

INTERACTION BETWEEN ENAMEL, PORCELAIN AND A GOLD ALLOY: AN IN VITRO WEAR STUDY



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PREFACE

This thesis reports on research work that was carried out during my PhD candidature at the School of Dentistry, The University of Adelaide, from August 2006 to 2010. The work initially aimed to investigate wear between enamel and different indirect materials including four porcelain systems (a porcelain bonded to metal veneering system, a leucite-reinforced glass ceramic used for veneering, a leucite-reinforced pressable ceramic, a machinable ceramic) and a type III gold alloy under various pH conditions. However, an interesting finding from qualitative analysis of the machinable ceramic has led to a more detailed examination of this system. In addition, over the years for the project to be accomplished, zirconia has become more popular, therefore a preliminary study on the wear behaviour of this relatively new material has been conducted to make the thesis more complete.

This thesis consists of seven sections, starting with a review of the literature, leading to the aims and rationale of the study (Section 1). The next three sections (Section 2, 3 and 4) present on the studies of enamel/ceramic wear, effect of acid on machinable ceramic and preliminary study of zirconia wear, respectively, that have been carried out. Each of this section composes of four chapters including an introduction, materials and methods, results and discussion for that specific study. These were followed by a section of general conclusions (Section 5), references (Section 6) and appendices (Section 7).

ABSTRACT

In dental practice, wear of the natural dentition is commonly seen in patients of all ages. It can have a mild effect on teeth, or be severe enough to affect patients' quality of life. Although different indirect restorative materials such as gold alloy or porcelain have been used for many years to restore excessively worn teeth, the procedures are generally complex and challenging to the dentists as well as costly and time-consuming for the patients.

A good restorative material should be aesthetic, durable and not be abrasive to the opposing dentition. Gold has been reported to be "enamel-friendly", but the colour makes it un-aesthetic. On the contrary, porcelain is aesthetic, biocompatible, durable and has become a popular choice for both clinicians and patients. However, previous studies have shown that some of the porcelain systems can be abrasive to the opposing natural enamel. The use of such abrasive porcelain systems would therefore be harmful to a patient's dentition in the long term.

Four porcelain systems and a gold alloy have been selected for this study:

- a veneering porcelain normally used in porcelain bonded to metal restorations (PBM-veneering porcelain)
- a leucite-reinforced glass ceramic used for veneering (LR-veneering ceramic)

- a leucite-reinforced pressable ceramic (LR-pressable ceramic)
- a machinable ceramic
- a type III gold alloy (gold).

The aims of the study were to determine the wear rates of the selected porcelains and opposing enamel under controlled conditions which simulated two clinical conditions:

- heavy attrition at near neutral pH (pH 6.1)
- heavy attrition with gastric regurgitation (pH 1.2)

In addition, preliminary studies on the wear of zirconia and enamel were conducted.

In this study, electro-mechanical tooth wear machines were used to simulate wear. Wear volume loss was measured by scanning specimens with 3D profilometers and evaluating the data using a purpose-written software. The surface micromorphology of wear facets was also observed by scanning electron microscopy (SEM). As a result of this analysis a more detailed investigation of the machinable ceramic was undertaken.

The results revealed that at pH 6.1, while enamel wear caused by the PBM-veneering porcelain, LR-veneering ceramic, machinable ceramic and gold alloy were not significantly different to the control group in which enamel specimens were worn against each other, significantly increased enamel wear was associated with the LR-pressable. Although enamel wear rates increased dramatically in conditions simulating attrition combined with gastric regurgitation, the gold alloy did not wear the opposing

enamel more than the enamel controls. In addition, in this study the machinable ceramic became porous under acidic conditions.

The findings presented in this thesis have implications for selection of porcelain for specific clinical cases. Although the findings should be cautiously extrapolated to in vivo conditions, they contribute to the understanding of new porcelain materials in terms of wear and erosion. In addition, results from preliminary experiments with zirconia will provide data to inform the development of protocols for future research.

DECLARATION

This work contains no material which has been accepted for the award of any other degree or diploma in any other university or other tertiary institution to Uyen Tran Kieu Ha 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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UYEN TRAN KIEU HA

Dated this.....day of2011

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*I wish to dedicate this thesis to my parents, Manh-Thu Ha and Trung-Moc Tran, for
continued support and encouragement of their three daughters*

SECTION ONE

INTRODUCTION

CHAPTER 1

TOOTH WEAR

1.1 Historical background

Tooth wear research is not a new area of investigation. Anthropologists have described the pattern and the extent of worn dentitions in both contemporary and, in particular, pre-contemporary human populations for over 200 years. It is generally accepted that tooth wear was the result of diet and the use of teeth as tools (Molnar 1972) while the differences in the wear patterns observed is correlated to cultural, environmental and gender differences within and between populations. Wear of the dentitions of prehistoric humans was heavy enough to flatten the occlusal and interproximal surfaces (Molnar 1972). Such surfaces were thought to enhance the masticatory efficiency as they eliminated the interlocking of cusps, allowing increased lateral movement of the mandible (Kaifu et al. 2003). According to Saitou (1987) the main cause of tooth wear was “a combination of friction of exogenous material forced over tooth surfaces and an increase in the number of power strokes during mastication when less refined, tougher foods are consumed”. Considerable wear was also experienced by more modern hunter-gatherer populations such as indigenous Aboriginal Australians and Eskimo, and ancestors of the present-day populations, whose diet was mainly coarse food. In

addition, anthropologists supported the view that occlusal and interproximal wear was closely associated with growth and development (Begg 1954) and the dentition maintained its functionality as the wear proceeded (Barrett 1969). Tooth wear is also associated with some direct consequences such as mesial drift, continuous eruption, incisal lingual tipping, and the forward shifting of the mandibular teeth. However, with the development of the industrialized society and the mastication of softer and more processed food, wear has decreased (Kaifu et al. 2003).

Palaentological research on the worn dentitions of different species supported the anthropological evidence, indicating tooth wear to be a common and hence physiological phenomenon. However, it was also reported that excessive wear could lead to pathology in different parts of the stomatognathic system, in particular the temporomandibular joints (Richards 1990).

Interestingly, early anthropologists used the terms abrasion, attrition and erosion interchangeably describing the same mechanism, that is, the friction or abrasiveness of foreign material (eg. food) forced onto the tooth surface.

In contrast to the anthropological concepts based on form and function from an evolutionary perspective, dentists most often considered tooth wear as pathology relating to tooth grinding and occlusal discrepancies. Dental focus was on repairing and rehabilitating broken-down dentitions in an environment where caries and periodontal disease was at plague proportions. However, in recent times dental opinion is changing towards the anthropological model. The words attrition, abrasion and erosion are now specific dental terms referring to dental mechanisms that will be described later.

Currently, improvements in oral health in contemporary populations has led to people keeping their teeth longer and therefore restoring worn dentitions is a common dental requirement.

1.2 Aetiology, mechanisms and prevalence of tooth wear

1.2.1 Aetiology and mechanisms of tooth wear

Tooth wear is a multifactorial process (Smith and Knight 1984; Smith et al. 1997; Al-Omiri et al. 2006). In the past, the terms attrition, abrasion and erosion have been used interchangeably to denote general tooth wear, but in recent times these terms have been more clearly defined to describe the different wear mechanisms.

Attrition is the wear that results from tooth to tooth contact without the presence of food (Pindborg 1970; Lambrechts et al. 1984; Smith 1989; Kaidonis 2008). The manifestations of active attrition are the well-defined, shiny wear facets that match with facets in the opposing arch (Kaidonis 2008). Cusp tips, incisal edges, occlusal or palatal surfaces, and even the interproximal contact points could be involved in attrition (Lambrechts et al. 1984; Mair 1990;1992). At an advanced stage, attrition may cause dentinal exposure (Litonjua et al. 2003); however, the dentinal surface remains flat with no “cupping” or “scooping” (Kaidonis 2008). The microwear detail found within wear facets reveals parallel striations typically within the facet border (Fig. 1.1). Attrition can be physiologic or pathologic, depending on the extent of wear relative to the age of the patient. Dental opinion in the past considered tooth grinding as a pathological habit that

was triggered by occlusal interferences, while others consider this to relate to a sleep disorder. However, opinion is slowly changing to the extent that some consider tooth grinding to be a physiological behaviour observed in many different species (Every 1972). In addition, it is acknowledged the behaviour is related to stress (Kaidonis et al. 2003).

