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## **TESI DI DOTTORATO**

## TITOLO

Innovative Dental Technologies and Restorative Materials in Prosthodontics: New Perspectives and Future Developments

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#### Chapter 1

#### **1.1 Introduction**

Dentistry has had an increased interest in new materials and new technologies that goes back decades. Soon after the discovery of anesthetics, the dental drill was invented, which meant that filling materials such as silicates and amalgams became widely used. In the early 20th century Dr. William H. Taggart introduced the loss-wax casting process in dentistry for the construction of crowns and bridges, which was adapted from the method till then used in the jewellery business. The developments in new polymers during the 1940s and 1950s resulted in the use of acrylic resins for dentures, acidic polymers for restorative cements and monomers for composite resin restorative materials. The lasting contributions of Michael Buonocore, Dennis Smith, Raphael Bowen, John McLean, Alan Wilson and many others in this respect are well known. The discovery by Branemark of the special properties of titanium metal did not take long to be translated into an explosion in dental implantology. Thus dentistry has shown itself to lead the medical disciplines in embracing new materials and new technologies and it has also proved in making use of new technologies such as CAD/CAM (Computer Aided Design/Computer Aided Manufacturing).

# 1.2 Occlusal Bite On Implant-Supported Restorations: a Digital Dentistry Approach with Digital Workflow and CAD-CAM Fabrication Leone R, Sorrentino R, De Stefano L, Zarone F. European Journal of Oral Implantology, 2017 (in press).

**Objectives -** The present case report aimed at describing the designing, digital workflow and CAD-CAM fabrication of an occlusal bite on implant-supported prosthesis.

**Materials and methods** - A 54-year old dysfunctional female patient presented with improper implant-supported restorations in the posterior regions of both arches. Such restorations were unsatisfactory for both function and esthetics. The patient complained about the impossibility to achieve correct occlusion, mastication and phonetics. The objective clinical examination pointed out a severe alteration of the oral functions; moreover, the oral discomfort had caused serious psychological and behavioral problems to the patient. Temporo-mandibular joint (TMJ) disorders were evidenced; consequently, it was decided to fabricate an occlusal bite plane to decondition masticatory muscles and recover a proper position of the mandible, so as to restore correct oral functions. Conventional impressions and intermaxillary relationships were recorded and the master casts were scanned to import all information in a virtual environment. Moreover, a digital facebow was used to set a digital articulator.

The static and dynamic occlusal contacts were studied and balanced on the

virtual casts and the occlusal bite was entirely designed in the digital environment. Then, CAD-CAM procedures were used to fabricate the bite, so as to take advantage of the optimal mechanical properties and wear resistance of CAD-CAM resin materials. Similarly, long-term poly-methyl-meta-acrylate (PMMA) interim restorations were produced to finalize the implant-supported rehabilitation.

**Results** - The CAD-CAM occlusal bite allowed the patient to recover correct oral functions in about 2 months; the TMJ disorder symptomatology disappeared and proper mandibular posture was restored. Due to the excellent mechanical resistance of the CAD-CAM materials, despite the initial severe parafunctions of the patient, no worn areas were evidenced on the surfaces of the bite.

Once the occlusion was considered stable, long-term implant-supported interim restorations made up of PMMA were luted onto implants, in order to allow for occlusal adjustments over time due to the function-related adaptability of the stomatognathic system.

**Conclusions** - The digital workflow made the registration and transfer of occlusal dynamics easier. Moreover, the designing of the occlusal bite in the virtual environment resulted in less time-consuming procedures. The CAD-CAM materials provided the occlusal bite with excellent mechanical properties and wear resistance.



Fig. 1. Digital Workflow



# Fig. 2. CAD/CAM resin Bite

#### Chapter 2

## 2.1 The coronal restoration of endodontically treated teeth

The coronal restoration of an endodontically treated tooth is a challenge for any dental practitioner. The traditional way to restore severely damaged endodontically treated teeth is to place a cast post- and-core restoration and a subsequent crown. An alternative method using prefabricated metal posts and composite resin as a core material was introduced around the 1970s.

Along with the less time-consuming procedure, the main advantage of a prefabricated post compared to the cast post and core is that undercuts of the pulp chamber can be maintained, thus preserving tooth material. Today, the use of a post is questioned even with the use of adhesive buildup materials in the reconstruction of endodontically treated teeth. Omitting a post is the optimal way to preserve tooth material; neverthless, the use of fiber posts was proved to prevent catastrophic root fractures. For crowned teeth, it was demonstrated that post placement did not increase the longevity of the teeth but the issue is still controversial.

2.2 Complications of endodontically treated teeth restored with fiber posts and different prosthetic systems: a systematic review.

Sorrentino R., Di Mauro M., Ferrari M., Leone R., Zarone F. Clinical Oral Investigations, 2016.

#### Introduction

One of the main functions of fiber posts is to ensure the retention of restoration after the loss of a large amount of dental structure, in order to secure the filling material to the tooth and build up a prosthetic core. It is well recognized that the presence of a post and core has the function to improve the retention of a restoration but does not improve at all the strength of dental roots [1, 2].

In the past decades, cast or prefabricated metal posts made up of materials with high moduli of elasticity (E) were used, just like gold alloys, stainless steel, or titanium since they were considered strong and clinically effective [3]. Nowadays, fiber posts are the most clinically used; they were introduced about 20 years ago, made up of materials with lower moduli of elasticity, such as glass, quartz, polyethylene, and carbon-reinforced composites [4].

Several studies reported that root fractures occur more frequently with metal than fiber posts [2–4], the main reason being the difference between the modulus of elasticity of metal (220 GPa) and dentin (42 GPa). On the contrary,

the modulus of elasticity of fiber posts (25–57 GPa) is closer to that of dentine [5–8] and this is considered a favorable property from the biomechanical point of view [6–8]. Recently, a systematic review with meta-analysis reported that the incidence of fractures was equal using either fiber or metal posts [5], so the topic remains controversial.

As to fiber posts, the most frequent complication is debonding; it is influenced by many factors, such as amount of residual tooth tissues, occlusal scheme and number of opposing teeth in function, periodontal status, signs of parafunctions, presence or absence of ferrule, and quality of adhesion [1, 8, 9].

Moreover, the characteristics of fiber posts as well as the adopted clinical procedures thoroughly influence the performances of the restorations, just like length and diameter of posts, integrity of the adhesive surface [10], and thickness of adhesive cements [11]. The presence of full crowns reduces the influence of the diameter of prefabricated posts on the occurrence of root fractures with either metal or fiber posts [12].

According to the literature, the prognosis of fiber posts is better in molars than in the anterior teeth. A high incidence of vertical fractures was noticed in the maxillary premolars, probably related to the small mesiodistal canal diameter, which could increase stress within the root. A higher amount of coronal structure and the presence of interproximal contacts improve the survival rates of post restorations [13].

The survival rates of fiber post restorations also depend on the type of the final prosthesis. Endodontically treated teeth restored with single crowns (SCs) were reported to show higher survival rates, probably for a shielding effect of crowns, while in the presence of fixed dental prostheses (FDPs) or removable partial dentures (RPDs) the failure rates were reported to be higher, probably due to unfavorable bending moments of the prosthetic systems [12].

Several investigations pointed out that crown coverage could positively affect the outcome of post-endodontic restorations with fiber posts. The survival rates of endodontically treated teeth restored with fiber posts and crowns was six times higher than those of teeth restored without crowns [14], achieving a survival rate of 85.1 % after 10 years of function [15]. The placement of a crown seemed to be more significative than the type of abutment buildup for the survival of the teeth [14].

The present systematic review aimed at investigating the relationships between the presence of fiber posts in abutment teeth and the occurrence of endodontic and prosthetic complications in the presence of SCs and FDPs, in patients with periodontal status *ad integrum*.

#### Search methods

The primary objective of the present systematic review was to analyze the incidence of clinical complications in SCs and FDPs using abutment teeth restored with fiber posts.

The secondary objective was to compare differences in failure rates between subgroups regarding the following variables:

- Presence of ferrule
- Type (incisor, canine, premolar, molar) and location (maxilla or mandible) of the teeth

The systematic review was based on a literature review of papers published between 1990 and 2015 and available in electronic databases (Pubmed, Evidence-Based Dentistry, BMJ Clinical Evidence, Embase, and Dynamed), since the earliest fiber-reinforced composite posts were introduced in the USA in the1990s; only articles written in English were considered, since it is considered to be the universal language of science. Gray literature was analyzed as well on the website www.opengrey.eu.

The strategy of the search included the use of different keywords and boolean operators, as follows:

- 1. Fiber post/post
- 2. Fiber post and/or single crown

- 3. Fiber post and/or fixed partial denture
- 4. Fiber post and/or fixed dental prosthesis
- 5. Fiber post and/or prosthetic restoration
- 6. Fiber post and/or prosthesis
- 7. Fiber post and/or complication

A manual search was performed as well, looking for eligible papers and reference lists of articles. Researchers and authors of non-published studies or of published studies that were not available electronically were contacted by the reviewers. Data extraction was carried out independently by two experienced reviewers; any disagreement was resolved by discussion with a third experienced reviewer.

#### **Inclusion criteria**

This systematic review was structured on the basis of the PRISMA guidelines.

The eligibility of investigations was assessed according to the P.I.C.O. as follows:

- 1. Participants
  - Patients with periodontal status *ad integrum*, endodontically treated to a sound state permanent teeth (absence of periodontal disease) and restored with SCs or FDPs

- 2. Interventions
  - Randomized clinical trials (RCTs) evaluating fiber posts and prosthetic complications over a minimum observational period of 36 months
  - RCTs evaluating failure rates of posts and/or final restorations in a single group but considering different types of teeth
  - RCTs reporting clearly defined inclusion and exclusion criteria and description of clinical procedures.
- 3. Comparison
  - Studies comparing failure rates of fiber posts and other prosthetic systems (i.e., direct composite)
- 4. Outcomes
  - Failure rates of fiber posts and prosthetic restorations in each group
  - Comparison of failure rates between different groups

All the studies not fulfilling the inclusion criteria were not included in the systematic review.

#### **QUALITY ASSESSMENT**

The quality of the included studies was evaluated using the criteria reported by the Cochrane Handbook for Systematic Reviews of Interventions [16]. Consequently, the quality assessment was carried out using the following criteria:

- Was there a randomization of the participants?
  - 0: Not randomized
  - 1: Inadequate
  - 2: Unclear
  - 3: Adequate
- Was a calculation of sample size undertaken?
  - 0: No/not mentioned
  - 1: Yes
- Were inclusion/exclusion criteria clearly defined?
  - 0: Not defined
  - 1: Poorly defined
  - 2: Well defined
- Was follow-up achieved?
  - 0: No/not mentioned
  - 1: Yes (<80 %)
  - 2: Yes (≥80 %)
- Was treatment blind to patients, operators, or assessors recorded?
  - 0: No/not possible
  - 1: Unclear

- 2: Yes

- Were the outcomes of the people who withdrew described by study group and included in the analysis (intention-to-treat or ITT analysis)?
  - 0: Not mentioned
  - 1: States numbers and reasons for withdrawal by study group but no analysis
  - 2: primary analysis based on all recruited cases
- Were the control and treatment groups comparable at entry?
  - 0: Large potential for confounding or not discussed
  - 1: Confounding small–mentioned but not adjusted for
  - 2: Unconfounded-good comparability of groups or confounding adjusted for

The risk of bias was assessed according to the Cochrane Handbook for Systematic Reviews of Interventions considering quality criteria, allocation concealment, blinding of outcome assessor, and follow-up [16].

#### Search results

Database search produced 4230 records, many of which were duplicates. Manual research did not produce any other relevant article. After duplications were removed, all the selected databases produced 3670 records. Analyzing titles, abstracts, and keywords, the reviewers excluded 3664 papers that did not meet the inclusion criteria; the main reasons for exclusion were as follows: not the topic of interest, in vitro studies, and studies without control. Four studies were not available electronically, thus the authors were contacted by email and the response rate was 100%. The full-texts of the remaining six articles were read, and the reviewers excluded two papers, as they focused on endodontic treatment and complications. The reasons for the exclusion of these articles are reported in Table 1.

Study	Reason for exclusion		
Mancebo et al. [17]	Excluded because the study focuses on endodontic treatments but does not report prosthetic failures and complications.		
Juloski et al. [18]	Excluded because the study focuses on endodontic treatments and materials but prosthetic complications were not reported.		

 Table 1
 Excluded studies and reasons for exclusion

The two independent reviewers' agreement rate was 97%. Any disagreement was discussed with a third reviewer. The workflow of the paper screening process is reported in Fig. 1.



On the basis of the reported inclusion criteria, only four studies were included in the present systematic review [19–22].

### Results

Only the following four studies met the inclusion criteria and were systematically reviewed: Mannocci et al. [19], Schmitter et al. [20], Ferrari et al. [21], and Sterzenbach et al. [22].

In a RCT, Mannocci et al. [19] compared the survival rates of endodontically treated premolars restored with fiber posts (Composipost, RTD, St Egreve, France), full cast crowns or composite fillings. In the study, 117 patients were recruited and randomly divided into two groups by tossing a coin: 60 teeth were restored with direct composite restorations and 57 with porcelain-fused-to-metal (PFM) SCs. All the restorations were made by the same operator. All the details regarding root canal treatments, crowns preparations, and criteria of success/failure were reported in the study. At the baseline, the patients were healthy and received oral hygiene instructions.

In a RCT, Schmitter et al. [20] investigated the 5-year results of two post systems: titanium screw posts (BKS, Brasseler, GA, USA) and glass fiber posts (ER dentine post, Brasseler). In this study, 100 patients requiring a SC, a FDP, or a RPD were recruited. At the baseline, all the patients did not show signs of periodontal disease. Forty-two patients were treated with metal screw posts and 39 patients with fiber posts. Post assignment was randomized and all the posts were placed by undergraduate students 6 months to 1 year prior to graduation. The metal screw posts were cemented with a zinc phosphate cement (Harvard cement, Harvard dental, Hoppegarten, Germany), whereas the fiber posts were luted using a composite resin cement (Variolink II, Ivoclar Vivadent, Schaan, Liechtenstein). All the details regarding the clinical procedures were described. The patients were recalled after 1 year and 5 years. Both the patients and the

dentists were blinded about the type of post used after endodontic treatment. The 5-year recall was performed by another blind dentist who had not been involved before in the study.

In a RCT, Ferrari et al. [21] evaluated the 6-year survival of endodontically treated premolars restored with fiber posts and PFM SCs. In this study, a sample of 345 patients was recruited and 360 premolars were divided into six groups on the basis of coronal residual dentine (4 to 1 residual coronal walls, presence of a minimum of 2 mm of ferrule, absence of ferrule). Each group was randomly divided into three subgroups on the basis of the restorative procedures as follows: no posts, prefabricated fiber posts (DT Light posts, RTD), and customized fiber posts (EverStick fibers, Stick Tech Ltd., Turku, Finland). The DT posts were luted using Calibra (Dentsplay Ltd., Kostanz, Germany), whereas the EverStick posts were luted using BisCore (Bisco, Schaumburg, IL, USA). All the details about the clinical procedures were reported in the study. At the baseline, all the patients did not present periodontal disease. All the selected teeth were in occlusal function with a natural tooth and in interproximal contact with two natural teeth. The clinical procedures were performed by the same experienced operator. Two examiners evaluated independently success and failure rates.

In a RCT, Sterzenbach at al. [22] evaluated the 7-year outcomes of endodontically treated teeth restored with glass fiber or titanium posts

(Fiberpoints Roots Pins Glass and Fiberpoints Root Pins Titanium, Schutz Dental Group, Rosbach, Germany). A sample of 91 patients was included in the study and randomly assigned to titanium (n = 45) or glass fiber post (n = 45) groups. Either the posts or PFM SCs were luted using a self-adhesive resin cement (RelyX Unicem, 3M ESPE, Seefeld, Germany). The clinical procedures were made by undergraduate students, while the 7-year follow-up was performed by an experienced blinded dentist. At the baseline, no signs of periodontal disease were observed. The incisors, canines, premolars, and molars were included in the study. The final prosthetic restorations were SCs, FDPs, SC-supported RPDs, and FPD-supported RPDs.

#### **Methodological quality**

The methodological quality assessment used in the present systematic review was reported in Table  $\underline{2}$ . Two independent reviewers evaluated the adequacy of records using a specific quality assessment protocol [16]. Any disagreement was discussed with a third experienced reviewer.

Sterzenbach et al. [22]	Ferrari et al. [21]	Schmitter et al. [20]	Mannocci et al. [19]	
L	L	L	L	Randomization
Н	Н	Н	Н	Calculation of sample size
L	L	L	L	Inclusion/ exclusion criteria
L	L	L	L	Follow-up achieved
L	Н	L	Н	Blind treatment
L	Н	U	Н	Withdrawing
L	L	L	L	Groups comparability

 Table 2
 Quality assessment of the studies included in the review

L low risk of bias, U unclear, H high risk of bias

Mannocci et al. [19] got an adequate randomization level by tossing a coin; the inclusion and exclusion criteria were properly defined and the final follow-up interested more than 80% of study subjects; all the restorations were made by a single experienced operator and a good comparability between groups was obtained.

In the study by Schmitter et al. [20], the randomization of the participants was adequate; computerized randomization was made by a professional nurse. The calculation of the sample size was not mentioned but the inclusion and exclusion criteria were properly defined: type of interventions, type of outcomes, and type of participants were specified. The 5-year follow-up was achieved for more than 80 % of the study sample. All the patients and the operators were blinded about the type of post used. The drop outs were adequately reported and there was a good comparability between groups.

In the study by Ferrari et al. [21], there was an adequate randomization of the participants. The inclusion and exclusion criteria were well defined and the follow-up regarded more than 80 % of the patients. The adopted clinical protocols did not allow blinding of the operators; intention-to-treat (ITT) correction was not applied and the sample size was not calculated. Nonetheless, a good comparability between groups was obtained.

In the study by Sterzenbach et al. [22], a satisfactory level of randomization of the participants was achieved by means of a computer-generated random list. The inclusion and exclusion criteria were clearly defined. No sample size calculation was performed and 80 % of the study population completed the follow-up period. Withdrawing was adequately described and included in the analysis, and there was a good comparability between the groups.

#### **Comparison of results**

#### **COMPARISON BETWEEN FIBER POSTS AND PROSTHESIS FAILURE EVENTS**

In the study by Mannocci et al. [19], no failures were observed at the 1-year recall. Conversely, after 2 and 3 years of function in group 1 (only direct composite restorations), one loss of retention of a fiber post and three marginal

gap openings were found; in group 2 (composite buildup + SC), two decementation of fiber posts and one marginal gap opening were observed. The failure rate was 6 % and no statistically significant differences were found between groups.

In the study by Schmitter et al. [20], in the fiber post group, 11 failures were observed: 2 losses of retention of fiber posts, 2 chipped or fractured SCs, 1 apical alteration and 6 teeth in need to be extracted due to post-core-crown complex loosening; thus, the survival rate of the teeth treated using fiber posts was 71.8 %. In the metal screw post group, 21 failures were observed: 1 post and 1 crown needed recementation, 1 tooth needed a new post, 17 teeth were extracted due to root fractures, 1 apical alteration, and 1 crown failure occurred; consequently, the survival rate of the teeth restored with metal posts was 50 %. Cox regression was performed in order to evaluate the influence of the analyzed variables, and it revealed that anterior teeth, the teeth with a significant loss of coronal structure and the teeth restored with metal screw posts, showed higher risk of failure.

