An investigation into surface texture and *in-vitro* two-body wear of CAD/CAM dental materials antagonised by acrylic denture teeth

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This thesis reports on research completed during my Doctorate of Clinical Dentistry (Prosthodontics) at the University of Adelaide, from January 2012 to December 2014.

Two primary aims of the project were firstly, to investigate surface texture analysis technology use on dental materials, and secondly, to complete a two-body in-vitro wear study of modern dental restorative materials opposing a standardized artificial acrylic tooth.
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Abstract

Introduction:

Wear of teeth and of dental materials is a normal chemical and biomechanical process and is one of the physical and behavioral characteristics of dental restorative materials that need to be understood when planning the incorporation of a dental restoration into the stomatognathic system. The aims for this investigation were to analyse and compare two-dimensional (Ra) and three-dimensional (Sa) methods for surface texture analysis and to apply these methods in a two-body in-vitro wear study using PMMA-acrylic denture teeth as antagonists and a range of commonly used dental restorative materials as test specimens. The material wear characteristics of the antagonist and sample materials were analysed both qualitatively and quantitatively in order to better improve the decisions made by clinicians.

Methods and Materials:

The dental materials analysed are all commercially available CAD/CAM materials for clinical dentistry. They included three monolithic zirconia materials (BruxZir™, Lava Plus™, DenZir™), a lithium disilicate ceramic (eMax™), a nano-ceramic composite resin (Lava Ultimate™), and PMMA-acrylic denture teeth (Gnathostar™, Ivoclar Vivadent).

The first investigation involved SEM surface texture analysis of the materials using commonly applied 2-D (Ra) methodology and a novel 3-D (Sa) surface profile generation protocol (Me-X). Qualitative and quantitative information was considered to determine which method gave the most accurate and consistent results for dental material surface topography characterisation.
The second part of the investigation involved *in-vitro* two-body wear investigations using PMMA-acrylic denture teeth as an antagonist and samples from each of the test groups. A purpose-build wear simulator applied a load of 99.5N for 240,000 “chewing” cycles with distilled water lubrication. The total material volume loss for the test materials and PMMA-acrylic antagonist were analysed via 3-D surface profile methods. Qualitative analysis was undertaken via SEM.

**Results:**

Ra and Sa surface texture analysis and quantification indicated that the 3-D Sa method was more sensitive to surface topography variation in the materials than the 2-D Ra method. Both the Ra and Sa methods gave the same ranking of the materials in terms of which had more pronounced surface topography variation. The highest values were obtained for the PMMA-acrylic group, followed by Lava Plus™, DenZir™, Lava Ultimate™, eMax™, and BruxZir™ in descending Ra and Sa order. Post two-body wear testing analysis showed qualitative and quantitative changes in the materials. Obvious wear facets were noted for the acrylic antagonist and control groups and the Lava Ultimate™ test material. No wear facets were noted in the zirconia or eMax™ ceramic test samples.

The acrylic antagonist material showed the highest volume loss when opposing the nano-ceramic Lava Ultimate™ sample material, followed by the eMax™ group, then the acrylic control group. The Lava Plus™ group showed the least acrylic antagonist material volume loss. The highest overall test material volume loss occurred in the nano-ceramic composite resin (Lava Ultimate™), group followed closely by the acrylic-acrylic control group.
Conclusions:

The $Ra$ and $Sa$ surface texture analysis and quantification results indicated that either method could be used to analyse the surface roughness of dental materials. The $Ra$ method was quicker and more cost effective than the $Sa$ method. The highest values in descending $Ra$ and $Sa$ order were obtained for the PMMA-acrylic group, followed by Lava Plus™, DenZir™, Lava Ultimate™, eMax™, and BruxZir™. The monolithic zirconia material samples (BruxZir™, Lava Plus™, DenZir™) showed significant intergroup variation both qualitatively and quantitatively.

The two-body in-vitro wear analysis showed that the acrylic antagonist cusp had a clear wear facet for each test group. Results showed that the nano-ceramic composite resin material was more abrasive to the acrylic antagonist material than the other materials tested.
This work contains no material which has been accepted for the award of any other degree in any other tertiary institution, and to the best of my knowledge and belief contains no material previously published or written by another person, except where due reference has been made in the text.

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Jonathan M Innes

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Section 1

Introduction
Introduction

1.1  Tooth Wear

1.1.1  Background

Anthropologists have investigated tooth wear in contemporary and pre-contemporary human populations and have identified tooth wear as resulting from diet and the use of teeth as tools (Molnar 1972). The differences in observed tooth wear patterns have been attributed to cultural, gender, and environmental influences between and within different population groups (Molnar 1972; Richards 1984). Observations have also been made on modern populations such as Australian Aboriginals and associations drawn to geographical locations and ingested food types (Beyron 1964; Richards 1984).

Begg (1954) reported that tooth wear occurred on occlusal surfaces and inter-proximally, both of which were associated with human subject growth and development, and the maintenance of function (Barrett 1969). More recently, it has been reported that human diets have changed with an increased intake of softer food types and less coarse foods, which has resulted in less tooth wear (Kaifu et al. 2003). However, in 2008 the UN reported that the life expectancy of humans in western countries is increasing annually, which means that both natural and artificial teeth will need to survive for longer than was previously the case.

Clinically, attention must be paid to this “normal process” in order to minimise or cease its effect on the natural tooth structure and the stomatognathic system in general. Smith and Robb (1996) reported on the prevalence of tooth wear and provided a system for identifying tooth wear that should be considered excessive for the patients’ age and required treatment increases. Richards (1990) reported on the link between what was considered “excessive”
tooth wear and pathological developments in the stomatognathic system including the temporomandibular joints.

Dental clinicians aim to restore form, function, comfort and aesthetics to broken down teeth, however in order to do this, many artificial materials must and can be utilised. An understanding of the individual physical and behavioural characteristics of these materials is key when planning the incorporation of a restoration into the functional and dynamic oral and stomatognathic system.

1.1.2 Aetiology of Tooth Wear

Tooth wear is a natural and multifactorial process of which there are three main categories, attrition, abrasion, and erosion (Smith and Knight 1984).

Tooth wear progresses throughout life as a normal phenomenon and is accelerated in some cases by events such as changes in diet or stress leading to bruxism (Berry and Poole 1974). Therefore it is important in clinical practice to identify an accelerated rate of tooth wear at an early stage, diagnose its cause, apply preventive measures and monitor carefully over time (Smith 1996).

Al-Omiri et al (2006) investigated the influence of tooth wear on patients’ quality of life and satisfaction with their dentition. Of the subjects referred to a “tooth wear clinic”, 36% were dissatisfied with their teeth and aesthetics, compared to only 3.9% in a group of patients with tooth wear within “normal” physiological limits. These patients also reported that their tooth wear had a deleterious effect on chewing and eating ability and that the tooth wear caused pain, and oral discomfort. Thus, there was a clear impact on daily life activities for subjects suffering abnormal tooth wear.
1.1.2.1  Attrition

Attrition is recognised as tooth wear that results from direct tooth-to-tooth or material-to-material contact (Pindborg 1970) and is characterised by distinctive, well-defined, “shiny” wear facets on opposing teeth (Kaidonis 2008). That is, there is a lack of cupping or scooping on the worn surface (Figure 1) and microscopically, parallel striations can be seen (Figure 2) within the wear facet borders (Kaidonis 2008).

Figure 1. Clinical images of a 22-year-old male demonstrating generalised attrition associated with a parafunctional habit. Note the loss of the cusp tips, flattened occlusal surface, equal dentine and enamel tissue height, and communication of opposing tooth wear facets in lateral mandibular movement. (Courtesy Dr M Zaninovich)
Figure 2. Scanning electron microscope image of wear facet showing parallel striations. The dentine (d) is not scooped out and is at the same level as the enamel (e) (Kaidonis 2008)

Attrition has been reported to affect a tooth’s interproximal, occlusal, palatal, and/or labial surfaces as well as cusp tips and incisal edges (Lambrechts et al 1984). Depending on the severity, all dental hard tissues can be affected.

Attrition can be classified as either physiologic or pathologic depending on the extent of the loss tooth structure relative to the patient's age. Historically, tooth attrition was attributed to “parafunctional” activity such as tooth grinding, or the presence of “occlusal interferences”, that is tooth to tooth contacts that occur outside a theorised central or single occlusal position. However there is growing support for the idea that tooth grinding is a normal physiological behaviour as it has been observed in many different species (Every 1972; Kelleher and Bishop 1997).

Bruxism is defined in the Glossary of Prosthodontic Terms, 2005, as “the parafunctional grinding of teeth…an oral habit consisting of involuntary rhythmic or spasmodic nonfunctional gnashing, grinding, or clenching of teeth in other than chewing movements”
(Glossary of prosthodontic terms 8th edition, 2005). Xhonga (1977) demonstrated that the amount of enamel loss in an individual who is a “bruxer”, is three to four times greater than that found in individuals identified as non-bruxers. It has been suggested that there is a correlation between an individual’s bite force and the amount of tooth wear he or she demonstrates. For instance, Dahl et al (1993) noted that males have a higher bite force and thus a higher severity and prevalence of attrition-related tooth wear compared to females. However, the same group (Dahl et al 1985) had shown that muscle activity was not related to maximum bite force and in fact no difference in maximum bite force was found between groups who had “pathologic attrition” and those that had a “healthy dentition”.

Dentists have theorised that pathologic tooth attrition may also be related to a reduced number of functional dental units. For instance, Bernhardt et al (2004) showed that the increase in dental attrition was proportional to the number of occluding dental units lost. This finding refuted earlier evidence by Poynter and Wright (1990) who suggested that, due to a tooth’s periodontal proprioception, remaining teeth could not be subjected to increased load and found no evidence of increased incisor tooth wear when posterior teeth were lost.

1.1.2.2 Abrasion

Abrasion occurs when exogenous materials are forced over the surfaces of occluding teeth (Kaidonis 2008). Abrasion-related tooth wear results in larger worn areas than attrition as it results from abrasive, coarse materials being forced over the entire occlusal surface of teeth. Anthropological studies have shown that this type of wear has reduced in incidence in recent times due to our contemporary diet being made up of less coarse foods (Kaidonis 2008). The exogenous material can be any material, including food or dirt, and can be associated with
habits including items individuals place between their teeth such as tools, pipes, or bobby pins (Kaidonis 2008; Hattab and Tassin 2000).

Richards and Brown (1981) suggested that abrasion-related tooth wear increases with age and suggested that the rate is correlated with the abrasiveness or coarseness of foods consumed.

Abrasion-related tooth wear is characterised by pitted or gouged areas in enamel, and shows a “scooped out” appearance when dentine is involved (Figures 3 and 4 from Kaidonis 2008). Microscopically, the area of abrasive wear shows “haphazard scratch marks” (Figure 5).

Figure 3. Clinical presentation of abrasion lesions on the occlusal surfaces in a 43 year old patient with a history of gastric oesophageal reflux disease, severe parafunctional activity, and a hard bristled tooth brush. Note the striations of the enamel and scooping of the dentine. (Courtesy Dr Zaninovich)
Figure 4. An example showing the effect of an abrasive diet on the teeth of a pre
contemporary Australian Aboriginal. Note the gouged and pitted enamel and
the scooping of the dentine (Kaidonis 2008)

Figure 5. Microwear detail of an abrasion area showing a haphazard scratch marks
(Kaidonis 2008)
Non-carious cervical lesions (NCCLs) are lesions of tooth wear at a tooth’s cervical area and are the most common type of tooth wear lesion reported in human populations (Litonjua et al 2003). A common presentation for this type of lesion is the “wedge-shaped” NCCL, the aetiology of which is controversial with some believing that it is solely an abrasion lesion associated with tooth brush and toothpaste abrasion (Dawson 2007; Litonjua et al 2003). Others refer to these lesions as “abfraction lesions” and hypothesise that they result from concentrated tensile stress at the tooth's cervical area due to flexure under loading (Addy and Shellis 2006). Nguyen et al (2008) investigated these lesions and found that they are in fact the result of two processes, abrasion and erosion. Horizontal furrows were identified microscopically which were related to toothbrush-type abrasion. However, the authors highlighted that tensile stress cannot be completely ruled out.

1.1.2.3 Erosion

Dental erosion is the loss of tooth structure that results from attack by acids that are not of bacterial origin (Pindborg 1970). Smith (1989) stated that erosion is a chemical process, however mechanical factors (abrasion and attrition) contribute to the loss of tooth structure as acid-softened surfaces are more susceptible to mechanical wear. Acids that are commonly associated with erosive tooth wear include the endogenous acids resulting from gastric acid regurgitation (GORD) and bulimia nervosa and exogenous (e.g. acid drinks, fruit). Occupation-related sources including wine tasting and working in dusty environments are also implicated (Wiegard and Attin 2007). Saliva also plays a key role with those with diminished salivary buffering capacity showing increased rates of erosive tooth wear (Dahl et al 1993; Meyers 2008).
Several researchers have identified a “high” and increasing prevalence of erosion-related tooth wear. For example Auad et al (2009) highlighted the increasing prevalence in the adolescent UK population with 37% of 14 year olds being affected. Signs of dental erosion that are evident in early stages include: “glossy enamel” (i.e. smooth, glazed); yellowing of teeth from underlying dentine; increased incisal translucency; and cupping of the occlusal surface (Vialati and Belser 2012). Dentinal scooping is characterised by intact dentinal tubules and may present as tooth sensitivity (Kaidonis 2008). The characteristic erosion lesions are microscopically smooth and lack caries as shown in Figures 6 and 7.

![Figure 6](image_url)  
*Figure 6. A micrograph of an erosion lesion. The lack of mechanical wear characteristics is noteworthy (image courtesy of Dr S. Ranjitkar).*
1.1.3 The Chewing Cycle

The chewing cycle can be divided into three distinct phases; preparatory, crushing, and gliding (Begg 1954; Barrett 1969; Kaifu et al 2003; Smith and Robb 1996).

The initial preparatory phase involves positioning the mandible for food bolus contact. The jaw opens and continues through the closing movement until the teeth contact the food bolus. No occlusal forces are applied during this phase except with sticky foods (Barrett 1969; Kaifu et al 2003).

Next is the crushing phase in which there is a three-body interaction of the opposing teeth with the food bolus. It begins when teeth contact the food bolus and ends when there is tooth-to-tooth contact or until the mandible opens or re-enters the preparatory phase (Barrett 1969; Kaifu et al 2003). At initial food contact, forces are directed through the food bolus and the
force magnitude experienced by the teeth is dependent on the type or stiffness of the food bolus (Barrett 1969; Kaifu et al 2003; Smith and Robb 1996). The food bolus is compressed and the masticatory force is distributed over the surface of the food bolus which is in contact with the maxillary and mandibular teeth, thus as the contact surface area increases, the force per unit area decreases (Barrett 1969).

The final phase is the gliding phase which may or may not occur during mastication. The gliding phase starts with tooth-to-tooth contact after complete penetration of the food bolus (if food is present) and continues until the jaw re-enters the preparatory phase (Barrett 1969; Kaifu et al 2003; Smith and Robb 1996). The force of mastication is concentrated through the tooth contact, and thus two-body wear occurs (Barrett 1969). At this time, three-body wear is also occurring when a food bolus is present (Barrett 1969). DeLong and Douglas (1983) described a formula for the subsequent tooth wear as being equal to volume loss which is a result of the wear mechanism multiplied by the occlusal force and total sliding distance divided by the substrate hardness and pressure. These are physiological, dental material and food dependent variables that are dynamic rather than constant in nature.

1.1.4 Methods of assessment of tooth wear

Tooth wear can be assessed qualitatively and quantitively both in vivo and in vitro.

Richards et al (2003) presented a “simple” mathematical model of occlusal and incisal tooth reduction for the clinician to predict the progress of tooth wear in individual patients over time. The investigators used a diagram-based system for scoring the extent of tooth surface loss and the age of the patient to predict tooth wear outcomes from an age-based graph which classified the wear rate as low, medium, or high.
Azzopardi (2000) described how in vivo tooth wear can be assessed using tooth wear indices such as that described by Smith and Knight (1984), or by comparing longitudinal changes in tooth form to a series of models or photographs. They stated that tooth wear indices cannot detect subtle changes in tooth wear and are thus indicated only for epidemiological studies. They discussed four quantitative methods for investigating in vitro and in situ wear which were: chemical methods; microradiography; digital image analysis; profilometry and surface mapping. The methods have been reported in literature and each have inherent advantages and disadvantages.

There are two forms of profilometry available. Firstly, contacting, which consists of a mechanical stylus sensor which contacts the sample with low force and secondly, non-contacting which commonly uses a laser to scan in either two or three dimensions (Mehl et al 1997, Azzopardi 2000, DeLong 2006). The latter is much quicker in recording data than the contacting devices. However it has stringent sample opacity requirement and can record “over shoots”, thus providing inaccuracy.

A key aspect of investigating changes in tooth wear, is maintaining the correct orientation of the sample to the scanner. Azzopardi (2000) reported that profilometry is able to measure surfaces with precision of 2.2µm and an accuracy of approximately 10µm (Azzopardi 2000).

Errors in wear measurements can be introduced when a stylus instrument is used by factors that include: the size of the stylus; stylus load; stylus speed; and, lateral deflection by asperities. The influence of stylus size is illustrated in Figure 8. The effect of stylus size on measurements becomes more significant as the curvature of the peaks and valleys decreases, or the magnitude of the slope increases (Thomas 1998). It has been reported that stylus speed and lateral deflection are generally only minor sources of error (Thomas 1998). In addition the contacting stylus may damage the surface of a specimen, especially if the surface is demineralised or has a low hardness (Attin 2006; DeLong 2006)
Figure 8. Distortion of a surface profile due to the effect of stylus size. This Figure is a schematic diagram comparison of an actual profile against the traced profile (Thomas 1998).

Scanning electron microscopy (SEM) has been used as a tool for both quantitative and qualitative analysis as it produces high resolution and magnification images (Attin 2006). SEM can now be used in conjunction with computer software to produce an image of the three dimensional surface characteristics of the sample material surface and wear facet. This image can be analysed to produce both quantitative and qualitative data (Bello et al 2011).

1.1.5 Surface texture analysis

1.1.5.1 Two dimensional surface texture analysis

High quality characterisation of surface topography is important in applications involving friction, lubrication, and wear (Thomas 1998). Generally, it has been found that friction increases with average roughness and therefore the roughness of ceramics and dental hard tissue is an important variable, as it plays a major role in the wear process.
The characterisation of a material’s surface topography, texture and/or roughness can be done in two principle planes (Thomas 1998). Using a sinusoidal curve as a simplified model of the surface profile, material's surface roughness can be measured at right angles to the surface in terms of the wave amplitude, and/or parallel to the surface in terms of the wavelength. The latter plane is also recognised as texture. The technique used to measure roughness in either of these two planes will inevitably have limitations.

Instrument limitations include vertical and horizontal resolution, which correspond to the smallest amplitude and wavelength the instrument can detect. Also, the vertical and horizontal range of the instrument correspond to the largest amplitude and wavelength that can be measured (Thomas 1998).

In statistical terms, the parameters used to characterise material surface topography are divided into central, second, third and fourth “moments”. Central moments are parameters which assess the distribution of heights such as centre average line (CLA) or average surface roughness (Ra or Sa) and root mean square (RMS) (Mair 1999). The second moment is known as the “variance” and represents the deviation of the distribution from its mean. The third moment is the “skewness” and is a measure of the asymmetry of the distribution. The fourth moment is known as the “kurtosis” which represents the shape of the distribution curve (Thomas 1998).

Amplitude parameters are the most important parameters to characterise surface topography. They are used to measure vertical characteristics of surface deviations (Gadelmawla et al 2002).

In addition to the amplitude parameters, other parameters are used to characterise or evaluate material texture. The high-spot count (HSC) is the number of peaks per unit length. Its reciprocal, Sm, is the mean spacing between the peaks. The profile length ratio RL is the
length of the profile divided by its nominal length. Currently there are more than 100 two-dimensional parameters that have been described in the literature (Thomas 1998). Common surface texture parameters are listed in Table 1.

Table 1: Surface texture parameters. (*From Leigh 1992*)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra</td>
<td>Roughness Average (Ra)</td>
</tr>
<tr>
<td>Rq</td>
<td>Root Mean Square (RMS) Roughness</td>
</tr>
<tr>
<td>Rt</td>
<td>Maximum Height of the Profile</td>
</tr>
<tr>
<td>Rv, Rm</td>
<td>Maximum Profile Valley Depth</td>
</tr>
<tr>
<td>Rp</td>
<td>Maximum Profile Peak Height</td>
</tr>
<tr>
<td>Rpm</td>
<td>Average Maximum Profile Peak Height</td>
</tr>
<tr>
<td>Rz</td>
<td>Average Maximum Height of the Profile</td>
</tr>
<tr>
<td>Rmax</td>
<td>Maximum Roughness Depth</td>
</tr>
<tr>
<td>Rc</td>
<td>Mean Height of Profile Irregularities</td>
</tr>
<tr>
<td>Rz(iso)</td>
<td>Roughness Height</td>
</tr>
<tr>
<td>Ry</td>
<td>Maximum Height of the Profile</td>
</tr>
<tr>
<td>Wt, W</td>
<td>Waviness Height</td>
</tr>
<tr>
<td>S</td>
<td>Mean Spacing of Local Peaks of the Profile</td>
</tr>
<tr>
<td>Sm, RSm</td>
<td>Mean Spacing of Profile Irregularities</td>
</tr>
<tr>
<td>D</td>
<td>Profile Peak Density</td>
</tr>
<tr>
<td>Pc</td>
<td>Peak Count (Peak Density)</td>
</tr>
<tr>
<td>HSC</td>
<td>Height Spot Count</td>
</tr>
<tr>
<td>λa</td>
<td>Average Wavelength of the Profile</td>
</tr>
<tr>
<td>λq</td>
<td>Root Mean Square (RMS) Wavelength of the Profile</td>
</tr>
<tr>
<td>Δa</td>
<td>Average Absolute Slope</td>
</tr>
<tr>
<td>Δq</td>
<td>Root Mean Square (RMS) Slope</td>
</tr>
<tr>
<td>Lo</td>
<td>Developed Profile Length</td>
</tr>
<tr>
<td>Ir</td>
<td>Profile Length Ratio</td>
</tr>
<tr>
<td>Rsk,Sk</td>
<td>Skewness</td>
</tr>
</tbody>
</table>

1.1.5.1.1 Material Roughness Amplitude Parameters

Average Material Roughness (*Ra*) describes the overall mean roughness of a surface. *Ra* is the area between the roughness profile and its mean line over a sampling length or the integral of the absolute value of the roughness profile height over the evaluation length. It is also known as arithmetic average, centre line average, and arithmetical mean deviation of the profile (Poon and Bhushan1995).
Average roughness ($Ra$) is recognised as one of the most effective and common surface roughness measures. It is simple to define, easy to measure, requires the least sophisticated instruments and also gives a good general description of the height variations of a surface. Average roughness ($Ra$) does however provide limited information on the assessed profile and the interpretation of the given $Ra$ value is difficult. This is because it is a true amplitude parameter and gives no information of the profile data. Specifically it does not give any information on the spatial structure such as the wavelength, does not differentiate between peaks/valleys (Gadelmawla et al. 2002) and is not sensitive to small changes in profile (Thomas 1998) (Figure 9).