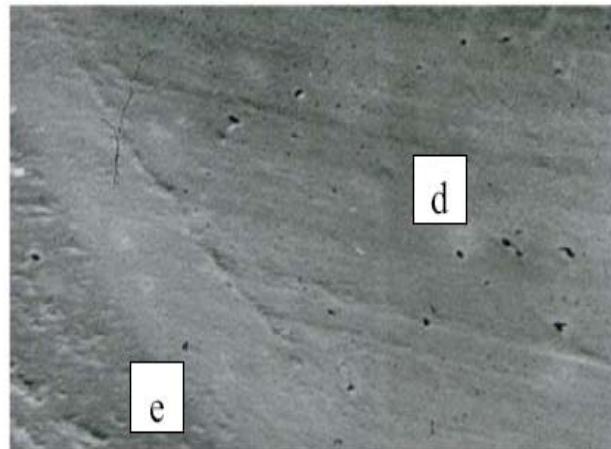


Figure 1. 1: Microwear detail of a facet showing parallel striations. The dentine (d) is not scooped out and is at the same level as the enamel (e) (adapted from Kaidonis 2008).

Some researchers believe the most common cause of attrition is associated with parafunctional activity (Kelleher and Bishop 1997; Abrahamsen 2005). Xhonga (1977) showed that the amount of annual enamel loss in bruxers was three or four times greater than that in non bruxers. Moreover, the higher bite force in males possibly contributed in part to their higher prevalence and severity of tooth wear (Dahl et al. 1993; Bernhardt et al. 2004). However, this was not supported by a study in which it was showed that the muscle activity was not necessarily associated with an increased maximal bite force

(Dahl et al. 1985) and there was no difference in maximal bite force between people with pathologic attrition and people with healthy dentitions.

In addition, the number of occlusal contacts has also been shown to be associated with the amount of wear. The decrease in the number of occluding contacts was proportional to the increase of attrition (Bernhardt et al. 2004). However, this concept has not been supported by Poynter who found no relationship between missing posterior teeth and incisal tooth wear (Poynter and Wright 1990). They suggested that teeth could not be overloaded due to a protective feedback mechanism from the periodontal ligament noniceptors.

Abrasion is caused by the friction of exogenous materials forced over tooth surfaces. For example a coarse diet produces abrasive wear, more frequently observed in pre-contemporary populations in comparison with contemporary populations, who consume more processed and refined food (Mair 1999; Kaidonis 2008). Abrasive food causes wear over the whole occlusal surface, producing a wear area that is distinct from the wear facet resulting from attrition (Kaidonis 2008). Abrasion may also result from habitual or occupational activities such as smoking pipes or using teeth as tools (eg. finger biting) and in professional people such as carpenters or tailors who might hold nails or bobby pins between their teeth for convenience (Hattab and Yassin 2000; Kaidonis 2008). Generally, abrasion resulting from food produces surfaces that appear pitted and gouged and when the dentine is exposed, it becomes scooped out (Fig. 1.2). Micrographs of such abraded surfaces show haphazard scratch marks (Fig. 1.3). It has

also been shown that abrasion is linearly associated with age (Richards and Brown 1981) and the rate observed reflects the diet consumed.

Some researchers prefer the term **demastication** in the situation where the abrasive food particles wear away the tooth substance; however, this is not commonly used in dentistry (Litonjua et al. 2003).

It has been reported that the most common lesions in modern human populations are found at cervical tooth surfaces, often called non-carious cervical lesions (NCCL); however, their aetiology is controversial. Some believe that “wedge-shaped” NCCLs are related to tooth brush abrasion (Litonjua et al. 2003) where the extent depends on technique, duration and frequency of brushing, bristle design and the abrasiveness of dentifrices (Hattab and Yassin 2000). Other researchers refer to this process as **abfraction** which is hypothetically resulted from tensile stress that concentrates in the cervical areas when the tooth is flexed by non-axial loading (Addy and Shellis 2006). According to this hypothesis, abfraction propagates microcracks between hydroxyapatite crystals in enamel and dentine, causing tooth tissue loss. However, a recent study by Nguyen et al. (2008) found evidence of horizontal furrows caused by toothbrush abrasion and evidence of erosion on NCCLs in extracted teeth (Figs. 1.4 and 1.5), which indicated that the common association of abrasion and erosion rather than the flexing of teeth play a significant role in the formation of wedge-shaped NCCLs. Though the “abfraction” hypothesis has not been supported by the above data, still one cannot discount that abfraction may play a role in NCCLs.



Figure 1. 2: 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 (adapted from Kaidonis 2008).

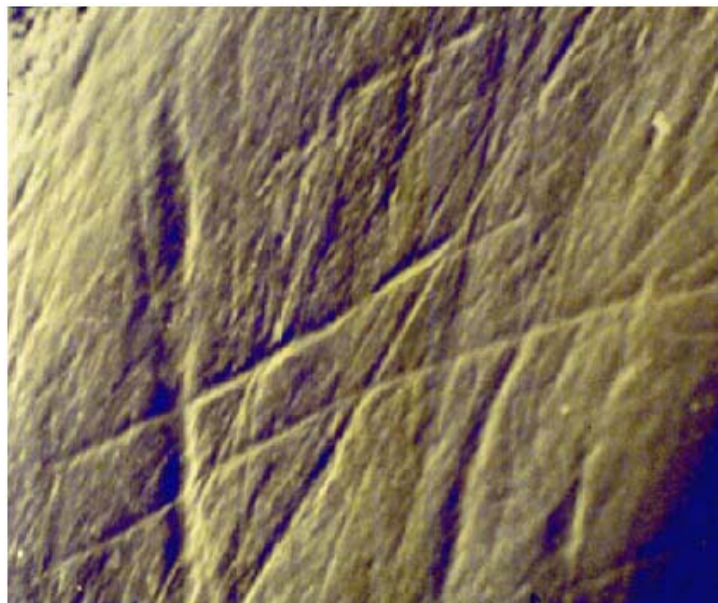


Figure 1. 3: Microwear detail of an abrasion area showing haphazard scratch marks (adapted from Kaidonis 2008).

The term dental **erosion** describes the process of gradual destruction of a surface by the attack of acids which are not produced by bacteria (Pindborg 1970). This is a chemical process although superimposed mechanical factors can contribute to the removal of acid-softened surfaces (Smith 1989).

Dental erosion is different from caries. While the former involves degradation of the surface, the later starts as subsurface demineralization of the tooth structure (Litonjua et al. 2003). At an early stage, the erosion lesion has a smooth, glazed unstained appearance. Dentinal scooping is commonly seen with some degree of sensitivity because dentinal tubules remain patent (Kaidonis 2008). Loss of enamel makes the incisal edges and proximal surfaces of anterior teeth become more translucent and the teeth appearing darker because of the translucency of the underlying dentine (Smales and Kaidonis 2006). At high magnification, lesions appear smooth and clean (Fig. 1.6)



Figure 1. 4: Variation in the appearance of NCCLs (adapted from Nguyen et al. 2008).

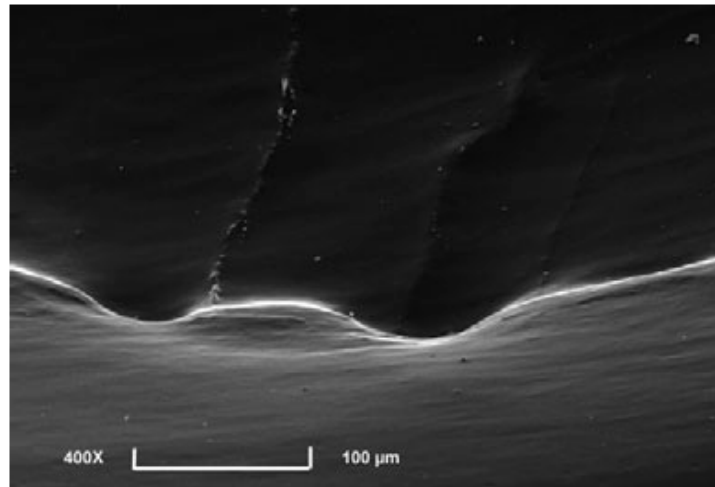


Figure 1. 5: A micrograph showing the evidence of abrasion and erosion occurring concurrently in an NCCL (x4000 magnification). The wavy line at the centre of the micrograph represents the upper margin of the NCCL. Faint horizontal scratch marks below the line are indicative of abrasion over an erosive background (adapted from Nguyen et al. 2008).

