A SURFACE TOPOGRAPHY STUDY OF THE UNTREATED AND HEAT-TREATED ZIRCONIA DENTAL CROWNS MANUFACTURED THROUGH CNC DENTAL MILLING MACHINE

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

CHE WAN JASLAN BIN CHE WAN JAAFAR

FACULTY OF MANUFACTURING ENGINEERING

2021

DECLARATION

I hereby, declared this report entitled "A Surface Topography Study of the Untreated and Heat-Treated Zirconia Dental Crowns Manufactured Through CNC Dental Milling Machine" is the result of my own research except as cited in references.

Signature : ………………………………………

Author's Name : CHE WAN JASLAN BIN CHE WAN JAAFAR

Date : 8 February 2021

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

……………………………………………………

(DR. RAHIMAH BINTI ABDUL HAMID)

DR. RAHIMAH BINTI HJ. ABDUL HAMID
Senior Lecturer
Faculty of Manufacturing Engineering
Universiti Teknikal Malaysia Melaka
Hang Tuah Jaya
76100 Durian Tunggal, Melaka

ABSTRAK

Zirconia adalah salah satu seramik pergigian yang paling banyak digunakan, dan menjadi popular sejak beberapa tahun kebelakangan ini kerana sifat mekaniknya yang sangat baik, yang membolehkannya digunakan sebagai bahan rangka untuk pembuatan mahkota gigi, jambatan atau penggabungan gigi dan implan gigi. Selain itu, penemuan sistem pengilangan medan gigi CAD/CAM telah menjadikan seramik zirkonia sebagai bahan asas untuk pemulihan gigi. Walau bagaimanapun, pemulihan mahkota gigi semasa penggilingan CNC yang tidak dirawat menunjukkan sifat mekanik permukaan yang sederhana kerana pesongan reflektif antara bahan kerja dan alat pemotong semasa proses penggilingan. Tujuan kajian ini adalah untuk membandingkan topologi permukaan pemulihan mahkota gigi yang dirawat haba dan tidak dirawat yang dihasilkan melalui mesin penggilingan gigi CNC. Blok Y-TZP Zirkonia telah digunakan untuk membuat 12 sampel mahkota gigi. Sampel mahkota gigi telah dibahagikan kepada dua kumpulan iaitu 3 sampel untuk mahkota gigi yang tidak dirawat dan 9 sampel untuk mahkota gigi dengan rawatan haba. 9 sampel mahkota gigi telah menjalani rawatan haba pada tiga suhu yang berbeza iaitu 850°C, 950°C, and 1050°C. Pada setiap tetapan suhu, 3 replikasi sampel telah digunakan. Selepas itu, sampel yang dipilih telah diperiksa dengan menggunakan alat ujian kekasaran permukaan. Pengukuran kekasaran permukaan telah dijalankan dengan menggunakan 2 sampel dari setiap kondisi mahkota gigi.Selepas itu, mahkota gigi yang dipilih telah disaluti dengan lapisan emas. Perbandingan permukaan topografi mahkota gigi yang tidak dirawat dan dirawat haba telah dilaksanakan menggunakan mesin pengimbas mikroskop elektron dengan kuasa pembesaran 50x. Keputusan dari pembelajaran ini telah membuktikan bahawa rawatan haba menggunakan suhu 850°C, 950°C, and 1050°C tidak dapat memperbaiki permukaan gigi. Mikrostruktur analisis telah menunjukkan kesan permukaan tidak rata masih lagi kelihatan selepas rawatan haba. Daripada keseluruhan keputusan, suhu yang digunakan adalah tidak mencukupi untuk mengurangkan kekasaran permukaan pada mahkota gigi Zirkonia.

ABSTRACT

Zirconia is one of the dental ceramics most widely used, which has become popular over recent years due to its excellent mechanical properties, which allow it to be used as a frame material to manufacture crowns, bridges or dental abutments and dental implant. On the other hand, the invention of CAD/CAM dental-field milling systems has made zirconia ceramics a standard material for dental restoration. However, the untreated CNC milled restoration shows moderate surface quality because of the reflective deflection between the workpiece and the tool cutter during milling operation. The aim of this study is to compare the surface topology of the untreated and heat-treated dental crown restoration manufactured through CNC dental milling machine. Y-TZP Zirconia block was used to manufacture 12 crown samples. The crown samples were divided into two groups, which 3 samples are for the untreated and 9 samples are for the heat-treated condition. 9 crowns samples were conducted for a heat treatment at three different temperature setting which is 850°C, 950°C, and 1050°C. At each temperature setting, three replication of the sample were adopted. After that, the selected samples were examined using a surface roughness tester. The measurement was carried out using two samples from each condition. After that, the selected crowns were sputter-coated. The surface topography comparison of the untreated and heat-treated crown restoration was executed using the Scanning Electron Microscope (SEM) machine with 50x power magnification. The result from this study showed the heat treatment of 850°C, 950°C, and 1050°C does not improve the surface quality of the crowns. The microstructure analysis shows the scallop-height effects still exist after the heat treatment. In conclusion, the temperature used in this study is not sufficient to improve the surface quality of the Zirconia dental crown. Also, the surface roughness tester plays a significant role in measuring the crown's surface. As a recommendation, a higher temperature in between 1300°C-1500°C shall be used for heat treatment.

DEDICATION

To

My late father, Che Wan Jaafar bin Che Wan Endut

My beloved mother, Nor Isah binti Abdullah

My brothers and sisters, Che Wan Nor Azlina

Che Wan Jasni

Che Wan Junaidi

Che Wan Juliana

Che Wan Nor Aida

for giving me moral support, money, cooperation, encouragement and also understandings

Thank You

ACKNOWLEDGEMENT

In the name of ALLAH, the most gracious, the most merciful, with the highest praise to Allah that I manage to complete this final year project successfully without difficulty.

I would like to express my deep and sincere gratitude to my respected supervisor, Dr. Rahimah binti Abdul Hamid, for giving me the opportunity to do this study and providing invaluable guidance throughout this study. Her kind supervision, sincerity, and motivation have deeply inspired me. She has taught me many kinds of things and exposed me to meaningful experiences during my final year project. It was a great privilege and honor to work and study under her guidance.

Next, a special thanks of gratitude to En Mohd Hanafiah bin Mohd Isa, Pn Siti Aisah binti Khadisah, and En Bahatiar bin Zaid, who have been so warm and kind to provide sincere assistance and good cooperation during the final year project period.

Last but not least, my deepest gratitude and thankfulness are dedicated to my beloved mother for her prayers, support, and sacrifices for educating and preparing me for my future. Also, I express my thanks to my brothers and my sisters for their support and valuable motivation. Not forgetting to all of my friends and all of the people who have helped, support, and contributed to complete my final year project. I have no valuable words to express my thanks, but my heart is still full of favors received from every person. Thank you very much.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

CAD/CAM	-	Computer-Aided Design & Computer-Aided Manufacturing
CNC	-	Computer Numerical Control
SEM	-	Scanning Electron Microscope
FPM	-	Porcelain fused to metal
Au	-	Gold
3D	-	Three-dimensional
FPD	-	Fixed Partial Dentures
Y-TZP	-	Yttria-Stabilized Zirconia
PMMA	-	Poly (methyl methacrylate)
ZrO2	-	Zirconium Dioxide
LMGC	-	Lithium Metasilicate Glass
CIP	-	Cold Isostatic Pressing
LM	-	Light Stereomicroscopy
DCP	-	Direction of Crack Propagation
EDS	-	Energy Dispersive X-Ray Spectroscopy
EBSD	-	Electron Backscatter Diffraction
FEG-SEM	-	Field Emission Gun Scanning Electron Microscopy
PSM	-	Projek Sarjana Muda
STL	-	Stereolithography
AC	-	Alternating Current
FKP	-	Fakulti Kejuruteraan Pembuatan
LCD	-	Liquid Crystal Display
WD	-	Work Distance

LIST OF SYMBOLS

µm	-	Micrometre
m	-	Metre
%	-	Percent
mm	-	Millimetre
MPa	-	Mega Pascal
°C	-	Degree Celsius
nm	-	Nanometre
kg	-	Kilograms
mm/min.	-	Millimetre per Minute
kN	-	Kilo Newton
m	-	Metre
°C/min	-	Degree Celsius per Minute
mm	-	Milimetre
rpm	-	Revolution per Minute
v_c	-	Cutting Speed
a_p	-	Depth of Cut
V_f	-	Feed Rate
Q′	-	Specific Material Removal Rate
Ra	-	Average Roughness, Arichmetic Mean
σ	-	Standard Deviation
Sa	-	Mean Arithmetic Deviation
Rz	-	Average Maximum Peak to Valley
m/s	-	Metre per Second
mm/min	-	Millimeter per Minute
P	-	Dynamic Viscosity
kV	-	Kilovolt
N `	-	Newton
HV	-	High Voltage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Dental crown restorations are made to cover a damaged or cracked tooth. Dental crowns can even be used to dress up a tooth that is very worn or discolored. Basically, a dental crown restoration is defined as a tooth-shaped "cap" that is placed over a damaged or cracked tooth. The purpose of the crowns is to cover the damaged tooth to restore its size and shape, strength, and get better appearances. Other than that, the crowns are made to protect a weak tooth from fracturing or to maintain together parts of a cracked tooth. For children, a crown restoration is usually used on primary teeth to guard a child's teeth at a high risk of tooth decay, especially when a child has trouble keeping up with regular oral hygiene.

In the dentistry sector, many types of material are used for manufacturing of crown restoration. For example, stainless steel crowns are prefabricated crowns primarily used as temporary measures on permanent teeth. The crown covers the tooth or the filling, while the permanent crown is made of a different material. For kids, the stainless steel crown is typically used to fit over the primary tooth that has been ready to fit it. The crown protects the whole tooth and prevents it from further decay. Other than that, there is much other material that is used for a crown restoration, such as metal alloys, porcelain-fused-to-metal, gold, porcelain, lithium disilicate, and Zirconium. Zirconium has become a relatively new material that combines metal strength with porcelain crown aesthetics. High translucent Zirconia and layered Zirconia crowns have recently become a more popular choice.

The Zirconia crowns are made from Zirconia blocks, a type of metal that is incredibly tough, and that is related to titanium, but it is categorized as a ceramic crown.

Crown restoration manufactured from Zirconia has a high demand among people because they provide many advantages compared to other materials. One of the main challenges in the dental field is ensuring the use of biocompatible materials for restoration methods that can stand up with the pressure executed by force during chewing. Zirconia is much stronger than porcelain, which has five times the strength compared to porcelain and other types of crowns. For patients that have fractures, cracks, or other broken crown problems, Zirconia is an excellent choice. Zirconia is much more resistant to stain than ceramic composites or acrylic crowns. The ability to better resist warm, hot, and cold temperatures can help protect against other crowns' often seen hyper-sensitivity. Zirconia crowns have low thermal conductivity and do not pass extreme temperature fluctuations as different crowns have.

For decades, the dental prosthesis has been a typical method of restoration, but like many other medical sectors, the strategy, material and technology that want to create these restorations have evolved. The invention of CAD/CAM dental milling systems has made Zirconia ceramics a standard material for dental restoration. A quick and individual method to manufacture Zirconia dental restorations is provided by the CAD/CAM milling systems. Smoother and hydrophilic surfaces of the CAD/CAM restoration crowns must have more advanced than traditional dentures. The drawbacks of these methods are limited accuracy and the potential of microscopic cracks.

The untreated CNC milled restoration has shown moderate surface roughness because of the reflective deflection between the workpiece and the tool's cutter during milling operation. Surface roughness is one of the critical characteristics of the microbial attraction of tooth surfaces and must be controlled in order to prevent any patient distress during use. The increasing surface roughness between 0.1 and 0.4 μm enhances the microbial adhesion. Kim *et al.* (2015) confirmed that the in-vitro study had shown the different amounts of bacterial adhesion of materials with extremely low surface roughness. Bollen *et al.* (1996) stated the surface roughness of the Ra-cut-off value of 0.2 μm is not affected by bacterial adhesion or colonization, which is less than this Ra-value. Therefore, in this study, the untreated surface roughness and the heat-treated CNC milled Zirconia restorations were examined using SEM machine in order to see the impact of heat treatment towards the surface quality of the crown.

1.2 Problem Statements

The surface roughness of the dental restorations is one of the important factors which are believed could contribute to the accumulation of microbial in the mouth during use. Therefore, it is vital to ensure the surface roughness of any restoration is within the acceptable limit. CNC milled dental crown exhibits a variation of surface roughness, which is mainly depending on the complex profile of the crown, in addition to the physical contact of the tool and the Zirconia block during the machining process. The first hypothesis of this study is there will be some scratch grooves and also a scallop height effect that could be encountered on the untreated crown as the outcome of the milling operation.

In order to overcome these milling operation drawbacks, the heat treatment of the dental crown is expected to reduce the pores and subsequently improve the surface texture. However, the appropriate temperature of the heat treatment is still unknown and could be based on the phase diagram or from the recommendation by other researchers. Therefore, the second hypothesis of this study is there will be an improvement of the surface roughness of the dental crown after the heat treatment. As a result, a study on the surface topography of the untreated and heat-treated dental crown was carried out using a Scanning Electron Microscope (SEM) machine in order to record the differences. On the other hand, a measurement of the surface roughness for both conditions was executed using a surface roughness tester. This was challenging due to the complex geometrical of the dental crown. In addition to the capability of the used surface roughness tester, a selection of where to place the measurement is also important in order to reduce any possible measurement errors.

1.3 Objectives of Study

a) To compare the surface topography of the untreated and heat-treated Zirconia dental crowns using the Scanning Electron Microscope (SEM) machine.

b) To analyze the surface roughness of the untreated and heat-treated dental crown using a surface roughness tester.

1.4 Scopes of Study

a) The Zirconia crown specimen was heated at 850-1050°C for the heat treatment.

b) The number of crown replications is three for each condition due to the expensive Zirconia blocks.

c) A CAD file of the dental crown in the machine was directly used to fabricate the specimens. The scanning of new crowns does not require due to the limitation of a capable 3D scanner.

d) The powder coat used is gold (Au) to coat 12 samples of the dental crown restorations.

e) The measurement of the surface roughness test was repeated five times at each side of the crown's sample. The occlusal surface is not examined due to the complex geometric profile.

f) Two samples were used from each condition during surface roughness measurement, and one sample was used from each condition during the surface topography analysis.

CHAPTER 2

LITERATURE REVIEW

In this chapter, recent theories of dental crown restoration and publications are reviewed. Different means to produce the dental crown have resulted in different mechanical properties in terms of microstructure and strength of the fabricated crowns. Information about these variations has been studied and summarized. In addition to that, a different manufacturing method towards dimensional accuracy, surface roughness and the heat treatment of the fabricated crowns are also reviewed.

2.1 Overview of the Dental Crown

Crown and bridge restoration has been one of the general practitioner's key methods for preserving their shape and function authentically. The recent introduction of integrated Osseo implants has expanded crown and bridge restorations for partially edentulous patients. Dental crowns act as a restoration protecting or coating teeth. This also preserves the standard size, form and color of the tooth. In the meantime, it does not only offer a good look, it also strengthens a tooth. These are two different crown types, one alternative is to cover a tooth that can boost the existing structure and the other one is placed over a damaged tooth. The other choice is the full reconstruction of a dental implant and it is enormously strong and usable with both options.

Aspros (2015) suggested that the metal filling in the teeth is expanding and contracting when people are exposed to hot and cold drinks or food. This can weaken teeth essentially and lead to cracking of the whole tooth. The inlay or onlay is a tooth repair just like a metal lining and even a crown. An inlay is a fill that is placed on the cusp of the tooth, while the onlay is used to preserve the cusp region that stretches over more of the tooth. The onlay of the dental crown is more preferred and has become the main focus in this study. Inlays and onlays are usually made of porcelain, acrylic resin and can also be made of gold. On the other hand, Bammani *et al*. (2012) has illustrated the difference between dental crown and bridges, as shown in Figure 2.1.

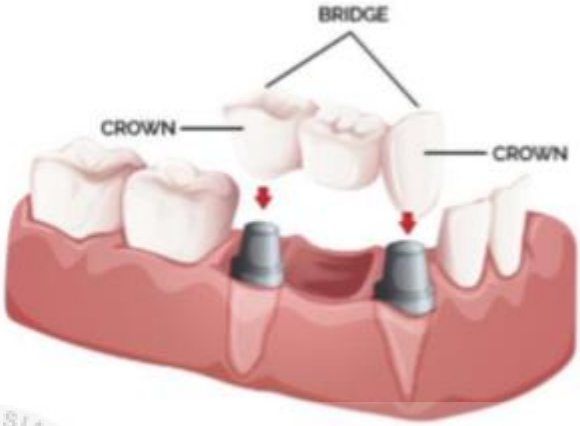


Figure 2.1: Diagram of dental crown and bridges (Bammani *et al*., 2012).

Bammani *et al*. 2012 highlighted that crowns could be classified into two types, which are posterior crowns and anterior crowns. Longer and thinner teeth are built at the front of the mouth, including the lower and top cuspid incisors of both primary and secondary teeth. The posterior crowns are designed for thicker teeth in the bottom of the mouth, the bottom and top molars.

2.2 Dental Crown Material

Different materials have been used for the manufacture of dental crown restorations. For example, Hian (2017) stated the progress in the last ten years in dental ceramic and processing technology is substantial and most advances have been associated with the new CAD-CAM method and new micro-structures. The intermediate properties of the composite materials between pottery and polymer make it a preferred process by means of CAD/CAM due to the simple milling and polishing of its components. Porcelain is one of the dental ceramics used in the production of dental crowns.