In the study by Ferrari et al. [21], after 6 years, the overall survival rate was 94.1 %. In the group without posts, the largest number of root fractures and crown dislodgements was observed. In the group with prefabricated posts, no crown dislodgement was observed but 12 post decementations and 1 root fracture were noticed. The teeth with four coronal residual walls were failure

free. In this study, root canal retention was a significant factor for survival of the teeth, as assessed by Cox regression. The interaction between the type of restoration and the residual dentin was not statistically significant.

In the study by Sterzenbach et al. [22], over 7 years of observation, the overall survival rate was 89 %. In the titanium post group, the two maxillary lateral incisors and one mandibular molar showed endodontic failures. In the glass fiber post group, two root fractures, one tooth mobility (score 3), and one core fracture were observed.

There were no studies reporting the incidence of complications of endodontically treated teeth restored with fiber posts and FDPs.

#### **COMPARISON BETWEEN FAILURE RATES OF SUBGROUPS**

The secondary objective of the present systematic review was not fulfilled because there were no studies reporting differences in failure rates between subgroups (i.e., fiber posts and SC or fiber posts and FDP) in relation to ferrule height, type, and location of the teeth.

#### Discussion

Intraradicular posts were introduced in clinical practice to ensure the retention of restorations to the teeth missing a significative amount of their structure [23].

Several studies demonstrated that fiber posts performed better than metal posts due to their lower modulus of elasticity (E) compared to metal posts and similar to that of dentine (42 GPa) [24], with a lower incidence of root fractures in the long-term [25]. However, in some cases, such a modulus of elasticity was associated with excessive stress and strains, causing marginal gap opening and post debonding, which were reported to be the most frequent failures [10].

Different factors can influence the survival rates of post systems, just like type of post, luting cement, tooth position, shape of root canal, and final prosthetic restoration. In particular, the luting system of fiber posts significantly affected their clinical performances; self-adhesive resin cements proved to be most effective in the long-term in vitro, probably because less sensitive to the skill of the operator [26]. Conversely, several clinical studies suggested that the bond strength of self-adhesive cements is lower than the bond strength generated by traditional adhesive cementation techniques [27]. Moreover, it has been pointed out that resin cements could achieve poor adhesion to prefabricated fiber posts just like FRC posts due to the presence of a cross-linked polymer matrix between their fibers; in such cases, adhesive failures could be reduced by means of the interpenetrating polymer network (IPN) mechanism, as suggested by La Bell et al. [28].

According to the selected inclusion and exclusion criteria, four RCTs were included in this systematic review and the results were reported in the Table 3.

Sterzenbach et al. [22]	Ferrari et al. [21]	Schmitter et al. [20]	Mannocci et al. [19]	Study	
71.2 months	72 months	61.37 months	36 months	Mean observational period	
91	360	100	117	No. of included teeth	
0-2 walls; 2 mm-ferrule	1-4 walls; absence/ presence ferrule	At least 60 % of coronal structure	Class II premolars	Amount of residual coronal structure	
Fiberpoints Root Pins Glass, Schutz Dental Group, Rosbach, Germany [Glass fiber posts]	DT Light Posts, RTD, St. Egreve, France [Quartz fiber posts]; EverStick Fibers, Stick Tech Ltd., Turku, Finland [Glass fiber posts]	ER dentine post, Brasseler, GA, USA [Glass fiber posts]	Composipost, RTD, Sr Egreve, France [Carbon fiber posts]	Fiber post type, brand name and manufacturer	
PFM SCs, FDPs, SC-supported RDPs, FDPs-RPDs	PFM	SCs, FDPs, SC-supported RPDs	Direct composite, PFM	Type of restoration	
Incisors, canines, premolars, and molars	Premolars	Incisors, canines, premolars, and molars	Premolars	Tooth type	
9.8 %	0 % (4 walls); 21.1 % (3 walls); 30.2 % (2 walls); 47 % (1 wall); 66 % (ferrule present); 77.2 % (ferrule absent)	28.2 %	6 %	Failure rate	

 Table 3
 Characteristics of the studies included in the review

The shortest mean observational period (36 months) was reported in the study by Mannocci et al. [19]; differently, in the RCT by Ferrari et al. [21], 360 teeth were analyzed over a period of 72 months, which was the longest among the included studies. The lowest number of teeth (n = 91) was analyzed by Sterzenbach et al. [22]. Mannocci et al. [19] and Ferrari et al. [21] evaluated only the premolars, whereas Sterzenbach et al. [22] and Schmitter et al. [20] assessed both the anterior and posterior teeth.

As to the length of posts, different approaches were used in the studies included in the present review. In the study by Mannocci et al. [19], the fiber post length was 7 mm; Schmitter et al. [20] extended fiber posts to at least 50 % of the length of the root canal; Ferrari et al. [21] and Sterzenbach et al. [22] left an apical seal of at least 4 mm of root canal filling. The results of the included studies pointed out an adequate resistance of fiber posts placed with intermediate length, which is in agreement with previous investigations [29, 30]. The most frequently reported failures were fiber post debonding and crown dislodgements; only Schmitter et al. [20] described crack or chipping of the restorations and post-core-crown complex loosening.

Both the highest and the lowest failure rates were reported by Ferrari et al. [21]: 0 % in the group of 60 premolars with 4 residual coronal walls and 77.2 % in case of ferrule absence, respectively. The same authors evidenced that, in the teeth without residual dentine walls, there were no significant differences in the failure rates with or without ferrule. The preservation of at least one coronal wall significantly reduced the risk of failure, which is also pointed out by current published literature [31]; however, the relationship between residual coronal structure and type of prosthetic complication were not statistically significant.

Mannocci et al. [19] reported no statistically significant differences in failure rates between the teeth restored with direct composite restorations and metal-ceramic SCs. However, several studies in the literature suggested that SCs would be desirable to improve the survival rates of the restorations involving endodontically treated teeth restored with fiber posts, reducing the risk of fracture [12].

In the study by Schmitter et al. [20], the anterior teeth with a significant destruction of coronal structure and the teeth restored with metal screw posts showed higher risks of failure and these results were in agreement with previous

investigations [13]. However, in this study, the quality assessment reported a high risk of bias: neither calculation of the sample size nor intention-to-treat analysis was performed.

In the studies by Schmitter et al. [20] and Sterzenbach et al. [22], the number of opposing teeth in occlusion, type of antagonist, and presence of malocclusions at the baseline were not specified. Operator blinding was not possible in the studies by Mannocci et al. [19] and Ferrari et al. [21], since the clinical protocols were different in relation to the type of post. In the research by Sterzenbach et al. [22], the clinical procedures were performed by operators with different experience and this could have affected the final results. Moreover, in the study by Sterzenbach et al. [22], it was not clear which type of prosthetic restoration was referred to a specific failure event.

Only in the study by Sterzenbach et al. [22], FDPs were used as final restorations but the failure rates of fiber posts were reported without taking into account the differences between SCs, FDPs, and RPDs supported by SCs or FDPs. No study reported a calculation of the sample size, and only in the RCT by Sterzenbach et al. [22] the intention-to-treat (ITT) analysis was applied. If ITT analysis and calculation of the sample size are not performed, an effect that is not truly present could be wrongly detected, leading to a distortion of the results of the analysis (i.e., false positive) and to a difficult interpretation of the results of an investigation. Furthermore, only Mannocci et al. [19] reported the

maxillary and mandibular location of the teeth. Differences in failure rates between the mono and multiradicular teeth are not discussed; the biomechanical behavior of a maxillary incisor is highly different from that of a mandibular molar and, consequently, not reporting the location of the teeth represents a bias leading to an ambiguous interpretation of the results of an investigation. Furthermore, no study considered the so-called "pseudoferrules": indeed, the ferrule may exist but the form of its preparation is not correctly made and this could affect the resistance for tilting. All these factors could have affected the results of the included studies.

Recently, a systematic review and meta-analysis by Figueiredo et al. [5] showed that fiber and metal posts resulted in similar incidence of root fractures and survival rates, not supporting the indications for fiber posts based on the reduction of failures; however, the authors reported that the included studies presented high risks of bias. Similarly, the present systematic review shows some limitations, such as language bias, strict inclusion criteria and few included RCTs presenting high risk of bias. The lack of studies with high methodological quality was confirmed by Schmitter et al. [32], which noticed that the reviews with the highest R-AMSTAR scores reported lower failure rates for fiber posts; however, no definitive clinical conclusions can be found due to the limited number of available high quality studies.

For future research, further RCTs focused on biological, technical, and esthetical prosthetic complications of endodontically treated teeth restored with fiber posts would be desirable to better understand how different types of prosthetic restorations could affect the survival of fiber posts.

#### Conclusions

According to the inclusion and exclusion criteria selected in the present systematic review of the literature, the included studies were too heterogeneous and scarcely comparable to achieve clear clinical statements; furthermore, to date, a univocal correlation between failure rates of fiber posts and typology of prosthetic restorations (SC or FDP) cannot be found.

Within the limitations of this systematic review and the lack of available clinical data, the majority of failure events were due to post debonding and dislodgements of SCs.

Further clinical data are needed in order to establish a possible correlation between failures and typology of restoration, so as to postulate predictable guidelines in the restoration of endodontically treated teeth.

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#### Chapter 3

#### 3.1 Innovative Dental Technologies: CAD/CAM

CAD/CAM began its dental life in 1970s with the first workers to explore its application in dentistry being Duret and Preston [1]. This was followed by the work of Moermann in the 1980s, which led to the development of the CEREC<sup>®</sup> system. CAD/CAM has now become a well accepted technology in most modern dental laboratories and for some enterprising clinicians at the chairside [2].

The development of CAD/CAM is based on three elements, namely: (1) data acquisition, (2) data processing and (3) manufacturing (Fig. 1). The exponential increase in power of computers has resulted in major advances in all of these areas. This is particularly exemplified by the recent introduction of intra-oral scanners. Thus it is possible to create a 3D model of the oral cavity directly with such a system, without the need to take an impression, pour a model and then digitize indirectly the model with one of the many laser scanner that are now available. The digital model can now be used to design the restoration and there are now many software packages available for the design of dental restorations such as crowns, bridges and partial denture frameworks. Some software providers are now able to claim that their partial denture software can survey,
design and wax a partial denture framework in less than 20 min [4].

A further development in the CAD/CAM technologies used in dentistry is the transition from closed to open access systems. Whereas in the past the digitizing, designing and manufacturing came as a closed system (e.g. CEREC<sup>®</sup>), more and more the technology is being opened up and the component part of a CAD/CAM system can be purchased separately [5]. This creates much greater flexibility in that data can be acquired from a range of sources (intra-oral scanner, contact or laser model digitizer, Computer Tomography, Magnetic Risonance Imaging), appropriate design software can be matched to the object to be manufactured (e.g. crown and bridge frameworks, partial denture frameworks, customized implants and implant abutments). Another very important consequence of the transition from closed to open systems is that this opens up access to a much wider range of manufacturing techniques such that the most appropriate manufacturing processes and associated materials can be selected. Thus, it is no longer necessary to be limited by the computer numerically controlled machining technologies that are currently used in most dental CAD/CAM systems.

#### 3.1.1 Subtractive manufacturing

If we look at where we are today then CAD/CAM in dentistry is primarily based around the process of subtractive manufacturing. The technology most people will be familiar with is computer numerically controlled machining, which is based on processes in which power-driven machine tools, such as saws, lathes, milling machines, and drill presses, are used with a sharp cutting tool to mechanically cut the material to achieve the desired geometry with all the steps controlled by a computer program. Thus the process starts out with a block of the material and the machine cuts away the bits that are not wanted. It has been demonstrated that by using this method the overall production time will be reduced considerably and complex models, which are otherwise difficult and/or impossible to make by the conventional dental processes, could be built up rather easily. These technologies have achieved a high degree of sophistication with new technologies such as electrical discharge machining, electrochemical machining, electron beam machining, photochemical machining, and ultrasonic machining [6,7]. Nowadays all these processing routes come under the umbrella of subtractive machining. However, as one might imagine this method of manufacturing is very wasteful as more material is removed compared to what is used in the final product.

In the aerospace industry there is currently much talk about the weight to flight ratio of an airplane. What this refers to is the weight of material that has to be used in relation to the weight of material in the final product such as an airplane. The aerospace industry uses expensive materials and is thus concerned about saving cost by reducing the weight to flight ratio. For example, a 1 kg reduction in weight can save \$3000 in fuel per annum and this means potential savings in the longer term of many billions of dollars [9]. Similarly this is a concern in the automotive industry. As a consequence of this drive for saving costs there has been a major transition from making parts by subtractive manufacturing to what is referred to as additive manufacturing. Using additive methods for manufacturing is more advantageous as many problems associated with milling can be readily overcome. The main advantage of this type of manufacturing is the ability of the technique to create fine detail such as undercuts, voids, and complex internal geometries. Another limitation of the current dental CAD-CAM systems is that the process does not easily lend itself to mass production such as crowns and bridges, since only one part can be machined at any one time.

#### 3.1.2 Additive manufacturing

So what is additive manufacturing and what are its benefits? Additive manufacturing is defined by the American Society for Testing and Materials (ASTM) as: The process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies.

In principle the process works by taking a 3D computer file and creating a series of cross-sectional slices. Each slice is then printed one on top of the other to create the 3D object. One attractive feature of this process is that there is no waste. Traditionally additive manufacturing processes started to be used in the 1980s to manufacture prototypes, models and casting patterns. Thus it has its origins in rapid prototyping (RP), which is the name given to the rapid production of models using additive layer manufacturing. Today additive manufacturing describes technologies that can be used anywhere throughout the product life cycle from pre-production (i.e. rapid prototyping) to full scale production (also known as rapid manufacturing) and even for tooling applications or post production customization. It is a remarkably rapidly changing field with a huge investment in developing enhanced manufacturing technologies and it is changing the way we make things. Today, additive manufacturing is used for a much wider range of applications and is even used to manufacture production-quality parts in relatively small numbers. Some

sculptors use the technology to produce complex shapes for fine arts exhibitions [10].

Thus additive manufacturing is transitioning from rapid prototyping models to manufacturing real parts for use as final products. The equipment is becoming competitive with traditional manufacturing techniques in terms of price, speed, reliability, and cost of use. This, in turn, has led to the expansion of its use in industry and there has been explosive growth in the sales and distribution of the equipment. In addition a new industry is emerging to create software to enable more effective use of the technology. The use of the technology is likely to grow especially as it is now possible to purchase a 3D printer for less than \$3500.00 [11]. Consequently centers providing 3D printing services are springing up around the globe.

Alongside these developments the number of materials that the industry uses has increased greatly and modern machines can utilize a broad array of polymers, metals and ceramics. As the industry makes the transition from prototypes to functional devices the materials available will begin to play a much bigger role. When producing a prototype it is enough for it to look good, but as we move to functional objects such as customized implants and oral prostheses the materials and their properties become much more important.

It is worth noting that the process of additive manufacturing is in fact ideally suited to dentistry, which has a tradition of producing customized parts made to fit the patient and not the other way around. This is a great opportunity for dentistry and there is already a huge array of additive manufacturing technologies that we can use and these include:

- Stereolithography (SLA)
- Fused deposition modeling (FDM)
- Selective electron beam melting (SEBM)
- Laser powder forming
- Inkjet printing

This list is by no mean exhaustive and every day something new is added. Below some of these technologies are described as to how they work and how they are or might be used in dentistry.

# 3.1.2.1 Stereolithography (SLA)

The term "stereolithography" was first introduced in 1986 by Charles W. Hull, who defined it as a method for making solid objects by successively printing thin layers of an ultraviolet curable material one on top of the other. A concentrated beam of ultraviolet light is focused onto the surface of a vat filled with liquid photopolymer and, as the light beam draws the object onto the surface of the liquid, each time a layer of resin is polymerized or crosslinked. The item is built up layer by layer, to give a solid object [13]. The basic manufacturing process is as follows:

- a 3D model of the desired object is created in a CAD program;
- a software package slices the CAD model up into thin layers, which may be anything from 5 to 20 layers per millimeter and the more layers the better the resolution;
- the laser scans the liquid resin in the vat and it sets, thus creating the first layer;
- the platform drops down into the vat by a fraction of a millimeter and the laser scans the next layer;
- this process is repeated layer by layer until your model is complete.

• once the run is complete, the objects is rinsed with a solvent to remove uncured resin and then placed in an ultraviolet oven that thoroughly cures the resin.

This is not a particularly quick process and depending on the size and number of objects being created, the laser might take a minute or two for each layer. If the object is small, you can produce several of them at the same time as they sit next to each other on the tray. A typical run might take anything from 6 to 12 h and for large objects runs over several days are possible. One of the first applications of additive manufacturing technologies was the production of physical models of the human anatomy based on CT data using SLA. SLA models started to be used in medicine and dentistry for the planning of surgical procedures and as a means of constructing customized implants such as cranioplasties, orbital floors and onlays [7,14–19]. In the early work the focus was on developing the models, which were used either as a template for swaging a titanium implant [18] or the implant was machined [7,16,19]. SLA is now routinely used to produce surgical guides for the placement of dental implants. Its use is gradually being extended to include the manufacture of temporary crowns and bridges and resin models for loss wax casting [20].

#### **3.1.2.2 Fused deposition modeling (FDM)**

There are a number of techniques that come under the umbrella of fused deposition modeling. Generally speaking these methods rely on the materials being extruded from a nozzle and include Fused Filament Fabrication (FFF) where a wire of a thermoplastic material is fed through a heated nozzle. Another approach is for the material to be fed from a reservoir through a syringe such as in the case of the bioplotter.

FFF is an additive manufacturing technology commonly used for modeling, prototyping, and production applications. The technology was developed by S. Scott Crump in the late 1980s and was commercialized in 1990 [21].

FFF works on the principle of laying down material in layers. A plastic filament or metal wire is unwound from a coil and supplies material to an extrusion nozzle which can turn the flow on and off. The nozzle is heated to melt the material and can be moved in both horizontal and vertical directions by a numerically controlled mechanism, directly controlled by a software package. The model or part is produced by extruding small beads of a thermoplastic material to form layers as the material hardens immediately after extrusion from the nozzle [22]. Stepper motors or servo motors are typically employed to move the extrusion head.

Several materials are available with a range of strength and thermal properties.

As well as acrylonitrile butadiene styrene (ABS) polymer, polycarbonates, polycaprolactone, polyphenylsulfones and waxes, a water-soluble material can be used for making temporary supports while manufacturing is in progress. This soluble support material is quickly dissolved with specialized mechanical agitation equipment.

It is interesting to note that this technology has not as yet shown itself in the dental journals for the fabrication of dental restorations, other than as an intermediary to produce wax patterns for subsequent casting. This does not mean that it is not thought about and it is probable that research is ongoing that has not yet reached the stage of being published. An example is a patent assigned to Jeneric/Pentron Inc., which describes the use of a ceramic paste in the form of a filament or wire that can used to fabricate dental restorations using fused filament fabrication (e.g. zirconia restorations) [28].

Whereas fused filament fabrication is based on a wire feed, the alternative is to use a reservoir of material that can, as with FFF, be extruded through a nozzle and put down in layers to create a 3D structure. The bioplotter uses this approach and is capable of printing in multiple materials to build up a 3D structure. The main application of the bioplotter is in the modeling of scaffolds for tissue engineering and organ printing. One attraction of the bioplotter is its ability to use a wide range of materials, including ceramic pastes (HAP and TCP) for creating porous bone scaffolds, bioresorbable polymers such

polycarpolactone and/or poly-l-lactide for drug delivery and agar, gelatine, chitosan, collagen, alginate and fibrin as carriers for cells that are used in organ printing. The bioplotter has a resolution of just a few micrometers, which means that it is able to create microstructural patterns that enhance cell invasion, proliferation, distribution and differentiation into the porous structure [23]. Human body parts being created include blood vessels [24], bone [25] and soft tissue [26]. By using a microsyringe the detail that can be printed can produce patterns fine enough to provide guidance to blood vessels growing into the scaffold [27].

