

# The Effect of Non-Thermal Plasma Treatment on the Shear Bond Strength of Monolithic Zirconia

A thesis submitted for the partial fulfillment of the requirements of Master's Degree in fixed prosthodontics, Faculty of Dentistry, Ain-Shams University

### Presented by:

### **Nancy Adel Hamed El-Gohary**

B.D.S (Misr International University 2010)

Faculty of Dentistry Ain-Shams University

2020

# **Supervisors**

### Dr/Marwa Mohamed Wahsh

Associate Professor of Fixed Prosthodontics Faculty of Dentistry, Ain-Shams University

### **Dr/Ahmed Ezzat Sabet**

Associate Professor of Fixed Prosthodontics Faculty of Dentistry, Ain-Shams University

# Acknowledgment

### Acknowledgement

First and forever thanks and gratitude to ALLAH for guiding me to the right way.

It's a great honor to express my sincere gratitude and appreciation to my professor Dr/ Marwa Mohamed Wahsh, Associate professor of fixed prosthodontics, faculty of Dentistry, Ain-shams University for her guidance, motivation, patience and for sharing knowledge.

I am greatly thankful to my advisor Dr/Ahmed Ezzat Sabet, Associate professor of fixed prosthodontics, faculty of Dentistry, Ain-shams University for his great support, patience, encouragement and help during this research.

I would like to thank Dr/Khaled Keraa Biostatistics and Quality management specialist, faculty of Dentistry, Misr international university for his sharing in the statistical analysis part in the thesis.

A very special gratitude goes to the department of fixed prosthodontics at Misr International University & Ain-Shams University.

To my best friends and sisters Sara El-debeeky and Omnia Ismail for their support and assistance throughout this work.

# Dedication

#### **Dedication**

To my grandmother, our family support system. Your prayers and unconditional love will be always surrounding me throughout my life.

To my beloved father and mother who are my source of power, giving me strength and emotional support in my weak moments. You are the reason of what I become today.

To my lovely sisters, life is full of changes but sisters' love is forever.

To my husband, friend and soul mate. Thank you for being my strength when I am weak, my calm when I am angry, and my everything I need.

To my lovely son, you will always be my sunshine and my little angel.

To my dear friends, you are the best part of me.

# **List of Contents**

Item	Page
List of figures	IV
List of tables	V
Introduction	1
Review of literature	3
Statement of the problem	32
Aim of the study	33
Materials and methods	34
Results	54
Discussion	66
Summary	75
Conclusion	77
References	78
Arabic summary	

