention of
zirconia hybrid abutments**

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presented by
Nalah Alghanaim, M.Sc.
From Jeddah, Saudi Arabia

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1st Reviewer: Prof. Dr. Hans-Jürgen Wenz, Klinik für Zahnmedizinische Prothetik

2nd Reviewer: Prof. Dr. Christian Graetz, Klinik für Zahnerhaltungskunde und
Parodontologie

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Signed: Prof. Dr. Hans-Jürgen Wenz

(Chairperson of the Examination Committee)

Table of Contents

Tables of Figures:	III
Table of tables:	III
Abbreviations	IV
1.1 Different materials of implant abutments	1
1.2 Method of fabrication of implant abutments.....	3
1.3 Factors affecting the retention of hybrid abutments	5
1.3.1 Surface treatment	5
1.3.2 Type of adhesive luting resin.....	7
1.3.3 Luting gap	7
1.3.4 Ti-base height	8
1.4 Cleaning and sterilization of implant abutments.....	8
1.4.1 Importance of sterilization	8
1.4.2 Methods of sterilization	9
2. Aim of the study	11
3. Materials and Methods	12
3.1 Experimental design and study groups	12
3.2 Surface conditioning of titanium bases and zirconia abutments	15
3.2.1 Bonding surfaces of titanium bases	15
3.2.2 Bonding surfaces of zirconia abutments	16
3.3 Bonding procedures	19
3.4 Disinfection and Sterilization.....	24
3.5 Sterilization process	27
3.6 Artificial aging	29
3.7 Axial retention test	31
3.8 Mode of failure.....	32
3.9 Statistical analysis	32
4. Results	34
5. Discussion	38
5.1 Discussion of Methodology	38
5.1.1 Zirconia hybrid abutments	38
5.1.2 Dual-curing luting resin	38
5.1.3 Surface conditioning of titanium bases and zirconia abutments	39
5.1.4 Sterilization process.....	39
5.1.5 Thermal aging.....	40
5.2 Discussion of Results	40
5.2.1 Influence of surface conditioning of titanium bases and zirconia abutments.....	40

5.2.2 Influence of sterilization	42
5.2.3 Influence of thermal aging.....	42
5.2.4 Failure mode of luting resin.....	43
6. Conclusions	44
7. Summary.....	45
8. Zusammenfassung.....	46
9. References.....	47
10. Acknowledgements	52
11. Dedication	53
12. Appendices	54

Tables of Figures:

Figure 1: Sky hybrid abutments, all single components of the test specimens before assembling. (A) Holding screw, (B) zirconia abutment, (C) titanium base, and (D) implant analogue.	12
Figure 2: Flowchart of study design.....	13
Figure 3: Alumina particle air-abrasion of titanium bases with 50 μ m particles at an angle of 90 degrees.....	15
Figure 4: <i>The dull appearance of the titanium base after alumina particle air-abrasion.</i>	15
Figure 5: Protect the outer surface of the zirconia abutment from alumina particle air-abrasion	16
Figure 6: Waterproof marker used to mark the inner surface of zirconia abutments.	17
Figure 7: Stained intaglio surface of zirconia abutments.....	17
Figure 8: Alumina particle air-abrasion of the inner surface of zirconia abutments	18
Figure 9: Removing the black rubber from the zirconia abutment.	18
Figure 10: DTK adhesive resin luting agent and MKZ primer	19
Figure 11: Bonding device (Kern and Thompson 1993)	19
Figure 12: MKZ primer applied to a titanium base.....	20
Figure 13: MKZ primer applied to zirconia abutments.	21
Figure 14: Application of DTK resin (A) to the titanium base, (B) to the zirconia abutment,(C) removal of the plastic screw, (D) placement of a prosthetic screw, (E) using the screwdriver	22
Figure 15: Removal of luting resin the excess.	23
Figure 16: Light curing the margins with a curing light.	23
Figure 17: Water bath incubator	24
Figure 18: FINOVO cleaning system.	24
Figure 19: FINEVO liquid set.....	25
Figure 20: Steps of the FINEVO cleaning system (A) Step 1, (B) Step 2, and (C) Step 3.....	26
Figure 21: Vacuklav 24 autoclave sterilizer.....	27
Figure 22: Specimens in sterilization pouches.....	27
Figure 23: Torque tester	28
Figure 24: Sealing the screw channel with polytetrafluoroethylene tape	28
Figure 25: Sealing the screw channel with flowable composite resin.	29
Figure 26: Groups that were subjected to thermal aging.	30
Figure 27: Thermocycling machine	30
Figure 28: Universal testing machine Zwick Z010 (1) Opening for the ceramic sleeve (2) Opening for titanium bases (3) Chains for torsion-free.....	31
Figure 29: Optical light microscope connected to a digital camera for failure mode evaluation.	32
Figure 30: Boxplot of the retentive strength (N) of test groups	35
Figure 31: Mode of failure of the luting resin (cohesive failure) (A) luting resin adhering to titanium bases, and (B) luting adhering to zirconia abutments.	37

Table of tables:

Table 1: List of study groups and study factors	14
Table 2: The retentive strength of the test groups.....	34
Table 3: Results of the three-way ANOVA test.....	36

Abbreviations

Abbreviation	Description
Al₂O₃	Aluminum oxide
APA	Airborne particle abrasion
°C	Degree Celsius
μm	Micrometer (0.001 millimeter)
CAD/CAM	Computer-aided design and computer-aided manufacturing
DC	Degree of conversion
MDP	Methacryloyloxydecyl dihydrogen phosphate
Min	Minute
Mm	Millimeter
MPa	Megapascal
N	Newton
PEEK	Polyetheretherketone
SD	Standard deviation
Sec	Second
Ti	Titanium
ZrO₂	Zirconia

1. Introduction

Dental implants and implant-supported restorations are the most promising and widely used prosthetic treatments for completely or partially edentulous patients to replace missing teeth with high survival rates (1). The ultimate goal of treatment with implants is to achieve optimal functional as well as esthetic outcomes (1). Many implant brands and various components are available in the dental market because of the growing demand for implant prosthesis. There are many local and systematic factors related to the success of implant restorations, such as osseointegration, proper case selection, abutment selection and maintenance. Proper selection of implant abutments is very critical to the success of implant prosthesis. Implant abutments are different in implant-abutment connection, type of material, type of retention, and method of fabrication (1, 2).

1.1 Different materials of implant abutments

The implant abutment is the part that supports and retains the implant restoration and connects it to the implant. Different materials are available for the fabrication of implant abutments, such as cast gold alloy, pure titanium, lithium disilicate ceramic, zirconia, and high-strength polymers (2).

Cast gold abutments were very popular but have lost popularity due to the introduction of stock abutments and CAD/CAM milled abutments (2, 3). Titanium abutment is the most commonly used material in dentistry. Titanium abutments (Ti) have been well-established for years and are considered the gold standard material for implant abutments. They were reported to have high success and survival rates in long-term clinical studies due to their biocompatibility and good physical properties (3, 4). However, titanium abutments can compromise the esthetic results due to the greyish discoloration of the surrounding soft tissues in patients with the thin gingival phenotype (2 mm or less in thickness) or inadequate depth of the emergence profile, particularly in the esthetic zone (5).

The high demand for esthetic restorations and increased patient awareness and expectations have led to the introduction and fabrication of implant-supported fixed dental prostheses from more esthetic ceramic-based materials (6). Ceramic abutments (alumina reinforced ceramics)

had been developed as an esthetic alternative to titanium abutments due to their natural-looking color. However, they did not show acceptable longevity, as alumina-based ceramics had relatively low mechanical strength and tended to undergo static fatigue and fracture at implant-abutment junction (6, 7).

In recent years, new materials including ceramics and polymers have been used in prosthetic implant dentistry. The challenge in ceramic materials is that they should combine sufficient strength and enough translucency that is required for optimum esthetics and longevity. Lithium disilicate ceramics and zirconia are the most commonly used for implant abutments. Lithium disilicate ceramics are considered the strongest glass ceramics, with an average flexural strength of 530 MPa. However, there was a high risk of lithium disilicate abutment fracture at the implant-abutment connection site due to their relatively low mechanical strength (6, 8).

High-strength ceramics, particularly yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was introduced as an abutment material in 1995 (9). Owing to its excellent biocompatibility with less plaque affinity, good esthetics and high fracture strength, approximately twice that of alumina (9). Nowadays, zirconia (ZrO_2) has become the most favorable material of choice in implant dentistry, especially in the anterior esthetic zone with thin gingival biotype. Zirconia abutments minimize bacterial and plaque accumulation, thereby reducing risk of soft tissue inflammation. However, it is weaker than titanium and should be avoided in mechanically challenging areas with limited interocclusal distance (10). A systemic review by Sailer *et al.* showed that 5-year survival rates and incidence of complications of ceramic abutments appeared to be similar to metal abutments (11).

With the advancement of technology, high-performance polymer materials have been developed. These materials are semi-crystalline polycyclic thermoplastic materials and have been proposed as a substitute for metals in biomaterials. They include PEEK (polyetheretherketone) and PEKK (polyetherketoneketone) (12). They have been introduced in many applications due to their outstanding physical, thermal and biological properties. They have been used successfully as healing abutments, provisional restorations, the framework of removable partial dentures, abutment screws, and implant abutments (12, 13). Newer modifications by strengthening with ceramic fillers or carbon fibers have been developed to improve their strength and to be used as final implant restoration. Besides, PEEK is believed to decrease the strain and stress within the restoration and at the implant-bone interface because of its lower modulus of elasticity (12, 13, 14).

Moreover, hybrid dental ceramics have been newly developed, featuring a dual ceramic network reinforced by a polymer network structure. The material is considered a resin ceramic composite material. The main advantages of this material are that its elastic modulus is closer to tooth dentin, more easily milled, adjusted and repaired with composite resin. Due to their inferior optical properties, it is indicated in posterior region (15). This hybrid material can be used to fabricate single-tooth restorations and implant-supported single crowns and is assumed to combine the positive properties of ceramic and composite materials, however, without available data on its longevity (16, 17).

1.2 Method of fabrication of implant abutments

The selection of implant abutments is based on many factors, including implant position and angulation, interocclusal distance, esthetic requirements, axial load distribution, soft and hard tissue profile and occlusion (18).

Implant abutments can be manufactured as standardized components (prefabricated) or customized. Prefabricated or stock abutments are made by the manufacturer with different heights, various tapers and angulations (19). Their advantages are simple utilization and inexpensiveness. However, their main problem is the platform diameter, and it still needs modifications and corrections of the height and wall alignment, which can be done in the lab or chairside. Compared to customized abutments, prefabricated ceramic abutments have disadvantages of limited ability for individualization, they cannot be modified to follow the gingival contour, and their use is limited to posterior non-esthetic areas (20, 21, 22).

Customized implant abutments are either fabricated in the lab by using computer-aided design/computer-aided manufacturing (CAD/CAM) technology or by copy-milling procedures (23, 24). CAD/CAM customized zirconia abutments have the advantages of a better emergence profile and they enable the fabrication of abutments with the desired shape and size (25). Moreover, customized abutments are indicated in cases with not ideal implant position, wrong implant angulation, and in the anterior esthetic zone for better esthetic outcomes (21).

Furthermore, the prefabricated zirconia stock abutments can be classified into two categories: One-piece and two-piece zirconia abutments. One-piece zirconia abutments are entirely made of zirconia, including the implant-abutment connection manufactured entirely in

a central production process by CAD/CAM technology (26). Studies have shown that one-piece zirconia abutments cause an increased marginal gap that can lead to bacterial colonization, as well as marginal misfit that can cause wear of the implant-abutment interface and lead to screw loosening as well as to abutment fracture at the connection area (27).

Therefore, the contemporary abutment design is the “two-piece” zirconia abutment, which consists of a zirconia abutment and a titanium insert connected to the implant (hybrid abutment). This type of implant abutment was developed to overcome the disadvantages of single-piece zirconia abutments. Hybrid abutments combine the mechanical stability of titanium to overcome the brittleness of zirconia. The titanium insert reinforces the fragile connection between the abutment and the implant and reduces the wear and damage at the implant-abutment interface. In addition, hybrid abutments have superior biological compatibility but have improved optical properties of zirconia. However, optimal adhesive bonding between titanium insert and zirconia abutments is required for good retention (28, 29). Another advantage is that the zirconia coping can be bonded to titanium inserts extra-orally and then screwed as a 1-piece restoration to the implant, reducing the risk of biological complications in terms of excess cement (30, 31).

A study by Gehrke *et al.* showed that two-piece zirconia abutments demonstrated superior resistance to fracture compared to one-piece and stock zirconia abutments and might even be clinically applicable in high load areas such as premolars and molars (32). Moreover, a systematic review showed that hybrid abutments had high fracture resistance that prevented the wear of implant connection and resulted in better marginal fit compared to one-piece zirconia abutments (33).

There are two designs of two-piece zirconia abutments, either hybrid-abutment-crown or hybrid abutment with a separate crown. In the hybrid-abutment-crown design, the crown and the abutment are made as one piece and bonded to titanium base that will be screwed to the implant. While in the hybrid abutment design, the abutment will be bonded to a titanium insert that will be screwed to the implant, and then a separate all-ceramic crown will be cemented on top of it (33).

1.3 Factors affecting the retention of hybrid abutments

The long-term clinical success of hybrid abutments depends mainly on the adhesive connection between the titanium insert and zirconia copings. Adhesive bonding has a great effect on the longevity of dental restorations by increasing the fracture strength, marginal adaptation, and retention of the restorations (29). A strong and durable bonding to the zirconia surface can be achieved by the combination of micromechanical retention as well as chemical reaction between the zirconia surface and adhesive system (34). The advantage of the hybrid abutment retention mechanism (screwed and cemented) is that it improves the light curing of adhesive system around the margins of the abutments and removes all the excess extra-orally before screwing it to the implant (21).

The retentive bond between zirconia abutments and titanium inserts is influenced by the abutment height and their convergence angle (taper), type of luting material, amount of luting material, luting material degradation rate and methods of surface conditioning (4, 26, 35, 36). A study by Ebert et al. found that the surface conditioning method, luting gap size, and storage time significantly influenced the bond strength and its durability (26).

1.3.1 Surface treatment

In order to achieve good adhesion in hybrid abutments, surface conditioning of zirconia surface and titanium inserts must be performed. Surface treatment and roughening to increase the surface area of the bonding surfaces depend on the material itself. Metallic restorations require air abrasion with alumina particles, while glass-ceramic restorations require etching with hydrofluoric acid. Zirconia ceramics are characterized by high mechanical properties due to high crystalline content and the absence of the glassy phase. For this reason, etching the zirconia surface with hydrofluoric acid (HF) is not an effective method for surface alteration to achieve adhesive bonding (37). To overcome this challenge, zirconia adhesion protocol involves micromechanical treatment followed by chemical bonding techniques.

Several methods were suggested for the surface treatment of zirconia, like air-borne particle abrasion (APA) with alumina particles and tribochemical silica coating. Various alternative surface conditioning methods have been developed, such as laser irradiation, selective infiltration etching, and applying low-using porcelain pearl layer. However, these alternative methods did not show any evidence of durable bonding. In addition, they are complicated and time-consuming procedures (38, 39, 40, 41).

The most common and effective method for surface treatment of zirconia is the APA with alumina particles with different particle sizes, pressures, and time durations. A study by Hallmann et al. recommended the APA using alumina particle size of 50 μm or 110 μm at a pressure of 0.15 MPa or 0.25 MPa (34). Kern reported that APA of the zirconia surface before resin bonding is a very effective method to enhance the micromechanical interlocking between the luting resin and zirconia surface when moderate pressure and small alumina particle size were used (40). It is recommended to use 50 μm alumina particles with a pressure of 0.25 MPa or less to provide the required micro roughening of zirconia surface. In addition, there is strong evidence that APA of zirconia surface at moderate pressure and using MDP containing primers or/and luting resin produce long-term durable bonding (40, 41).

There are many benefits of surface treatment of zirconia, including cleaning the intaglio surface from organic contaminations, expanding the bonding area and increasing surface roughness (41). This will improve the micromechanical retention by creating microporosity and roughing the surface of zirconia, increasing the surface energy, and enhancing the wettability of the zirconia surface (35, 42, 43, 44). Additionally, APA affects the integrity of the zirconia surface, especially when using large alumina particle size and high pressure. The effect of APA on the flexural strength of zirconia is controversial in the literature. Some of the studies showed a positive strengthening effect of APA on the zirconia surface because of the phase transformation of Y-TZP from tetragonal to monoclinic; this would increase the fracture toughening of the zirconia and protect cracks propagation (40, 45, 46). On the other hand, some studies reported that APA with high air pressure will increase the microcracks formation in the intaglio surfaces of zirconia abutments; these microcracks can be stress concentration sources and have a damaging effect on the flexural strength and decrease the mechanical properties of zirconia. However, the adhesive system can prevent the negative effect of surface alterations and wreaking effect by its ability to seal all microcracks of the roughened surface of zirconia (37, 40, 46, 47).