#### 3.1.2.3 Selective electron beam melting

Selective electron beam melting (SEBM) is a type of additive manufacturing for producing near net shape metal parts. The technology manufactures parts by melting metal powder layer per layer with an electron beam in a high vacuum [29]. The stream of electrons is created by heating a tungsten filament and the beam is then directed using a magnetic field. Because it uses electrons rather than light, the energy in the beam is very high. Consequently, unlike some metal sintering techniques, the parts are fully dense, void-free, and extremely strong.

This technology has already found wide application in orthopedics and maxillofacial surgery for the construction of customized implants. One of the main attractions is the ability to create highly porous structures in a range of alloys including cp-titanium, Ti-6Al-4V and Co/Cr [30,31]. The potential benefit of porous mesh or foam structures is that the mechanical properties can be adapted to conform more closely to that of bone, particularly the elastic modulus, which may help to prevent stress shielding. In addition, the porous structure permits the ingrowth of bone and provide better fixation of the implant. The accuracy of SEBM is in the range of 0.3-0.4 mm and the surface finish tends to be rough with an  $R_a$  value in the range of  $25 \,\mu$ m. This may be adequate for the manufacture of larger implants such as used in orthopedic and maxillofacial reconstruction but would not be good enough to make crown and bridge frameworks.

# 3.1.2.4 Laser powder forming techniques

Laser based additive manufacturing, such as Selective Laser Melting (SLM) and Selective Laser Sintering (SLS), is accomplished by directing a high power laser using mirrors at a substrate consisting of a fine layer of powder [32,33]. Where the beam hits the powder it creates a melt pool and the powder particles fuse together. After each cross-section is scanned, the powder bed is lowered by one layer thickness, a new layer of material is applied on top, and the process is repeated until the part is completed. This technology is in wide use around the world due to its ability to make very complex geometries directly from digital CAD data. While it began as a way to build prototype parts early in the design cycle, it is increasingly being used in limited-run manufacturing to produce enduse parts. The terminology used can be somewhat confusing, especially as it is still evolving and there is no common agreement on how to differentiate clearly between the various techniques. When processing polymers and ceramic the industry generally refers to this as selective laser sintering whereas for metals the terms used are SLM or DMLS (Direct Metal Laser Sintering).

Compared to other methods of additive manufacturing, SLS/SLM can produce parts from a relatively wide range of commercially available powder materials. These include a wide range of polymers such as polyamide to produce a facial prosthesis [34], ultra high molecular weight polyethylene [35], polycaprolactone to provide functionally graded scaffolds [36], mixtures of polymers such as

polycaprolactone and drugs to act as drug delivery devices [37] and composites such as mixtures of hydroxyapatite and polyethylene and polyamide to produce customized scaffolds for tissue engineering [38]. A range of metal powders can be used that include steel, titanium, titanium alloys, and Co/Cr alloys. The physical process can involve full melting, partial melting, or liquid-phase sintering. Depending on the material, up to 100% density can be achieved with material properties comparable to those from conventional manufacturing methods. In many cases large numbers of parts can be packed within the powder bed, allowing very high levels of productivity. The technology is beginning to find wide acceptance for the construction of implants such as bone analogs [39], orthopedic [40] and dental implants [41] with porous surface features for bone ingrowth, dental crowns and bridges [42] and partial denture frameworks [43]. When considering the use of selective laser sintering to produce medical and dental parts from pure ceramic powders there is still some way to go and many problems that need to be resolved. Nevertheless, there are some promising studies ongoing such as the production of bone scaffolds made from a porous apatite-wollastonite glass ceramic [44]. At the Fraunhofer Institute for Laser Technology in Germany they appear to have managed to produce a zirconia bridge framework using selective laser melting [45].

# 3.1.2.5 Inkjet printing technologies

Inkjet printers are capable of printing at a very high resolution by ejecting extremely small ink drops. Inkjet printing works by propelling individual small droplets of "ink" toward a substrate. In this context the ink can be anything from an aqueous solution of coloring agents and binders to a ceramic suspension, such as used in some studies to produce zirconia dental restorations [46,47] or a cell solution to produce tissue constructs [48]. The ink is forced through a small orifice by a variety of means including pressure, heat, and vibration. One approach consists of building up the object layer by layer from depositing droplets to form a layer of the material and then depositing the next layer. To be used for additive manufacturing, the liquid droplets must change phase to solid upon deposition on the substrate when printing a pattern. Depending on the deposited material, the phase change could be by drying, heat transfer, UV light or chemical reaction. Another method operates in a manner similar to the SLS/SLM approach where a thin layer of powder is spread out, but instead of using a laser, an inkiet head prints a binder. The latter technology is the only one that allows for the printing of multicolored objects across the whole color spectrum.

The polyjet range of printers from Objet is an example of a commercially available inkjet printing technology that builds up layer by layer by depositing droplets of a polymer and as each layer is formed it is cured by UV light [49].

Already the company is exploring a wide range of dental applications such as reproduction of dental models, orthodontic bracket guides, surgical guides for implant placement, mouth guards, sleep apnea appliances and even try-in veneers. A particular feature of this technology is that it can print an object using two materials with quite distinctively different properties. Thus it would be possible to produce a mouth guard with hard and soft regions and it can make them with different colors.

An example of the powder/binder approach is the Z-Corp machines [50], which use a colored binder in up to four inkjet heads and thus is able to produce any color you like. The powder is typically a fine grained silica and the binder is made up of an aqueous solution of coloring agents, usually magenta, cyan and yellow and a resin to act as the glue for the powder particles. The product once made is quite fragile until the porosities between the powder particles has been infiltrated with another resin such as a cyanoacrylate. This technology has been used to produce porous calcium polyphosphate (CPP) structures for tissue engineering [51]. The CPP particle are mixed with polyvinyl acetate and are bonded to each other by injection with the binder which dissolves the PVA producing connecting bridges between the CPP particles. The binder is burnt off and then the article is sintered, producing a porous scaffold. By using a different combination of materials, the University of Sheffield is using this technology to develop colored soft tissue prostheses.

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# Chapter 4

### 4.1 CAD/CAM Materials in Prosthodontics

#### **4.1.1 Metals in Prosthodontics**

Metal-ceramic restorations are commonly provided in dental practice, primarily because of their acceptable biological, mechanical, and esthetic properties. The success of these restorations depends on the presence of a strong bond between the porcelain and metal substructure. Noble metal alloys are generally preferred for the metal frameworks, because of their biocompatibility, good mechanical properties, and excellent ceramic-to-metal bond; but, base metal (Ni-ti ,Ni–Cr and Co–Cr) casting alloys are extensively used worldwide because of economic considerations.

However, casting of base metal alloys is more technique sensitive compared to casting of noble alloys because of the high melting range and oxidation of base metal alloys during casting. In addition, due to their high hardness, grinding of cast base metal alloys to finish castings is time consuming for dental laboratories and also they tend to form thicker, darker oxide layers that may present esthetic problems. But new technologies, as CAD/CAM laser welding or spark erosion, may replace casting of the base metal alloys reducing time, increasing the precision of fit and reducing aesthetic problems.

Among the base metals, two main categories of alloys exist: nickel based and cobalt based. Alloys in both systems contain chromium as their second largest constituent and depend upon it for corrosion resistance.

Recent epidemiological data suggests over 65 million people in Europe have undergone at least one reaction to nickel (Ni) in their lifetime. Oral exposure to Ni has been definitively shown to induce dermatitic flares in Ni-sensitised persons. In addition, Ni hypersensitivity reactions from the leaching of metallic elemental components into the surrounding oral mucosal tissues have been identified to be associated with Ni-based fixed prosthodontic and orthodontic dental appliances. Conversely, the Co-Cr appears to be more biocompatible than Ni.

# 4.1.1.1 Posterior CAD/CAM cobalt-chromium alloy single crowns: 4-year prospective clinical study

Leone R., Zarone F., Piombino P., Sorrentino R. Dental Materials

#### Introduction

Due to the increasing cost of noble metals, the use of cobalt-chromium (Co-Cr) alloys for dental restorations has become more and more widespread with various and successful clinical applications (Al Jabbari, J Adv Prosthodont 2014).

According to the classification proposed by the American Dental Association (ADA) in 1984 (ANSI/ADA Specification No. 38, J Am Dent Assoc 1984), the Co-Cr alloys are predominantly base metals (noble metal content < 25%); they are composed of 75 wt% or more of base metal elements and of 25 wt% or less of noble metals (Au, Ir, Os, Pt, Rh, Ru), although in clinical practice they do not contain noble metal elements at all (ISO 6871-1, 1994; Wataha, J Prosthet Dent 2002; Roberts et al. J Prosthodont 2009).

The binary Co-Cr alloy was proved to be very strong and stain resistant. It is characterized by high strength, heat resistance, limited fatigue damage and excellent biocompatibility; it is non-magnetic (so particularly indicated in patients undergoing magnetic resonance imaging or MRI) and demonstrated favorable resistance to corrosion, wear and tarnish (Viennot et al., Eur J Oral Sci 2005; Okazaki and Gotoh, Biomater 2005; Serra-Prat et al., J Prosthet Dent 2014). Moreover, the Co-Cr alloy shows a high modulus of elasticity (E: 200-220 GPa), providing reliable rigidity for intraoral use with no need for heavy cross-sections even in case of long span fixed dental prostheses (FDPs), so reducing the weight and room of metal frameworks (Svanborg et al., Int J Prosthodont 2013; Al Jabbari, J Adv Prosthodont 2014).

However, casting of base metal alloys is more technique sensitive compared to that of noble alloys, mainly because of the high melting range and oxidation of base metal alloys during casting (Lucchetti et al., J Prosthet Dent 2015).

Base metal alloys tend to form thicker and darker oxide layers that could cause esthetic drawbacks (Eliasson et al., J Prosthet Dent 2007). Moreover, increased oxidation could cause poor bond strength between Co-Cr and the veneering porcelain due to chromium ions diffusion (Wu et al., J Prosthet Dent 1991). Consequently, other metallic components, such as Ce, Ga and Nb, can be added in Co-Cr alloys to control thermal expansion, provide fluidity and modify the oxidation characteristics, so improving the metal-ceramic bond. Molybdenum (Mo) and tungsten (W) can be used as strengthening agents (Al Jabbari, J Adv Prosthodont 2014).

The high solidus temperature of Co-Cr alloys, different from the ceramic sintering temperature, reduces the risk of framework distortion after sintering (Kelly and Rose, J Prosthet Dent 1983). Nonetheless, the high coefficient of thermal expansion and melting temperature could cause technical drawbacks during the dental laboratory procedures (Bezzon et al., J Prosthet Dent 2004). The stiffness of Co-Cr alloys makes it more difficult to grind or cut the frameworks, making finishing more time consuming (Eliasson et al., J Prosthet Dent 2007; Svanborg et al., Int J Prosthodont 2013).

As to dental applications, the Co-Cr alloys were first used in the 1930s to fabricate the substructures of removable partial dentures (RPDs) (Al Jabbari, J Adv Prosthodont 2014). Their popularity increased rapidly, since they have almost half the density of gold-based alloys and consequently the weight of dental restorations was significantly lighter (Wataha, J Prosthet Dent 2002).

The use of the Co-Cr alloys for the fabrication of porcelain-fused-to-metal (PFM) FDPs began in the 1970s, due to the rapid escalation of the price of gold. Nowadays, Co-Cr alloys are mainly used to produce the frameworks of RPDs, single crowns (SCs) and FDPs as alternatives to other metals: they are cheaper than gold and free from the risk of Ni-related allergic responses (Vermeulen et al., J Prosthet Dent 1996; Leinfelder, J Am Dent Assoc 1997; Bertrand and Poulon-Quintin, J Prosthodont 2010; van Noort, Dent Mater 2012; Syed et al. J Clin Diagn Res 2015).

Recently, different technologies alternative to conventional casting were proposed to produce Co-Cr frameworks and reduce handling difficulties: Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) and Selective Laser Melting (SLM), commonly known as the laser sintering technique or spark erosion (Beuer et al., Br Dent J 2008; van Noort, Dent Mater

2012). Both fabrication methods could limit the weakening due to internal porosities and represent viable alternatives to conventional casting (Al Jabbari, J Adv Prosthodont 2014). Furthermore, CAD/CAM and laser sintering were proved to reduce production time, improve the precision of fit and limit estethic problems due to oxidation of Co-Cr frameworks (Ucar et al., J Prosthet Dent 2009; Ortorp et al., Dent Mater 2011; Keul et al., Dent Mater 2014).

Although Co-Cr alloys have been used as an alternative to conventional noble metals in fixed prosthodontics, to date only a few studies investigated the clinical performances of Co-Cr prostheses (Eliasson et al., J Prosthet Dent 2007; Hjalmarsson et al., Int J Prosthodont 2011; Tara et al., Int J Prosthodont 2011; Svanborg et al., Int J Prosthodont 2013). No adverse reaction to Co-Cr were reported but some patients experienced both biological and technical problems; a few ceramic fractures were reported after 3 to 7 years of clinical service (Eliasson et al., J Prosthet Dent 2007). Conversely, no ceramic chipping was reported after 47 months of function in laser sintered Co-Cr SCs and the clinical results were comparable to those obtained with conventional metal-ceramic restorations (Tara et al., Int J Prosthodont 2011). Similar results were also achieved on implants with Co-Cr prostheses veneered with ceramics and titanium-acrylic restorations after 5 years of function; veneering chipping were noticed in both groups (Hjalmarsson et al., Int J Prosthodont 2011).

The present prospective clinical study aimed at evaluating the 4-year clinical outcomes of ceramic veneered CAD/CAM Co-Cr single crowns supported by

natural teeth in posterior regions.

#### **Materials and Methods**

#### *Recruitment of patients*

Eighty-nine consecutive patients in need of single restorations in posterior areas of both maxilla and mandible were enrolled in the present prospective clinical protocol.

Fifty-two male and 37 female patients were recruited from May to July 2012 at the Department of Fixed Prosthodontics of the University "Federico II" of Naples (Italy) and were included in the present prospective study; their ages ranged from 21 to 68 years (mean age 41.2±8.4). All patients were in good general health; none of them showed parafunctional habits and 37 were smokers. The requirements of the Helsinki declaration were fulfilled; before being included in the study, all patients underwent an informative interview and had to sign a written consent form. The present prospective clinical study was approved by the Ethical Committee of the University "Federico II" of Naples.

## Inclusion and exclusion criteria

The following inclusion criteria were used to recruit patients:

- good general health;
- ASA I or ASA II according to the American Society of Anesthesiologists;
- periodontal health;

- Angle class I occlusal relationship;
- minimum of 10 couples of opponent teeth;
- good oral hygiene;
- no evident signs of parafunctions and/or temporomandibular disorders.

Furthermore, the abutment teeth had to fulfill the following inclusion criteria:

- periodontal health (absence of tooth mobility, absence of furcation involvement);
- proper positioning in the dental arch (tooth axis adequate for a SC);
- sufficient occlusal-cervical height of the clinical crown ( $\geq 4$  mm) for the retention of a SC;
- vital or endodontically treated to a clinically sound state;
- opposing natural teeth.

Conversely, in the presence of the following conditions, patients were excluded from the study:

- high caries activity;
- presence of periodontal disease on the abutment tooth;
- occlusal-cervical height of the abutment tooth < 4 mm;
- reduced interocclusal distance or supererupted opposing teeth;
- unfavorable crown-to-root ratio;
- severe were facets, clenching and/or bruxism;
- presence of RPDs;
- pregnancy or lactation;

- alcohol and/or drug addiction.

#### **Prosthodontic procedures**

A total of 120 ceramic veneered CAD/CAM Co-Cr SCs replacing either premolars and molars in both maxilla and mandible were fabricated; each patient received only 1 crown. The distribution of the restorations was reported in Table 1.

All the prosthodontics procedures were performed by 2 experienced and calibrated prosthodontists and by 1 expert dental technician. Oral hygiene procedures as well as any necessary core build-up, endodontic treatment and/or post-and-core placement were carried out before the prosthodontics steps.

Preliminary alginate impressions were made to get study gypsum casts, diagnostic wax-ups, light-cured resin customized impression trays and acrylic temporary restorations. Silicone indexes were fabricated from the diagnostic wax-ups to check a proper tooth structure reduction during the procedures of abutment preparation that were standardized as follows, according to the requirements of the CAD-CAM framework production:

- margin design: 1 mm circumferential rounded chamfer;
- cavo-surface angles: rounded;
- axial reduction: 1.5 mm;
- occlusal reduction: 1.5-2 mm;
- total occlusal convergence angle: 10°-14°.

The margins of the preparations were slightly subgingival, never violating the biologic width. The acrylic resin temporary restorations were relined intraorally with self-polymerizing resin and then cemented with a eugenol-free luting agent (Temp Bond NE, Kerr Corporation, Orange, CA, USA); careful occlusal adjustment of the provisional restorations was performed.

Two weeks were waited after tooth preparation before taking final impressions in order to allow the soft tissues to recover from preparation trauma. The final impressions were taken placing 2 non-impregnated retraction cords (Ultrapak, Ultradent, South Jordan, UT, USA) around the abutment teeth to displace the gingival tissues and taking full-arch impressions with customized light-cured acrylic impression trays and polyether materials (Impregum and Permadyne-L, 3M ESPE, Seefeld, Germany). Intermaxillary registrations were taken by means of a self-polymerizing A-silicone. Then, the provisional restorations were relined and cemented again as previously described.

The master casts were fabricated with super hard gypsum and mounted in semiadjustable articulators. A die spacer (thickness: 30 microns) was applied at the occlusal and axial surfaces of the abutment, starting 1 mm above the preparation margin. The master casts were digitized by means of the *Sweden&Martina* CAD-CAM system. Co-Cr single frameworks were designed according to the manufacturer's instructions and providing room for an even thickness of the veneering ceramic. The frameworks were milled from Co-Cr blanks at the *Sweden&Martina* center. The framework thickness was checked at occlusal,

axial and marginal surfaces with a digital caliper with an accuracy of 0.01 mm.

The Co-Cr structures were tried-in intraorally and evaluated for accuracy of fit with a silicone disclosing agent (Fit Checker, GC, Tokyo, Japan); if necessary, any pressure spot was transferred to the tooth surface and the adjustment made on the abutment tooth. The marginal precision was checked by means of standardized periodical radiographs.

All the frameworks were veneered by the same experienced dental technician. A conventional powder build-up veneering technique was performed using a feldspathic ceramic specifically dedicated to Co-Cr structures.

CAD/CAM Co-Cr frameworks were fabricated and veneered with ceramics. The restorations were cemented using a eugenol-free zinc oxide luting agent. The patients were recalled after 1, 6, 12, 24, 36 and 48 months. The survival and success of the restorations were evaluated. The technical and esthetic outcomes were examined using the United States Public Health Service criteria. The biologic outcomes were analyzed at abutment and contralateral teeth and descriptive statistics were performed.

	Maxilla	Mandible	Total
1st premolar	26	7	33
2nd premolar	12	5	17
1st molar	20	23	43
2nd molar	11	16	27
TOTAL	69	51	120

**Table 1 -** Locations of the Co-Cr single crowns.

#### Results

All of the 120 patients and, consequently, all of the 120 Co-Cr single crowns were examined during 4 years of clinical function. No patient was lost at followup or censored.

As to the technical problems, neither fractures of the frameworks nor losses of retention were observed in all of the samples. The cumulative survival rate was 100% while the cumulative success rate was 99.1% after 4 years according to Kaplan–Meier, considering veneering ceramic chippings as events.(Table 2) During the entire observational period, one minor cohesive fracture of veneering ceramic was noticed: the chipping was detected, at the recall after 1 year of clinical service, on the occlusal surface of a mandibular molar. Such cohesive fracture did not impair function, neither was it noticed by the patient. Consequently, the chipped areas was carefully rounded and polished so that the SC remained in situ for further observation.