In relation to that, Dolidze (2016) describes the most common aesthetic restoration material used is porcelain fused to metal (FPM) in manufactured crown and bridge restoration work because of its excellent mechanical properties. Many dentists are reluctant to use a new or moderately tested form of restoration. However, FPM has already proved itself and has been used in dentistry for more than 50 years. Nonetheless, dental work has started in order to enhance the aesthetic result for the metal-free ceramic restorations. So, the introduction of Zirconia and E-max would make the development of the perfect crown easier. E-max crown is one of the kind of ceramics crown that makes it suitable for its enduring appearance, which comes in a single block of lithium disilicate ceramic and can be graded as a highly prized ceramic for its strength, durability and opaque performance.

According to Alfawaz (2016), due to the high appearance, inertness and biocompatibility, ceramics have become popular as a dental restoration. For dental ceramics, Zirconia is one of the alternatives for ceramic materials used as a dental biomaterial for contemporary restorative dentistry. Zirconia gains clinical popularity due to excellent mechanical features and the simple use of CAD/CAM technology for green manufacturing. Moreover, the previous survey suggests that Zirconia crowns have provided an excellent substitute for restorations of prosthodontics in premolar and molar regions.

2.3 Zirconia Crown Preparation

In this chapter, the preparation of the Zirconia crown is elaborated. Firstly, the scan of the tooth has to be carried out using any 3D optical scanner to acquire an automatically accurate 3D model of the tooth, as discussed by Jang *et al.* (2011). Figure 2.2 shows the resin die and crown manufactured through CNC dental milling machine.

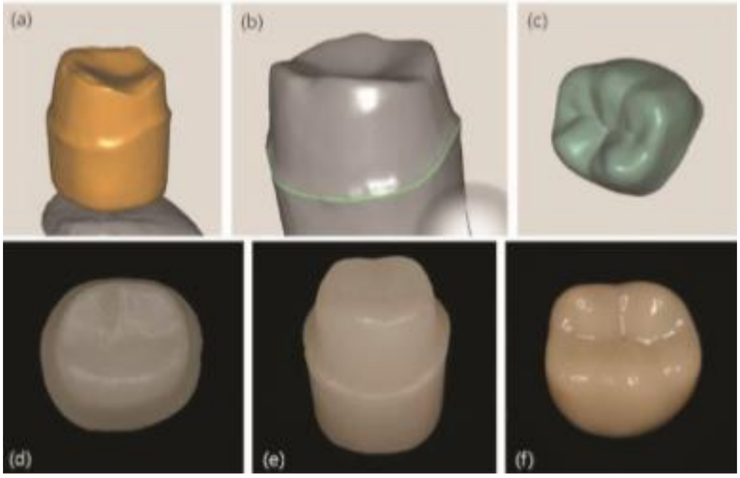


Figure 2.2: (a,b) resin die design in CAD software, (c) dental crown design in CAD software, (d, e) manufactured resin die, (f) manufactured Zirconia crown (Jang *et al.*, 2011).

A dental clinician provided a 24 h in vivo anterior fractured (canine to canine) six-unit fixed partial dentures (FPD). A dental technician in a private laboratory that trained in CAD/CAM techniques and handling Cercon System for the Zirconia framework had manufacture the Zirconia The frame of the Cercon consisted of a Y-TZP sintered with 1350°C and furnished with a porcelain feldspar. According to the dentist, the frame was manually modified by remolding the palatal surface after CAD/CAM machining (Lohbauer *et al.*, 2010). Figure 2.3 shows the anterior Zirconia bridge manufactured from Cercon System.

Figure 2.3: Cercon veneered six-unit anterior Zirconia bridge (Lohbauer *et al.*, 2010).

Cho *et al.* (2011) highlighted in the first mandibular acrylic model tooth, the 3D contour measurements were scanned using Optical Scanner (S600 Zirkonzahn) and then a typical CAD (Zirkonzahn. Modellier, Zirkonzahn, Italy) preparation for the curvature was created at a maximum 6-degree convergence angle (3 grade on each side), a deep 1.0 mm chamfer, 5 mm height preparation and 1.5-2.0 mm occlusal reduction. Occlusal reduction CAD file on the prepared tooth (CAD/CAM M5, Zirkonzahno, Italy) was inserted in a milling machine and acrylic (PMMA) dies were produced and then used as a master die in order to make all-ceramic crowns. Figure 2.4 shows the preparation dimension of the master dies, Zirconia coping design, and the fabricated Zirconia crown.

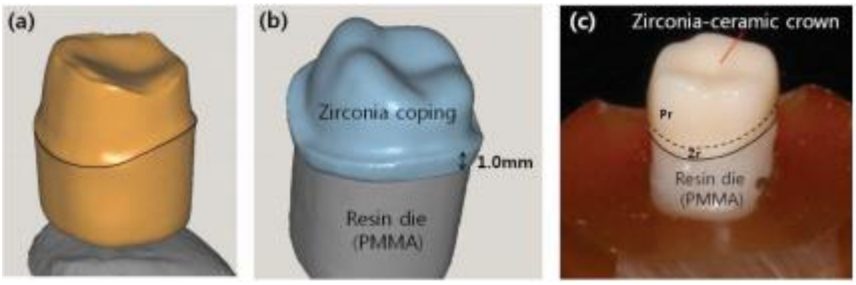


Figure 2.4: (a) Preparation dimensions of the master die, (b) Zirconia coping design with 1mm marginal collar width, (c) Fabricated Zirconia crown (Cho *et al.*, 2011).

Schmitter *et al.* (2014) have discussed the Zirconia veneer was developed with commercial dental software CAD/CAM (Dental Designer; 3Shape, Copenhagen, Denmark) and modified at the edge of the incisor. Performing two modifications, which is, the oral part of the veneer beneath the edge of the incisor was made planar and subsequently, an ellipsoidal bump (4mm× 1.5mm× 0.5mm) was placed at the location of the planned load application point (Schmitter *et al.*, 2014). The CAD-constructed maxillary incisor crown is shown in Figure 2.5.

Figure 2.5: CAD-constructed maxillary incisor crown with the modified area (raised ellipsoid) on the oral side (Schmitter *et al.*, 2014).

Anna *et al.* (2014) stated the two Zirconia blocks Ivoclar IPS e.max ZirCAD (LOT:PX0075) of three bar-shaped specimens (28 mm x 4 mm x 2 mm) and Wieland Zeno Zr were sintered to maximum density using CAD/CAM technology, polished with diamond bur of 3 and 1 m in running water until a mirror-like surface has been reached and cut into four equal pieces (7 mm x 4 mm x 2 mm) with a diamond bur under water cooling.

2.4 Zirconia Crown Milling Process

Hamid *et al.* (2019) conducted the milled dental crown restorations that were performed using Ardenta DT 100 milling machine. The milling process was executed with a carbide tool under dry machining. During this machining experiment, the NexxZr T and also known as dental zirconium oxide (Y-TZP ZrO2) was used as work material for dental crown restorations. The NexxZr T was obtained from Sagemax Bioceramics, Inc. The Zirconia disc was sintered at a temperature between 1500-1530°C.

Jang *et al.* (2011) stated that the prepared tooth CAD file was inserted into a milling machine, and acrylic resin block (PMMA) was milled to render resin die. Crowns were designed to fit into each resin die based on data from plastic models in CAD software. Crowns were designed to fit into each resin die based on data from plastic models in CAD software. Specimens were grouped by occlusal thickness into five classes. Zirconia blocks were milled in a partially sintered process given a shrinkage of 20 percent and then completely sintered at 1600°C for 12 hours. Sandblasting was used at 3 bars with 50 μm aluminum oxide and then glazed the crown's outer surface. CNC milling machine (DWX 50, Roland, Japan) of 29,000 rpm spindle speed was used for 72 cylindrical (7.5 mm diameter and 5 mm height) Zirconia substrates. The structures have been divided into two groups by surface treatment type which is CNC-milling treatment and grit-blasting.

Lan *et al.* (2017) stated that the 24 KMUZ block experiments were selected with 30 mm diameter and 14 mm thickness. From the center of the Zirconia block, each specimen was milled. Figure 2.6 shows the size of the different milling shapes. All samples have been milling by a single CNC system milling center opened with tungsten carbides burs new for each sample during the milling procedure (Ardenta CNC mill, DT100-4A, Tainan, Taiwan). The KMUZ blocks were sintered at a temperature between 1350-1520°C.

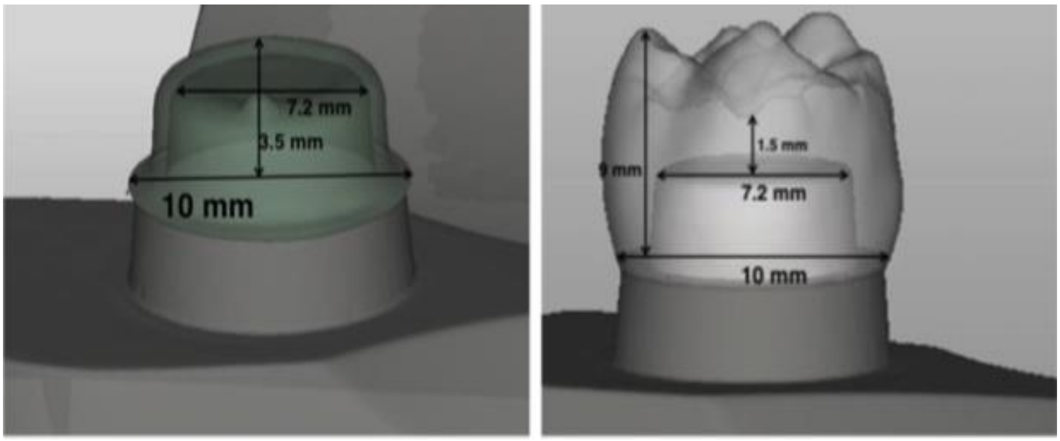


Figure 2.6: The dimension of various milling forms: (left) copping and (right) crown (Lan *et al.*, 2017).

A study by Alao *et al.* (2017) employed the blocks of the lithium metasilicate glass (LMGC) which is IPS e.max CAD was milled using with a stepped bur 12S and cylindrical bur 12S, both of which are of the same composition and properties, using the chairside CAD/CAM milling unit, (CEREC MC XL, Sirona, Germany). Figure 2.7 shows the LMGC block that was milled during this experiment. Wet milling has been performed according to the manufacturer's plan, which simulates crowns' surface milling, the most challenging phase of the CAD/CAM process. The LMGC blocks were undergone for annealing at 480°C for 1 hour.

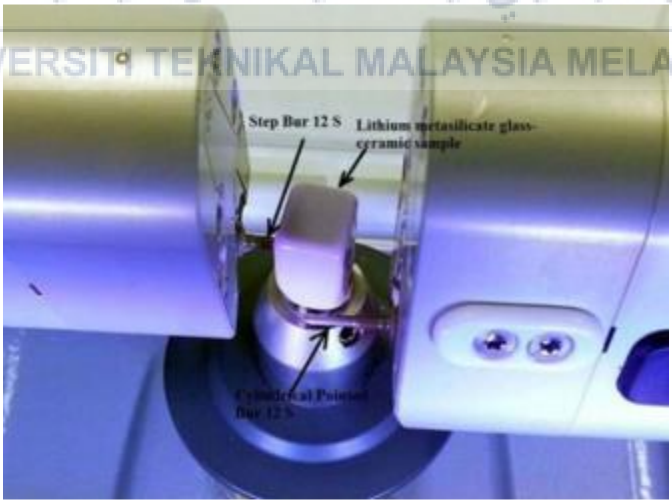


Figure 2.7: Chairside CAD/CAM milling of an LMGC block using two diamond burs (Alao *et al.*, 2017).

Denkena *et al.* (2017) discussed the experiments were carried out using a 5-axis CNC machine tool (Röders RFM 600). The speed of cutting, depth of cutting and abrasive grain are different, the rate of feed is constant. Table 2.1 summarizes the parameters. This study uses a coolant with 5 percent oil-in-water emulsion as a wet grinding technique. The specimens are attached to a workpiece carrier with sticky dental wax. The specimens should be facing the same level before the major experiments can be conducted as preparatory work. This is necessary if comparable results are to be generated without any impact on manual specimen manufacturing.

Table 2.1: Parameter for milling process claimed by (Denkena *et al.*, 2017)

Process Parameter	
Cutting speed	v_c = 10, 30 m/s
Depth of cut	a_p = 0.005, 0.1 mm
Feed rate	V_f = 800 mm/min
Specific material removal rate	Q′ = 0.07; 1.33 mm³/s·mm

Schmitter *et al.* (2014) discussed the veneer was then milled for 48 crowns with an elevated height and the veneer for 16 crowns combined with the Cercon base cast (DeguDent) without an elevated height. The base cast of Cercon is a polyurethane blank to make coping without leaving a residue.

Yttria-stabilized Zirconia was used in order to fabricate the Zirconia block. A five-axis open system CAM (Dentaswiss, Biodenta, Germany) milling machine was used to machine the Zirconia blocks into a crown frame. The Zirconia blocks were conducted for the sintering process between 900-1200°C with 2 hours holding time and the cooling rate at 3°C/min. Figure 2.8 shows the milled Zirconia crown restoration. After the machining process, a FESEM (Zeiss Merlin) Field Emissions electron Microscope observed the morphological characteristics of milled zirconium surfaces to evaluate the effect of zirconium surface quality pre-sintering temperature (Faeizah *et al.*, 2019).

Figure 2.8: CAD/CAM milled Zirconia crown (Faeizah *et al.*, 2019).

According to Kontonasaki *et al.* (2019), the monolithic and central Zirconia frameworks, which can be either pre-sintered or completely sintered, are manufactured through CAD/CAM technologies. CIP Zirconia blocks require a relatively easy and quicker milling procedure which causes less wear of the machining equipment, but frameworks have a 20–25% linear and non-uniform shrinkage during sintering. Based on this research, the flexural strength will decrease if the sintering process above 1550°C.

Mo *et al.* (2005) discussed the crown specimen preparation was designed with a specific dimension and performed on a mandibular first molar acrylic model tooth. The preparation has been tested by laser with a Cerec 3 scan unit (Serial no. 01014) and the crown developed with software V1.60 R980 and fabricated using Cerec 3 procurement and construction unit. The machining setting of all crowns was 1.5 mm for the thickness of the occlusal and the lateral walls.

2.5 Surface Roughness Test

A study on the surface roughness of the CNC milled Zirconia dental restorations is still lacking. Hamid *et al.* (2020) have examined the fabricated CNC milled restorations, but it was only limited to the untreated restorations. The measured roughness characteristics were the average arithmetical value (Ra). These were determined by using a contact-type stylus profilometer Mitutoyo surface tester. In order to set the sample number λ of three, the stylus traversed length, Lt, was set to 0.24 mm, with a cut, λc, to 0.08 mm. Figure 2.10 shows the points listed have been chosen on the buccal and the lingual region. Due to this complicated geometry profile in this area, the occlusal surface was not examined. On the basis of 3 traces each, the average surface roughness has been recorded. During the study, the random error was reduced by three measurement times for a point and the average data was calculated. If more traces are taken for each point, the Ra value can be improved, as the read outer is eliminated so that the standard deviation σ is less than 0.05 for each sample.

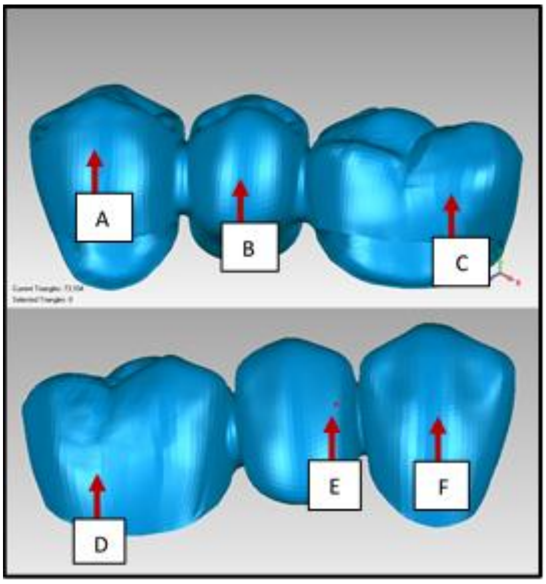


Figure 2.9: The measurement points (Hamid *et al*., 2019).

The Ra values are based upon the style location of the profilometer, as shown in Figure 2.10. This can explain the variation of Ra values in the multiple tracing for each point.

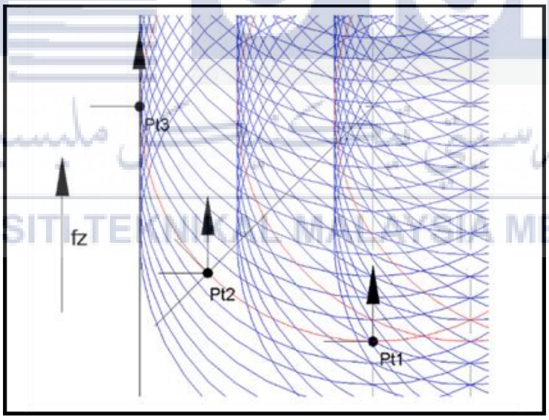


Figure 2.10: Schematic diagram shows the Ra value varies depending on the stylus location (Hamid *et al*., 2019).

Inokoshi *et al*. (2014) stated the surface roughness was determined by an optical interferometer with the magnification of 5x, on the three remaining specimens in each grade. The quantification of the surface rugging parameter 3D Sa (mean arithmetic deviation) was done using the software Vision32. For every specimen, the surface-treated sides were selected from five regions (the effective field of view was 1.210 mm or 0.921 mm). The aim of a surface roughness model was to determine the impact of different surface treatments by creating a linear mixing effects model. The measurement was carried out with three measurements at each region to get the average.

Cheng *et al.* (2018) stated a profilometer (Surfcorder SE-40 G, Kosaka, Tokyo, Japan) with a transverse length of 2.4 mm and a cut-off value von 0.8 mm was used to measure surface roughing (Ra) in the various roughening conditions of blasted Zirconia substrate values. This study of Zirconia surface roughness (Ra), subjected to various blasting parameters, is shown in Figure 2.11. Based on this study, 4 measurements were taken at each selected surface to get the average and 3 specimens were used in order to take the measurement.