![Figure 9](image)

**Figure 9.** The limitations of Average mean roughness. All graphical representations showing surface roughness have the same $Ra$ value. Differences between peaks and valleys (A) and variations in wavelength (B) are not evident in average mean roughness scores (Gadelmawla 2002; Thomas 1998).

The mathematical definition and the digital implementation of the arithmetic average height parameter are shown respectively in Figure 10 and correlate with the graphical representation shown in Figure 11.
Figure 10. Mathematical definition and the digital implementation of arithmetic average height parameter \((Ra)\). \(l\) = evaluation length, \(y\) = height, \(x\) = distance along measurement, \(n\) = number of samples, \(y(x)\) = profile ordinates of roughness profile (Mair 1999).

\[
Ra = \frac{1}{l} \int_0^l |y(x)| \, dx
\]

\[
Ra = \frac{1}{n} \sum_{i=1}^n |y_i|
\]

Figure 11. \(Ra\) is the arithmetic mean of the absolute departures of the roughness profile from the mean line. It is the most often used parameter of roughness (Mair 1999).

The Root Mean Square roughness (RMS or \(Rq\)) is the root mean square average of the roughness profile ordinates (Leitao and Hegdahl 1981). RMS represents the standard deviation (SD) of the distribution of surface heights, and is therefore an important statistical
parameter to describe the surface. RMS is more sensitive to large deviations from the mean line than the arithmetic average surface roughness (Ra). The mathematical definition and the digital implementation of this parameter are shown in Figure 12.

$$R_q = \sqrt{\frac{1}{l} \int_0^l \{y(x)\}^2 \, dx}$$

Figure 12. Mathematical definition and the digital implementation of the Root Mean Square roughness (RMS or $R_q$): $l = \text{evaluation length}$, $y = \text{height}$, $x = \text{distance along measurement}$, $n = \text{number of samples}$, $y(x) = \text{profile ordinates of roughness profile}$ (Leitao and Hegdahl 1981).

RMS provides statistical information on the symmetry of the surface distribution however, similar to average roughness average (Ra), its disadvantages are that it provides no spatial structure information and does not take into account the differences between peaks and valleys (Leitao and Hegdahl 1981). The RMS mean line is the line that divides the profile so that the sum of the squares of the deviations of the profile height from it is zero (Figure 13).

$$R_q = \sqrt{\frac{1}{n} \sum_{i=1}^n y_i^2}$$

Figure 13. The RMS mean line divides the profile so that the sum of the squares of the deviations of the profile height from it is equal to zero (Leitao and Hegdahl 1981).
The mean roughness depth parameter ($R_z$) is a surface texture parameter which is more sensitive to occasional high peaks or deep valleys than average surface roughness ($Ra$) as it examines maximum profile heights and not averages (Leitao and Hegdahl 1981). The Single Roughness Depth ($R_{zi}$) is the vertical distance between the highest peak and the deepest valley within a given sampling length. The Mean Roughness Depth ($R_z$) is the arithmetic mean value of the single roughness depths of consecutive sampling lengths (Leitao and Hegdahl 1981). The mean roughness depth, $R_z$, is defined by two methods according to the definition system. The international ISO system defines this parameter as the difference in height between the average of the five highest peaks and the five lowest valleys along the assessment lengths of the profile. The German DIN system defines $R_z$ as the average of the summation of the five highest peaks and the five lowest valleys along the assessment length of the profile (Azzopardi 2000; Leitao and Hegdahl 1981). The mathematical and graphical definition is shown in Figure 14.

$$R_z = \frac{1}{n} (R_{z1} + R_{z2} + \ldots + R_{zn})$$

![Diagram showing mathematical and graphical definition of mean roughness parameter $R_z$.](image)

**Figure 14.** The mathematical and graphical definition of mean roughness parameter $R_z$ where $n$ is the number of samples along the assessment line (Leitao and Hegdahl 1981).
The Maximum Roughness Depth ($R_{max}$) is the largest single roughness depth within the evaluation length. The $R_{max}$ parameter is useful for surfaces where a single defect is not permissible. $R_z$ and $R_{max}$ are used together to monitor the variations of surface finish in a production process. Similar values of $R_z$ and $R_{max}$ indicate a consistent surface finish, while a significant difference indicates a surface defect in an otherwise consistent surface (Thomas 1998).

Parameters which describe the roughness profile slope include the mean width of profile elements and the root mean square slope (Poon 1995). The mean width of profile elements ($R_{Sm}$) is the arithmetic mean value of the widths of the profile elements of the roughness profile, where a profile element is a peak and valley in the roughness profile (Poon 1995). The root mean square slope ($R_{sq}$) is the root mean square average of all local profile slopes and is seen in Figure 15. Each slope is calculated using a smoothing algorithm to reduce the effect of random noise on the value of $R_{sq}$ (Leitao and Hegdahl 1981).

Figure 15. The mean width of profile elements ($R_{Sm}$) is the arithmetic mean value of the widths of the profile elements of the roughness profile, where a profile element is a peak and valley in the roughness profile (Poon 1995).
The Mean Leveling Depth parameter \((Rpm)\) measures the mean value of the leveling depths of five consecutive sampling lengths. Exceptional profile peaks are thus only partly considered. By contrast to the surface parameters already described such as \(Ra\) and \(Rz\), the \(Rpm\) parameter gives reliable information on the profile shape. Small \(Rpm\) values characterise a surface featuring wide peaks and narrow valleys whilst greater \(Rpm\) values indicate a spiky, sharp ridge profile (Poon 1995). The ratio of RPM to \(Rz\) \((Rpm:Rz)\) is of special interest because the value quantifies the asymmetry of a given profile (Mair 1992; Mair 1996) and gives valuable information on profile shape. A \(Rpm:Rz\) ratio higher than 0.5 indicates a sharp ridge profile, whereas a ratio smaller than 0.5 indicates that the profile is rounded (Whitley et al 1987). The leveling depth \((Rp)\) is also the largest of the five leveling depths. The maximum roughness depth, \((Rt)\), peak to valley height, is the vertical distance between the highest peak and the lowest valley of the roughness profile, \(R\), within the evaluation length, \(l\). (Figure 16).

![Figure 16. Mean leveling depth \((Rpm)\) parameter measures the mean value of the leveling depths of five consecutive sampling lengths. The leveling depth \((Rp)\) is also the largest of the five leveling depths. The maximum roughness depth \((Rt)\), peak to valley height, is the vertical distance between the highest peak and the lowest valley of the roughness profile \(R\) within the evaluation length \(l\). \(Rpm = 1/5 \ (Rp1 + Rp2 + Rp3 + Rp4 + Rp5)\) (Mair 1992; Poon 1995)
1.1.5.2 Three Dimensional (3-D) Surface Texture Analysis

The SEM image formation process is based on “perspective projection” and, similar to conventional light microscopy, a three dimensional object is projected onto a two dimensional image plane with the loss of the information in the third dimension. SEM provides a large depth of focus, but does not allow depth measurements and thus only two dimensional measurements are feasible.

A eucentric tilting of the SEM stage, which means that the intersection of the primary electron beam with the specimen defines the centre of tilting, yields a second image of a material specimen observed from a different viewpoint. In this way a stereoscopic image is generated. Subsequently, overlay techniques including red-green colouring provide anaglyph viewing and thus depth perception but at this stage, no quantitative analysis can be undertaken.

Utilising analysis software (MeX, Alicona), reference points called “homologous points”, can be aligned on each image taken. This allows the true three dimensional (3D) coordinates of the observed points to be recovered. Each point is a single pixel. A third image, from a third tilt position, allows the software to generate a dense 3D model of the material surface, from which measurements can be made. Traditional surface texture analysis only utilises two images, and thus the third image allows improved accuracy by a reduction of error influencing variables such as the stage tilt angle. Figure 17 shows an overview of the imaging process.

The MeX software utilises EN/ISO standards for material surface area and texture analysis and can also measure material volume.
Figure 17. Image acquisition and construction of the 3-D representation of the material surface with MeX software. (from http://www.alicona.com)
This form of surface texture analysis has been employed in the analysis of bone and tooth surfaces in fossil specimens (Bello et al 2011) and perikymata (Bocaego et al 2011). Both reported that a detailed 3D model of the surface can be constructed from the series of images, calculating x, y, and z coordinates for any point within the scan images with a resolution of up to 0.4µm horizontally and 0.01µm vertically.

The tilting of the stage enables the elimination of errors related to the steepness of slopes. For instance, historically with microscopic analysis, slopes of 80° or more were unmeasurable. The stage tilting approach allows the accurate measurement of peaks with slopes greater than 80° (Bello et al 2011).

The 3D model is rendered as a rotatable, shaded image by the MeX software. Prior to analysis, the plane to which all the measured points are referenced is adjusted to correct for bias introduced by the stereoscopic images (Bello et al 2011). This process involves numerical algorithms based upon pixel-to-pixel image intensity and depth. Subsequently, a profile path oriented perpendicular to the line of the surface irregularities is defined on each image field. Following filtering of the high-frequency irregularities on the profile, which separates the roughness from the waviness and the overall geometrical form of the surface, the coordinates of the profile path are used to construct a quantitative profile diagram (Bocaego et al 2010). This diagram represents a selected part of the profile path that is delimited according to internationally standardised surface roughness parameters (ISO 4288). The profile diagram is able to reveal many structural irregularities on material surfaces that are quantifiable.

The two-dimensional measurements are made along a single line between two points at a given distance and the measurements are taken in a two-dimensional manner. The 3D method here requires a rectangular surface area to be scanned and selected which results in a significantly larger field and importantly a larger sample area from which 3D interpretation can be made (Figure 18).
Figure 18. 3D Diagram of material surface generated with MeX software.

The surface texture analysis using the 3D technique described here results in five measurable variables which are comparable to those attainable by 2D methods. The 3D variables are measured in nanometers (nm) and include:

- $S_a$: Average height of selected area
- $S_q$: Root-Mean-Square height of selected area
- $S_p$: Maximum peak height of selected area
- $S_v$: Maximum valley depth of selected area
- $S_z$: Maximum height of selected area
1.1.6 Tribology

Multiple definitions for tribology exist. In its simplest terms, Mair (1992) defined it as “the study of friction, lubrication, and wear”. An important, basic principle in wear studies that is not often appreciated is that no known surface is perfectly smooth, and under magnification surfaces will appear wavy and rough to varying degrees (Kragelskii 1965). When considering two surfaces being brought into contact, the contact is “point to point”, and the number of contacts increases as the surfaces are brought closer together by increasing force. In a dynamic process, these contacts also change over time.

Halling (1975) described how the interaction of two solid surfaces within a given environment results in two phenomena. Firstly, there is energy dissipation which is the resistance to motion and is indicated by the coefficient of friction. This energy dissipation results in a heat release at the contact and noise. Secondly, during the sliding process all surfaces are to a greater or lesser extent changed in their basic characteristics. They may become smoother or rougher, have physical properties such as their hardness altered, and some material may be lost in the process.

Halling (1975) also stated that the consequence of the dynamic interaction of two surfaces is the “wear process” which involves a variety of factors that are difficult to quantify. He went on to define wear as “the progressive loss of substance from the surface of a body brought about by mechanical action”. Earlier, Kragelskii (1965) had defined wear as "the destruction of material produced as a result of repeated disturbances on the frictional bonds”.

Generally in tribology, there are several definitions used for various types of wear. They have been classified by Pugh (1973) as: abrasive wear; adhesive wear; fatigue wear; corrosive wear; erosive wear; and fretting wear.
Importantly, even though some of the terminology overlaps with that used by dentists the definitions of the terms are very different.

1.1.6.1 Abrasive wear

Mair (1992) reported that abrasive type wear is the most common and defined it as the cutting away of a surface by abrasive asperities or particles. Abrasive wear occurs as a result of asperities ploughing into a softer surface which results in either plastic deformation without the removal of material, or low cycle fatigue which results in material loss as demonstrated in Figure 19 (Mair 1992, Mair 1996).

![Figure 19. Abrasive wear (from Mair 1996)](image)

Several factors have been noted by Mair (1996) and Halling (1975) as influencing abrasive wear. These include the hardness of the contacting materials; abrasive particle geometry; load applied; and the distance of the slide contact.

There are two forms of abrasive wear, namely “two-body” and “three-body” wear. Two-body wear occurs as a result of two opposing surfaces wearing against each other directly as demonstrated in Figure 20 (Mair 1992; 1999).
Figure 20. Two-body abrasive wear (from Mair 1999). Both surfaces are brittle and there is subsequent fracture of the asperities. The lower surface is “softer” than the upper surface which “plows” into the softer surface.

Figure 21 shows three-body wear, which occurs when a third “body” of abrasive particles is interposed on the wear interface between two opposing surfaces which are being rubbed together. Three body wear commonly follows an initial two-body wear period (Mair 1992; Mair 1996).

Figure 21. Three-body abrasive wear (from Mair 1999). The abrasive particles in the slurry flow under pressure and cut away the surfaces.
1.1.6.2 Adhesive wear

Adhesive wear is the result of “cold welding” of the protuberances of contacting surfaces due to friction between two moving surfaces (Mair 1992; Mair 1996; Halling 1975; Zum-Gahr 1987). As the movement continues, the welds are sheared, which if it occurs at the subsurface level results in the deposition of the separated material onto the opposing surface. With further movement, this material may form a loose particle “third-body” of material which results in abrasion. This process is illustrated in Figure 22 (Mair 1996).

![Figure 22](image)

Figure 22. Adhesive wear (from Mair 1996). This Figure shows the “cold welding” and detachment of material.

1.1.6.3 Fatigue wear

Fatigue wear results when two surfaces moving under dynamic loads cause the formation and propagation of subsurface micro-cracks. Loss of material occurs via delamination due to the growth and propagation of subsurface micro-cracks resulting from cyclical loading of the opposing surface layers with repetitive compressive, tangential and tensile stresses, as shown in Figure 23 (Mair 1992; Mair 1999).
Figure 23. Fatigue wear (from Mair 1996). A rolling action of two surfaces causes shear subsurface stresses which result in nucleation of cracks which propagate laterally to the material surface with the continued rolling action resulting in delimitation and fatigue wear or failure.

Suh (1973) introduced the theory of delamination to fatigue wear. Delamination is defined as the formation of fractured surface material or “chips” and occurs after a critical number of loading cycles when the surface material becomes surrounded by a network of linked cracks. The breakup of the material surface increases the risk of the formation of larger fragments which leads to larger pits on the surface (Suh 1973; Jahanmir 1994).

The rate at which fatigue wear occurs is non-linear and behaves much differently from adhesive or abrasive wear. With fatigue, negligible wear occurs prior to the point of surface fracturing, whereas in both adhesive and abrasive wear mechanisms, there is a gradual deterioration of the surface. The time to fatigue failure is dependent on several factors including the amplitude of the reversed shear stresses, the interface lubrication conditions, and the fatigue properties of the materials (Mair 1992; Mair 1999). It has been shown that adhesive wear can exacerbate surface fracturing and delamination by weakening the subsurface thus allowing adhesive forces to “pluck out” the surface fragments (Suh 1973).
1.1.6.4 Corrosive (Tribochemical) wear

Corrosive wear is the wear process involving the chemical degradation of a surface that is rubbed away by an opposing surface and differs from corrosion which is a static chemical degradation of a surface (Mair 1992; Helling 1973; Zum-Gahr 1987). In a static environment the products of corrosion form a barrier which slows the rate of corrosion. When there is a rubbing or sliding action, this protective barrier is worn away, exposing the original material and allowing the corrosive attack to continue (Mair 1996). This process is illustrated in Figure 24.

![Corrosive wear](image)

Figure 24. Corrosive wear. This process results from a sliding movement of opposing materials in a corrosive environment (from Mair 1996).

1.1.6.5 Erosive wear

Plugh (1973) described erosive wear as damage occurring in a solid body by the impact of external particles or fluid under pressure. The wear medium here is particles or fluid which acts as the second surface. An example is the erosion of rocks by water.
1.1.6.6 Fretting wear

Halling (1975) defined fretting wear as wear resulting from the low amplitude vibratory motion that takes place between two surfaces when they are loaded together. Mair (1992) stated that there is no evidence that fretting wear occurs in the mouth and is thus not relevant regarding dental wear.

Tribology concepts are based on the observations of tribologists on a micro- and nano-scale. The understanding of the mechanical and chemical engineering principles of the wear process aids the interpretation and understanding of the macro-level wear of dental tissue. Dental terminology has generally been used in this project other than when the alternative, more specific tribology terminology allows a clearer explanation of an observation or process.

1.2 Relevant Dental Materials

In this section relevant properties of each of the materials included in this study are reviewed to provide a background for the interpretation of the results.

1.2.1 Acrylic Prosthetic Teeth

Acrylic resin denture teeth present advantages over porcelain denture teeth. These include: being less brittle; superior bonding to acneic denture base materials; ease of adjustment and polishing, a more natural appearance; and, less noise produced during mastication (Mello 2009).

Historically, acrylic teeth have shown a higher wear rate compared with porcelain denture teeth which has longer term implications for the maintenance of function and occlusal vertical
dimension. Significant improvements have been made in the formulation of acrylic denture teeth to improve their mechanical properties. These improvements include an interpenetrated polymer network (IPN), polymers with double-cross links (DCL), and resin teeth with added inorganic agents (Mello 2009).

Polymethyl methacrylate (PMMA) was developed in the 1930s by chemists Rowland Hill and John Crawford and was first registered as Perspex®. It is a synthetic resin produced by the polymerisation of methyl methacrylate and is commonly used in the manufacture of denture teeth.

Methyl methacrylate is an ester of methacrylic acid (CH2=C(CH3)CO2H) obtained primarily from propylene, a compound refined from the lighter fractions of crude oil. Propylene and benzene are reacted together in order to form cumene, or isopropylbenzene; the cumene is then oxidized to cumene hydroperoxide, which is treated with acid to form acetone. Acetone is in turn converted in a three-step process to methyl methacrylate (CH2=C(CH3)CO2CH3). Methyl methacrylate, in bulk liquid form or suspended as fine droplets in water, is polymerized under the influence of free-radical initiators to form solid PMMA (Anusavice 2003).

The chemical structure of the polymer repeating unit is:

\[
\begin{array}{c}
\text{CH}_3 \\
\text{CH}_2 - \text{C} - \\
\text{CO}_2\text{CH}_3
\end{array}
\]

The presence of the pendant methyl (CH₃) groups prevents the polymer chains from packing closely in a crystalline fashion and from rotating freely around the carbon-carbon bonds. As a result, PMMA is a tough and rigid material.
1.2.2 Nano-Ceramic Composite Resin (LAVA Ultimate, 3M ESPE)

Lava Ultimate Resin Nano Ceramic (RNC) is a novel dental material designed specifically for CAD/CAM (Computer Aided Design/Computer Aided Manufacture) use for dental restorations (Figure 25).

![LAVA Ultimate CAD/CAM blocks](http://www.3m.com/3M/en_US/Dental/Products/Lava-Ultimate/)

The material consists of nano-ceramic particles (80%wt) embedded in a highly cross-linked resin matrix (20%wt). Aspects of the manufacturing are proprietary process secrets (3M Company information). The nanometer particles are mono-dispersed, non-aggregated, and non-agglomerated nanoparticles as shown in Figure 26.

![Nano-ceramic aggregates](http://www.3m.com/3M/en_US/Dental/Products/Lava-Ultimate/)
LAVA Ultimate restorative contains two types of nanoparticles: silica nanomers of 20nm diameter, and zirconia nanomers of 4-11nm diameter. The nanomers are treated with a silane coupling agent which bonds to the nano-ceramic surface and the resin matrix during block manufacture. The “nano-cluster particles” consist of bound aggregates of engineered nanoparticles. The nano-clusters have structural integrity that allow a high proportion of ceramic filler to be incorporated into the blocks which provides strength and both fracture and wear resistant properties. The average nanocluster size is 0.6-10μm. The LAVA Ultimate Flexural strength is reported to be 200MPa.