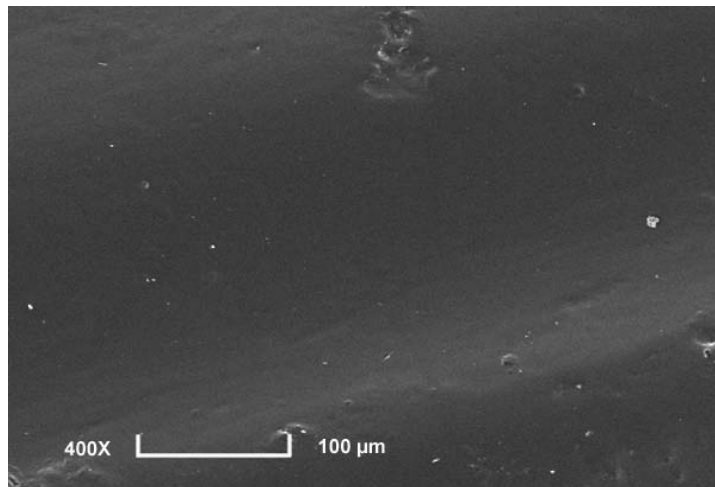


Figure 1. 6: Micrograph of erosion lesion (courtesy of Dr S. Ranjitkar). Note the lack of mechanical wear.

In addition to these commonly used dental terms, Dahl et al. (1993) and Litonjua et al. (2003) suggested the term perimylolysis or perimolysis to describe wear on palatal

surfaces of the maxillary teeth resulting from a combination of the low pH of gastric reflux and the hyperactivity of the tongue. This describes a mechano-chemical effect on the teeth, which is the effect of acid and the mechanical activity of the tongue.

Diets containing acidic foods and drinks play an important role in tooth wear (Dahl et al. 1993). Various types of acids such as citric acid, phosphoric acid or ascorbic acid in soft drinks and sports drinks can damage the teeth if they are consumed in sufficient quantities (Meyers 2008). Acids not only chelate and dissolve the mineral content of the tooth but also soften the tooth surface rendering it more susceptible to the harmful effects of attrition and abrasion.

Involuntary and voluntary regurgitation may occur in people with gastro-intestinal or psychological problems (Dahl et al. 1993; Kelleher and Bishop 1997). Gastric reflux typically causes erosion lesions on the palatal surfaces of upper incisors (Kelleher and Bishop 1997; Abrahamsen 2005; Zero and Lussi 2005). Also patients with eating disorders such as anorexia, bulimia nervosa and rumination commonly present with erosive lesions.

Saliva plays an important role in mineral loss and gain (Zero and Lussi 2005; Meyers 2008). It has a cleansing and buffering action and neutralizes potentially harmful acid (Dahl et al. 1993; Zero and Lussi 2005; Meyers 2008). In addition, it acts as a reservoir

of mineral ions such as calcium, phosphate and possibly fluoride that are needed for the remineralization process.

The main cause of environmental erosion is the presence of acidic material in the work place with the erosion lesion mainly localized to the labial surfaces of the maxillary and mandibular incisors (Dahl et al. 1993; Milosevic 1993; Kelleher and Bishop 1997). The severity of erosion increases when the acid concentration is higher and the exposure time longer. Some clinical studies have provided information on the prevalence of erosion in wine tasters and competitive swimmers (Wiegand and Attin 2007). Working in dusty environments (eg. quarries, mines etc) can be associated with high rates of tooth wear (Dahl et al. 1993). In today's modern culture, abrasion resulting from processed, softer food is less extensive; nevertheless the combined effects of erosion and attrition are common. Therefore the term "erosive wear" is often used to describe a mechano-chemical effect.

1.2.2 Prevalence of tooth wear

A number of studies have been conducted on the prevalence of tooth wear in contemporary populations (Al-Omiri et al. 2006), with a predominance of data obtained in children and adolescents, compared to that on adults (Van't Spijker et al. 2009). However, it is difficult to quantify the overall prevalence of tooth wear due to different methodologies and in particular the many different indices used to measure wear (Al-Omiri et al. 2006; Bardsley 2008; Meyers 2008; Van't Spijker et al. 2009). As a

consequence, there is no clear figure on the prevalence of wear in the literature (Shaw 1997; Kaifu et al. 2003).

Various indices have been used as epidemiological research tools to assess tooth wear in clinical and laboratory settings (Bardsley 2008). Perhaps the most popular one was the tooth wear index (TWI) system proposed by Smith and Knight (Smith and Knight 1984; Van't Spijker et al. 2009). This index was designed to measure and monitor multifactorial tooth wear. However, over time, the system has been modified and many other indices have been proposed and used (Dahl et al. 1989; Bardsley 2008). The lack of standardization has made it difficult to compare the reported prevalence of tooth wear between populations.

Furthermore, many of the prevalence studies conducted in the past measured general tooth wear without focusing on the different wear mechanisms involved. Kaidonis et al. (1993) found a high frequency of faceting in an Australian Aboriginal population, in which more than 90% of both anterior and posterior teeth presented with wear facets. In 2005, Casanova-Rosado and co-workers conducted a cross-sectional study on the prevalence and severity of attrition in 390 Mexican adolescents in the age group of 14 to 19 years old. The result showed that 33.3% of the study population had attrition and the severity was related to age, the presence of defective fillings and Class II malocclusion (Casanova-Rosado et al. 2005). However, it has generally been recognized that erosion is the primary wear process and its prevalence increases with age (Young 2001; Kaifu et

al. 2003; Bernhardt et al. 2004; Bardsley 2008; Johansson et al. 2008; Van't Spijker et al. 2009).

Adding to this, a study in 2006 found erosion to be the most prevalent form of wear affecting 84% of subjects; however, the cause of tooth wear was difficult to identify because of the involvement of other wear mechanisms. Interestingly, the wear process in general was also found to be gender-related, with the number of affected male subjects nearly double that of females. This was attributed to different lifestyles between sexes (Al-Omiri et al. 2006).

Another study carried out on a sample of 463 kindergarten children in Germany (age group 2 to 7 years old) found that 32% of the subjects had erosion and this increased with age (Wiegand et al. 2006).

A survey of 1002 individuals aged 45 years old or more in England showed that wear increased with age on cervical and occlusal/incisal tooth surfaces (Donachie and Walls 1995). It was also observed that more wear was seen in males than females, which is in agreement with the study by Al-Orimi et al. (2006); however, there was no variation between subjects of different social class backgrounds.

Wear was found to have certain impacts on daily living (Al-Omiri et al. 2006). The authors' study aimed to identify the effects of tooth wear on patients' quality of life and

satisfaction with their dentition. Thirty six percent of tooth wear patients who had been referred to a tooth wear clinic were not satisfied with their teeth and appearance compared with 3.9% of the control group, which composed of subjects with tooth wear that was considered within normal limits for their particular age group. In addition, wear, to a certain extent, also compromised chewing and eating ability, caused pain and oral discomfort.

To assist the clinicians in the diagnosis and treatment of tooth wear, a specific method has been proposed (Richards et al. 2003). The mathematical models developed enabled the clinician to predict the severity of tooth wear at different future ages based on the established relationships between age and tooth wear scores. It also helped to assess whether the wear rate of a patient was low, moderate or high so that the clinician could intervene with treatment when necessary.

1.3 Methods of assessment of tooth wear

There are a number of qualitative and quantitative methods to assess tooth wear in vivo and in vitro (Azzopardi et al. 2000). In vivo tooth wear can be assessed by comparing “longitudinal” changes that are visible on series of models or photographs or by using tooth wear indices. The most widely used index system is the tooth wear index (TWI) introduced by Smith and Knight in 1984. However, index systems are more suitable for epidemiology studies rather than for an individual assessment and they cannot detect minute amounts of wear (Azzopardi et al. 2000).