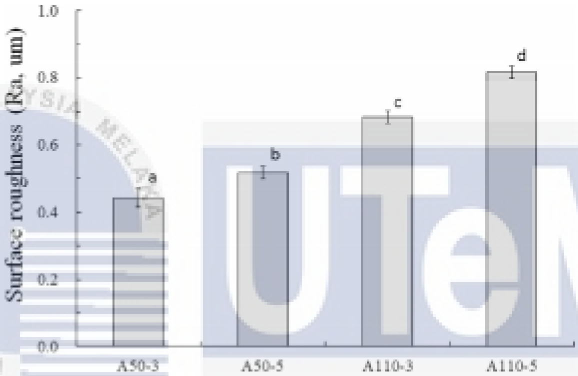


Figure 2.11: The Ra values of the Zirconia specimens with different blast conditions. The same letter are not significant differences (P > 0.05) (Cheng *et al.*, 2018).

Camila *et al.* (2017) stated the disc-shaped Y-TZP specimens were developed according to ISO 6872-2015 guideline for biaxial flexural force testing of ceramic materials. The Y-TZP (yttrium-stabilized tetragonal polycrystalline Zirconia Zenostar T) was used for dental restoration and the specimen was polished, sintered, and inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper) to ensure the dimensions were compliant with ISO 6872-2015 and randomly applied to the assessment of surface post-processing treatments. The roughness analysis on a standardized profilometer by considering the parameter of ISO:4287-1997 (Ra and Rz) has been performed on all samples of each evaluated state. Three readings were executed per specimen in the opposite direction of the one used during polishing. In two consecutive stages, polishing was performed to produce a perfect surface. The polishing treatment was carried out using fine (#3101F – grit size 46 µm) and extra-fine (#3101FF – grit size 30 µm diamond burs. The use of Optrafine System (Ivoclar Vivadent) was required. This polishing kit mainly consists of two tips and a nylon brush which is used to produce a smooth surface by combining with a diamond paste. The specimen was attached to a metal-based and the region to be polished was divided into two regions, taking into account the size of the polishing tips. This step procedure was carried out for 25 seconds at each region for each selected tips. Table 2.2 shows the variation of surface roughness tester and measurement taken at each selected surface used by other researchers.

Table 2.2: Method used by other researchers for surface roughness test

Source	Device used	The measurement taken at each selected surface to get the average
Hamid *et al*., (2019)	Profilometer (Mitutoyo surface roughness tester)	3 times
Masanao *et al*., (2014)	Optical interferometer	3 times
Chih-Wen *et al*., (2018)	Profilometer (Surfcorder SE-40 G)	4 times
Camila *et al*., (2017)	Digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper	3 times

2.6 Surface Topography Analysis (SEM)

Scanning Electron Microscope (SEM) system has been used to examine the surface structure of the dental restoration. Hamid *et al*. (2019) study the surface topography of the surface of the Zirconia untreated restoration. Initially, the sample was sputtered to prevent the non-metal sample from discharging electrostatically. Carl Zeiss Evo 50 at 15.00kV speeding voltage for 100x and 500x secondary electron magnification power was used. The untreated surface of the milled Zirconia has shown the scallop height effect from SEM images. The machined surface profile is determined by the reflective deflection during processing between the device and the workpiece. As the outcome of the SEM, the micro-pitting was shown in the image with 500x power magnification due to the compaction process of Zirconia-powder. Figure 2.12 shows the scratch grooves and micro-pitting after the untreated restorations and Figure 2.13 illustrates the restoration images and the scallop height effect as the outcome of the milling process.

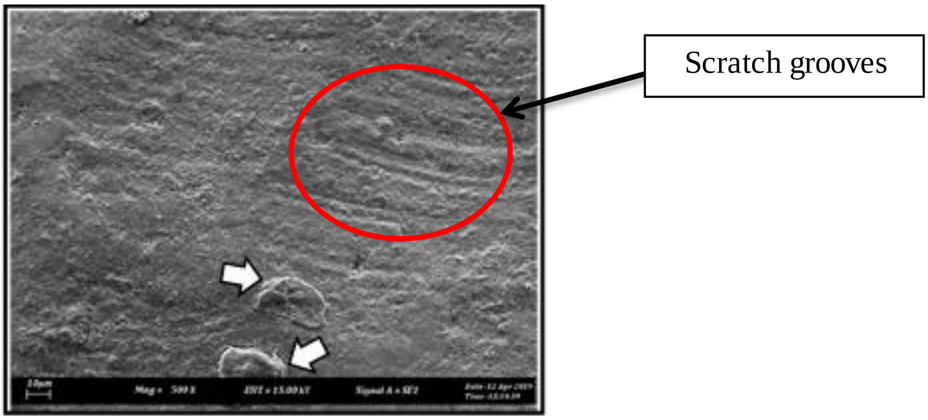


Figure 2.12: SEM image with scratch grooves of specimen (x500). Micro-pitting is shown in the arrow (Hamid *et al*., 2019).

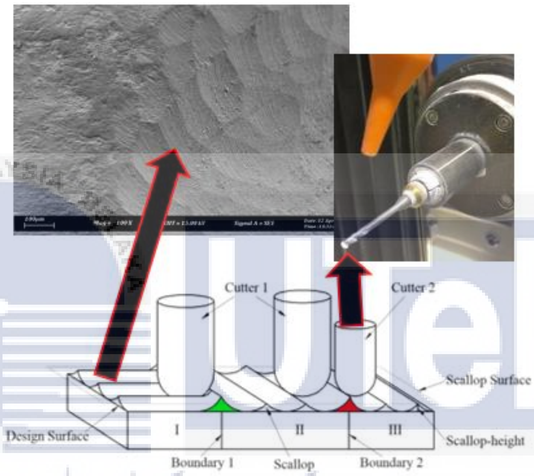


Figure 2.13: Restoration images and the scallop height effect as the outcome of the milling process with 100x magnification (Hamid *et al*., 2019).

Cheng *et al*. (2018) showed the surface morphology of the slashing Zirconia and interconnection between Zirconia and veneer ceramics. The fractured Zirconia surface was examined using an optical microscope with an accelerating voltage of 3.0 kV to assess the fracture mode of the specimens after a shear strength test. In the measurement of the remaining amounts of the veneer ceramics on the broken surface, image analysis software has been used. Figure 2.14 shows the image of surface morphology after the sintering and polishing process. The findings of fracture surface analysis showed that the fracture of the specimens mainly occurred in veneer ceramics and that the cross-linking between veneering and Zirconia was occurring.

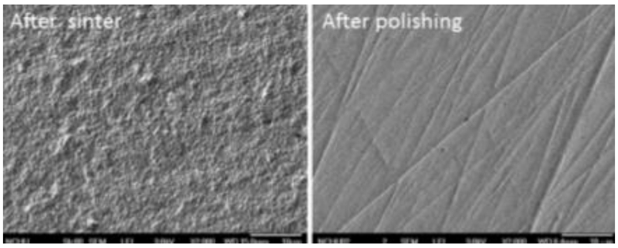


Figure 2.14: SEM photomicrographs with 2000x magnifications of blasted Zirconia surfaces showing surface morphology after sintering and after polishing process (Cheng *et al.*, 2018).

Lohbauer *et al.* (2010) investigated a systematic approach with a light stereomicroscope (LM) was taken to perform a fractoral analysis of the two retrieved fragments as well as scanning electron microscopy (SEM). The broken parts were cleaned 10 min in an ultrasound alcohol bath prior to the microscopic investigation. The macroscopic aspect was checked by the LM in different lighting conditions. For characterization of morphology, microstructure and fractured surface fractures, SEM (Leitz ISI SR 50, Akashi, Japan) was used. At low magnifications, the Zirconia core shows the arrows of two clearly defined lines of arrest and twist hackle, as shown in Figure 2.15. These two features that are easy to see provide a clear indication of the direction of crack propagation (dcp) from the top (incisal) to the bottom (gingival). In fact, the lines of arrest are dependent upon the spread of the crack and the origin of the crack is on the concave side, that's mean, upwards, at the tip of the incisal core. A big pore is in the veneering ceramic, next to those two arrest lines.

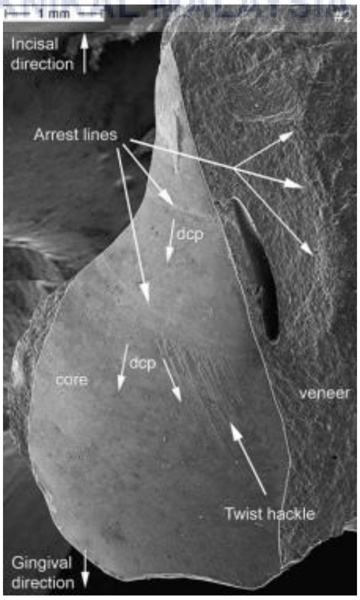


Figure 2.15: Fractographic characteristics are indicated as lines of arrest and twist hackle. The direction of crack propagation (dcp) is shown (Lohbauer *et al.*, 2010).

Alao *et al.* (2017) discussed the specimens were gold coated before the topography analysis. In order to study surface fracture, morphology and removal mechanisms, each surface processed was also seen under SEM (JEOL JSM 5410LV, Tokyo, Japan) with 10kV accelerating voltage. The CAD/CAM-milled LMGC morphology is shown in Figure 2.16(a), which is the details of machining grooves and scratched due to the diamond abrasives. Figure 2.16(b) shows the effect of the shear bands that cause the cracks and irregular fractures and also smeared areas on the surface. The pulverized areas accumulated with the microchips tested on the highly fractured areas are shown in Figure 2.16(c).

Figure 2.16: SEM LMGC Surface micrographs produced by the CAD/CAM process reveal (a) fractional fracturing tracks, fractures and pile-ups induced with the shear band to pulverize material (100x magnification), (b) surface fracture details (1000x magnification), and (c) pulverized and smeared area details (5000x magnification) (Alao *et al.*, 2017).

Guazzato *et al.* (2004) used an electronic scanning microscope (SEM) (XL 30 Philips, Eindhoven, Holland) with 15 kV accelerating voltage to observe the microscopic features of the treated surfaces. In addition, the light microscope and SEM located the origin of the fracture. The surfaces of the unheated specimens have different mechanical treatment functions. Both defections (measure up to 18 mm) and micro-cracks are unevenly distributed on the edge of the scratches, characterized by deformed and displaced material. Figure 2.17 shows the surface without heat treatment.

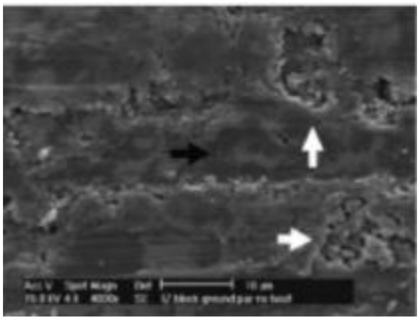


Figure 2.17: SEM images with 4000x magnification without heat treatment. The black arrow indicates the direction of grinding; the white arrows indicate major defects whose shape and orientation seem unrelated to the grinding orientation (Guazzato *et al*., 2004).

According to Denkena *et al*. (2017), the topography of the grinding tool is measured with SEM. The content of the monoclinic phase and resulting residual stress are measured after grinding surface roughness. SEM images of ground surfaces are shown in Figure 2.18 and Figure 2.19. The chip thickness measured for the surface area is 0.42 µm, which means that it is smaller than the critical uncut chip thickness measured at 0.895 µm. The result is a uniformly distributed surface with evenly distributed grinding points. The uncut chip's thickness calculated in Figure 2.19 is 2.52 µm, which makes it higher than the critical uncut chip thickness calculated. The surface displays broken intercrystalline breakouts, high ruggedness on the rims. With uncut chip thickness, the roughness increase.

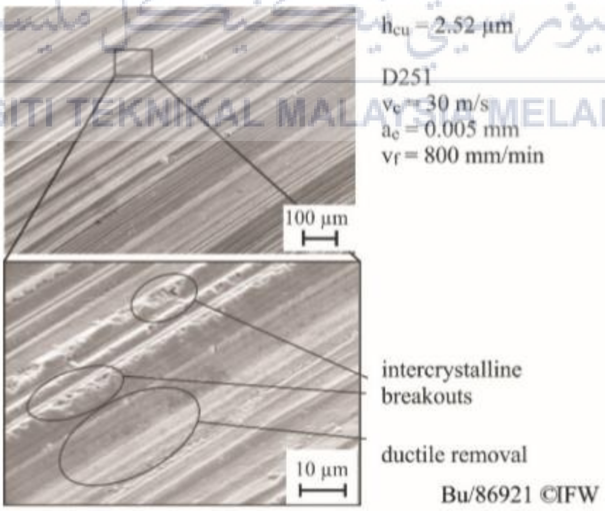


Figure 2.18: Surface topography of the ground surface with hcu = 2.52 µm (Denkena *et al*., 2017).

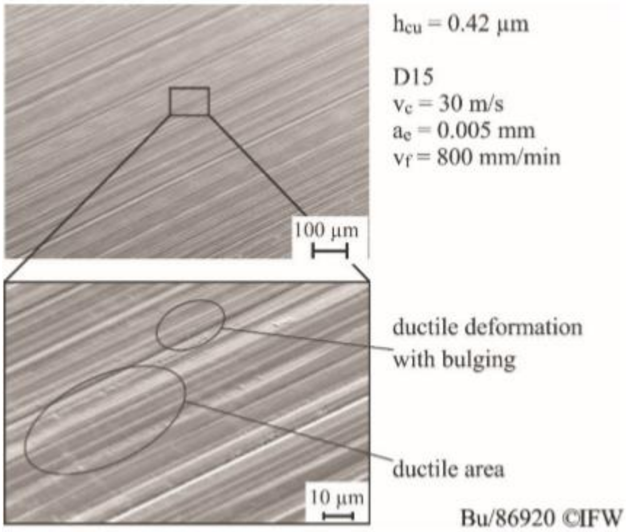


Figure 2.19: Surface topography of ground surface with hcu = 0.42 µm (Denkena *et al.*, 2017).

Smith *et al.* (2011) stated a stereomicroscope (SMZ-140, VWR International,West Chester, PA, USA) and a scanning electron microscope (SEM – Quanta 200, FEI, Hillsboro, OR, USA) were used to establish if the modes of failure were either adhesively, cohesively or mixed with broken surfaces of the samples (SMZ-140 VWR International, WC Chester, USA). Before SEM analysis, all fracture surfaces were coated with a gold sputter. A typical adhesive failure for micro tensile specimens was observed in Figure 2.20.

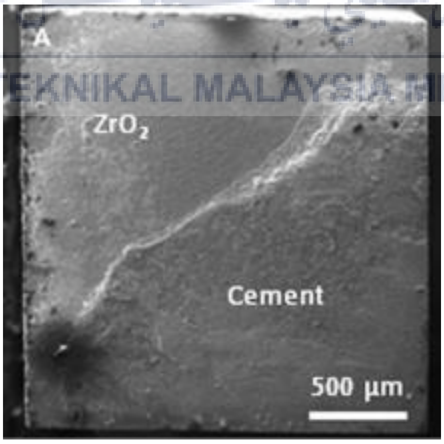
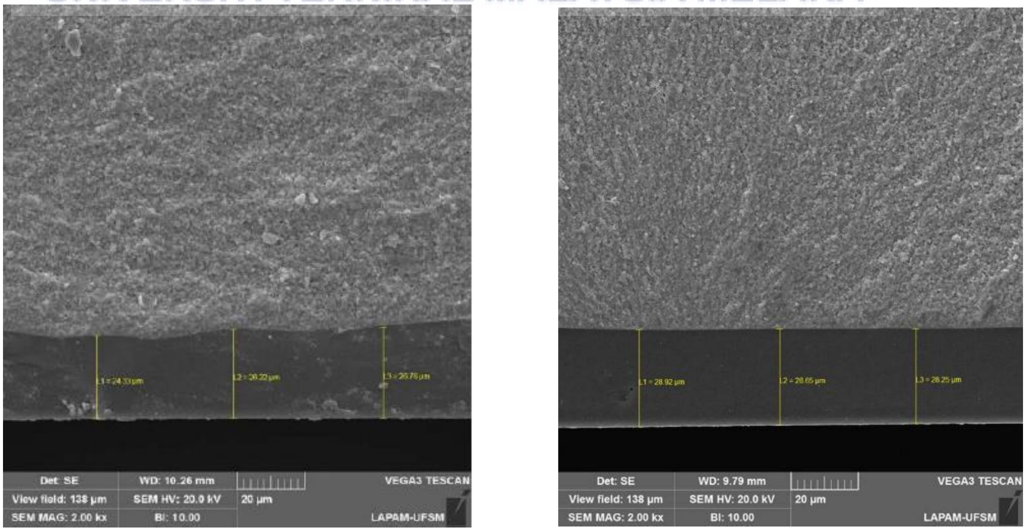


Figure 2.20: SEM of Zirconia fracture surface (Smith *et al.*, 2011).

Schriwer *et al.* (2017) stated to identify the origin of the fracture and to assess the spillage, the broken curves were analyzed with light-microscopic fracture methods. SEM was used to further examine some specimens. To assess the results' clinical relevance, the fractures modes have been compared to the fractures of comparable curves fractured during the clinical function. The microstructure was assessed and the fractographic analysis was validated using a split-crown in each SEM group.

