Faculty of Manufacturing Engineering

EFFECT OF HEAT TREATMENT ON COMPRESSIVE FRACTURE STRENGTH AND MICROSTRUCTURE OF CNC MILLED ZIRCONIA DENTAL RESTORATION

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A thesis submitted in fulfilment of the requirements for Master of Manufacturing Engineering (Industrial Engineering)

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DECLARATION

I declare that this thesis, headed "Effect of Heat Treatment on Compressive Fracture Strength and Microstructure of CNC Milled Zirconia Dental Restoration," is the result of my own study. The thesis was not accepted for any of the applicants, and it is not presently being submitted for any further candidates.

Signature : …………………………………..

Name : Mira Mazlina binti Mahdzir

Date : 28th January 2022

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Industrial Engineering

Signature :………………………

Name :………………………

Date :………………………

DEDICATION

To my beloved father, Mahdzir Bin Ahmad,

My precious mother, Norzila binti Abdul Kader,

My respected supervisor, Dr Rahimah binti Hj. Abdul Hamid,

I want to thank for all those supports, motivation, and guidance during the journey of my master project.

ABSTRAK

Zirkonia biasanya digunakan dalam pemulihan mahkota gigi kerana kekuatannya yang tinggi, sifat tidak reaktif dan estetik berbanding bahan lain. Mahkota zirkonia yang direka menggunakan mesin pengilangan CNC boleh menghasilkan produk akhir dengan ketepatan yang tinggi dan kurang memakan masa. Walau bagaimanapun, proses pengilangan menjejaskan kualiti permukaan zirkonia, hasil daripada sentuhan alat dan bahan kerja semasa operasi pengilangan. Ini boleh menyebabkan gangguan pada struktur mikro dan menjejaskan keliatan patah mahkota zirkonia. Kemerosotan ini menjadi lebih ketara apabila zirkonia tidak dirawat haba, tetapi suhu rawatan haba yang sesuai yang boleh meningkatkan kualiti permukaan pemulihan gigi secara serentak tidak diketahui. Oleh itu, matlamat kajian ini adalah untuk menganalisis kekuatan patah mampatan pemulihan gigi zirkonia giling CNC selepas terdedah kepada pelbagai suhu rawatan haba, dan untuk mengkaji keberkesanan rawatan haba dalam meningkatkan kualiti permukaan pemulihan gigi. Oleh itu, lapan belas sampel daripada zirkonia Y-TZP dikelompokkan kepada dua kumpulan, iaitu kumpulan yang dirawat haba dan kumpulan tidak dirawat. Tiga sampel ditetapkan untuk yang tidak dirawat dan baki 15 sampel dibahagikan kepada 3 kumpulan suhu berbeza iaitu 1400°C, 1500°C dan 1600°C, dengan tiga sampel setiap tetapan suhu. Selepas pemanasan, kekasaran permukaan setiap sampel diambil. Kemudian, spesimen tertakluk kepada ujian beban tunggal untuk menentukan kekuatan patah mampatannya. Seterusnya, spesimen disalut sputter untuk meningkatkan imej sebelum menganalisis fraktografi di bawah mesin SEM. Kajian ini menunjukkan bahawa rawatan haba mempengaruhi keliatan patah mampatan dan kekasaran permukaan berbanding sampel yang tidak dirawat. Namun begitu, tiada perbezaan ketara dapat dilihat apabila suhu meningkat. Namun begitu, ia menunjukkan saiz butiran bertambah apabila suhu meningkat. Oleh itu, suhu optimum untuk mencapai keliatan patah mampatan yang lebih baik dan kualiti permukaan tidak dapat ditentukan. Untuk kajian akan datang, mesin CNC mestilah lebih mesra pengguna dan bermaklumat data semasa membuat mahkota pergigian. Sementara itu, untuk menentukan kekasaran permukaan spesimen, profilometer optik harus digunakan pada masa hadapan. Relau rawatan pemanasan harus mempunyai kadar pemanasan dan penyejukan untuk setiap suhu. Akhir sekali, memandangkan eksperimen ini tidak membuahkan hasil tentang fasa transformasi sampel selepas dirawat haba, X-Ray Difraction (XRD) harus digunakan dalam penyelidikan akan datang.