Quantitative methods include:

- chemical methods
- microradiography
- digital image analysis, and
- profilometry and surface mapping.

These are mainly used for in vitro and in situ investigations (Azzopardi et al. 2000).

Erosion studies often use chemical methods to measure the degree of demineralization. Here, the concentration of calcium and phosphate in the solution is quantified after apatite dissolution in acid (Azzopardi et al. 2000; Attin 2006). The technique is sensitive and accurate and provides information on the concentration of ions released (Barbour and Rees 2004).

Similarly, microradiography allows the volume of mineral loss and lesion depth to be assessed based on the attenuation of X-rays by dental hard tissues (Azzopardi et al. 2000; Barbour and Rees 2004; Attin 2006). By using photo-counting X-ray detectors, X-ray sensitive photographic plates or film, the penetrating radiation is recorded and the mineral density of the enamel can be mapped.

With digital image analysis, images generated by computers can be compared to measure erosive or abrasive lesions. However, there exists a potential for errors with the method (Azzopardi et al. 2000).

Contacting and non-contacting profilometry are also used for the measurement of wear in dentistry. A mechanical or laser sensor traces the surface to record two or three dimensional coordinates (Mehl et al. 1997; Azzopardi et al. 2000; DeLong 2006). Contacting profilometers involve the use of a mechanical stylus with diameters of 0.1mm or larger and loaded with a force in the milliNewton range (Attin 2006; DeLong 2006). Non-contacting profilometers use laser or light of different colours. The advantages of the laser profilometers include the fact that they do not contact the surface and the scanning time is much shorter than systems using contacting sensors (Azzopardi et al. 2000; DeLong 2006; Heintze et al. 2006). However, laser systems require an opaque, diffuse reflecting surface and the laser stylus may produce “overshots” at the sharp edges, resulting in artifacts (Attin 2006; DeLong 2006). Although the contacting profilometer produces more accurate results and is not affected by differences in surface material properties such as colour or transparency, the stylus may damage surfaces, especially if they are demineralized (Attin 2006; DeLong 2006). If the orientation of the specimens can be exactly reproduced, tooth wear measurements can be determined by a profilometer with a precision of 2.2 μ m and an accuracy of 10 μ m (Mehl et al. 1997). However, the depth of undercuts could not be measured with profilometry techniques, so the positioning of the specimen is critical (Azzopardi et al. 2000). This has been improved with the use of a purpose-written software that has an option to re-align reference planes in order to eliminate the orientation issues (Liu et al. 2004).

Other methods have also been used in wear assessment. Hardness measurements can provide information about enamel erosive lesions because the erosive process weakens

and softens the enamel surface (Barbour and Rees 2004; Attin 2006). Commonly used measurement methods are microindentation and nanoindentation or ultra-microindentation. While microindentation gives the results in Knoop hardness number (KHN) or Vickers hardness number (VHN), nanoindentation results can be read in the SI unit of Pascals (Nm^{-2}). An advantage of nanoindentation is that it can measure enamel erosion lesions at an earlier stage because the measurement can detect a lesion that is as small as 200nm (Finke et al. 2001). Also, nanoindentation can explore both the plastic and elastic deformation for the surface while microindentation investigates only plastic deformation (Barbour and Rees 2004; Attin 2006). However, microindentation is less expensive and the process is faster and simpler. Both techniques require the specimens to be polished flat before subjected to experiment.

Scanning electron microscopy (SEM) can produce qualitative and quantitative results. It allows visualization of wear patterns in high resolution images (Attin 2006). SEM observation requires the specimens to be coated and it is performed under vacuum. With some materials, this may induce crack propagation that can be mistaken as resulting from the wear process. This problem can be overcome by “environmental SEM”, a technique in which the uncoated samples are placed in a pressurized container (Field et al. 2010). SEM is an expensive technique and requires training. However, the technique is reproducible and the tooth surface reproduction is good.

The atomic force microscope is one example of a scanning probe microscope. It has been used successfully in enamel erosion investigations (Kasas et al. 1993) and gives an

accurate quantitative assessment though it is time-consuming (Barbour and Rees 2004). One advantage is that it does not require the specimens to be hydrated, coated or vacuumed, therefore avoiding artifacts and damage due to sample preparation.

Secondary ion mass spectroscopy “is a form of mass spectroscopy in which a beam of ions is incident on a surface, causing the ejection of secondary ions which are spectroscopically analyzed” (Barbour and Rees 2004). The technique is extremely sensitive and is used as an effective tool to analyze major and trace elements in dental hard tissues (Lodding 1997).

In summary, methods for the assessment of tooth wear range from simple descriptive and imaging techniques for describing macro- and microscopic wear to complex elemental analysis. Each of the methods has different applications and provides different insights into tooth wear processes.

CHAPTER 2

TRIBOLOGY

The word tribology is derived from the Greek word “tribos” which means rubbing. It is defined as “the science and technology of interacting surfaces in relative motion and of related subjects and practices” (Halling 1975). More recently, Mair (1992) defined it as the “study of friction, lubrication and wear”.

A fundamental principle in understanding wear is that there is no perfectly smooth surface (Kragelskii 1965). Under magnification, surfaces are wavy and rough. The length of the waves varies from 1,000-10,000 μm and their heights range from a few to 20-40 microns. For example, the height of the irregularities on the surface of a cleavage of mica can be 20 \AA in size or 0.05 – 0.1 μm on the smoothest metallic surfaces. As a consequence, the contacting opposed surfaces are actually point to point contacts between the asperities (Mair 1992). The number of the contact points increases when the two surfaces come closer to each other under an applied load. In addition, different contact spots are formed during sliding at successive intervals of time (Kragelskii 1965).

The interaction of two solid surfaces within a given environment results in two manifestations (Halling 1975):

- *“(1) There is an 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 a small, but sometimes significant, amount of noise.*

- *(2) 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 so-called process.”*

A natural consequence of the interaction of the relative motion of two surfaces is the wear process (Halling 1975) involving a variety of factors that are difficult to quantify.

There is no perfect definition of wear. A committee of the Institution of Mechanical Engineers (Halling 1975) defined wear as: “the progressive loss of substance from the surface of a body brought about by mechanical action”. According to Kragelskii (1965), wear is “the destruction of material produced as a result of repeated disturbances of the frictional bonds”.

Nevertheless, the definitions of wear mechanisms used by tribologists are different to those used in dentistry, and have been classified by Pugh (1973) as abrasive wear, adhesive wear, fatigue wear, corrosive wear, erosive wear and fretting wear.

2.1 Abrasive wear

Abrasive wear is the most common type of wear (Mair 1992). It was described as “the cutting away of a surface by abrasive asperities or particles” and it occurs when the asperities plough into the softer surface (Mair 1992; Mair et al. 1996) (Fig. 2.1).

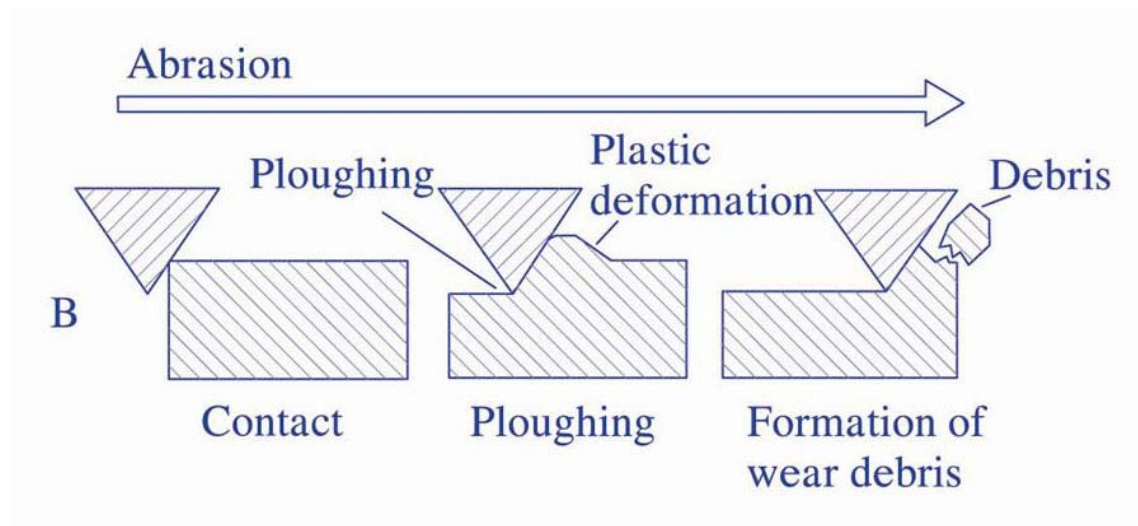


Figure 2. 1: Abrasive wear (adapted from Mair et al. 1996).