# **List of Figures**

(1) Katana Zirconia STML Disc (2) Panavia SA cement plus (3) Ulta etch 35% phosphoric acid gel (4) 50µm alumina oxide particles for air abrasion (5) 98%Hexamethoxydisioxane for NTP treatment (6) Komet saw used for cutting of zirconia discs (7) Milling of zirconia into cylindrical block (8) cylindrical specimen of 5 mm diameter before sintering process (9) cutting disc shaped specimens of 2.5mm thickness before sintering (10) 5 mm diameter before sintering (11) 2.5mm thickness after sintering (12) 4mm diameter after sintering (13) 2mm thickness after sintering (14) Air abrasion of zirconia disc (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment (20) Schematic drawing showing the equipment component (21) Quanta scanning electron microscope 48 (22) Gold spattered zirconia sample 48	Figure	Item	Page
(3) Ulta etch 35% phosphoric acid gel 38 (4) 50µm alumina oxide particles for air abrasion 38 (5) 98%Hexamethoxydisioxane for NTP treatment 38 (6) Isomet saw used for cutting of zirconia discs 39 (7) Milling of zirconia into cylindrical block 40 (8) cylindrical specimen of 5 mm diameter before sintering process (9) cutting disc shaped specimens of 2.5mm thickness before sintering 41 (11) 2.5mm thickness after sintering 41 (12) 4mm diameter before sintering 41 (13) 2mm thickness after sintering 41 (14) Air abrasion of zirconia disc 45 (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber 46 (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47 (20) Schematic drawing showing the equipment component 48	(1)	Katana Zirconia STML Disc	36
(4) 50µm alumina oxide particles for air abrasion 38 (5) 98%Hexamethoxydisioxane for NTP treatment 38 (6) Isomet saw used for cutting of zirconia discs 39 (7) Milling of zirconia into cylindrical block 40 (8) cylindrical specimen of 5 mm diameter before sintering process (9) cutting disc shaped specimens of 2.5mm thickness before sintering 41 (11) 2.5mm thickness after sintering 41 (12) 4mm diameter before sintering 41 (13) 2mm thickness after sintering 41 (14) Air abrasion of zirconia disc 45 (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting 46 (17) Placement of specimens in cylindrical chamber 46 (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47 (20) Schematic drawing showing the equipment component 48	(2)	Panavia SA cement plus	38
(5) 98%Hexamethoxydisioxane for NTP treatment (6) Isomet saw used for cutting of zirconia discs (7) Milling of zirconia into cylindrical block (8) cylindrical specimen of 5 mm diameter before sintering process (9) cutting disc shaped specimens of 2.5mm thickness before sintering (10) 5 mm diameter before sintering (11) 2.5mm thickness after sintering (12) 4mm diameter after sintering (13) 2mm thickness after sintering (14) Air abrasion of zirconia disc (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47 (20) Schematic drawing showing the equipment component	(3)	Ulta etch 35% phosphoric acid gel	38
(6) Isomet saw used for cutting of zirconia discs (7) Milling of zirconia into cylindrical block (8) cylindrical specimen of 5 mm diameter before sintering process (9) cutting disc shaped specimens of 2.5mm thickness before sintering (10) 5 mm diameter before sintering (11) 2.5mm thickness after sintering (12) 4mm diameter after sintering (13) 2mm thickness after sintering (14) Air abrasion of zirconia disc (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47 (20) Schematic drawing showing the equipment component	(4)	50 µm alumina oxide particles for air abrasion	38
(7) Milling of zirconia into cylindrical block (8) cylindrical specimen of 5 mm diameter before sintering process  (9) cutting disc shaped specimens of 2.5mm thickness before sintering  (10) 5 mm diameter before sintering (11) 2.5mm thickness after sintering (12) 4mm diameter after sintering (13) 2mm thickness after sintering (14) Air abrasion of zirconia disc (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47 (20) Schematic drawing showing the equipment component	(5)	·	38
(8) cylindrical specimen of 5 mm diameter before sintering process  (9) cutting disc shaped specimens of 2.5mm thickness before sintering  (10) 5 mm diameter before sintering  (11) 2.5mm thickness after sintering  (12) 4mm diameter after sintering  (13) 2mm thickness after sintering  (14) Air abrasion of zirconia disc  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle  (16) Ultrasonic cleansing of zirconia disc after sandblasting  (17) Placement of specimens in cylindrical chamber  (18) The glow discharge of plasma inside the cylindrical chamber  (19) Plasma equipment  47  (20) Schematic drawing showing the equipment component	(6)	Isomet saw used for cutting of zirconia discs	39