The tribochemical silica coating method has been shown to improve the stability of the bonded interface between the titanium inserts and zirconia abutments. This method includes APA of the zirconia surface followed by silica-coating and application of silane primer (40, 41, 47). The APA increases the surface area of zirconia and enhances the surface energy. This will improve the micromechanical adhesion (48, 49). In addition, after the silane compound is applied to the silica-coated surface, a chemical reaction occurs between the silica layer

deposited on the zirconia surface and the silane coupling agent forming the silica-silane bond (44, 45, 47).

1.3.2 Type of adhesive luting resin

The retention of conventional cements depends on the micromechanical retention between the filler particles of the cement and rough abutment surfaces. In contrast, the retention of luting resin is based on the chemical bonding between the resin and the bonding surfaces, which produces higher bond strengths and a better marginal seal (35, 50).

Zirconia abutments are bonded onto the titanium inserts using luting resins, which are composed of methacrylate monomers and inorganic particles treated with silane. There are three main types of luting resin, chemical cure, dual cure, and light cure systems. Chemical and dual-curing luting resin are mainly used to bond zirconia abutments on titanium bases because of the difficulty of sufficiently light curing under opaque zirconia abutments. Many factors may affect the polymerization mechanism of the luting resin, including the film thickness and chemical composition of the resin, opacity, and the shade of the ceramic material, which can affect the light penetration and decrease the polymerization activity of the luting material (51, 52).

A previous study by Silva et al. showed significantly greater tensile strength values for hybrid abutments bonded with resin-based system compared with glass-ionomer and temporary cements (53). Evidence shows that a long-term durable bond between titanium inserts and air-abraded zirconia surfaces can be achieved by using luting resins containing methacryloyloxydecyl dihydrogen phosphate (10-MDP) (54, 55). Moreover, it has been reported that using primers containing 10-MDP in combination with luting resins yield also high bond strengths by forming chemical bonding to the zirconia surface (46).

1.3.3 Luting gap

There is a negative correlation between the size of luting gap and the retention of hybrid implant abutments. This means that higher retention values are observed in smaller luting gaps between the titanium bases and zirconia copings. A larger luting gap with increased resin film thickness will decrease the bond strength of two-piece zirconia abutments (18). Likewise, a study by Ebert et al. reported that zirconia coping with 30 μm gap size showed significant increase in retention compared to others with 60 μm gap size (19, 26).

1.3.4 Ti-base height

The geometry of titanium inserts is considered one of the factors that affect the adhesive bond between titanium and zirconia copings, especially the height, in addition to the degree of taper and diameter (18, 19). A previous study by Abbo et al. reported that a 1 mm increase in the length of titanium bases results in significant increase in the retention of the zirconia supra-structure (56). Likewise, Zahoui et al. concluded that taller abutments provided greater retention than shorter ones (50). However, another study showed that the height of titanium inserts did not affect the retention of hybrid abutments (53).

1.4 Cleaning and sterilization of implant abutments

1.4.1 Importance of sterilization

The long-term success rate of implant restorations depends on the implant-abutment connection. Peri-implant soft tissue serves as a barrier between the oral environment and protects the peri-implant bone from any microbial contamination. Bonding procedure in hybrid abutments is technique-sensitive and should be carried out with caution to decrease the risk of bacterial contamination. Therefore, the contamination of customized abutments after the laboratory procedures may influence the inflammatory response of peri-implant tissues (57). The microbial contamination around the implant-abutment junction will increase the chance of peri-implantitis and implant bone loss (58).

Cleaning, disinfection or sterilization of implant abutments must be done in the clinical setting to remove all debris and contamination that might affect implant longevity. Bacterial contamination can be present at abutment external surface, at the internal connection and within the screw canal, and on customized prosthetic components after laboratory procedures (57, 59). The purpose of cleaning is to remove or decrease visible soils such as blood, protein, and debris on the material surface. The aim of sterilization is to eliminate or prevent the reproduction and proliferation of microorganisms, including spores, bacteria, and fungi (60). A clinical study by Canullo et al. suggested and advised to use only cleaned and sterilized customized abutments. In addition, the study showed that 12 min argon plasma treatment of titanium implant abutments resulted in significantly less marginal bone loss around the implants over a 2-year period, compared to non-sterile and 5-second steam cleaning in periodontally healthy patients (58).

However, there were some concerns about this method because argon plasma treatment is not approved as a sterilization method for semi-critical medical devices.

1.4.2 Methods of sterilization

Many sterilization methods have been used in dentistry, depending on the desired application and material properties. The main goal is to sterilize implant material and implant prosthetic components without affecting their mechanical and surface characteristics. Some implant manufacturers provide sterilized stock abutments and hybrid abutments. However, extra-oral bonding of zirconia to titanium copings will lead to contamination of the two-piece zirconia abutments and makes cleaning or sterilizing implant abutments necessary (61, 62).

According to the Centers for Disease Control and Prevention, dental instruments are classified into three categories depending on the risk of transmitting infection (57, 61). Dental instruments can be classified into critical, semi-critical and non-critical instruments. Critical instruments are those used to penetrate bone and soft tissues and must be sterilized after each use. They include implants, bone chisels, forceps, and burs. Semi-critical instruments are those that come in contact with skin or oral mucosa but do not penetrate bone or the surrounding soft tissues. They should be sterilized or, at a minimum disinfected. They include mirrors, amalgam condensers, and implant abutments. Non-critical instruments are those that come in contact only with intact skin but not a mucous membrane, such as external components of X-ray heads. They require only low-level disinfection (57, 63, 64).

Sterilization techniques can be divided into physical and chemical methods. Physical methods are the most suitable for sterilizing implants and implant components, such as dry heat sterilization, steam autoclave, Gamma irradiation, and ultraviolet radiation. Steam autoclaving is the most commonly used in the dental field due to its low cost, convenience, and reliable effects. Chemical sterilization methods include using ethylene oxide, ozone, and phenols. However, chemical sterilization methods are not recommended by the American Dental Association because of the toxic effect of residual chemicals left on the products and for operator safety (57, 59, 61, 65). A study by Fadanelli et al. showed that steam autoclaving increased the mean bond strength of a chemically curing luting resin compared to a dual-curing luting resin (66).

Clinically, it is highly recommended to sterilize the hybrid implant abutments after bonding and prior to intraoral insertion. However at present, there is no available evidence about the

influence of sterilization parameters (heat treatment and pressure) on the retention of the hybrid abutment and their effect on the retentive strength of resin luting material used in the zirconia-titanium interface.

2. Aim of the study

The aim of this laboratory study was to investigate the effect of surface conditioning methods, sterilization, and thermal aging on the retention of zirconia abutments bonded to titanium inserts.

In particularly:

1. To evaluate the effect of airborne-particle abrasion of the inner surfaces of zirconia abutments and titanium surfaces (different particle sizes and different pressures) on the retentive strength of the hybrid abutments.
2. To evaluate the effect of autoclave sterilization on the retentive strength of DTK resin material used to bond zirconia abutments onto titanium bases.
3. To evaluate the effect of artificial aging (thermal cycling and long-term water storage) on the retentive strength of zirconia hybrid abutments.

The null hypotheses tested were that: (1) different surface pretreatment would not influence the retentive strength, (2) autoclave sterilization would not affect the retentive strength, and (3) thermal aging would not affect the retentive strength of hybrid abutments.

3. Materials and Methods

3.1 Experimental design and study groups

The study was carried out of a total of 128 specimens of stock zirconia abutments (Sky hybrid abutments, bredent, Senden, Germany). The hybrid abutments consisted of zirconia abutments and titanium bases that were screwed onto the implant analogues ([Figure 1](#)). The zirconia abutments were straight (0° angled) measured 9.4 mm in height and 5.5 mm in outer diameter, while the height of the titanium bases was 4.5 mm. The specimen was attached with a screw to a lab analogue of a 3.5 mm dental implant.

This study tested the retentive strength between zirconia abutments and titanium inserts, luted with dual-curing luting resin (DTK-adhesive; bredent). The study factors were the different zirconia surface conditioning parameters, the effect of sterilization, and the effect of thermal cycling.

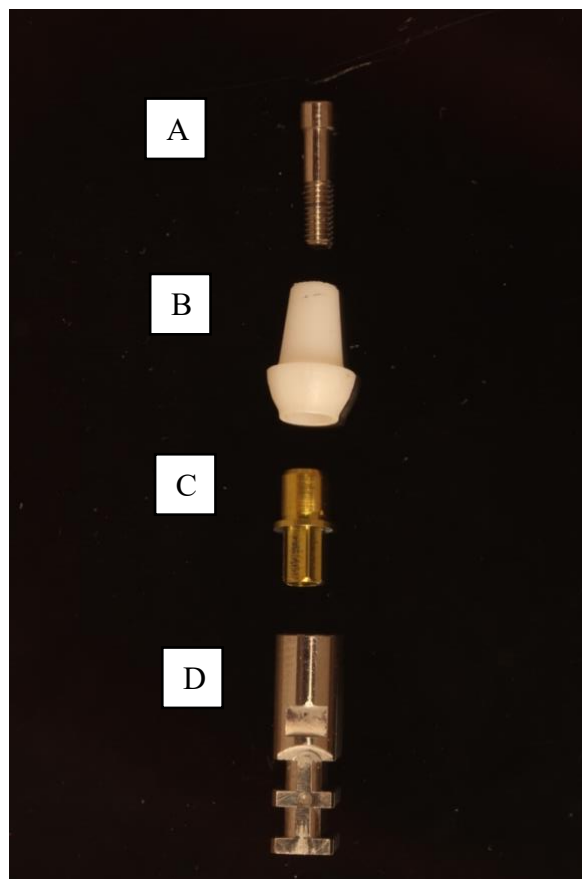


Figure 1: Sky hybrid abutments, all single components of the test specimens before assembling. (A) Holding screw, (B) zirconia abutment, (C) titanium base, and (D) implant analogue.

The total 128 zirconia abutments were subdivided into 16 subgroups, with 8 specimens in each group ($n=8$). The first variable was zirconia surface condition parameters, and the specimens were divided randomly into 4 main groups (A, B, C, D / $n=32$) according to the different zirconia surface conditioning parameters: Air-abrasion using (A) 50 μm alumina particles at 1.0 bar pressure, (B) 50 μm at 2.0 bar pressure, (C) 100 μm at 1.0 bar pressure or (D) 100 μm at 2.0 bar pressure. The second variable was the sterilization factor after 3 days of bonding. All the specimens were randomly subdivided into two subgroups (groups 1 and 2) with ($n=16$).

For subgroup 1, the specimens were only disinfected, whereas for subgroup 2, the specimens were subjected to disinfection and then to autoclave sterilization. Finally, according to the third variable, which is thermal ageing, half of the specimens in each subgroup (N) with ($n=8$) were subjected to an axial retention test in a universal testing machine, and the other half (T) with ($n=8$) was first thermally aged for 150 days and then subjected to the axial retention test.

The study design is illustrated in [Figure 2](#). The study groups and their division/subdivision according to the studied parameters are listed in [Table 1](#).

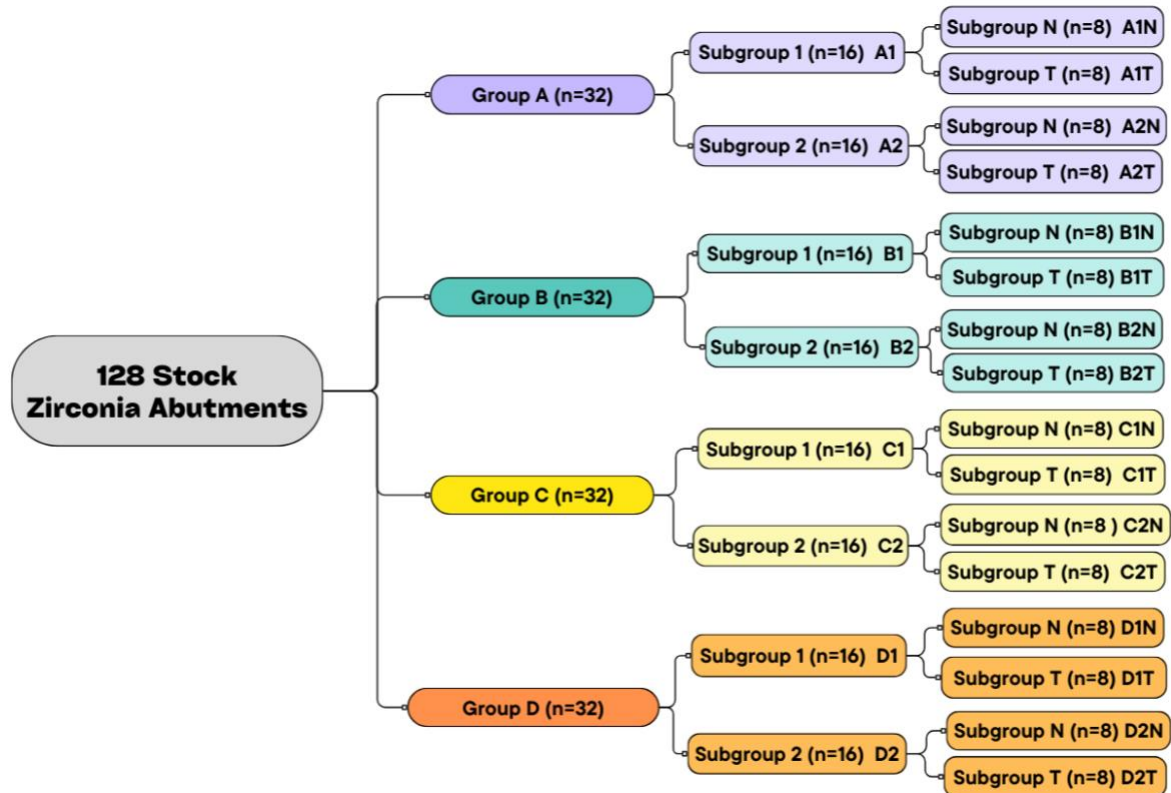


Figure 2: Flowchart of study design

Table 1: List of study groups and study factors

Groups (n=8)	Method of zirconia conditioning	Mode of sterilization	Thermal cycling (150 days)
Group 1 A1N	50 μm alumina at 1.0 bar	No sterilization	No
Group 2 A1T	50 μm alumina at 1.0 bar	No sterilization	Yes
Group 3 B1N	50 μm alumina at 2.0 bar	No sterilization	No
Group 4 B1T	50 μm alumina at 2.0 bar	No sterilization	Yes
Group 5 C1N	110 μm alumina at 1.0 bar	No sterilization	No
Group 6 C1T	110 μm alumina at 1.0 bar	No sterilization	Yes
Group 7 D1N	110 μm alumina at 2.0 bar	No sterilization	No
Group 8 D1T	110 μm alumina at 2.0 bar	No sterilization	Yes
Group 9 A2N	50 μm alumina at 1.0 bar	Sterilization	No
Group 10 A2T	50 μm alumina at 1.0 bar	sterilization	Yes
Group 11 B2N	50 μm alumina at 2.0 bar	sterilization	No
Group 12 B2T	50 μm alumina at 2.0 bar	sterilization	Yes
Group 13 C2N	110 μm alumina at 1.0 bar	sterilization	No
Group 14 C2T	110 μm alumina at 1.0 bar	sterilization	Yes
Group 15 D2N	110 μm alumina at 2.0 bar	sterilization	No
Group 16 D2T	110 μm alumina at 2.0 bar	sterilization	Yes

A = air-abrasion using 50 μm alumina particles at 1.0 bar pressure

B = air-abrasion using 50 μm alumina particles at 2.0 bar pressure

C = air-abrasion using 110 μm alumina particles at 1.0 bar pressure

D= air-abrasion using 110 μm alumina particles at 2.0 bar pressure

1 = no sterilization

2 = sterilization

N = no thermal cycling

T = with thermal cycling (150 days).

N = number of specimens

3.2 Surface conditioning of titanium bases and zirconia abutments

3.2.1 Bonding surfaces of titanium bases

First, the titanium bases were attached to the implant analogue with their screw. Then a plastic screw was placed onto the titanium bases to protect the screw channel. The bonding surfaces of titanium bases were air-abraded vertically using 50 μm alumina particles (Aluminum Oxide Abrasive; Kulzer, Wehrheim, Germany) at 2.5 bar pressure from a distance of 10 mm for 15 seconds at a right angle (90 degrees) ([Figure 3](#)). The titanium surface was air-abraded until it had a dull appearance (visual check) ([Figure 4](#)).