One hundred and two abutments (85%) were vital at the beginning of the study, and they all remained vital during the entire observational period. No significant differences in the average periodontal parameters between test and control teeth were detected at any follow-up examination. Neither radiographic evidence nor signs or symptoms of proximal decay or periapical pathologies were noticed during the entire follow-up period. According to the patients' VAS judgments, the overall function of the SCs showed a mean value of 9.1 ( $\pm$ 1.2) while the overall aesthetics scored a mean value of  $9.4 (\pm 0.4)$ .

The technical evaluation by means of the USPHS criteria revealed very good clinical performances of the Co-Cr SCs (Table 2). In terms of fracture resistance, all of the frameworks rated alpha. Regarding occlusal wear, two restorations rated bravo, and occlusal wear was detected mainly at the level of the opposing natural teeth.

According to the Wilcoxon test, the periodontal parameters of the test and the control teeth were not significantly different. Furthermore, the SCs had no effect on the periodontal parameters after 4 years of clinical function.

USPHS criteria	Alpha (A)	Bravo (B)	Charlie (C)	Delta (D)
Framework fracture	120(100%)	0	0	0
Veneering fracture	119(99,1%)	0	0	1(0,8%)
Occlusal wear	118(98,3%)	2(1,6%)	0	0
Marginal adaptation	119(99,1%)	1(0,8%)	0	0
Anatomical form	120(100%)	0	0	0

|--|

USPHS criteria	Alpha (A)	Bravo (B)	Charlie (C)	Delta (D)
Framework fracture	No fracture of framework	Ι	—	Fracture of framework
Veneering fracture	No fracture	Chipping but polishing possible	Chipping down to the framework	New restoration is needed
Occlusal wear	No occlusal wear on restoration or on opposite teeth	Occlusal wear on restoration or on opposite teeth <2 mm	Occlusal wear on restoration or on opposite teeth >2 mm	New restoration is needed
Marginal adaptation	No probe catch	Slight probe catch but no gap	Gap with some dentin or cement exposure	New restoration is needed
Anatomical form	ldeal anatomical shape, good proximal contacts	Slightly over- or undercontoured, weak proximal contacts	Highly over- or undercontoured, open proximal contacts	New restoration is needed

"United States Public Health Service" criteria

#### Discussion

Co-Cr made with CAD-CAM technique offers excellent flexural strength, fracture toughness, and good biocompatibility, together with acceptable marginal and internal adaptation of the restorations, all factors that undeniably contribute to the long-term success of SCs.(12)

Co-CR SCs were reported to show a certain amount of both biologic complications, like secondary caries, and technical problems, such as chippings of the veneering ceramic.

The primary requirement for the success of a metal-ceramic restoration is the development of reliable bonding between the veneering ceramic and the alloy.(13)

Stress concentration during ceramic cooling can result in ceramic chipping, either immediately or in a delayed response.(15,16)

Chipping and delaminating of veneering ceramics are critical problems in the fabrication of metal ceramic restorations,19 for both base metal and noble metal alloys. Chemical bonding is the primary mechanism of interaction between metal and ceramic.(17-15,18). Some studies have shown that satisfactory bond strength is obtained between Co-Cr and Ni-Cr alloys and veneering porcelain.(19,20)

Evidence also suggests that airborne-particle abrasion of bonding surfaces increases the metal surface energy, improving the wettability of opaque ceramic

and, consequently, the bond strength, through micromechanical bonding.(21)

All of the periodontal parameters did not significantly change over the entire observational period. These results agree with those of other clinical investigations and confirm the good biological response of the soft tissues to Co-Cr restorations (22). A slight gingival inflammation with positive BOP was noticed in a few cases, but no involvement of deep periodontal structures was detected until the end of the examination time.

Indubitably, a correct management of the prosthetic procedures is to be addressed as one of the main success factors in order to avoid possible biological complications like recurrent caries and periodontal problems: an accurate abutment preparation; a precise provisional prosthesis for an optimal soft tissue conditioning; a flawless impression, delayed from 10 to 14 days after tooth preparation for achieving stable and sound soft tissues; and a careful, conventional cementation are all paramount for the final results.

#### Conclusion

Within the limitations of the present study and its observational period, the excellent survival rate of single posterior Co-Cr frameworks allows to address this kind of restoration as a valid treatment option and a viable alternative to noble metal-ceramic SCs in clinical cases with favorable biomechanical conditions.

The following conclusions can be drawn:

 No framework fractures were detected while minor chippings of veneering ceramic were noticed in one SC;

 Co-Cr cores exhibited sufficient strength to ensure a predictable serviceability for posterior SCs in the absence of excessive or parafunctional loads;

 Tooth-supported posterior Co-Cr SCs showed very good mechanical performances in terms of clinical fracture resistance and marginal integrity;

The renowned biocompatibility of Co-Cr was confirmed by the evidence of sound support tissues;

The overall aesthetics and function were very satisfactory for the patients
 (Table 3) and very promising for the clinicians in the medium term.

Within the limitations of the present study and its observational period, the excellent survival rate of single posterior Co-Cr frameworks made with CAD-CAM technique allows to address this kind of restoration as a valid treatment option in posterior areas and a viable alternative to noble metal-ceramic single crowns.

Satisfaction score		Observational period (mo)		
	0 < x > 12	12 < x > 24	24 < x > 36	36 < x > 48
Nonacceptable	0	0	0	0
Acceptable	0	0	0	0
Good	0	2(1,6%)	3(2,5%)	3(2,5%)
Excellent	120(100%)	118(98,3%)	117(97,5%)	117(97,5)

Table 3


Fig. 1. Digital Cast



Fig. 2. Framework Try-in



Fig. 3. Ceramic Try-in



Fig. 4. 48-month Follow-up

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### **4.1.2 Ceramics in Prosthodontics**

Dental ceramics are materials that are part of systems designed with the purpose of producing dental prostheses that in turn are used to replace missing or damaged dental structures. The literature on this topic defines ceramics as inorganic, non-metallic materials made by man by the heating of raw minerals at high temperatures.[1]

Ceramics and glasses are brittle, which means that they display a high compressive strength but low tensile strength and may be fractured under very low strain (0.1%, 0.2%).

As restorative materials, dental ceramics have disadvantages mostly due to their inability to withstand functional forces that are present in the oral cavity. Hence, initially, they found limited application in the premolar and molar areas, although further development in these materials has enabled their use as a posterior long-span fixed partial prosthetic restorations and structures on dental implants.[2] All dental ceramics display low fracture toughness when compared with other dental materials, such as metals.[3]

Ceramics can be classified by their microstructure:

- Glass-based Ceramic
- Crystalline ceramics
- Polycrystalline ceramics

### 4.1.2.1 Glass Ceramics

Glass ceramics were first developed by Corning Glass Works in the late 1950s. According to McLean,[11] the first works on glass ceramics were performed by Mac Culloch, but his work did not receive much attention. Further investigations by Grossman and Adair[12,13] concluded with the development of a tetra silicic fluormica-containing ceramic system.

In principle, an article is formed while liquid and a metastable glass results on cooling. During a subsequent heat treatment, controlled crystallization occurs, with the nucleation and growth of internal crystals. This conversion process from a glass to a partially crystalline glass is called *ceraming*. Thus, a glass ceramic is a multiphase solid containing a residual glass phase with a finely dispersed crystalline phase. The controlled crystallisation of the glass results in the formation of tiny crystals that are evenly distributed throughout the glass. The number of crystals, their growth rate and thus their size are regulated by the time and temperature of the creaming heat treatment.

Its composition is as follows: 45-70% SiO<sub>2</sub>, 8-20% MgO, 8-15% MgF<sub>2</sub>, 5-35%  $R_2O + RO$ , where  $R_2O$  has a range between 5-25% and is composed of at least one of the following oxides: 0-20%  $K_2O$ , 0-23%  $Rb_2O$  and 0-25%  $Cs_2O$  to improve translucency and RO, which has a range between 0-20%, and is composed of at least one of the following oxides: SrO, BaO and CdO.

Additional components may account for up to 10% of Sb<sub>2</sub>O<sub>5</sub> and/or up to 5% of traditional glassy colorants.[12,13]

There are two important aspects to the formation of the crystalline phase: crystal nucleation and crystal growth. The thermal treatment known as ceraming[14] is composed of two processes: glass is heated up to a temperature where nuclei form  $(750^{\circ}-850^{\circ}C)$ , and this temperature is kept for a period of time ranging from 1 to 6 h so that crystalline nuclei form in the glass (process known as nucleation). Then, the temperature is increased to the crystallization point  $(1000^{\circ}-1150^{\circ}C)$  and this temperature is maintained for a period ranging from 1 to 6 h until the desired level of glazing is obtained (process known as crystallization).[12,15]

### 4.1.2.1.1 Glass-based systems with fillers

### Leucite-reinforced feldspar glass ceramics

Glass-based systems are made from materials that contain mainly silicon dioxide (also known as silica or quartz), which contains various amounts of alumina.

Aluminosilicates found in nature, which contain various amounts of potassium and sodium, are known as feldspars. Feldspars are modified in various ways to create the glass used in dentistry. Synthetic forms of aluminosilicate glasses are also manufactured for dental ceramics.[16,17]

Pressed glass ceramics are materials containing high amounts of leucite crystals (35% by volume).[14] The basic component of this ceramic is feldspathic porcelain, consisting of 63% SiO<sub>2</sub>, 19% Al<sub>2</sub>O<sub>3</sub>, 11% K<sub>2</sub>O, 4% Na<sub>2</sub>O and traces of other oxides. Leucite crystals are added to the aluminum oxide.[18,19]

This material is manufactured using a process known as heat pressing, which is performed in an investment mold. This mold is filled with the plasticized ceramic thus avoiding the sintering process and the subsequent pore formation.[20] This ceramic undergoes dispersion strengthening through the guided crystallization of leucite.

Dispersion strengthening is a process by which the dispersed phase of a different material (such as alumina, leucite, zirconia, etc.) is used to stop crack

propagation as these crystalline phases are more difficult to penetrate by cracks.[14,21]

Leucite crystals are incorporated during ceraming and hence performing this process again is unnecessary when inducing crystal growth.[19]

The construction of ceramic restorations using leucite-reinforced feldspars can be done either by sintering, using a modified version of the sintering process described earlier to construct the porcelain jacket crown, or by a process known as hot pressing.

### 4.1.2.1.2 Lithium Disilicate and Apatite Glass Ceramics

In order to be able to extend the use of resin-bonded ceramic restorations and possibly use them for bridge construction, a glass ceramic based on a SiO<sub>2</sub>-Li<sub>2</sub>O system has been developed (Empress II, Ivoclar-Vivadent). To increase the strength, thermal expansion and contraction behavior of ceramics, manufacturers have added crystalline filler particles.[22] Other types of filler additions include particles of high-melting glasses that are stable at the firing temperature of the ceramic.[23] Kelly[22] refers to a ceramic as a "glass-ceramic" when the filler particles are added mechanically during manufacturing precipitate within the starting glass by special nucleation and growth-heating treatments. The crystalline phase that forms is a lithium disilicate (Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>) and makes up about 70% of the volume of the glass ceramic. Lithium disilicate has an unusual microstructure, in that it consists of many small interlocking plate-like crystals that are randomly oriented. This is ideal from the point of view of strength because the needle-like crystals cause cracks to deflect, branch or blunt; thus, the propagation of cracks through this material is arrested by the lithium disilicate crystals, providing a substantial increase in the flexural strength.

A second crystalline phase, consisting of a lithium orthophosphate ( $Li_3PO_4$ ) of a much lower volume is also present. The mechanical properties of this glass ceramic are far superior to that of the leucite glass ceramic, with a flexural strength in the region of 350–450 MPa and fracture toughness approximately

three-times that of the leucite glass ceramic. The glass ceramic is claimed to be highly translucent due to the optical compatibility between the glassy matrix and the crystalline phase, which minimizes internal scattering of the light as it passes through the material.

The processing route is the same as the hot-pressing route described above, except that the processing temperature, at 920°C, is lower than for the leucite glass ceramic. The grain sizes of lithium metasilicate crystals range from 0.2  $\mu$ m to 1  $\mu$ m, rendering a flexural strength of 130 MPa to this material. This is comparable to the other mill-ready leucite-reinforced CAD/CAM (ProCAD, Ivoclar Vivadent) blocks and the feldspathic CAD/CAM blocks (Vitabloc Mark II).[24]

During the crystallization cycle, there is a controlled growth of the grain size  $(0.5-5 \ \mu m)$ . This transformation leads to a glass ceramic that is made up of prismatic lithium disilicate dispersed in a glassy matrix.[25] This alteration increases the flexural strength of the restoration to 360 MPa,[26] an increase of 170%. A random orientation of small interlocking plate-like crystals makes up the lithium-disilicate restoration. The orientation and size of the crystals can account for crack deflection and blunting, which, in turn, accounts for the increase in fracture toughness over the leucite-reinforced ceramics.[27]

There are two basic fabrication methods. The first method is to mill the restoration to full anatomical contour. Before crystallization, the incisal edge is preserved by creating a silicone index. The incisal edge is cut back, creating mamelons, and is layered with the appropriate incisal porcelains back to the original contour using the silicone index as the guide. The restoration is then crystallized in the furnace using the standard firing program. A variation of this technique is crystallizing before the layering steps. This method allows the operator to see the colour of the restoration before application of the layering ceramics. This does require a wash coat firing of the layering ceramic before the build-up ceramic is applied.

The second method is to mill the crown to full contour, then stain, glaze and crystallize. This method also has a variation that includes applying the stain and glaze after the crystallization step. This allows the operator to see the final color of the crown while applying the stains. It may be easier to apply the stains, but it involves a second 12-min firing cycle.

### 4.1.2.1.3 Crystalline-based Systems with Glass Fillers Glass-infiltrated high-strength ceramic core systems

The addition of alumina to the feldspathic glass during the pre-fritting process limits the amount of alumina that can be incorporated to about 40–50 vol.%. An alternative approach has been adopted in a system called In-Ceram (Vita). This core material has an alumina content of 85%. A ceramic core is formed onto a refractory die from a fine slurry of alumina powder by a process known as slip casting. After the die has dried, it is sintered for 10 h at 1120°C. The melting temperature of alumina is too high to produce full densification of the powder by liquid phase sintering, and solid phase sintering alone occurs. Consequently, the coping thus created is only just held together at the contact points between the alumina particles, and a porous structure is the result. The strength of this porous core is only about 6–10MPa. The porous structure is then infiltrated with a lanthanum glass, which has a low viscosity when fired at 1100°C for 4–6 h, which increases the strength. The molten glass is able to penetrate into the pores, producing a dense ceramic. The esthetics and functional form are then achieved by the use of conventional feldspathic dental ceramics. [28,29]

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### 4.1.2.1.4 Treatment Concept for a Patient with a High Smile Line and Gingival Pigmentation: A Case Report

## Zarone F., Leone R., Ferrari M., Sorrentino R. The International Journal of Periodontics and Restorative Dentistry. 2017 (*in press*)

The importance of healthy, uninamed, light coral pink-shaded gingival tissues is paramount in oral esthetics. It is undeniable that beautiful teeth adjacent to unesthetic gingival tissues cannot fulfill the requirements for an appealing smile[1]. In the last decade, the importance of pink esthetics has been stressed in relation to various gingival parameters affecting the esthetic result, such as the presence of mesial and distal papillae, curvature and level of the buccal mucosa, root convexity, and soft tissue color and texture[2,3]. In particular, the color of sound gingival or peri-implant mucosae can deeply influence the harmonic appearance of a smile; in a natural physiologic condition it should be light pink with a stippled, translucent, orange peel appearance.[4,5]. Such a color depends on several factors: thickness and keratinization of the epithelium; quantity, quality, and distribution of blood vessels; and presence of intraepithelial pigments. such melanin. carotene, reduced hemoglobin, as and oxyhemoglobin.[6,7]

The brown melanin pigment is synthesized in the epithelium at the basal and the suprabasal layers by melanocytes, whose number and distribution in the mucosa is strongly related to their presence in the skin, although their activity is somewhat reduced in the mucosa.[8] Melanin granules (melanosomes) determine the color of hair, skin, mucosae, and eyes, providing protection from stresses, free radicals, and UV radiation and having the ability to sequester metal ions and bind specific drugs and organic molecules.[9] The color of skin and mucosae is genetically determined by the number, size, and distribution of melanosomes and by the type of melanin (ie, eumelanin or pheomelanin).[9] The prevalence of melanin pigmentation is genetically determined, being present in all ethnicities but varying between 0% and 89% as a result of genetic factors and smoking habits.[10] An ethnic pigmentation rate of 38% (27% smokers and 11% non smokers) reported in a Turkish population is midway between the ratios reported for Asian and European populations.[11] A high level of oral pigmentation caused by melanin is frequently observed in Africans, East Asians, and Hispanics.[12]

Aside from genetic phenotype factors, oral hyperpigmentation can be caused or enhanced by conditions such as tobacco use, prolonged intake of drugs (particularly tricyclic antidepressants and antimalarial drugs) and systemic pathologies (eg, Peutz-Jeghers syndrome, Addison disease, neuro bromatosis, malignant melanoma).[13,15]

Although not a pathological condition, gingival hyperpigmentation is frequently considered a major esthetic concern, impairing the harmony of the smile especially in the presence of a high smile line (gummy smile), as in the

described clinical case.[1,14,16]

### **Case Description**

The present case report is related to a 35 year-old dark-skinned Cuban woman who expressed dissatisfaction with the shape of her maxillary central incisors, both of which were fractured at the margins because of previous parafunctional habits that were no longer evident at the time of treatment planning (Fig 1). The oral examination showed an intense brownish pigmentation of the gingival tissues in the maxilla and mandible, the negative effect of which was increased by a very high smile line (Fig 2). At the time of treatment planning, the patient did not accept any proposal of periodontal surgery to remove the pigmentations due to a previous experience of panic attack during a tooth extraction.

The analysis of periodontal tissues did not show signs of inflammation or significant attachment loss. After 15 days of careful oral hygiene procedures (scaling, root planing, and oral hygiene instructions), the patient's periodontal chart showed no signs of periodontal pathology. At the level of the maxillary central incisors, a probing depth of 4 mm was detected in the absence of periodontal bleeding. This was due to hyperplastic tissues further shortening the appearance of the incisors and resulting in a reverse smile line. On this basis, the first step of the treatment was crown-lengthening surgery, performed with a gingivoplasty for esthetic and hygienic reasons.

After inducing local anesthesia in the maxillary incisors region by means of mepivacaine 2% with epinephrine 1:200,000, the pseudopockets were removed using a BP-15 scalpel to an extent of 3 mm with an external bevel incision, taking care not to damage the dentogingival attachment. At that time, the patient mustered up the courage to ask for a last-minute partial pigmentation removal, so the gingival tissues above the maxillary incisors were de-epithelized and the exposed connective tissue was covered with periodontal dressing (Coe Pack, GC). The patient was instructed to regularly brush the teeth that were not involved in the surgery and regularly use a chlorexidine mouthrinse (Curasept 0.2%, Curaden Healthcare) for 1 minute, once per day, for 1 week. No antibiotics were prescribed.

After 30 days of healing, the gingival arches of the central incisors appeared symmetrical and more apically and properly positioned and shaped; moreover, the keratinized adherent gingiva showed no inflammation and a light pink color with a complete absence of any visible melanic pigmentation (Fig 3). An alginate impression was taken (Hydrogum 5, Zhermack) and study casts were poured with type IV dental stone (Elite Rock, Zhermack) to get a wax-up of the maxillary central incisors to be reshaped and resized to their natural length. A mock-up was done using a silicone template (Elite Glass, Zhermack) and self-curing acrylic resin (GC UniFast III, GC).