ABSTRACT

Zirconia is commonly used in dental crown restoration due to its high strength, non-reactive and aesthetic properties compared to other materials. Zirconia crown fabricated using CNC milling machine can produce a final product with high accuracy and less time-consuming. However, the milling process affects the quality of the zirconia's surface, as a result of the tool and workpiece contact during the milling operation. This can lead to disruption of the microstructure and affect the fracture toughness of the zirconia crown. This deterioration becomes more apparent when the zirconia is not heat treated, but the suitable heat treatment temperature which can simultaneously improve the surface quality of the dental restoration is not known. Hence, the aims of this study are to analyse the compressive fracture strength of CNC milled zirconia dental restorations after being exposed to various heat-treatment temperatures, and to study the effectiveness of heat treatment in improving the surface quality of dental restoration. Therefore, eighteen samples from Y-TZP zirconia are grouped into two groups, which are the heat-treated and the untreated group. Three samples are set for the untreated and the remaining 15 samples are divided into 3 groups of different temperature which are 1400°C, 1500°C and 1600°C, with three samples per temperature setting. After heating, surface roughness of each sample is taken. Then, specimens are subjected to a single-load-testing to determine their compressive fracture strength. Next, the specimens are sputter coated to enhance the image before analysing the fractography under the SEM machine. This study shows that heat treatment does influence the compressive fracture toughness and surface roughness compared to untreated samples. However, there is no significant differences can be seen when the temperature increases. Nevertheless, it shows that the grain size increases when the temperature increases. Hence, optimum temperature to achieve better compressive fracture toughness and surface quality could not be determined. For future study, CNC machine must be more user-friendly and data-informative when fabricating the dental crown. Meanwhile, in order to determine the specimen's surface roughness, an optical profilometer should be employed in the future. The heating treatment furnace should have a heating and cooling rate for each temperature. Last but not least, since this experiment yielded no results about the transformation phase of the sample after being heat treated, X-Ray Diffraction (XRD) should be used in future research.

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LIST OF ABBREVATIONS

UTeM	-	Universiti Teknikal Malaysia Melaka
FKP	-	Fakulti Kejuruteraan Pembuatan
CAD/CAM	-	Computer Aided Design & Computer Aided Manufacturing
CNC	-	Computer Numerical Control
3D	-	Three Dimensional
SEM	-	Scanning Electron Microscope
CaO	-	Calcium Oxide
MgO	-	Magnesium Oxide
Y_2O_3	-	Yttrium Oxide
CIP	-	Cold Isostatic Pressing
SC	-	Slip Casting
Mg-PSZ	-	Magnesium Partially Stabilized Zirconia
ZTA	-	Zirconia Toughened Alumina
3Y-TZP	-	Yttria Full Stabilized Tetragonal Zirconia Polycrystal
ZrO_2	-	Zirconia Oxide
ZTA	-	Zirconia Toughened Alumina
CVD	-	Chemical Vapour Deposition
LTD	-	Low-Temperature Degradation
CRM	-	CAD Reference Model
Au	-	Gold
Pd	-	Palladium
EDS	-	Energy Dispersive Spectroscopy
TC	-	Thermal cycling
MC	-	Mechanical Cycling

MZC	-	Monolithic Zirconia Crown
RBZ	-	Resin-Based Composite
POM-C	-	Polyoxymethylene-Copolymer
MZr	-	Monolithic Zirconia
MLD	-	Monolithic Lithium Disilicate
MZLS	-	Monolithic Zirconia-Reinforced Lithium Silicate
HL	-	Hackle Line
AL	-	Arrest Line
FAST	-	Field-Assisted Sintering Technique
ARG	-	Argen Z Esthetic
CER	-	Ceramill Zolid PS
CON	-	Cercon HT
COP	-	Copran Monolith
DDB	-	DD Bio ZX2
NEX	-	NexxZr-T
ZIR	-	Zirlux 16+
XRD	-	X-Ray Diffraction Analysis
FESEM	-	Field Emission Scanning Electron
Al_2O_3	-	Aluminum Oxide
HfO_2	-	Hafnium Oxide
Ag	-	Silver
Co	-	Cobolt
Cr	-	Chromium
Cu	-	Copper
Fe	-	Iron
Mo	-	Molybdenum
Ni	-	Nickel
Pt	-	Platinum
Ta	-	Tantalum
W	-	Tungsten

LIST OF SYMBOLS

°C	-	Temperature
°	-	Degree
gcm^{-3}	-	Gram per Cubic Centimetre
%	-	Percentage
µ	-	micro
μm^2	-	square micrometre
Ra	-	Surface Roughness
±	-	plus minus
cm	-	centimetre
m	-	metre
h	-	hour
s	-	second
Hz	-	frequency
mm/m	-	millimeter per minute
≥	-	more than equal
≤	-	less than equal
$MPam^{1/2}$	-	Mega pascal per metre square
MPa	-	Mega pascal
G-g	-	Force
H_v	-	Vickers hardness
P	-	Load
Kg	-	Kilogram
D	-	Diameter

