Assessment of Two Surface Treatment Protocols on Monolithic Zirconia at Presintered and Post-sintered Stages

A thesis submitted for the partial fulfillment of the Doctorate Degree requirements in Crown & Bridge, Faculty of Dentistry, Ain Shams University

By

Kamal Khaled Ebeid Ahmed

B.D.S, Faculty of Dentistry, Ain Shams University, 2009 M.sc, Faculty of Dentistry, Ain Shams University, 2014

Supervisors

Dr. Tarek Salah Morsi

Assistant professor and head of Crown and Bridge Department Faculty of Dentistry, Ain Shams University, Egypt

Dr. Marwa Mohamed Wahsh

Assistant professor, Crown and Bridge Department Faculty of Dentistry, Ain Shams University, Egypt

Dr. Maged Mohamed Zohdy

Lecturer, Crown and Bridge Department
Faculty of Dentistry, Ain Shams University, Egypt

Prof. Dr. med. dent. Matthias Kern

Head of Prosthodontics, Propaedeutics, and Dental Materials Department Christian-Albrechts University, Kiel, Germany

<u>Acknowledgment</u>

No words can express my deepest thanks and sincere gratitude as well as appreciation to *Dr. Tarek Salah Morsi*, Professor of Crown and Bridge, Faculty of Dentistry, Ain Shams University. His valuable advice, devoted effort and unique cooperation, will always be deeply remembered. This work could have never been completed without his extraordinary assistance and sincere guidance. His advice on both research as well as on my career have been priceless.

I would also like to express my special appreciation and thanks to *Prof. Matthias Kern*, Head of Prosthodontics, Propaedeutics, and Dental Materials Department, Christian-Albrechts University, whom have been a tremendous mentor for me. I would like to thank him for encouraging my research and for allowing me to grow as a research scientist.

I want also to express my profoundest gratitude to *Dr. Marwa Wahsh*, Assistant Professor of Crown and Bridge, Faculty of Dentistry, Ain Shams University. Tutoring me since I was an undergraduate student till today, Dr. Marwa offered her unreserved help, guidance, motivation, and immense knowledge.

The good advice, support and friendship of *Dr. Maged Zohdy*, Lecturer of Crown and Bridge, Faculty of Dentistry, Ain Shams University has been invaluable on both an academic and a personal level, for which I am extremely grateful. His devoted effort, close supervision and remarkable help are highly appreciated.

I wish to express appreciation for technical assistance and laboratory support of *Dr. Sebastian Wille, Frank Lehmann, Detlev Gostomsky, and Rüdiger Möller*, of the Department of Prosthodontics, Propaedeutics and Dental Materials, Christian-Albrechts University at Kiel.

Last but not least, deepest thanks to my dear professors, colleagues and staff members of Crown and Bridge Department, Faculty of Dentistry, Ain Shams University, and special thanks to my elder brothers *Dr. Amr el Etreby*, *Dr. Ahmed Sabet* and *Dr. Ahmed Abo El Fadl* for their great support, encouragement and cooperation.

<u>Dedication</u>

This work is dedicated to

My dear parents,

Precious sister,

Beloved wife,

and lovely son

Contents

List	of fig	gures	1
List	of ta	bles	IV
List	of ab	obreviations	V
Intro	duc	tion	. 1
Revi	ew c	of literature	4
Zi	rcon	ia restorations	4
Ef	fect	of different surface treatments on zirconia	6
Ef	fect	of surface treatment of zirconia on its surface roughness	6
		of surface treatment and aging on the phase transformation and flexural the of zirconia	
Ef	fect	of surface treatment of zirconia on the bond strength to resin cement	L3
Ef	fect	of stage of surface treatment on zirconia	26
Aim	of th	he study	29
Mat	erial	s and methods	30
I)	N	Naterials	30
II)	N	Nethods	31
	1-	Sample grouping	31
	2-	Fabrication of zirconia discs	33
	3-	Surface treatment of discs	37
	4-	Surface roughness evaluation	39
	5-	Phase transformation evaluation	11
	6-	Biaxial flexural strength	13
	7-	Tensile bond strength	16
	8-	Data management and analysis	58
Resu	ılts	6	50
1- or		ffect of type of surface treatment and the stage in which it is performed face roughness6	50
2- or		ffect of type of surface treatment and the stage in which it is performed ase transformation	56

Refe	rences	. 98
Sum	mary and Conclusions	. 96
Discu	ussion	. 87
5-	Correlations of results of different variable	. 86
	Effect of type of surface treatment and the stage in which it is performed the tensile bond strength of zirconia to resin cement	
	Effect of type of surface treatment and the stage in which it is performed the biaxial flexural strength	