After the patient accepted the new shape of the central incisors, the teeth were

prepared for ceramic veneers. The preparation design was minimally invasive, with a slight marginal chamfer (0.3 mm), a 0.5-mm reduction at the axial level, and an incisal butt joint, the latter allowing a natural translucency at the incisal margins (Fig 4). Two leucite-filled glass-ceramic veneers (Empress, Ivoclar Vivadent) were fabricated (Fig 5). After try-in and adjustment procedures, the two veneers were cemented using a dual-curing resin cement (Clear 1 Esthetic Cement, Kuraray Noritake), after choosing the proper color by means of try-in pastes (Fig 6).

After 2 months, the patient was satis ed with the results (Fig 7) and asked for a reduction of the high smile line. An experienced esthetic medical doctor selectively injected botulinum toxin to reduce the activity of the lip elevator muscles and lower the smile line (Fig 8). After 2 weeks, the esthetic result was satisfactory, achieving a more harmonic lip line that was well matched to the incisal curve (Figs 9 and 10).

After 3 years later, the patient asked for removal of the residua gingival pigmentation at both arches. The same surgical procedure was used to completely remove the remaining brownish gingival areas as previously described (Figs 11 and 12).

At the 3-year follow-up, no major modifications were detected either at the level of the restorations or of the gingival color (Figs 13 and 14).

### Discussion

Dark gums often represent a major esthetic problem, especially in the presence of a high smile line. A Gingival Pigmentation Index (GPI) has been proposed to define the level of pigmentation.[17] In this case report, the scalpel blade technique was used to remove the melanin pigmentations in a young adult patient, exhibiting an unappealing GPI.

The elimination of gingival pigmentations has been reported in the scientific literature as a viable option according to different procedures, although evidence-based data regarding a unique, more efficient clinical approach is not well recognized.[6,10,12,22] The choice of the most appropriate technique should take into account several factors, including patient compliance and pain tolerance, the physiopathologic situation, operator skill, and predictability over time.[14,15]

The use of diamond burs has been widely reported in the literature.[14,15,23] Under local anesthesia, de-epithelization of heavily pigmented gingival areas can be obtained through abrasion using flame-shaped or large round diamond burs with high-speed handpieces under saline irrigation. However, increased postoperative pain was noticed in some cases.[17]

A less invasive method for gingival depigmentation is based on the use of phenol 90% and alcohol 95%.[14,15,23] This technique was reported to be quite

simple and effective, although phenol should be used carefully to avoid tissue damage. Phenol penetrates the subepithelial connective tissue and produces necrosis or apoptosis of melanocytes; this process may be accompanied by a certain degree of inflammation. The procedure can be repeated until satisfactory depigmentation is achieved.[14,15]

Cryosurgery, based on local application of liquid nitrogen (-190°C) or other cryogen substances (eg, tetrafluoroethane) by means of cotton swabs or specific devices, can be effective in successful elimination of melanin pigmentations. The rapid freezing leads to destruction of the superficial layers and calls for particular care to avoid damage to the deeper tissues.[13,14,17,18] In a recent systematic review, cryosurgery exhibited optimal predictability for gingival depigmentation among the examined procedures.[14]

Electro- and radiosurgery require a higher level of expertise compared with the previous methods. Light, brushing strokes should be used when moving the electrode, which should be kept in motion throughout the procedure because its prolonged application could cause heat accumulation and subsequent tissue damage.[14,15,24]

Laser diode at 810 nm in pulse mode also was reported to be effective in gingival depigmentation.[13,15,21,22] Similar to electrosurgery, it was recommended to move the laser tip as a brush to prevent tissue heating. A one-step laser treatment is usually sufficient to remove pigmentation, achieving

tissue sterilization and efficient coagulation;[25] nonetheless, such an approach was reported to be highly dependent on operator skill because of the absence of tactile feedback while using lasers.[13] A delayed inflammatory reaction may occur, inducing mild postoperative discomfort lasting up to 1 to 2 weeks; furthermore, re-epithelialization can be delayed as compared with conventional surgery.[13,25]

Carbon dioxide (CO<sub>2</sub>) and erbium:Yag (Er:Yag) lasers have also been proposed for the treatment of gingival pigmentation. Although both techniques proved to be safe and effective in achieving gingival depigmentation with optimal esthetic results, when pain and wound healing were assessed Er:Yag laser outscored CO<sub>2</sub>.[26,29] Consequently, Er:Yag laser seemed to be the laser of choice for the treatment of gingival hyperpigmentation.[29]

To date, there is no unequivocal evidence for the best procedure to achieve gingival depigmentation, although the scalpel blade technique seems to be a simple and effective surgical approach with a low level of morbidity.[13,15,18,21,24,25] Further randomized controlled clinical trials would be useful to evaluate the effectiveness of the reported techniques in the long term.

As to the high smile line, the dental treatment was integrated with an esthetic medical approach: selective injection of botulinum toxin to limit overcontraction

of the patient's upper lip elevator muscles.[30,31] In the last decade, the use of botulinum toxin was extended to the treatment of some muscle-related disorders in the orofacial area, such as temporomandibular disorders, occlusal parafunctions, and masseter hypertrophy, and esthetic defects such as radial lip lines, deep nasolabial folds, and high smile line.[31-33]

To date, long-term data about the clinical effectiveness of botulinum toxin in the treatment of high smile lines is missing and only a few clinical reports are available in the literature.[30,31,34] The use of botulinum toxin should be carefully planned by trained and well-experienced operators. Although high levels of patient acceptance and satisfaction were reported,[30,31,34] the efficacy of botulinum toxin in reducing muscle overcontraction is subject-dependent and limited over time (to approximately 6 months). Consequently, this approach cannot be considered predictable in the long term and could necessitate re-entry.[30,31] However, such an approach is minimally invasive and without the risk of major complications compared with aggressive surgical treatments such as LeFort I osteotomy.[30,31,34]

### Conclusions

The described minimally invasive multidisciplinary approach based on surgical gingival depigmentation, adhesive ceramic veneers, and selective botulinum toxin injection corrected the undesired high smile line and achieved patient satisfaction. The scalpel technique proved to be safe and effective for gingival

depigmentation. The use of botulinum toxin can be useful to temporarily reduce high smile lines, but it cannot be considered a long-term solution. Periodic follow-up and treatment re-entries could be necessary over time. Further controlled clinical studies would be advisable to evaluate the predictability and maintainability of such esthetic treatments in the long term.



Fig 1 Preoperative extraoral view.



**Fig 3** Healing after surgical removal of the front gingival pigmentations.



Fig 2 Preoperative intraoral view.



Fig 4 Tooth preparations for ceramic veneers on the maxillary central incisors.



Fig 5 View of the veneers on the master cast.



Fig 6 The ceramic veneers 2 weeks after cementation.



Fig 7 The ceramic veneers at the 2-month follow-up.



Fig 8 Selective injection of botulinum toxin.



Fig 9 Follow-up 2 weeks after botulinum toxin injection (front view).



Fig 10 Follow-up 2 weeks after botulinum toxin injection (lateral view).

Fig 11 (left) Surgical removal of the lateral gingival pigmentations.Fig 12 (right) Detail of the scalpel technique.





Fig 13 Intraoral postoperative view at the 3-year follow-up.





**Fig 14** Extraoral postoperative view at the 3-year follow-up.

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# 4.1.2.1.5 "Digitally Oriented Materials": Focus on Lithium Disilicate Ceramics

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### Introduction

In the last decade, the development of new technologies has moved in parallel with a rapid evolution of restorative materials on the rails of Digital Dentistry, opening new horizons in the field of Prosthodontics. The implementation in the daily practice of the most advanced technologies, like CAD/CAM, lasersintering/melting, and 3D-printing, has got a synergic impulse from the enhanced mechanical and manufacturing properties of the new generation of dental materials: high strength ceramics, hybrid composites and technopolymers, high precision alloys, and so forth. Among these, metal-free ceramics offer unchallenged advantages like high esthetic potential, astounding optical characteristics, reliable mechanical properties, excellent consistency in terms of precision and accuracy due to the manufacturing technologies, lower costs, and more convenient production timing. In particular, lithium disilicate in the last years has gained maximum popularity in the dental scientific community, offering undeniable advantages.

### **Physical-Mechanical Properties and Fabrication Techniques**

Lithium disilicate (SiO2-Li2O) was introduced in the field of glass ceramics in

1998 as a core material, obtained by heat-pressing ingots (Empress 2, Ivoclar Vivadent, Lichtenstein), with a procedure similar to the lost-wax technique used for dental alloys (lithium disilicate heat extrusion at 920°), showing an optimal distribution of the elongated, small, needle-shaped crystals in a glassy matrix with a low number and small dimensions of pores [1]; the core is eventually veneered with fluorapatite-based ceramics, showing noticeable translucency and, at the same time, higher flexural strength (350 MPa) compared to older glass ceramics like the leucite-based ones [2, 3]. Such a material has been discontinued since 2009, replaced in the market by an upgraded typology of lithium disilicate, IPS e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein), in which both the optical and mechanical properties have been enhanced by introducing technical improvements in the production processes [4]. The crystals are smaller and more uniformly distributed; at the same time, this new, more versatile material has introduced the possibility of producing anatomically shaped, monolithic restorations, with no veneering ceramic, just colored on the surface; this innovative indication has become more and more popular in the last years, highly reducing technical complications like chippings and fractures, mainly used for restorations in the posterior areas, where such failures have been shown to be more frequent [5-11]. In order to accommodate the material to the needs of chairside CAD/CAM production processes, another technique has been introduced, based on the use of partially, precrystallized blocks (IPS e.max CAD, Ivoclar Vivadent), containing both 40% lithium metasilicate (Li2SiO3)

crystals and lithium disilicate (Li2Si2O5) crystal nuclei; it is available in different shades and degrees of translucency, depending on the size and density of crystals. In the initial condition, such machineable, bluish blocks show moderate hardness and strength (around 130 MPa); consequently, they are easier to mill, reducing wear of the machining devices at the same time, with evident advantages during chairside procedures [12]. After milling, heat treatment (840–850° for 10 min) determines full crystallization of the material: lithium metasilicates tend to evolve to form lithium disilicates (70%) [13], increasing the flexure strength up to MPa [14] with a fracture toughness of 2.5 MPa $\cdot$  [15]. Compared to the e.max CAD, hot-pressed lithium disilicate exhibits better mechanical properties, like higher flexure strength (440 MPa) and fracture toughness (2.75 MPa $\cdot$  - IPS e.max Press, Ivoclar Vivadent) [16].

The fabrication processes and machinability affect the restorative quality of monolithic lithium disilicate glass ceramics. A recent investigation analyzed the diamond tool wear, chip control, machining forces, and surface integrity of lithium disilicate after occlusal adjustments. Minimum bur wear but significant chip accumulation was evidenced; furthermore, machining forces were significantly higher than with other glass ceramics. Although the final surface roughness of lithium disilicate was comparable to other glass ceramics, occlusal adjustment caused intergranular and transgranular microcracks, resulting in shear-induced plastic deformations and penetration-induced brittle fractures;
such behavior is distinctive of lithium disilicate and very uncommon in other glass ceramics. Consequently, lithium disilicate should be considered the most difficult to machine among glass ceramics for intraoral adjustments [17]. Moreover, thermal processing can influence crystallization kinetics, crystalline microstructure and strength of lithium disilicate restorations. Particularly, extended temperature range (820–840°C versus 750–840°C) and protracted holding time (14 min versus 7 min) produced significantly higher elastic-modulus and hardness properties but showed flexural strength and fracture toughness properties similar to controls (i.e., 750–840°C for 7 min). Rapid growth of lithium disilicates happened when the maximum formation of lithium metasilicates had ended [13].

Recently, innovative fabrication techniques have been proposed to improve the microstructure of lithium disilicate ceramics. Particularly, spark plasma sintering (SPS) was developed specifically for CAD-CAM dental materials. This fabrication process allowed refining the microstructure of lithium disilicate; its densification resulted in textured and fine nanocrystalline microstructures with major lithium disilicate/lithium metasilicate phases and minor lithium orthophosphate and cristobalite/quartz phases [18].

#### **Mechanical Testing and Fracture Resistance**

Due to its intrinsic brittle behavior, lithium disilicate suffers from fatigue failure

during clinical service. Microcracks usually initiate in load bearing and/or stress concentration areas, eventually fusing under dynamic loads and creating major flaws that could weaken the lithium disilicate structure; when the ultimate mechanical strength is overcome, catastrophic failures occur [19–22].

Several laboratory studies investigated the fatigue resistance of lithium disilicate single crowns (SCs) and fixed dental prostheses (FDPs) to evaluate experimental designs and testing parameters [20–24]. Different laboratory variables were proved to influence the fatigue resistance of lithium disilicate restorations, such as magnitude of load, number of cycles, abutment and antagonist material, wet environment, and thermocycling; conversely, chewing frequency, lateral movements, and aging technique were considered not influential factors [23]. Single load to fracture after fatigue tests (i.e., combination of dynamic and static loading until fracture) reported highly variable ultimate strength values for this material: from 980.8 N to 4173 N for monolithic SCs and from 390 N to 1713 N for posterior FDPs [23, 24]. Significant comparisons between data were not possible because of the heterogeneity of research designs and testing modalities [24].

Fairly consistent agreement between in vitro and in vivo results was reported. As to SCs, after 2 years of simulated or real service, 100% survival rates were noticed in both laboratory [25] and clinical investigations [26]; in in vitro studies 100% survival rate was reported after 5 years of simulated function as well [20, 27] while the percentage changed to 97.8% in in vivo clinical investigations [26]. Differently, as regards FDPs, the cumulative survival rates at 5 years ranged from 75% to 100% in vitro [28, 29] while the equivalent clinical rate was 78.1% [26]; long-term laboratory investigations simulating more than 10 years of service showed 70% survival rate [30], comparable to the in vivo cumulative survival rate of 70.9% after 10 years of function [26]. The sound level of agreement between in vitro and in vivo data confirmed that laboratory investigations could represent a good simulation of the clinical scenario; nonetheless, this conclusion has to be considered only indicative, since the amount of data is not large enough to indicate consolidated clinical guidelines [24].

A recent systematic review showed significant heterogeneity leading to data inconsistency, because of different study setups and testing parameters. The lack of testing standardization made it almost impossible to perform consistent comparisons between laboratory studies. Consequently, to date, indicative and comparable data about dynamic mechanical testing of lithium disilicate restorations remain still controversial; further investigations with specific standardization criteria are needed [24].

According to in vitro results of dynamic loading, CAD-CAM lithium disilicate

SCs should have a thickness of at least 1.5 mm to withstand occlusal loads in posterior areas [22]. Being a filled glass-ceramic, lithium disilicate's final performance as a dental material is strongly related to the type of adhesive cement and accuracy of procedure [31]. To achieve the highest microtensile bond strength ( $\mu$ -TBS) values and best clinical performances, the restorations have to be adhesively luted to the substrates [32, 33]. CAD-CAM monolithic posterior SCs made of lithium disilicate and luted with self-adhesive resin cements showed significantly higher fatigue resistance than feldspathic ceramic restorations. Particularly, lithium disilicate SCs effectively bore the physiological range of masticatory loads, mainly showing repairable fractures. Catastrophic failures were noticed only after load-to-failure tests up to 4500 N [33, 34].

As to implant-supported restorations, although this material showed the highest ultimate strength when compared to feldspathic ceramic and resin nanoceramic onto implant titanium abutments in vitro, no accordance was found between the initial and maximum fracture resistance values of lithium disilicate after chewing simulation with thermocycling simulating 5 years of clinical service [35].

Furthermore, CAD-CAM monolithic lithium disilicate SCs showed an optimum in vitro stiffness and strength values when cemented onto both prefabricated titanium abutments and customized zirconia abutments [36].

# Machinability, Wear Mechanism, and Behavior

Friction and wear effects of lithium disilicate on the opposing natural tooth enamel have been also investigated, with and without fluorapatite coating, showing that they were less severe in unveneered specimens [37]. The initial surface roughness did not influence the final wear but the topography of the wear pattern affected the corresponding wear loss, since a smoother final wear aspect was associated with lower wear. Moreover, superficial wear of lithium disilicate was reported to be sensitive to environmental pH, showing higher friction and wear behavior in basic pH conditions; this was due to the fact that wettability, surface charge, and dissolution trend of lithium disilicate are pHdependent. The presence of fluorapatite veneering resulted in increased wear of both lithium disilicate crowns and opposing natural teeth; therefore, veneering of the occlusal surface should be avoided.

These results are in agreement with another recent in vitro investigation reporting that zirconia showed less wear than lithium disilicate; in any case, the latter showed occlusal wear equivalent to sound enamel. Enamel wear was reduced after ceramic surface polishing and this supports that this procedure is advisable after performing occlusal adjustments of both lithium disilicate and zirconia restorations. Veneering porcelain significantly increased enamel abrasion; consequently, the use of monolithic zirconia and lithium disilicate should be preferred in areas of strong occlusal contact, in order to limit enamel damage of the opposing teeth over time [38].

After friction against dental enamel, lithium disilicate and monolithic zirconia specimens did not become as rough as feldspathic ceramics. Particularly, when comparing wear effects onto rough, smooth, and glazed surface finishing, eventually rough lithium disilicate became significantly smoother than fine feldspathic porcelain [39].

However, when compared to type III gold, lithium disilicate was more abrasive against human enamel. Enamel opposing lithium disilicate in vitro showed cracks, plow furrows, and surface loss typical of abrasive wear mechanism, resulting in worse wear resistance and friction coefficient than in the presence of antagonist gold [40].

Opposing steatite in chewing simulations, monolithic lithium disilicate yielded higher antagonistic wear and worse wear behavior than monolithic translucent and shaded zirconia, but about half as high as the enamel reference (274.14  $\mu$ m); particularly, more severe wear patterns on both ceramics and opponents were observed after grinding and glazing [41].

Initial surface finishing and occlusal loads significantly affected the surface roughness, friction, and wear mechanisms of lithium disilicate: as the load increased, surface roughness became more severe and friction coefficient and wear volumes increased in turn. The abrasive wear process can be divided into 2 typologies: 2-body and 3-body abrasive wear. Particularly, in 2-body abrasion wear is caused by hard protuberances on one surface sliding over another while in 3-body abrasion particles are trapped between 2 surfaces but are free to roll and slide. In the presence of smooth lithium disilicate surfaces, 2-body abrasion was dominant while, in case of rough surfaces, 3-body abrasive wear was more significative. Worn lithium disilicate surfaces demonstrated higher sensitivity to delaminations, plastic deformations, and brittle fractures [42].

Two-body wear of lithium disilicate ceramic was found to be comparable to that of human enamel. Furthermore, abrasive toothbrushing significantly reduced gloss and increased roughness of all materials except zirconia [43]. When evaluating mechanical and optical properties, CAD-CAM lithium disilicate glass-ceramic (IPS e.max CAD) demonstrated the most favourable discoloration rate and the lowest 2-body wear on the material side when compared to CAD-CAM composites, hybrid materials, and leucite ceramic; in this study, the wear rate was analyzed in a chewing simulator using human teeth as antagonists [44].

Similarly to other glass ceramics, lithium disilicate can be intraorally repaired in

case of chipping. In vitro results using resin composites as restorative materials demonstrated that lithium disilicate can be effectively repaired with hydrofluoric acid etching followed by silanization and adhesive bonding [7, 8, 45].