Abrasive wear includes two-body and three-body wear. **Two-body wear** can occur when there are two opposing surfaces wearing against each other. (Mair 1992;1999) (Fig. 2.2).

NOTE:
This figure is included on page 23 of the print copy of
the thesis held in the University of Adelaide Library.

Figure 2. 2: Two-body abrasive wear (adapted from Mair 1999).

Three-body wear occurs when two opposing surfaces wear against each other in the presence of third body abrasive particles at the wear interface (Mair 1992; Mair et al. 1996). The wear process usually starts as a two-body wear process but eventually becomes a three-body one (Fig. 2.3).

There are a number of factors that can influence abrasive wear:

- the hardness of the materials in contact
- the geometry of the abrasive particles, and
- the load and the sliding distance (Halling 1975).

Collectively, the variables involved and the dynamic nature of these processes make mathematical modelling (Appendix 1) difficult and beyond the scope of this project.

NOTE:
This figure is included on page 24 of the print copy of
the thesis held in the University of Adelaide Library.

Figure 2. 3: Three-body abrasive wear (adapted from Mair 1999).

2.2 Adhesive wear

Adhesive wear occurs when the friction between the moving surfaces causes cold welding of the protuberances of the contacting surfaces (Halling 1975; Zum-Gahr 1987; Mair 1992; Mair et al. 1996). When the surfaces continue moving, the welds are sheared. If shearing occurs at a subsurface level, then there is a deposition of the detached material onto the opposing surface. With further rubbing, some of the detached material forms loose third body particles contributing to three-body abrasion (Fig. 2.4).

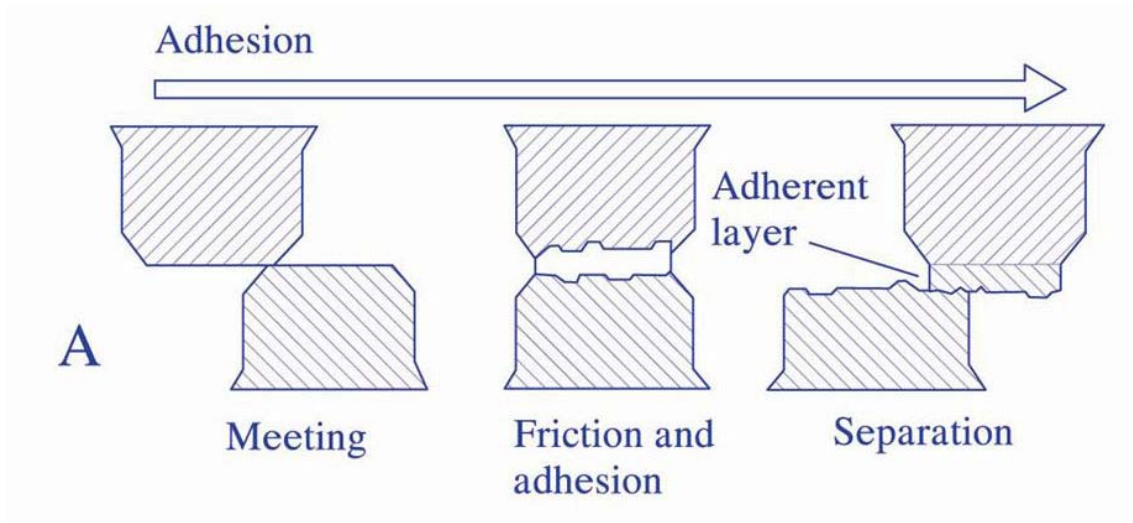


Figure 2. 4: Adhesive wear (adapted from Mair et al. 1996).

2.3 Fatigue wear

Fatigue wear occurs when two surfaces move under dynamic loads resulting in the formation and propagation of subsurface microcracks. Cyclic loading of surface layers with repetitive compressive, tangential and tensile stresses causes subsurface cracks to grow and propagate to the surface with subsequent loss of material (Mair 1992;1999) (Fig 2.5).

The theory of delamination was first introduced to fatigue wear in 1973 (Suh 1973). It was assumed that the wear process involved surface dislocation followed by the formation of sub-surface cracks and voids, which would eventually propagate to the surface (Suh 1973;1977).

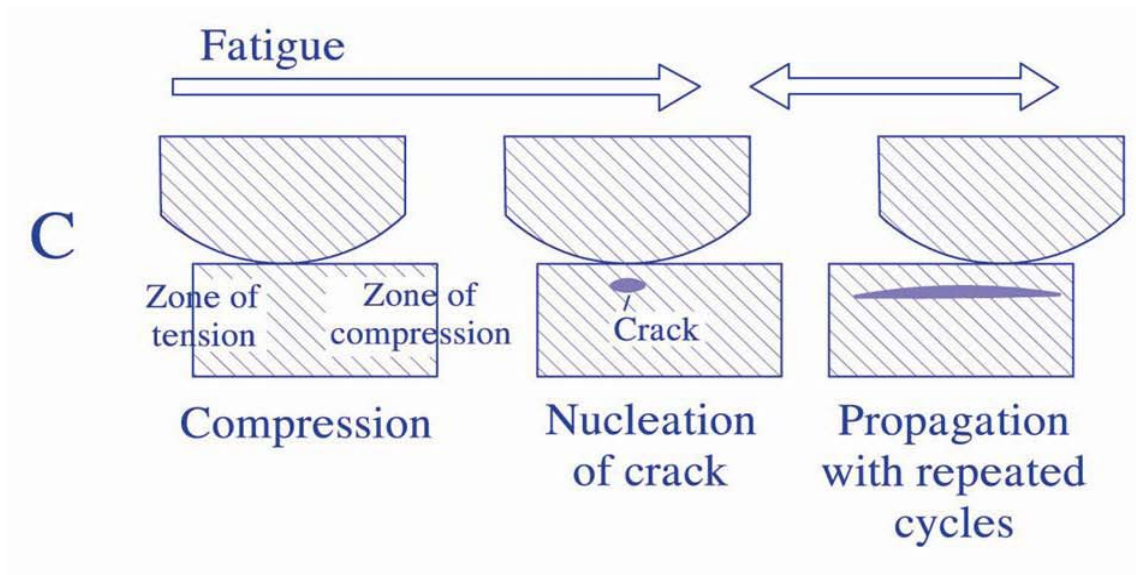


Figure 2. 5: Fatigue wear (adapted from Mair et al. 1996).

2.4 Corrosive wear

Corrosive wear, also termed “tribochemical wear”, refers to the wear process involving chemical degradation of a surface that is then rubbed away by the opposing surface (Halling 1975; Zum-Gahr 1987; Mair 1992) (Fig. 2.6). It is different from “corrosion” which relates to static chemical degradation of the surface (Zum-Gahr 1987).

2.5 Erosive wear

Erosive wear was described by Pugh (1973) as the damage occurring in a solid body by the impact of external particles or fluid under pressure. In this situation, the particles or

fluid act as the second surface. For example, the erosion of sand and rocks can be caused by wind and waves.