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Dental crown restoration is the great way to enhance the colour of the teeth where the technologies nowadays can make the colour of the crown to natural colour of actual teeth. Besides, people who have a gap between teeth can choose dental implant to restore the space (Horne, 2020). There are two types of techniques for dental restoration which are conventional and atraumatic. Conventional method requires expensive material, electrical supply and trained dental health personal while atraumatic uses limited electrical driven equipment and applies adhesive material such cements and resins for dental restoration (Dorri *et al.*, 2017).

There are three types of materials commonly used for dental crown restoration: all-metal, all-ceramic and metal-ceramic (Rathi and Verma, 2018). In all metal, mostly it is under the category of metal alloy and titanium alloy. However, there are several disadvantages of these alloy which are porous casting and corrosion. The corrosion that happens in the oral cavity usually is wet corrosion due to the humid environment in the mouth. In addition, the release of metal ions will negatively affect the body (Noumbissi, Scarano and Gupta, 2019).

Meanwhile, metal ceramic crown is a composite of metal substructure and porcelain veneer. This porcelain veneer could resist the flexure where the tensile strength can be developed among the veneer resulted to prevent of fracture. Common alloy used for metal ceramic crown is palladium alloy where it can provide high modulus elasticity. However, the problem is when silver compound in palladium alloy diffused resulted to the greenish hue of porcelain veneer. Metal ceramic dental crown is much more different compared to ceramic crown because it might trigger allergic reaction towards metal on some people (Rathi and Verma, 2018).

In addition, there are two group of dental ceramic which are glass and polycrystalline. In glass ceramics, polycrystals or crystalline act as a filler to enhance the monolithic restoration of the dental crown. Leucite, lithium disilicate, and alumina are examples of polycrystals used to strengthen the matrix of glass ceramics. Zirconia and alumina are the component that include in the creation of polycrystalline dental crown restoration (Butt *et al.*, 2019). Among those three materials, ceramic is the chosen material for dental application because it has chemical stability, biocompatibility, high compressive and aesthetic and CAD/CAM is used to manufacture the dental crown to achieve high efficiency (Cheng, Yang and Yan, 2018).

Computer Aided Design or Computer Aided Manufacturing (CAD/CAM) is one of the technologies uses on restorative dentistry that has given a big impact towards the discipline of dentistry. The presence of this technology has contributed to the technology from machine copy milling to fully computer-controlled system allows the crowns and bridges to be manufactured easily. The user only needs to create the design of crown restoration through the CAD/CAM software then converted to CAD/CAM machine before milling process is continued (Alghazzawi, 2016).

CAD/CAM provides numerous advantages towards dentistry application, especially on dental crown restoration in terms of productivity, precision, accuracy, and better restoration than traditional fillings (Susic, Travar and Susic, 2017). However, previous research mentioned that the manufacturing process of dental crown restoration using

CAD/CAM affects the surface roughness, which contributes to the mechanical behaviour that is important for fracture strength (Fraga et al., 2017). Besides, the type of axis for the milling process also affects the accuracy of the dental crown because different axis have different levels of precision (Al-Aali *et al.*, 2020).

The most common method for fabricating zirconia restorations is to use partially sintered zirconia blanks, made in a semi-sintered, porous form using soft-milling procedures, making them easier to mill in computer-assisted manufacturing (CAM) machine. Zirconia prosthesis must be sintered after milling to get the optimum density and strength. This sintering technique is frequently linked with a volumetric shrinkage of 20% to 30%. In addition, fully sintered blanks have a high strength which requires longer milling durations and increases milling tool attrition. Furthermore, fully sintered zirconia milling produces high surface temperatures, which cause surface damage and defect formation, reducing the expected lifespan of the prosthesis dramatically (Ahmed *et al.*, 2019).

Fabrication of zirconia creates defect on the surface and one of the procedures that can overcome this drawback is sandblasting. Sandblasting is a typical treatment for cleaning ceramic materials' surfaces and increasing micro retentive structures for the bonding process. It's a delicate procedure in which just a little quantity of surface material is removed and heat and stress on the surface are kept to a minimum (Çağlar and Yanıkoğlu, 2016). Furthermore, heat treatment, polishing, and glazing are commonly used to restore surface smoothness to avoid mechanical performance degradation and prevent tooth wear (Zucuni *et al.*, 2017).