1.2.3 Ceramics

1.2.3.1 Historic Background

Ceramic use in dentistry originated in 1774 when Alexis Duchateau, a French apothecary, and a Persian dentist, Dubois de Chemant fabricated porcelain paste for denture construction as an alternative to ivory which absorbed oral fluids, stained and was unhygienic (Jones 1985; Van Noort 1994; Craig 2006). Following de Chemant’s immigration to Britain, he collaborated with the Wedgewood porcelain factory to manufacture porcelain paste for dentures (Jones 1985; Craig 2006).

In 1808 an Italian dentist, Giuseppangelo Fonzi fabricated the first porcelain dental crown for use in complete dentures (Craig 2006). These early porcelains were opaque, and it was not until 1838, when an American dentist Dr Elias Wildman improved the formula, that translucency and “tooth colours” were introduced. This formula had increased amounts of feldspar and eliminated kaolin (Kelly et al 1996).
In 1849, the Dentist Supply Company made a significant advancement by improving translucency and colour through the introduction of vacuum firing (Kelly et al 1996) and later an American dentist Dr Charles H Land patented a technique of fusing porcelain to thin platinum foil pioneering the process for fabricating the porcelain - metal crown (Zeilgler 1905).

1.2.3.2 Dental Ceramics

The word “ceramics” originates from the Greek word “keramos” which means “burnt material” (Giordano 1996; Rosenblum and Schulman 1997). The use of the word “ceramic” in dentistry refers to inorganic crystalline materials that are fired at high temperatures in order to achieve desired properties (Craig 2006; Rosenblum and Schulman 1997). The dental term encompasses materials used for veneering, core structure, and one piece (monolithic) restorations (Naylor 2009).

Dental ceramics generally exhibit many desirable properties for dental restorations such as: biocompatibility; high compressive strength; aesthetics; low thermal conductivity and diffusivity; abrasion wear resistance; colour stability; and reduced plaque accumulation compared with other dental materials (McLean 1979; 1984). Dental ceramics do have limitations however, including: brittleness; low fracture toughness; and low tensile strength (Zheng and Zhou 2006; 2007; Anusavice 2003).

1.2.3.3 Structures of Dental Ceramics

Dental ceramics or porcelain consist of a crystalline silicon-oxygen network such as quartz, feldspar and/or alumina, and an amorphous glass-forming matrix. These form a large three-dimensional network of silica tetrahedra which are connected by oxygen atoms (Figures 27,
The amount of glass and crystal varies depending on the porcelain formulation (McLean 1979).

Figure 27. Diagram of a silica unit with each SiO terahedra sharing an oxygen atom. (from McLean 1979).

Figure 28. Three dimensional drawing of a silica unit in which the silicon atom Si is surrounded by four oxygen atoms (from McLean 1979).
When considering improving properties of dental ceramics such as low-fusing temperature, high viscosity, and resistance to detriviation, oxides of potassium, sodium, aluminium, and boron can be added (McLean 1979). Metal oxides can also be added to provide the porcelain with various colours. In addition of starch and sugar may be added to the porcelain powder for easier manipulation (Van Noort 1994).

Following firing, there are two main “phases” that make up the ceramic. These are the “crystalline phase” and the “glass phase”. The relative amount of each of these phases varies between porcelain type. The proportion of leucite in the crystalline phase, and the heat treatment time are affected by the K$_2$O content (McLean 1979). Feldspar (potash feldspar), a potassium aluminium silicate (K$_2$O.Al$_2$O$_3$.6SiO$_2$), is the main ingredient of classic porcelains used for veneers and metal-ceramic crowns. At 1150°C (considered a “high” temperature) feldspar melts and fuses with quartz to form leucite (KAlSi$_2$O$_6$ or K$_2$O.Al$_2$O$_3$.4SiO$_2$) with a tetragonal structure (crystalline phase) and molten glass with an amorphous structure (glass phase) (Craig 2006). As much as 85% of total volume can be comprised of glass matrix. The volume of leucite may range from 10%-35% depending on formula. This process where a material melts to create a liquid and a different crystalline material is called “incongruent
melting” (Anusavice 2003; Denry 1996). The leucite crystals are formed by a process called “surface crystallisation” as the porcelain is heated to between 700°C and 1200°C, where crystals grow slowly along grain boundaries towards the centre of the grain (Anusavice 2003; Denry 1996).

Each phase of the ceramic material contributes to its individual mechanical and optical properties. The glass phase is responsible for properties including brittleness, translucency, and a non-directional fracture pattern. The feldspathic leucite crystalline phase is responsible for the thermal and mechanical behaviour of the porcelain which are dependent on the distribution and size of the crystals. Leucite crystal phase enhances porcelain strength, optical properties, fracture toughness, and hardness (Rosenblum and Schulman 1997; Craig 2006). The high thermal expansion of leucite improves control of the porcelain’s thermal expansion coefficient, depending on the amount present which generally ranges 10-20%. Matching the thermal contraction characteristics between metal crown copings and overlaying porcelain reduces internal stresses which are a common cause of porcelain cracking and delamination which is a common cause of failure of metal ceramic restorations (Anusavice 2003). Leucite has limitations as it is a relatively unstable phase and thus, repeated firing cycles, slow cooling, and extended heat soaks can affect the leucite content and the overall mechanical properties of porcelain (Kelly et al 1996).

The amount of potash present, the firing temperature, and the length of time porcelain is held at high temperatures affect the extent of leucite crystal formation (Kelly and Benetti 2011). The leucite content can be increased or decreased depending on the number of firings and slow cooling. “Secondary crystallisation” is the process whereby the leucite crystal quantity increases with subsequent firings, thus increasing the coefficient of thermal expansion with each firing cycle. Firing at temperatures that are too high can cause the leucite crystals to
dissolve into the glass matrix which reduces the crystalline phase volume, thus lowering the coefficient of thermal expansion and weakening the ceramic (Anusavice 2003).

Quartz (SiO₂) or “silica” possesses a high fusion temperature and acts as a framework around which the other porcelain ingredients can flow. Quartz stabilises the porcelain at high temperatures and, along with potash feldspar, prevents the porcelain from undergoing pyroplastic flow on the metal substructure during the sintering process and results in a stronger fired porcelain (Muia 1982). The hardest and strongest component of porcelain is alumina (Al₂O₃). The water molecules naturally attached to alumina are removed resulting in alpha alumina, which is ground into a fine powder (McLean 1979). The addition of alumina increases the viscosity of the melt and the overall strength of the porcelain.

Dental porcelain is considered chemically stable and thus does not deteriorate over time. Porcelain also has a similar thermal conductivity and coefficient of thermal expansion to tooth enamel and dentine, as well as high compressive strength and low tensile strength (Van Noort 1994). Craig (2006) outlined selected physical properties of dental porcelain and enamel (Table 2).

Table 2. Selected physical properties of dental porcelain and enamel. (from Craig 2006).

<table>
<thead>
<tr>
<th></th>
<th>Feldspathic Porcelain</th>
<th>Enamel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength (MPa)</td>
<td>25</td>
<td>10</td>
</tr>
<tr>
<td>Compressive Strength (MPa)</td>
<td>149</td>
<td>384</td>
</tr>
<tr>
<td>Elastic Modulus (GPa)</td>
<td>69-70</td>
<td>84</td>
</tr>
<tr>
<td>Fracture Toughness (KIC)</td>
<td>0.9-1.0</td>
<td>0.7-1.3</td>
</tr>
</tbody>
</table>
Although very strong, dental porcelain has two concerns. Firstly, it is brittle and can fail catastrophically, and secondly, it has been shown to adversely wear opposing teeth which is a general concern in restorative dentistry (Giordano 1996; Rosenblum and Schulman 1997). Kelly (2011) formulated an overview of dental ceramics based on the physical properties of fracture toughness and bending strength (Figure 30).

![Classification of dental ceramics based on the physical properties of fracture toughness and bending strength](from Kelly and Benetti 2011)

**1.2.3.4 Classification of dental ceramics**

Many methods have been proposed in order to classify dental ceramics including: fusion temperature; fabrication method; crystalline composition and phase; and clinical application.
1.2.3.4.1 Fusion temperature

Fusion temperature outlines the temperature range at which the ceramic particles fuse together, as the melting process does not occur at a specific temperature. This has been used to classify ceramics since the 1940’s and has been described by Craig (2006) in four main groups:

- High-fusing: 1315°C - 1370°C
- Medium-fusing: 1090°C - 1260°C
- Low-fusing: 870°C - 1065°C
- Ultra low-fusing: <870°C

1.2.3.4.2 Clinical Application

Craig (2006) outlined three major applications for ceramics in dentistry:

- Veneering metal substructures
- Indirect all-ceramic restorations including crowns, bridges, inlays, onlays and veneers
- Ceramic denture teeth

1.2.3.4.3 Crystalline composition

Ceramic classification based on composition or microstructure results in four main groups (McLaren 2009):

- Glass based systems (mainly silica)
- Glass based systems (mainly silica) with fillers (crystalline such as leucite or more recently lithium disilicate)
- Crystalline-based systems with glass fillers (mainly alumina)
- Polycrystalline solids (alumina and zirconia)
As an alternative, Kelly and Benetti (2011) proposed three main categories including: predominantly glass; particle filled glass (with a subgroup called “glass ceramics”); and fully polycrystalline (Figure 31).

![Figure 31](image)

**Figure 31.** Schematic representation of three basic classes of dental ceramics. (a) Predominantly glass-based ceramics, the weakest of all groups, are lightly filled with opacifiers and colorants to mimic natural tooth aesthetics. (b) Glass containing 35-70% filler particles to improve strength are moderately aesthetic as full contour restorations. (c) Completely polycrystalline (no glass) ceramics. These can be used for frameworks or full contour restorations (from Kelly and Benetti 2011).

### 1.2.3.4.3.1 Glass-based systems

Predominantly glass-based ceramics are an amorphous three-dimensional network of atoms with an irregular pattern of spacing and arrangement. These are derived primarily from feldspar and are based on silica (silicon oxide) and alumina (alumina oxide). They are known as “feldspathic porcelains” (glass ceramics) and are a member of the aluminosilicate family of
materials. This group of ceramics most closely resemble the optical properties of natural enamel and dentine (Kelly and Benetti 2011; McLaren 2009).

Some of the advantages of glass-based ceramics include resistance to devitrification (crystallisation) during firing and an extended temperature firing range, which resists slumping when temperatures are higher than optimal. They are also biocompatible.

These materials have multiple uses including veneering core structures (including metal, zirconia, or alumina), use as monolithic veneer materials, and in a CAD/CAM block form (e.g. Vita mark 2™ for Cerec™) (McLaren 2009).

These ceramics are composed primarily of: silica (SiO$_2$) 64%; alumina (Al$_2$O$_3$) 18%; and potash (K$_2$O) and soda (Na$_2$O) in various amounts (Giordano 1996).

1.2.3.4.3.2 Glass-Based Systems with fillers

These ceramics have a large range of crystal types and glass-crystalline ratios and are commonly subdivided into three categories. Although the glass composition is essentially the same as the predominantly glass category, the crystal phase consists of different types and/or amounts which have been added or grown in the glass matrix (McLaren 2009). The primary crystal types used are leucite, lithium disilicate, or fluorapatite (McLaren 2009).

Leucite crystals are created by increasing the potash (K$_2$O) content of the alumino-silicate glass matrix, which also lowers the ceramic melting temperature.

1.2.3.4.3.2.1 Low-to-moderate leucite containing (<50%) feldspathic glass

This sub-category has become commonly known as “feldspathic porcelains”. Leucite is added to raise the coefficient of thermal expansion (CTE) of the material to allow addition to metals
and polycrystalline substructures (the amount of leucite is dependent on the material being veneered). This group of materials can also be used for veneers. They are usually in a liquid and powder form and have a random size and distribution of leucite crystals with a high average size of 20µm (McLaren 2009; Kelly and Benetti 2011).

The large particle size and random distribution results in relatively low fracture resistance and high abrasivity towards natural tooth enamel. The later generations of this material (such as Vita VM13™) have a finer leucite crystal and consistent particle distribution throughout the glass matrix which results in improved flexural strengths and reduced abrasivity (McLaren 2009).

1.2.3.4.3.2.2 High-leucite containing (50%) glass

This sub-categories glass matrix is also based on alumino-silicate glass and is known as a “glass ceramic” which has had the crystalline phase grown within the glassy matrix through a “controlled crystallisation of glass” process. This material is available in three forms; powder-liquid, machinable, and pressable. The most widely used commercial product in this group is IPS Empress™ (Mclaren 2009; Kelly and Benetti 2011).

The fracture resistance of this group is based primarily on the material fabrication processing technique than the crystal type, amount, and distribution of the crystals within the glass matrix. The pressable and machinable forms have much higher fracture resistance than the powder-liquid variant (Mclaren 2009; Kelly and Benetti 2011).

1.2.3.4.3.2.3 Lithium Disilicate containing (70%) glass

The commercial version of this material is known as eMax™ (formerly IPS Empress II™). The alumino-silicate glass has lithium oxide added and the crystals that form are needle-like
in shape and make up approximately 70% of the volume of the glass ceramic. This needle-like shape and the high volume of crystals contribute to the higher flexural strength and fracture resistance compared with the other subgroups (McLaren 2009; Kelly and Benetti 2011). The crystals measure 3-6µm in length.

This material comes in two forms, pressable and machinable. It also has the desirable property of translucency which means it can be used for monolithic restorations or can be veneered with an alumino-silicate glass porcelain which has fluorapatite added which contributes to the veneering porcelains coefficient of thermal expansion (CTE) and optical properties that are matched to the underlying eMax™ material. The standard chemical composition of both forms are seen in Table 3. The physical properties of the two materials can be seen in Table 4.

Table 3. Standard chemical composition of IPS eMax CAD and Press materials.

<table>
<thead>
<tr>
<th>IPS e.max Press</th>
<th>Ingots for the ceramic press technique</th>
<th>(in wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard composition:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>57.0 – 80.0</td>
<td></td>
</tr>
<tr>
<td>Li₂O</td>
<td>11.0 – 19.0</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>0.0 – 13.0</td>
<td></td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.0 – 11.0</td>
<td></td>
</tr>
<tr>
<td>ZrO₂</td>
<td>0.0 – 8.0</td>
<td></td>
</tr>
<tr>
<td>ZnO</td>
<td>0.0 – 8.0</td>
<td></td>
</tr>
<tr>
<td>+ other oxides</td>
<td>0.0 – 10.0</td>
<td></td>
</tr>
<tr>
<td>+ coloring oxides</td>
<td>0.0 – 8.0</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>IPS e.max CAD</th>
<th>Ceramic blocks for the CAD/CAM technique</th>
<th>(in wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard Composition:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>57.0 – 80.0</td>
<td></td>
</tr>
<tr>
<td>Li₂O</td>
<td>11.0 – 19.0</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>0.0 – 13.0</td>
<td></td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.0 – 11.0</td>
<td></td>
</tr>
<tr>
<td>ZrO₂</td>
<td>0.0 – 8.0</td>
<td></td>
</tr>
<tr>
<td>ZnO</td>
<td>0.0 – 8.0</td>
<td></td>
</tr>
<tr>
<td>Other and colouring oxides</td>
<td>0.0 – 12.0</td>
<td></td>
</tr>
</tbody>
</table>

(Source: R and D Ivoclar Vivadent AG, Schaan, Liechtenstein)
Table 4. Physical properties of IPS eMax Press and CAD

<table>
<thead>
<tr>
<th>Physical Properties of IPS eMax™</th>
<th>Press</th>
<th>CAD</th>
</tr>
</thead>
<tbody>
<tr>
<td>CTE (100-400°C [10⁶/K])</td>
<td>10.2</td>
<td>10.2</td>
</tr>
<tr>
<td>CTE (100-500°C [10⁶/K])</td>
<td>10.5</td>
<td>10.5</td>
</tr>
<tr>
<td>Flexural strength (biaxial) [MPa]</td>
<td>400</td>
<td>360</td>
</tr>
<tr>
<td>Fracture toughness [MPa M¹/²]</td>
<td>2.75</td>
<td>2.25</td>
</tr>
<tr>
<td>Modulus of elasticity [GPa]</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Vickers hardness [MPa]</td>
<td>5800</td>
<td>5800</td>
</tr>
<tr>
<td>Chemical resistance [µg/cm³]</td>
<td>40</td>
<td>40</td>
</tr>
<tr>
<td>Press temperature EP 600 [°C]</td>
<td>920</td>
<td></td>
</tr>
</tbody>
</table>

(Source: R and D Ivoclar Vivadent AG, Schaan, Liechtenstein)

1.2.3.4.3.3 Crystalline-based systems with glass fillers

The first material in this category was introduced in 1988 as glass-infiltrated, partially sintered alumina (In-Ceram™). This material has a sintered crystalline matrix that is 85% of the volume with a junction of the particles in the crystalline phase. This contrasts with the previously discussed ceramics which have a glass matrix and no junctions between crystals. The crystalline phase may consist of alumina, alumina-zirconia, or an alumina-magnesia mixture known as “spinell” and can be manufactured by slip casting or milling from a pre-sintered block. The alumina or spinell framework is then infiltrated at high temperature with a low-viscosity lanthanum glass (McLaren 2009; Kelly and Benetti 2011).

This material has a very high flexural strength (up to four times that of the previously mentioned materials), which is due to the mainly crystal nature of the material. It is important to note that the spinell version (alumina-magnesia) has half the strength of the alumina-zirconia version (McLaren 2009; Kelly and Benetti 2011).
1.2.3.4.3.4 Polycrystalline solids

Polycrystalline solid-sintered monophase ceramics are formed by directly sintering crystals together without an intervening matrix to form a dense, glass-free, air-free polycrystalline structure. The frameworks may be either solid-sintered aluminous-oxide or zirconia-oxide (McLaren 2009). There were two significant developments in the 1980’s which allowed the use of these materials in dentistry; the availability of highly controlled starting powders; and the use of computers in ceramic processing (Ritter 2010).

There are three techniques for fabricating solid-sintered, mono-phase, ceramic frameworks for porcelain application. These are:

1. DCS Precident™. This process machines the final desired framework shape from a solid sintered block of material.

2. Procera™. This uses a virtual oversized die where a slurry of either aluminous oxide or zirconia-oxide is applied and subsequently fired. It fully sinters and shrinks to fit the die.

3. Zirconia. This process machines an oversized coping from a partially sintered block of zirconia-oxide material, which is subsequently fired to full sintering temperature. The material shrinks to fit the die.

The zirconia form has improved physical properties (see Table 5) compared with the alumina variant which results in a material that is twice as tough and strong. Its flexural strength range is 900-1,100MPa, although there is no correlation between flexural strength and clinical performance of materials even though some authors suggest this is an advantage (McLaren 2009; Anusavice 2003; Kelly and Benetti 2011). Zirconia ceramic also has a fracture toughness, which is a measure of a material’s ability to resist crack growth, of 8-10MPa
which is significantly higher than other dental ceramics and has been reported as a benefit for this material.

Table 5. Tetragonal Zirconia Polycrystals 3Y-TZP: Mechanical and Physical Properties.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equiaxed grains μm D</td>
<td>0.2-0.5</td>
</tr>
<tr>
<td>Flexural strength MPa</td>
<td>800-1200</td>
</tr>
<tr>
<td>Fracture toughness MPa m$^{1/2}$</td>
<td>6.1-8.5</td>
</tr>
<tr>
<td>Weibull modulus</td>
<td>10-18</td>
</tr>
<tr>
<td>Porosity %</td>
<td>8.1-11.5</td>
</tr>
<tr>
<td>Threshold Intensity factor MPa m$^{1/2}$</td>
<td>3.1</td>
</tr>
<tr>
<td>Critical crack Intensity MPa m$^{1/2}$</td>
<td>7.4</td>
</tr>
<tr>
<td>Thermal Conductivity Wm$^{-1}$K$^{-1}$</td>
<td>2</td>
</tr>
</tbody>
</table>

One of the reported concerns with zirconia framework restorations that are veneered has been a relatively high incidence of fracture of the veneering porcelain (Al-Amleh et al 2010). These limitations have led to the introduction of several monolithic or “full contour” zirconia dental restoration materials along with the development of CAD/CAM systems for the manufacture of these restorations. There is little clinical data on the performance of these monolithic zirconia restorations, however anecdotal evidence suggests its use in restorative dentistry is growing.
1.3 In-Vitro Dental Material Tribology

1.3.1 General Considerations in in-vitro acrylic denture tooth testing

There is significant complexity in the process of opposing dental material wear as there are many factors which are difficult to control in the laboratory. Although, in-vitro wear testing does provide a valuable research tool as a large number of tests can be run safely and thus a large amount of relevant information and data can be obtained within a relatively short period of time. This type of research design can also assess individual factors which may be related to material wear.

In-vitro testing does however have some short comings for instance when trying to compare data or findings from different studies or when trying to extrapolate in vitro study data to the clinical situation (Ghazal et al 2008). This shortcoming is a concern as there is significant variability in the study instrumentation, materials and protocols, and each of these parameters can influence wear mechanisms (Jahanmir and Fischer 1994; Lawn et al 1984; Fisher et al 1983). Examples relevant here include: the wide range of wear machines; the contact geometry; the loading protocols; chemistry; and flow of lubricants in the in-vitro wear studies (Seghi et al 1991; DeLong et al 1986; Hacker et al 1996; Krejci et al 1994; Mahalick et al 1971; Monasky and Taylor 1971; White 1993). Other influencing factors may include: contact pressure; material microstructures and physical properties; friction; third body characteristics; and chemicals or medium, all of which will influence the mechanism(s) of wear.
1.3.2 Material Wear Quantification

Tooth and material wear quantification has been historically undertaken via a number of methods. In 2001, ISO standards relating to two or three body wear were updated regarding recommendations and eight different wear assessment methods are discussed. (ISO 2001 Guidance on testing of wear) If different instruments are used, inaccuracies can result which reduces the validity of the data when comparing different studies and when determining dental material rankings.