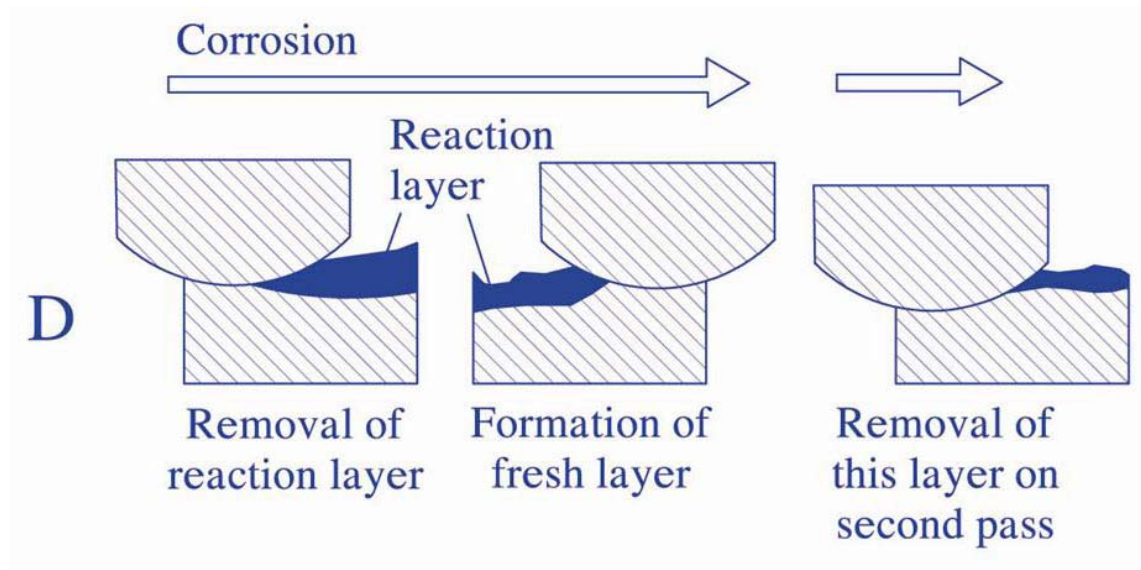


Figure 2. 6: Corrosive wear (adapted from Mair et al. 1996).

2.6 Fretting wear

Fretting wear results from “the low amplitude vibratory motion that takes place between two surfaces when they are loaded together” (Halling 1975). However, there is no evidence that this occurs in the mouth (Mair 1992).

The terminology derived by tribologists based on their detailed knowledge of wear on a micro- and nano-scale provides an important context for interpreting and understanding the terminology generally used by dentists which is based on macro-level clinical

observations. In this project the dental terminology has generally been used other than when the alternative, more specific terminology allows a clearer explanation of an observation or process.

CHAPTER 3

CERAMICS

3.1 Historical background

The art of producing fine translucent porcelain was developed in Europe in the 18th century (Kingery and Vandiver 1986). The first person who made porcelain paste for use in dentistry for denture work was a French apothecary, Alexis Duchâteau, with the assistance of a Parisian dentist, Dubois de Chemant in 1774 (Jones 1985; van Noort 1994; Craig 2006). This was to replace ivory dentures, which absorbed oral fluids, stained badly, were porous and therefore highly unhygienic (van Noort 1994). De Chemant's continuing work on improving porcelain formulations was granted a patent initially in France. However, this encountered opposition from a Parisian dentist who accused him of stealing Duchâteau's idea, and as a consequence, de Chemant had to emigrate to England (Craig 2006). In Britain his work was again patented and he continued to collaborate with the well known Wedgwood porcelain factory to manufacture porcelain paste for dentures (Jones 1985; Craig 2006).

In 1808 the first single porcelain crown was introduced in Paris by an Italian dentist, Guiseppangelo Fonzi, but the aesthetics were suboptimal. The dental profession did not master the art of ceramics until the end of the 19th century (Craig 2006). Over the years, advances in ceramics have been made and many new porcelain materials have appeared on the market with continuous improvement in strength and optical qualities (van Noort 1994).

3.2 Definitions

The term ceramics originates from the Greek word “keramos”, which means “burnt material” (Giordano 1996; Rosenblum and Schulman 1997). These are specified, non-metallic and inorganic products. They are solid objects which are processed by baking raw materials at high temperature to achieve the desirable properties (Rosenblum and Schulman 1997; Craig 2006).

Porcelain is a ceramic that has a specific composition (Rosenblum and Schulman 1997; Craig 2006) made from a mixture of kaolin (pure white clay), quartz, and feldspar ($K_2O \cdot Al_2O_3 \cdot 6SiO_2$). These three natural minerals are blended and fired at high temperature to form what is called “white-ware” and porcelain is a type of “white-ware” with a relatively high strength and translucency (Rosenblum and Schulman 1997; Craig 2006).

3.3 Structures of dental ceramics

Dental porcelain comprises of a basic silicon-oxygen network with a glass-forming matrix (McLean 1979), which is a large three-dimensional network of silica tetrahedra, connected by oxygen atoms (oxygen bridging Si-O-Si) (Oh et al. 2002) (Figs 3.1, 3.2 and 3.3).

Other oxides such as potassium, sodium, calcium, aluminum and boric oxides are incorporated to give additional properties such as low-fusing temperature, high viscosity, and resistance to detrieviation (McLean 1979). In addition, metal oxides are also added to provide porcelain with various colours, and a binder consisting of starch and sugar, may be added for easier manipulation of the powder (van Noort 1994).

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This figure is included on page 31 of the print copy of
the thesis held in the University of Adelaide Library.

Figure 3. 1: Diagram of a silicate unit with each SiO tetrahedra sharing an oxygen atom (adapted from McLean 1979).

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This figure is included on page 32 of the print copy of the thesis held in the University of Adelaide Library.

Figure 3. 2: Three dimensional drawing of a silicate unit in which the silicon atom Si is surrounded by four oxygen atoms (adapted from McLean 1979).

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This figure is included on page 32 of the print copy of the thesis held in the University of Adelaide Library.

Figure 3. 3: Three dimensional drawing of linked silicate units which form the continuous network in glass (adapted from McLean 1979).

3.4 Composition of ceramics

There are two main phases in the composition of ceramics after baking: the crystalline phase (leucite, and/or other alumino-silicate crystals) and a glass phase. The amount of

crystal and glass varies in different types of porcelain, while the proportion of leucite and the time of heat treatment are affected by the K_2O content (McLean 1979).

The main raw ingredient of the classical porcelains for ceramic-metal crowns is feldspar, a potassium aluminum silicate ($K_2O \cdot Al_2O_3 \cdot 6SiO_2$). At high temperature (about $1150^{\circ}C$), feldspar melts to form leucite ($KAlSi_2O_6$ or $K_2O \cdot Al_2O_3 \cdot 4SiO_2$) with a tetragonal structure and molten glass with an amorphous structure (Craig 2006).

Each phase of the ceramics contributes to the optical and mechanical properties of the material. The glassy phase gives porcelain properties of glass such as brittleness, a non-directional fracture pattern and translucency. The leucite crystalline phase not only elevates porcelain strength, hardness and resistance to cracking but also affects the optical properties. In addition, the high thermal expansion of leucite helps to control the thermal expansion coefficient of the porcelain, depending on the amount present (10-20%) (Rosenblum and Schulman 1997; Craig 2006).

Finally, although very strong, dental porcelain has two major shortcomings: it is brittle and can fail catastrophically, and also has the potential to cause wear of opposing teeth, which is a major concern in clinical dentistry (Giordano 1996; Rosenblum and Schulman 1997).

3.5 Properties of ceramics

Dental porcelain is chemically stable and does not deteriorate with time. Its thermal conductivity and coefficient of thermal expansion are similar to those of enamel and dentine. Furthermore, ceramics have high compressive strength and low tensile strength (van Noort 1994). Representative physical properties of dental porcelain in reference to enamel are presented in Table 3.1 (Craig 2006).

Table 3. 1: Selected physical properties of dental porcelain and enamel.

	Feldspathic porcelain	Enamel
Tensile strength (MPa)	25	10
Compressive strength (MPa)	149	384
Elastic Modulus (GPa)	69-70	84
Fracture toughness (K_{IC})	0.9-1.0	0.7-1.3

3.6 Classification of dental ceramics

Ceramic materials can be classified by either fusion temperature or major applications in dentistry. Some authors categorized them based on composition and fabrication; however, this will not be presented here.

Since the early 1940s ceramics have been classified into three groups based on fusion temperature (Craig 2006):

- High-fusing ceramics: 1315⁰C to 1370⁰C.
- Medium-fusing: 1090⁰ C to 1260⁰ C
- Low-fusing: 870⁰ C to 1065⁰ C.