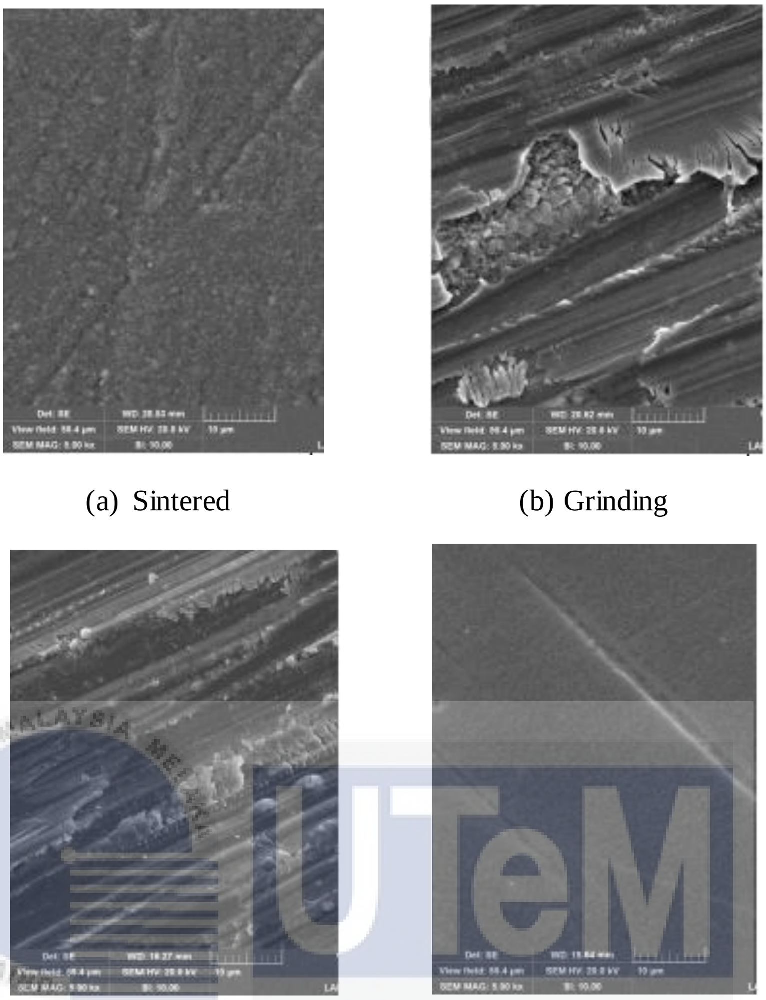
After the heat treatment of the crown, the specimens were carbon-coated before carried out the observation of surface topography analysis using scanning electron microscopy energy dispersive spectroscopy (SEM-EDS). The analysis was performed with 20kV accelerating voltage and 0.4 mA probe current and pure Co was used as an optimizing element. SEM-EDS tests showed similar morphological characteristics with dull surfaces after aging and similar elements such as composition for both pottery products. But three sample weights were reduced by about 25 percent after aging compared to the control samples in analyze of three sample areas, which were presented indicated for IV ceramics (Anna *et al.*, 2014).

Camila *et al.* (2017) stated the specimen was coated with the gold-sputtered and topography analysis was carried out using Scanning Electron Microscopy (SEM - Vega3, Tescan, Czech Republic) with 20kV accelerating voltage and 2000x magnification for cross-sectional topography observation and 5000x power of magnification for the superficial topographical pattern observation as shown in Figure 2.21 and 2.22. Table 2.3 shows the summary of the parameter setting for surface topography analysis carried out by other researchers.



(a) Grinding + glazing (b)Grinding+glazing+polishing

Figure 2.21: Representative micrographs (2000x magnification) demonstrating a thin and apparent homogeneous surface of glaze layer (ranging between 24-28 m) (Camila *et al.*, 2017).



(a) Sintered (b) Grinding

(c) Grinding+heat treatment (d) Grinding+polishing

Figure 2.22: (a) Representative SEM images (5000x magnification) of Y-TZP surface, where it notices that grinding. (b) introduced scratches with varying depths (surface topographical alterations), whose alterations were kept after heat treatment (c). Also, it notes that the polishing and glazing promoted smoother surfaces (d) (Camila *et al*., 2017).

Table 2.3: Parameter setting for SEM analysis by other researchers

Source	**Accelerating voltage (kV)**	**Magnification**
Hamid *et al*., (2019)	15	100x and 500x
Chih-Wen *et al*., (2018)	3	2000x
Alao *et al*., (2017)	10	100x, 100x, and 5000x
Massimiliano *et al*., (2004)	15	4000x
Anna *et al*., (2014),	20	2000x
Camila *et al*., (2017)	20	2000x and 5000x

2.7 Heat Treatment

Alao *et al.* (2017) investigated the glazing process was performed in the programmed dental furnace at a standby temperature of 403 °C, by firing samples without glass beads. The samples were then heated at 90°C/min and kept for 10 min at a heating rate of 820 °C. Then heated up to 840 °C at a heating speed of 30 °C/min and kept for 7 minutes. The samples were then refrigerated to a room temperature of 700 °C.

Guazzato *et al.* (2004) discussed the samples were sandblasted, and then two cycles of heat-treated were conducted, which is first, one minute at 960 ◦C, and second at 940 ◦C in one minute. Such terms are the same as those for the auto-glazing and furnace porcelain that is used for Vita on In-Ceram Zirconia.

Anna *et al.* (2013) carried out the heat treatment test to carry out the mechanical and microstructural properties of Zirconia dental crowns. Zirconia core is veneered with feldspathic porcelain coating to achieve extremely aesthetic Zirconia reconstruction and subsequently firing at high temperatures (750-900 ◦C). Three specimens from 2 Zirconia blocks, which is Ivoclar IPS e.max ZirCAD and Wieland ZENO Zr are milled using CAD/CAM technology and sintered with full density and polished with diamond pastes and then the specimen was heat treated. The applied heat-treatment corresponded to 4 firing cycles with parameter settings (standby temperature 403◦C, pre-heating temperature-time 4s, heating rate 50◦C/min, final temperature 850◦C, vacuum in 450◦C, and vacuum out 749◦C.

Camila *et al.* (2017) stated the heat treatment test could cause the returns m-phase effectively into t-phase and removes existing residual stress. The study's objective is to compare and evaluate the effect of various surface post-processing treatments such as polishing, glazing, heat treatment+polishing, and glazing+polishing on the micromorphology, roughness, phase transformation, and fatigue strength on the Zirconia restorations. The specimen was heat treated on the Vacumat 600MP furnace (Vita Zahnfabrik). The test procedure was executed according to the manufacturer's guideline (closing time 18s, heating rate 65°C/min, final temperature 1050°C, maintenance of 15 min, followed by slow cooling 25°C/min). Table 2.4 shows the summary of the parameter setting for the heat treatment test executed by other researchers.

Table 2.4: Parameter setting for heat treatment test based on other researchers

Source	Alao *et al*., (2017)	Massimiliano *et al*., (2004)	Anna *et al*., (2013)	Camila *et al*., (2017)
Device used for heat treatment test	Dental furnace	Not stated	Kavo autoclave sterilizer 2100	Vacumat 600MP furnace
Standby temperature (◦C)	403	Not stated	403	Not stated
Pre-heating temperature time (s)	Not sated	Not stated	4	Not stated
Heating rate (◦C/min)	90		50	65
Final temperature (◦C)	840	940	850	1050
vacuum in (◦C)	Not stated	Not stated	450	Not stated
vacuum out (◦C)	Not stated	Not stated	749	Not stated
Slow cooling (◦C)	Not stated	Not stated	Not stated	25

2.8 The Effect of Surface Treatment on the Friction and Wear Behaviour

Buciumeanu *et al.* (2017) investigated the procedures for effect surface treatment on the wear behavior and friction of Yttria-tetragonal Zirconia poly-crystal towards the human enamel under reciprocating sliding in the presence of artificial saliva..Reciprocal pin-up layer wear research was carried out for the tooth repaired material pin and layer to reflect the repaired material (Zirconia) for a simplified representation check. The wear characteristics of the tooth/Zirconia tribopair were tested using an interconnecting Bruker-UMT-2 plate tribometer. Figure 2.23 illustrates the schematic representation of the reciprocating wear test.

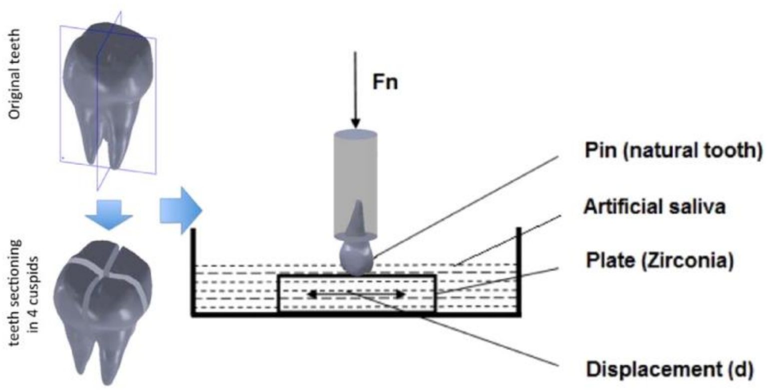


Figure 2.23: Schematic representation of the reciprocating wear test (Buciumeanu *et al*., 2017).

The pins and the Zirconia plates were put in the oven for 2 days at 35°C before and after testing to ensure the same dehydration amount during weighting. Since the maximum force value of a single dent during chewing is between 20 and 120 N, a load of 49N was chosen. The reported enamel microhardness is around 300 HV, while the Zirconia hardness is around 1378 to 1354 HV. The ruggedness was measured using a 3.5 mm measuring length and 0.1 mm/s surface profilometer. For each research sample, six measurements were performed in different areas. Detailed knowledge of X-ray diffraction patterns was collected using the Rietveld system of refinement. To evaluate the wear pattern on the surface in the presence of artificial saliva after reciprocating wear check of Zirconia plates against the dent. Photos were analyzed using (SEM) in SE mode (x100 magnification). FEG-SEM images of the wear line (x100 magnification) are seen in Figure 2.24 after reciprocal wear checks of Zirconia plates against dents in the presence of artificial saliva.

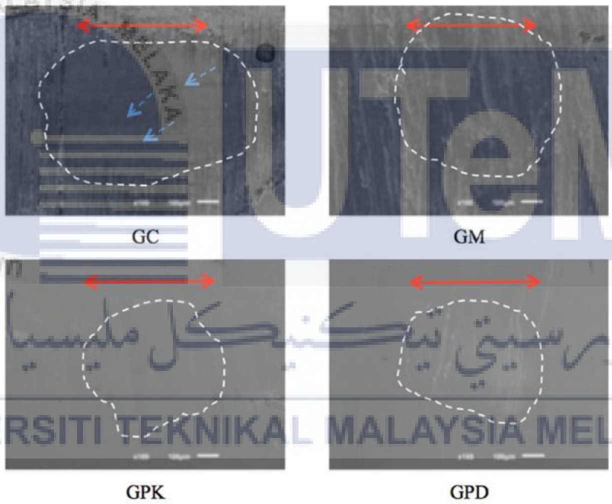


Figure 2.24: FEG-SEM images of the wear track (100x magnification) formed after wear tests (Buciumeanu *et al.*, 2017).

2.9 Summary of the Literature Review

In this chapter, various machines and materials used were studied in order to fabricate the crown restorations, as summarized in Table 2.5. The temperature for the sintering process for the crown restorations also helps in completing this study. Other than that, the other researcher mostly used the Mituyoto surface roughness tester during conducting the surface roughness test. Table 2.2 shows the method that was found for the surface roughness test. The critical part during this study, which is the setting parameter of the heat treatment test, such as final temperature, initial temperate, and temperature rate, was also found. The setting parameter of the heat treatment test from other researchers is summarized in Table 2.4. On the other hand, the magnification used for the SEM was around 100x-5000x magnification.

Table 2.5: Summarization of crown preparation

Source	Machined used for dental restorations	Material used	Sintering temperature(◦C)
Hamid *et al*., (2019)	Ardenta DT 100 milling machine	NexxZr T Zirconia block	1500-1530
Jang *et al*., (2011)	CNC milling machine (DWX 50, Roland, Japan)	Acrylic resin block (PMMA)	1600
Ting-Hsun *et al*., (2017)	Ardenta CNC mill, DT100-4A	KMUZ block	1350-1520
Alao *et al*., (2017)	CEREC MC XL	IPS e.max	480°C (annealing for an hour)
Amat *et al*., (2019)	Five-axis open system CAM (Dentaswiss, Biodenta	Yttria-stabilized Zirconia	900-1200
Eleana *et al*., (2019)	Unknown	CIP Zirconia blocks	1550
Andreas *et al*., (2005)	Cerec 3	Acrylic block	Not stated

CHAPTER 3
METHODOLOGY

This chapter defines the suggested methodology of this research, which consists of the techniques to complete the study. Basically, the process flow of the study is discussed in terms of the machining method, heat treatment, roughness analysis, and the surface topography of the untreated and heat-treated crowns. The parameter settings for each method used was discussed briefly in this section.

3.1 Project Planning

This section explains the procedures need to complete in PSM 1 and PSM 2. In order to achieve the objectives, this study should succeed in the process stage according to the engineering method used, detailed design, machining process and testing method.

Figure 3.1 shows the guidelines of the project from the beginning until the end of this project. It is divided into two-stage whereby PSM 1 consists of the introduction, literature review and methodology while PSM 2 consists of the machining process, result and discussion, and conclusion and recommendation

.

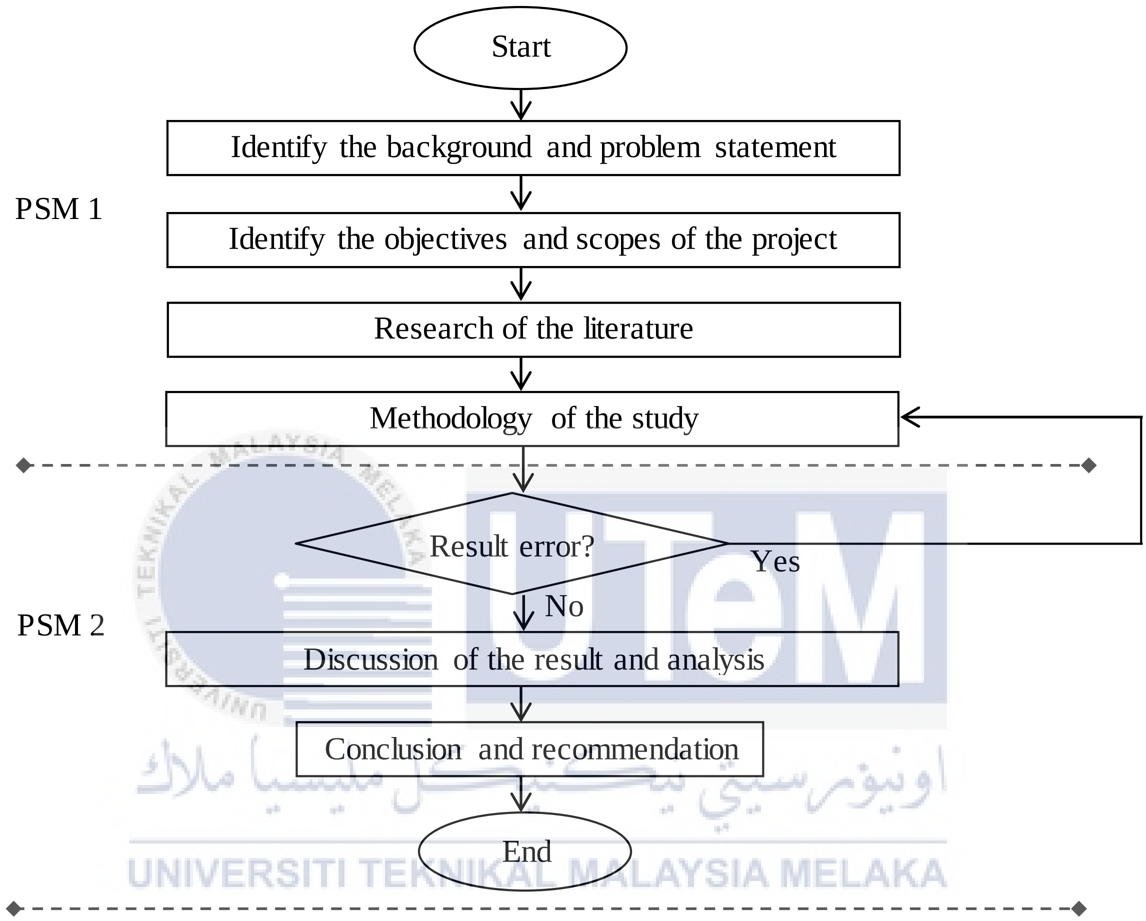


Figure 3.1: Flowchart of the overall project.