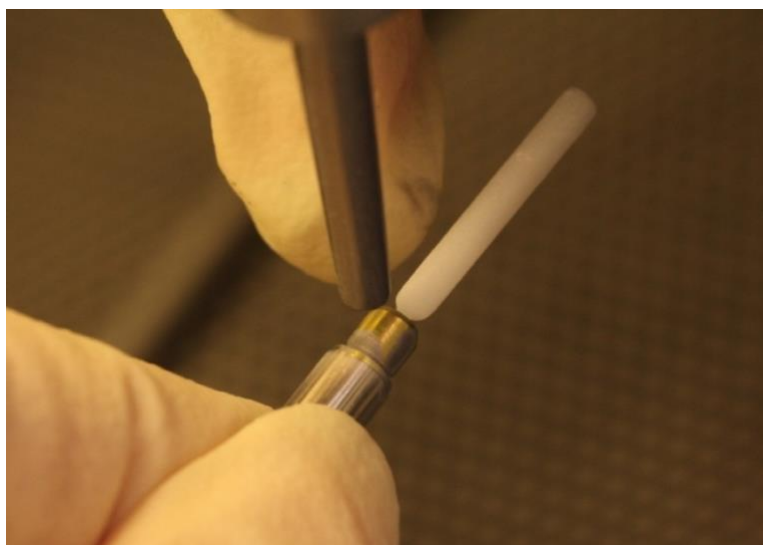
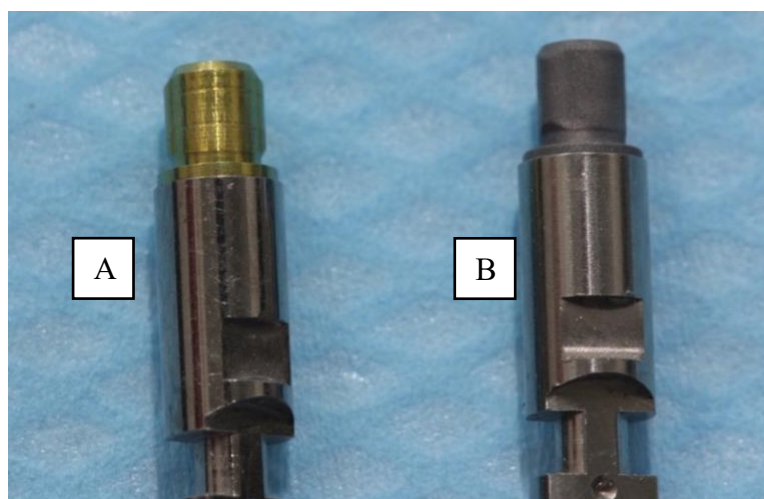


Figure 3: Alumina particle air-abrasion of titanium bases with 50 μm particles at an angle of 90 degrees.



*Figure 4: The dull appearance of the titanium base after alumina particle air-abrasion.
(A) Titanium base before alumina particle air-abrasion (gold shiny appearance).
(B) Titanium base after alumina particle air-abrasion (gray dull appearance)*

3.2.2 Bonding surfaces of zirconia abutments

The outer surface of zirconia abutments was protected by covering it with black rubber material ([Figure 5](#)). The intaglio surfaces of zirconia abutments were marked/colored with a blue waterproof marker before the air-abrasion (67) ([Figure 6](#) and [Figure 7](#)).

The air-abrasion of the inner surface of zirconia abutments was conducted using alumina particles in an oblique direction (45 degrees) inside the canal and at right angle in the shoulder area from a distance of 10 mm. Alumina particle air-abrasion was conducted until the blue marking was removed ([Figure 8](#)). Each group was conditioned according to the specific parameters.

- Group A: air-abrasion using 50 μm alumina particles at 1.0 bar pressure.
- Group B: air-abrasion using 50 μm alumina particles at 2.0 bar pressure.
- Group C: air-abrasion using 110 μm alumina particles at 1.0 bar pressure.
- Group D: air-abrasion using 110 μm alumina particles at 2.0 bar pressure.

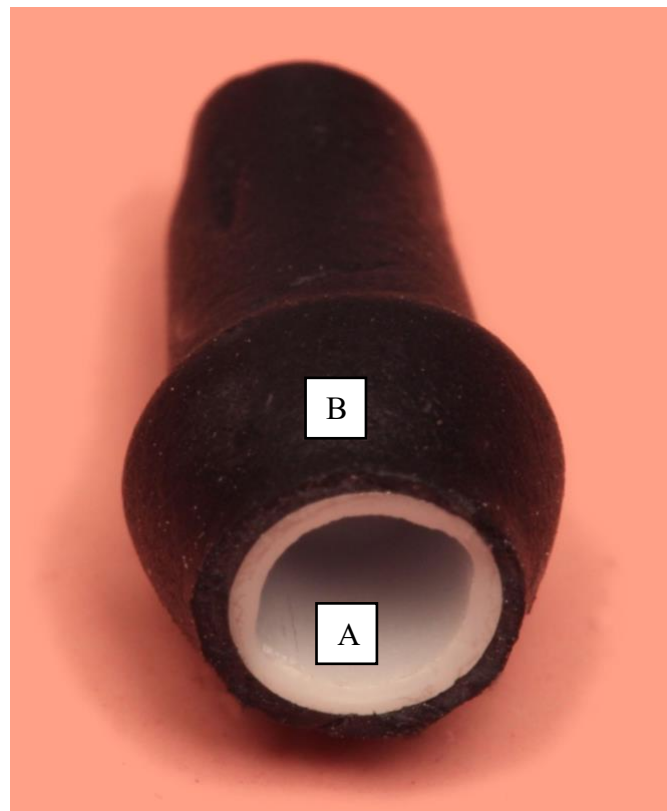


Figure 5: Protect the outer surface of the zirconia abutment from alumina particle air-abrasion .

(A) Intaglio surface of zirconia abutments

(B) The black rubber material on the outer surface of zirconia abutments



Figure 6: Waterproof marker used to mark the inner surface of zirconia abutments.



Figure 7: Stained intaglio surface of zirconia abutments.

After air-abrasion of the zirconia abutments, the black rubber protector was removed with a blade ([Figure 9](#)). Then all air-abraded titanium inserts and zirconia abutments were cleaned in an ultrasonic bath (Sonorex super RK 510 H; Bandelin, Berlin, Germany) with 99% isopropanol for 3 minutes. Blast off any alcohol supernatant with dry and oil-free air.

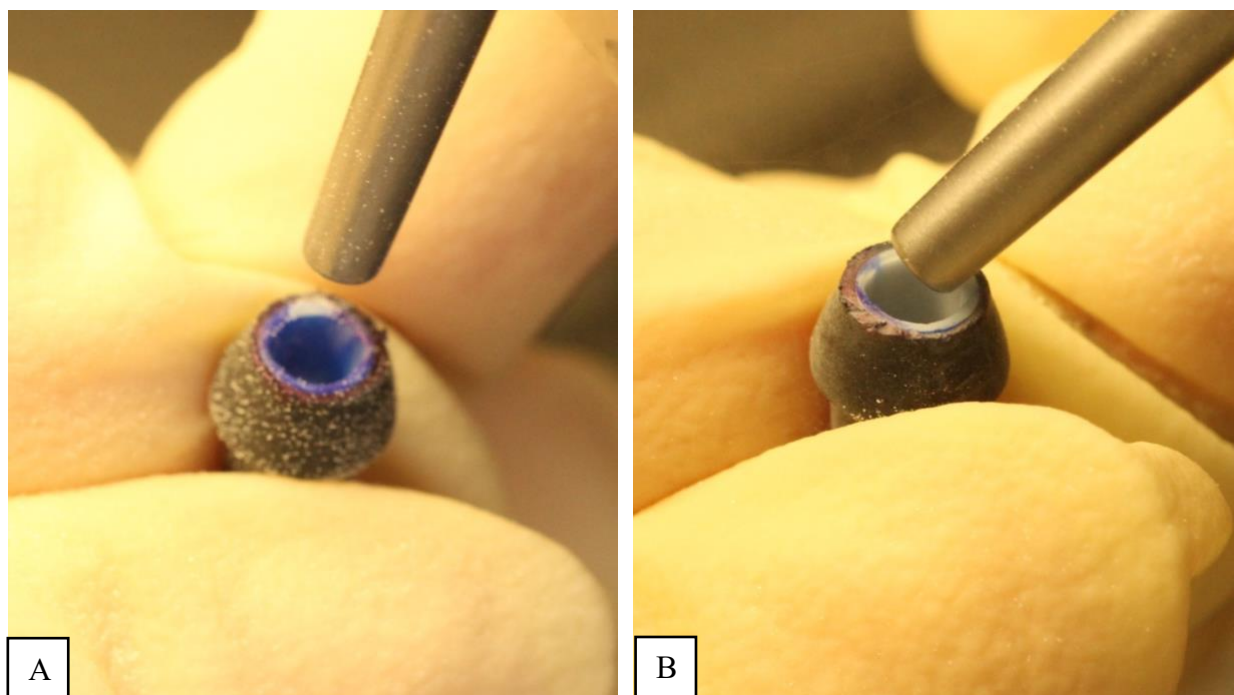


Figure 8: Alumina particle air-abrasion of the inner surface of zirconia abutments
(A) Vertical direction (90 degrees) at the shoulder
(B) Oblique direction (45 degrees) inside the canal

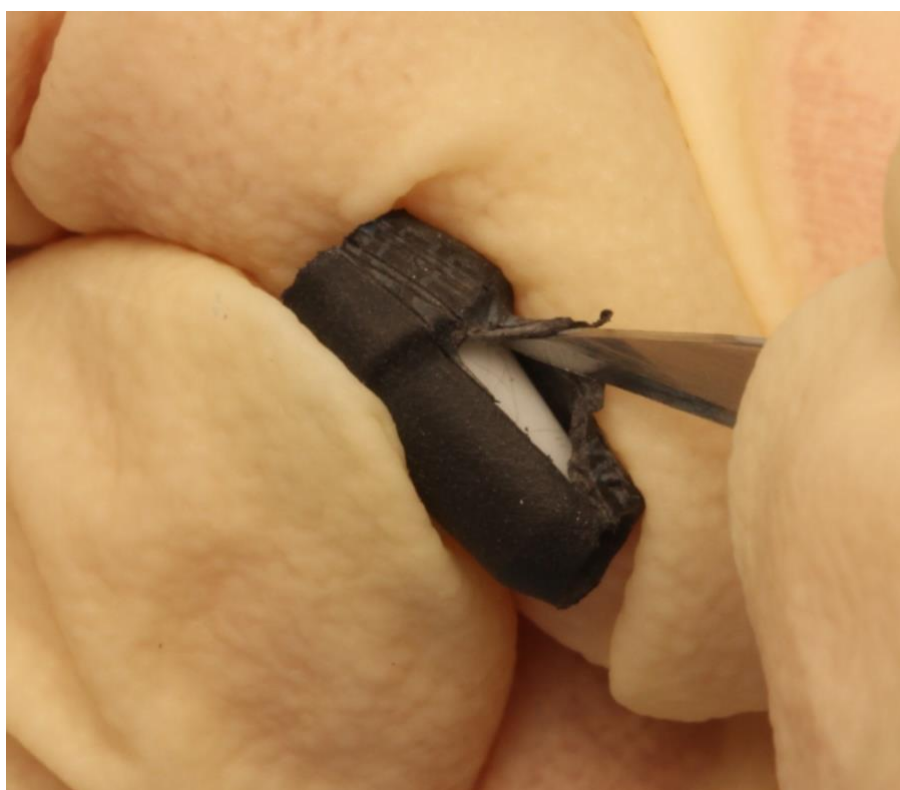


Figure 9: Removing the black rubber from the zirconia abutment.

3.3 Bonding procedures

All zirconia abutments were adhesively luted on the titanium bases using a dual-curing luting resin (DTK-adhesive; bredent) according to the manufacturer's instructions ([Figure 10](#)). The bonding device was a customized bonding apparatus shown in [Figure 11](#) (Kern and Thompson 1993).



Figure 10: DTK adhesive resin luting agent and MKZ primer

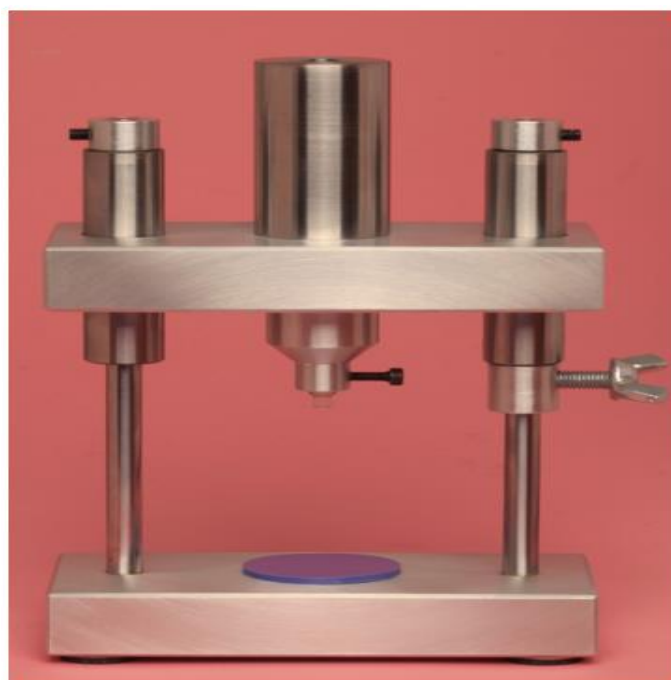


Figure 11: Bonding device (Kern and Thompson 1993)

Before the luting procedure, the correct end position of the zirconia and titanium inserts was marked with a waterproof pen.

The titanium inserts were attached again to the implant analogue and were positioned in the clamp of the customized bonding apparatus. A plastic screw was placed onto the titanium bases to protect the screw channel. Using a disposable micro brush, a universal MKZ primer was applied to the air-abraded titanium bases and zirconia abutments ([Figure 12](#) and [Figure 13](#)) The primer was applied for 60 seconds and then dried thoroughly with mild, dry, and oil-free air. A new microbrush was used for each specimen.

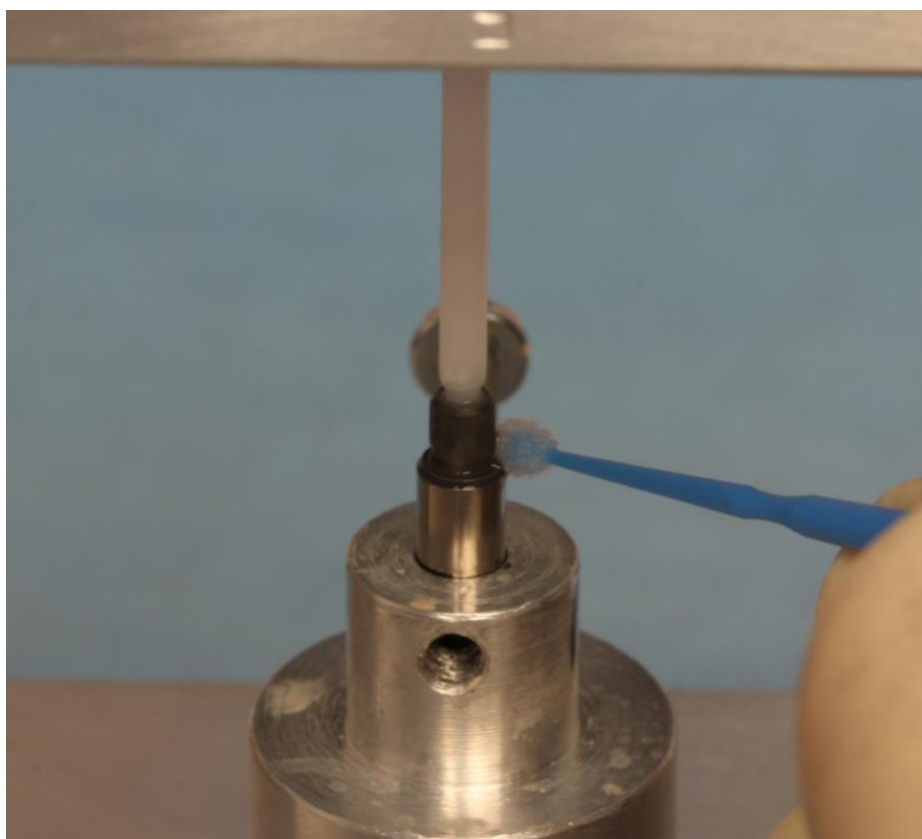


Figure 12: MKZ primer applied to a titanium base.

Before using the luting resin, a small amount was extruded and discarded from the luting paste tubes. DTK-adhesive resin was added to the titanium bases and zirconia abutments. Afterwards, the zirconia was placed over the titanium bases and the plastic screw was removed. The prosthetic screw was placed with a screwdriver to attach the hybrid abutment to the implant analogue ([Figure 14](#)).