Early studies of tooth wear investigated individual pre- and post-wear sample weight. However, over time, methods with improved accuracy were introduced to overcome errors associated with water uptake by tooth structure or dental materials during the testing process. The more accurate methods involve microscopic specimen contour/volume assessments, and recently, scanning specimens and the use of individualized software to quantify the material sample volumes. The literature generally includes assessment methods such as sample height loss (Ghazal et al 2008, Al-Hiyastat et al 1998; Elmaria et al 2006: Esquivel-Upshaw et al 2006: Etman et al 2008; Fisher et al 1983; Kadokawa et al 1906; O’Kray and O’Brian 2005; Ramp et al 1999; Ramp et al 1997), volume loss (Ghazal et al 2008; Alarcon et al 2009 ; Metzler et al 1999; Palmer et al 1991), weight loss (Monasky and Taylor 1971), and wear surface area (Clelland et al 2003). Height and volume measurements have been performed with a stereomicroscope with a micrometer-calibrated movable platform (Palmer et al 1991) or a reflex microscope (Al-Hiyasat et al 1998), although the most common and effective technique for measuring height and volume loss is scanning profilometry (Ghazal et al 2008; Elmaria et al 2006; Esquivel-Upshaw et al 2006; Etman et al 2008; Kadokawa et al 2006;
1.3.3 Standardising wear simulation research protocols

The lack of standardization of in vitro material wear protocols has been cited as being a limitation to the results attained (Ghazal et al 2008). It is generally concluded that the standardisation of protocols would significantly improve the quality of dental wear studies by facilitating comparisons and improving extrapolation to the clinical situation. Despite efforts in the dental community, the International Standards Organization (ISO) Technical Committee 106 (dentistry) has been unable to reach a consensus on the standardisation of dental related material wear testing protocols (Condon and Ferracane 1996). The committee’s statement outlined that despite the many wear tests being used and proposed for dental materials, there is no one generally accepted method and none of the in vitro wear simulators used have been developed based on direct intra-oral observations of mechanisms of damage accumulation (Condon and Ferracane 1996). The committee’s report also concluded that due to the extensive variability in research methodologies, it is not surprising that differences in results and outcomes occur even when studies are examining the same materials (Krejci et al 1994; Grossman 1991; DeLong et al 1992).

1.3.4 Dental Sample Material Test Parameters

In vitro wear simulation and testing between dental materials and natural dental hard tissues commonly use prepared enamel specimens from extracted molars or premolars where the tooth structure acts as the antagonist and is worn against a flat material sample. Other research
and testing parameters lack any standardisation and thus no thorough analysis including meta-
analysis has been performed on specific categories of natural and/or artificial dental materials.
There are several test parameters which vary significantly in the literature. These include: the
type of tooth contact or chewing simulator; the applied force; the cycle number and
frequency; the form of lubricating medium if any; the pH of the lubricant; the inclusion of
exogenous abrasive materials; the geometry of samples (Czichos 1986; Denry 1996; DeLong

1.3.4.1 Tooth contact (chewing) simulators

Many different chewing or wear simulators are described in literature. These include: pin to
disc simulators (Alarcon et al 2009; Clelland et al 2003; Kadokawa et al 2006); Willytec
(Ghazal et al 2008); University of Alabama (Kadokawa 2006); Oregon health sciences
university (Clelland et al 2003; Clelland et al 2001); Leinfelder (Ramp et al 1999; Alarcon et
al 2009); the closed loop servo hydraulics (Magne et al 1999), as well as various custom wear

1.3.4.2 Loads applied to specimens

Past wear studies have published a wide variety of applied loads during testing. The units for
measuring load also vary depending on the origin of the study and include: grams; kilograms;
pounds; or Newton (Elmaria et al 2006; Ranjitkar et al 2008; Shabanian and Richards 2002).
The range of loads is large with 0.75N being the lowest reported, and 80N being the highest
(Ramp et al 1999; Ramp et al 1997; Kadokawa et al 2006; O’Kray and O'Brian 2005; Metzler
Alarcon et al 2009). Loads less than 32N have been reported as being insignificant as very low wear rates have been reported, and loads greater than 160N have also been excluded as they often result in catastrophic failure of the test material. Numerous authors have reported that a load of approximately 50N is the mean value for physiologic biting force in non-bruxing patients (Gibbs et al 1981) It has also been well demonstrated that by increasing the load, the resultant material wear increases (Lutz et al 1992).

1.3.4.3 The number of cycles

The number of cycles or study duration of in vitro experiments must be planned to result in sufficient wear or material loss to allow consistent and reliable measurements and comparison within the study and between consecutive studies using the same parameters. Wear testing between various natural and artificial dental materials have been reported to run from 5,000 to 1,200,00 cycles depending on the overall study protocol and material as well as study aims (Fisher et al 1983; Elmaria et al 2006; O’Kray and O’Brian 2005; Ramp et al 1999; Ramp et al 1997; Palmer et al 1991; Al-Hiyastat et al 1997; Al-Hiyastat et al 1999; Taylor et al 1989; Hacker et al 1996; Monasky et al 1971; Kadokawa et al 2006; Ghazal et al 2008; Metzler et al 1999; Alarcon et al 2009; Magne et al 1999, Albashaireh et al 2010; Stawarczyk et al 2012; Preis et al 2011; Ghazal et al 2009; Beuer et al 2012; Heintze et al 2006; Albrecht et al 2011; Ghazal et al 2008).

It has been uniformly cited that the amount of material wear increases with the increasing number of loaded cycles (Fisher et al 1983; Elmaria et al 2006; O’Kray and O’Brian 2005; Ramp et al 1997 and 1999; Palmer et al 1991; Al-Hiyastat et al 1997 and 1999; Taylor et al 1989; Hacker et al 1996; Monasky et al 1971; Kadokawa et al 2006; Ghazal et al 2008;
Metzler et al 1999; Alarcon et al 2009; Magne et al 1999). This increasing wear generally follows a linear pattern but variation has been described (Hacker et al 1996; Richards 1990).

1.3.4.4 Lubrication and pH variations

Distilled water is the most commonly reported lubricating medium in dental material wear studies (Ghazal et al 2008; Al-Hiysat et al 1998; Fisher et al 1983; Ramp et al 1999; Ramp et al 1997; Metzler et al 1999; Palmer et al 1991; Magne et al 1999). Artificial saliva has been used as has tap water and even natural saliva (O’Kray and O’Brian 2005; Elmaria et al 2006; Kadokawa et al 2006; Monasky et al 1971; Hacker et al 1996; Kaidonis et al 1983). All approaches have both theoretical and actual advantages and disadvantages. Distilled and tap water offers a low cost and easy to use medium which has low corrosion qualities, however they don’t perfectly mimic natural saliva. Artificial saliva also doesn’t mimic natural saliva and also adds the variable of potential remineralisation (when testing enamel or dentine) and thus influences results. Natural saliva denatures within a few minutes potentially influencing results and making analysis and extrapolation difficult.

1.3.4.5 Material Specimen Geometry

In vitro studies mostly analyse flat specimens which as the specimens are easy to fabricate and can be standardised. It is noteworthy that the flat specimens have been associated with more wear of both the specimens and the antagonist material when compared to crown specimens. This is believed to be the result of a greater surface contact area (Heintze et al 2008).
Studies have varied the form of the antagonist material. Various forms include various cusp forms (Stawarczyk et al 2012) or balls (Ghazal et al 2009).

1.3.4.6 The outcome of in-vitro test parameter variation

Significant efforts have been made to standardise test parameters, methodologies, materials and sample preparations. However, the variation and lack of consistency between studies have made this impossible to date. The result is a lack of agreement on the ranking of specific materials for tooth and dental material wear. Regarding the surface finish of ceramics, there has been limited consistency between studies, however, more recently studies have agreed that polishing or glazing are both effective methods in reducing surface roughness and thus optimising the surface texture of the material (Heintze et al 2008, DeLong et al 1992; Jagger et al 1995; de Silva et al 2006; Sarikaya and Guler 2010).

1.3.4.7 Factors influencing material wear when opposed by ceramic

Dental ceramics are generally considered resistant to wear and are abrasive to opposing materials (McLaren 2009). Historically, the importance of polishing and reducing the surface texture of ceramic occlusal surfaces was not appreciated and the impact on opposing dentitions was noteworthy. Heintze et al (2008) reported that the severity of tooth surface wear is influenced by dental materials as well as patient factors (Heintze et al 2008). Ceramic material factors which can influence the rate of opposing material wear include physical, micro-structural, chemical, and surface characteristics (Etman et al 2008; Esquivel-Upshaw et al 2006). It has been reported that harder materials with higher surface roughnesses result in
higher opposing material wear. Two factors that contribute to this are crystal phase concentrations and crystal sizes, with those that are smaller being less abrasive (Derand and Vereby 1999). Patient factors that have been implicated in tooth wear include acidic or erosive diets, dysfunctional occlusion, increased biting force, and parafunctional habits (Heintze et al 2008).

Although investigators have proposed several physical factors as being predictors of wear, no specific physical factors of ceramic materials have been universally identified (Lawn et al 1984; Heintze et al 1998; Jahanmir et al 1994; Fischer et al 1989). In the 1980’s, dental ceramic surface hardness was thought to determine enamel wear (Lawn 1984). However research during the 1990’s demonstrated that surface hardness alone is not a reliable predictor of opposing dentition wear (Heintze et al 1998; Jahanmir et al 1994; Fischer et al 1989). More recent studies have indicated that surface texture, especially surface roughness, has the most influence on opposing material wear when compared to other dental ceramic physical properties (Elmaria et al 2006; Monasky and Taylor 1971; Seghi et al 1991). It must be noted that any reported “influencing” factors are derived from in vitro wear studies which have shortcomings and may not have a direct clinical correlation (Etman et al 2008; Esquivel-Upshaw et al 2006).

1.3.4.8 Dental ceramic physical micro structural properties

Physical properties of dental ceramics influence the polishability of the surface and also the possible wear of the opposing material, whether it be an artificial or natural dental material (Martinez-Gomis et al 2003). The exact influence of material physical properties including hardness, fracture toughness, frictional resistance, leucite content, porosities, crystal
characteristics, creaming, and surface texture still face disagreement (Al-Wahadni and Martin 1999).

1.3.4.8.1 Hardness

The term hardness describes the resistance a material has to plastic deformation. It was previously assumed that higher hardness values were associated with an increased rate of opposing material wear. Recent studies, however, have shown that there is in fact no strong correlation between hardness value and opposing material wear (Monasky and Taylor 1999). The wear process appears to be more closely related to a materials microstructure, the surface roughness of both contacting materials, and environmental factors including lubrication and pH (Oh et al 2002; Magne et al 1999). Several reasons have been given in literature for the weak correlation between material hardness and its ability to wear an opposing material. One is that when materials such as dental ceramic and enamel wear against each other, the wear process is not via plastic deformation as with metals, but is in fact via a process of fracturing (Heintze et al 2008, Elmaria et al 2006). Another reason is that the micro-hardness of crystalline ceramics varies depending on the orientation of the indenter to the crystals. Finally, it may be due to the variability in the hardness between the glass matrix and the crystals (De Long et al 1986). Thus, it is not surprising that relatively soft ceramics exhibit more abrasive action against human enamel than harder ceramics, and the same may be said for any opposing materials undergoing a wear process (De Long et al 1986).
1.3.4.8.2 Frictional Resistance

The term frictional resistance describes the resistance to lateral sliding of two materials in contact with each other (Al-Wahadni and Martin 1999). This depends on a number of factors including the relative motion, the properties and structure of the materials, and the environment. An example is the friction between porcelain-to-porcelain contact points has been reported at almost twice that of porcelain-to-acrylic contact points and approximately three times that of enamel-to-enamel contacts (Zheng and Zhou 2006). Zheng stated that roughness, high loads, and high sliding speeds have all been shown to increase the coefficient of friction and thus materials wear (Zheng and Zhou 2006). All known finishing and polishing methods still leave microscopic irregularities and it is the interaction between these irregularities on opposing materials that determines the friction between them (Heintze et al 2008, Elmaria et al 2006). Aqueous environments such as saliva or lubricants are known to reduce the surface hardness of dental ceramic materials which results in increased friction due to adhesion at a microscopic level. Therefore, the outcomes of in vitro wear studies can be influenced by the lubrication medium and may cause issues with wear result extrapolation (Zheng and Zhou 2007).

1.3.4.8.3 Fracture Toughness

Fracture toughness refers to the resistance a material has to rapid crack propagation under tensile stress (Oh et al 2002). This is an intrinsic property that depends on two factors. Firstly, the stress intensity at a crack tip in the material, and the second factor is the crack size relative to the micro structural features of the material in question including crystal size, aspect ratio, porosity, and orientation and distribution of the glass phase. The fracture of ceramic materials
starts when the applied load produces a stress at the tip of a crack or material flaw. This type of wear can start with the formation of a crack followed by slow crack propagation over time, and then eventually catastrophic failure and fracture of the material. As plastic deformation doesn’t apply, the influence of fracture toughness on ceramic material wear is significant (Oh et al 2002, Zheng and Zhou 2007).

A materials surface roughness may increase as a result of brittle fracture if the material has insufficient toughness to resist fracture. The brittle chips that are formed during the abrasion process can further contribute to the wear process. Brittle fracture of ceramic materials can be more pronounced when they are positioned adjacent to processing defects including porosity and impurity inclusions, and they can also contribute to crack propagation under loading (Zheng and Zhou 2007).

Heintze et al (2008) stated that as enamel has a lower hardness and fracture resistance when compared to leucite and lithium dislicate dental ceramics, the enamel undergoes more antagonistic wear when opposed by these two ceramics. The roughness of the ceramic then further contributes to the wear process. Enamel also has a lower fracture toughness compared with these ceramics, which further enhances the wear of the enamel (Heintze et al 2008).

1.3.4.8.4 Porosity

Sintered porcelains are produced by powder and liquid being mixed together, which is then condensed and sintered. This process can allow the accumulation of porosities within the porcelain structure that are difficult or in some cases impossible to remove, and may contribute to inferior material characteristics such as reduced strength and aesthetics. If during the wear process porosity is exposed, the sharp edge of the defect present an area of stress concentration which can increase the wear of the opposing material and add to deterioration
of the ceramic surface (Zheng and Zhou 2007). Porosity or air entrapment can however be minimised in the manufacture process by casting or pressing ceramic ingots as supplied by manufacturers or by utilising industrially manufactured ceramic CAD blocks (Oh et al 2002).

1.3.4.8.5 Crystals

Dental ceramics or porcelain is composed of silica glass that is modified by the addition of fluxes including CaO, K₂O, Na₂O, B₂O₃, and/or Al₂O₃. With a specific composition of oxides, leucite crystals are formed to improve optical and mechanical properties of the porcelain, as well as control the coefficient of thermal expansion. Dental ceramics indicated for ceramo-metal restorations contain approximately 15-25%vol leucite as their main crystalline phase, whereas leucite based all-ceramic restorations contain up to 90%vol leucite as the main crystal phase. Leucite crystals consist mainly of alumina, magnesia, zirconia or lithia within a glassy matrix (Ritter 2010). These crystals are 5-7μm in diameter and are responsible for wear of the opposing dentition or material due to their high hardness.

Factors related to the crystals that influence the potential abrasiveness of a dental ceramic include the type, content, morphology, and distribution of the crystalline particles (oh et al 2002; De Long et al 1989). Some studies however have shown that crystals within a glassy matrix may not necessarily adversely impact the wear of opposing dental enamel (Oh et al 2002). Contention exists in the literature about the exact influence of the ceramics of different compositions on the wear of opposing materials and thus standardisation of in-vitro testing is required (Oh et al 2002; De Long et al 1989).
1.3.4.8.6 Ceram Layer

“Ceramming” is the process of controlled crystallisation of the glass resulting in the formation of crystals which are evenly distributed throughout the glass structure. The crystal size, amount, and rate of growth are determined by the time and temperature of the ceramming heat treatment. Ceramming results in crystals which are oriented perpendicular to the external surface of a glass ceramic and the outermost layer of the ceramic is called the “ceram” layer. The ceram layer has been reported as being up to twice as abrasive to the opposing dentition and four times as wear resistant than a polished ceramic (De Long et al 1989).

1.3.4.8.7 Ceramic chemical degradation due to acid attack

Oh et al (2002) reported that the wear rate of dental enamel opposed by ceramic is significantly higher in a highly acidic environment versus neutral situations. There were two reasons cited for this. The first being that the acidic environment affects the solubility of tooth structure (at pH 5.5 vs pH 6.5) by up to eight times for enamel. The second is that forms of chemical degradation including glass corrosion, etching, and crystal deposition increase the degree of surface roughness and reduce the ceramics wear resistance by continually exposing a rough surface (Oh et al 2002). Occlusal forces accelerate this process further (De Long et al 1986).

1.3.4.8.8 Effect of surface finish
An uneven distribution of dental ceramic crystals within the glassy matrix can result in flaws in the ceramics internal structure. Dental laboratory processing can further enhance these defects which can result in a reduction in material strength and thus increased wear rate on opposing materials (Metzler et al 1999). The processes of polishing and glazing can reduce or seal respectively the ceramic surface which reduces the abrasiveness of the ceramic. This may only influence the early stages of wear though (Monasky and Taylor 1971).

The surface texture of dental porcelain should be as smooth as possible to enhance the function, aesthetics, and biologic compatibility of a ceramic restoration (Cook et al 1984; Ma et al 1999). Rough surfaces have numerous undesirable outcomes including reduced flexural strength (Rosenblum and Shulman 1997), increased antagonistic abrasion of opposing materials (Al-Hiyasat et al 1997; Wright et al 2004; Kramer et al 2006), accumulation of stain and plaque (Tholt de Vasconcellos et al 2006; Clayton and Green 1970).

1.4 Literature Review of Acrylic Denture Teeth Wear Testing

A literature search was conducted via PubMed and Embase for published studies which investigated the wear of highly cross-linked acrylic denture teeth. The search resulted in ten in vitro two-body wear simulation studies. One group lead by Ghazal have published seven of the in-vitro studies. A summary of each two-body in vitro wear study including wear testing machine, number of cycles, load, lubricant, antagonist, and denture teeth test materials is outlined in Table form in Appendix 1.

The literature search revealed a number of studies which have looked at in vitro wear testing and surface texture analysis of artificial denture teeth. Various methods of testing were reported with significant variation in all aspects of the wear studies.
The artificial teeth testing included various types of acrylic (PMMA) based denture teeth such as PMMA, Highly cross linked PMMA, direct cross-linked (DCL)-PMMA, PMMA with interpenetrating polymer network (IPN), PMMA with fillers, urethane dimethacrylate (UDMA), as well as composite resin, and feldspathic ceramic denture teeth. Various studies indicated that the PMMA artificial teeth may suffer more wear than other materials depending on test parameters (Ghazal et al 2008; Hahnel et al 2009; Heintze et al 2012; Hirano et al 1998; Mello et al 2009; Reis et al 2008; Hao et al 2014; Stober et al 2006; Suwan 2001).

The antagonist materials tested against the artificial acrylic test materials included: natural tooth enamel; steatite; zirconia oxide; alumina; aluminium oxide; silicone nitride; feldspathic ceramic; stainless steel; Ni-Cr Alloy; composite resin; and, acrylic denture teeth (Ghazal et al 2008; Hahnel et al 2009; Heintze et al 2012; Hirano et al 1998; Mello et al 2009); Reis et al 2008; Hao et al 2014; Stober et al 2006; Suwanaroop et al 2001). The form that the antagonists took varied significantly from balls, to cusps, sandpaper, or natural tooth forms.

The wear machines that were used included Dual-axis chewing simulator (Willytec, Munich, Germany), Pin-on-disc (EGO, Regensburg, Germany, and Abrasive tester (TE88; Phoenix Tribology Ltd)), Customised wear testing machine (Tsurumi University), Customised wear testing machine developed by Wagner and Hacker (1996), a “simulated brushing machine”, Ducom Friction wear machine (custom), and Mello et al (2009) used another custom variant. There is clearly no consensus nor consistency within this literature regarding a standardised machine for wear testing. Heintze et al (2012) aimed to correlate laboratory testing protocols with clinical wear results for the same materials. Only the results of the experimental setup with standardised antagonists of the same denture tooth material against flat specimens were similar to the clinical wear results with a comparable relative difference in mean wear between the materials.
There was significant variation in the test parameters regarding lubricant, cycle number, and load applied to the material samples. The lubricants utilised included nothing, tap water, distilled water, and human saliva. Some studies used thermo-cycling of lubricants whereas others did not.

Regarding the number of cycles and rate of cycles, these ranged from 1,000 to 1,200,000 total cycles at varying rates from constant contact up to 265 cycles per minute. One study reported a rate of 1,000rpm. The nature of the cycles varied as well, with some samples being in constant contact with the antagonist with either linear or rotating actions, while others enacted the gliding phase of mastication with period of no contact then contact with a horizontal slide. The slide distances varied from 0.2mm to 20mm.

The force applied to the samples is also an important variable. Here, the forces varied from 0.2kg (2N) up to 50N.

Methods of sample and antagonist analysis included qualitative methods such as SEM which was also used for surface texture analysis. Methods for analysis of material loss due to antagonistic wear included height change measurements, volume assessment via weight, and 3-D surface profilometry via stylus and laser methods. DeLong (2006) reported that 3D analysis is the most accurate method for measuring wear.

Surface roughness analysis was undertaken by several studies (Ghazal et al 2008). All of these studies utilised two-dimensional analysis and reported in $Ra$ values. An understanding of surface roughness is important (as outlined by Ghazal et al (2008)) who studied zirconia antagonists of varying roughness and their effects on opposing samples. The investigators found a positive correlation between the roughness of the zirconia antagonist and the degree of wear of the opposing sample material.
Hahnel et al (2009) stated that the outcome of *in vitro* studies on the wear resistance of artificial resin teeth appears to be influenced markedly by the antagonist material used for mastication simulation. Thus, for the prevention of vertical height loss, clinicians might consider *in vitro* studies using ceramic antagonist materials for mastication simulation, which allow for the better differentiation between various artificial resin teeth with regard to wear resistance.