3.2 Flowchart of Study

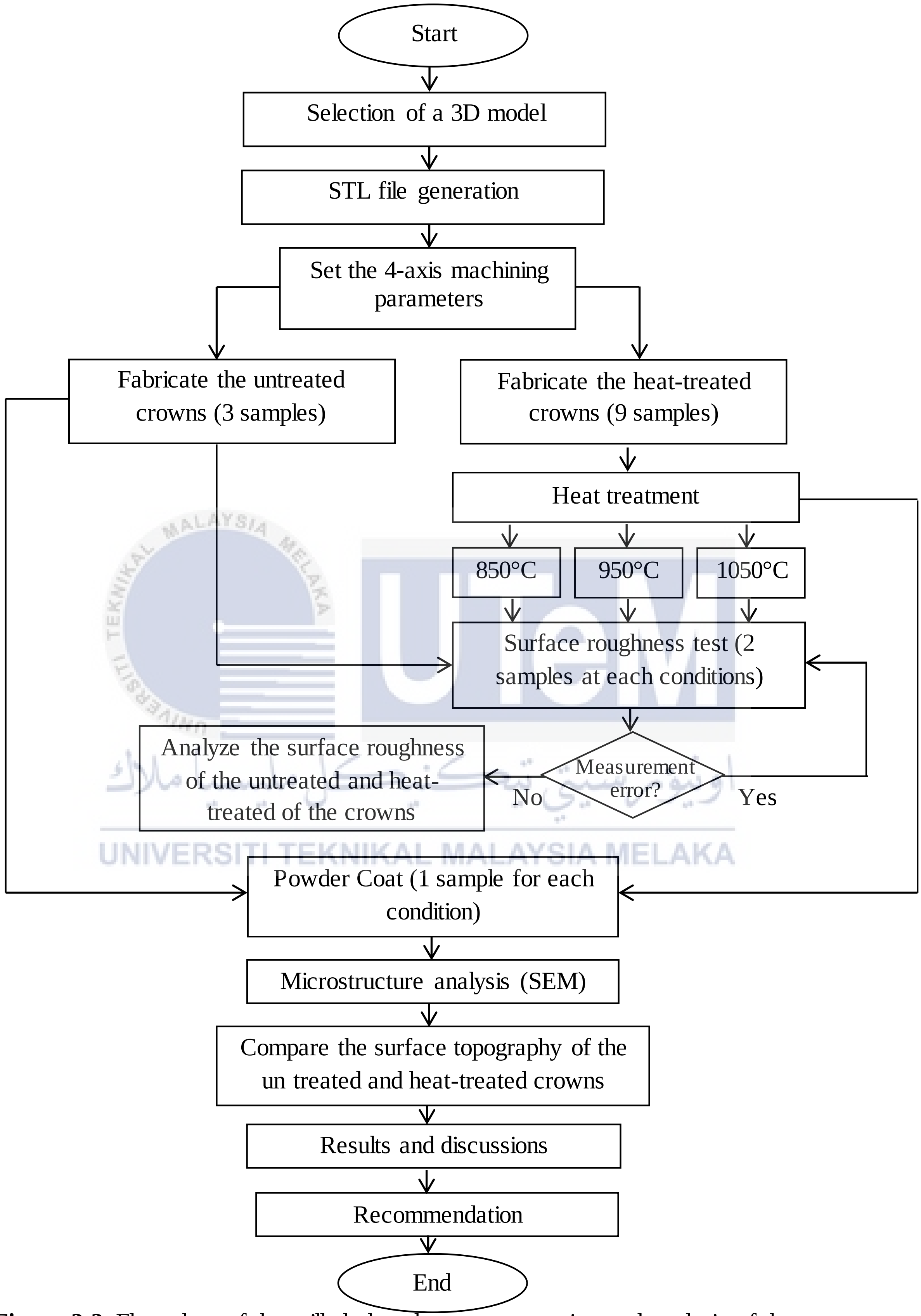


Figure 3.2: Flow chart of the milled dental crown restoration and analysis of the crown restorations.

A flowchart is defined as a formalized graphic that able to represent a sensible arrangement, work or manufacturing process, organization chart, or similar formal arrangement. The flowchart in Figure 3.2 shows the sequence of processes and flow upon completion of this study.

3.3 Relationship between Objectives and Methodology

Table 3.1 shows the relationship between the objectives and the methodology that was carried out to fulfill the aim of this project. To complete this study, the best methods were used for each of the objectives to meet the quality issue's requirement in the future.

Table 3.1: The relationship between objective and methodology

Objectives	Method Used
To compare the surface topography of the untreated and heat-treated Zirconia dental crowns	➢ Microstructure analysis using SEM Machine (Carl Zeiss Evo 50) ➢ Powder coat (SC 7620 Mini Sputter Coater).
To analyse the surface roughness of the untreated and heat-treated dental crown	➢ Profilometer (Mitutoyo Surftest SJ-410) ➢ Muffle furnace ➢ ARDENTA 3D dental CNC milling machine.

In order to achieve all of the objectives, there is a several software and machine that need to be used to accomplish the objectives. The instrument and the machine required for this study are provided by Faculty of Manufacturing Engineering, UTeM. Other than that, the assist from the supervisor and technician was also required to complete this study.

3.4 Dental Crown Specimen/Sample Preparation

During this study, the material used for the experiment was NexxZr T, which is dental Zirconia oxide (Y-TZP ZrO_2) from Sagemax Bioceramics, Inc. There were 12 samples of Zirconia crown restoration that were fabricated using Ardenta dental milling machine. Each sample was tested differently after the milling process to compare the surface topography between the heat-treated and untreated Zirconia crown. At the end of this study, the comparison was carried out. Figure 3.3 shows the example of Zirconia crown restoration and Figure 3.4 shows the NexxZr T Zirconia block.

Figure 3.3: Zirconia crown restorations.

Figure 3.4: NexxZr T, dental Zirconia oxide (Y-TZP ZrO2) from Sagemax Bioceramics, Inc.

The first three samples were the untreated crown. Two samples from the untreated crown were tested with a profilometer for these measurement. The other sample was employed for the surface topography analysis. For the heat-treated condition, there were 9 samples. During this study, three temperature setting were used which is 850°C, 950°C, and 1050°C. At each temperature setting, three replications of the sample were adopted. The procedures are still the same with the untreated crowns, two samples were used for the surface roughness measurement and one sample was further executed for the SEM analysis.

3.4.1 NexxZr-T Zirconia Block

NexxZr T is a dental Zirconia block used for dental crown restoration during this study, which is dental Zirconia oxide (Y-TZP ZrO2) from Sagemax Bioceramics, Inc. This Zirconia block has high translucent zirconium oxide for perfect aesthetics. It has multiple color selection, which is up to 20 colors. For monolithic restructuring, NexxZr T is the dental zirconium oxide (Y-TZP ZrO2) for single-tooth to multiple-unit bridge restoration. The perfect balance between translucentness and strength (1270 MPa) for the production of all-ceramic restorations of aesthetics is impressive. The indications for this Zirconia disc are for single-tooth restorations (anterior and posterior), 3-unit bridges (anterior and posterior), multi-unit bridges (anterior and posterior), implant-supported restorations (multi-unit screwed on titanium bases), and anatomically reduced crowns and bridges. Table 3.2 shows the composition and the properties of the disc obtained from the manufacturer's website.

Table 3.2: Composition of the Zirconia disc as claimed on the manufacturer's website

Raw Material	**NexxZr T**
Zirconium oxide ZrO_2	≥ 89%
Yttrium oxide + Y_2O_3	4-6%
Hafnium oxide HfO_2	≤ 5%
Aluminium oxide AI_2O_3	< 1%
Chemical solubility [μg/cm2]	< 100

3.5 3D Virtual Model in CAD

The software of the CAD/CAM dental laboratory was used to produce a 3D dental crown restoration digital model, and then the use of ARDENTA is required to produce the digital dental restoration model. CAD software for dental laboratories is designed to guide the technician through the design of a 3D digital crown, bridge, protection or another dental crown restoration model. CAM program for dental laboratories is the link between the device and a mill that manages how materials are used. Dental laboratory CAD/CAM software can be opened or closed with open applications that work only with selected scanners, mills or printers while working with various hardware and software systems. During this study, the CAD file for the crown was provided by Sagemax Bioceramics, Inc. After purchasing the Zirconia blocks from Sagemax Bioceramics, Inc, they give several files of crowns and bridges model in CAD file, so selected one of the model of the crown that was provided in order to manufacture the crowns restoration using the CNC dental milling machine. The CAD file of the crowns was used to generate the STL file before starting the milling operation.

3.6 CNC Dental Milling Machine

The use of the ARDENTA DT 100 CNC dental milling machine that is available at UTeM was required in order to manufacture the crown. Figure 3.5 shows the ARDENTA DT 100 that was used. This is one of the latest modern digital dentistry generations, which offers the best option for dentistry. The high-speed light precision mill combines tool is one of the best devices for optical laboratory. The automation by ARDENTA 3D Milling Machine can reduce the load in the dental laboratory.

ARDENTA has a horizontal spindle that is capable of providing good control of the chip and great precision. This machine has 0.001 mm location resolution and an accuracy of 0.01 mm positioning. ARDENTA has a minimum margin thickness of 0.15 mm while a minimum coping thickness is 0.3 mm and a cement gap of 0.02 mm. From 'ARDENTA.pdf' article (n.d.), ARDENTA is the laboratory-oriented CNC controller with its own features such as a simple one-touch graphical interface, which allows the operator to use without CNC experience or training, and process automation is complete due to the measuring system for tool lengths. ARDENTA also has 4 or 5-axis milling machines with high precision and speed.

Figure 3.5: ARDENTA DT 100 dental CNC milling machine.

3.6.1 Machining Procedure

During this study, the fabrication of the Zirconia dental crowns was done using the Ardenta Dental Milling Machine in the faculty. The Ardenta works with the supply of the air pressure from the compressor and also electricity current in order to move the tool and the orientation of the material platform to produce a dental crown.

Table 3.3 shows the step procedure in using the Ardenta DT 100. The appropriate procedure must be adapted in order to avoid human error and to get a good outcome from the milling process.

Table 3.3: Step by step procedure for preparing the crowns

No	Step
1	The Zirconia blocks were attached to the material platform and the ARDENTA CAM software was opened.
2	The STL file of the crowns and the type of restoration was selected. The selected restoration is anatomic crown.
3	The parameter setting, such as the diameter of stock, the thickness of stock, and the number of support, was adjusted before the machining. After that, all of the data from ARDENTA CAM Software was sent to the ARDENTA CNC milling machine.
4	The machining started after clicking the start button.
5	Wait for the machining process until the restoration was done. The other parameter setting of the machine, such as feed rate and spindle speed, will automatically appear on the screen during the machining. After the machining was done, the ARDENTA will automatically stop. After that, the crown samples are ready.

To manufacture the dental crown, there are 3 types of tool used which are ball the nose 1, the ball nose 2, and the ball nose 3 as shown in Figure 3.6. Ardenta is an automatic machine, which the user does not need to change the tools. The tools are automatically changing during the machining. Other than that, the feed rate and spindle speed are automatically generated once the machining started.

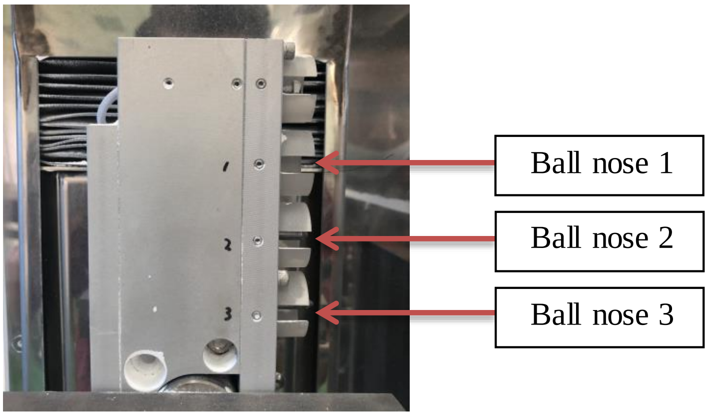


Figure 3.6: Tools used by Ardenta.

In this study, the selected design for the crown sample is shown in Figure 3.7. There are a lot of design of the crowns, but the selected design has a wide surface on each side of the crown which appropriates for the measurement of the roughness.

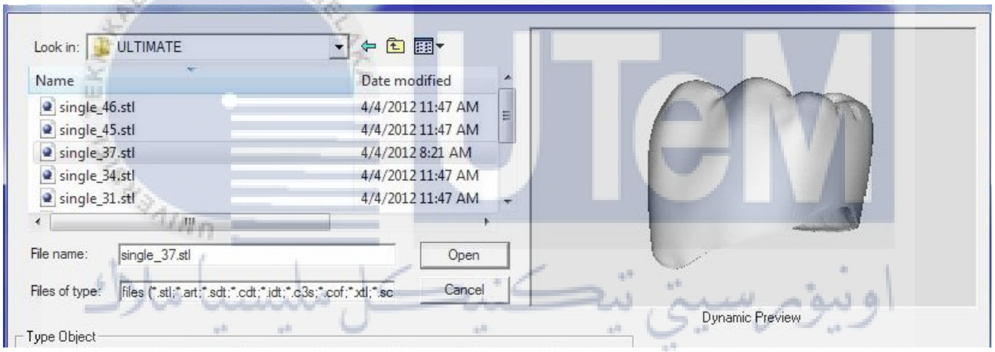


Figure 3.7: Crown designs available in the laboratory.

Before the machining, there are a few parameter settings that need to be adjusted, such as the thickness and diameter of the stock, the material of the stock, the type of crowns that need to be machined, and the number of support needed, as shown in Figure 3.8. To define the thickness and diameter of the stock, the work material is measured using a digital vernier caliper. If the measurement of the stock has some errors, the outcome of the milling varies from the actual sample size. Other than that, the number of support structures affects the surface of the crowns. After adjusting these parameters and setting, the manufacturing of the dental crowns started. For Zirconia material, the time needed to manufacture a dental crown is around 40-50 minutes, which is longer than other materials. This is due to the brittleness features of the Zirconia material, which easy to initiate a crack. The complex geometrical surfaces on the dental crowns also influence the time needed to manufacture the crowns.

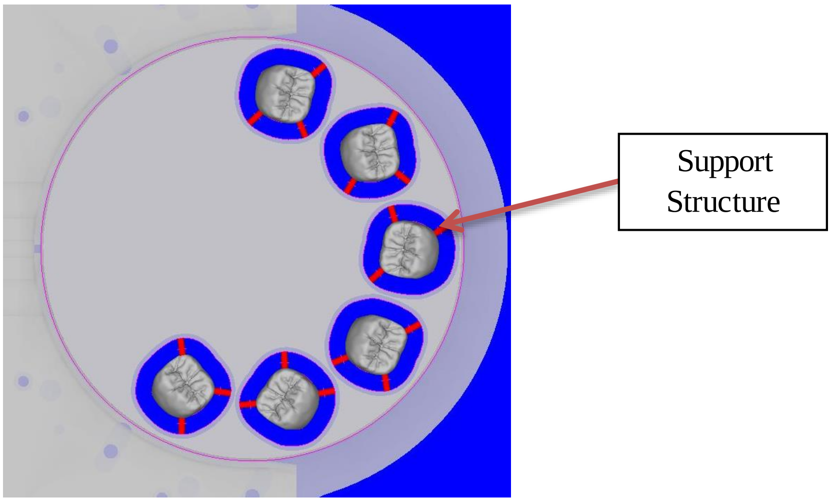


Figure 3.8: Support structure setting.

The Zirconia blocks must be attached and locked on the material platform, as shown in Figure 3.9. The material needs to be locked to ensure the workpiece is not moving during the machining. The machine needs to be monitored during machining is in progress to prevent the failure of the tool during the machining. After the machining is done, the machine stops automatically. After that, the user just needs to open the door of the machine and pick up the crowns and keep it under room temperature in order to avoid the environmental temperature affects the samples during the topography analysis. The crowns are wrapped with a fabric or tissue in order to avoid the cracks due to the brittleness of Zirconia. Figure 3.10 shows the Zirconia blocks after the crowns were manufactured.

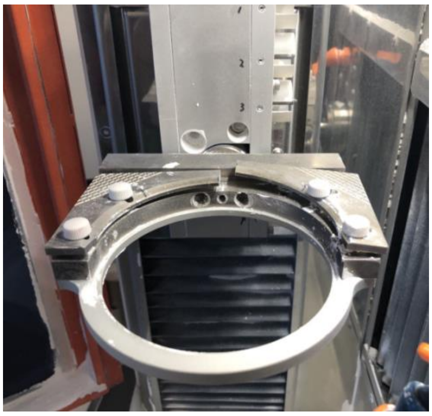


Figure 3.9: Material platform.

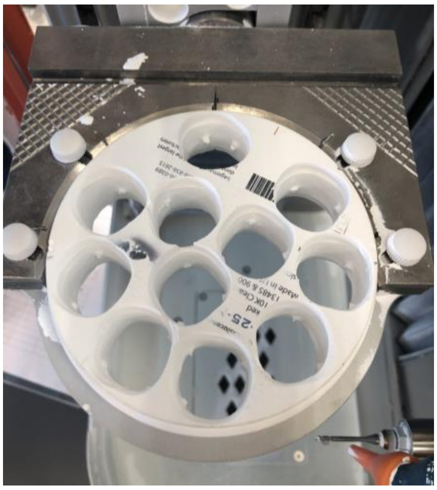


Figure 3.10: Zirconia block after machining.

3.7 Heat Treatment on Zirconia Dental Crown

After the crown specimens were milled from the ARDENTA dental CNC milling machine, the specimens need to be heat-treated. During this treatment, 9 samples are required. Based on Anna *et al.* (2013), the heat treatment test was conducted at the final temperature of 850°C with a heating rate of 50°C per minute. On the other hand, Camila et al. (2017) stated that the heat treatment test was executed with a final temperature of 1050°C and a heating rate of 65°C per minute. Based on the previous work, we decided to heat the specimens at three different temperatures, which is 850°C, 950°C, and 1050°C. Each of these temperatures was tested by three replications, as summarized in Figure 3.9. Three samples were heated at 850°C, three samples at 950°C, and three samples at 1050°C. Three replications used at each of the temperatures setting were to avoid human error during the heat treatment. Figure 3.11 illustrates the replications of the sample at each condition.

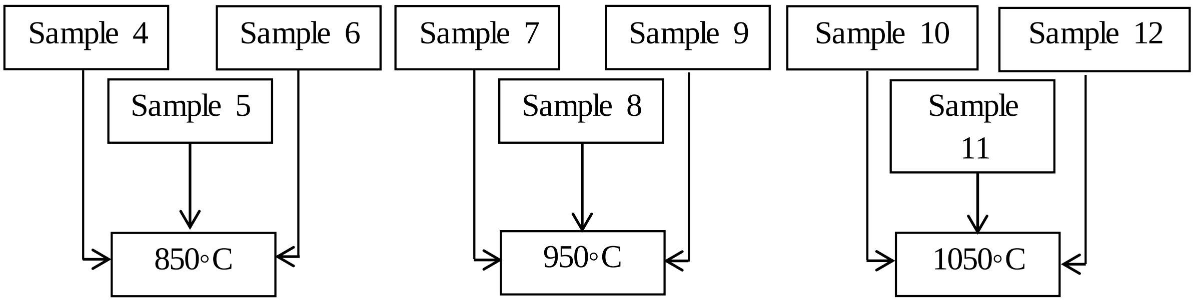


Figure 3.11: The replications of the samples at each temperature.