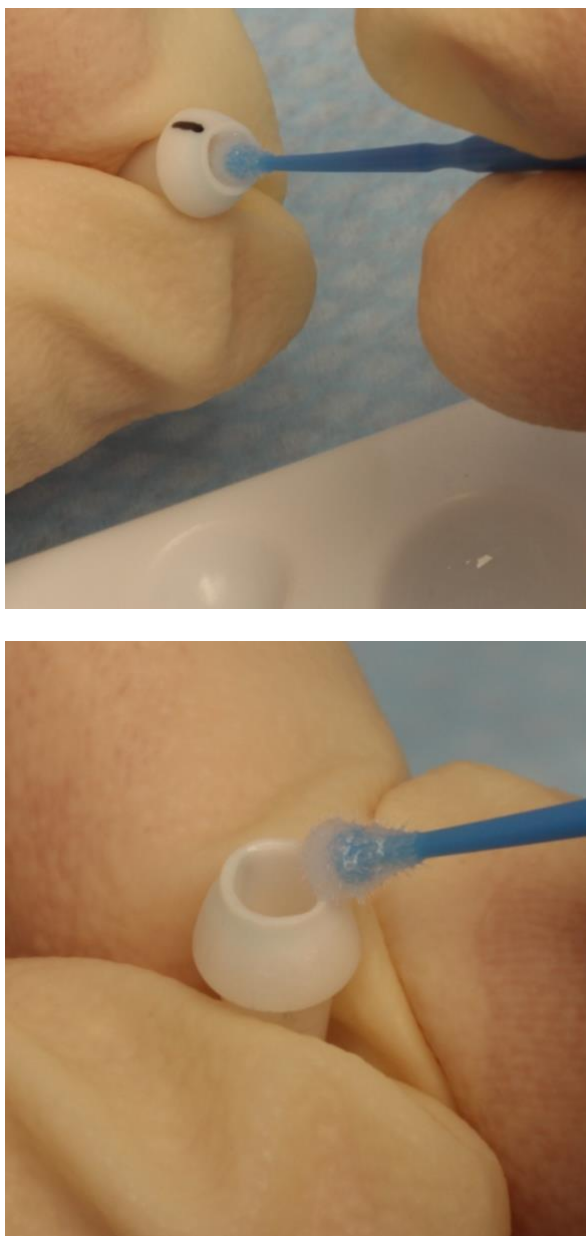


Figure 13: MKZ primer applied to zirconia abutments.

The silicon pad in the device supported the ceramic sleeves, and the specimens were squeezed firmly for 2 seconds. A new mixing tip for the luting resin was used for each specimen. All the excess luting at the margins was removed with a foam pellet (Schaumstoff-Pellets PluLine Gr.3 500St, Pluradent, Germany) ([Figure15](#)). Light curing of the margins was done by using a curing light (Elipar 2500 Halogen curing light) for 20 seconds on each surface ([Figure16](#)). The specimens were fixed at a pressure of 7.5 N in the bonding apparatus for 5 minutes. Later, the specimens were placed for 10 min in a furnace at a temperature of 37 °C to enhance chemical polymerization.

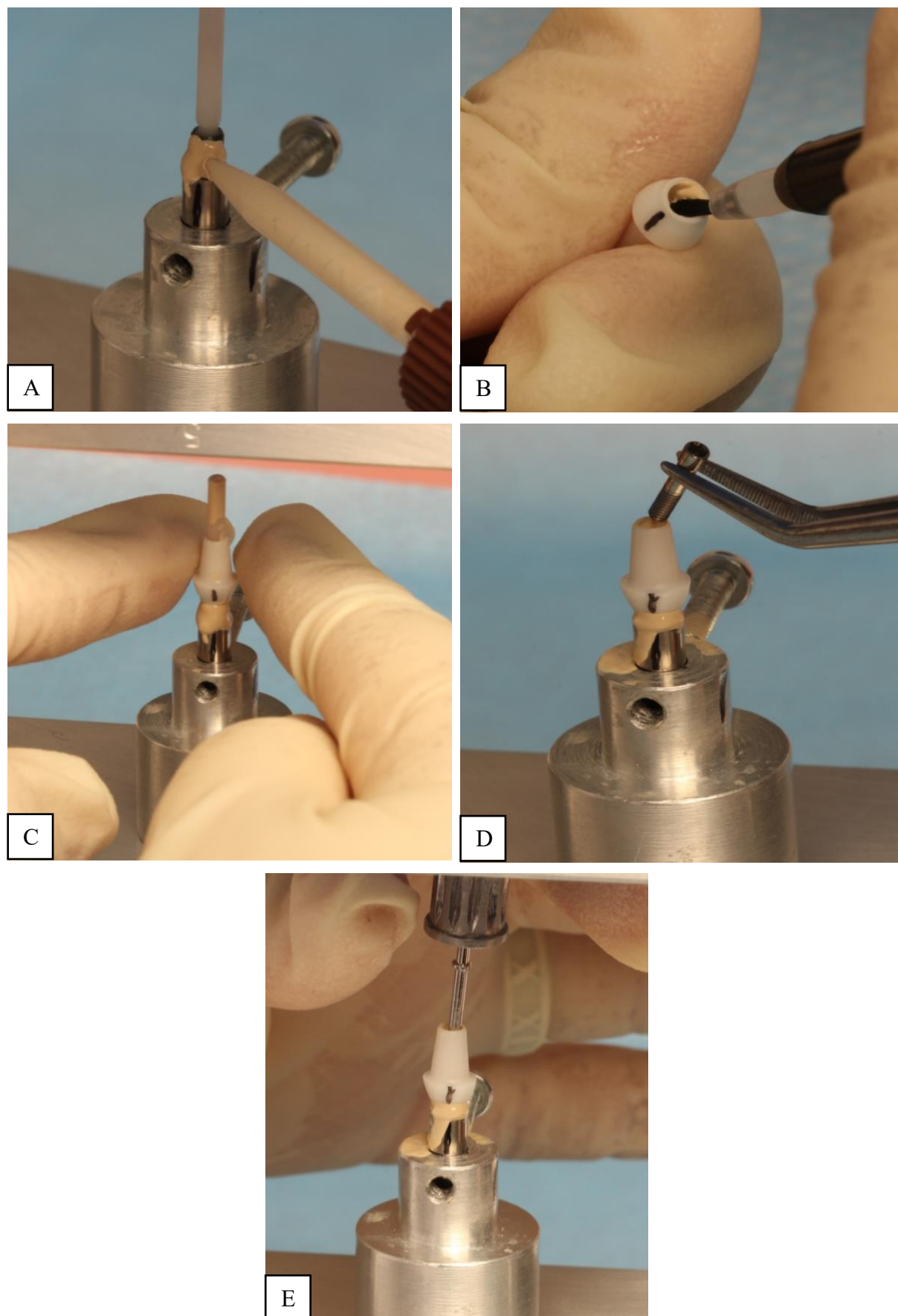


Figure 14: Application of DTK resin (A) to the titanium base, (B) to the zirconia abutment, (C) removal of the plastic screw, (D) placement of a prosthetic screw, (E) using the screwdriver

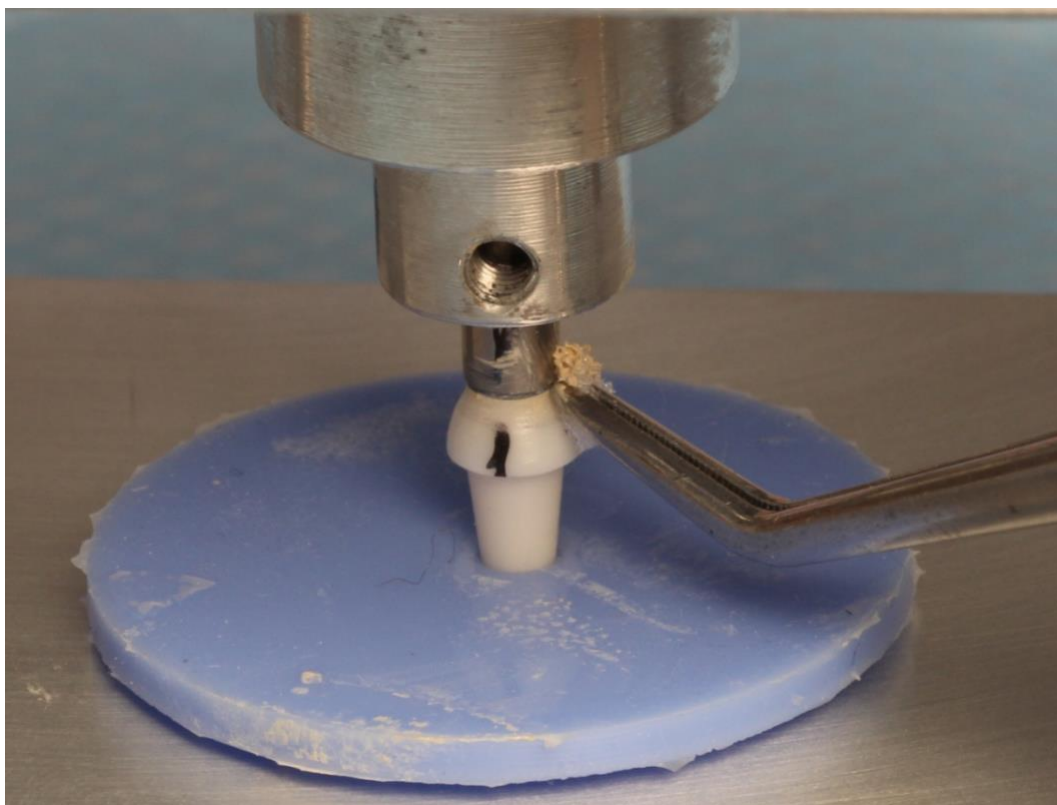


Figure 15: Removal of luting resin the excess.



Figure 16: Light curing the margins with a curing light.

After the complete setting of the luting resin, all the specimens were stored in small containers with normal saline in their groups (A, B, C, D) and kept in a 37°C water bath (incubator) for three days ([Figure 17](#)).



Figure 17: Water bath incubator

3.4 Disinfection and Sterilization

After three days, each group was randomly further subdivided into 2 subgroups (n=16); for subgroup 1, the specimens were only disinfected, whereas for subgroup 2, the specimens were subjected to disinfection and then to autoclave sterilization. The disinfection was conducted using the FINOVO cleaning system (bredent) ([Figure 18](#)) according to the manufacturer's instructions.



Figure 18: FINOVO cleaning system.

FINEVO cleaning system is an ultrasonic method with three different liquids to remove the residues from titanium, zirconia, and hybrid ceramic surfaces. The FINEVO liquid set ([Figure 19](#)) consists of:

- 1) FINEVO 1: Cleaning liquid with disinfectant action
- 2) FINEVO 2: Ethanol alcohol 80%
- 3) FINEVO 3: Purified water



Figure 19: FINEVO liquid set.

The protocol of the FINEVO cleaning system ([Figure 20](#)) was as follows:
Deep ultrasonic cleaning of the implant abutments in 3 short stages,
Step 1: Started with FINEVO solution 1 for 5 min at 30 C.
Step 2: Followed by placing the abutments in Ethanol alcohol for another 5 min.
Step 3: Placed the abutments in the distilled water for 5 min.
After the cycle was completed, all the abutments were dried with dry, oil-free air and placed in clean pouches. Half of the specimens only were sterilized (subgroup 2), while the other half were not sterilized (subgroup 1).



Figure 20: Steps of the FINEVO cleaning system (A) Step 1, (B) Step 2, and (C) Step 3

3.5 Sterilization process

The specimens of the second subgroup were placed in heat-sealable pouches and loaded into a class B dental autoclave (Vacuklav 24 autoclave sterilizer, Melag Medizintechnik, Berlin, Germany) ([Figure 21](#)) Specimens from each group (A, B, C, D) were packed individually and labelled ([Figure 22](#)). They were sterilized in steam autoclaving at a short cycle (5 minutes at 134°C under a pressure of 2 bar).



Figure 21: Vacuklav 24 autoclave sterilizer



Figure 22: Specimens in sterilization pouches

After disinfection and sterilization, the torque wrench was calibrated to ensure accurate torque (25 Ncm) was delivered to the screw, which was further screwed again after 10 minutes ([Figure 23](#)). All the screw channels were sealed with polytetrafluoroethylene tape ([Figure 24](#)) and flowable composite resin ([Figure 25](#)).



Figure 23: Torque tester

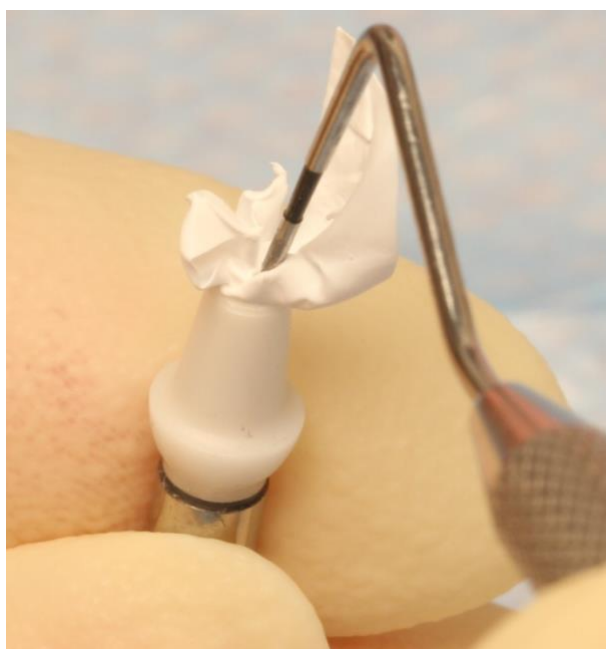


Figure 24: Sealing the screw channel with polytetrafluoroethylene tape

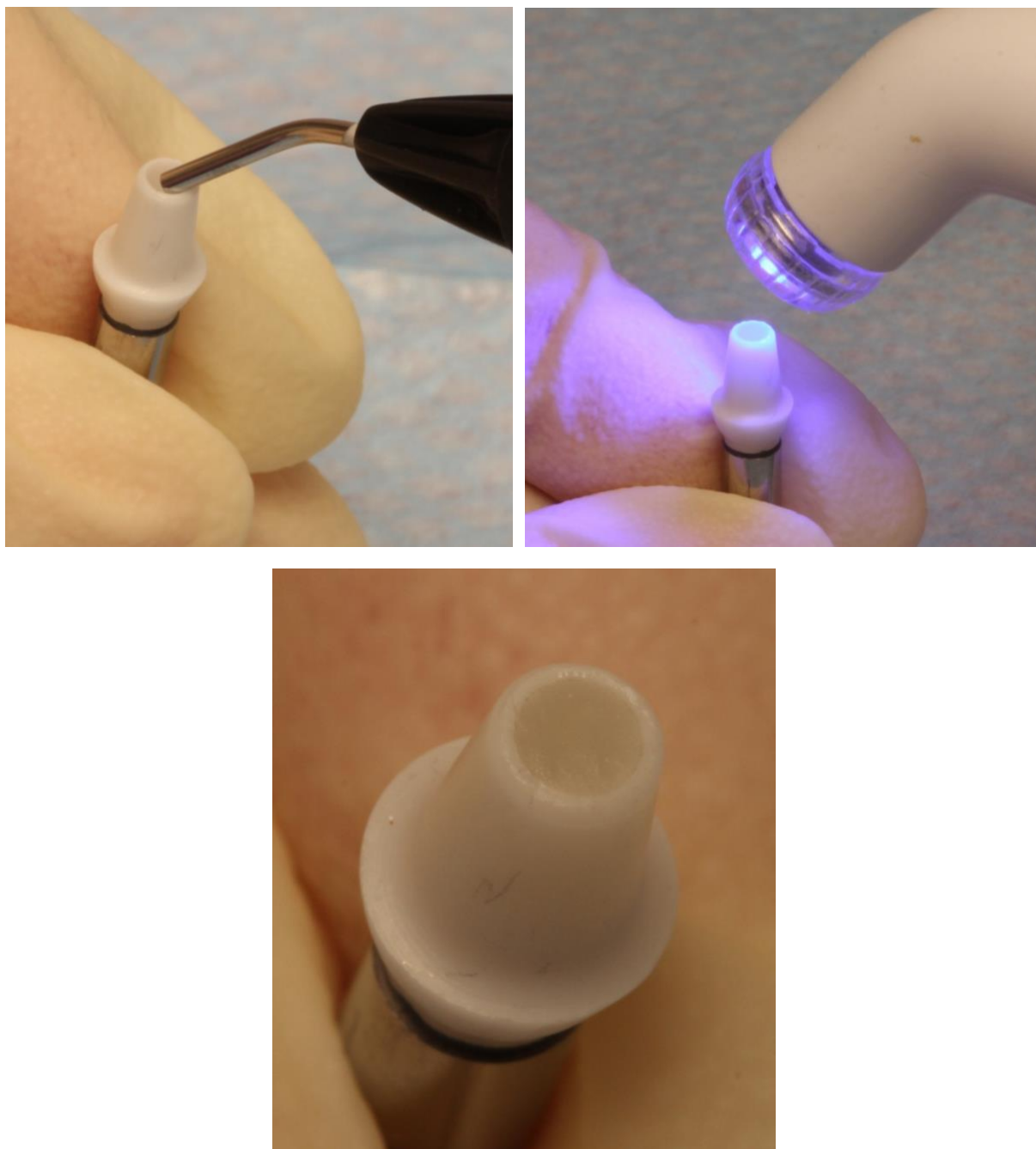


Figure 25: Sealing the screw channel with flowable composite resin.

3.6 Artificial aging

Finally, each subgroup was randomly further subdivided into two subgroups ($n=8$). The first half of the specimens in each subgroup (N) were directly subjected to the axial retention test, without undergoing artificial aging (Groups (A1N, B1N, C1N, D1N, A2N, B2N, C2N, D2N)). The other half (T) were first thermally aged for 150 days and then subjected to the axial retention test. These groups were (A1T, B1T, C1T, D1T, A2T, B2T, C2T, D2T) wrapped in a small piece of fabric and marked with a tag made of acrylic materials carved with the number of each group ([Figure 26](#)). The specimens were stored in a thermocycling machine for 150 days

for the thermal aging. Within this time, the specimens were subjected to 75,000 cycles of thermal cycling from 5°C to 55°C, with regular rearrangement of the specimens, 30 seconds of residence time in the pools, and 5 seconds of dripping time ([Figure 27](#)).