Only one author has investigated zirconia opposing denture tooth materials, however, only feldspathic ceramic and nano-filled composite resin denture teeth were assessed. No studies have been reported which have investigated zirconia, eMax™ or Lava Ultimate™ opposing PMMA denture teeth. Ghazal (2008) investigated composite resin denture teeth alongside human enamel and feldspathic ceramic denture teeth which opposed three antagonist materials including steatite (6mm balls), zirconia (inceram™), and alumina (inceram AL™). They found that composite resin denture teeth had more wear than human enamel and discussed the mechanism of composite resin material degradation and the influence it has on the wear process.

“In the abrasion mechanism of composite resin materials, the size, shape, volume and hardness of fillers, the bonding between fillers and polymer matrix, and the polymerisation dynamics, all have an effect on the wear characteristics. During the wear process of composite resin, inorganic filler particles are exposed after the softer resin is abraded, which causes a high friction coefficient and leads to high internal shear stresses in the polymer matrix. This corresponds to increased sensitivity to wear which might have accelerated the wear of composite resin teeth during long term testing.”

Ghazal et al (2008) also stated that the wear resistance of all PMMA based teeth is less than the composite resin teeth in their study and highly cross-linked PMMA and conventional
PMMA teeth showed the same overall wear. Feldspathic ceramic showed a similar wear rate to enamel.

1.5 Rationale for this research

Understanding the behaviour of dental materials subjected to “chewing-like” actions is essential to understanding how and when each material should be used. The introduction of new dental materials into the market place often comes with a lack of laboratory and/or clinical testing which may have implications for not only a patients’ existing restorations and teeth, but also any dental restorations which are undertaken in the future.

It has been well documented that different dental materials have varying effects on other opposing dental materials which may in turn result in a shorter life of restorations or prostheses. There has been numerous studies into the wear behaviour of various artificial denture teeth, however, no studies have investigated the behaviour of these teeth when opposing novel materials, namely, monolithic zirconia, lithium dislocate ceramic, and nano-ceramic composite resins for CAD/CAM use.

Multiple studies have identified the correlation between in vitro and in vivo attrition and abrasion wear testing of dental materials in a chewing simulator (Finger and Thiemann 1987; Heintze et al 2006). This evidence validates to some extent the use of in vitro wear testing in the prediction of in vivo performance of materials. However due to the dynamic nature, including multiple influencing factors in vivo, a clinical observation of material performance is paramount in making final decisions about material choice (Heintze et al 2006 and 2012).
Section 2

Study Aims and Hypotheses
2.1 Study Aims

1. Qualitative assessment of the dental material surfaces prior to wear testing

2. Investigate a novel three-dimensional surface roughness analysis method, $Sa$, for use in dental material analysis as opposed to conventional two-dimensional $Ra$ values.

3. Investigate the surface texture before and after wear testing of dental materials including:
   - Monolithic Zirconia
     - Lava Plus™ (non-HIP)
     - BruxZir™ (non-HIP)
     - DenZir™ (HIP)
   - Monolithic Lithium Disilicate Ceramic (eMax™)
   - Lava Ultimate™ Nano-Ceramic Composite Resin
   - Conventional Acrylic (PMMA) Artificial Denture Teeth

4. Investigate the *in vitro* two-body wear behavior of the sample dental materials opposing the conventional acrylic (PMMA) artificial denture tooth antagonist. Tested materials will include:
   - Monolithic Zirconia (one representative material)
     - Lava Plus™ (non-HIP)
   - Monolithic Lithium Disilicate Ceramic (eMax™)
o Lava Ultimate™ Nano-Ceramic Composite Resin

o Conventional Acrylic (PMMA) Artificial Denture Teeth

5. Measure the volume loss (wear) of the acrylic antagonist and the opposing sample dental materials using three-dimensional profilometry.

6. Analyse material sample wear characteristics using SEM in the post-testing state.

2.2 Null Hypotheses

1. The two-dimensional (Ra) values for materials and three-dimensional (Sa) values will provide comparable information about surface roughness.

2. There is no relationship between Ra and Sa values regarding dental material surface topography characterisation.

3. The dental sample materials will have similar Ra and Sa values.

4. Surface roughness of all tested materials will increase following wear simulation testing.

5. The monolithic zirconia material will cause more antagonistic wear of the opposing artificial acrylic denture tooth material (PMMA) compared to all other materials tested.
Section 3

Methods and Materials
3.1 Sample Preparation

3.1.1 Monolithic Zirconia

Lava Plus™ (3M ESPE) is a “translucent” all-zirconia (monolithic) based ceramic restoration material. It is a tetragonal polycrystalline zirconia (ZrO₂) partially stabilised with 3mol yttria and has a lower alumina dopent content (0.1%) compared to LAVA™ framework zirconia materials, as such it is a “non-HIP’d” zirconia material. The original Lava Zirconia material was brought to market in 2001. Lava Plus™ is a new generation material which is claimed to have superior physical properties and aesthetics. Lava Plus™ is indicated for various dental restorations including partial or complete crowns, bridges, and implant restorations.

These restorations are CAD/CAM-based and are designed using the Lava Design 7™ (3M ESPE) software and digital workflow system. The restorations are milled in the “pre-sintered” state using a LAVA CMC 500™ (3M ESPE) CAD/CAM milling machine with a series of diamond burs. Following characterisation, the restoration undergoes final sintering in a LAVA Furnace 200™ (3M ESPE) to achieve its final dimensions. Following this, the restorations can be finished by polishing, glazing, and/or staining with subsequent sintering as required.

In this study, the material samples were manufactured using the “monolithic” restoration work flow without glazing or staining. The material samples were in the post-sintered state prior to polishing in order to demonstrate the characteristics of the material in its potentially
roughest state. The samples were 10mm x 8mm and were 1.5mm thick as recommended by the manufacturer.

“BruxZir™” (Glidewell) is also a non-HIP’d all-zirconia (monolithic) based ceramic restoration material. It is a tetragonal polycrystalline zirconia (ZrO$_2$) partially stabilised with yttria. Restorations fabricated from this material are CAD/CAM-based and are compatible with a number of CAD software systems including CEREC™, E4D™, TRIOS™, and 3M ESPE®. The manufacture process is the same as that for the Lava Plus™ material in that partially sintered zirconia blanks are used in the milling process and are subsequently sintered to the desired restoration dimensions (Table 6).

Table 6. BruxZir manufacturer instructions for sintering.

<table>
<thead>
<tr>
<th>Ramp up cycle</th>
<th>Ramp down cycle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Start temp: 25°C</td>
<td>Start temp: 1,530°C</td>
</tr>
<tr>
<td>Heat rate: 10°C/min</td>
<td>Ramp time: 180mins</td>
</tr>
<tr>
<td>Ramp rate: 151 mins</td>
<td>Final temp: 70°C</td>
</tr>
<tr>
<td>Final temp: 1,530°C</td>
<td>Hold time: 120 mins</td>
</tr>
</tbody>
</table>

In this study, BruxZir™ samples were manufactured following the same basic process as was used to prepare the Lava Plus™ specimens except that the final samples were 10mm x 10mm and were 1.5mm thick due to the original block dimensions and as recommended by the manufacturer.

DenZir™ (DenZir, Stockholme, Sweden) is a ceramic material consisting of HIP (Hot Isostatically Pressed) Yttrium Oxide stabilised zirconium dioxide. These restorations have
similar indications to the two previously mentioned systems. This material is also designed for CAD/CAM fabrication techniques. DenZir™ utilises the “Cad.esthetics System™” (DenZir) or the “open system” which allows image capture and design from using any company’s system.

DenZir™ restorations are fabricated in a two-step process. First, the manufacturing of the DenZir blocks of material, and second, the “grinding” phase in which the dental restoration is fabricated. The block manufacturing phase involves the refinement of zirconia dioxide powder. This powder is pressed and shaped into the desired block dimensions and subsequently sintered under a proprietary process. Following this, in order to achieve improved physical properties regarding maximum density, strength and aging resistance, the blocks are hot isostatically pressed (HIP), and finally inspected as part of the quality control process.

Unlike the LAVA™ and BruxZir™ materials these restorations are milled, in the completely sintered state. The second “grinding” phase involves shaping of the HIP blocks into the desired dental restoration form. DenZir™ proprietary CAD/CAM software is used to input the desired restoration shape and dimensions and calculate the milling time. After the restoration is milled, it is inspected and vapour cleaned, and logged according to zirconia dioxide batch number.

In this study, the material samples were manufactured following the “monolithic” restoration work flow without glazing or staining. The material samples were in the post-sintered state prior to polishing in order to demonstrate the characteristics of the material in its potentially roughest state. The samples had a 10mm diameter and were 1.5mm thick as per manufacturer instructions.
3.1.2 Lithium Disilicate Specimen

Milled and pressable lithium disilicate is indicated for any restorative prosthesis that is less than three units in total, for example inlays, inlays, crowns, and bridges. The standard chemical composition of the IPS eMax CAD™ (Ivoclar Vivadent) which was used in this study is listed in Table 3 (page 47) and the physical characteristics of both the pressable version and CAD/CAM version are seen in Table 4 (page 47).

In this study, the IPS eMax™ samples were manufactured from CAD/CAM blocks. As with the zirconia specimens, these samples were in the post-sintered state prior to polishing in order to demonstrate the characteristics of the material in its potentially roughest state. These samples were 10mm x 6mm and were 2mm thick.

3.1.3 Resin Nano-Ceramic (RNC)

The LAVA Ultimate™ is delivered from manufacturer (3M ESPE) in CAD/CAM block form. The blocks were sectioned with a diamond cutting wheel to dimensions of 10mm x 10mm with a thickness of 3mm. The samples were subsequently wet polished with 2000 grit silicon abrasive paper to achieve the desired polished finish.

3.1.4 Artificial acrylic denture tooth material (PMMA)

In this study “Gnathostar™ (Ivoclar Vivadent) artificial denture teeth were sectioned to provide PMMA samples. Upper first molars were chosen, the cusps were removed to produce a flat surface and the testing surface was wet polished with 2000 grit silicone abrasive paper
to achieve the desired polished finish. The samples were approximately 10mm x 8mm and had a thickness of 4mm.

3.1.5 Antagonist Preparation (PMMA)

In each wear study an (PMMA) artificial acrylic denture tooth material (“Gnathostar™”, Ivoclar Vivadent) was used as an antagonist. Premolar artificial teeth were chosen and sectioned to leave the buccal cusp tip. The tips were then wet polished with 2000 grit silicon abrasive paper to achieve a standardised polished finish.

3.2 Mounting

All prepared samples were mounted on SEM mounting studs for subsequent SEM analysis and wear testing procedures. The bonding surfaces were prepared according to the cementation instructions for Rely-X Ultimate™ resin cement (3M ESPE). The bonding surfaces of the PMMA, resin nano-ceramic and zirconia specimens, and the SEM studs were subjected to airborne particle abrasion with Rocatec (3M ESPE) at 4 bar, and the Lithium Disilicate samples (eMax™) were acid etched with 6% hydrofluoric acid (Kerr) according to the manufacturers recommendations.

The SEM studs and the material sample bonding surfaces were treated with Scotchbond Universal Adhesive™ (3M ESPE) for twenty seconds and subsequently air-dried for five
seconds. Scotchbond Universal Adhesive™ is a mild self-etch adhesive consisting of phosphorylated monomers in a water/ethanol solution which provides acidity (pH 2.7). There are three “adhesion promoters” in the formulation: Vitrebond™ copolymer for dentine adhesion; MDP phosphate monomer which provides self-etching properties and chemically bonds to zirconia, alumina, and metal alloys; and silane for chemical adhesion to glass-based ceramic systems. The system also consists of dimethacrylate resins and HEMA for matrix formation around the filler components. The constituents of Rely-x ultimate™ resin cement are listed in Table 7 and the physical properties are listed in Table 8.

Table 7. Rely-X Ultimate™ resin cement constituents

<table>
<thead>
<tr>
<th>Base paste</th>
<th>Catalyst paste</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methacrylate monomers</td>
<td>Methacrylate monomers</td>
</tr>
<tr>
<td>Radiopaque, silanated fillers</td>
<td>Radiopaque alkaline (basic) fillers</td>
</tr>
<tr>
<td>Initiator components</td>
<td>Initiator components</td>
</tr>
<tr>
<td>Stabilizers</td>
<td>Stabilizers</td>
</tr>
<tr>
<td>Rheological additives</td>
<td>Pigments</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>RHEOLOGICAL ADDITIVES</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Fluorescence dye</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>DARK CURE ACTIVATOR FOR</td>
</tr>
<tr>
<td></td>
<td>SCOTCHBOND UNIVERSAL ADHESIVE</td>
</tr>
</tbody>
</table>
Table 8. Rely-X Ultimate™ resin cement physical properties.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Rely-x Ultimate (lc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexural strength (MPa)</td>
<td>98</td>
</tr>
<tr>
<td>Compressive strength (MPa)</td>
<td>262</td>
</tr>
<tr>
<td>Modulus of elasticity (GPa)</td>
<td>7.7</td>
</tr>
<tr>
<td>Surface hardness (HV 0.2)</td>
<td>40</td>
</tr>
<tr>
<td>Film thickness (µm)</td>
<td>12</td>
</tr>
<tr>
<td>Water sorption (µg/mm³)</td>
<td>21</td>
</tr>
<tr>
<td>Solubility (µg/mm³)</td>
<td>0</td>
</tr>
<tr>
<td>Expansion after 1 month (%)</td>
<td>0.5</td>
</tr>
</tbody>
</table>

The bonding surfaces were coated with a thin film of Rely-x ultimate™ cement. Three 2mm diameter stainless steel balls were cemented adjacent to the samples for orientation during surface profilometry. The material samples with applied cement were then light cured (3M
ESPE) for twenty seconds. Figure 32 shows examples of mounted zirconia and acrylic specimens.

![Mounted specimens](image)

Figure 32. Mounted specimens. *Left:* Zirconia. *Right:* Acrylic.

### 3.3 Scanning electron microscopy (SEM) observations and Surface texture quantification

Scanning electron microscopy (SEM) was used for two main purposes in this investigation:

- To investigate a novel three-dimensional surface roughness analysis method (Sa) for use in dental material analysis as opposed to conventional two-dimensional Ra values. Assessment was undertaken prior to and after completion of in-vitro wear testing. This is the first time this technology has been used for the study of dental materials.

- To investigate the surface roughness of the materials before and after wear testing.

All specimens were mounted on SEM studs as described. All material samples were subject to nitrogen air spray under high pressure to remove surface contaminants. The materials were then coated with a 15nm layer of carbon prior to analysis with the Phillips XL30 FEG Scanning Electron Microscope (Figure 33). Images were first taken for surface characterisation at up to 650x magnification. The samples were then subjected to the 3-D
surface texture analysis protocol for the MeX software analysis which required three scans from 0°, +11.5°, and -8.5° in order to generate the data set for analysis. Ra measurements were taken from identical 10 pixel wide lines drawn horizontally across the centre of the image region. Areas for Sa and Ra analysis were kept identical from sample to sample. All data was stored on removable data storage devices.

Figure 33. Phillips XL30 FEG Scanning Electron Microscope

3.3.1 MeX for Sa Analysis

Three dimensional reconstructions were performed in Alicona MeX®. In order to carry out the reconstructions, stereoscopic tilt-series images were acquired at Adelaide Microscopy, The University of Adelaide, on a Philips XL30 FEG-SEM. Samples were coated in a layer of carbon prior to imaging in order to make the sample conductive. The imaging process involved acquiring secondary electron (SE) images at zero, +11.5 and -8.5 degree tilts, on multiple regions per sample. Secondary electron imaging provides detail on topography and
surface features. Contrast and brightness settings can be altered in the SEM software, and were optimised for each area imaged to provide the greatest contrast possible without saturating the image.

Once acquired, images are imported into the MeX® software, and calibrated for X-Y distance using the scale bar on the SEM image. Z-distance is resolved by the software using the tilt-series images and is partly dependent on the changing brightness of pixels as they are tilted towards and away from the SE detector. Once the 3D model is created (Figure 34), before measurements are carried out a 2nd order polynomial “form removal” step is carried out. This removes any contribution to the roughness measurement from any large-scale surface variations, such as a non-flat surface as seen in some of the implant samples. Following this both $Ra$ and $Sa$ measurements were performed. $Ra$ measurements were taken from identical parts of every reconstruction, in which a 10 pixel wide line was drawn horizontally across the centre of the imaged region, and data output. Areas for $Sa$ measurements were kept identical from sample to sample, and comprised the entire imaged area.

![Figure 34. MeX generated image of a material sample for Sa analysis.](image)

### 3.3.2 Comparison of $Ra$ and $Sa$
One of the study aims was to undertake surface roughness (texture/topography) analysis of each sample utilising the most commonly used approach in dental material analysis ($Ra$) and a potentially more accurate three-dimensional method ($Sa$) in order to determine if there is a relationship between $Ra$ and $Sa$. Both are measured in nanometers (nm). Six measurements were taken in order to improve the confidence of the given $Ra$ and $Sa$ values for each material. The results in terms of means and standard deviations (SD) are presented in Tables 9 and 10 (the complete set are shown in Appendix 2). They are demonstrated in Figures 35 and 36.

Table 9. Baseline $Ra$ measurements. Mean and SD values are shown for the materials of interest. Acrylic sample material (A), Acrylic Antagonist material (AA), BruxZir™ Zirconia (BZ), Lava Plus™ (LP), DenZir™ Zirconia (DZ), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

<table>
<thead>
<tr>
<th>$Ra$ (nm)</th>
<th>A</th>
<th>AA</th>
<th>BZ</th>
<th>LP</th>
<th>DZ</th>
<th>eMax</th>
<th>LU</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>457.2</td>
<td>627.0</td>
<td>148.2</td>
<td>375.3</td>
<td>297.6</td>
<td>160.5</td>
<td>217.9</td>
</tr>
<tr>
<td>s.d.</td>
<td>161.3</td>
<td>138.6</td>
<td>12.5</td>
<td>84.7</td>
<td>83.1</td>
<td>31.9</td>
<td>47.1</td>
</tr>
</tbody>
</table>

Table 10. Baseline $Sa$ measurements. Mean and SD values are shown for the materials of interest. Acrylic sample material (A), Acrylic Antagonist material (AA), BruxZir™ Zirconia (BZ), Lava Plus™ (LP), DenZir™ Zirconia (DZ), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

<table>
<thead>
<tr>
<th>$Sa$ (nm)</th>
<th>A</th>
<th>AA</th>
<th>BZ</th>
<th>LP</th>
<th>DZ</th>
<th>eMax</th>
<th>LU</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>708.6</td>
<td>837.0</td>
<td>390.4</td>
<td>690.9</td>
<td>546.8</td>
<td>410.5</td>
<td>480.0</td>
</tr>
<tr>
<td>s.d.</td>
<td>93.2</td>
<td>74.3</td>
<td>86.1</td>
<td>53.4</td>
<td>84.5</td>
<td>42.8</td>
<td>74.7</td>
</tr>
</tbody>
</table>
Figure 35. Baseline $Ra$ value graph. Mean and SD values are shown for the materials of interest. Acrylic sample material (A), Acrylic Antagonist material (AA), BruxZir™ Zirconia (BZ), Lava Plus™ (LP), DenZir™ Zirconia (DZ), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

Figure 36. Baseline $Sa$ value graph. Mean and SD values are shown for the materials of interest. Acrylic sample material (A), Acrylic Antagonist material (AA), BruxZir™ Zirconia (BZ), Lava Plus™ (LP), DenZir™ Zirconia (DZ), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).
Data for both $Ra$ and $Sa$ values were plotted and a liner regression model (Excel, Microsoft Corporation) was fitted to assess the relationship between the two methods (Figure 37).

![Ra vs Sa Scatter Plot](image)

Figure 37. Scatter plot and $R^2$ correlation of the baseline Ra and Sa values.

The $R^2$ value (coefficient of determination) of 0.89 indicates a close relationship between the $Ra$ and $Sa$ values. This suggests that the $Ra$ and $Sa$ methods of surface texture analysis are providing very similar information. Experience with the two methods suggests that in a relatively homogenous specimens such as dental materials used here, the $Ra$ method is adequate, whereas for more heterogeneous specimens the $Sa$ method has advantages because it draws on data from a wider area of the specimen.

### 3.4 Profilometry

A 3-D scanner (Pix-4, Roland DG, Tokyo, Japan) with a piezo sensor that detects contact between its stylus and the surface being scanned was utilised for material surface scanning prior to and following the wear testing. The scanner was interfaced with a Windows-based PC to record mesh points of the scanned material. The software, “Dr PICZA” (Roland DG,
Tokyo, Japan), was provided with the scanner in order to define the scanning area, set the
scanning limits, define the resolution, and calibrate the scanner prior to each scan. The
resolution of the Pix-4 scanner was set for the finest values or “highest resolution” which was
50µm for the X and Y coordinates and 25µm for the Z axis. This required up to five hours for
scanning of each sample. The Dr PICZA software also allowed data visualisation and export
for further analysis (Ha 2011). Figure 38 shows the 3-D scanner used in this study.