(9) cutting disc shaped specimens of 2.5mm thickness before sintering  (10) 5 mm diameter before sintering  (11) 2.5mm thickness after sintering  (12) 4mm diameter after sintering  (13) 2mm thickness after sintering  (14) Air abrasion of zirconia disc  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle  (16) Ultrasonic cleansing of zirconia disc after sandblasting  (17) Placement of specimens in cylindrical chamber  (18) The glow discharge of plasma inside the cylindrical chamber  (19) Plasma equipment  47  (20) Schematic drawing showing the equipment component  (21) Quanta scanning electron microscope  48	(7)	Milling of zirconia into cylindrical block	40
(10) 5 mm diameter before sintering 41 (11) 2.5mm thickness after sintering 41 (12) 4mm diameter after sintering 41 (13) 2mm thickness after sintering 41 (14) Air abrasion of zirconia disc 45  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber 46 (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component 48	(8)		40
(11) 2.5mm thickness after sintering 41 (12) 4mm diameter after sintering 41 (13) 2mm thickness after sintering 41 (14) Air abrasion of zirconia disc 45  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting 45  (17) Placement of specimens in cylindrical chamber 46 (18) The glow discharge of plasma inside the cylindrical chamber 46  (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component 47	(9)	l	40
(12) 4mm diameter after sintering 41 (13) 2mm thickness after sintering 41 (14) Air abrasion of zirconia disc 45  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber 46 (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component (21) Quanta scanning electron microscope 48	(10)	5 mm diameter before sintering	41
(13) 2mm thickness after sintering (14) Air abrasion of zirconia disc 45  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber 46 (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component 47  (21) Quanta scanning electron microscope 48	(11)	2.5mm thickness after sintering	41
(14) Air abrasion of zirconia disc 45  (15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle  (16) Ultrasonic cleansing of zirconia disc after sandblasting 45  (17) Placement of specimens in cylindrical chamber 46  (18) The glow discharge of plasma inside the cylindrical chamber 46  (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component 47  (21) Quanta scanning electron microscope 48	(12)	4mm diameter after sintering	41
(15) Suction tip was sectioned to standardize the distance of 10mm between the disc and the nozzle (16) Ultrasonic cleansing of zirconia disc after sandblasting (17) Placement of specimens in cylindrical chamber (18) The glow discharge of plasma inside the cylindrical chamber (19) Plasma equipment (19) Schematic drawing showing the equipment component (20) Quanta scanning electron microscope 48	(13)	2mm thickness after sintering	41
distance of 10mm between the disc and the nozzle  (16) Ultrasonic cleansing of zirconia disc after sandblasting  (17) Placement of specimens in cylindrical chamber 46  (18) The glow discharge of plasma inside the cylindrical chamber  (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component  (21) Quanta scanning electron microscope 48	(14)	Air abrasion of zirconia disc	45
sandblasting  (17) Placement of specimens in cylindrical chamber 46  (18) The glow discharge of plasma inside the cylindrical chamber  (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component  (21) Quanta scanning electron microscope 48	(15)	-	45
(18) The glow discharge of plasma inside the cylindrical chamber  (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component  (21) Quanta scanning electron microscope 48	(16)		45
Chamber  (19) Plasma equipment 47  (20) Schematic drawing showing the equipment component  (21) Quanta scanning electron microscope 48	(17)	Placement of specimens in cylindrical chamber	46
(20) Schematic drawing showing the equipment component  (21) Quanta scanning electron microscope 48	(18)		46
(21) Quanta scanning electron microscope 48	(19)	Plasma equipment	47
	(20)		47
(22) Gold spattered zirconia sample 48	(21)	Quanta scanning electron microscope	48
	(22)	Gold spattered zirconia sample	48