To accomplish this study, the use of a furnace, which is muffle furnaces with bricks insulation that available at UTeM was required during heat treatment, as shown in Figure 3.12. Heating elements on tubes that radiate freely in the furnace chamber provide for these furnaces with very short heating times. For this furnace, the robust lightweight refractory brick insulation can achieve a maximum operating temperature of 3000°C. This furnace has multilayer isolation in the furnace chamber with robust lightweight refractory bricks. It also has two shell housings for lower external temperatures and stability.

Figure 3.12: Muffle furnaces with bricks insulation.

The first replication of the samples was put into the furnace, followed by the other two replications. The heat treatment was conducted with the parameter setting as shown in Table 3.4. After the heat treatment was done, the samples was kept in an airtight container. Two samples from each condition were conducted for the surface roughness test and 1 sample from each condition was conducted for the SEM analysis.

Table 3.4: Parameter setting for heat treatment

Machine Parameter	First Replication	Second Replication	Third Replication
Waiting Time	1 minute	1 minute	1 minute
T (heating rate)	50°C/minute	50°C/minute	50°C/minute
T (final temperature)	850°C	950°C	1050°C
Holding Time (min)	15 minute	15 minute	15 minute

3.8 Surface Roughness Testing on Zirconia Dental Crown

Mitutoyo Surftest SJ-410, which is a profilometer, was used to masure the surface, as shown in Figure 3.13. The setup that is crucial in this analysis is the travel length that set up, which 0.08 mm x 3 due to the surface of the dental crown is small and limited. The experiment was repeated several times because of the limitation of the machine that only can yield the measurement on the flat surface. During this study, there are four conditions of the crowns which are untreated, 850°C heat treated, 950°C heat-treated, and 1050°C heat treated. The measurement was carried out using two samples from each condition of the crowns.

Figure 3.13: Mitutoyo Surftest SJ-410 surface roughness tester at Metrology Laboratory.

This instrument was calibrated first before the measurement is taken. The measurement method (calibration test) is performed on a reference workpiece (accuracy roughness specimen) and is adjusted to the difference between the measured value and the value reference (accuracy ruggedness sample). The measurement process is based on the calculation of the surface roughness. The measurement flow for this surface roughness test is shown in Figure 3.14.

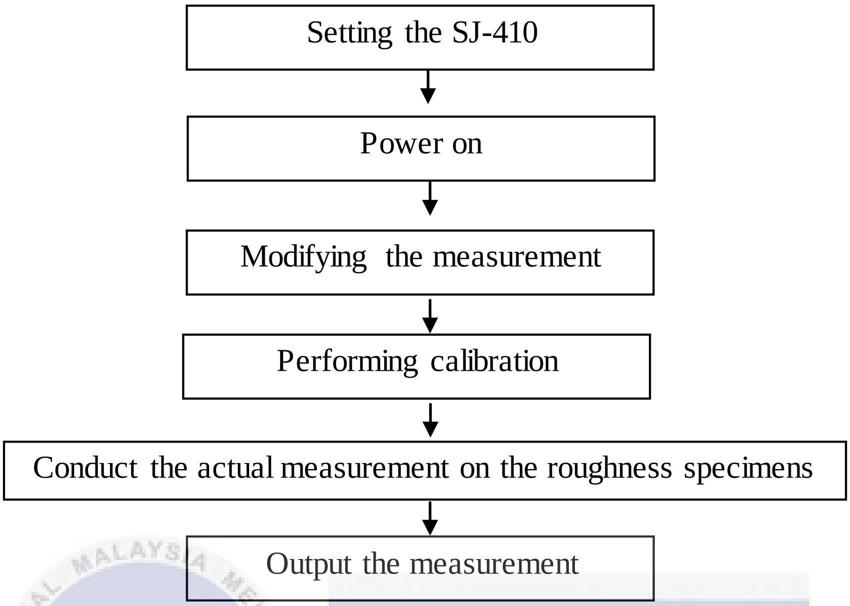


Figure 3.14: The measurement flow for surface roughness test.

Based on the flowchart, the first step is to set up the Mitutoyo Surftest SJ-410 surface roughness tester according to the feature of the crown to be measured. Next is power on the tester. Pick the AC adapter or battery built-in as the source of power. The third step is to change the measuring conditions for information on possible changes to measurement conditions. The fourth stage, which is the critical process, is performing the calibration. Calibration is a way to change the detector gain so that accurate measurements can be made by the SJ-410. The measurement of a supplied accuracy specimen can easily accomplish this. After that, the measurement is performed. In other words, the roughness at the selected point was measured, which is at each side of the crown for all of the selected specimens and the result was display.

The surface roughness measurement area is shown in Figure 3.15. The four areas that have been selected are because they have wide and flat surfaces which is easy to be measured. The top area of the crowns is not measured due to the complex geometrical and occlusal surfaces and also because of the limited machine capability that can only take the measurement on the flat surface. Five measurements are taken at each selected point to reduce the measurement errors and to get the average, Ra value. Ra is the arithmetic average of the absolute values of the height deviations which is from the mean line. In other words, the Ra value is the average of a set of individual measurements of a surface valley and peak. To make sure the Ra measurement is done on the same side for all of the samples, the selected points are initially marked to avoid the measurement error. The results of the measurement can be stored, written, extracted as SPC data and transmitted to a personal computer. The selected samples, which are two samples from each condition, were employed for this test.

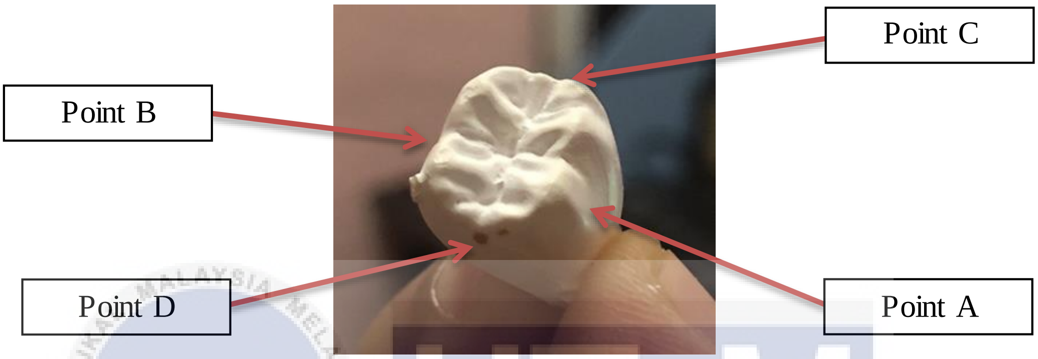


Figure 3.15: Measurement area on each side of the crowns.

3.8.1 Calibration Process

As mentioned above, the critical process which is performing the calibration, the appropriate procedure must be adapted. The calibration method involves a reference workpiece (specimen of accuracy roughness) and adjusting the difference (adjustment of gain), if the measured value is located between the measured and the reference (specimens of accuracy). The calibration measurement on the SJ-410 can easily adjust this difference. Calibration should be carried out regularly depending on the application of the SJ-410. Furthermore, calibration will be essential if the instrument is first used or if the detector has been installed/replaced. Correct measurements cannot be achieved without properly calibrating the instrument. The calibration process is described in the Table 3.5.

Table 3.5: Calibration procedure

Step	Description
1	In the measuring, mode click on the [CAL / STD / RANGE] key. The current calibration value is displayed in the calibration mode. The calibration value can be adjusted in this state. 
2	If the values displayed vary from the one shown on the accuracy set, change the value of the calibration. Proceed to the next step if the calibration value requires no change. 
3	Confirm the displayed value, then press the [n/ENT] key. The value entered is defined. 
4	The value of the calibration is now set. Press the [START/STOP] key. During the calibration measurement (the detector crosses), calibration measurement with the specimen of accurate ruggedness is carried out and "——" is shown. 
5	The measured value is displayed when the calibration measurement is complete. Then Press the [n/ENT] key. The calibration factor is updated and the entire calibration process is completed. 

SJ-410 was set on a workpiece to start the measurement, press the [START / STOP] button. The measurement results are shown on the LCD for confirmation after the measurement has been completed. In order to be accurate in calculating surface roughness, a solid foundation that is insulated from all sources of vibration must be carried out as well as possible. The results may be unreliable when the measurement is carried out subject to significant vibration. Figure 3.16 shows the example of a measurement that will be provided by Mituyoto Surftest SJ-410. The standard deviation for the result of each surface roughness measurement for each sample is calculated using Equation 3.1.

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

Where x is measurement value, Equation 3.1

$\bar{x}$ is measurement mean,

n is number of samples

Figure 3.16: Example of measurement on Mitutoyo Surftest SJ-410.

3.9 Powder Coat/Sputtering Process

SEM machines can scan various types of samples, such as semiconductors, metals and alloys, polymers, ceramics and biological specimens. Some samples can be harder to picture and must be sputter-coated to provide an image of high quality. Sputtering consists of the layer of a thin sample (about 10 nm), such as chrome, platinum, gold, or silver, of conductive metals. In order to improve the SEM images, non-conductive materials also require sputter coating. Non-conducting materials may serve as an electron trap, meaning that electrons accumulate on the surface by means of a mechanism called charging. Gold has a small grain size that facilitates high-resolution picking and high conductivity, which is the conventional favorite for sputter coating. Other than that, chromium, tungsten or iridum have extremely fine grains and are very popular options for ultra-high-resolution imaging.

During this study, all of the selected samples, which are four samples, initially need to be coated in order to improve the image. The aim of this process is to avoid electrostatic discharge of the non-metallic samples during the SEM analysis. All of the samples will be coated with gold (Au) using the SC 7620 Mini Sputter Coater provided by FKP as shown in Figure 3.17. The powder coat is required to create a conductive layer of metal on the sample, inhibit charging, minimize thermal damage, and improve the secondary electron signal required for topographic analysis in the SEM.

Figure 3.17: SC 7620 Mini Sputter Coater at the laboratory.

The coating process consumes 3 minutes to make sure the sample is fully coated. After the coating process was done, the sample can't be touched and need to keep in an airtight container to avoid contamination. Figure 3.18 shows the samples after the coating process.

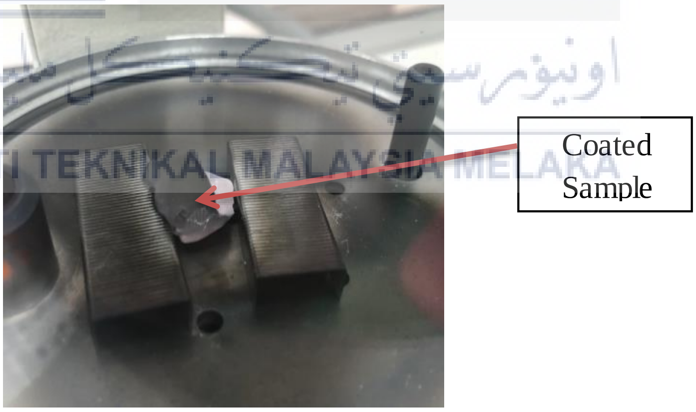


Figure 3.18: Crown sample after the coating process.

3.10 A Study on the Surface Topography of the Untreated and Heat-Treated Dental Crowns

SEM machine scans a focused beam of the electron over a surface for the production of an image. The beam electrons communicate with the sample and produce different signals, which can be used to get information on surface topography and composition.

Carl Zeiss Evo 50 was used to examine the surface structure of the milled dental restoration, as shown in Figure 3.19. The objective of this study is to analyze the surface topography of the untreated and heat-treated dental crown restoration. All of the samples, which are four samples of heat-treated and untreated crown, were analyzed.

Figure 3.19: Carl Zeiss Evo 50 SEM Machine at FTKMP laboratory.

3.10.1 SEM Procedure

SEM is a very delicate device and should be used very carefully. Following the proper procedure can avoid damage to the microscope. The procedures below describe how to get a basic picture.

a) A prepared sample was obtained. Use a sample that has been prepared. During this study, the total sample is 4. Each sample needs to be examined.

b) To open the sample door, bring the SEM to air pressure. Understand that the sample chamber is always vacuumed and the SEM is always power on. In other words, there are no gas molecules in the chamber, so that electrons have a clear road towards the sample. Hold on the VENT button and when the flash button is flash, a click sound can be heard. The VENT button will continue to flash until the temperature in the chamber raised to atmospheric pressure. The chamber can be opened at atmospheric pressure once the VENT button is solid orange.

c) The gloves must be put on the hand. The SEM chamber must stay very clean, and this is extremely important. Slide-out the door slowly with the handles on either side of the door. After the door has opened, take the sample holder carefully and slide it to the right. It comes out of its grooves and the sample can be loaded. Two set screws hold the sample on each of the four corners of the sample holder. Slightly loosen these screws. When the screws are tightened, the sample can be placed into the holder. If the sample is in place, go ahead and drag the sample holder on the sample stage back to its grooves. Squeeze the door back into the room by pressing it.

d) The EVAC button was pressed while the door shut is hold, until it starts to blink to make sure the chamber back under vacuum. The click sound will exposed and then the sounds of the pump turbines dragging up will be heard after a minute or so. These pumps remove all gas particles from the chamber and bring about 10 ^ -6 torr internal pressure. This means that the chamber is vacuumed while the EVAC button is glowing firmly green. Wait a further 5 minutes now before the next step continues.

e) The electron beam was turned on after the five minutes have ended. At the same time, the images were taken. Go to the SEM computer and activate the microscope software. On the top right of the screen, the green button says OFF that indicates the electron beam is off. To turn on the beam, click and it will ON. Next, press the button ACB (automatic contrast and brightness) and see the outcome image—zoom in to the desired magnification with the magnification knob on the control panel. Use the focus knob when zoomed in to focus the image.

f) The height of the sample is set. The sample is now concentrated, but it may not look as good as expected. The working distance specified as "WD x mm" is shown underneath the image of the sample on the computer screen. The numbers are usually about 10-20. Notice and take note of that number. The use of a working distance of 10 mm for decent pictures is required. The beam is therefore focused 10 mm away from the lens. Click on "WD x mm" to set the working distance and will be able to change any x number to 10. Slider will appear. Click 10 and go ahead. Use the Z-button to change the sample height. It could be a tricky part.

Whatever the original working distance, remove 10 from it and how much the Z-knob needs to move. Watch the screen when pressing the Z button, as it begins to concentrate. The image is ready once it's focused.

g) Zoom the desired magnification back and use the X and Y knobs if want to move to a different area of the sample. Contrast and brightness can also be adjusted to our preferences. To find the desired area, click the camera icon which says STORE and then take a picture. This opens a dialog box to save the image file. The name and the file can be saved in a directory folder or on a flash drive.

h) Turn off the electron beam by clicking the green button that says "ON" on the top right corner. Before continuing, make sure that it says "OFF." Turn the knobs X, Y and Z to a value of 25 mm each. Now press and hold the VENT button until the sample chamber blinks and returns to atmospheric pressure.

i) When the VENT button is solid orange, open the sample chamber. Put the blank sample back in the holder and slip it back into the sample stage once the sample is removed from the holder. Run back to the closed position by sliding the door. Then press the EVAC button and hold it until it blinks. NEVER leave the SEM chamber vented. This can cause damage to the parts inside. Once the EVAC button glows solid green, it is saved to leave the SEM.

The parameter settings for the SEM machine are shown in Table 3.6. After conducting the SEM test for all of the samples, the objective was achieved, which is to compare the surface topography of the untreated and heat-treated Zirconia dental crowns. A comparison between these two conditions was carried out. The image of the untreated and heat-treated crown samples was captured.

Table 3.6: Parameter setting for SEM machine

Carl Zeiss Evo 50 Scanning Electron Microscopic Machine (SEM)				
	Accelerating Voltage (kV)	**Work Distance (WD)**	**1^{st} Magnification**	**2^{nd} Magnification**
Untreated Crown	15.00	7 mm	50×	200×
Heat-treated Crown	15.00	7 mm	50×	200×

3.11 Summary of Chapter 3

The heat treatment experimentation is expected to reduce the scallop height effect on the crown's surface. The comparison between the heat-treated and untreated dental crown restoration was successfully carried out. In conclusion, the tool path and the cutter's size during dental milled restoration produced the scallop height effect and scratch grooves on the surface due to the complex geometrical feature on the crown surface.. This study has shown the surface roughness tester plays a significant role in the surface roughness measurement. At the end of this study, the SEM images are expected to support the hypothesis stated in the problem statements section.

CHAPTER 4

RESULT AND DISCUSSION

This chapter provides the results of the experiments that were conducted in PSM 2. The fabrication of the Zirconia crown is done using Computer Numerical Controlled (CNC) dental milling machine provided by Faculty. Then, the fabricated crowns were heat treated. After that, all of the heat-treated and untreated crowns were analyzed using a surface roughness tester and SEM in order to observe and analyze the surface roughness and topography of the crowns. All the results are presented in this chapter to discuss the effect of the heat treatment on the dental crown's surface. At the end of this chapter, the comparison of the surface roughness from the untreated and heat-treated dental crown restoration was discussed.

4.1 Heat Treatment of the Zirconia Dental Crowns

After the machining, the samples are divided into four batches, which contain three samples per batch. The first batch is the untreated sample, the second batch is heat-treated samples with 850°C, the third batch and the fourth batch is the heat-treated samples with 950°C and 1050°C, respectively. The sample setup is shown in Figure 4.1.

In the heat treatment, the use of a muffle furnace provided by Faculty is required. The furnace can produce heat more than 3000°C, so precautions must be considered in order to keep the safety. Before start-up the treatment, firstly, the furnace needs to be set into the desired parameter, such as waiting time, heating rate, final temperature, and holding time.

Figure 4.1: Sample set up for a batch of heat-treated samples.