![3-D scanner](image)

Figure 38. The 3-D scanner (Roland DG, Tokyo, Japan)

Purpose-written MATLAB 6.5 software (The Mathworks Inc, Natick MA, USA) was used to
analyse the data sets (Liu et al 2004). This software enabled the production of a 3-D graphic
of the scanned sample, on which the reference plan defined by the three stainless steel balls
could be identified (Figure 39). The software calculated the volume of the scanned sample
above this reference plan. For this study the volume was determined as a mean of three
separate scans of each sample and software analysis.
The validity and reliability of this scanning system has been confirmed in experiments reported by Liu et al (2004). It was shown that the difference in the scanning volume compared to the true volume was between 3.7% and 8.5%, depending on the geometry of the specimen. Wear volumes were recorded in cubic millimeters and the pre- and post-wear scans were tabulated in Microsoft Excel (Microsoft, USA) spreadsheets for subsequent analysis.

3.5 Wear testing machine

In the mid 1990’s an electro-mechanical wear machine was designed and fabricated by the Department of Mechanical Engineering at The University of Adelaide. The original machine, along with its sister machine have been used in tribology studies of various types and have resulted in a series of publications on tooth and dental restorative material wear (Kaidonis et al 1998; Shabanian and Richards 2002; Ranjitkar et al 2008). The wear machine and was originally described by Kaidonis et al (1998) in the following way:

**Figure 39.** A snapshot of a 3-D graphic of a scanned specimen
“The tooth wear apparatus consists of a stainless steel base and frame onto which all parts are secured. The machine is driven by a 75-watt D.C. electric motor configured to operate at variable speeds. The motor powers a 10:1 reduction gearbox that moves a series of interchangeable cams controlling the movement of one of two opposing specimen holders. These holders allow the specimens to be accurately repositioned at all times. Depending on the selected cam, movement can be controlled in either or both of two dimensions (horizontal and vertical). A simple adjustment screw on the cam follower, accurately establishes the degree of movement in the horizontal plane, allowing for control of the duration of contact of the enamel surfaces of specimen teeth (and material samples). A magnetic counter, attached to the drive of the gearbox, records the number of cycles of the machine”

“The upper mobile section of the machine was designed to support weights for applying loads to the specimens. Without the addition of load, the inherent weight of the upper component is 3.2 kg. Therefore, for load below 3.2 kg, the moveable component of the machine is attached to a counterbalanced overhead pulley system.”

“Experiments were based on uni-directional movements where a moving upper specimens was rubbed against a fixed lower specimen in one direction for a specified duration, after which the cam lifted the upper specimen and repositioned it at the beginning of the stroke. This action constituted one cycle.”

Stainless steel specimen holders accommodate stainless steel cylinders which are secured to the stationary base by two screws in an anti-rotational fixture. The upper specimen holder is fixed in place and receives the stainless steel cylinder which holds the antagonist sample material. The cylinders themselves are secured by lateral-directed screws.
The upper, moveable section, was designed to support additional weight which is desired to be applied to the sample(s). The weight range allowed load between 2.5N and 162N, which was stated as the range which represents a physiologic functional range (Kaidonis et al 1998). The wear machine is shown in Figure 40.

![Image](image_url)

**Figure 40.** a. The University of Adelaide customised wear machine. b. The set Acrylic Antagonist opposing an Acrylic sample material.
3.6 Wear machine protocols

3.6.1 Load (Applied Force)

There is no agreement in the literature on the most appropriate load for in vitro wear studies (Li 2001) and the relevant ISO standard provides no guidelines. Kelly (1996) stated that loads during mastication and swallowing ranges from 5N to 364N, and Nishigawa (2001) stated that nocturnal bite force may be up to 423N, while Gibbs et al (1981) stated that maximum bite forces may be as high as 1270N. Eisenburger and Addy (2002) found that researchers have used loads ranging from 2N to 162N. While previous studies using the University of Adelaide wear machines have utilised 99.5N (Kaidonis et al 1998; Shabanian and Richards 2002; Ranjitkar et al 2008), a literature review in this study on two-body in vitro, denture tooth wear found loads from 2N to 50N were commonly used.

3.6.2 Lubrication

Several lubricants have been used for in vitro two-body wear testing of human dental tissues and restorative dental materials. These have included: nothing (ie dry testing); tap water; distilled water; and human saliva. Some studies used thermo-cycling of lubricants (5°C to 55°C) whereas others are conducted at either room temperature or 37°C. The purpose of two-body in vitro wear testing is to investigate the influence of direct contact between the materials without a third body present, thus the lubricant acts to “simulate” a moist oral environment as well as to remove wear debris generated.

Ha (2011) investigated the potential effect of water uptake or loss during testing on the dimensional change of natural tooth enamel, and restorative materials including dental ceramics and composite resin materials. She found no significant dimensional changes.
In this study the lubrication system as described by Kaidonis et al (1998) was employed. It consisted of a plastic lubricant reservoir which was connected by plastic tubing to a pipette tip. The reservoir was placed 0.5m above the pipette tip and a flow device was attached to the tubing to control the flow rate. The tubing was suspended using laboratory stand and the pipette was positioned for direct flow on to the wearing interface of both upper antagonist and lower sample material. The flow rate was fixed at 0.5ml/min as previously described, and the water lubricant that had passed over the wearing materials was collected and discarded. In this study all specimens were lubricated with distilled water.

3.6.3 Type of Movement

The wear machine is adjustable for either uni- or bi-directional movements. Kaidonis et al (1998) concluded that measured wear rates for enamel specimens were the same for both movements at 32N and 99.5N. Shabanian and Richards (2000) confirmed when testing composite resin, glass ionomers and enamel at pH 7 under loads of 32N, 67N, and 99.5N.

3.6.4 Cycles (duration of experiment)

As outlined in the literature review of denture teeth tribology, there is a wide range in testing cycles depending on the study with as little as 1,000 cycles noted for one study and up to 1,200,000 being employed in another study (Ghazal et al 2008; Heintze et al 2012). The number of cycles depends on the minimum detectable tooth reduction (i.e. the measurement system), applied load, nature of the lubricant, and the wear of the specimens under the test conditions. On this particular wear machine, Kaidonis et al (1998) utilized 32N of load for 89,000 cycles at a speed of 80 cycles/min with data collected at intervals during the study. This provided discernable differences in wear in enamel worn with lubricant at a range of
pHs. Kaidonis et al (1998) found that enamel showed two phases of wear: Phase one in the first 20,000 cycles and phase two which continued up to the 89,000 cycles and beyond in a linear fashion.

A preliminary investigation in this study established that 240,000 cycles at 80 cycles/min under 97N of load utilising distilled water lubricant at room temperature produced detectable differences in wear.

### 3.6.5 Cycling rate

Kaidonis et al (1998) demonstrated that there was no statistically significant difference in mean enamel loss when the machine was run at either 80 cycles/min or 160 cycles/min. These study conditions included an applied load of 32N and were conducted for 89,000 cycles. Kaidonis et al (1998) also noted that at a speeds over 160 cycles/min, the upper specimen impacted the lower specimen with greater force during the return movement which resulted in an increase in surface breakdown. He concluded that the return movement was “controlled” at a rate of 80 cycles/min which was later confirmed by Ha (2011).

### 3.6.6 Area of specimen contact

The shape and size of natural teeth and artificial tooth restorations and denture teeth vary significantly, and as a result the facet areas vary. Kaidonis et al (1998) described the pattern of change in facet area during the wear process over 15,000 cycles. He found that an increase in facet area was linearly related to the increase in the number of cycles the samples were subjected to. This was also observed by Ratledge et al (1994).
Shabanian and Richards (2000) tested the effect of facet area on the rate of material wear under 99.5N with a water lubricant using SEM to quantify facet area at intervals up to 40,000 cycles. Height changes were also measured. The results indicated a low correlation between initial area and the change in height, but no significant relationship between facet area and wear rate could be established (Shabanian and Richards 2000).

### 3.7 Experimental protocol

#### 3.7.1 In vitro two-body Wear Study

The study involved the antagonistic wear of an acrylic antagonist denture tooth (polished) opposing various dental material samples. The acrylic material itself acted as the control (acrylic antagonist opposing acrylic sample material) with which the other material groups were compared. A single representative specimen material was chosen for each material group for the wear testing component of the study (ie Lava Plus™ represented the zirconia group with BruxZir™ and DenZir™ excluded). The following experiments were conducted:

1. Acrylic Antagonist opposing acrylic sample material (control series)
2. Acrylic Antagonist opposing nano-ceramic composite resin (Lava Ultimate™, 3M ESPE)
3. Acrylic Antagonist opposing monolithic zirconia (Lava Plus™, 3M ESPE)
4. Acrylic Antagonist opposing lithium disilicate (eMax™, Ivoclar-Vivadent)

There was 8 pairs in each test group and the control group. There were three measurements taken prior to testing (baseline) of the material volume as per the above mentioned protocol for the acrylic antagonist and its paired opposing material. Following testing, three further
measurements were taken for comparison. A mean and standard deviation was calculated from the three measurements.

For the material volume loss (wear) experiments, data sets for both the acrylic antagonist material and sample dental materials were summarised in tabular form with material sample, all measurements, and mean measurements. These results were plotted on a bar chart to demonstrate the material volume loss for each group as a whole, the respective acrylic antagonist for each group, and its respective opposing dental material sample.

The material samples of interest were tested against the acrylic material which acted as a control for all groups. The material specimen groups were also statistically tested against each other to determine if significant differences in the amount of material and/or antagonist specimen wear volumes occurred.

The null hypothesis was that all groups displayed the same amount of material wear and acrylic antagonist material wear.
3.7.2 Conclusions and Antagonistic Wear Study Protocols

The final wear experimental protocols were:

- Wear machine: The University of Adelaide customised wear machine

- Load: 97N (9.9kg)

- Cycles: 240,000 cycles at 80 cycles/min to maintain consistency and minimise effects of impact stress

- Movement: Uni-directional

- Lubricant: Distilled water at room temperature

- Sample Mounting: Antagonist PMMA denture teeth were mounted as antagonists and material samples (flat) were mounted as “samples”. All were mounted on SEM studs which were utilised for all objectives of the study. The Rely-x ultimate™ Resin cement was used to the manufacturer's instructions and three orientation stainless steel 1mm diameter balls were mounted alongside the materials.

- Samples were analysed before and after wear testing for surface texture (qualitative assessment and quantitative (Ra and Sa)) and volume changes for wear analysis.

3.7.3 Statistical Analysis

For each quantitative experiment, data sets were summarised in tabular form with sample size, raw measurements, and mean measurements.

Statistical significance testing was undertaken with student’s *t*-tests using a significance level of p<0.05 and these data were tabulated. This method was also utilized for the analysis of the wear testing volumetric changes for each of the material specimen groups.
Section 4

Results
4.1 Qualitative Surface Texture

4.1.1 Pre-wear Surface Evaluation

Qualitative surface texture characteristics are presented for the acrylic antagonist, acrylic sample, Lava ultimate™, Lava Plus™, DenZir™, BruxZir™, and eMax™ samples. At a gross view level (naked eye), the surfaces of all materials appeared “smooth” with no irregularities, with the zirconia materials being most light reflective. The sample were subjected to analysis by SEM at magnifications of 120x, 150x and 650x and the MeX software was utilised to develop a three-dimensional (3-D) image of the surface topography of each material.

The various materials showed characteristic surface topographies which subsequently underwent quantitative surface topography analysis using the two-dimensional method (Ra) and the three-dimensional method of (Sa). The images are presented below in the Figures outlined.

4.1.1.1 Acrylic Antagonist and Acrylic Test Samples (PMMA)

The Acrylic Antagonist (AA) material is an unfilled PMMA material, as is the acrylic Sample Material (A). The gross view of the AA and A specimens is seen in Figure 41. AA material was analysed at 120x and 650x magnification and the MeX 3-D image generated for quantitative analysis of the surface topography which is shown in Figures 42, 43 and 44. At 120x magnification, the area of the cusp tip which was hand polished shows parallel striations in various directions up to the border with the unpolished part of the sample (Figure 42). 650x magnification, as seen in Figure 43, Shows more detail of the polished area and the evidence of surface contamination by what is most probably debris from the polishing of the surface.
The MeX generated 3D image in Figure 44 gives a very clear projection of a sample area of the specimen and demonstrates the surface topography in high resolution. Here, the striations are clearly seen and the peaks and troughs are clearly evident. Figures 45, 46 and 47 show the surface topography of the acrylic sample material specimen, which as expected displays the same characteristics as the acrylic antagonist surface with both having been prepared in a same manner by hand polishing with abrasive paper.

Figure 41. Gross views of the acrylic antagonist (a) and acrylic sample material surface (b).
Figure 42. Acrylic Antagonist material at 120x magnification.

Figure 43. Acrylic Antagonist material at 650x magnification. The red arrows indicate possible surface contaminants left from the polishing process. The blue arrows indicate the striations left in the materials surface following hand polishing.

Figure 44. MeX 3-D image generated of the acrylic antagonist specimen for quantitative analysis of the surface topography. Dimensions are 468µm (x-axis) x 318µm (y-axis) x 27µm (z-axis).
Figures 45 and 46 show the acrylic sample specimen at 120x and 650x magnification and the MeX 3-D image (Figure 47) generated for quantitative analysis of the surface topography.

Figure 45. Acrylic Sample material at 120x magnification.

Figure 46. Acrylic sample material at 650x magnification.
4.1.1.2 Lava Ultimate™ (LU)

The Lava Ultimate™ Sample material was examined at gross level (Figure 48), 150x and 650x magnification, and a MeX 3-D image was generated for qualitative and quantitative analysis of the surface topography. The Lava Ultimate™ sample material was hand polished in the same manner as the acrylic material sample. The same striation pattern can be seen at both magnifications and in the MeX generated 3D image as seen in the acrylic images which is expected. The images are shown in Figures 49, 50, and 51.
Figure 48. Gross view of the Lava ultimate™ sample material specimen.

Figure 49. Lava Ultimate™ sample material at 150x magnification

Figure 50. Lava Ultimate™ sample material at 650x magnification.
4.1.1.3 eMax™

The eMax™ samples were examined at the gross level (Figure 52), 150x and 650x magnification by SEM and a MeX 3-D image was generated of the surface topography. These are demonstrated in Figures 53, 54, and 55. Figure 51 shows the sample at 150x magnification. Here, there are obvious differences between areas within the image. Some areas appear to have little or no surface texture variation and others seem to have significantly more variation. There may also be surface contamination either left over from the milling process and sintered to the surface, or debris left on the surface after finishing procedures were completed. Figure 55 which shows the MeX 3D image clearly demonstrates the surface variation as seen in the 2D SEM images.
Figure 53. eMax™ sample material at 150x magnification. The red arrows demonstrate areas of little surface topography variation and the blue arrows show areas of pronounced surface topography variation.

Figure 54. eMax™ sample material at 650x magnification. The red arrows demonstrate areas of little surface topography variation and the blue arrows show areas of pronounced surface topography. The orange arrows show the striations in the surface of the material left by the milling process.
Figure 55. MeX 3-D image generated for quantitative analysis of the surface topography. Dimensions are 466µm (x-axis) x 317µm (y-axis) x 11.85µm (z-axis). The red arrows demonstrate areas of little surface topography variation and the blue arrows show areas of pronounced surface topography.

4.1.1.4 Lava Plus™ (LP)

The Lava Plus™ samples were examined at 150x and 650x magnification via SEM and a MeX 3-D image was generated of the surface topography. These are demonstrated in Figures 57, 58, and 59. Figure 56 shows the gross view of a Lava Plus specimen material surface which has a high luster. Figure 57 shows the sample at 150x magnification. Here, there are obvious striations in the material surface within the image. These striations are very pronounced. There may also be surface contamination either left over from the milling process and sintered to the surface, or debris left on the surface after finishing procedures were completed. Figure 59, which shows the MeX 3D image clearly demonstrates the surface variation as seen in the 2D SEM images.
Figure 56. Gross view of Laval Plus™ sample with high surface luster evident.

Figure 57. Lava Plus™ sample material at 150x magnification. Red arrows show the obvious striations in the material surface. The blue arrows show surface contamination or debris.
Figure 58. Lava Plus™ sample material at 650x magnification.

Figure 59. Lava Plus™ MeX 3-D image generated for quantitative analysis of the surface topography. Dimensions are 466µm (x-axis) x 317µm (y-axis) x 20µm (z-axis).
4.1.1.5 BruxZir™

The BruxZir™ sample was examined at 150x and 650x magnification via SEM and a MeX 3-D image was generated of the surface topography. These are demonstrated in Figures 61, 62, and 63. Figure 60 shows the gross view of the material surface which has a mat type finish with little luster. Figure 61 shows the sample at 150x magnification. Here, there are obvious striations in the material surface within the image. These striations are very pronounced. There may also be surface contamination either left over from the milling process and sintered to the surface, or debris left on the surface after finishing procedures were completed. Figure 63 which shows the MeX 3D image clearly demonstrates the surface variation as seen in the 2D SEM images.

Figure 60. Gross view of BruxZir™ sample
Figure 61. BruxZir™ sample material at 150x magnification. The red arrows demarcate the striations in the material surface and the blue arrows show possible debris on the surface.

Figure 62. BruxZir™ sample material at 650x magnification.
Figure 63. MeX 3-D image of a BruxZir™ specimen generated for quantitative analysis of the surface topography. Dimensions are 468µm (x-axis) x 318µm (y-axis) x 17µm (z-axis).

4.1.1.6 DenZir™

The DenZir™ sample was examined at 150x and 650x magnification via SEM and a MeX 3-D image was generated of the surface topography. These are demonstrated in Figures 65, 66, and 67. Figure 64 shows the gross view of the material surface which has a high luster finish. Figure 65 shows the sample at 150x magnification. Here, there are no obvious striations in the material surface within the image. There are areas of inclusion in the material surface as shown in Figure by the red arrows. There may also be surface contamination either left over from the milling process and sintered to the surface, or debris left on the surface after finishing procedures were completed. Figure 67 which shows the MeX 3D image clearly demonstrates the surface variation as seen in the 2D SEM images.
Figure 64. Gross view of DenZir™ sample

Figure 65. DenZir™ sample material at 150x magnification.
Figure 66. DenZir™ sample material at 650x magnification. The red arrows indicate surface defects and the blue arrows show possible surface contamination.

Figure 67. MeX 3-D image generated for quantitative analysis of the surface topography. Dimensions are 467µm (x-axis) x 317µm (y-axis) x 12µm (z-axis).
4.1.2 Post wear surface evaluation

The SEM qualitative analysis was also undertaken for the materials post testing which are seen in Figures outlined in the following section.

4.1.2.1 Acrylic antagonist and test material samples

*Group 1: Acrylic Antagonist opposing Acrylic material sample*

The acrylic antagonist is shown in Figure 68 at the pre-testing and post-testing intervals at the gross level. Figure 68 (b) shows a clear wear facet in the acrylic antagonist as indicated by the red arrow. Figure 69 demonstrates the acrylic sample specimen at the completion of antagonist wear testing and a clear wear facet with a change in surface topography is shown by the red arrows.

![Figure 68](image)

Figure 68. a. The acrylic antagonist pre-testing, and b. the acrylic antagonist post testing (representative of the group). The red arrows indicate the area of change on the cusp tip.
Figure 69. The gross view of an acrylic sample post testing with red arrows clearly showing a wear facet area.

Figure 70 shows the worn surface of the acrylic antagonist following 240,000 cycles. The red arrows indicates the wear facet area where attrition type striations are obvious. The blue arrow shows the polished surface which has not been subjected to two-body wear. There are clear differences between the two areas of the material surface. Breakdown in the surface can also be seen adjacent the green arrows and debris or surface artifact is visible at the orange arrows. The Me-X generated image of the surface is seen in Figure 71 which highlights the surface texture.
Figure 70. The worn surface of acrylic antagonist (versus acrylic) at 120x magnification. a. The edge of the wear facet, b. The polished, but unworn area of the antagonist. The red arrows indicates the wear facet area where attrition type striations are obvious. The blue arrow shows the polished surface which has not been subjected to two-body wear. There are clear differences between the two areas of the material surface. Breakdown in the surface can also be seen adjacent the green arrows and debris or surface artifact is visible at the orange arrows.

Figure 71. The worn surface of acrylic antagonist (opposing acrylic).
Figure 72. The worn area of the acrylic sample (opposing acrylic antagonist) at 120x magnification SEM. a. The contact wear facet. b. The non-contacted polished area.

Figure 72 demonstrates the worn surface of acrylic sample (opposing acrylic antagonist) showing parallel striations up to the border with the unpolished part of the sample which was described by Kaidonis (2008) as being representative of attrition. The Me-x generated image of the acrylic sample material specimen wear facet is shown in Figure 73.

Figure 73. Me-X generated image of the wear facet within the acrylic material specimen.
Group 2: Acrylic Antagonist opposing Lava Ultimate material sample

The acrylic antagonist is shown in Figure 74 (page 123) at the pre-testing and post-testing intervals at the gross level. Figure 74 (b) shows a clear wear facet in the acrylic antagonist as indicated by the red arrow.

![Figure 74. Acrylic versus Lava Ultimate. a. The acrylic antagonist pre-testing, and b. the acrylic antagonist post-testing in a specimen representative of the group.](image)

Figure 75 shows the worn surface of the acrylic antagonist (versus Lava ultimate) at 120x magnification. No obvious striations are present, however the red arrows indicate areas of surface degradation in the acrylic antagonist material. The Me-X generated image of the AA specimen is represented in Figure 76.
Figure 75. An SEM image taken at 150x magnification of the worn surface of acrylic antagonist (versus LU). a. The wear facet area. b. The unworn polished area.

Figure 76. The MeX 3D image of the acrylic antagonist material surface opposing the lava ultimate sample™ material.

Figure 77 shows a representative specimen of the lava ultimate sample™ at the completion of antagonist wear testing and no clear wear facet is present. There was no change in surface topography obvious at the gross level in most samples. Figure 78 demonstrates images from a lava ultimate™ specimen where relatively deep grooves or striations are present. This only
occurred in two of the tested specimens. SEM images were taken at a higher magnification of 650 x (Figure 79) which highlights these “grooves”. Figures 80 and 81 respectively represent
the Me-X images of the wear facet of lava ultimate showing the area of little surface
topography variation marked by the letter ‘a’ in Figure 78 and the area of significant surface
topography variation indicated by the letter ‘b’ in Figure 78.