(22)		
(23)	Teeth embedded in acrylic resin blocks exposing the experimental surface	4.
(24)	Plastic mounting template	4
(25)	Application of 35% phosphoric acid etchant on the exposed enamel surface of the specimen	5
(26)	Leaving the phosphoric acid etchant for 15 seconds	5
(27)	Application of SA cement plus on the treated surface of the disc	5
(28)	Placement of the treated disc surface covered with the resin cement above the tooth specimen	5
(29)	Constant load of 1Kg was placed above the specimen	5
(30)	Light curing of the specimen	5
(31)	Thermo-cycling machine	5.
(32)	universal testing machine	5.
(33)	A mono-beveled chisel shaped metallic rod parallel to the interface of the bonding surface.	5.
(34)	Bar chart representing mean and standard deviation values for shear bond strength of surface treatments regardless of thermocycling	5
(35)	a Bar chart representing mean and standard deviation values for shear bond strength before and after thermocycling regardless of surface treatment	5
(36)	Bar chart representing mean and standard deviation values for shear bond strength with different interactions of variables	5

(37)	Scanning electron microscope image of control group 3000X	60
(38)	Scanning electron microscope image of air abrasion group 3000X	61
(39)	Scanning electron microscope image of non- thermal plasma group 3000x	62
(40)	Energy dispersive spectroscopy of control group	63
(41)	Energy dispersive spectroscopy of air abrasion group	64
(42)	Energy dispersive spectroscopy of non-thermal plasma group	65

### **List of Tables**

table	<i>Item</i>	Page
(1)	Materials used in this study	35
(2)	Chemical composition of the super translucent multilayered Zirconia (STML)	36
(3)	Technical data of katana Zirconia STML ceramic discs	37
(4)	Chemical composition of Panavia SA cement plus	37
(5)	Sintering cycle	42
<b>(6</b> )	Samples grouping	44
(7)	Two-way ANOVA results for the effect of different variables on mean shear bond strength	55
(8)	The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between shear bond strength of surface treatments regardless of thermocycling	55
(9)	The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between shear bond strength before and after thermocycling regardless of surface treatment	56
(10)	The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between shear bond strength values with different interactions	58

# Introduction

#### **Introduction**

For many years, it was known that metal ceramic restorations used in dentistry combine both the esthetic properties of ceramic material and mechanical properties of metals. But with the use of metals either as a full metal restoration or in combination with ceramics, some drawbacks have been showed that lead to patient dentist un-satisfaction.

All ceramic restorations are metal free alternatives from the metal ceramic restorations. Zirconium oxide (ZrO<sub>2</sub>) ceramics is considered the strongest ceramic material that has been used in the recent years. It is characterized by high fracture resistance and durable viability in comparison to porcelain or metallic substrates (1).

High strength ceramic restorations have been popularly used in dentistry due to increasing patient's demands for a more esthetic restoration with the new era of dental technology and research. They are biocompatible materials, highly esthetic, and fulfill highly mechanical properties.

With the evolution of zirconia materials, zirconia-based restorations have been more frequently used. Until even a few years ago it was challenging to produce a zirconia restoration that accurately replicates the natural translucency and opalescence of real teeth. Now, the newest generation of materials can provide patients with even better treatment outcomes due to increased translucency, better wear characteristics, and strength with significantly improved translucency, the technician can fabricate restorations that more closely replicate natural teeth. (2)

This ceramic restorative material by the help of computer aided design and computer aided manufacturer(CAD/CAM) innovation has been widely used in dentistry, zirconia can be used in so many clinical aspects as orthodontic brackets, post and cores, implants, and fixed partial dentures (3).

Adhesive cementation of dental restorations is highly requested, since it enhances retention, marginal adaptation, fracture resistance, and decreases the possibility of recurrent decay and require more conservative tooth preparation. Different surface treatments are launched to improve the adhesion and retention to the tooth structure.

The conventional way of cementation which includes H-F acid and salinization treatment for the surface of zirconia before adhesive cementation is not effective to achieve proper adhesion due to the lack of silica and glass phase in the microstructure. Due to the difficulty in achieving chemical and mechanical bonding with zirconia, alternative methods have been performed.

The most commonly used micromechanical surface treatment method is air abrasion using alumina oxide particles to improve mechanical bonding with abrasive particle size ranging from 50 to 250 µm. (1) Another form of surface treatment to zirconia ceramics is the use of non-thermal plasma treatment (NTP) to improve the surface chemistry of zirconia for bonding/cementation mechanism. Non thermal plasma is a partially ionized gas containing mixture of ions, electrons, free radicals in non-equilibrium state to be applied to the ceramic surface. This treatment produces a thin silica layer that chemically changes the surface of ceramics improving its wettability and enhancing the bond strength by creating chemical active sites. (4)

# Review of Literature