List of figures

Figure 1: Illustration showing how samples were divided in each subgroup	32
Figure 2: Disc design on CAD software	33
Figure 3: Datron dental milling machine	35
Figure 4: Zirconia blanks after milling the discs	35
Figure 5: Robocam dryer	36
Figure 6: The two disc designs before and after sintering	36
Figure 7: Apparatus to standardize nozzle specimen distance	37
Figure 8: Rocatec silica coating machine	38
Figure 9: Specimens placed under the apparatus and surface treated	38
Figure 10: Keyence VX-100 laser scanning microscope	40
Figure 11: 100X magnification scanning lens	40
Figure 12: Zirconia disc placed in specimen holder	42
Figure 13: X-ray source, disc, and receptor	42
Figure 14: Metallic platform with steel balls	43
Figure 15: Zirconia disc placed on metallic platform	44
Figure 16: Close-up view of zirconia disc, metallic platform, and piston	44
Figure 17: Fractured zirconia disc	45
Figure 18: Resin cement, universal primer, and composite core material	46
Figure 19: Plexiglass tube	47
Figure 20: Filling of plexiglass with composite core material	47
Figure 21: Plexiglass overfilled with composite core material	48
Figure 22: Removal of excess composite and formation of a flat surface	48
Figure 23: Rubbing of universal primer on zirconia disc	50
Figure 24: Air drying of universal primer	50
Figure 25: Insertion of plexiglass tube with composite into bonding apparatus	.51
Figure 26: Placing of resin cement	51
Figure 27: Applying the load using the bonding apparatus to bond the resin	
cement to zirconia	52
Figure 28: Close-up view of zirconia disc, resin cement, and plexiglass tube	52
Figure 29: Removal of excess cement using pellet	53
Figure 30: Light curing of resin cement	53
Figure 31: Bonded plexiglass containing composite core to the zirconia disc us	ing
resin cement	54
Figure 32: Water incubator with temperature set at 37 degrees Celsius	55
Figure 33: Thermocycling machine	55
Figure 34: Loop apparatus for tensile bond strength testing	56
Figure 35: Close-up view of loop apparatus	56
Figure 36: Gold sputtered zirconia specimen	57

Figure 37: Scanning electron microscope	58
Figure 38: Box plot of Ra for all subgroups	61
Figure 39: 3D surface image of control group (unsintered)	62
Figure 40: 3D surface image of control group (Sintered)	62
Figure 41: 3D surface image of group air abraded in pre-sintered stage	
(Unsintered)	63
Figure 42: 3D surface image of group air abraded in pre-sintered stage (Sinter	ed)
	63
Figure 43: 3D surface image of group silica coated in pre-sintered stage	
(Unsintered)	
Figure 44: 3D surface image of group silica coated in pre-sintered stage (Sinte	-
Figure 45: 3D surface image of group air abraded in post sintered stage	
Figure 46: 3D surface image of group silica coated in the post sintered stage	65
Figure 47: Illustration of graph showing only tetragonal phase characteristic	
peaks	
Figure 48: Illustration of graph showing tetragonal and monoclinic characteris	
peaks	
Figure 49: Box plot of monoclinic phase % for all subgroups	
Figure 50: Box plot of BFS for all subgroups	
Figure 51: Box plot of TBS for all subgroups (Before thermocycling)	
Figure 52: Box plot of TBS for all subgroups (Before and after thermocycling) Figure 53: Graph showing mode of failure for all subgroups before thermocycling.	
rigure 33. Graphi showing mode of failure for all subgroups before thermocyc	_
Figure 54: Graph showing mode of failure for all subgroups after thermocyclin	
Figure 55: Stereomicroscope image of control group (Before thermocycling)	_
Figure 56: SEM image of control group (Before thermocycling)	
Figure 57: Stereomicroscope image of control group (After thermocycling)	
Figure 58: SEM image of control group (After thermocycling)	
Figure 59: Stereomicroscope image of group air abraded in pre-sintered stage	
(Before thermocycling)	
Figure 60: SEM image of group air abraded in pre-sintered stage (Before	
thermocycling)	78
Figure 61: Stereomicroscope image of group air abraded in pre-sintered stage	!
(After thermocycling)	
Figure 62: SEM image of group air abraded in pre-sintered stage (After	
thermocycling)	79
Figure 63: Stereomicroscope image of group silica coated in pre-sintered stag	е
(Before thermocycling)	80
Figure 64: SEM image of group silica coated in pre-sintered stage (Befroe	
thermocycling)	80

Figure 65: Stereomicroscope image of group silica coated in pre-sintered stage	
(After thermocycling)	81
Figure 66: SEM image of group silica coated in pre-sintered stage (After	
thermocycling)	. 81
Figure 67: Stereomicroscope image of group air abraded in post sintered stage	
(Before thermocycling)	82
Figure 68: SEM image of group air abraded in post sintered stage (Befroe	
thermocycling)	. 82
Figure 69: Stereomicroscope image of group air abraded in post sintered stage	
(After thermocycling)	83
Figure 70: SEM image of group air abraded in post sintered stage (After	
thermocycling)	. 83
Figure 71: Stereomicroscope image of group silica coated in post sintered stage	2
(Before thermocycling)	84
Figure 72: SEM image of group silica coated in post sintered stage (Before	
thermocycling)	. 84
Figure 73: Stereomicroscope image of group silica coated in post sintered stage	•
(After thermocycling)	85
Figure 74: SEM image of group silica coated in post sintered stage (After	
thermocycling)	. 85