Figure 77. The worn surface of the lava ultimate material sample post testing

Figure 78. The worn surface of the lava ultimate™ (opposing acrylic antagonist) at
120x magnification. a. The wear facet. b. Significant texture change within
the wear facet area. ‘C’. The unworn polished surface area. The red arrow
indicates an area of deep grooves or striations in the material surface.
Figure 79. The worn surface of the lava ultimate (opposing acrylic antagonist) at 650x magnification. The blue arrow indicates significant surface topography changes were identified in the wear facet area of the lava ultimate material. The orange arrow indicates the filler particles are clearly visible at 650x magnification. The red arrow demarcates an area of deep grooves or striations in the material surface.

Figure 80. MeX generated image of the worn surface of lava ultimate (versus acrylic antagonist).
Group 3: acrylic antagonist opposing eMax material sample

The acrylic antagonist is shown in Figure 82 at the pre-testing and post-testing intervals at the gross level. Figure 82 (b) shows a clear wear facet in the acrylic antagonist as indicated by the red arrow. The acrylic antagonists’ wear facet following the wear study is shown in the SEM image at 150x magnification in Figure 83. The worn surface of the acrylic antagonist is represented by the Me-X generated image of Figure 84. Figure 85 shows the eMax™ sample at the completion of antagonist wear testing and no clear wear facet is present. There was no change in surface topography obvious at the gross level in most samples (Figure 86). Figure 87 is representative of Me-X images from an eMax™ sample specimen where no striations or grooves are present.
Figure 82. Acrylic antagonist opposing eMax™. a. The acrylic antagonist pre-testing, and b, the acrylic antagonist post testing. This is a representative sample from the group.

Figure 83. The worn surface of the acrylic antagonist (opposing eMax™) at 120x magnification. a. The wear facet. b. The unworn polished area. The red arrow indicates areas of surface degradation. The blue arrow shows an area of debris on the surface.
Figure 84. The worn surface of acrylic antagonist (opposing eMax™)

Figure 85. The worn surface of the eMax™ material sample post wear testing.

Figure 86. The worn surface of the eMax (opposing acrylic) at 120x magnification. a. Surface texture changes following a unidirectional pattern of striations within the wear “facet” area (red arrows). Blue arrows show areas of debris on the surface of the material.
Figure 87. The worn surface of the eMax™ sample (opposing acrylic antagonist)

*Group 4: Acrylic Antagonist opposing Lava Plus material sample*

The acrylic antagonist is shown in Figure 88 at the pre-testing and post-testing intervals at the gross level. Figure 88 (b) shows a clear wear facet in the acrylic antagonist as indicated by the red arrow. The SEM image of the worn surface of the acrylic antagonist at 120x magnification clearly demonstrates a wear facet (Figure 89) and the Me-X generated image (Figure 90) highlights the striations of the wear facet. The Lava Plus™ specimen at the completion of wear testing and no clear wear facet (Figure 91). There was no change in surface topography obvious at the gross level in most samples. Figure 92 shows images from a Lava Plus sample where striations or grooves are present which are consistent with those that were present in the pre-testing state. These are also obvious in the Me-X generated image (Figure 93).
Figure 88. acrylic versus Lava Plus™. a. The acrylic antagonist pre-testing, and b, the acrylic antagonist post testing.

Figure 89. The worn surface of the acrylic antagonist (opposing Lava Plus™) at 120x magnification. Blue arrow - The wear facet area. Red arrow - The unworn polished area.
Figure 90. The worn surface of acrylic antagonist (versus LP). Red arrows indicate striations (unidirectional) in the acrylic antagonist material surface.

Figure 91. The worn surface of the Lava Plus™ material sample.
Figure 92. The worn surface of the Lava Plus (opposing acrylic) at 120x magnification. a. Similar surface topography as seen in the baseline scan.

Figure 93. MeX generated 3D image of the worn surface of Lava Plus™ (opposing acrylic antagonist). The surface topography of the Lava Plus sample is as it was prior to the testing phase with clear unidirectional features in the material surface. As discussed earlier, these may be the result of the milling process in manufacturing this material.
4.2  Quantitative (Post Wear) Surface Texture Analysis

Following the active two-body wear testing phase, a representative range of materials were analysed for material surface roughness and the volumetric change.

4.2.1  *Ra* measurements post two body *in-vitro* wear testing of acrylic and sample materials

*Ra* measurements were undertaken of the acrylic antagonist and sample materials following two body wear testing. These are shown in Tables 11 and 12, representing the acrylic antagonist and the tested sample materials respectively. Figures 94 and 95 show a comparison of *Ra* values from baseline measurements to the post tested state for both groups.

<table>
<thead>
<tr>
<th>acrylic antagonist material <em>Ra</em> (nm) measurements</th>
<th>Acrylic</th>
<th>eMax</th>
<th>LU</th>
<th>LP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>387.15</td>
<td>245.82</td>
<td>311.75</td>
<td>568.87</td>
</tr>
<tr>
<td>SD</td>
<td>106.52</td>
<td>51.91</td>
<td>124.02</td>
<td>251.72</td>
</tr>
</tbody>
</table>

Table 11. acrylic antagonist *Ra* values post two body wear testing

<table>
<thead>
<tr>
<th>Sample Materials <em>Ra</em> (nm) measurements</th>
<th>Acrylic</th>
<th>eMax</th>
<th>LU</th>
<th>LP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>545.51</td>
<td>169.49</td>
<td>134.49</td>
<td>313.76</td>
</tr>
<tr>
<td>SD</td>
<td>180.37</td>
<td>37.42</td>
<td>16.00</td>
<td>63.78</td>
</tr>
</tbody>
</table>

Table 12. Sample Material *Ra* values following two body wear testing

On review, the areas of gauging in the lava ultimate™ sample materials were not included in the initial data as they only occurred on a few of the tested samples, not all.
Figure 94. Sample material $Ra$ values (nm) at baseline and post two-body wear testing. Acrylic sample material (A), Lava Plus™ (LP), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

Figure 95. Acrylic Antagonist material $Ra$ values (nm) at baseline and post two-body wear testing. Acrylic sample material (A), Lava Plus™ (LP), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).
Student’s t-test was undertaken to determine if the differences in $Ra$ values for the acrylic antagonists opposing the sample materials of interest and the sample materials themselves were significantly different from each other before and after two-body wear testing. The t-test was done for all the materials within the tested groups. The results are shown in the Tables outlined below.

When the $Ra$ values in the post wear tested sample materials were compared, the pairwise comparisons (Table 13) show that apart from the eMax™ with a surface roughness intermediate between that of the lava ultimate™ and Lava Plus™ (and not significantly different to either) all other difference were statistically significant. Thus the null hypothesis that all the materials would have the same $Ra$ values post testing is rejected. It is evident that after 240,000 cycles of two-body wear against and acrylic antagonist that:

- The acrylic specimens were rougher than all other materials
- The Lava Plus™ specimens were smoother than the acrylic but rougher than the lava ultimate™
- The eMax™ was smoother than the acrylic but similar in roughness to both the Lava Plus™ and lava ultimate™
- Lava ultimate™ was similar in roughness to the eMax™ but smoother than all of the other materials.

Table 13. P-values for pairwise comparisons of $Ra$ values within the post-wear testing sample materials

<table>
<thead>
<tr>
<th>Post Testing Ra differences between acrylic and the other materials (p=0.05)</th>
<th>acrylic</th>
<th>eMax</th>
<th>Lava ultimate</th>
<th>Lava Plus</th>
</tr>
</thead>
<tbody>
<tr>
<td>acrylic</td>
<td></td>
<td>0.022654</td>
<td>0.019352</td>
<td>0.022654</td>
</tr>
<tr>
<td>eMax</td>
<td>0.1595</td>
<td></td>
<td>0.159515</td>
<td>0.012102</td>
</tr>
<tr>
<td>ultimate</td>
<td></td>
<td></td>
<td></td>
<td>0.00885</td>
</tr>
<tr>
<td>plus</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
When the Ra values in the post wear tested acrylic antagonists (see Table 14) were compared the results show that none of the compared Ra values differed significantly (Table 14). Thus the null hypothesis that after 240,000 cycles of two-body wear the Ra values for the acrylic agonists will not differ significantly is supported.

Table 14. P-values for pairwise comparisons of the Ra values within the post-wear testing acrylic antagonists

<table>
<thead>
<tr>
<th>Post Testing Ra differences between acrylic antagonists (p=0.05)</th>
<th>acrylic</th>
<th>emax</th>
<th>Lava ultimate</th>
<th>Lava Plus</th>
</tr>
</thead>
<tbody>
<tr>
<td>acrylic</td>
<td>0.070398</td>
<td>0.445639</td>
<td>0.253731</td>
<td></td>
</tr>
<tr>
<td>emax</td>
<td></td>
<td>0.460952</td>
<td>0.080086</td>
<td></td>
</tr>
<tr>
<td>ultimate</td>
<td></td>
<td></td>
<td>0.141833</td>
<td></td>
</tr>
<tr>
<td>plus</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4.2.2 Ra values of acrylic antagonists and material specimens before and after two-body wear testing

A positive difference between pre- and post-wear Ra values between the acrylic antagonists indicates and increase in roughness.

P-values for the pairwise comparisons of the pre-and post-test data for the acrylic agonists (see Figure 95) for each material specimen group are shown in Table 15. The results show that for the acrylic, eMax™, and lava ultimate™ groups the opposing acrylic antagonists increased significantly in roughness. Only the Lava Plus™ specimens did not result in a significant increase in roughness of the opposing tooth. Therefore, the null
hypothesis that the acrylic antagonists in all groups would have the same $Ra$ values following wear testing can be rejected.

Table 15. P-values for comparisons of $Ra$ values of the acrylic antagonists before and after two-body wear testing

<table>
<thead>
<tr>
<th>Pre- and Post-Testing $Ra$ differences of acrylic antagonists between groups ($p=0.05$)</th>
<th>acrylic</th>
<th>eMax</th>
<th>Lava ultimate</th>
<th>Lava Plus</th>
</tr>
</thead>
<tbody>
<tr>
<td>acrylic</td>
<td>0.015649</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>eMax</td>
<td></td>
<td>0.000529</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lava ultimate</td>
<td></td>
<td></td>
<td>0.020995</td>
<td></td>
</tr>
<tr>
<td>Lava Plus</td>
<td></td>
<td></td>
<td></td>
<td>0.693907</td>
</tr>
</tbody>
</table>

P-values for the comparison of pre-and post-wear Ra values of the tested sample materials (see Figure 94) are shown in Table 16. The results showed no significant change in surface roughness with 240,000 cycles of wear against an acrylic antagonist for that the acrylic sample material, eMax™, and Lava Plus™ supporting the null hypothesis that there would be no significant differences. The lava ultimate™ group, however, showed a significant reduction in $Ra$ indicating that it becomes smoother with wear.

Table 16. Significance testing of $Ra$ values of the tested material samples before and after two-body wear testing.

<table>
<thead>
<tr>
<th>Post Testing $Ra$ differences of sample materials between groups ($p=0.05$)</th>
<th>acrylic</th>
<th>eMax</th>
<th>Lava ultimate</th>
<th>Lava Plus</th>
</tr>
</thead>
<tbody>
<tr>
<td>acrylic</td>
<td>0.459246</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>eMax</td>
<td></td>
<td>0.706658</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ultimate</td>
<td></td>
<td></td>
<td>0.005903</td>
<td></td>
</tr>
<tr>
<td>plus</td>
<td></td>
<td></td>
<td></td>
<td>0.254077</td>
</tr>
</tbody>
</table>
4.3 Volumetric analysis: acrylic antagonist (AA) and material sample specimen volume loss following 240,000 cycles of tribology testing (Volume loss in mm³).

Paired opposing surfaces were profile mapped and digitally recorded at the beginning and end of the wear cycle tests. There were three measurements taken of the antagonist material and its paired sample material from which a mean value for each acrylic antagonist and its paired material sample were formulated. These measurements were done prior to and after the wear testing cycles. The mean and standard deviations were formulated from these data sets. The mean volume losses for each opposing material are presented in Table 17 for both the acrylic antagonist and its opposing material sample. These are presented graphically in Figure 96. The complete results are shown in Appendix 3. Each antagonist material pairing consisted of eight (8) pairs of acrylic antagonist material and sample test material.

The material volume change data following the two body wear testing phase indicated that the Lava Ultimate™ material was the most abrasive of the acrylic antagonist, and the Lava Plus™ was the least abrasive. Statistical significance testing was undertaken for the acrylic antagonist material and the sample materials regarding their respective changes following testing when compared to the acrylic control group. The null hypotheses here were that the acrylic antagonists were worn equally by each opposing sample material and also, that the sample materials were equally affected by the acrylic antagonist when compared to the acrylic control group.
Table 17. Post testing material loss (wear) in mm³. Acrylic sample material (A), Lava Plus™ (LP), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

<table>
<thead>
<tr>
<th>Material</th>
<th>Mean (mm³)</th>
<th>s.d.</th>
<th>Antagonist</th>
<th>Mean (mm³)</th>
<th>s.d.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acrylic</td>
<td>0.792</td>
<td>0.309</td>
<td>Acrylic</td>
<td>0.446</td>
<td>0.249</td>
</tr>
<tr>
<td>Lava Ultimate</td>
<td>0.030</td>
<td>0.020</td>
<td>Lava Plus</td>
<td>1.318</td>
<td>0.324</td>
</tr>
<tr>
<td>eMax</td>
<td>0.012</td>
<td>0.017</td>
<td>eMax</td>
<td>0.676</td>
<td>0.117</td>
</tr>
<tr>
<td>Lava Plus</td>
<td>0.024</td>
<td>0.045</td>
<td>Lava Plus</td>
<td>0.405</td>
<td>0.102</td>
</tr>
</tbody>
</table>

Figure 96. Total mean material volume loss (mm³) for acrylic antagonists and sample materials by test group and total overall combined antagonist and sample material volume loss for each test group. Acrylic sample material (A), Lava Plus™ (LP), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

P-values for the comparisons of the volumetric changes for the acrylic antagonists opposing the sample materials of interest and the sample materials themselves are shown in Tables 18 and 19.
The data clearly show that when opposed to Lava Ultimate™ and eMax™, the acrylic antagonist wear was significantly greater than the acrylic and Lava Plus™ groups. The Lava Plus™ and acrylic versus acrylic antagonists wore at comparable rates. The acrylic antagonist wear rates were greatest when worn against Lava Ultimate™ and least when worn against Lava Plus™, with the differences between Lava Ultimate™, Lava Plus™ and eMax™ being significantly different from each other. Ultimately Lava Plus™ is the same as the denture tooth as highlighted in Table 18.

For the sample materials (Table 19), none of them wore significantly when compared with acrylic.

Table 18. P-values for pairwise comparisons of acrylic antagonist wear when opposing different sample materials.

<table>
<thead>
<tr>
<th>AA wear RE</th>
<th>Acrylic</th>
<th>eMax</th>
<th>Lava ultimate</th>
<th>Lava Plus</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA-A Control</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acrylic</td>
<td>0.039</td>
<td>0.000</td>
<td>0.677</td>
<td></td>
</tr>
<tr>
<td>eMax</td>
<td></td>
<td>0.001</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Lava ultimate</td>
<td></td>
<td></td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Lava Plus</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 19. P-values for pairwise comparison of different sample material wear when opposing acrylic antagonist.

<table>
<thead>
<tr>
<th>Sample wear RE</th>
<th>Acrylic</th>
<th>eMax</th>
<th>Lava ultimate</th>
<th>Lava Plus</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA-A Control</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acrylic</td>
<td></td>
<td>0.0002</td>
<td>0.0002</td>
<td>0.0002</td>
</tr>
<tr>
<td>eMax</td>
<td></td>
<td>0.0803</td>
<td>0.5259</td>
<td></td>
</tr>
<tr>
<td>Lava ultimate</td>
<td></td>
<td></td>
<td>0.7418</td>
<td></td>
</tr>
<tr>
<td>Lava Plus</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Section 5

Discussion
Section 5.0 - Discussion

5.1 Baseline/Pre-testing material analysis

The acrylic antagonist material and the material samples were examined prior to testing at the “naked eye” level up to 650x magnification via SEM and by 3-D reconstruction of the surface texture profile.

5.1.1 Qualitative Analysis

On visual examination the DenZir™ monolithic zirconia material demonstrated the highest lustre of any of the materials. It is noteworthy that the DenZir™ material is a “HIP” zirconia which was prepared in the post sintered state, unlike the other monolithic zirconia materials, BruxZir™ and Lava Plus™ which are non-HIP zirconia materials which are initially prepared in the pre-sintered state. The remaining zirconia materials also demonstrated a lustrous finish similar to the eMax™ material. The Lava Ultimate™ and the acrylic material used for the antagonist and control sample appeared smooth to the naked eye, however they lacked the lustre of the ceramic materials.

SEM analysis of the surface texture of the materials was conducted at 120 x, 150 x, and 650 x magnification. Under these magnifications it became obvious that the DenZir™ zirconia material which showed the highest lustre to the naked eye, also demonstrated a surface which generally lacked surface topography changes. The BruxZir™ and Lava Plus monolithic zirconia materials had obvious peaks and rises in their respective surfaces which may be the result of the manner in which they are manufactured with diamond burs to reduce a solid block of this material to the crown form designed on the relevant software system. The Lava Plus material demonstrated what appeared to be larger peaks and troughs in the surface topography when compared to the BruxZir™ material. The eMax™ material demonstrated a
surface which was closer to the DenZir™ material than the other materials. The eMax™ surface demonstrated minimal variations in the surface texture, however there were a number of isolated rises which may be particles that have been sintered to the surface as a result of the milling process. The Lava Ultimate™ and the acrylic antagonist and sample were all polished with 2000 grit silicone abrasive paper prior to testing. All three materials demonstrated a similar surface finish under magnification with obvious peaks and troughs in the surface texture.

The 3-D surface texture images clearly demonstrated variations in the surface topography of each of the materials used within this study. The striations present in the acrylic, Lava Ultimate™, Lava Plus™, and BruxZir™ materials were clearly reproduced in the generated 3-D images.

The 3-D images for the DenZir™ material samples showed little surface texture variation as with the preceding SEM images. From a qualitative perspective, the DenZir™ material clearly lacked the surface topography of the other materials. This was followed by the eMax™ sample, the BruxZir™ material, then the Lava Plus™, Lava ultimate™, and acrylic materials.

The SEM MeX generated images of the surface topography of dental materials provided a useful tool for qualitative assessment of a materials surface and gave a valuable insight into the changes in surface topography from a pre-testing state to the post-testing state. Clear patterns emerged for the different materials regarding the development of a wear facet and the degradation of a given materials surface following antagonistic two-body wear.
5.1.2 Quantitative Surface Texture Analysis

Quantitative analysis via SEM images following the Ra and Sa protocol of the acrylic antagonist and sample material, eMax™, Lava Ultimate™, Lava Plus™, BruxZir™, and DenZir™ materials was undertaken. Historically, the two-dimensional Ra method has been used to quantify surface texture. However, a novel three-dimensional approach is now available, the Sa (3-D), which was also undertaken in this study to assess if this can provide more accurate surface texture analysis results for dental materials of interest. To firstly characterize the materials of interest and secondly compare the Ra and Sa values for given materials, Ra and Sa analysis was undertaken on the material samples prior to any testing phase.

To allow a direct comparison of the Ra and Sa data analysis was undertaken here on the same images. The results showed that the Sa method appeared to be more sensitive to variations in surface topography but both the Ra and the Sa methods indicated the same overall order of the materials regarding the values for the surface topography and the Ra and Sa were highly correlated (r=0.94). In terms of surface texture or roughness, the highest values were attained for the Acrylic group followed by Lava Plus™, DenZir™, Lava Ultimate™, eMax™, and BruxZir™ in descending Ra and Sa value order.

Given that the same order was achieved in terms of ranking the materials of interest in terms of most to least rough, it is logical to suggest that both methods can be used to rank materials and give a quantifiable value of the relative surface roughness or texture of a given material of interest. One important factor to consider is that the Ra method is much quicker and cheaper...
to undertake when compared to the 3-D \textit{Sa} method and thus can be more readily used in dental material surface texture analysis.

\section*{5.2 Post two-body \textit{in-vitro} wear testing analysis}

Two-body in-vitro wear testing was undertaken utilising an acrylic antagonist cusp-form opposing four materials including acrylic as control, Lava Ultimate\textsuperscript{TM}, Lava Plus\textsuperscript{TM}, and eMax\textsuperscript{TM}. The samples were subjected to 240,000 cycles of wear testing in a customised wear machine. Following testing, the materials were subjected to the same methods of analysis used for baseline assessments including qualitative analysis via visual inspection, SEM imaging up to 650x magnification, and 3-D surface texture profile reproduction. Quantitatively, the tested materials underwent \textit{Ra} and \textit{Sa} surface texture analysis in order to quantify any changes in the worn or wear facet area. The materials were also examined via 3-D profilometry to assess material volume loss over the 240,000 testing cycles. This was conducted in order to quantify the wear (material loss) relationship between these opposing materials.

\subsection*{5.2.1 Qualitative analysis}

Visual assessment of the materials revealed that a clear wear facet developed on the acrylic antagonist material for all tests undertaken. There were obvious wear facets in the control acrylic material and the Lava Ultimate\textsuperscript{TM} material, albeit to a much lesser extent, and no clear wear facets were seen on the eMax\textsuperscript{TM} or Lava Plus\textsuperscript{TM} materials.