#### **Impression Techniques and Accuracy of Fit**

Both conventional and digital impression techniques allow for the fabrication of lithium disilicate restorations but the results in terms of marginal accuracy are still controversial [46–51].

An in vitro study reported similar marginal accuracy between conventional and digital impression techniques (112.3  $\pm$  35.3  $\mu$ m and 89.8  $\pm$  25.4  $\mu$ m, resp.) and no statistically significant differences were noticed among the different approaches [51]. Differently, the results of a recent in vitro study suggested that pressed and milled lithium disilicate SCs from digital impressions had a better internal fit to the abutment tooth than pressed SCs from polyvinylsiloxane impressions in terms of total volume of internal space, average thickness of internal space, and percentage of internal space at or below 120 µm [50]. Similarly, another in vitro investigation proved that the fully digital workflow provided better margin fit than the conventional fabrication [48]. These results were not in agreement with other investigations demonstrating that the combination of polyvinylsiloxane impressions and Press fabrication techniques for lithium disilicate SCs produced the most accurate 2D and 3D marginal fits [46] and that the combination of digital impressions and pressed lithium disilicate SCs produced the least

accurate internal fit [49].

To date, in general, marginal and internal fit of lithium disilicate restorations is significantly influenced by the employed digital impression technique. Although almost all actual digital impression systems show accuracy values within the thresholds of clinical acceptability, significant fit discrepancies are still evident among different digital systems [52].

In vitro microscopical analyses demonstrated that CAD-CAM lithium disilicate SCs had significantly smaller marginal gaps than CAD-CAM anatomic contour zirconia restorations. As to the absolute marginal discrepancy, lithium disilicate SCs showed some overextended margins. Both finish line geometry and fabrication systems significantly influenced the absolute marginal discrepancy [53].

In vivo results by means of the replica technique showed that CAD-CAM lithium disilicate SCs had significantly larger internal axial and occlusal gaps than porcelain-fused-to-metal (PFM) SCs; conversely, marginal gaps were not significantly different. Nevertheless, both PFM and lithium disilicate SCs showed clinically acceptable marginal fit [54]. As regards the restoration adaptation (i.e., marginal and internal fit) of the different manufacturing

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techniques, evidence is growing that these parameters are more favourable with the hot-pressing technique than with the precrystallized, CAD/CAM milled blocks [46, 55, 56].

#### **Biocompatibility**

Biologic safety of dental ceramics is another main topic on which dental research has been focusing in the last years; such a property can be different even within the same class of materials. Lithium disilicate exhibited more severe in vitro cytotoxicity than dental alloys and composites and became more cytotoxic after polishing [57].

In vitro, human gingival fibroblasts cellular response may reflect variability in soft tissue reaction to different surface materials for prosthetic restorations. In a study by Tetè et al., polished zirconia showed a better integration in respect to the other materials [58]. Analysis on human epithelial tissue cultures, on the other side, demonstrated that lithium disilicate showed the best biocompatibility when compared to zirconia and cobalt-chromium alloys. Consequently, lithium disilicate can be considered a suitable material even for subgingival restorations directly contacting the sulcular epithelial tissues [59]. As to in vivo evidences, the presence of all-ceramic restorations did not induce inflammatory reactions in periodontally healthy patients; no differences between gingival reactions to lithium disilicate and zirconia restorations could be shown [60, 61].

#### **Clinical Indications and Outcomes**

For its outstanding optical properties, mechanical characteristics, ease of processing, and possibility of etching/adhesive bonding, ensuring a minimally invasive approach, lithium disilicate glass ceramics have rapidly become some of the most popular restorative materials in almost all the indications of fixed Prosthodontics [8].

Their primary use was addressed for single crowns (SCs). The first clinical studies were conducted on the early typology of lithium disilicate (IPS Empress, Ivoclar Vivadent) and reported quite promising short-term results for the veneered crowns [62, 63]; in particular, Marquardt and Strub, in their prospective clinical trial on both crowns and anterior FDPs, showed for the SCs a survival rate of 100% after 5 years of clinical service [63]. Gehrt et al. [6] analyzed the medium-long term clinical performance of 74 lithium disilicate full-coverage, anterior and posterior crowns after a service time of at least 5 years; all the frameworks, made with the hot-pressing technique from ceramic ingots (IPS e.max Press), were at least 0.8 mm thick and were eventually veneered with a fluorapatite ceramic. The survival rate was 97.4% after 5 years and 94.8% after 8 years of clinical service; among the technical complications, 3 crowns resulted affected by minor chipping. The study revealed that the survival rate was not influenced by cementation type (conventional versus adhesive) or by crown location (anterior versus posterior); on the other hand, in vitro researches have clearly demonstrated that lithium disilicate can bear high stress conditions, like in posterior crowns [64, 65]. Esquivel-Upshaw et al. [66] conducted a 3-year clinical study comparing the performance of veneered lithium disilicate (Empress 2), monolithic lithium disilicate (e-Max Press, glazed), and metal-ceramic crowns (IPS d.SIGN veneer); they observed similar, highly positive results, although a higher degree of surface roughening was detected in the veneered lithium disilicate-based crowns, compared to metalceramics, between years 2 and 3. This problem was probably due to degradation/water corrosion of glaze ceramic. Another retrospective, multicentric study on 860 lithium disilicate restorations, both tooth- and implant-supported, including full crowns, laminate veneers, and onlays, reported cumulative survival and success rates beyond 95% for an observational period ranging from 12 to 72 months [8]. The analyzed restorations were both bilayered and monolithic type. More recently, other retrospective studies, with longer observational times, have confirmed low failure rates and very favourable cumulative survival rates with lithium disilicate crowns [65, 67, 68]. Positive clinical outcomes of lithium disilicate reinforced glass ceramics have been confirmed by a recent systematic review [11], showing that 5-year survival rates of all-ceramic SCs made out of lithium disilicate or oxide ceramics (i.e., alumina and zirconia) were similar to the gold standard, metal-ceramic crowns. The widespread diffusion in the daily practice of full-anatomic, monolithic lithium

disilicate restorations, characterized by favourable mechanical properties, together with the possibility of manufacturing low thickness restorations adhesively bonded to the dental substrate, has introduced the use of inlays, onlays, and "tabletops" made of this material in the posterior sites, taking advantage of a minimally invasive approach and of a resistant, biocompatible ceramic (Figures 1–4). In that research, low fracture rates were reported: 0.91% for monolithic and 1.83% for bilayered single crowns (twice the rate of the monolithic); 4.55% for monolithic FDPs; 1.3% for monolithic; and 1.53% for bilayered veneers (Figures 5-9). Guess et al. [69] conducted a 7-year prospective "split-mouth" study on both pressed lithium disilicate (IPS e.max Press, Ivoclar Vivadent) and CAD/CAM leucite-reinforced glass-ceramic (ProCAD, Ivoclar Vivadent) partial-coverage restorations. The preparation was performed reducing the entire occlusal surface for a 2 mm thickness, creating a butt joint design at level of the nonsupporting cusps and a rounded shoulder for the supporting cusps. The authors reported high survival rates with both types of restorations, recommending them for a minimally aggressive treatment of extended lesions in posterior teeth. In a recent in vitro research, Sasse et al. [70] advised the need of a lithium disilicate minimum thickness of 0.7–1.0 mm when nonretentive, full-coverage adhesively retained occlusal veneers are used. As regards 3-unit FDPs, according to the manufacturer's recommendations, the use of lithium disilicate should be limited to the replacement of anterior teeth or premolars. Clinical data on this topic is quite controversial. The early,

short/medium-term studies, mainly conducted on Empress 2 bilayered lithium disilicate bridges, suggested a certain cautiousness for such an indication: Taskonak and Sertgöz [71] reported a 50% survival rate at 2 years; a prospective clinical trial by Marquardt and Strub showed a fracture rate of 30% after 5 years of clinical service [63]. Makarouna et al. [72], in a randomized controlled trial, after 6 years observed a survival rate of 63% for lithium disilicate FDPs, compared to a much more favourable 95% in the control group (metal-ceramic FDPs).

In a 10-year prospective study conducted by Solá-Ruiz et al. on Empress 2 FDPs, a survival rate of 71.4% was detected, the most frequent complications being postoperative sensitivity, recessions, and marginal discolorations [73]. The introduction of the monolithic, anatomically shaped lithium disilicate FDPs has recently made achieving more favourable outcomes possible.

Some in vitro studies [29, 74, 75] have pointed out that lithium disilicate monolithic crowns and FDPs, both CAD/CAM and hot-pressed, are more resistant to fatigue fracture compared to bilayered, hand veneered ones, showing higher fracture loads (1900 N), that are comparable to the metal-ceramic standard. The lack of the esthetic, weaker veneering material allows a thicker bulk of high strength disilicate; in any case, as regards FDPs, it has to be pointed out that their mechanical performance is multifactorial, being strongly related to many factors, like shape of the structure and size and radius of the connectors

among others.

In a long-term prospective study, Kern et al. [5] evaluated the clinical performance of 3-unit, monolithic lithium disilicate FDPs (IPS e.max Press, Ivoclar Vivadent). In this research, the bridges were used not only for the replacement of anterior teeth or premolars (as suggested) but also for missing molars. After 5 years, the survival and success rates were 100% and 91.1%, respectively; after 10 years, they were reduced to 87.9% and 69.8%. Considering that 10-year survival rates of 87.0 to 89.2% have been reported for the "reference" metal-ceramic FDPs by some systematic reviews [11, 76] and that the major, catastrophic failures occurred lately in FDPs replacing missed molars (beyond the manufacturer's recommendations), these evidences advise that the monolithic lithium disilicate can be regarded as a promising candidate to replace metal-ceramics for short-span freestanding bridges.

In the last years, in the light of the concepts of minimal invasivity, economy, and long-term durability, alternative treatment strategies for the anterior single tooth replacement have become more and more popular, taking advantage of the materials' high strength and of the possibility of a reliable adhesive bonding to dental substrates. In particular, cantilevered, all-ceramic, resin-bonded, fixed partial dentures (RBFPD) have been increasingly gaining approval from the dental community, offering a feasible alternative to implant therapy in many cases, particularly when indications for implant therapy are not present, due to general, anatomic, economic, or patient's compliance factors. In such cases, instead of a complete crown, a single veneer adhesively bonded to the lingual side of the support tooth can be used; a careful occlusal check is mandatory, in order to get a proper distribution of stress and a stress limitation on the cantilevered tooth, avoiding lateral and protrusive contacts on the pontic. Also, for this kind of restoration, clinical outcomes are highly encouraging, although data is quite limited to medium-term studies and case series [77–80].

In the last years, the chairside production workflow is gaining more and more interest in the prosthodontic realm, for the speed of delivery and cost reduction of SCs and inlays. The first clinical trials report encouraging results. In the study by Reich and Schierz, besides a survival rate of 96.3% after 4 years, a few biological complications (secondary caries below the crown margin, changing of sensibility perception) and technical complications (need of cervical composite filling) were observed [81].

Recently, Sulaiman et al. [82] have analyzed the clinical outcomes of different IPS e.max lithium disilicate prostheses (SCs, FDPs, veneers, inlays, and onlays), both in the bilayered and monolithic forms, in a 4-year retrospective study on a total of 21.340 restorations. In that research, low fracture rates were reported: 0.91% for monolithic and 1.83% for bilayered single crowns (twice the rate of the monolithic); 4.55% for monolithic FDPs; 1.3% for monolithic and 1.53% for

bilayered veneers; and 1.01% for monolithic inlays/onlays. Finally, in the last years, the use of lithium disilicate single crowns bonded onto CAD/CAM zirconia abutments has become increasingly widespread, taking advantage of the high strength and biocompatibility of zirconia, in contact with the peri-implant soft tissues, together with the prosthetic versatility and optical characteristics of lithium disilicate. In vitro studies have demonstrated that these prosthetic solutions exhibit high fracture loads [27, 83] and, at the same time, short-term clinical studies have shown fairly positive outcomes [84], also onto one-piece zirconia implants (Spies). Another clinical approach, also supported by favourable short-term outcomes, makes use of zirconia implant-supported full-arch frameworks ("implant bridges") on which monolithic lithium disilicate crowns are adhesively bonded [7, 85].

### Conclusions

It is a far from indisputable fact that all of the innovative solutions offered by lithium disilicate are widening the restorative scenario more and more; thanks to the excellent optical properties, the high mechanical resistance, the unique restorative versatility, and the different manufacturing techniques, it is no doubt one of the most promising dental materials in the realm of Digital Dentistry, although more light is still to be shed on some clinical and technical aspects.



Figure 1: Case 1 (Monolithic Lithium Disilicate Onlays). Maxillary posterior teeth in a 25-year-old female patient affected by severe food behavior disorder (bulimia). One year before the dental treatment, she was considered healed by a psychotherapist and declared recovered. The teeth were not prepared; only minimal smoothing of some sharp edges was performed.



Figure 2: Case 1 (Monolithic Lithium Disilicate Onlays). After conventional impressions, the casts were scanned by a 3-Shape D700 (3 Shape, Copenhagen, Denmark) digital scanner and analyzed by means of a Dental System 15.5.0 software (3 Shape) and the restorative finish lines were detected. Then, occlusal shape design and contacts were defined.



Figure 3: Case 1 (Monolithic Lithium Disilicate Onlays). The wax patterns of the posterior onlays were milled out of a wax disk (Cera SDD98A18RWC, Sintesi Sud, Avellino, Italy) using a Roland DWX-50 Dental Milling Machine (Whip Mix GmbH, Louisville, KY, USA) and then repositioned on the cast. After careful checking, the lithium disilicate heat pressed onlays (IPS e.max Press MT, Ivoclar Vivadent) were made and eventually polished.



Figure 4: Case 1 (Monolithic Lithium Disilicate Onlays). The onlays after adhesive cementation.



Figure 5: Case 2 (Bilayered Lithium Disilicate Veneer Replacement). A female patient asked for the replacement of 6 porcelain laminate veneers with discolored and fractured margins. After the study of the case, done with the aid of digital software programs, a crown lengthening procedure was performed.



Figure 6: Case 2 (Bilayered Lithium Disilicate Veneer Replacement). The old veneers were carefully removed under stereomicroscopic control; after the new supragingival preparations, an intraoral scanning device (3-Shape D700) was used to take digital impressions of both dental arches.



Figure 7: Case 2 (Bilayered Lithium Disilicate Veneer Replacement). The new smile design was cut away and inserted in the patient's physiognomic image. After designing the new veneers, they were pressed with lithium disilicate (IPS e.max Press MT) and veneered.



Figure 8: Case 2 (Bilayered Lithium Disilicate Veneer Replacement). The new veneers at the end of the treatment.



Figure 9: Case 2 (Bilayered Lithium Disilicate Veneer Replacement). The patient's smile.

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#### **4.1.2.2** Cristalline Ceramics

#### In-Ceram Spinell, Alumina, Zirconia

Infiltrated ceramics are made through a process called slip-casting, which involves the condensation of an aqueous porcelain slip on a refractory die. This fired porous core is later glass infiltrated, a process by which molten glass is drawn into the pores by capillary action at high temperatures. Materials processed in this way exhibit less porosity, fewer defects from processing, greater strength and higher toughness than conventional feldspathic porcelains.[2]

This glass-infiltrated core is later veneered with a feldspathic ceramic for final esthetics. These have excellent translucency and esthetic qualities, but have poor physical properties and require the high-strength core that the already-mentioned infiltrated ceramics can provide. The Vita In-Ceram slip-casting system makes use of three different materials to gain a good compromise between strength and esthics.

#### **In-Ceram Spinell**

Spinell (MgAl<sub>2</sub>O<sub>4</sub>) is a natural mineral that is normally found together with limestone and dolomite. It is of dental significance because of its extremely high melting point (2135°C) combined with its high strength. Spinell is also chemically inert and has low electrical and thermal conductivity but, most importantly, it has unique optical properties. It has moderate strength of about 350 MPa and good translucency.

It is more than twice as translucent as In-Ceram alumina due to the refractive index of its crystalline phase being close to that of glass. Glass infiltrating in a vacuum environment results in less porosity, ensuring this high level of translucency. Often, however, this level of translucency can be excessive and can lead to an overly glassy, low-value appearance.

# **In-Ceram Alumina**

Aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) is most widely known under the term corundum. As a result of the homogeneous framework structure made of ultrafine Al<sub>2</sub>O<sub>3</sub> particles, whose cavities are filled with a special glass, the degree of tensile bending strength is significantly higher than that of all other ceramic systems.[1] With a weight percentage of 10–20%, aluminum oxide is a component of feldspar, which is the starting material for metal–ceramic veneering materials. The ceramic materials for substructures of jacket crowns have been enriched by up to 60% by weight with aluminum oxide crystals with a grain size of 10–30 um to increase stability. Because of the large difference in the refraction index (feldspar n = 1.53; corundum n = 1.76), intense refraction of light occurs at the aluminum oxide crystals in the feldspar, which results in the opaque effect of such Al<sub>2</sub>O<sub>3</sub>-enriched ceramic materials. Therefore, they are only suitable for

fabrication of crown frames with subsequent veneering. In-Ceram alumina has a strength of around 500 MPa and poor translucency.

Synthetically produced corundum with a grain size of 2–5 um is used for In-Ceram alumina. In the solid phase, it is sintered at 1100°C, well below the melting point of 2040°C, and it is then infiltrated with dentine-coloured glass at 1120°C.

#### **In-Ceram Zirconia**

The zirconia system uses a mixture of zirconium oxide and aluminum oxide as a framework to achieve a marked increase in the flexural strength in the core framework. Aluminum oxide makes up about two-thirds of the crystalline structure as seen in the scanning electron micrograph to the right. The remaining crystalline structure consists of tetragonal zirconium oxide (round white particles). The proportion of glass phase amounts to approximately 20–25% of the total structure. This leads to the high strength as already seen in In-Ceram alumina. The increase however over alumina is due to the zirconium oxide particles that protect the structure against crack propagation. It has a very high strength of around 700 MPa and very poor translucency.

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#### 4.1.2.3 Polycrystalline ceramics: ZIRCONIA

Zirconium oxide–based materials, especially yttria-tetragonal zirconia polycrystals (Y-TZP), were recently introduced for prosthetic rehabilitations as a core material for single crowns, conventional and resin-bonded fixed partial dentures (FPDs) [1], and, in dental implantology, as abutments or implants [2]. The raw materials of the zirconia are the minerals zircon (ZrSiO4) and baddelyite ( $\beta$ -ZrO2), whose mines are located in South Africa, Australia and USA. Zirconia was discovery by the German chemist Martin Heinrich Klaproth in 1789. The term zirconium refers to the metal, while zirconia ceramic ("zirconia") refers to zirconia-dioxide-ceramic (ZrO2).

Zirconia takes up a peculiar place amongst oxide ceramics due to its excellent mechanical properties. This condition is due to the huge amount of the research that has been performed since the discovery of the transformation toughening capabilities of this material. The different stages of polymorph zirconia are temperature dependent: at ambient pressure, unalloyed zirconia can assume three crystallographic forms. At room temperature and upon heating up to 1170°C, the symmetry is monoclinic (P21/c). The structure is tetragonal (P42/nmc) between 1170 and 2370°C and cubic (Fm 3m) above 2370°C and up to the melting point [3] [4]. The transformation from the tetragonal (t) phase to the monoclinic (m) phase upon cooling is accompanied by a substantial increase in volume (~4.5%), sufficient to lead to catastrophic failure [5]. The ceramic

shows a hysteretic martensic t  $\rightarrow$  m transformation during heating and cooling. This transformation is reversible and begins at ~950 °C on cooling. Alloying pure zirconia with stabilizing oxides such as CaO,MgO,Y2O3 or CeO2 allows the retention of the tetragonal structure at room temperature and therefore the control of the stress-induced. Zirconia has a high temperature stability and melting point (2680°C), high hardness (1200-1350 HVN), high thermal expansion (>10 x 10-6 1/K), low thermal conductivity (<1 W/mK) and a good thermo-shock resistance ( $\Delta$ T=400-500°C).