The parameter setting for each batch of the sample is shown in Table 4.1. For this furnace, there is not too much parameter setting that needed to be set. Waiting time is the initial time needed before the temperature rise. Basically, the waiting time used is 1 minute. It is to make sure the sample is in good condition before the heating process started. The heating rate that is used in this heat treatment is 50°C per minute, so the time needed to reach the desired temperature is around 17 to 21 minutes for all of the samples. This setting is taken from the other study by Anna *et al.* (2013). When the temperature reached at the desired temperature, the furnace stops raising the temperature and then hold the temperature for 15 minutes. The holding time is adopted from Camila *et al.* (2017). Basically, the holding time depends on the material used and the size of the samples since the Zirconia crown has tiny size, the holding time that is normally used is 15 minutes. The bigger the size of the part, the more time needed to hold the temperature. After all the samples are heated, the surface roughness test is conducted subsequently. The strategy of experimentation for the surface roughness and The SEM analysis are shown in Table 4.2.

Table 4.1: Parameter setting for heat treatment

	Heat treated	**Heat treated**	**Heat treated**
Waiting Time (min)	1	1	1
Heating Rate (°C /min)	50	50	50
Final Temperature (°C)	850	950	1050
Holding Time (min)	15	15	15

Table 4.2: Strategy of the experimentation for all samples

Sample	Condition/ Heat Applied	Surface Roughness Test	Scanning Electron Microscope
1	Untreated	/	Not Required
2	Untreated	/	Not Required
3	Untreated	Not Required	/
4	Heat-treated (850°C)	/	Not Required
5	Heat-treated (850°C)	/	Not Required
6	Heat-treated (850°C)	Not Required	/
7	Heat-treated (950°C)	/	Not Required
8	Heat-treated (950°C)	/	Not Required
9	Heat-treated (950°C)	Not Required	/
10	Heat-treated (1050°C)	/	Not Required
11	Heat-treated (1050°C)	/	Not Required
12	Heat-treated (1050°C)	Not Required	/

4.2 Surface Roughness Analysis

The analysis of the surface roughness was performed using Mituyoto Surftest Roughness Tester. Two samples from untreated crowns and two samples from each condition of the heat-treated crown required for the surface roughness test. The four areas that have been selected are discussed in the methodology section. The top area of the crowns is not to be measure due to the complex geometrical and occlusal surfaces and also because of the limited machine capability.

During the apparatus setup, there was a problem occur which is the size and the weight of the samples were too tiny and lightweight, which can affect the result during the measurement. When the stylus starts to take the measurement, the sample moves from the current position because the sample is not in a stable condition. In order to overcome the problem, the use of plasticine is employed to maintain the position of the samples during the measuring process, as shown in Figure 4.2.

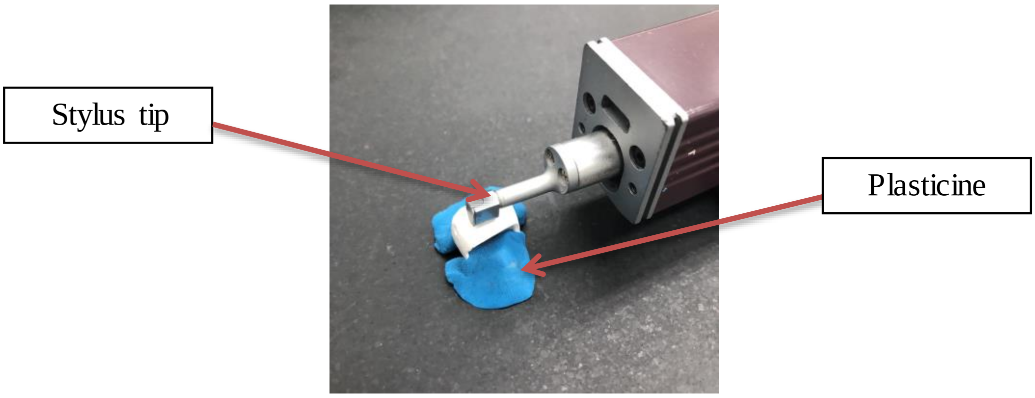


Figure 4.2: Sample setup with plasticine support.

After the calibration is done, the instrument is ready to be used. During the measurement, the position of the stylus tip is very important in order to have a correct measurement. In a standard stylus tool, a conispherical diamond tip is projected over the measured surface, and the vertical motion of the tip is determined in response to the surface. In determining the profile (line) of the surface, the vertical movement of the stylus tip and the displacement of the tip perpendicular to the plane are used (Leach *et al.*, 2015). The position of the stylus also can affect the result during the measurement. The arithmetic average height, Ra parameter is used while taking the measurement. This parameter is easy to measure, easy to define, and provides an excellent general description of height variations (Gadelmawla *et al.*, 2002). For this study, 5 readings were taken at each of the measurement points. The result is shown in Table 4.3, Table 4.4, Table 4.5, and Table 4.6.

Table 4.3: Result of surface roughness test for the untreated crown crowns

Reading / Point	1(µm)	2 (µm)	3 (µm)	4 (µm)	5 (µm)	Average,Ra (µm)	Standard Deviation (µm)
	Sample 1						
A	0.94	0.91	0.72	0.76	0.61	0.8	0.137
B	0.59	0.65	0.89	0.58	0.72	0.7	0.127
C	0.43	0.83	0.85	0.78	0.74	0.7	0.171
D	0.62	0.53	0.56	0.54	0.65	0.6	0.052
	Sample 2						
A	0.66	0.59	0.70	0.67	0.61	0.7	0.045
B	0.71	0.78	0.71	0.82	0.69	0.7	0.055
C	0.74	0.56	0.54	0.61	0.69	0.6	0.055
D	0.65	0.68	0.72	0.72	0.88	0.7	0.055

Table 4.4: Table: Result of surface roughness test for 850 °C heat-treated crowns

Point \ Reading	1 (µm)	2 (µm)	3 (µm)	4(µm)	5 (µm)	Average, Ra(µm)	Standard Deviation (µm)
	Sample 4						
A	0.69	0.81	0.91	0.87	0.77	0.8	0.09
B	0.70	0.68	0.59	0.67	0.72	0.7	0.05
C	0.66	0.67	0.59	0.64	0.55	0.6	0.05
D	0.62	0.69	0.78	0.71	0.68	0.7	0.06
	Sample 5						
A	0.58	0.53	0.58	0.49	0.55	0.6	0.04
B	0.45	0.47	0.49	0.59	0.45	0.5	0.06
C	0.76	0.93	0.98	1.15	1.11	0.9	0.16
D	0.96	0.74	0.67	0.73	0.97	0.8	0.13

Table 4.5: Result of surface roughness test for 950°C heat-treated crowns

Point \ Reading	1 (µm)	2 (µm)	3 (µm)	4 (µm)	5 (µm)	Average, Ra(µm)	Standard Deviation (µm)
	Sample 7						
A	0.63	0.77	0.48	0.65	0.63	0.6	0.10
B	0.45	0.55	0.47	0.68	0.57	0.5	0.09
C	0.87	0.93	0.87	0.80	0.81	0.8	0.05
D	0.34	0.59	0.65	0.43	0.61	0.5	0.13
	Sample 8						
A	0.94	0.91	0.96	1.11	0.90	0.9	0.09
B	0.42	0.58	0.42	0.52	0.43	0.4	0.07
C	0.93	1.07	1.09	0.87	0.95	0.9	0.09
D	0.83	0.90	0.83	0.94	0.83	0.8	0.05

Table 4.6: Result of surface roughness test for 1050°C heat-treated crowns

Point \ Reading	1 (µm)	2 (µm)	3 (µm)	4 (µm)	5 (µm)	Average, Ra(µm)	Standard Deviation (µm)
	Sample 10						
A	0.60	0.63	0.55	0.62	0.45	0.5	0.13
B	0.66	0.64	0.69	0.45	0.60	0.6	0.09
C	0.65	0.46	0.64	0.48	0.66	0.6	0.10
D	0.63	0.81	0.62	0.72	0.65	0.6	0.08
	Sample 11						
A	0.79	0.70	0.78	0.67	0.76	0.7	0.05
B	0.51	0.46	0.54	0.44	0.63	0.5	0.08
C	0.58	0.65	0.57	0.67	0.55	0.6	0.05
D	0.64	0.68	0.66	0.66	0.56	0.6	0.05

Based on Table 4.3, point A, B, C, and D from sample 1 and sample 2 are very close, which the difference is between 0.1 µm and below 0.1 µm, respectively. Based on the result, both the untreated samples have a similar surface outcome from the milling process.

Table 4.4 shows the heat-treated samples with 850°C heat applied. Point A, B, C, and D from sample 4 have an average reading of 0.8 µm, 0.7 µm, 0.6 µm, and 0.7 µm and for sample 5 are 0.6 µm, 0.5 µm, 0.9 µm, and 0.8 µm. This result shows that most of the point from both samples has differences above 2.0 µm. The result is supposed to be under 1.0µm difference. As an example, the measurement at point C for sample 4 and sample 5 has 33.33% differences between them.

Based on Table 4.5, which is the heat-treated samples with 950°C, the result shows that the point A, B, C, and D from sample 7 are 0.6µm, 0.5µm, 0.8µm, and 0.5µm. At the other hand, the result from sample 8 give the reading of 0.9µm, 0.4µm, 0.9µm, and 0.8 for point A, B, C, and D. From sample 7 and sample 8, both of them also have two points that have differences above 0.2 µm. The measurement at point A for both samples also has 33.33% differences between them.

For the last heat-treated samples of 1050°C, Table 4.6 shows the result for sample 10 and sample 11. The average reading for point A, B, C, and D from samples 10 are 0.5µm, 0.6µm, 0.6µm, and 0.6µm while the average reading for sample 11 is 0.7µm, 0.5µm, 0.6µm, and 0.6µm. They have a similar average reading at point C and point D, which is 0.6µm. The measurement at point B for sample 10 and sample 11 have 16.67% differences, which is less than the other condition.

4.2.1 The Comparison of the Ra Average Reading

Based on the surface roughness measurement, it can be concluded that the difference between the untreated and heat-treated crown samples is little. The comparison of the average reading at points A, B, C and D are shown in Table 4.7.

At point A, the comparison between sample 2 and sample 10 is discussed. Sample 10 shows the lowest average reading, which is 0.5 µm and sample 2 has an average of 0.7 µm during the measurement. We can conclude there is some surface improvement for sample 10. At point B, sample 8 has the lowest average reading, which is 0.4 µm. Based on sample 1 and sample 2, there is a decrease from 0.7µm to 0.4µm, which also proved that there is some effect from the heat treatment.

At point C, sample 8 shows the average reading is high, which is 0.9µm compared to sample 2, 0.6µm (untreated). At point D, sample 8 also shows the reading is high (0.8µm) compared to the untreated samples (sample 2, 0.7µm). From overall observations, during the measurement of sample 8, some errors occur due to machine capability limitation. The average measurement at all points of sample 8 is high compared to the other samples.

On the other hand, the comparison between sample 1 (untreated) and sample 10 (1050°C heat-treated) was discussed. Based on Table 4.7, the surface roughness was decreased after the heat treatment. At point A, the surface roughness decreased 37.5%. At point B and C, there was a decrease of 14.28% which is lower than point A. At point D, there was no changes in the measurement of the roughness.

Table 4.7: Comparison of the average (Ra) reading at point A, B, C, and D

	Untreated		**850°C**		**950°C**		**1050°C**	
Sample / Point	1 (µm)	2 (µm)	4 (µm)	5 (µm)	7 (µm)	8 (µm)	10 (µm)	11 (µm)
A	0.8	0.7	0.8	0.6	0.6	0.9	0.5	0.7
B	0.7	0.7	0.7	0.5	0.5	0.4	0.6	0.5
C	0.7	0.6	0.6	0.9	0.8	0.9	0.6	0.6
D	0.6	0.7	0.7	0.8	0.5	0.8	0.6	0.6

Based on the overall result, we can conclude the temperature used, which are 850°C, 950°C, and 1050°C are not sufficient to reduce the surface roughness of the Zirconia dental crown. All of the samples that are heat-treated are not resulting in a better surface finish. The temperature used needs to be higher than 1200°C. In addition, the surface roughness tester could also influence the result while measuring the surface of the crowns. The Mituyoto surface roughness tester that available at the Faculty is not suitable for the uneven surface. The instrument also does not provide the actual measurement of the surface roughness due to the limitation of the machine capability. The result from the surface roughness test is supported by the SEM images.

4.3 Scanning Electron Microscope Analysis

Scanning electron microscope analysis is conducted to capture the image of the crown's surface in order to make the comparison between the untreated and heat-treated crowns. The selected sample which is sample 3 (untreated), sample 6 (850°C), sample 9 (950°C), and sample 12 (1050°C), are used for this analysis. Before the SEM analysis started, all of the samples need to be sputter-coated. The samples are coated using SC 7620 Mini Sputter Coat that is available at the Faculty. The material used for the sputtering process is gold coated, which contains 80% gold and 20% palladium to provide a better image during the SEM analysis. The images captured are shown in Figure 4.3.

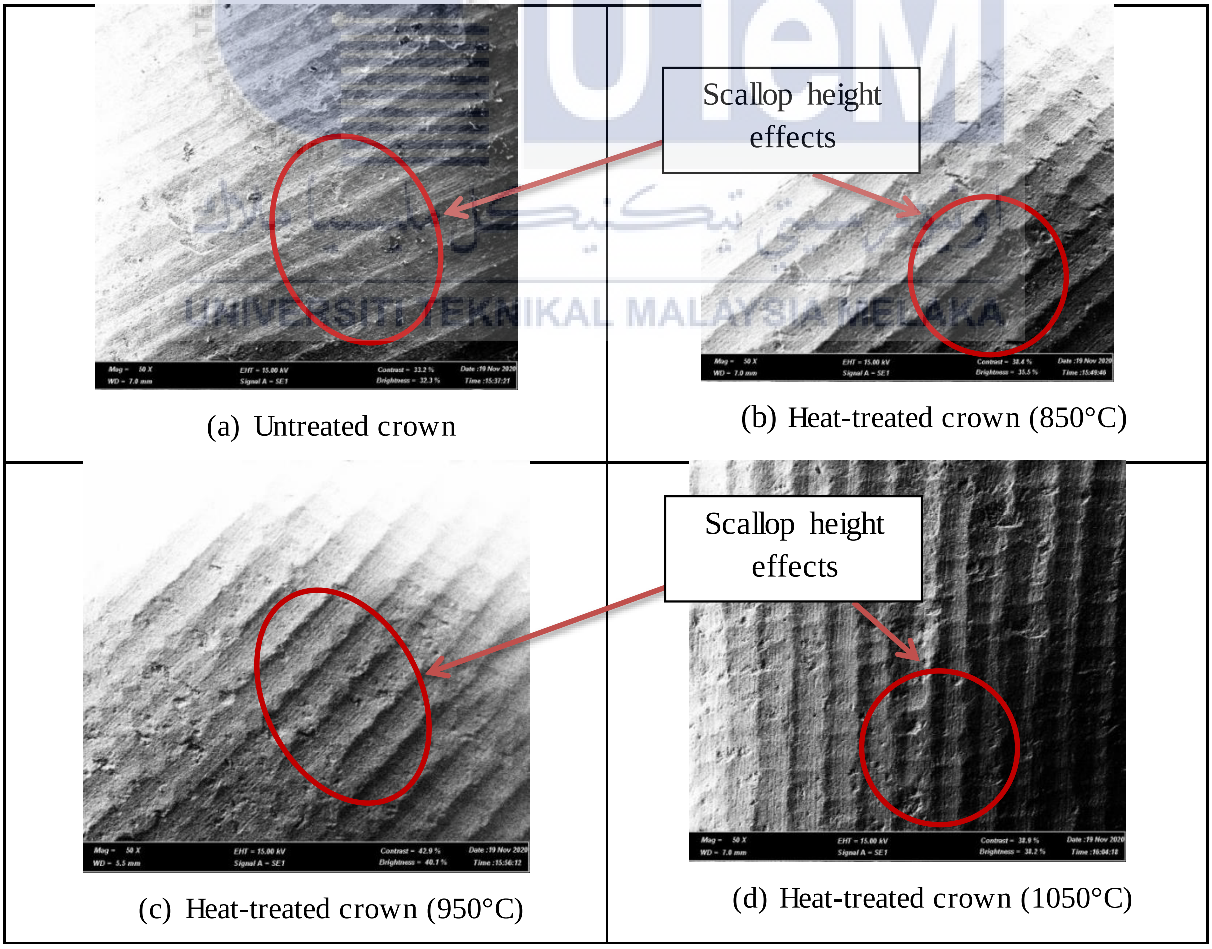


Figure 4.3: SEM image at 50x magnification power for (a) Untreated crown, (b) Heat-treated crown (850°C), (c) Heat-treated (950°C), and (d) Heat-treated (1050°C).

As the outcome of the SEM, Figure 4.3 (a) shows the image of the surface from the untreated crown sample with 50x secondary electron magnification power. The surface topography of the untreated crowns shows the scallop height effect caused by the tools during the machining process. At 50x power magnification, the scallop height effects are clearly seen through the secondary electron. Secondary electron basically used for topography contrast in the scanning electron microscope, such as for the visualization of roughness and surface texture (Zhou *et al.*, 2015). Other than that, a secondary electron can resolve the surface topography or surface structure at 10nm or better. Before capturing the image, the accelerating voltage used for SEM is 15kV. According to Zhou *et al.* (2015), the resolution of the surface can be lost when the accelerating voltage is too high. On the other hand, the work distance (WD) used is 7mm. Zhou *et al.* (2015) has suggested that in order to analyze a sample or a specimen with a large variety of topographical, a long WD is preferred to bring as much of the image into focus as possible.

Figure 4.3 (b) shows the SEM image of the 850°C heat-treated crowns. As we can see, it is similar to the untreated crown where the scallop height effect can be seen. From this observation, the temperature used is too low which is unable to eliminate or reduce the scallop height effect. Figure 4.3 (c) and Figure 4.3 (d) show the 950°C and 1050°C temperature are not capable to improve the surface roughness. The gap of all temperature used is too close where we can't see the effect of the heat treatment. Obviously, the temperature of 850°C, 950°C, and 1050°C is not relevant in order to improve the scallop height effect from the Zirconia dental crown surface. In order to get the suitable temperature for the heat treatment of the Zirconia dental crown, a phase diagram is required. After that, the prediction of the suitable final temperature and holding time for the furnace during the heat treatment can be carried out.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

This chapter summarizes the overall findings of this study. Also, the recommendation for the future study of the Zirconia dental crown surface treatment is discussed in order to improve the method used.