Figure 26: Groups that were subjected to thermal aging.

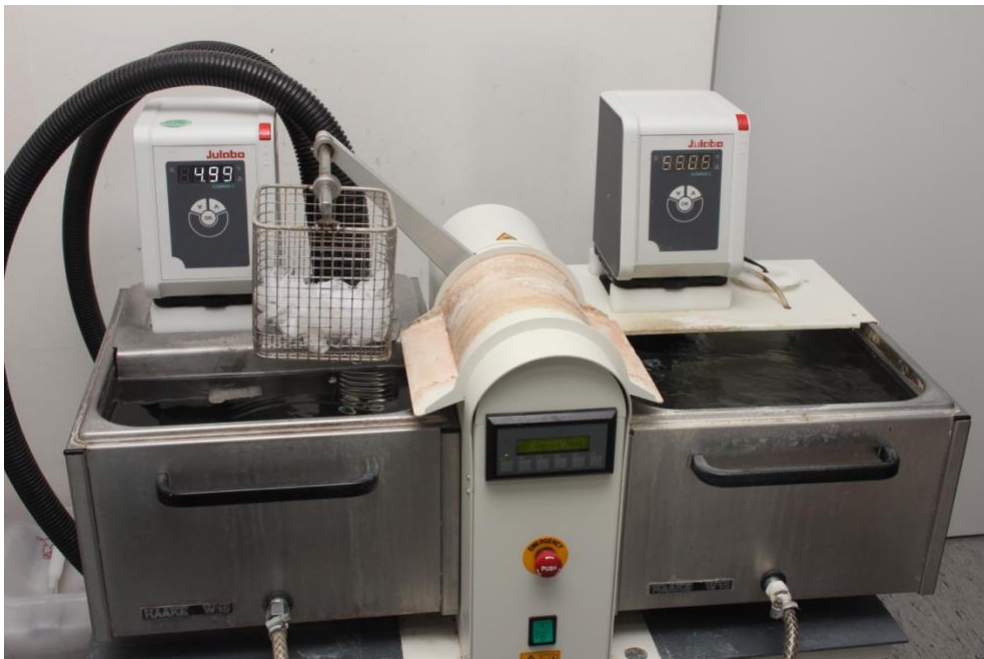


Figure 27: Thermocycling machine

3.7 Axial retention test

The axial retention test was applied using a universal testing machine, Zwick Z010 (Zwick, Ulm, Germany) ([Figure 28](#)). For the test to be done securely, a custom-made holding device with two chains was fabricated to position the hybrid abutment assembly, which allowed tilting-free testing. The abutments were taken out and pulled apart at a tension speed of 2 mm/min to measure the force developed until the adhesive bond failed. The load required to dislodge components of each hybrid abutment was recorded in Newton (N).

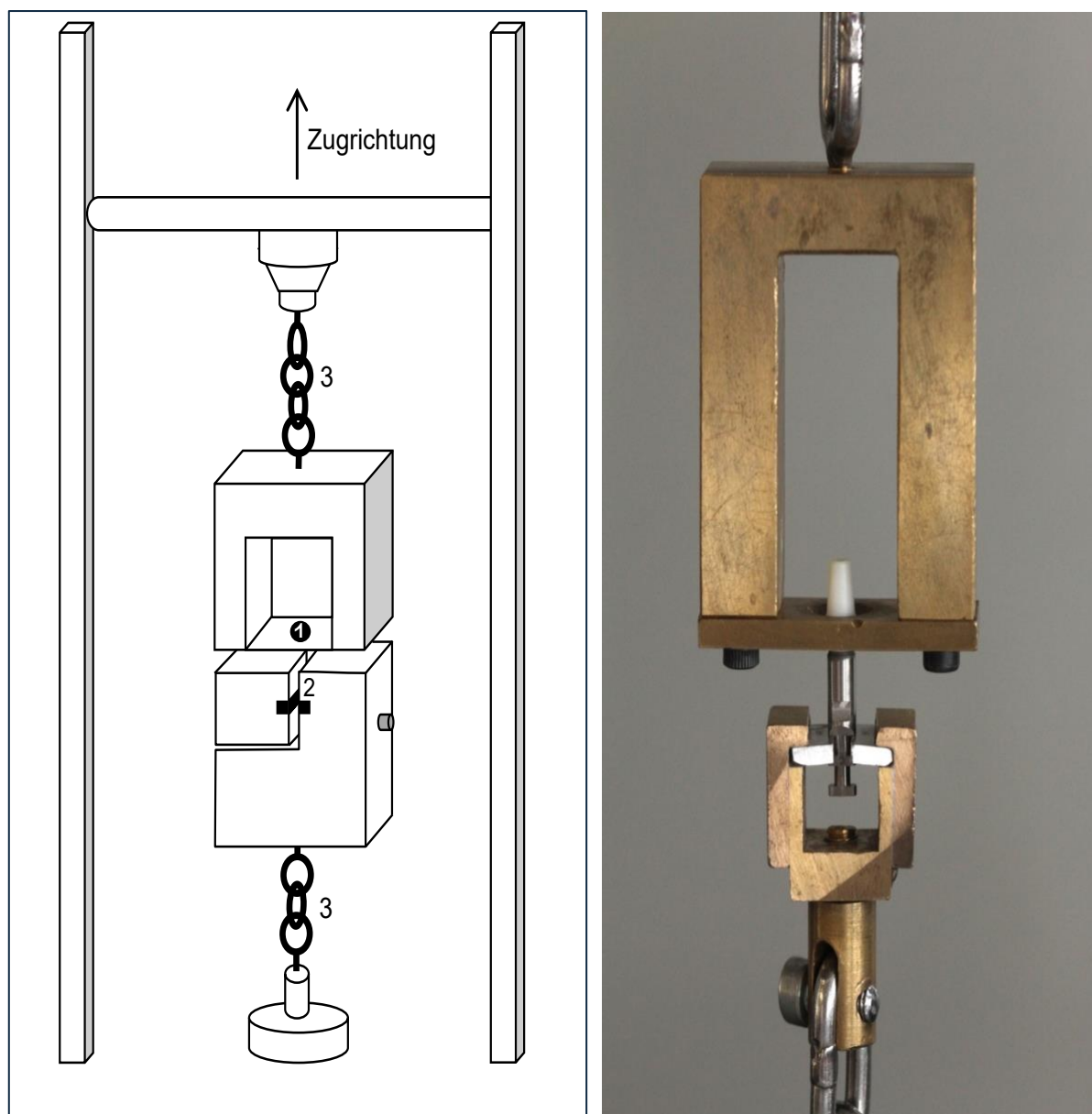


Figure 28: Universal testing machine Zwick Z010 (1) Opening for the ceramic sleeve (2) Opening for titanium bases (3) Chains for torsion-free.

3.8 Mode of failure

All specimens were inspected after the axial retention test to evaluate the failure mode. The failure modes were analyzed by using an optical light microscope at $\times 25$ magnification (Carl Zeiss, Oberkochen, Germany) and a connected digital camera (Canon EOS 60D, Canon, City, Japan) ([Figure 29](#)). The failure mode was categorized as follows:

- Type 1: Luting resin remained on the titanium insert (adhesive failure between zirconia and luting resin).
- Type 2: Luting resin remained on the zirconia surface (adhesive failure between titanium and luting resin).
- Type 3: Luting resin remained on both components (cohesive failure).

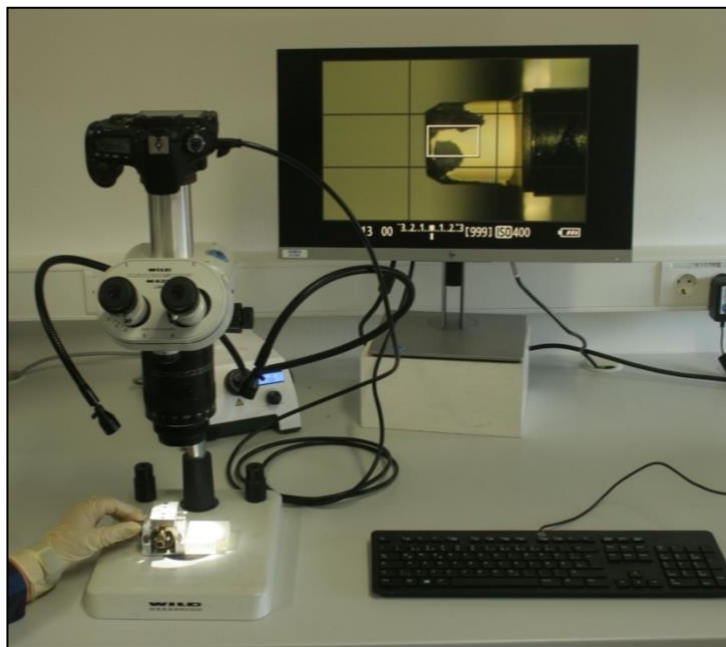


Figure 29: Optical light microscope connected to a digital camera for failure mode evaluation.

3.9 Statistical analysis

The collected data was revised, coded, tabulated, and introduced to a PC using Statistical Package for Social Science (SPSS 20.0 for Windows, SPSS Inc, Chicago, IL, USA). The Shapiro-Wilk test revealed that the data was not normally distributed. Four values (out of 128) were considered outliers (The distance is $3 \times \text{IQR}$ from the median, and they were in total $< 5\%$). Therefore, these four values were removed from the analysis. Statistical analysis was performed

using three-way ANOVA to detect interaction between different variables ($p=0.415$), additional statistical analysis was performed by using separate one-way ANOVAs, followed by Tukey test for post hoc pairwise comparisons among groups regarding the treatment variable. The level of significance (p value) was set at 0.05 (95% confidence interval) for all tests.

4. Results

The median values, the mean values, as well as the corresponding standard deviations of the retention tests were calculated for each group, respectively, and are shown in [Table 2](#) and in addition are illustrated as a boxplot in [Figure 30](#)). The median retentive strength ranged from a minimum of 688 N for group C1T to a maximum of 1390 N for group B2N.

Table 2: The retentive strength of the test groups

Gr. Nr.	Group	Retentive strength (N)	
		Median	Mean $\pm$ SD
1	A1N	1165	1123 $\pm$ 168A _a α
2	A2N	1140	1145 $\pm$ 108B _a α
3	B1N	1230	1215 $\pm$ 72A _a α
4	B2N	1390	1339 $\pm$ 154A _a α
5	C1N	931	901 $\pm$ 185B _a α
6	C2N	1120	1063 $\pm$ 123B _a α
7	D1N	1180	1154 $\pm$ 128A _a α
8	D2N	1175	1162 $\pm$ 168AB _a α
9	A1T	882	929 $\pm$ 200A _a β
10	A2T	1235	1256 $\pm$ 185A _a α
11	B1T	988	983 $\pm$ 84A _b α
12	B2T	1120	1158 $\pm$ 221A _a α
13	C1T	688	709 $\pm$ 128B _b β
14	C2T	1005	996 $\pm$ 254A _a α
15	D1T	933	866 $\pm$ 138AB _b β
16	D2T	1070	1136 $\pm$ 211A _a α

Note: Different superscript uppercase letters indicate significant differences ($p \leq 0.05$) between different treatments for the same mode of sterilization and thermal cycling protocol. Different subscript lowercase letters indicate significant differences ($p \leq 0.05$) regarding the followed thermal cycling protocol for the same mode of sterilization and the same treatment. Different

superscript Greek letters indicate significant differences ($p \leq 0.05$) regarding the followed sterilization protocol for the same thermal cycling protocol and same treatment.

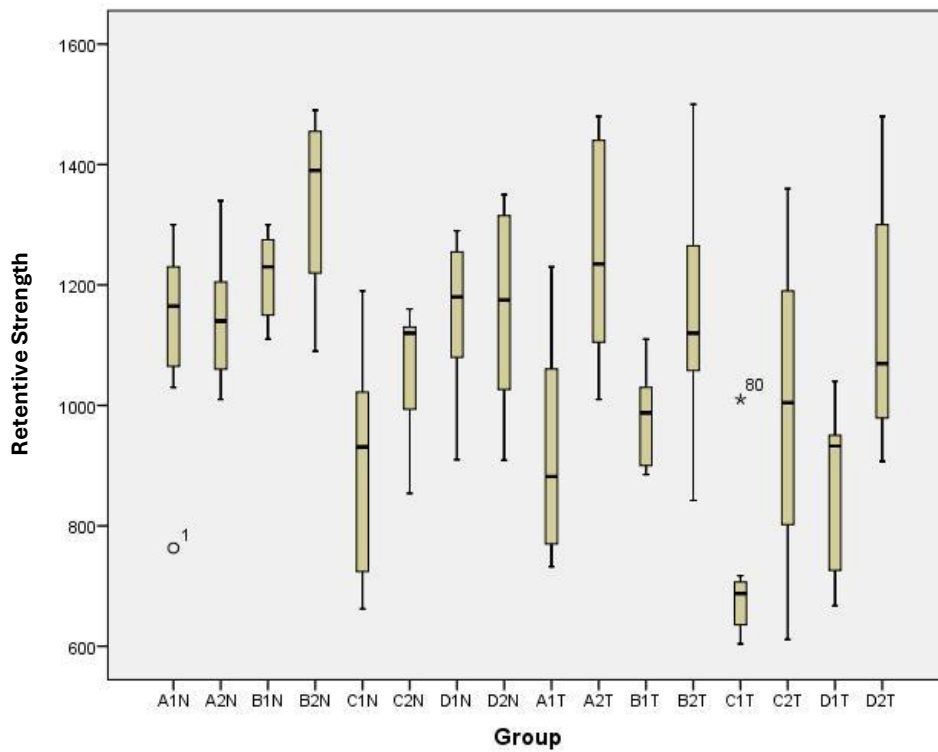


Figure 30: Boxplot of the retentive strength (N) of test groups

The results of the three-way ANOVA test are shown in (Table 3). No significant interaction ($p > 0.05$) was detected between the three different variables (treatment, sterilization and thermocycling) regarding the effect on the resulting retentive strength. The sterilization as a variable had a statistically significant positive effect ($p \leq 0.05$) on the retentive strength of groups that underwent thermocycling with the exception of sub- groups B. However, the sterilization had no significant effect on the retentive strength when thermocycling was not applied.

Whereas, thermocycling as a variable had a statistically significant negative effect ($p \leq 0.05$) on the retentive strength only in the absence of sterilization with the exception of subgroups A. For the groups that underwent the sterilization process, thermocycling had no statistically significant effect on the retentive strength. The followed treatment protocol had no effect on the resulting retentive strength for the groups that underwent thermocycling and followed the sterilization protocol.

Table 3: Results of the three-way ANOVA test

Test of between-subjects effects					
Dependent variable: retentive strength					
Source	Type III sum of Square	Df	Mean square	F	Sig
Corrected	3135651.753 ^a	15	209043.450	7634	.000
Intercept	141305325.708	1	141305325.708	5.160.195	.000
Thermocycling	548395.255	1	548395.255	20.026	.000
Sterilization	908336.099	1	908336.099	33.171	.000
Treatment	1117447.036	3	372482.345	13.602	.000
Thermocycling * sterilization	265504.397	1	265504.397	9.696	.002
Thermocycling * treatment	108020.588	3	36006.863	1.315	.273
Sterilization* treatment	34261.836	3	11420.612	.417	.714
Thermocycling * Sterilization * treatment	78859.719	3	26286.573	.960	.415
Error	2957441.690	108	27383.719		
Total	148769917.000	124			
Corrected total	6093093.444	123			

^a $R^2 = .515$ (adjusted $R^2 = .447$).

The observed failure mode was completely type 3 (mixed) ([Figure 31](#)), except for the detected screw fracture in 2 specimens from group B2N and 1 specimen from group D2T. The percentages of adhesive failure mode and cohesive failure mode were determined (appendixes).