Meanwhile, fracture toughness of zirconia crown also influenced by fabrication process. This is because during fabrication, cracks and flaws and void could happen. These fracture usually happen at the incisal tip of zirconia crown (Abed and Bassim, 2020). Besides, zirconia's microstructural grain size and physical properties are also impacted by sintering (Kaizer *et al.*, 2017). The temperature at which zirconia materials are sintered changes the grain size. Greater grain size achieved is due to the longer of holding time and higher the temperature during sintering. The larger the grain size, the better the fracture toughness (McCullagh *et al.*, 2019).

1.2 Problem Statement

Chewing is the common mechanism for human to eat and it involves the strength of their teeth. Since dental crown restoration is the alternative for people with damaged or broken teeth, they need to ensure that the dental crown is safe and able to sustain high pressure of the load. Hence, the fracture strength of dental crown restoration is an important factor for the chewing mechanism. Fracture toughness that is suitable for clinical testing is estimated 4.7365 ± 2.2676 kN. CAD/CAM affects mechanical behaviour, essential for fracture strength (Fraga et al., 2017). Grain size influenced by the heat treatment is significant for the microstructural properties of zirconia (Sen & Isler, 2020). Zirconia prosthesis must be sintered after milling to get the optimum density and strength (Ahmed et al., 2019). Density is a significant physical property related to the porosity of the microstructure. Generally, a dense ceramic exhibits a packed microstructure with limited porosity. Pores in the microstructure are detrimental to the mechanical properties of dental restoration. Good densification can help to improve the grain growth of a microstructure (Hao et al., 2016).

Moreover, the CNC milling process of zirconia crown created defects and scratches on the surfaces. This destroys the aesthetic value for the zirconia crown, besides affecting its microstructure and physical properties. Hamid et al. (2018) showed a scallop height and scratch grooves effect on the restoration without finishing. CNC milling produces defects and scratches on the surfaces. This destroys the aesthetic value for the zirconia crown, besides affecting its microstructure and physical properties (Matei et al., 2019). Surface roughness also can increase the wear of the material (Buciumeanu et al., 2017). Theoretically, sintering could improve the grain size and simultaneously reduce the porosity. However, the optimum heat treatment temperature to densify the microstructure grains and topography and porosity of the zirconia dental restoration is unknown. According to Miyazaki et al. (2013), a sintering process at 1000˚C to 1100˚C does not affect the zirconia bond. Ting et al. (2019) added, good mechanical properties can be achieved if the zirconia is sintered at a temperature >1500°C but, temperatures >1600°C can lead to excessive grain development and increased porosity (Öztürk and Can, 2019). Therefore, this study sets the sintering temperature to 1400˚C – 1600˚C.

1.3 Research questions

1. Can sinter the milled zirconia restoration improve the surface quality and eliminate the machining effect?
2. Will sintering at the optimum temperature improve the grain size and increase the strength?
3. Will sinter the milled zirconia restoration improve the grain size and simultaneously reduce the porosity?

1.4 Objectives of Study

a) To study the effectiveness of heat treatment in improving the surface quality of dental restoration.
b) To analyse the compressive fracture strength of zirconia dental restorations at different heat-treatment temperatures.
c) To determine the influence of temperature and load on the microstructure grain.

1.5 Scopes of Study

1. The pre-sintered zirconia disc, which is dental zirconia oxide (Y-TZP ZrO_2) is used as the workpiece material. Three types of zirconia disc from different manufacturers with different disc thickness is adopted, which are:
   (a) Product 1 – Samy (98mm x 10mm)
   (b) Product 2 – Zahndent (98mm x 10mm)
   (c) Product 3 – Honchon (98mm x 12mm)
2. The sintering temperature used is set to 1400˚C – 1600˚C due to the limitation of the disc.
3. Porosity analysis was conducted using a densimeter due to the lack of porosity equipment at the laboratory. The Archimedes Principle is explored in this method, where less density indicates a higher porosity level.
4. A cylindrical shape is used in this study for the surface roughness, porosity test, fracture test and fractography analysis; not the actual dental feature, to avoid the difficulty of the surface roughness measurement due to the uneven surfaces of the actual restoration.
5. Holding time is set to 2 hours, according to the manufacturer's recommendation.
6. Each temperature will have two replications to reduce the random errors.