5.1 Summary of the Study

The initial expected result from this study is the heat treatment with specific temperature can reduce the scallop height effect and scratch grooves as the outcome of the milling operation. The first objective, which is to compare the surface topography of the untreated and heat-treated crowns using the SEM machine, was achieved. The image on both untreated and heat-treated crowns was analyzed. The second objective was also achieved. The data from the surface roughness test was successfully analyzed in a table.

At the beginning of this study, the selection of the design for the crown restoration plays a significant role. This selection was made due to the limitation of the available surface roughness tester at the laboratory., Since the traveling length of the tester was set to 0.08 mm, the surface roughness tester was capable to measure the selected surface of the crowns. Other than that, determining the number of support for the crowns before the machining is also significant. This is because when the support is taken out, it causes a poor surface on the crown. It is also to ensure a controllable machining environment.

In this study, the effect of the heat treatment with the selected temperature which is 850°C, 950°C, and 1050°C are not affecting the surface roughness. The selected temperatures are not high enough to close the pore and reduce the surface roughness of the Zirconia dental crowns. The following conclusions were drawn from this study:

a) The temperature of 1050°C does not improve the surface roughness of the Zirconia dental crowns. A higher temperature within 1200°C-1500°C is recommended.

b) The surface roughness tester plays a significant role in measuring the crown's surface.

c) SEM analysis of the crown samples proves that heat treatment of 1050°C does not affect the surface quality of the crowns. The image of 50x magnification shows the scallop height effects still exist after the heat treatment.

5.2 Recommendations

A possible future work that is recommended is for fabricating the dental crown. It is more convenient to be able to scan the dental crown to complete the dental digital data creation process flow, which includes 3D scanning, 3D virtual model in CAD, point cloud data editing and STL file generation. Other than that, the sample for each condition must have 4 samples, which are three samples for the surface roughness and one sample for SEM. The three samples for the surface roughness are to ensure the measurement taken is averaged and to reduce the measurement error. The samples that have gone through the surface roughness test are not able to go through the scanning electron microscope analysis because of the scratch effect from the stylus during the surface roughness test. It will ruin the image during the scanning electron microscope.

The furnace used should be more advance in parameter setting. The current furnace used does not have the cooling rate parameter. The cooling rate also plays a significant role towards the surface roughness of the Zirconia dental crown. In the future study, the temperature used must be higher than 1200C. During this study, it is proven that the temperature of 1050C and below does not give effect to the surface of the crowns. To find out the heat treatment test with multiple temperatures, there must be a long gap between the selected temperatures to obtain a reasonable result during the surface roughness and SEM analysis. In order to achieve the current objective, a phase diagram using an appropriate method is also required to obtain the suitable temperature for the heat treatment. For the surface roughness measurement in the future study, the profilometer used must be suitable in taking the measurement of the complex geometrical surfaces. The selection of the profilometer must be considered for this study. In this study, due to the limitation of the surface roughness tester at the laboratory and the incapability to use the appropriate tester at the other university because of Covid-19, the obtained data is accepted. This is because the traveling length set in relation to the selected crown was made to compensate the lackness.

5.3 Life Long Learning Element

The lifelong learning element defines as the development of human potential through a continuously supportive process that stimulates and allows individuals to acquire all the knowledge, values, skills and understanding that will require throughout the lifetimes and able to apply the learning in all roles. The knowledge gained through this study is to develop an understanding of CNC milling technology in machining dental crown restoration.

5.4 Complexity Element

It can be defined as the complexity that presented when undergoing this study. For example, the initial planning of the methodology in achieving the objectives of this study is involving with 3D scanner for the fabrication of the crowns. Instead, due to the limitation of the machine, the process of scanning the pre-fabricated dental crown cannot be done. As the design of the dental crown has high complexity and it is not compatible to the function of 3D scanner that available here.

REFERENCES

Alao, A., Stoll, R., Song, X., Abbott, J. R., Zhang, Y., & Abduo, J. (2017). *Journal of the Mechanical Behavior of Biomedical Materials Fracture , roughness and phase transformation in CAD / CAM milling and subsequent surface treatments of lithium metasilicate / disilicate glass- ceramics*. *June*.

Alfawaz, Y. (2016). Zirconia crown as single unit tooth restoration: A literature review. *Journal of Contemporary Dental Practice*, *17*(5), 418–422.

Aspros, A. (2015). Inlays & Onlays Clinical Experiences and Literature Review. *Journal of Dental Health, Oral Disorders & Therapy*, *2*(1), 26–31.

Bammani, S. S., Birajdar, P. R., & Metan, S. S. (2012). Dental Crown Manufacturing using Stereolithography Method. *Proc. of Int. Conf. on Advances in Industrial and Production Engineering*, 7–10.

Buciumeanu, M., Queiroz, J. R. C., Martinelli, A. E., Silva, F. S., & Henriques, B. (2017). *Tribology International The effect of surface treatment on the friction and wear behavior of dental Y-TZP ceramic against human enamel*. *116*(June), 192–198.

Cheng, C., Yang, C., & Yan, M. (2018). *Bond strength of heat-pressed veneer ceramics to Zirconia with various blasting conditions.*

Cho, H. Y., Won, H. Y., Choe, H. C., & Son, M. K. (2011). *Fracture Characteristics of Dental Ceramic Crown according to Zirconia Coping Design*. *10*, 1561–1566.

Denkena, B., Breidenstein, B., Busemann, S., & Moritz, C. (2017). *ScienceDirect Impact of hard machining on Zirconia based ceramics for dental applications*. *65*, 248–252.

Dolidze, T., & Bitarova, I. (2016). Advantages and Dısadvantages of E-MAX and Zirconia Crowns. *European Scientific Journal*, *7881*(December), 1857–7881.

Faeizah, N., Muchtar, A., Zenn, H., & Sufiyan, M. (2020). *Machinability of a newly developed pre-sintered Zirconia block for dental crown applications*. *261*.

Guazzato, M., Albakry, M., Quach, L., & Swain, M. V. (2010). *Influence of surface and heat treatments on the flexural strength of a glass-infiltrated alumina / Zirconia-reinforced dental ceramic*. *2005*, 454–463.

Hian, L. (2017). Critical review Dental Materials/Dentistry Dental ceramics: a review of new materials and processing methods. *Braz. Oral Rest*, *31*, 133–146.

Hamid, Idayu, N., Ali, M. A., & Kasim, M. S. (2020). Intelligent Manufacturing and Mechatronics. *Symposium on Intelligent Manufacturing and Mechatronics*, *1*, 387–398.

Inokoshi, M., Vanmeensel, K., Zhang, F., Munck, J. De, Eliades, G., Minakuchi, S., Naert, I., Meerbeek, B. Van, & Vleugels, J. (2014). Aging resistance of surface-treated dental Zirconia. *Dental Materials*, 1–13.

Irvine, D., & Matthews, J. (2020). *ScienceDirect The The analysis analysis of of tool tool wear wear mechanisms mechanisms in in the the machining machining of of Zirconia dental dental crowns crowns pre-sintered Zirconia*. *00*(2019), 1–8.

Jang, G. W., Kim, H. S., Choe, H. C., & Son, M. K. (2011). Fracture strength and mechanism of dental ceramic crown with Zirconia thickness. *Procedia Engineering*, *10*, 1556–1560.

Kontonasaki, E., Giasimakopoulos, P., & Rigos, A. E. (2020). *Strength and aging resistance of monolithic Zirconia : an update to current knowledge*. *56*, 1–23.

Lan, T. H., Wang, C. H., Chen, K. K., Wang, M. C., & Lee, H. E. (2017). Milling properties of low temperature sintered Zirconia blocks for dental use. *Materials Science and Engineering C*, *73*, 692–699.

Lohbauer, U., Amberger, G., Quinn, G. D., & Scherrer, S. S. (2010). Fractographic analysis of a dental Zirconia framework : A case study on design issues. *Journal of the Mechanical Behavior of Biomedical Materials*, *3*(8), 623–629.

Mo, W. H., Bindl, A., & Lu, H. (2006). *Strength and fracture pattern of monolithic CAD / CAM-generated posterior crowns*. 29–36.

Schmitter, M., Lotze, G., Bömicke, W., & Rues, S. (2015). Influence of surface treatment on the in-vitro fracture resistance of Zirconia-based all-ceramic anterior crowns. *Dental Materials*, 1–9.

Schriwer, C., Skjold, A., Gjerdet, N. R., Øilo, M., & Cam, C. A. D. (2017). *ScienceDirect Monolithic Zirconia dental crowns . Internal fit , margin quality , fracture mode and load at fracture*. *3*(5009), 1012–1020.

Schünemann, F. H., Galárraga-vinueza, M. E., Magini, R., & Fredel, M. (2019). *Materials Science & Engineering C Zirconia surface modifications for implant dentistry*. *January*.

Smith, R. L., Villanueva, C., Rothrock, J. K., Garcia-godoy, C. E., Stoner, B. R., Piascik, J. R., & Thompson, J. Y. (2018). Long-term microtensile bond strength of surface modified Zirconia. *Dental Materials*, *27*(8), 779–785.

Vatali, A., Kontonasaki, E., Kavouras, P., Kantiranis, N., Papadopoulou, L., Paraskevopoulos, K. K. M., & Koidis, P. (2014). Effect of heat treatment and in vitro aging on the microstructure and mechanical properties of cold isostatic-pressed Zirconia ceramics for dental restorations. *Dental Materials*, *30*(10), e272–e282.

Bhushan, B. (2001). 2.1 The Nature of Surfaces. *Modern Tribology Handbook*, *1*, 49–120.

Coggins, L. W. (2001). Electron microscopy. *BioPharm*, *14*(12 SUPPL.), 19. https://doi.org/10.1002/0471443395.img111

Gadelmawla, E. S., Koura, M. M., Maksoud, T. M. A., Elewa, I. M., & Soliman, H. H. (2002). Roughness parameters. *Journal of Materials Processing Technology*, *123*(1), 133–145. https://doi.org/10.1016/S0924-0136(02)00060-2

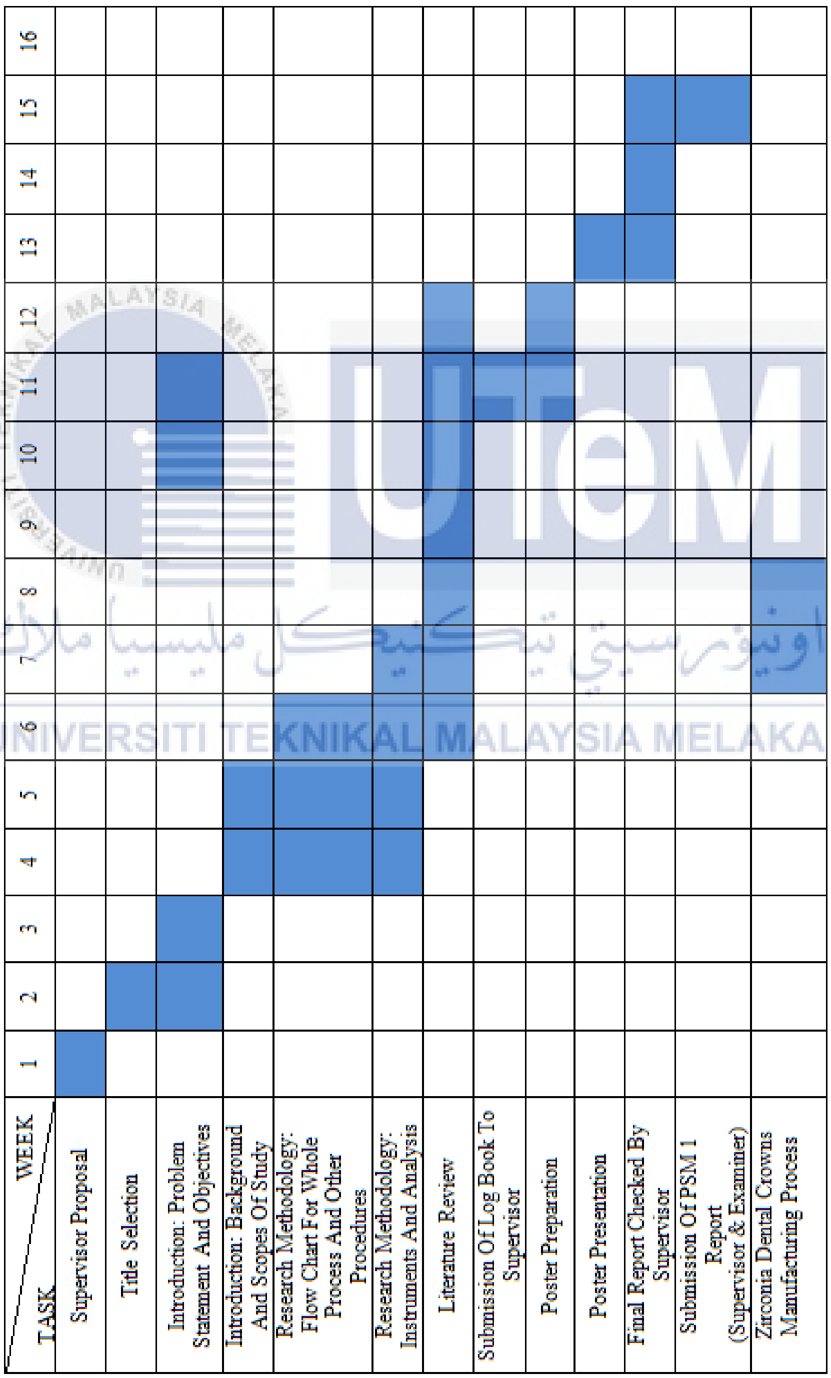
Leach, R. K., Giusca, C. L., Haitjema, H., Evans, C., & Jiang, X. (2015). Calibration and verification of areal surface texture measuring instruments. *CIRP Annals - Manufacturing Technology*, *64*(2), 797–813. https://doi.org/10.1016/j.cirp.2015.05.010

Ramos, G. F., Pereira, G. K. R., Amaral, M., Valandro, L. F., & Bottino, M. A. (2016). Effect of grinding and heat treatment on the mechanical behavior of Zirconia ceramic. *Brazilian Oral Research*, *30*(1). https://doi.org/10.1590/1807-3107BOR-2016.vol30.0012

APPENDICES

Appendix A

Gantt Chart for FYP I



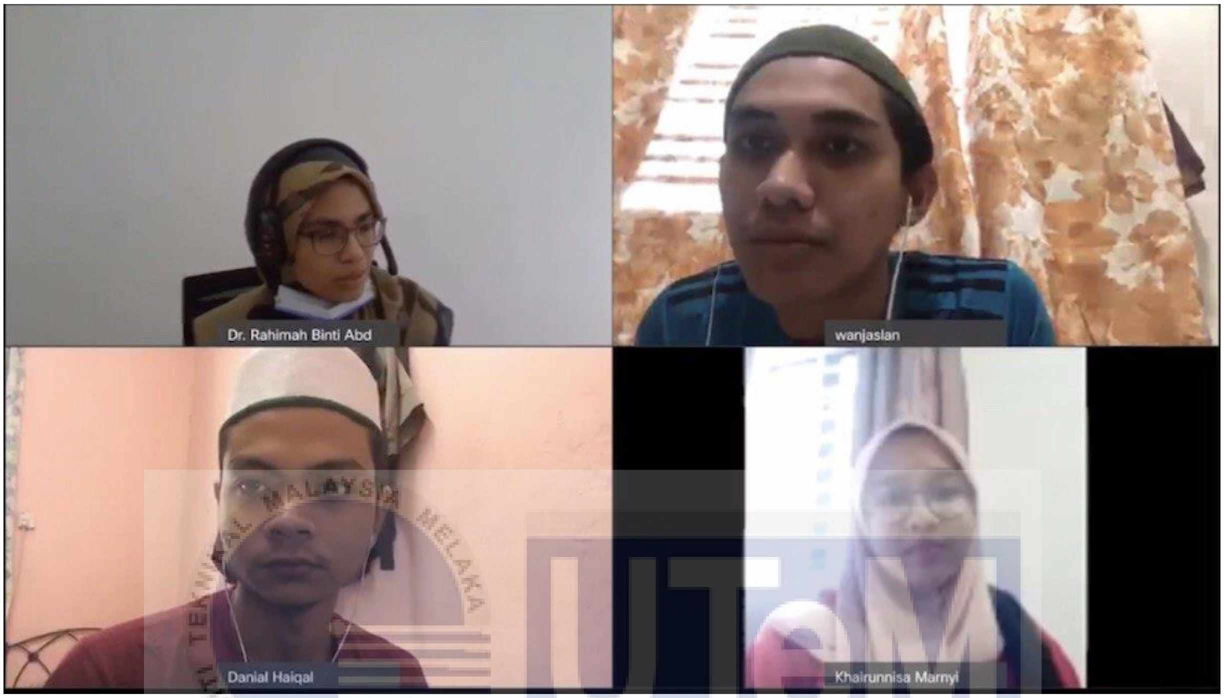
Appendix B

Gantt Chart for FYP II

TASK \ WEEK	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Dental Crowns Manufacturing Process	■	■															
Heat Treatment Test			■	■													
Surface Roughness Test					■	■											
Coating Process and Scanning Electron Microscope Analysis							■										
Analyse the Data from Test's Result			■	■	■	■	■	■	■	■	■	■					
Submission Of Log Book To Supervisor											■						
Video Preparation for Presentation										■	■	■	■				
Video Presentation														■			
Final Report Checked by Supervisor													■	■	■		
Submission Of FYP Report (Supervisor & Examiner)															■		
Submission of Final FYP Report to Supervisor																	■

Appendix C

Online Meeting with Supervisor



Appendix D

Question and Answer Session with Panels