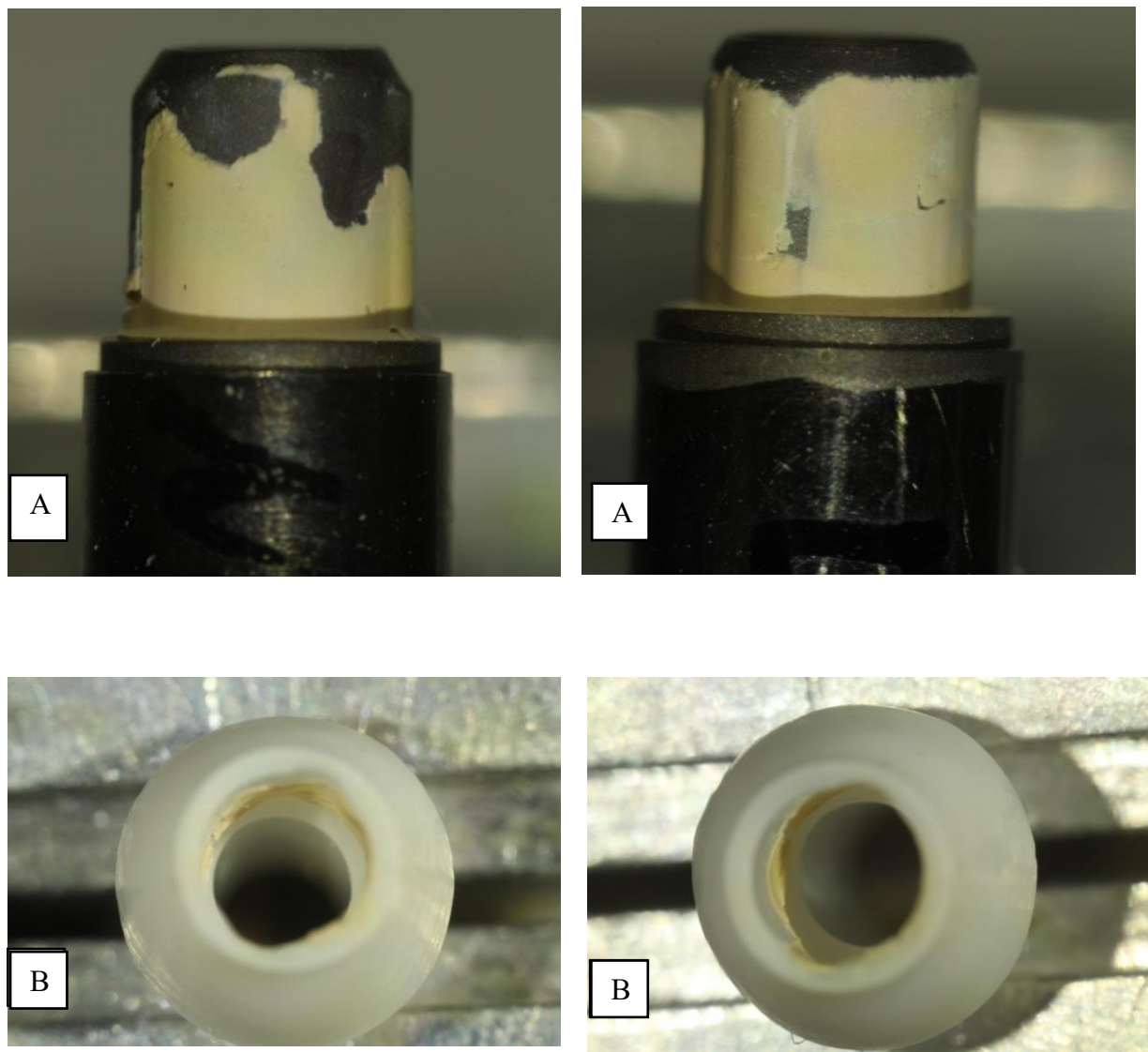


Figure 31: Mode of failure of the luting resin (cohesive failure) (A) luting resin adhering to titanium bases, and (B) luting adhering to zirconia abutments.

5. Discussion

5.1 Discussion of Methodology

This laboratory study aimed to evaluate the retention of zirconia hybrid abutments bonded to titanium inserts under three variables. The study factors were the different zirconia surface conditioning parameters, the effect of sterilization, and the effect of thermal cycling.

5.1.1 Zirconia hybrid abutments

This in-vitro study was conducted using prefabricated hybrid zirconia abutments, or “two-piece” zirconia abutments, which consist of zirconia copings bonded to titanium inserts. These abutments were selected because they have the advantages of both zirconia and titanium abutments, including better esthetics, improved mechanical properties and superior biological response, with no adverse effects on the implant-abutment interface (28, 29). Moreover, several studies showed that hybrid abutments had high fracture resistance and less wear of implant connection compared to one-piece zirconia abutments (32, 33). Moreover, due to the large number of specimens, same-charged prefabricated hybrid zirconia abutments were used to ensure the reliability of the results and maintain standardization of all parameters. All parameters of the hybrid abutment were consistent, including the height of the titanium bases, the height and diameter of the zirconia abutments, angulation, geometry, and the luting gap. In addition, commonly clinically used short titanium bases (4.5 mm) were used to test the ability of the luting resin to retain the abutment without relying on the height of the titanium bases.

5.1.2 Dual-curing luting resin

Composite resin luting agents are more suitable than glass ionomer cement or conventional cement for long-term durable bonding of zirconia abutments to titanium inserts under clinical conditions. (35, 75) Chemical and dual-curing bonding systems have been used to bond zirconia abutments to titanium inserts because of the difficulty of using light cure under the opaque zirconia surface (66). In this study, all zirconia abutments were adhesively luted on the titanium bases using a dual-curing luting resin (DTK adhesive resin and MKZ primer), which is indicated for bonding of restorations for implant abutments. The reason for the durable bonding of resin luting agents is that they have phosphate monomer (MDP), which is the best documented and most commonly used phosphate monomer. MDP monomers contribute mainly to the chemical bond to zirconia surface through the chemical interaction between the phosphate ester group of MDP and the hydroxyl group on the zirconia surface. (35, 47, 75) Moreover, the

bonding device used for all specimens was a customized bonding apparatus (Kern and Thompson 1993), with a 7.5 N load for 5 minutes to ensure the standard film thickness of the luting resin until its complete setting.

5.1.3 Surface conditioning of titanium bases and zirconia abutments

Air particle abrasion of the bonding surfaces of titanium and zirconia components with aluminum oxide particles is one of the most effective methods to increase the retentive and bonding strength of the luting resin (4, 26, 35). In the present study, the bonding surfaces of titanium bases were air-abraded vertically using 50 μm alumina particles at 2.5 bar pressure from a distance of 10 mm for 15 seconds at a right angle (90 degrees), as recommended by many studies to enhance the mechanical retention between the luting resin and titanium bases. It has been shown that mechanical treatment of titanium surfaces using air particle abrasion with 50 μm alumina particles provided the most stable bonding to luting resin (10, 36).

Air abrasion of zirconia surfaces had an important role in enhancing the bonding strength of resin to oxide ceramics, as has been shown in previous studies. This is achieved by increasing the surface roughness and activating the zirconia surface (67). A study by Wolfart et al. demonstrated that conditioning the zirconia surface with alumina particles at 0.25 MPa enhanced the bonding strength between zirconia and luting resin (68).

In this study, we evaluated the retentive strength of hybrid abutments under different surface pretreatments (varying particle sizes and pressures) to assess the impact of various surface conditioning parameters on the long-term resin bond strength of zirconia ceramics. Air abrasion of zirconia surfaces was conducted using 50 μm alumina particles at 1.0 bar pressure, 50 μm at 2.0 bar pressure, 100 μm at 1.0 bar pressure, or 100 μm at 2.0 bar pressure. The inner surfaces of the zirconia abutments were marked with a blue waterproof marker prior to the air abrasion. The purpose of the blue marker was to facilitate effective conditioning of the zirconia by visualizing the surface roughening effect through the disappearance of the blue color. The air abrasion of the inner surface of zirconia abutments was performed in an oblique direction (45 degrees) to access the zirconia inner canal for effective air abrasion, and at a right angle (90 degrees) in the shoulder flat area from a distance of 10 mm.

5.1.4 Sterilization process

Many cleaning and sterilization methods have been used for the implant abutments to improve the peri-implant attachment of the mucosa and, consequently, the long-term success

of the implant treatment (57). The microbial contamination of the hybrid abutments can increase the chance of the inflammatory response and peri-implant bone loss and affect implant longevity (57, 58).

The laboratory bonding procedure for hybrid abutments is technique-sensitive as it may lead to contamination of the two-piece zirconia abutments. Numerous sterilization methods have been employed in dentistry, aiming to eliminate the reproduction of microorganisms, such as bacteria and fungi, to safeguard the implant-abutment junction (61, 62). Steam autoclaving is the sterilization method recommended for sterilizing implant parts because of its effectiveness, low cost and convenience (61). It is recommended to sterilize hybrid abutments before inserting them intraorally. In this study, after bonding procedures, we disinfected hybrid abutments using the FINOVO ultrasonic cleaning system. After disinfection, specified groups of specimens were sterilized in steam autoclaving at a short cycle (5 minutes at 134 °C under a pressure of 2 bar). However, luting resins may be affected by moisture or heat during autoclave sterilization (64). To date, there is no evidence about the long-term bonding durability and the effect of sterilization on the retentive strength of zirconia hybrid abutments.

5.1.5 Thermal aging

Water storage and thermocycling are suitable artificial aging methods for evaluating the long-term durability of the bonding strength of luting resin to the zirconia surface (69,71). This is due to the fact that water storage and thermal cycling can result in water absorption and volume expansion, which may damage or affect the retention of luting agents (69, 71,76).

To assess the bonding retention in clinical conditions, we utilised a reliable well-established thermocycling artificial ageing protocol. The specimens in this study were placed in a thermocycling machine and underwent 75,000 cycles of thermal cycling, ranging from 5°C to 55°C, over a period of 150 days.

5.2 Discussion of Results

Statistical analysis of the present study detected no significant interaction ($p>0.05$) between the three different variables (surface treatment, sterilization and thermocycling) regarding the effect on the resulting retentive strength (4, 26, 35).

5.2.1 Influence of surface conditioning of titanium bases and zirconia abutments

Regarding the surface pretreatment factor, the highest retentive strength was recorded for group that was air-abraded with 50 µm alumina particles at 2 bar pressure, subjected to

sterilization, but not exposed to thermal cycling (B2N), with a median value of 1390 N. In contrast, the lowest was recorded for group that was air-abraded with 110 μm alumina particles at 1 bar pressure, not sterilized, but subjected to thermal aging for 150 days (C1T), with a median value of 688 N. There was a notable difference in retentive strength between the groups abraded at 1 bar and 2 bar pressure. The groups in which zirconia surfaces were air-abraded with 50 μm and 100 μm alumina particles at 2 bar pressure (Groups B and D) exhibited higher median retentive strength compared to the groups abraded with 50 μm and 100 μm particles at 1 bar pressure (Groups A and C). The main reason for the better retentive strength in groups abraded at 2 bar pressure is the angle of air-abrasion of the intaglio surfaces of zirconia abutments. The air abrasion was performed in an oblique direction toward the canal (intaglio surface of the zirconia) to allow better cleaning and to increase the bonding surface area of the zirconia surface. Due to the oblique orientation of alumina particles, the actual pressure effecting the zirconia surface is lower than the actual pressure generated by the blasting unit. Therefore, air-abrasion at 2 bar pressure in oblique orientation produces better surface roughness and increases the bonding surface area without any possible surface damage from microcracks that can be stress concentration sources (46).

Nevertheless, the current study found no significant difference between different conditioning methods on the resulting retentive strength. Several studies were in accordance with the results of the present study, such as a study by Gehrke et al. that reported no significant difference in the retentive strength of hybrid implant abutments bonded with different luting resins with different surface conditioning methods of zirconia copings (4). In addition, a previous study concluded that using primers containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) in combination with airborne-particle abrasion led to a significant increase in the bond strength of luting agent to zirconia (24). This is in concert with the results of the present study, where an MDP-containing primer (MKZ) was used. This conclusion could be explained by forming a chemical bond between metal oxides of the zirconia surfaces and the phosphate monomers in the primer, enhancing the long-term bond between zirconia and titanium inserts (26, 47, 68). A study by Hansen et al. investigated the influence of alumina particle air-abrasion on the zirconia surface with different pressures applied and found no significant effect on the retentive strength between zirconia copings and titanium bases luted with MDP-containing luting resin and primers (69). Thus, the first null hypothesis that different surface pretreatment would not influence the retentive strength was accepted.

5.2.2 Influence of sterilization

Regarding the sterilization factor, autoclave sterilization increased the retentive strength significantly for all groups regardless of the conditioning methods, whether thermocycling was applied or not ($p \leq 0.05$). An explanation for this could be that heat and pressure applied during the sterilization process improved the cross-linking within the polymer network, which would improve the mechanical characteristics of the luting resin and thus increase the retentive strength (70). Also, a study by Bergamo et al. concluded that autoclave sterilization increased the retention of zirconia crowns bonded to titanium-base abutments when a self-adhesive resin and no titanium-base surface pretreatment was used. However, that study showed that sterilization did not influence the bond strength when a conventional luting resin or air-abraded titanium bases were used (62). Furthermore, another study evaluated the effect of steam autoclaving on the bonding strength using chemically curing or dual-curing luting resin for luting two-piece zirconia abutments. The study showed no weakening effect on the retention of the components. However, the authors reported increased retentive strength for adhesively bonded groups using chemically curing luting resins (66). A study by Wiedenmann et al. investigated the retentive strength between zirconia abutments and titanium inserts luted using two different luting resins. The study reported that other cleaning procedures, including autoclave sterilization, did not affect the retentive strength (57). A similar study revealed no weakening or negative effect of autoclave sterilization on the retention of zirconia abutments adhesively luted to titanium bases in two-piece implant abutments (59). In the current study, sterilization increased the retention of the hybrid abutments. Therefore, the second hypothesis was rejected.

5.2.3 Influence of thermal aging

Water storage and thermocycling are adequate artificial aging methods for testing the long-term durability of resin bonding (71). Regarding the thermal aging factor as a variable, an adversely significant effect on the retentive strength was detected only in the absence of sterilization. The retentive strength decreased after 150 days of water storage with thermal cycling. Thermocycling had no statistically significant effect on the retention values for the groups that underwent the sterilization process. The rationale behind this result may lie on the high degree of conversion (DC) of the molecules within the luting resin due to the thermal energy supplied by the sterilization process. The resulted improved DC will increase the hardness, lower the solubility, and increase the compressive and flexural strength values of

luting resin. Therefore, it is reasonable that luting resin that polymerized at high temperatures (sterilized specimens) showed higher DC than luting resin polymerized at room temperatures (non-sterilized specimens). A study by Daronch et al. reported that pre-heating the resin composite to 50 °C or 60 °C increases their degree of conversion (72, 73). A similar study found that increased temperatures of resin luting agent before polymerization increased the DC and improved the mechanical properties of resin luting agent (74).

The results of the current study agreed with those of a previous study that evaluated the bond strength of hybrid abutments after thermocycling. A study by Mehl et al. evaluated the tensile strength of zirconia abutments on titanium bases using four different self-adhesive luting resins. They reported a significant reduction in retention after 150 days of thermocycling; the most excellent retentive values were achieved in groups luted with auto-curing luting resin containing an MDP monomer (75). A study by Gungor et al. tested the effect of luting resin type and thermal aging on the retentive strength of zirconia abutment bonded to titanium inserts. The study found that Zirconite composite resin had the highest retentive strength and that thermal ageing resulted in significantly lower retention compared to the control groups for all luting resins, which is in accordance with the results of our study (76). An explanation for the low retentive strength values recorded in the study above might be the absence of air-particle abrasion of the bonding surfaces of the zirconia abutments. Therefore, the third hypothesis was partially rejected because thermal aging decreases the retentive strength only in the absence of sterilization. Thermal aging had no adverse effect on sterilized specimens.

5.2.4 Failure mode of luting resin

After the retention test, the failure modes at the titanium and zirconia bonding surfaces were found to be a completely cohesive failure with luting resin retained on both zirconia and titanium surfaces. This observation supports the benefit of alumina particle air-abrasion and using primers to improve the adhesion between the luting resin and bonding surfaces. This finding is consistent with the results of another study that compared the retention of hybrid abutments luted with two types of resin materials after steam sterilization and found that the mode of resin failure was mainly the mixed type (66). Furthermore, the failure modes of air-abraded groups of zirconia abutments in a previous study were cohesive failures (26). This shows the correlation between higher retention of luting resin to zirconia surfaces and higher percentages of cohesive failures.

6. Conclusions

Based on the findings of this current laboratory study, the following conclusions can be drawn:

1. Airborne-particle abrasion of the intaglio surfaces of zirconia abutments with alumina particles at 1.0 to 2.0 bar pressure, and using MDP-containing primers, provided durable bonding of zirconia abutments to titanium inserts.
2. Autoclave sterilization significantly positively affected the retentive strength between zirconia abutments and titanium inserts. Therefore, steam autoclaving is highly recommended before the final installation of hybrid abutments onto implants.
3. Using DTK resin luting agent in two-piece zirconia abutments resulted in a good performance after sterilization and thermocycling without any significant reduction in their retentive strength.

7. Summary

The objective of this study was to assess the impact of sterilisation, conditioning methods, and thermal ageing on the retention strength of two-piece zirconia abutments.

A total of 128 stock zirconia abutments were divided into 4 groups, each including 32 abutments. The groups were categorized based on the conditioning parameters used: Air-abrasion with (A) 50 μm alumina particles at 1.0 bar pressure, (B) 50 μm particles at 2.0 bar pressure, (C) 100 μm particles at 1.0 bar pressure, and (D) 100 μm particles at 2.0 bar pressure. All abutments were bonded to titanium bases using DTK adhesive resin and stored in a water bath at a temperature of 37°C for a duration of 72 hours.

The groups were divided into two subgroups (n=16) each. Group 1 was disinfected, while Group 2 underwent both disinfection and autoclave sterilisation. Half of the specimens from each grouping (n=8) were directly submitted to the axial retention test (group N), whereas the remaining half underwent 150 days of thermocycling before the retention test (group T).

The data was subjected to statistical analysis using a three-way analysis of variance (ANOVA) followed by Tukey's post-hoc tests.