The wear facets in the acrylic antagonist material all appeared smooth, except in several cases where it was opposing Lava Ultimate\textsuperscript{TM} where gouged areas were obvious. There were also
gouge marks seen in the opposing Lava Ultimate™ material sample specimen. This type of deterioration was only seen in three (3) of the tested Lava Ultimate™ groups.

The ceramic materials appeared unchanged following testing.

**SEM analysis** of the surface texture of the materials was conducted at 120 x, 150 x, and 650 x magnification. Under these magnifications it was evident that the surface topography of the acrylic antagonist materials had changed from the baseline, where there was a clear demarcation between the wear facet which now appeared much smoother than it did at baseline. The exceptions to this were the gouge marks which appeared in some of the acrylic antagonists opposing the Lava Ultimate™ material. It is speculated that this could be the result of breakdown between the filler and matrix components in the Lava Ultimate™ surface which creates irregularity in the opposing surfaces, and thus a gouging effect. This irregularity is then exaggerated over the course of the wear cycles.

Where the acrylic opposed acrylic, there were obvious gouges which followed the unidirectional movement of the antagonist material. The acrylic antagonists for the acrylic control and the Lava Ultimate™ material sample clearly demonstrated more variation in surface texture than the acrylic antagonists opposing the ceramic materials.

SEM imaging of the Lava Plus™ and the eMax™ material samples showed only minor changes from the baseline images. The eMax™ images showed minor texture changes along the unidirectional path of the wear machine. It is apparent that the ceramic materials had “polishing” effect on the opposing acrylic antagonist material as less surface texture variation was seen after the wear cycles.
The 3-D surface texture images clearly demonstrated variations in the surface topography of each of the materials used within this study which corresponded to those evident is the 2-D SEM images.

The 3-D image regenerated the surface profile of the Lava Ultimate™ samples and clearly demonstrated the gouge marks in the material surface post testing. The 3-D images of the eMax™ and Lava Plus™ materials demonstrated little qualitative change from the baseline images. The MeX images provided a high resolution of the material surfaces which aided in the qualitative assessment of the materials tested.

5.2.2 Quantitative Analysis

Ra measurements were undertaken of the acrylic antagonist and sample materials following two body wear testing.

The Ra values for the Acrylic Antagonists changed significantly from the baseline values as the result of 240,000 cycles of in vitro wear. In this form of testing, the 240,000 cycles corresponds to approximately 24 months in situ (Mello et al 2009).

The Ra data for the tested sample materials revealed that:

- The acrylic specimens were rougher than all other materials
- The Lava Plus™ specimens were smoother than the acrylic but rougher than the Lava Ultimate™
- The eMax™ was smoother than the acrylic but similar in roughness to both the Lava Plus™ and Lava Ultimate™
- Lava Ultimate™ was similar in roughness to the eMax™ but smoother than all of the other materials.
5.2.3 Material Wear and Volume Changes

The volumetric analysis of material loss indicate that the acrylic antagonist suffered more aggressive attrition-type wear and thus material loss when opposing the Lava Ultimate™ material compared to the control acrylic group and the eMax™ and Lava Plus™ groups. The least acrylic antagonist wear was demonstrated for the acrylic versus Lava Plus group followed by the acrylic control and the eMax groups. One possible reason for the Lava Ultimate™ resin nano-ceramic composite causing a higher amount of wear on the acrylic antagonist material is that the softer resin matrix is broken down quicker than the nano-ceramic clusters, thus leaving the ceramic clusters exposed which results in an increase in surface texture and a resulting increase in attrition effect.

Statistical significance testing via student’s t-test was undertaken regarding the differences in Antagonist (Acrylic) wear (volume loss) when opposing different sample materials when compared to the Acrylic versus Acrylic group which acted as a control. The \( p\)-value for significance testing was set at \( p<0.05 \). The results of the testing are shown in Table 18 and the data revealed the following:

- The acrylic antagonists in the Lava Plus™ group were not significantly different from the acrylic versus acrylic group.
- The acrylic antagonists in the eMax™ and Lava Ultimate™ groups had \( p\)-values \(<0.05 \) and were thus were worn significantly more than the acrylic antagonists in the acrylic versus acrylic group.
- The null hypothesis that the acrylic antagonist would be equally affected and thus worn by each opposing material sample, was rejected.
The ceramic materials, eMax™, Lava Plus™, and the nano-ceramic composite resin Lava Ultimate™ showed no significant change in volume from baseline levels. Statistical significance testing was undertaken regarding the differences in sample material wear (volume loss) when opposing the acrylic antagonist material when compared to the Acrylic opposing Acrylic group. The p-value for significance testing was set at p<0.05. The results of the testing are shown in Table 19 and the data revealed the following:

- All of the material sample groups, eMax™, Lava Ultimate™, and Lava Plus™ had p-values <0.05.
- The null hypothesis that the material specimens would be as equally affected as the acrylic specimen in the acrylic versus acrylic group was rejected.

The results for the overall combined material volume loss for the material specimen and the corresponding acrylic antagonist showed the following:

- The highest overall material volume loss that occurred (when both the acrylic antagonist material and the material samples were combined) was found for the acrylic-Lava Ultimate™ group.
- This was followed closely by the acrylic-acrylic group.
- The Lava Plus™ and eMax™ groups had the least amount of combined material wear (volume loss).

Therefore, the ceramic materials, Lava Plus™ and eMax™ had the most favourable interaction with the acrylic antagonist material in terms of the sample materials ability to wear the opposing acrylic antagonist material.
Conclusions
6.1 Surface texture analysis

6.1.1 Baseline/Pre-Wear testing

- Qualitative assessment of the material sample specimens indicated that the DenZir™ material demonstrated a higher luster and little surface texture variation.
- Two-Dimensional SEM imaging reviewed all materials have surface inclusions and despite efforts to clean the surfaces, surface contaminants remained.
- The surface texture analysis revealed that the BruxZir™ monolithic zirconia material specimen had the lowest baseline surface texture values for Ra and Sa analysis and was thus the smoothest material.
- The Sa method of dental material surface texture analysis displays a higher sensitivity to surface texture variation when compared to the simpler and less complex Ra method.
- Both the Ra and Sa methods resulted in the same ranking of the tested materials in terms of roughness or surface texture even though they gave different values.
- The R-squared analysis revealed a value of 0.8858 which indicates a strong relationship between the Ra and Sa values in terms of measuring dental material surface texture.
- The Sa method is more complex, time consuming and expensive than the Ra method.

6.1.2 Post two-body in-vitro wear testing

- The acrylic antagonist materials showed a significant reduction in surface texture following wear testing.
- The sample material specimens didn’t show a significant reduction in surface texture following wear testing opposing acrylic antagonist material.
6.2 Volumetric changes of acrylic antagonist specimens and material sample specimens following

- The ceramic materials, eMax™, Lava Plus™, and the nano-ceramic composite resin Lava Ultimate™ showed no significant change in volume from baseline levels.
- The material volume loss of the acrylic test sample material showed statistically significant changes when compared to the ceramic and resin nano-ceramic material specimens.
- The acrylic antagonist had significantly higher volume loss when opposing the resin nano-ceramic composite material compared to the other tested materials.
- The use of an acrylic tooth cusp form in in-vitro two-body dental material wear analysis allows for simple preparation and good quality control of the antagonist material.
Section 7

References


75. Ivoclar R D Vivadent AG, Schaan, Liechtenstein


Appendices
### 8.1 Appendix 1 – Literature review table summary of in-vitro wear studies on artificial denture teeth

<table>
<thead>
<tr>
<th>Author</th>
<th>Denture tooth materials Tested</th>
<th>Antagonist material and type</th>
<th>Wear Machine</th>
<th>Load (N)</th>
<th>Cycle number</th>
<th>Lubricant</th>
<th>Wear measurement method</th>
<th>Findings Summary</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hao et al 2014</td>
<td>Seven: 1. Cross-linked PMMA with PMMA pearls; 2. MPM-PMMA with polymerised PMMA; 3. 10% IPN &amp; 90% HXL polymers; 4. Composite resin; 5. PFMA with particle fillers; 6. PMMA; 7. PMMA</td>
<td>12mm diameter silicone nitride ceramic ball</td>
<td>Abrasive tester; Pin-on-Block design (TE**; Phoenix Tribology Ltd)</td>
<td>49</td>
<td>50,000 cycles</td>
<td>Thermocycling 5-55º water @ 2min cycles</td>
<td>Height and volume via laser scanning microscope</td>
<td>Composite resin and highly cross-linked teeth showed better wear resistance versus the conventional PMMA teeth</td>
</tr>
<tr>
<td>Ghazal et al 2010</td>
<td>Four: 1. Feldspathic ceramic; 2. Acrylic (UDMA/PMM A); 3. Composite resin; 4. IPN-PMMA</td>
<td>Human enamel</td>
<td>Dual-axis chewing simulator (willytec)</td>
<td>49</td>
<td>300,000</td>
<td>Thermodyclining 5-55º water @ 2min cycles</td>
<td>Laser scanning profilometry, SEM. Microscopy</td>
<td>Acrylic teeth showed less wear resistance versus composite resin teeth and ceramic teeth. Composite resin teeth showed more wear versus ceramic teeth. Acrylic shows fatigue type wear and composite resin shows attritional wear with loss of filler particles.</td>
</tr>
<tr>
<td>Shetty et al 2010</td>
<td>Three: 1. Surana Ultradent; 2. Premadent; 3. Dentek.</td>
<td>600 Grit silicone carbide paper</td>
<td>Ducom friction wear machine (custom)</td>
<td>2</td>
<td>10,000</td>
<td>Nil</td>
<td>Volume change</td>
<td>Surana Ultradent acrylic denture teeth showed the least wear</td>
</tr>
<tr>
<td><strong>Mello et al 2009</strong></td>
<td>Seven in total: 1. High-density Cross-linked PMMA; 2. PMMA, EDMA; 3. Cross-linked PMMA; 4. PMMA, EDMA, Pigments; 5. Synthetic polymer-based on PMMA, double cross-linked; 6. Synthetic polymer based on PMMA (Gnathostar); 7. Microfilmed composite resin, UDMA.</td>
<td>Two: 1. Ni-Cr Alloy (Vera Bond 2, Aalba Dent Inc, Cordelia, CA, USA); 2. Indirect Composite Resin “Solidex” (Shofu Inc. Tokyo, Japan)</td>
<td>“Customised”</td>
<td>5</td>
<td>40,000 @ 265 cycles/min (equivalent to 120 days)</td>
<td>Distilled water at 37°C</td>
<td>Nikon 6C profile projector measuring height change.</td>
<td>1. Most samples (inc Gnathostar) showed no significant difference in wear rate between metal &amp; composite resin antagonists. 2. Antagonists showed no measurable wear</td>
</tr>
<tr>
<td>Study</td>
<td>Type of Teeth/Resin</td>
<td>Treatment Details</td>
<td>Duration</td>
<td>Temperature</td>
<td>Methodology</td>
<td>Findings</td>
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<tr>
<td>Hahnel et al 2009</td>
<td>Six types of teeth: 1. PMMA (Gnathostar); 2. UDMA; 3. DCL-PMMA; 4. IPN-PMMA; 5. Composite resin; 6. PMMA with fillers</td>
<td>Three: 1. Steatite (soap stone); 2. Steel Balls; 3. Artificial resin teeth (PMMA)</td>
<td>EGO Pin-on-block design</td>
<td>120,000@ 1.2Hz, 1mm horizontal movement</td>
<td>Distilled water, thermocycled 5º &amp; 55º</td>
<td>Volume via optical 3D surface profilometer (willytec). Surface analysis via SEM</td>
<td>Steatite showed higher wear rates. Ceramic antagonist resulted in larger “wear tracks” versus artificial teeth. Composite resin was more wear resistant than other materials versus ceramic but no significant difference was noted for other antagonists.</td>
<td></td>
</tr>
<tr>
<td>Ghazal et al 2009</td>
<td>Two: 1. Human enamel; 2. Composite resin</td>
<td>Zirconia ceramic balls 6mm diameter in three groups: 1. As supplied; 2. Air abraded with 50µm alumina particles at 0.5bar; 3. ABA at 1bar.</td>
<td>Dual-axis chewing simulator (willytec)</td>
<td>300,000</td>
<td>Thermocycling 5-55º water @ 2min cycles</td>
<td>Ra, Laser scanning profilometry of PVS-epoxy resin copies via SEM</td>
<td>Increased surface roughness increased the friction coefficient which in turn resulted in increased wear.</td>
<td></td>
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<tr>
<td>Ghazal et al 2008</td>
<td>Three: 1. Human Teeth; 2. Composite resin; 3. Feldspathic ceramic</td>
<td>Steatite 6mm balls, Zirconia (inceram), alumina (inceram AL)</td>
<td>Dual-axis chewing simulator (willytec)</td>
<td>49</td>
<td>600,000 cycles</td>
<td>Thermocycling 5-55º water @ 2min cycles</td>
<td>Vertical height loss and volume loss. Custom profilometer with 5µm stylus with 0.02µm SEM surface analysis</td>
<td>Zirconia and alumina showed less wear than nano-filled composite resin but weren’t significant.</td>
</tr>
<tr>
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</tr>
<tr>
<td>Ghazal et al 2008</td>
<td>Seven: 1. Feldspathic ceramic; 2. Composite resin; 3. IPN-PMMA; 4. PMMA; 5. TCR resin; 6. DCL resin; 7. PMMA U-derivative. Polished with 4000 grit abrasive paper. Human premolar as control</td>
<td>Steatite 6mm balls</td>
<td>Dual-axis chewing simulator (willytec)</td>
<td>49</td>
<td>1,200,00 at 1.3Hz, 0.3mm horizontal slide</td>
<td>Thermocycling 5-55º water @ 2min cycles</td>
<td>Vertical height loss and volume loss. Custom profilometer with 5µm stylus with 0.02µm SEM surface analysis</td>
<td>CR teeth had more wear than enamel. The wear resistance of all PMMA teeth was less than composite resin. Feldspathic ceramic showed similar wear to enamel.</td>
</tr>
<tr>
<td>Ghazal et al 2008</td>
<td>Three types: 1. Feldspathic ceramic; 2. Nano-filled composite resin; 3. UDMA/PMMA</td>
<td>Matched the same materials for control and human enamel</td>
<td>Dual-axis chewing simulator (willytec)</td>
<td>49</td>
<td>200,000 cycles (two body, then three-body of 100,000 each)</td>
<td>Distilled water at 21° then a “food bolus material” (PMMA powder + distilled water)</td>
<td>Vertical height loss and volume loss. Custom profilometer with 5µm stylus with 0.02µm SEM surface analysis</td>
<td>CR-CR and feldspathic - feldspathic showed similar wear and both were significantly less than acrylic-acrylic.</td>
</tr>
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</tr>
<tr>
<td>Reis et al 2008</td>
<td>Three: 1. Cross-linked PMMA; 2. Highly cross-linked IPN-PMMA; 3. Highly cross-linked PMMA</td>
<td>Two: 1. Duceram glazed and polished; 2. Duceram glazed and air abraded</td>
<td>“customised”</td>
<td>3</td>
<td>100,000 @ 4.5 cycles/min for 20mm</td>
<td>Distilled water at 37°C</td>
<td>Height loss</td>
<td>All teeth showed more wear against the air abraded ceramic antagonist. Highly cross linked teeth showed less wear for the abraded group only.</td>
</tr>
<tr>
<td><strong>Stober et al 2006</strong></td>
<td>Ten materials of: 1. PMMA; 2. MPM-PMMA; 3. DCL-PMMA; 4. IPN-PMMA; 5. INPEN-PMMA; 6. Isostit (UDMA + Inorganic fillers); 7. PMMA + fillers; 8. Nano-composite resin; 9. Human enamel control</td>
<td>$\text{Al}_2\text{O}_3$ balls of 4.8mm diameter</td>
<td>Two-axis chewing simulator (willytec)</td>
<td>40</td>
<td>100,000 cycles @ 8mm/sec</td>
<td>Distilled water at room temperature</td>
<td>Volume, 3-D measurements with optical 3D surface profilometer “laserscan 3D” (willytec) with matched software</td>
<td>Enamel was highly variable. No significant difference between different denture tooth types compared to enamel.</td>
</tr>
<tr>
<td><strong>Zeng 2005</strong></td>
<td>Composite resin with various fillers</td>
<td>The same material as tested</td>
<td>Customised wear testing machine (Tsurumi University)</td>
<td>50</td>
<td>50,000, 60 strokes/min, 2mm distance</td>
<td>Water at 37°C</td>
<td>Height and volume via weight calculations</td>
<td>The composite resin teeth with more fillers showed less wear</td>
</tr>
<tr>
<td><strong>Suwannaroop 2001</strong></td>
<td>Five denture teeth: 1. PMMA; 2. UDMA; 3. IPN-PMMA; 4. FM-inorganic filler CR; 5. Porcelain</td>
<td>Aluminium oxide 3mm diameter balls (Ra 2.28µm)</td>
<td>Pin-on-disc</td>
<td>15</td>
<td>10,000 cycles at 1,000rpm rotating/spinning</td>
<td>Distilled water at room temperature</td>
<td>Weight and volume loss with contact stylus profilometer and matched software</td>
<td>Highly-cross-linked PMMA teeth had the greatest surface loss. The volume losses of ceramic teeth, composite teeth, and acrylic teeth weren’t significantly different.</td>
</tr>
<tr>
<td><strong>Hirano et al 1998</strong></td>
<td>Two materials; 1. PMMA; 2. Composite resin denture teeth</td>
<td>“Enamel abraders”</td>
<td>Customised developed by Wagner and Hacker 1996</td>
<td>13.4</td>
<td>10,000</td>
<td>Human Saliva</td>
<td>Volume. Surfanalyser system 400 Profilometer</td>
<td>PMMA showed more wear than the composite resin teeth</td>
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### 8.2 Appendix 2 – Complete set of baseline $Ra$ and $Sa$ values for all examined materials

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<th>$Ra$ (nm/μm)</th>
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<td>Mean 457.24</td>
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<td>Mean 627.04</td>
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<td>Mean 148.23</td>
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</table>
8.3 Appendix 3 – Post two-body wear testing material loss (wear) in mm$^3$ for each two-body wear testing group (material sample and acrylic antagonist). Acrylic Antagonist (AA), Acrylic sample material (A), Lava Plus™ (LP), Lithium Disilicate (eMax™), and Lava Ultimate™ nanoceramic composite resin (LU).

Table – Acrylic Antagonist (AA) opposing Acrylic sample material (A). AA volume change.

<table>
<thead>
<tr>
<th>AA - A</th>
<th>Volume change (mm$^3$)</th>
<th>Post Wear Volume (means)</th>
<th>Mean volume loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA Values</td>
<td>Pre Wear Volume (means)</td>
<td>Post Wear Volume (means)</td>
<td>Mean volume loss</td>
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<td>Test group 1</td>
<td>87.212</td>
<td>86.245</td>
<td>0.9672</td>
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<td>109.51</td>
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<tr>
<td>Test group 3</td>
<td>83.197</td>
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<td>Test group 4</td>
<td>94.103</td>
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<td>Test group 5</td>
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<td>Test group 6</td>
<td>95.978</td>
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<td>Test group 7</td>
<td>50.427</td>
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<td>91.0337</td>
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Table – Acrylic Antagonist (AA) opposing Acrylic sample material (A). A volume change.

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<th>AA - A</th>
<th>Volume change</th>
<th>Post Wear Volume (means)</th>
<th>Mean volume loss</th>
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<tr>
<td>A Sample Values</td>
<td>Pre Wear Volume (means)</td>
<td>Post Wear Volume (means)</td>
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<td>61.317</td>
<td>60.452</td>
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<td>96.892</td>
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<td>71.308</td>
<td>70.172</td>
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<td>110.11</td>
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Table – Acrylic Antagonist (AA) opposing Lava Ultimate sample material (LU). AA volume change.

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<td>AA Values</td>
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Table – Acrylic Antagonist (AA) opposing Lava Ultimate sample material (LU). LU volume change.

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</table>
Table – Acrylic Antagonist (AA) opposing eMax. AA volume change.

<table>
<thead>
<tr>
<th>AA values</th>
<th>Pre Wear Volume (means)</th>
<th>Post Wear Volume (means)</th>
<th>Mean volume loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test group 1</td>
<td>100.558</td>
<td>99.739</td>
<td>0.8192</td>
</tr>
<tr>
<td>Test group 2</td>
<td>111.904</td>
<td>111.144</td>
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<tr>
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<td>0.6309</td>
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<td>87.992</td>
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<td>0.5285</td>
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<td>94.365</td>
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Table – Acrylic Antagonist (AA) opposing eMax. eMax volume change.

<table>
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<tr>
<th>eMax values</th>
<th>Pre Wear Volume (means)</th>
<th>Post Wear Volume (means)</th>
<th>Mean volume loss</th>
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<td>Test group 1</td>
<td>84.497</td>
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**Table – Acrylic Antagonist (AA) opposing Lava Plus (LP). AA volume change.**

<table>
<thead>
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<th>AA values</th>
<th>Pre Wear Volume (means)</th>
<th>Post Wear Volume (means)</th>
<th>Mean volume loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test group 1</td>
<td>87.409</td>
<td>86.913</td>
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<td>Test group 2</td>
<td>93.268</td>
<td>92.833</td>
<td>0.4347</td>
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<td>135.79</td>
<td>135.492</td>
<td>0.2985</td>
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<td>115.432</td>
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<td>0.2645</td>
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<td>101.305</td>
<td>100.751</td>
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**Table – Acrylic Antagonist (AA) opposing Lava Plus (LP). LP volume change.**

<table>
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<th>AA - LP</th>
<th>Volume change</th>
<th>Volume change</th>
<th>Mean volume loss</th>
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</tr>
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<td>26.8</td>
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