# **Transformation toughening**

However, the metastability of tetragonal zirconia could increase the susceptibility to aging because some stress-generating surface treatments such as grinding or sandblasting can trigger the t $\rightarrow$ m transformation with volume increase and formation of compressive stresses on the surface, thereby modifying the phase integrity though increasing the flexural strength [7]. The increase of volume determines a local stop of the crack propagation. This process is called "transformation toughening", with the resistance against crack propagation that increases with the length of the crack [8].

Only three types of zirconia systems are used in dentistry although currently there are many systems of zirconia available on the market. Thee first is yttrium cation-doped tetragonal zirconia polycrystals (3Y-TZP), the second is

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magnesium cation-doped partially stabilized zirconia (Mg-PSZ) and finally the zirconia-toughened alumina (ZTA). The partly stabilized zirconia (PSZ) is stabilized with magnesia and in addition to the cubic phase, a transformable tetragonal phase is available. Its microstructure at room temperature is mostly cubic with portions of monoclinic and tetragonal phases. While the Tetragonal Zirconia Polycrystals (TZP) have a ultra-fine, nanometre-scaled structure that allows the transformation during cooling from the cubic to the tetragonal phase, but not to the monoclinic phase. [10]

# Low temperature degradation (LTD)

One of the aging process is well-described in the literature and is called "low temperature degradation" (LTD) of the zirconia. This is a phenomenon due to the presence of water [10] [11] [12]. The consequences of this aging process are determine the degradation of the zirconia surface with the grain pullout and subsequently microcracking of the structure. This phenomena represent an strength degradation.

Three are the hypotheses of the low temperature degradation. The first speculate that there is the diffusion of water species (here OH-) into the lattice via oxygen vacancies and (b) resulting change of lattice parameters [13].

The second hypothesis claim that H2O reacts with Y2O3 to form clusters rich in Y(OH)3 [14].

The last hypothesis sustain that the water vapor attacks the Zr-O bond, breaking it and leading to a stress accumulation due to movement of -OH. This in turn generates lattice defects acting as nucleating agents for the subsequent T-M transformation [15]

#### **Mechanical Properties**

Zirconia-based materials have higher strength, fracture toughness [16] in comparison to the feldspathic ceramics [17]. The failure mechanism of the zirconia, like others ceramic materials, is due to sub-critical crack-growth. The metal-oxide bonds which were destroyed when the stress is present near the tip of the initial crack with a water-assisted mechanism [18]. Cyclic loading during the biting or chewing simulation can slowly cause the degradation of the toughening mechanisms [19] determine the fracture of the zirconia framework because a toughened material could be more susceptible to rupture. The cracks can originate inside of the zirconia framework or close to the ceramic veneer interface and propagate to the interface [20].

## Configuration

The zirconia in dentistry is usually milled in pre-sintered stage. This configuration is a soft, chalk-like stage that is called "green" stage. During the sintering process, the material shrinks and reduces the volume shrinkage of about 20-25%. It's very important to know the exact volume shrinkage

information for the individual zirconia blank blocks in order to optimize the fitting of the restoration. The zirconia is called hipped (hot iso-static pressed) when the material is industrially sintered, and then is CAD-milled at its final high strength. Hipped zirconia has a constant grading and thus a more homogeneous quality. As expected, milling time and wear of the milling tools is higher in comparison to the pre-sintered variants. The zirconia for dental applications, zirconia is stabilized at room temperature with the addition of 3 mol% yttria. This configurations reach high strength (800-1200 MPa) good fracture toughness (6-15 MPa x m1/2).

## **Fabrication process**

The fabrication of the framework or the monolithic zirconia requires rapid prototyping procedures such as milling with a CAD/CAM [21]. The different manufacturers use milling-machines directly in the dental laboratories or centralized production center. The process starts with optically digitizing the clinical abutment with an intraoral camera or with 3D-scanning devices using gypsum models or wax models. Afterwards the substructure is designed on the computer (CAD) and the core is anatomically shaped to support the ceramic veneering material. In the last few years, the use of zirconia for monolithic restoration has increased. This approach is now possible because the burs can mill the anatomic occlusal design with fissures.

The properties of the zirconia substructures could depend by the manufacturing

process. The use of insufficient preparations, or frameworks with imprecise dimensions / thickness could reduce the integrity of zirconia restorations. The design of the core, when it is a simple cap or an occlusal supporting design, has a strong influence on the lifetime of the veneering [22-23]

The zirconia framework has to support completely the veneering ceramic material in order to avoid chipping or delamination of the aesthetic porcelain

The dimensions and design of FPDs, especially in the connector areas in posterior and in anterior fixed partial dentures, is important to increase the clinical life of the all-ceramic restoration. The volume of the connector area in anterior and posterior FPDs must be minimum of  $3x3mm^2$  [24-25].

For the quality of the marginal fit, besides the well-known clinical parameters, the CAD/CAM fabrication process may play a decisive role. Different milling devices, milling strategies, and software capabilities may contribute to the results even more than the different types of ceramic materials. Based on the assumption that the clinically acceptable marginal fit extends to 200µm, CAD/CAM fabricated restorations with values between 64-83µm and 245µm [26-28] are in most cases good to acceptable. The results for the marginal fit are in the range of porcelain- fused-to-metal (PFM) restorations or pressed-ceramics and vary widely depending on the abilities of the dental technician.

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# 4.1.2.3.1 Hydrothermal degradation of monolithic zirconia: guidelines for finishing treatments.

Sorrentino R., Leone R., Camposilvan E., Chevalier J., Ferrari M., Zarone F. Dental Materials, 2016.

## Introduction

Polycrystalline zirconium dioxide (zirconia) became more and more popular in prosthodontics due to high biocompatibility, excellent mechanical properties, natural-looking esthetics and wear rate comparable to that of human enamel.

Pure zirconia is chemically an oxide but technologically it can be considered a ceramic material. At ambient pressure, it is polymorphic and allotropic, presenting 3 crystallographic phases at different temperatures: monoclinic (from room temperature to 1170°C), tetragonal (from 1170°C to 2370°C) and cubic (from 2370°C to 2680°C, the melting point of the material). Upon cooling, a spontaneous transformation from the tetragonal shape to the more stable monoclinic phase occurs with 4-5% volume increase of zirconia crystals and consequent compressive stresses within the material. This phenomenon, called "phase transformation toughening (PTT)", could be useful to improve fracture toughness and limit crack propagation under clinical service.

The manufacturers' choice to use Yttria-stabilized Tetragonal Zirconia Polycrystals (Y-TZP) was mainly aimed at matching its optical and mechanical properties, particularly to take advantage of the inherent PTT mechanism of the

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material keeping it in the tetragonal or cubic shape at room temperature in a thermodynamically metastable state by means of dopant oxides (just like yttria or ceria) used as crystal stabilizers.

Nonetheless, both manufacturing processes as well as clinical procedures could cause the removal of stabilizing oxides, resulting in surface degradation and/or onset of microcracks. Moreover, zirconia is susceptible to a spontaneous, slow PTT toward the monoclinic shape at room temperature in absence of any mechanical stress; this phenomenon, called "Low Temperature Degradation (LTD)" causes the aging of zirconia with a decrease of its physical properties and a potential risk of spontaneous, catastrophic failures. Since LTD can be accelerated by mechanical stress and wetness, it can be considered a kind of hydrothermal degradation. Furthermore,

Computer Aided Design-Computer Aided Manufacturing (CAD-CAM) monolithic zirconia was developed to avoid the occurrence of fracture of the veneering ceramic (i.e. chipping).

The present in vitro study aimed at investigating the influence of surface polishing, regeneration and aging on the phase transformation of monolithic zirconia by means of XRD analysis.

The null hypothesis stated that there was no association between surface polishing, regeneration procedures and aging on the monoclinic phase content of

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3Y-TZP.

### **Materials and Methods**

Thirty CAD-CAM monolithic zirconia copings were used in the study. The copings were designed as circular truncated cones according to the average dimensions of maxillary premolars (height: 7 mm, diameter: 8 mm); they were milled with a thickness of 0.5 mm flat occlusal surfaces and a total occlusal convergence of 10°.

The specimens were divided into 3 experimental groups of 10 copings each according to the constitutive 3Y-TZP material and manufacturing system, as follows:

- Group 1 (n=10): Nobel Procera Zirconia (Nobel Biocare Management AG, Zurich-Flughafen, Switzerland);
- Group 2 (n=10): Lava Classic (3M ESPE, Seefeld, Germany);

- Group 3 (n=10): Lava Plus (3M ESPE).

The occlusal surface of each coping was ideally divided in half and 2 different test areas, one per half, were univocally identified by means of an arbitrary coordinate reference system used in all the subsequent experimental analyses; similarly, a control area was identified on the axial wall of each specimen. The copings were spatially oriented by means of aluminium holders provided with a notch, used to reposition the samples in both the SEM and the XRD analyzer. Before performing the experimental analyses, the test and control areas of all the samples were subjected to light microscopy inspections, in order to record the morphological surface characteristics and identify any dishomogeneous area due to the CAM fabrication procedures. Moreover, an environmental SEM was used to analyze the microstructure and grain size of each specimen. Finally, each coping was subjected to XRD analysis, in order to assess the monoclinic phase content (vol%).

Then, the occlusal surfaces of all the specimens were ground by an experienced prosthodontist under stereomicroscope (10x) with medium grit diamond burs mounted on a high speed handpiece under constant water cooling; grinding was performed with minimal hand pressure for 5 seconds. Subsequently, half of the occlusal surface of each coping was polished with coarse, medium and fine grit diamond impregnated rubber polishers according to the manufacturer's instructions; the polishing was considered completed when high luster was achieved.

All the samples were thoroughly cleaned in an ultrasonic bath with ethanol for 10 minutes and dried in a vacuum desiccator.

At this time, each experimental group was divided into 2 subgroups and half of the specimens were subjected to a regeneration process, as follows:

- Subgroup 1A (n=5): Nobel Procera Zirconia regenerated;
- Subgroup 1B (n=5): Nobel Procera Zirconia not regenerated;
- Subgroup 2A (n=5): Lava Classic regenerated;
- Subgroup 2B (n=5): Lava Classic not regenerated;

- Subgroup 3A (n=5): Lava Plus regenerated;
- Subgroup 3B (n=5): Lava Plus not regenerated.

The regeneration firing at 1200°C for 1h was intended to restore the tetragonal phase of zirconia before artificial aging; the not regenerated and the regenerated samples were used as tests and controls respectively.

At this time (T0), the test (i.e. ground vs polished) and control areas of all the copings were subjected to XRD analyses to quantify the monoclinic phase content (vol%) as previously described.

Then, the samples underwent accelerated artificial aging in autoclave at 134°C for 2h (T1), 6h (T2), 18h (T3) and 54h (T4) to roughly simulate a reasonable lifespan of dental restorations. After each cycle of hydrothermal degradation, the test (i.e. ground vs polished) and control areas of all the copings were subjected to light microscopy and XRD analyses as previously described. The optical inspections allowed for a qualitative, morphological classification of the specimens on the basis of the presence or absence of detectable surface degradation. Differently, the XRD analyses allowed for a quantitative evaluation of tetragonal-to-monoclinic phase transformation of zirconia (vol%) after accelerated artificial aging.

The recorded data were statistically analyzed with a dedicated software (IBM SPSS Statistics 24, IBM, Armonk, NY, USA). The Kolmogorov-Smirnov test was used to verify the normality of data distribution and the Levene's test was performed to analyze group variances. The two-way ANOVA and the Tukey's

post-hoc test for multiple comparisons were applied to strength data from the experimental groups in order to assess the significance of the effect of the factors "material", "polishing" and "regeneration", as well as of the interactions between these factors. In all the analyses, the level of significance was set at p<0.05.



Fig. 1. SEM images showing the microstructure of Group1



Fig. 2. SEM images showing the microstructure of Group2



Fig. 3. SEM images showing the microstructure of Group 3



Fig. 4. Sample analyzed surfaces



Fig. 5. X-Ray diffraction

# Results

SAMPLE DESCRIPTION		ARTIFICIAL AGING TIME								
		Т0	T1 (2h)	T2 (6h)	T3 (18h)	T4 (54h)				
DROCERA	Regenerated	0	0	0	0	0				
ZIRCONIA	Not regenerated	0	0	0	0	20				
LAVA CLASSIC	Regenerated	0	0	0	0	0				
	Not regenerated	0	0	0	0	0				
LAVA PLUS	Regenerated	0	0	0	0	0				
	Not regenerated	0	0	0	0	0				

Table 1. Percentage of samples showing surface degradation

SAMPLE DESCRIPTION			ARTIFICIAL AGING TIME							
			Т0	T1 (2h)	T2 (6h)	T3 (18h)	T4 (54h)			
	Querrad	Regenereted	0	0	2.65	13.91	38.09			
	Ground	Not regenereted	0	8.33	13.51	15.8	33.62			
PROCERA		Regenereted	0	0	4.74	14.3	35.35			
ZIRCONIA	Polisned	Not regenereted	0	3.74	4.23	9.61	21.03			
	0 a set set	Regenereted	0	0.69	11.37	22.31	41.15			
	Control	Not regenereted	0	9.63	21.38	33.51	42.1			
	Ground	Regenereted	0	0	0	7.44	28.49			
		Not regenereted	0	5.58	10.68	19.22	35.56			
LAVA	Polished	Regenereted	0	0	1.65	12.45	31.72			
CLASSIC		Not regenereted	0	1.12	4.65	12.18	24.12			
	Control	Regenereted	0	0	12.69	26.44	38.89			
		Not regenereted	0	9.38	17.78	29.93	41.55			
		· · · · ·								
LAVA PLUS	Ground	Regenereted	0	0	0	0	8.57			
		Not regenereted	0	3.52	5.53	13.4	29.95			
		Regenereted	0	0	0	1.05	13.01			
	rolisned	Not regenereted	0	0	0.86	6.51	17.1			
	Control	Regenereted	0	0	0	3.16	30.02			
	Control	Not regenereted	0	0	1.72	18.6	41.91			

Table 2.vol% tetragonal-to-monoclinic phase transformation of zirconia samples



Fig. 6. Evolution of the monoclinic content (vol%) measured by XRD after artificial aging on Control surfaces



Fig. 7. Evolution of the monoclinic content (vol%) measured by XRD after artificial aging on Ground surfaces



Fig. 8. Evolution of the monoclinic content (vol%) measured by XRD after artificial aging on Polish surfaces



Fig. 9. Differential interference contrast illumination (DIC) microscopy (Nomarsky contrast) images showing the distribution of degradation and its relation with the presence of scratches and polished areas after artificial aging

## Discussion

The literature indicate that procedures to adjust the zirconia surface, such as grinding or polishing, can induce superficial modifications, damage, and phase transformation from the tetragonal (t) to the monoclinic (m) phase (Karakoca and Yilmaz, 2009, Maerten et al., 2013 and Pereira et al., 2016a).

# Conclusion

Commercial zirconia with small grain size can be recommended to hinder the hydrothermal degradation, as well as a regeneration process after the last superficial treatment. Control, ground and polished surfaces showed similar phase characteristics and behavior. 4.1.2.3.2 In vitro aging and mechanical properties of translucent monolithic zirconia.

Leone R., Sorrentino R., Camposilvan E., Chevalier J., Zarone F., Ferrari M. Dental Materials, 2016.

# Introduction

Restorative dentristry has been experiencing in the recent years a progressive shift from the use of metal-ceramic solutions to fully ceramic dental crowns, bridges and fixed-partial dentures. Among ceramic materials, zirconia has become the state-of-the-art, due to its excellent mechanical properties and good aesthetics thanks to the white color. Especially in terms of strength, zirconia is the only single-oxide ceramic that guarantees values above 1GPa in flexure, allowing the design of thin walled restorations. These restorations are normally shaped from pre-sintered milling blocks by CAD-CAM technology, which gives good results in terms of dimensional accuracy and absence of defects of the final piece.

Nonetheless, when zirconia was introduced in the market, its opacity obliged dental technicians to apply several layers of veneering ceramics in order to increase the superficial translucency and so improve the aesthetics. Other more translucent full-ceramic materials, based on the glass-ceramic systems Lithium Disilicate or Leucite (revise) became therefore a competitive alternative to zirconia especially for front teeth. Even if these materials have a strength much lower than zirconia (approximately 400 MPa), they can be placed without veneering, so offering a comparable performance for the same wall thickness. Finally, novel zirconia grades have been developed in the last years, offering better resistance to hydrothermal degradation, improved sinterability and higher translucency. Moreover, some of the pre-sintered blocks are pre-shaded in several tonalities and even multilayered with a gradient of chroma or translucency. The restorations made with such materials can be placed either without coating or with a thin glaze layer also in visible regions, allowing reducing the wall thickness.

Tetragonal zirconia exhibit moderate translucency due to residual porosity and to the effect of birefringence: since the refractive index n is different along the main crystal axes, both reflection and refraction occur at grain boundaries, so the light loses coherence leading to a reduction of the light transmittance [1], [2].

High translucency in zirconia can be pursued with several strategies: a) reducing the residual porosity, for example when adding a glassy phase or some sintering additives, b) refining the microstructure so that grain boundaries do not interfere with light, c) increasing considerably the grain size so to have less grain boundaries, and d) introducing significant amounts of cubic phase, which is optically isotropic and do not induce birefringence [1]. Indeed, these variations imply substantial changes in the performance of zirconia. To understand how the translucency challenge is tackled in actual materials, the microstructural characteristics and phase composition of monolithic crowns with different degrees of translucency are explored in this work.

The zirconia grade with 3 mol% of yttria (3Y-TZP) is known to be susceptible to hydrothermal degradation, a superficial aging phenomenon that may take place, depending on microstructure, composition and stress state, when the surface is exposed to humid environment and moderate temperatures, including those found in the human body [3]. This slow process is triggered by the diffusion into the zirconia lattice of water species, which destabilize the tetragonal phase, allowing the formation of monoclinic (Laths-twins) on the surface through a nucleation-and-growth mechanism. After the degradation has progressed on the whole surface, it starts slowly to propagate into subsuperficial regions towards the bulk of the piece [4].

The result is a microcracked layer, with the formation of uplifts on the surface and eventually grain pull out, with a progressive loss in contact mechanical properties such as hardness, wear and scratch resistance [5]–[7]. It should be underlined here that hydrothermal degradation only interests a thin layer of few micrometers in well densified bodies, leading to a very slight change in the mechanical strength [8]. The situation might be different in presence of cyclic contact loading, for example associated to mastication, where the effects of concentrated stresses and aging overlap.

In non-glazed monolithic crowns the surface is exposed to saliva, food and

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moderate temperatures, so there is the potential risk for hydrothermal degradation. One open question is whether materials that are currently employed are still suffering from this phenomenon and, if so, whether it may lead to a substantial reduction of the reliability or surface state in dental restorations. These subjects are treated in our work, which compares both the aging behavior and the mechanical properties of zirconia materials of different translucency currently employed in the elaboration of monolithic dental crowns. Special emphasis is given to novel "cubic" highly-translucent dental crowns, which have a short clinical history and for which the aging behavior has not been assessed yet. Moreover, their transformability must be substantially different than in conventional 3Y-TZP. Results are interpreted taking into account the observed differences in microstructure and phase composition.