Group that was air-abraded with 50 μm alumina particles at 2 bar pressure, subjected to sterilization, but not exposed to thermal cycling (group B2N) exhibited the highest median retention strength of 1390 N, whereas group that was air-abraded with 110 μm alumina particles at 1 bar pressure, not sterilized, but subjected to thermal aging for 150 days (group C1T) had the lowest strength of 688 N. No statistically significant interaction ($p>0.05$) was seen between the different variables in relation to their impact on the resulting retention.

However, the sterilization always showed a positive effect. Thermocycling had a substantial negative effect only when sterilisation was not performed ($p\leq 0.01$). Thermocycling did not have a statistically significant impact on the retention of the sterilized groups. For the sterilized groups, thermocycling had no statistically significant effect on the retention.

In conclusion, our findings indicate that steam autoclaving significantly enhanced the retention of hybrid zirconia abutments. The DTK adhesive resin demonstrated excellent performance during sterilisation and thermocycling when used with two-piece zirconia abutments.

8. Zusammenfassung

Ziel dieser Studie war es, die Auswirkungen der Sterilisation, der Konditionierungsmethoden und der thermischen Alterung auf die Retentionsstärke von zweiteiligen Zirkoniumdioxid-Abutments zu untersuchen.

Insgesamt 128 Abutments aus Zirkonoxidkeramik wurden in 4 Gruppen mit jeweils 32 Abutments eingeteilt. Die Gruppen wurden anhand der verwendeten Konditionierungsparameter kategorisiert: Abstrahlen mit (A) 50 µm Aluminiumoxidpartikeln bei 1,0 bar Druck, (B) 50 µm Partikel bei 2,0 bar Druck, (C) 100 µm Partikel bei 1,0 bar Druck und (D) 100 µm Partikel bei 2,0 bar Druck. Alle Abutments wurden mit DTK-Adhäsivsystem auf Titanbasen geklebt und für 72 Stunden im Wasserbad bei 37 °C gelagert.

Die Gruppen wurden in jeweils zwei Untergruppen (n=16) weiter aufgeteilt. Gruppe 1 wurde desinfiziert, während Gruppe 2 sowohl desinfiziert als auch im Autoklav sterilisiert wurde. Die Hälfte der Proben aus jeder Gruppe (n=8) wurde direkt dem axialen Retentionstest unterzogen (Gruppe N), während die andere Hälfte vor dem Retentionstest für 150 Tage lang thermozykliert wurde (Gruppe T).

Die Daten wurden anhand einer dreifachen Varianzanalyse (ANOVA) mit anschließendem Tukey-Post-hoc-Test statistisch ausgewertet.

Die Gruppe, die mit 50 µm Aluminiumoxidpartikeln bei einem Druck von 2 bar sandgestrahlt, sterilisiert, aber nicht thermischen Zyklen ausgesetzt wurde (Gruppe B2N), wies die höchste mittlere Retentionskraft von 1390 N auf, während die Gruppe, die mit 100 µm Aluminiumoxidpartikeln bei einem Druck von 1 bar sandgestrahlt, nicht sterilisiert, aber einer thermischen Alterung über 150 Tage ausgesetzt wurde (Gruppe C1T), wies die niedrigste Retentionskraft von 688 N auf. Es wurde keine statistisch signifikante Interaktion ($p \geq 0,05$) zwischen den verschiedenen Variablen in Bezug auf ihre Auswirkungen auf die resultierende Retentionskraft festgestellt.

Die Sterilisation zeigte jedoch immer eine positive Wirkung. Das Thermocycling hatte nur dann einen wesentlichen negativen Effekt, wenn keine Sterilisation durchgeführt wurde ($p \leq 0,01$). Bei den sterilisierten Gruppen hatte das Thermocycling keine statistisch signifikante Auswirkung auf die Retentionskraft.

Zusammenfassend zeigen die Ergebnisse, dass die Sterilisation die Retention von Hybrid-Zirkonoxid-Abutments deutlich verbessert hat. Der DTK-Adhäsivsystem zeigte bei der Sterilisation und beim Thermocycling eine hervorragende Leistung, wenn es mit zweiteiligen Zirkonoxid-Abutments verwendet wurde.

9. References

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11. Dedication

This doctorate thesis is dedicated to my beloved mother, father, sisters, and family and friends for their unconditional love and support.

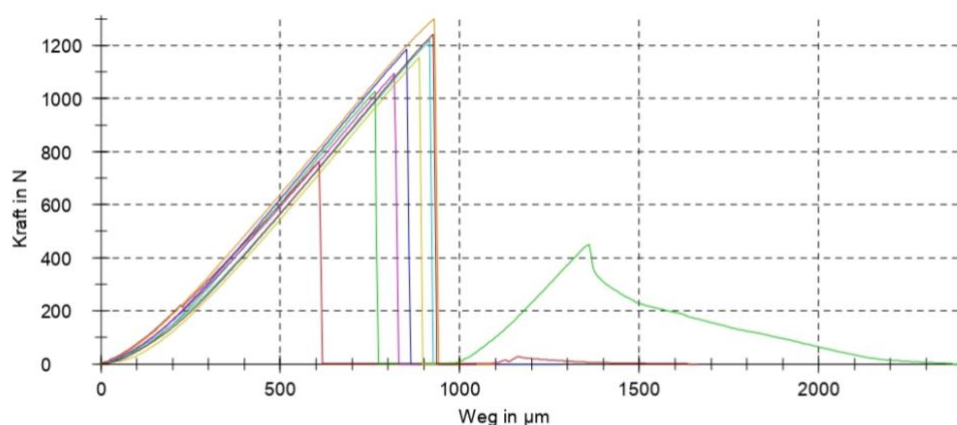
I would also like to dedicate my work to my lovely husband, Dr. Ahmed Bawahab, and my intelligent kids, who believed in me and inspired me through my long journey.

Finally, and most importantly, I thank my young sister, Dr. Nebras Alghanaim, for her support, encouragement, quiet, patience, and unwavering love, which were undeniably the bedrock on which I always relied. I could not have accomplished this without her support.

12. Appendices

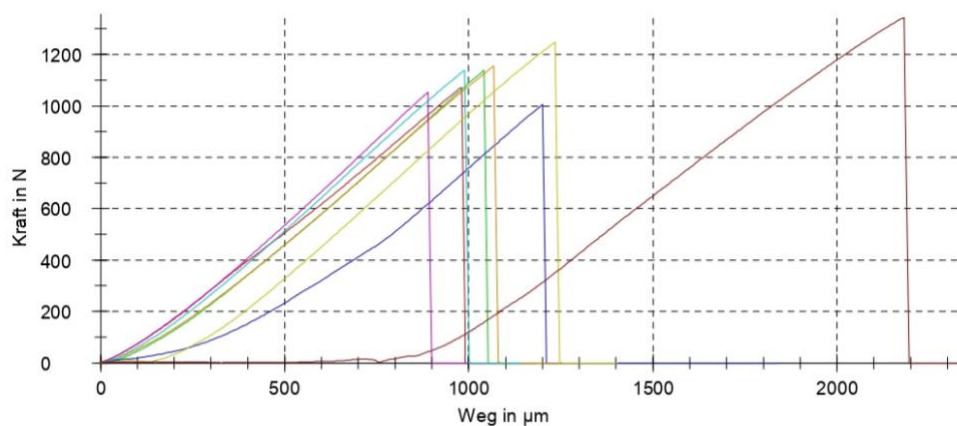
Retentive strength and failure mode of group (A1N)

Specimen Number	Specimen in (A1N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
1	1A1N	763	60%	40%	40%	60%	1123	± 168	1165
2	2A1N	1030	70%	30%	30%	70%			
3	3A1N	1180	50%	50%	50%	50%			
4	4A1N	1300	80%	20%	20%	80%			
5	5A1N	1100	70%	30%	30%	70%			
6	6A1N	1220	40%	60%	60%	40%			
7	7A1N	1150	70%	30%	30%	70%			
8	8A1N	1240	80%	20%	20%	80%			



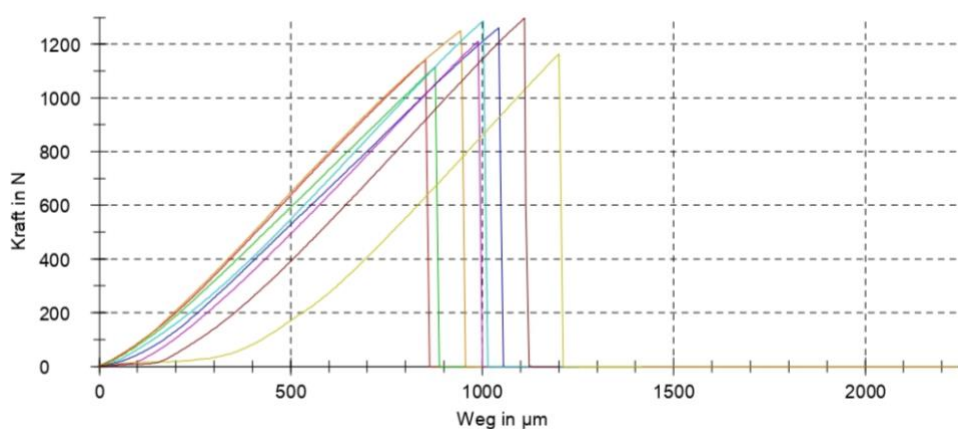
Retentive strength and failure mode of group (A2N)

Specimen Number	Specimen in (A2N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
9	1(A2N)	1070	50%	50%	50%	50%	1145	± 108	1140
10	2(A2N)	1140	40%	60%	60%	40%			
11	3(A2N)	1010	40%	60%	60%	40%			
12	4(A2N)	1160	50%	50%	50%	50%			
13	5(A2N)	1050	60%	40%	40%	60%			
14	6(A2N)	1140	30%	70%	70%	30%			
15	7(A2N)	1250	20%	80%	80%	20%			
16	8(A2N)	1340	70%	30%	30%	70%			



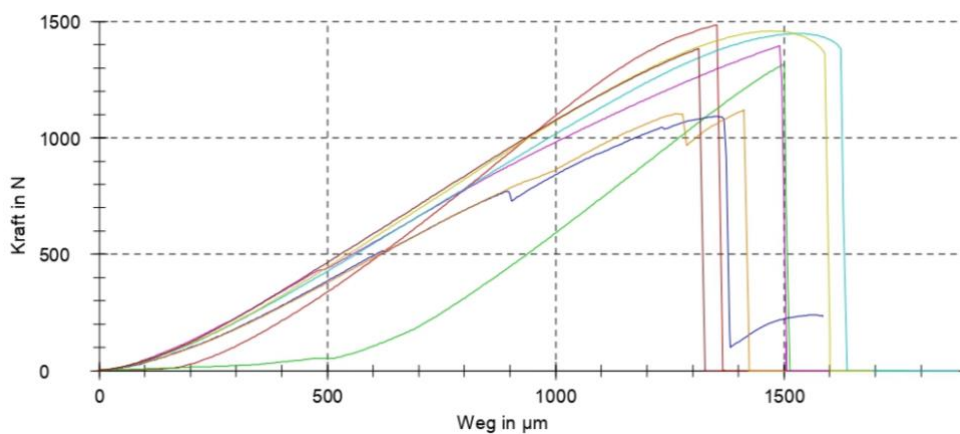
Retentive strength and failure mode of group (B1N)

Specimen Number	Specimen in (B1N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
17	1B1N	1140	70%	30%	30%	70%	1210	± 72	1215
18	2B1N	1110	80%	20%	20%	80%			
19	3B1N	1260	50%	50%	50%	50%			
20	4B1N	1250	60%	40%	40%	60%			
21	5B1N	1210	70%	30%	30%	70%			
22	6B1N	1290	80%	20%	20%	80%			
23	7B1N	1160	70%	30%	30%	70%			
24	8B1N	1300	70%	30%	30%	70%			



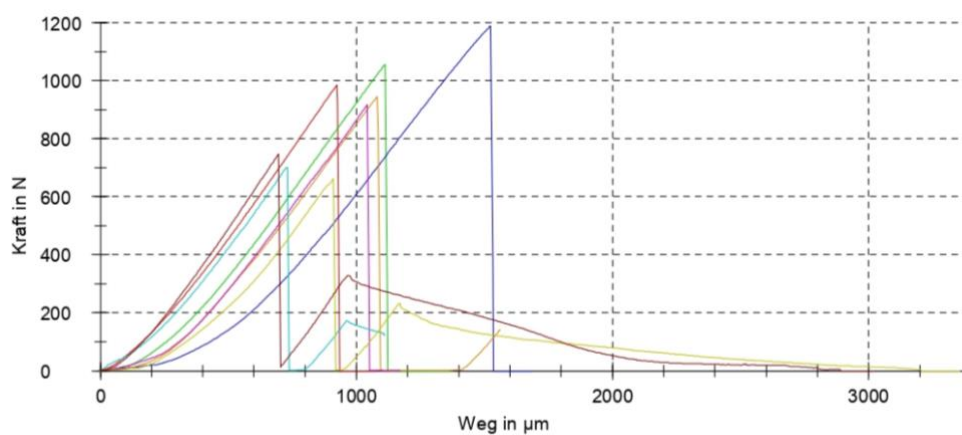
Retentive strength and failure mode of group (B2N)

Specimen Number	Specimen in (B2N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
25	1B2N	1490	50%	50%	50%	50%	1339	± 154	1390
26	2B2N	1320	50%	50%	50%	50%			
27	3B2N	1090	60%	40%	40%	60%			
28	4B2N	1120	40%	60%	60%	40%			
29	5B2N	1400	50%	50%	50%	50%			
30	6B2N	1450	screw fracture	screw fracture	screw fracture	screw fracture			
31	7B2N	1460	screw fracture	screw fracture	screw fracture	screw fracture			
32	8B2N	1380	50%	50%	50%	50%			



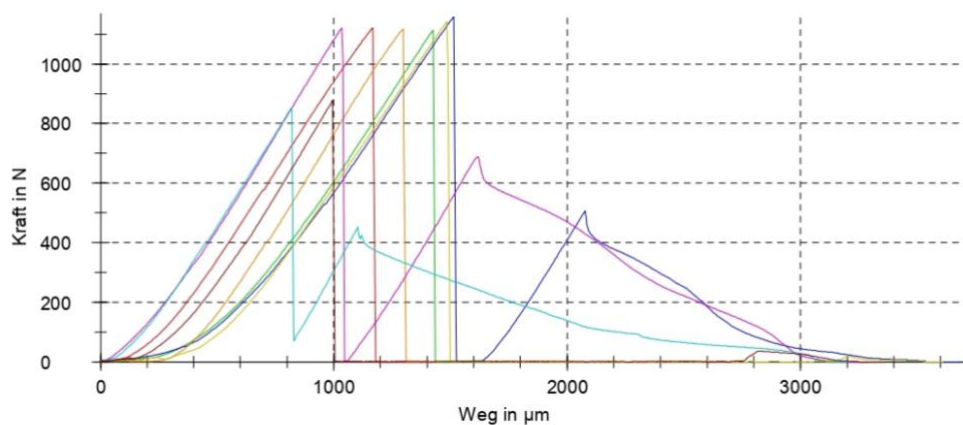
Retentive strength and failure mode of group (C1N)

Specimen Number	Specimen in (C1N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
33	1C1N	984	30%	70%	70%	30%	901	± 185	931
34	2C1N	1060	50%	50%	50%	50%			
35	3C1N	1190	60%	40%	40%	60%			
36	4C1N	945	30%	70%	70%	30%			
37	5C1N	917	20%	80%	80%	20%			
38	6C1N	702	20%	80%	80%	20%			
39	7C1N	662	15%	85%	85%	15%			
40	8C1N	746	10%	90%	90%	10%			



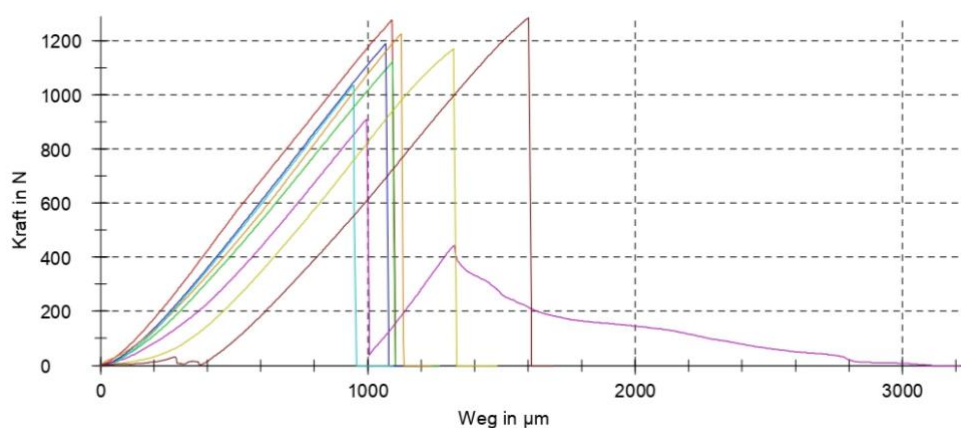
Retentive strength and failure mode of group (C2N)