List of tables

Table 1: Materials used in this study	30
Table 2: Experimental factorial design	32
Table 3: Effect of sintering on mean(SD) of Ra of group treated in pre-sinte	red
stage and control group	60
Table 4: Percentage decrease in surface roughness due to sintering	60
Table 5: Mean(SD) of Ra for all subgroups	60
Table 6: Effect of sintering stage on mean(SD) of Ra	61
Table 7: Effect of surface treatment on mean(SD) of Ra	61
Table 8: Effect of sintering on mean(SD) of monoclinic % of group treated in	n pre-
sintered stage and control group	66
Table 9: Mean(SD) of monoclinic % for all subgroups	66
Table 10: Mean(SD) of BFS in MPa for all subgroups	69
Table 11: Effect of sintering stage on mean(SD) of BFS	69
Table 12: Effect of surface treatment on mean(SD) of BFS	70
Table 13: Mean(SD) of initial TBS for all subgroups	71
Table 14: Mean(SD) of TBS for all subgroup before and after thermocycling	73

List of abbreviations

Al₂O₃: Aluminum trioxide

BFS: Biaxial flexural strength

CAD: Computer aided design

CAM: Computer aided manufacturing

FDP: Fixed dental prosthesis

LTD: Low temperature degradation

MDP: 10-methacryloxydecyl dihydrogen phosphate

Ra: Surface roughness

SBS: Shear bond strength

TBS: Tensile bond strength

XPS: X-ray photoelectron spectroscopy

XRD: X-ray diffraction

ZrO₂: Zirconium dioxide

Introduction

The increased popularity of all-ceramic materials as an alternative to metal-ceramic restorations is mainly due to their excellent esthetics, chemical stability and biocompatibility. However, the brittleness and low tensile strength of conventional glass ceramics limit their long-term clinical application. Several glass ceramics have been introduced such as high alumina-content glass-infiltrated ceramic material and lithium disilicate glass-ceramic which has been successfully used for crowns, anterior fixed dental prostheses (FDPs) and three-unit FDPs replacing the first premolar. However, these materials do not have sufficient strength to allow reliable use for FDPs, especially in the molar region.

Recently, the development of advanced dental ceramics has led to the application of partially stabilized zirconia in restorative dentistry which can be produced using a computer aided design/computer-aided manufacture (CAD/CAM) systems. The use of zirconia-based ceramics for dental restorations became more popular due to their superior fracture strength and toughness when compared to other dental ceramic systems.⁽⁴⁻⁶⁾

Nowadays, the application of zirconia in the production of all-ceramic restorations has become one of the most focused on topics in dental research. Such increase in the interest is mainly due to its high mechanical strength and exceptional biocompatibility.^(7, 8) The success of zirconia-based all-ceramic restorations is highly dependent on the establishment of a strong adhesion between zirconia and the luting cement. Compared with traditional luting cements, resin cement has some irreplaceable advantages including higher mechanical strength and better esthetic properties.⁽⁹⁾ However, without any surface treatment, the resin zirconia integration was found to be susceptible to aging conditions.⁽¹⁰⁾ Meanwhile, the conventional bonding approaches, such

as acid etching followed by the application of silane coupling agents, could not effectively improve the bond strength between zirconia and resin cement due to the chemical inertness of zirconia and the absence of a glass content. (11, 12) Thus, zirconia ceramics cannot be etched with commonly used acids, such as hydrofluoric (HF) and phosphoric (H₃PO₄) acids for adding the surface roughness. Furthermore, it is also very cumbersome to form a strong chemical integration between zirconia and resin cement by using solely the conventional silane coupling agents. (13-15)

Air-abrasion with alumina particles followed by an appropriate chemical bonding process was recommended to achieve long-term retention to zirconia. The incorporation of 10-methacryloxydecyl dihydrogen phosphate (MDP) in primers or resin cements was a major factor in producing durable resin zirconia bonding which has already been confirmed in the related clinical trials. Other surface treatments, such as the tribochemical silica coating, selective infiltration etching, heating with a hot etching solution, laser surface treatment, plasma treatment and surface fluorination, have been developed to enhance resin zirconia bonding. The tribochemical method has been proven not only to increase the values of surface roughness, but also to add silica content on zirconia surface. Silica content is vital for activating the functions of silane coupling agents. Both mechanical interlocking and chemical integration between resin cement and zirconia have thus been enhanced. (19)

However, there are still concerns about the influence of air abrasion and tribochemical silica coating on the mechanical properties and long-term stability of zirconia ceramic since it has been reported that they induce some flaws and phase transformation on the surface, thus promoting low temperature degradation. The generation of such flaws and transformation might produce some detrimental effects on the liability of zirconia ceramic. (21)

There are also some concerns about the durability of the bond strength to silica-coated zirconia ceramic as many laboratory studies showed that it decreased significantly after a few months of artificial aging. (22, 23, 13, 15, 24)