However, ultra low-fusing dental ceramics with firing temperature below 870⁰C have been recently introduced, giving ceramics a wide range of applications.

Ceramics have three major applications in dentistry (Craig 2006):

- Ceramo-metal crowns and fixed partial dentures
- All-ceramic crowns, inlays, onlays and veneers, when aesthetics is a priority, and
- Ceramic denture teeth.

3.6.1 Metal-ceramic restorations

These ceramics contain conventional feldspathic porcelain and are composed primarily of:

- SiO₂ (silica, 64%)
- Al₂O₃ (alumina, 18%), and
- various amounts of K₂O (potash) and Na₂O (soda) (Giordano 1996).

The leucite content determines the thermal and mechanical behaviour of the porcelain. In particular, it increases the coefficient of thermal expansion of the porcelain to match that of the metal copings. This helps to avoid internal stress, a common cause for cracking and failure. However, leucite is an unstable phase. Repeated firing, slow cooling and extended heat soaks can affect the leucite content and mechanical properties of porcelain (Kelly et al. 1996).

3.6.2 All-ceramic restorations

The all-ceramic restorations permit light transmission and allow for an optimal aesthetic result (van Dijken 1999).

The materials used to fabricate this type of restoration can be classified into the following categories:

- glass ceramics
- alumina-based ceramics, and
- zirconia-based ceramics.

The non-metallic ceramic restorations are often composed of strong ceramic copings veneered with aesthetic veneering porcelains, which typically consist of glass and crystalline phase of fluoroapatite, alumina oxide, or leucite (Conrad et al. 2007).

Glass ceramics were first developed by Corning Glass Works in the late 1950s (van Noort 1994) and since then new systems have come into existence. Currently a commonly used glass ceramic indicated for single anterior and posterior restorations is Authentic™ (Ceramay GmbH+Co.KG, Stuttgart, Germany) which is a leucite-reinforced glass ceramic. The material comes in the form of ingots or layering powder.

Alternative glass ceramic is IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein), a lithium disilicate glass ceramic ($\text{SiO}_2\text{-Li}_2\text{O}$) which is veneered with fluoroapatite-based veneering porcelain (IPS Eris, Ivoclar Vivadent, Schaan, Liechtenstein). It has sufficient strength for the construction of three-unit bridges in the anterior segment. In 2005, the same company launched IPS e.max Press, which is also a lithium disilicate porcelain but with improved physical properties and translucency (Giordano 1996; van Dijken 1999; Stappert et al. 2006).

Vita Mark II (VITA Zahnfabrik, Bad Säckingen, Germany) is a machinable feldspathic porcelain which came onto the market in 1991. It has fine grain size ($4\mu\text{m}$) and primarily contains a mixture of 60-64% SiO_2 and 20-23% Al_2O_3 . The advantage of this porcelain is that it can be etched by hydrofluoric acid to create micromechanical retention for resin cement cementation (van Dijken 1999; Conrad et al. 2007).

The first all-ceramic system In-Ceram Alumina (VITA Zahnfabrik, Bad Säckingen, Germany), an **alumina-based ceramic**, was introduced in 1989. It is an opaque core, fabricated through the slip-casting technique and veneered with feldspathic porcelain

(Giordano 1996; Bindl and Mormann 2002). In 1994, In-Ceram Spinell (VITA Zahnfabrik, Bad Säckingen, Germany) was launched, with a specific crystalline structure referred to as “spinell” (magnesium aluminate- $MgAl_2O_4$) in the composition. The spinell core has a better translucency but reduced flexural strength, allowing it to be used directly or to be veneered with a feldspathic porcelain (Giordano 1996; Bindl and Mormann 2002). The In-Ceram Alumina was later modified with the addition of 35% zirconium oxide to strengthen the core, forming In-Ceram Zirconia (VITA Zahnfabrik, Bad Säckingen, Germany). However, this core is also opaque and lacks translucency (Giordano 1996).

Another alumina-based product that has been more commonly used lately is the Procera All-ceramic System (Procera-Snadvik, Stockholm, Sweden). Copings produced from this system composed of a very high purity aluminium oxide (>99.9%) (van Dijken 1999; McLean 2001), giving the porcelain optimal strength.

The three types of **zirconia-containing ceramic** systems commonly used in dentistry in recent times are yttrium cation-doped tetragonal zirconia polycrystals (3Y-TZP), magnesium cation-doped partially stabilized zirconia (Mg-PSZ) and zirconia-toughened alumina (ZTA) (Denry and Kelly 2008). 3Y-TZP has the highest flexural strength and fracture toughness of the three in the range of 800-1000 MPa and 6-8 $MPa\ m^{0.5}$, respectively. These mechanical properties exceed those of other available ceramic systems.

Materials in this category are composed primarily of zirconium oxide and a small percentage of Yttrium oxide (Luthardt et al. 1999) and have a structural form that changes with changing temperature during manufacture (Denry and Kelly 2008). Between room temperature and 1170⁰C, the structure exists in the monoclinic phase, that transforms to a tetragonal phase when the temperature increases above 1170⁰C. A cubic phase forms at temperatures between 2370⁰C and the melting point. Upon cooling, the tetragonal phase reverses to the monoclinic phase with an associated increase in volume of about 3 to 5%, creating an induced compression stress onto the surface (Luthardt et al. 1999; Denry and Kelly 2008), that significantly slows down crack propagation and leads to high fracture toughness (Kosmac et al. 1999; Denry and Kelly 2008). However, the heterogeneity of the structural phase adversely affects the surface integrity and compromises strength (Denry and Kelly 2008). To overcome this, stabilizing oxides including CaO, MgO, Y₂O₃ or CeO₂ were added to retain the tetragonal phase at room temperature and to control volume change (Denry and Kelly 2008). However, surface treatment such as grinding or sandblasting can trigger the phase transformation from tetragonal to monoclinic, which could be harmful to the long-term performance of the material (Denry and Kelly 2008).

3.7 Wear studies of ceramics

Both in vivo and in vitro wear studies have been conducted on wear between porcelain and enamel by a number of authors with the majority of them occurring in the laboratory under controlled conditions. It is very difficult to simulate the complex nature of the

oral environment, therefore in vitro studies are needed to investigate individual factors that cause tooth wear. However, in vitro findings should be extrapolated to clinical situations with caution (Ghazal et al. 2008).

There were only a few previous in vivo studies of wear rates of enamel and porcelain, largely because clinical studies are time-consuming and difficult to conduct (Ghazal et al. 2008). One study evaluated the wear of IPS Empress ceramic (Ivoclar-Vivadent, Licheinstein) inlays, onlays, enamel and luting cement over eight years of clinical service (Kramer et al. 2006). It was found that the opposing enamel wore significantly faster than ceramic inlays regardless of the location of inlays and antagonists in the oral cavity (Kramer et al. 2006).

Another in vivo study evaluated the wear of enamel opposing a new heat-pressed core ceramic IPS e.max Press (Ivoclar Vivadent) over a period of one year (Esquivel-Upshaw et al. 2006). This core ceramic was made of lithium disilicate with high fracture toughness. The result showed that the mean occlusal wear rate of enamel was 88.4 μ m for premolars and 88.3 μ m for molars, with a range of 29 to 255 μ m (Esquivel-Upshaw et al. 2006). This wear rate was faster than the reported annual normal enamel wear rate of 18 μ m for premolars and 38 μ m for molars (Lambrechts et al. 1989).

The most recent in vivo study assessing the wear of enamel opposing metal (Simidur S2, Panadent, a high noble ceramo-metal alloy composing gold, platinum, palladium and

silver used for metal-ceramic crown), a metal ceramic material (IPS Classic, Ivoclar Vivadent), an experimental hot-pressed ceramic with lithium disilicate crystals (Ivoclar Vivadent) and Procera Allceram (Nobel Biocare) was reported in 2008 (Etman et al. 2008). It was found that the Procera AllCeram produced more wear on the opposing enamel and the material itself was worn down more than other selected materials over a period of 24 months.

In vitro studies of opposing porcelain and enamel wear have been conducted using wear simulators under controlled conditions for loads, number of cycles and the lubricating medium. However, there is no global consensus regarding the design of tooth wear machines and as a result different wear simulators have been used in different studies. (Table 3.2).