Specimen Number	Specimen in (C2N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
41	1C2N	1120	10%	90%	90%	10%	1063	± 123	1120
42	2C2N	1110	30%	70%	70%	30%			
43	3C2N	1160	30%	70%	70%	30%			
44	4C2N	1120	25%	75%	75%	25%			
45	5C2N	1120	20%	80%	80%	20%			
46	6C2N	854	20%	80%	80%	20%			
47	7C2N	1140	30%	70%	70%	30%			
48	8C2N	877	10%	90%	90%	10%			



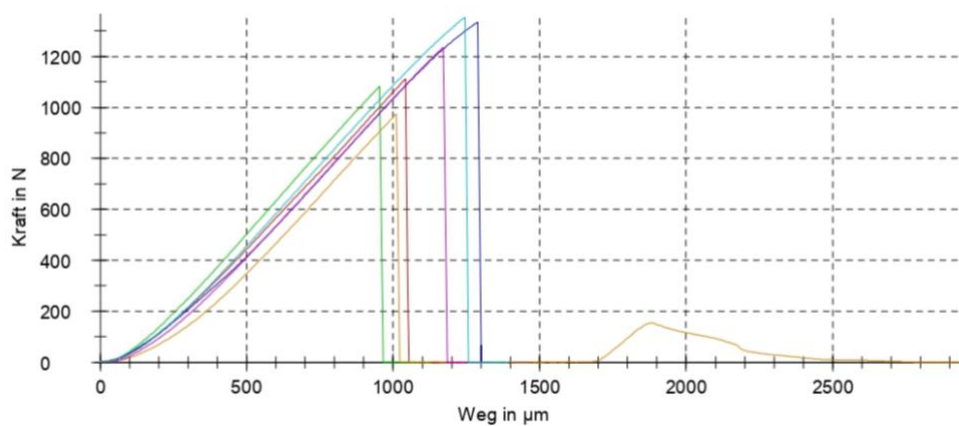
Retentive strength and failure mode of group (D1N)

Specimen Number	Specimen in (D1N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
49	1D1N	1280	60%	40%	40%	60%	1154	± 128	1180
50	2D1N	1120	70%	30%	30%	70%			
51	3D1N	1190	70%	30%	30%	70%			
52	4D1N	1230	50%	50%	50%	50%			
53	5D1N	910	50%	50%	50%	50%			
54	6D1N	1040	40%	60%	60%	40%			
55	7D1N	1170	70%	30%	30%	70%			
56	8D1N	1290	50%	50%	50%	50%			



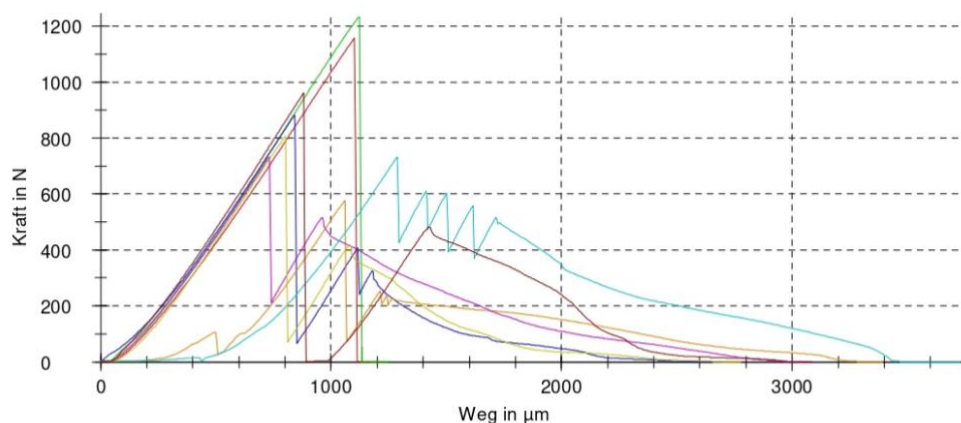
Retentive strength and failure mode of group (D2N)

Specimen Number	Specimen in (D2N)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
57	1D2N	909	50%	50%	50%	50%	1162	± 168	1175
58	2D2N	1110	25%	75%	75%	25%			
59	3D2N	1080	50%	50%	50%	50%			
60	4D2N	1340	40%	60%	60%	40%			
61	5D2N	973	50%	50%	50%	50%			
62	6D2N	1240	40%	60%	60%	40%			
63	7D2N	1350	50%	50%	50%	50%			
64	8D2N	1290	10%	90%	90%	10%			



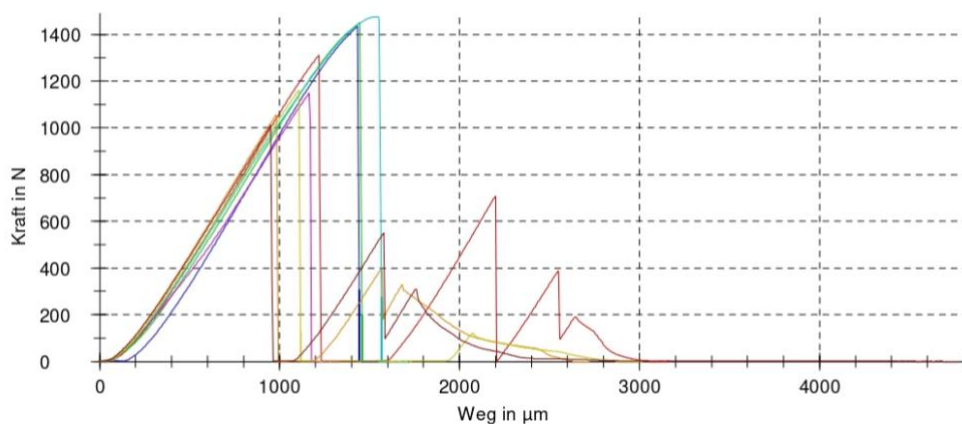
Retentive strength and failure mode of group (A1T)

Specimen Number	Specimen in (A1T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
65	1A1T	1160	80%	20%	20%	80%	929	± 200	882
66	2A1T	1230	85%	15%	15%	85%			
67	3A1T	882	70%	30%	30%	70%			
68	4A1T	577	30%	70%	70%	30%			
69	5A1T	732	50%	50%	50%	50%			
70	6A1T	733	80%	20%	20%	80%			
71	7A1T	807	75%	25%	25%	75%			
72	8A1T	961	80%	20%	20%	80%			



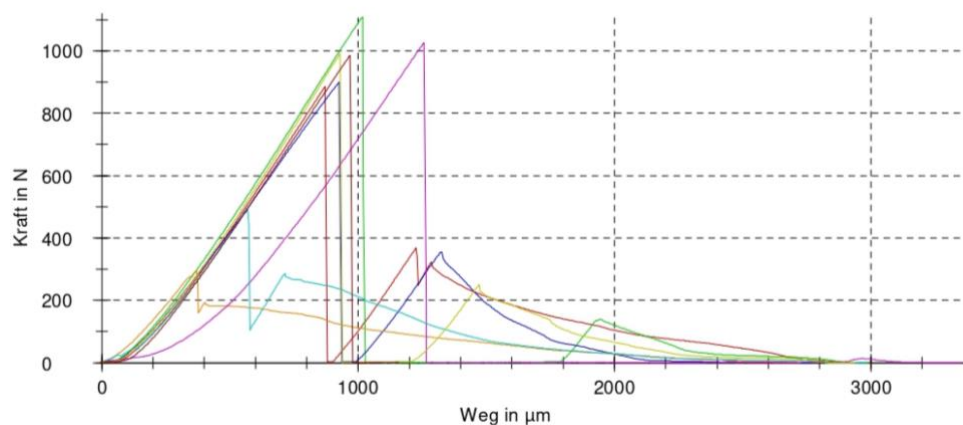
Retentive strength and failure mode of group (A2T)

Specimen Number	Specimen in (A2T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
73	1A2T	1310	70%	30%	30%	70%	1256	± 185	1235
74	2A2T	1450	50%	50%	50%	50%			
75	3A2T	1430	65%	35%	35%	65%			
76	4A2T	1060	70%	30%	30%	70%			
77	5A2T	1150	75%	25%	25%	75%			
78	6A2T	1480	60%	40%	40%	60%			
79	7A2T	1160	50%	50%	50%	50%			
80	8A2T	1010	70%	30%	30%	70%			



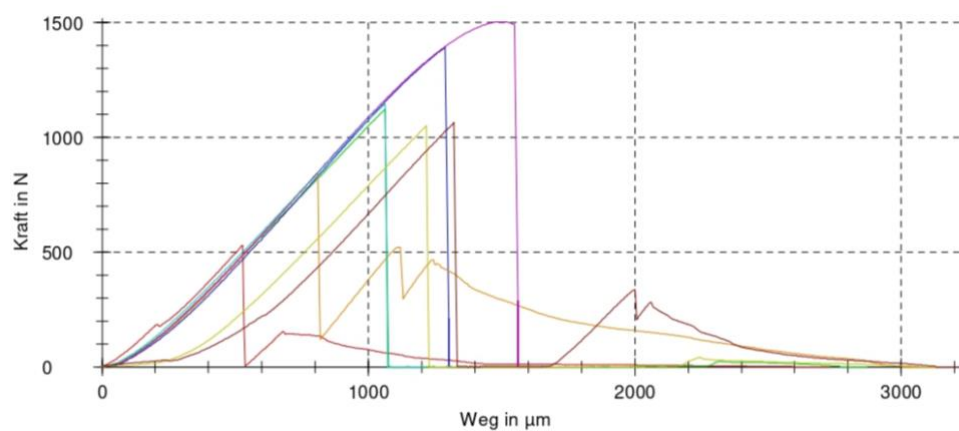
Retentive strength and failure mode of group (B1T)

Specimen Number	Specimen in (B1T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
81	1B1T	885	50%	50%	50%	50%	983	± 84	988
82	2B1T	1110	40%	60%	60%	40%			
83	3B1T	900	80%	20%	20%	80%			
84	4B1T	295	30%	70%	70%	30%			
85	5B1T	1030	65%	35%	35%	65%			
86	6B1T	492	65%	35%	35%	65%			
87	7B1T	990	60%	40%	40%	60%			
88	8B1T	985	70%	30%	30%	70%			



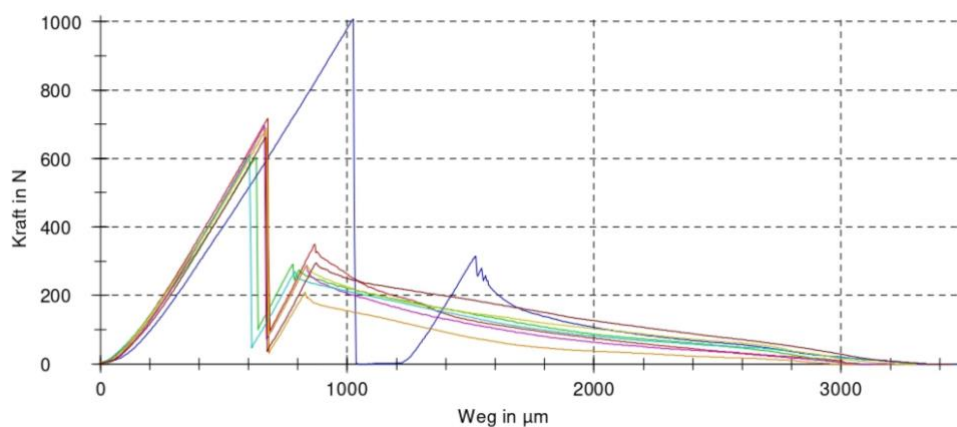
Retentive strength and failure mode of group (B2T)

Specimen Number	Specimen in (B2T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
89	1B2T	530	30%	70%	70%	30%	1158	± 221	1120
90	2B2T	1120	80%	20%	20%	80%			
91	3B2T	1390	50%	50%	50%	50%			
92	4B2T	842	70%	30%	30%	70%			
93	5B2T	1500	70%	30%	30%	70%			
94	6B2T	1140	75%	25%	25%	75%			
95	7B2T	1050	65%	35%	35%	65%			
96	8B2T	1066	50%	50%	50%	50%			



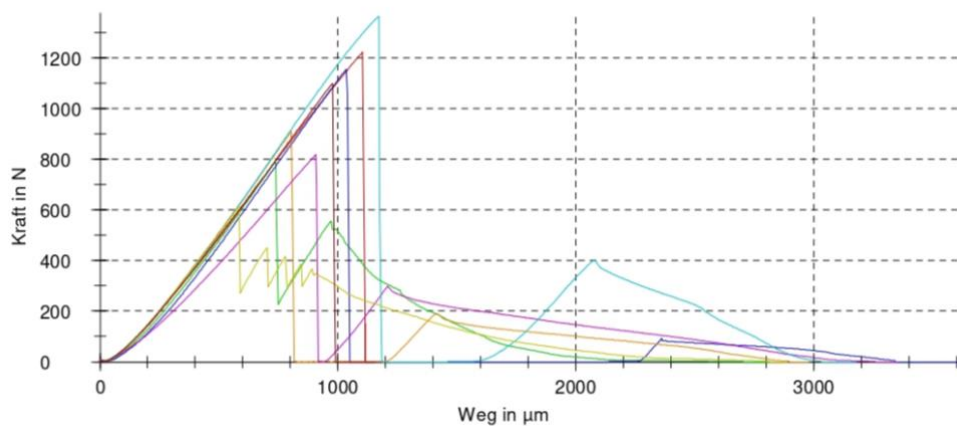
Retentive strength and failure mode of group (C1T)

Specimen Number	Specimen in (C1T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
97	1C1T	717	25%	75%	75%	25%	709	± 128	688
98	2C1T	604	5%	95%	95%	5%			
99	3C1T	1010	10%	90%	90%	10%			
100	4C1T	689	15%	85%	85%	15%			
101	5C1T	696	10%	90%	90%	10%			
102	6C1T	610	25%	75%	75%	25%			
103	7C1T	686	15%	85%	85%	15%			
104	8C1T	661	5%	95%	95%	5%			



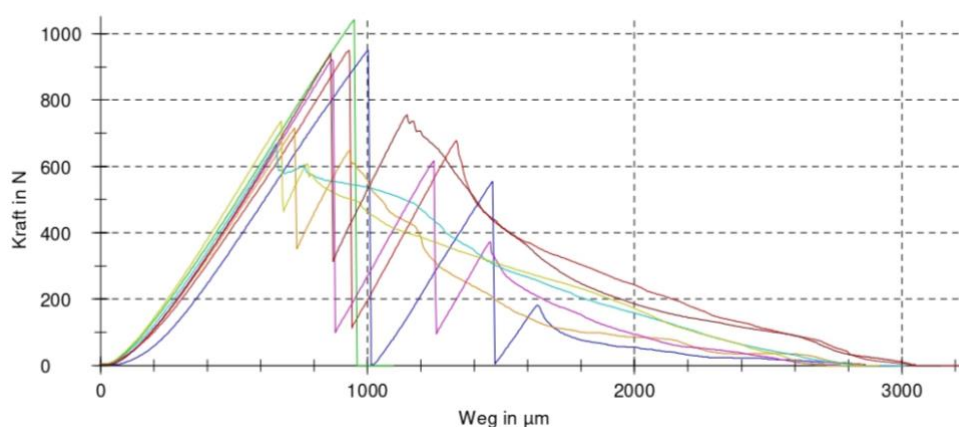
Retentive strength and failure mode of group (C2T)

Specimen Number	Specimen in (C2T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
105	1C2T	1220	70%	30%	30%	70%	996	± 254	1005
106	2C2T	786	75%	25%	25%	75%			
107	3C2T	1160	20%	80%	20%	80%			
108	4C2T	909	30%	70%	70%	30%			
109	5C2T	818	20%	80%	80%	20%			
110	6C2T	1360	65%	35%	35%	65%			
111	7C2T	611	35%	65%	65%	35%			
112	8C2T	1100	65%	35%	35%	65%			



Retentive strength and failure mode of group (D1T)

Specimen Number	Specimen in (D1T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
113	1D1T	950	75%	25%	25%	75%	866	± 138	933
114	2D1T	1040	30%	70%	70%	30%			
115	3D1T	951	35%	65%	65%	35%			
116	4D1T	715	70%	30%	30%	70%			
117	5D1T	923	75%	25%	25%	75%			
118	6D1T	667	40%	60%	60%	40%			
119	7D1T	737	50%	50%	50%	50%			
120	8D1T	942	40%	60%	60%	40%			



Retentive strength and failure mode of group (D2T)

Specimen Number	Specimen in (D2T)	Retentive Strength (N)	(Ti) Adhesive failure	(Ti) Cohesive failure	(ZrO ₂) Adhesive failure	(ZrO ₂) Cohesive failure	Mean	SD	Median
121	1D2T	1150	80%	20%	20%	80%	1136	± 211	1070
122	2D2T	1210	70%	30%	30%	70%			
123	3D2T	989	40%	60%	60%	40%			
124	4D2T	1390	85%	15%	15%	85%			
125	5D2T	985	30%	70%	70%	30%			
126	6D2T	907	30%	70%	70%	30%			
127	7D2T	973	85%	15%	15%	85%			
128	8D2T	1480	screw fracture	screw fracture	screw fracture	screw fracture			