In the case of glazed monolithic crowns, the role of glazes with respect to hydrothermal degradation is still unknown to our knowledge, so this will be another topic of our study. Nonetheless, even if the surface is coated, some regions can be accidentally left unglazed during the procedure (which is manually performed), or may become exposed after chipping or grinding due to chairside manual adjustments. This is especially true in the marginal area, which is the most delicate region in the dental crown, as it is shown in Fig. 1., where some chipping is observed, as well as unglazed spots that have been studied in detail by FIB observations.



Fig. 1. SEM image and FIB study of marginal region of a dental crown retrieved after in-vivo fracture. Some chipping can be observed in the marginal region, as well as unglazed zones, as confirmed by the FIB trench in A). In B), a detail of the subsuperficial microstructure is shown, observing the presence of defects and debris induced by the roughening process and a  $\sim$ 1.5 um thick recrystallized layer.

# **Experimental**

Four different zirconia materials were studied: one conventional grade "Aadva ST" (Standard Translucency – ST group), one with improved translucency "Aadva EI" (Enamel Intensive – EI group), one highly translucent and partially cubic "Aadva NT" (Natural Translucent – NT group) (Aadva, GC Tech, Leuven, Belgium) and one higly translucent, partially cubic and multi-layered in chroma "Katana UTML" (Ultra Translucent Multi Layered – ML group, Kuraray

Noritake Dental Inc., Aichi, Japan). 2 full-contour molar crowns preforms were milled by CAD-CAM technology from the pre-sintered 98.5 mm disks of each material. The preforms were sintered according to the manufacturer's specification, as summarized in Table 1. The outer surface was split into 2 halves, each comprising 1 supporting and 1 chewing cusp; one half was glazed ( $\sim$ 10-100 µm), while the other half was left uncoated. Two different glazes were used: CeraFusion, based on Lithium disilicate (Komet) which was fired at 1005°C during 15', and the Magic Glaze Flu(Wieland Zenostar, Ivoclar Vivadent) which was fired at 1005°C during 15'. Similarly, 20 disks 1.5 mm in thickness and 12 mm in diameter were prepared from each material and mirror polished with conventional ceramic procedures.

Label	Commercial name	Cathegory	Y <sub>2</sub> O <sub>3</sub> content (mol%)	Al <sub>2</sub> O <sub>3</sub> content (wt%)	Heating	Hold	Cooling
ST	Aadva ST	Full-strength	3	0.2	8°C/min to		9°/min to
EI	Aadva El	Enhanced translucency	3	0.05	°C/min to 1450°C	2 h	Tamb
NT	Aadva NT	High translucency	5.5	traces	?	?	?
ML	Katana UTML	High translucency multy-layered	5.5	traces	10°C/min to 1550°C	2 h	10°C/min to Tamb

Table1. Summary of specimens' preparation procedures.

The microstructure was analyzed by scanning electron microscopy (SEM) after thermal etching at 1200°C, 1h. 10°-75° 2theta X-ray diffraction (XRD) patterns were collected from polished disks to quantify the phase composition by Rietvield analysis employing the software Topaz. 26°-33° and 53°-65° 2theta XRD scans were performed at level of the outer surface of crowns (both glazed and non-glazed) at time 0 and after 2, 6, 18, 54 hours of artificial aging in autoclave (134°C at 2 bars steam pressure). The surface monoclinic content was quantified by measuring the intensity of selected peaks and applying the formula of Toraya et al. [9]

Total transmittance (Tt%) and Contrast Ratio (CR) were measured by a spectrophotometer (Jasco Y-670, France) as measures of the optical properties. The CR gives an indication of the opacity and is the ratio between the luminous reflectance of a specimen over a black background to that over a white background of a known reflectance. CR ranges

from 0 (total translucency) to 1 (total opacity) [10]. The values measured at 555 nm wavelength were chosen to compare the different materials, according to the definition of the International Commission on Illumination [11].

Half of the disks were subjected to artificial degradation for 18 hours, in order to reproduce a reasonable lifespan of a dental crown. We remind that, according to the activation energy of the hydrothermal degradation process measured on typical zirconia materials, 1 hour in autoclave at 134 °C corresponds roughly to 1 to 3 years at the human body temperature [4].

Biaxial strength was measured by means of piston-on-three-balls strength tests, using 10 disks per group. Indentation fracture (IF) toughness and hardness tests were performed on polished disk surfaces by using a Vickers indenter and a load of 10 Kg, applied. The IF toughness values were calculated from the surface length of the cracks developing at the corners of Vickers indent using the Niihara formula [12].

The results were treated statistically with one-way analysis of variance (ANOVA) with a Tukey post-hoc test, employing the software Minitab. p-value was set at 0.05. Mean values plus standard deviations are presented in the results, while letters on the carts charts indicate the statistically significant differences.

Focused Ion Beam (nVision40 dual beam station - Carl Zeiss AG, Germany) was employed to cut and polish a trench in the border between a glazed area and a non-glazed spot of selected aged samples, after depositing a strip of protective Carbon. The subsurface microstructure was then observed by SEM, obtaining secondary electron (SE) and backscattered images.

#### Results

SEM images of the samples microstructure after sintering are shown in Fig. 2. The grain size, measured by the intercept method, is reported in Table 2. ST shows a classical grain 3Y-TZP grain size of 0.5  $\mu$ m, while this value is slightly reduced in EI. Both microstructures are quite homogenous, while a bimodal appearance is found in the highly translucent materials, especially for NT. Moreover, the grain size is much bigger, especially in ML.



Fig. 2. SEM images showing the microstructure of the 4 materials studied. From a) to d): ST, EI, NT, ML.

Table	1:	Grain	size,	phase	analysis	after	Rietvield	refinement	and	Optical
propert	ies	(CR ar	nd Tt%	6) for th	he four m	aterial	s			

Grade	Grain Size (µm)	Group	Tetrag. (%)	Cubic (%)	CR	Tt%
ST	500±35	а	76.8	23.2	0.74±0.01	36.9±0.15
EI	400±20	b	80.3	19.7	0.70±0.01	38.4±0.07
NT	980±40	С	67.8	32.2	0.62±0.01	43.4±0.13
ML	1800±580	d	68.9	31.1	0.69±0.01	36.0±0.07



Fig. 3. XRD representative patterns of the materials studied



Fig. 4. Biaxial strength before and after 18 h artificial aging for the four materials studied are reported in a), where the suffix "–A" indicates aged materials. Hardness and IF toughness values are shown in b) and c), respectively. Letters indicate statistically significant differences.

The biaxial strength of cubic crowns were definitely lower than for tetragonal ones, as shown in Fig. 4. ST exhibit strength values well above 1000 MPa, while EI shows slightly lower values. For NT and EI the strength is similar, around 450 MPa.

Hardness was similar for the four materials tested, observing minor differences of less than 0.5 GPa. In order of increasing hardness we find ST, EI, ML, NT. IF toughness was similar between ST and EI (~4.7 MPa $\sqrt{m}$ ) and between NT and ML (~3.8 MPa $\sqrt{m}$ )

The results of aging tests are reported in Fig. 5.

Indeed, NT and ML are fully resistant to hydrothermal degradation for the aging times applied here. One second observation is that tetragonal materials suffer from the aging phenomenon, starting also from very short aging times (2h). EI is aging faster than ST until 18h of exposure, while they reach similar values at 54h.

The biaxial strength did not show any variation after 18 h of artificial aging for ST, NT and ML materials. Results suggest a slight increase (even if not statistically significant) for EI samples.



Fig. 5. Evolution of the monoclinic content measured by XRD after artificial aging.

Diffraction patterns taken from the glazed side of dental crowns were readable and allowed to measure the amount of monoclinic phase, since X-rays could penetrate through the glaze layer and reach zirconia surface. No monoclinic phase was found both for CeraFusion and MagicGlaze Flu types, except for some isolated cases. Optical observations revealed that in these cases a significant portion of the surface was left unglazed, as it is shown in Fig. 6. The subsurface around some unglazed spots was therefore studied by FIB as shown in Fig. 7, where SEM analysis revealed that aging progressed from the exposed spot both towards the bulk and the sides, in a hemispherical volume of radius ~16  $\mu$ m.



Fig. 6. Optical images of monolithic dental crowns, showing the presence of partially-glazed regions



Fig. 7. FIB study of an unglazed spot in the area close to the margin of an EI monolithic crown. The spot is visible to the left of the trench in C), where the presence of glaze is also recognizable by the darker color with respect to the zirconia surface. By imaging the FIB trench with backscattered electrons, the transformed volume around the spot becomes clearly visible in D). Unaffected microstructure is observable on the right of the trench, well below the glazed region. Arrows indicate the direction of spreading of the degradation from the spot.

#### Discussion

## Microstructure, composition & translucency

Important differences are found in terms of microstructure for the materials studied. The first reason is indeed the yttria content, which is much higher in NT and ML (5.5 mol%) with respect to ST and EI (3 mol%). These additions allows to obtain a combined tetragonal-cubic microstructure where the translucency improvement is twofold: cubic grains do no exhibit birefringence since they are optically isotropic, and being the tetragonal grains supersaturated in yttria, their tetragonality is reduced and so the refractive index and the amount of birefringence [1]. While the addition of yttria tends normally to reduce the grain size with respect to pure zirconia, once cubic phase is nucleated, grain growth is boosted as shown by the micrographs of FIG 2.

Moreover, there is a difference in grain size and distribution between ML and NT, which can be attributed to the different sintering protocols. Their CR and Tt% is significantly different, probably in relation to the presence of a chroma gradient in ML, as it was shown for colored and non-colored 3Y-TZP [13].

Having ST and EI similar amount of yttria, there is only a slight difference in grain size, which is smaller for EI, and in composition. Therefore, the observed substantial difference in CR and Tt% is justified by the absence of second

phases (Al2O3) and smaller grains for EI. Alumina grains are randomly present in ST, like in standard 3Y-TZP, due to the fact that tetragonal zirconia is supersaturated in alumina to improve its aging behavior [14]. A reduction of the residual porosity might also justify the better optical properties for EI. In fact, light scattering is greatly affected by the residual porosity, where only some fractions % can lead to an opaque material, if the pores are bigger than approx. 50 nm[15].

Indeed, the translucency of all the materials studied here is still below the one of enamel (CR  $\sim 0.45$ ) and only comparable to the one of dentine (CR  $\sim 0.65$ ) for the highly translucent ones. According to the classification introduced by Vichi et al. [16], these materials can be classified as "low-translucent" (ST) and "medium-translucent" (EI, NT, ML), and their application as monolithic crowns in aesthetic frontal areas may find some limitations also for the NT and ML.

XRD patterns in Fig. 3 show substantial differences between ST, EI and NT, ML. For the first two materials, the spectra are quite similar, with a strong tetragonal component, as indicated by the presence of several tetragonal doublets. Similar spectra are found also among NT and ML, this time with a strong cubic component. After Rietveld refinement, it was shown that all the materials are composed by a mixture of cubic and tetragonal phases, where approximately 50% of cubic phase is found in highly translucent crowns, and

about 30% is found both in ST and EI materials.

# Aging behavior

The first striking evidence is that highly translucent zirconia with mixed tetragonal, cubic composition and 5.5 mol% yttria do not suffer from hydrothermal degradation even after 54 h of artificial aging. It can therefore be stated that these materials are fully resistant to aging in in vitro conditions and no external load applied. This imply that the tetragonal phase must be supersaturated in yttria with respect to 3Y-TZP, and so less transformable. The presence of an intrinsically non-transformable cubic matrix may also help in hindering the aging process. This is true for the thermal treatments applied here, while a similar behavior cannot be assured for longer sintering times or higher temperatures. In fact, the enhanced diffusion process in the latter case would promote a more complete separation of tetragonal and cubic phases, the cubic volume fraction would increase, absorbing yttium from the tetragonal phase, which would become less stable, and, eventually, transformable [17]–[19].

Another important point to underline is that hydrothermal degradation was observed in ST and EI starting from very short aging times (2h). Indeed, 3Y-TZP used nowadays in dentistry are still susceptible to aging when prepared in clinical conditions and cannot offer high aging resistance. Having EI smaller grain size than ST, a slower aging process is expected. [20] Nonetheless, the observed kinetics is faster for EI, which imply that the effect of alumina addition is more important than the difference in grain size, as shown in Refs. [14], [21]. Very little dispersion was observed in terms of  $V_m$ % among different samples of the same group, indicating that CAD-CAM processing induce a highly comparable surface state for what concerns hydrothermal degradation.

It was proven that glazing 3Y-TZP effectively protects it against aging for both the glazes examined here and even for glazing thicknesses of few micrometers and 54h of exposure. On the other hand, it was observed that a perfect glazing, coating 100 % of the surface, is practically impossible to obtain. This can be the result of: a) insufficient coverage during the glaze precursor application, especially for glazes that are applied by spray, which tend to leave some unglazed spots; b) impossibility of glaze application on the marginal edge; c) chipping of the glaze in the marginal area; d) chairside adjustments made by the dentist before placing the crown. Therefore, if the material is susceptible to hydrothermal degradation, after glazing it will inevitably undergo the aging process in some localize areas, as it has been demonstrated by the FIB-SEM study summarized in Fig. 7. Aging will spread from the non-glazed spot in radial direction, both towards the bulk and adjacent surface regions, so also below the glazed areas. The study was performed on an EI sample at 54 h exposure, so the one aging faster after the more severe aging treatment, but we can expect qualitatively similar results for other 3Y-TZP grades. The first implication of this process is that we can expect to create subsurface microcracking and damage under the glaze, which may result in a proneness to
chipping due to the poor strength of degraded zirconia especially when a tensile load is applied perpendicularly to the surface (separation load) [22]. This would, in turn, extend the unglazed spot, exposing neighboring areas to a similar phenomenon. A second implication is that this process creates damaged localized spots instead of a uniform degraded layer. While in the case of polished-and-aged zirconia the mechanical strength of 3Y-TZP is only slightly reduced after long and severe hydrothermal exposure [8], the presence of these microcracked spots could lead to stress concentrations, which might have critical consequences especially in the marginal area. Therefore, even if it was shown that the glaze serves as a protection for the surface beneath, its application cannot improve the crown aging resistance if considered as a whole.

## **Mechanical properties**

The observed slight variations of hardness can be attributed to variations of microstructure and phase composition. It is well known from the Hall-Petch relation that, for the same composition, the smaller the grain size, the harder the material, as it happens for EI with respect to ST. NT is even harder due to the high amount of cubic phase, which is harder than the tetragonal [23]. ML has a lower hardness with respect to NT due to the bigger grain size, being the cubic phase content approximately equal for both.

Important differences were registered in terms of biaxial strength, showing that ST and EI are at least twice as strong as NT and ML, implicating a dramatic

difference between full-strength and last generation highly-translucent zirconias. This proves that the tetragonal phase in NT and ML is overstabilized, having insufficient transformability for inducing the beneficial effects of the transformation toughening process. Therefore, in these materials the final strength is determined mainly by the size of defects present in the microstructure and the intrinsic fracture toughness of zirconia. Anyhow, it is surprising that both NT and ML have practically the same strength values. Given that the grain size is quite different, one should expect lower strength for bigger grains since grain size and defect size are normally associated. On the other hand, the decrease of strength might be compensated in ML as a consequence of a toughening mechanism mediated by crack deflection, which is also more important for bigger grains. The main conclusion is that, with these novel materials, the flexural strength and the transformation toughening unique to zirconia have been sacrificed in name of aesthetics. The biaxial strength for highly translucent zirconias is just little better than for the best glass-ceramics available on the market, which are indeed more translucent [REF], so the choice of these materials for producing dental crowns is not obvious. Therefore, it is important for the dentist to avoid expecting a similar mechanical performance from both opaque and translucent zirconias. On the other hand, both ST and EI fulfill the requirements for dental ceramics capable of supporting 4 and more unit, even though the biaxial strength is slightly lower for EI. This evidence might be explained by the fact that smaller grain size means normally lower

transformability. So EI is less mechanically transformable but more susceptible to hydrothermal degradation than EI.

As expected, after 18h of aging exposure, NT and ML did not show any strength variation, since no aging took place for these materials. The situation is different for ST and EI: as demonstrated, they are both susceptible to aging, with  $\sim 37$ Vm% and ~46 Vm%, respectively, recorded at 18h exposure. Anyway, no strength variation was recorded for ST after aging, while a slight strength increase was measured for EI. This variation can have two possible justifications: a) the aged layer induces some degree of "tip blunting" for the biggest defects/cracks naturally present in the material: in this case, either the defects of EI are different in nature than the ones of ST and so more responsive to the blunting effect (bigger, sharper), or the higher amount of monoclinic phase in EI induces a deeper blunting effect; b) since aging has progressed deeper in EI, the compressive layer which normally accompanies the degradation process in the transition zone between degraded and non-degraded material is placed close to the critical defects tip, inducing a reduction in their stress-intensity factor. This thickness can be estimated in approximately 10-15 um by considering standard fracture mechanics solutions for surface cracks and a failure stress of 1000-1200 MPa. We can expect therefore to not observe the strength increase for longer and shorter aging exposures, since the compressive layer would be placed deeper or less deep, respectively, than the defects tip. In both cases, the degraded layer after 18h artificial aging is not thicker than the

critical defects, since there no strength decrease was recorded.

IF toughness results support the above discussion, indicating sensibly lower values for NT and ML with respect to ST and EI. This difference can be interpreted as lower transformability for the highly translucent materials. On the other hand, similar results were found for ST and EI, so the difference in biaxial strength among the two groups may be not imputable to a different transformability. Nonetheless, we prefer not to speculate further on this aspect due to the intrinsic limitations of the IF toughness test and its precision.

### **Clinical implications**

Some concerns surround the application of zirconia in dentistry due to the possibility for hydrothermal degradation. We have shown that hydrothermal degradation takes place in state-of-the-art materials prepared clinically and shaped as normal dental crowns. We have also shown that this phenomenon is not detrimental for strength even after long exposures, at least in vitro. Even if the possibility of a much more severe – and maybe localized – aging process when cyclic contact load is applied remains still an open question, these results should reassure dentists and patients about the use of zirconia. Future studies implying zirconia crowns retrieved from in vivo patients may give an answer to this question.

On the other hand, the use of highly-translucent non-transformable zirconia materials should be done carefully. If it is true that these materials are more

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aesthetic and do not suffer from in vivo hydrothermal degradation, their toughness and strength is dramatically lower than in 3Y-TZP. Therefore, manipulation and crown preparation should be done carefully, avoiding thin walls and the presence of sharp edges as much as possible. Having similar mechanical strength as for lithium disilicate glass-geramics, the latter may also be evaluated as an alternative when considering the use of these materials.

The glaze does not represent a real protection against aging, so it should be considered only for aesthetical purposes.

# Conclusion

In tetragonal zirconia monolithic crowns, hydrothermal degradation can be observed also for short aging times, while the glaze act as a protective layer against it. The effect of glaze is protective for the zirconia underneath, nonetheless this is generally not beneficial since some regions are always left unglazed, especially in the marginal regions.

The presence of cubic phase in highly translucent materials entails two main advantages, a sensible increase in translucency and the complete absence of hydrothermal degradation. On the other hand, the lack of transformation toughening for cubic zirconia and the coarser microstructure cause a severe drop in mechanical properties, which can represent a limitation for their application in conditions where high mechanical stresses are applied.

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## Conclusions

Digital technology is advancing rapidly in dentistry. Computers are making what were previously manual tasks easier, faster, cheaper and more predictable. Layered manufacturing processes can produce complex shapes at affordable prices with little or no waste. The challenge for the dental materials research community is to mach the new technology with materials that are suitable for use in dentistry. This can potentially take dental materials research in a totally different direction.

The new generation of ceramic materials presents interesting options, both in terms of material selection and in terms of fabrication techniques. A closer understanding of the dynamics of the materials with respect to design of the restoration and the intended use is required to enable these restorations to perform productively.

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