The loads applied in previous wear studies have been reported in either grams, kilograms or Newtons, and ranged broadly from as small as 180g or 1.8N to 100N (Shabaniyan and Richards 2002; Elmaria et al. 2006; Ranjitkar et al. 2008). A range of loads that have been used in studies of wear of porcelain are presented in Table 3.3.

The number of cycles used in wear studies has varied considerably with an upper limit of 300,000 cycles (Magne et al. 1999), which is equivalent to 62.5 hours at a rate of 80 cycles per minute. This also indicates that such studies are time consuming to conduct in the laboratory. The range of cycles used in various studies are as follows (Table 3.4)

Table 3. 2: Some of the wear machines used in previous porcelain wear studies.

Name of wear machine	Studies
Pin on disc	(Fisher et al. 1983) (Palmer et al. 1991) (O'Kray and O'Brien 2005), (Metzler et al. 1999)
The University of Alabama	(Kadokawa et al. 2006)
The Oregon Health Sciences University	(Clelland et al. 2001;2003)
The Willytec	(Ghazal et al. 2008)
The Leinfelder	(Alarcon et al. 2009) (Ramp et al. 1999)
Closed-loop servohydraulics	(Magne et al. 1999)
Custom-made	(al-Hiyasat et al. 1998b)

Table 3. 3: Some of the loads (in increasing order) used in previous porcelain wear studies.

Load	Studies
0.75N	(Kadokawa et al. 2006)
	(Ramp et al. 1997; 1999)
4.5N	(O'Kray and O'Brien 2005)
4.536N (1 pound)	(Hacker et al. 1996)
6N	(Metzler et al. 1999)
10N	(Abe et al. 2001)
13.5N	(Magne et al. 1999)
	(Palmer et al. 1991)
20N for the cycles and direct contact with a static load of 70N at the end	(Clelland et al. 2003)
30N	(Fisher et al. 1983)
40N	(al-Hiyasat et al. 1998b)
49N	(Ghazal et al. 2008)
76 to 80N	(Alarcon et al. 2009)

Table 3. 4: Some of the cycles (in increasing order) used in previous porcelain wear studies.

Number of cycles	Studies
10,000 cycles	(Fisher et al. 1983) (Elmaria et al. 2006) (O'Kray and O'Brien 2005) (Ramp et al. 1997; 1999)
25,000 cycles	(al-Hiyasat et al. 1998b)
43,200 cycles	(Monasky and Taylor 1971) (Hacker et al. 1996)
100,000 cycles	(Kadokawa et al. 2006) (Ghazal et al. 2008)
172,800 cycles	(Metzler et al. 1999)
250,000 cycles	(Alarcon et al. 2009)

The most popular lubricating medium used in studies on wear rates of porcelain and enamel has been distilled water (Fisher et al. 1983; Palmer et al. 1991; Ramp et al. 1997; al-Hiyasat et al. 1998b; Magne et al. 1999; Metzler et al. 1999; Ramp et al. 1999; Ghazal et al. 2008). Some researchers have used artificial saliva (Monasky and Taylor 1971; O'Kray and O'Brien 2005) or tap water (Elmaria et al. 2006; Kadokawa et al. 2006). One study reported utilized fresh, natural saliva as a lubricant (Hacker et al. 1996). This has also been used in a previous wear study of enamel (Kaidonis et al. 1998). All these approaches have their advantages and disadvantages. While tap water and distilled water are a convenient approach, they really do not mimic the oral environment.

Artificial saliva does not mimic real saliva and also adds another variable, that of remineralization, which potentially changes the results. Natural saliva, on the other hand, although seemingly a good approach, denatures within minutes and makes the results of long experiments skewed.

Most of the wear studies between enamel and porcelain have been carried out in neutral or near neutral pH and only two papers by the same group of authors were found to address the wear in lower pH (al-Hiyasat et al. 1997; al-Hiyasat et al. 1998a). Their experiments investigated wear of three types of porcelain (Vitadur Alpha (Vita Zahnfabrik, Germany), Duceram-LFC (Ducera Dental GmbH, Germany) and Vita Mark II (Vita Zahnfabrik, Germany)) against enamel in Coca-Cola (pH in the range of 2.28 to 2.37). It was found that exposure to the carbonated beverage increased the amount of enamel wear by 19% against Alpha porcelain, 13% against Vita Mark II and 74% against Duceram-LFC compared with enamel wear produced in water.

Most of the wear machines described above simulate the dental behaviour of tooth grinding and very few studies relating to wear during mastication have been reported. A number of studies used a slurry of artificial materials as a third particle (three-body wear studies) to simulate the food bolus during the chewing cycle. In one study a poly methyl methacrylate (PMMA) slurry was used between a high-fusing porcelain (Ceramco II, Densply, USA) and composite resin (Premise, Kerr, USA), type IV gold alloy (Argenco 56, Argen, USA) and enamel (Kadokawa et al. 2006). Non-plasticized PMMA powders with a mean particle size of 40µm for wear testing between ceramic and enamel was also

used in the study by Ghazal et al. (2008). However, other slurries consist of totally different materials, such as a mixture of glass pearls, aluminum oxide powder (105 μ m) and water (Derand and Vereby 1999) or a mixture of cornmeal grit and wholemeal flour in distilled water (al-Hiyasat et al. 1999).

Another difficulty confronting wear studies was the methods used to quantify wear. The different approaches have been:

- height loss (Fisher et al. 1983; Ramp et al. 1997; al-Hiyasat et al. 1998b; Ramp et al. 1999; O'Kray and O'Brien 2005; Elmaria et al. 2006; Esquivel-Upshaw et al. 2006; Kadokawa et al. 2006; Etman et al. 2008; Ghazal et al. 2008), and
- volume loss (Palmer et al. 1991; Metzler et al. 1999; Ghazal et al. 2008; Alarcon et al. 2009).
- Some other authors assessed weight loss (Monasky and Taylor 1971) or wear area (Clelland et al. 2003).

Measurements of height or volume loss were performed with either a stereoscopic microscope with a micrometer-calibrated movable platform (Palmer et al. 1991) or a Reflex microscope (al-Hiyasat et al. 1998b). However, the most popular technique used in recent studies was by scanning with a profilometer (Ramp et al. 1997; Metzler et al. 1999; Ramp et al. 1999; Clelland et al. 2003; O'Kray and O'Brien 2005; Elmaria et al. 2006; Esquivel-Upshaw et al. 2006; Kadokawa et al. 2006; Etman et al. 2008; Ghazal et al. 2008; Alarcon et al. 2009).

Finally, the range of materials tested for their wear characteristics can be briefly grouped as follows:

- Conventional porcelain (Seghi et al. 1991; DeLong et al. 1992; Hacker et al. 1996; Magne et al. 1999; Imai et al. 2000; O'Kray and O'Brien 2005; Kadokawa et al. 2006)
- Low-fusing dental porcelain (Metzler et al. 1999; Imai et al. 2000; Clelland et al. 2003; O'Kray and O'Brien 2005)
- Pressable ceramic (Imai et al. 2000; Elmaria et al. 2006)
- Veneering porcelain for aluminum oxide cores (Hacker et al. 1996; Elmaria et al. 2006)
- Machinable ceramic (Seghi et al. 1991; DeLong et al. 1992; al-Hiyasat et al. 1999; Ramp et al. 1999)

It is difficult to compare the results between in vitro wear studies due to different wear conditions and tested materials. Nevertheless important conclusions have been generated. First, porcelain has a potential to induce wear to the opposing enamel, with effects correlated more with the surface roughness than with the material hardness (Monasky and Taylor 1971; Seghi et al. 1991; Elmaria et al. 2006). Furthermore, low-fusing temperature porcelain was not less abrasive than the traditional feldspathic porcelain (Clelland et al. 2003), and porcelain denture teeth would cause more vertical loss to the opposing enamel compared to those made from acrylic resin or nano-filled

composite (Ghazal et al. 2008). Interestingly, the wear in a two-body condition is significantly higher than that in a three-body environment for both porcelain and opposing materials (gold, composite resin and enamel) (Kadokawa et al. 2006). Last but not least, though various liquids have been used in wear studies as the lubricating medium, effects of the extreme pH have hardly been studied.

CHAPTER 4

AIMS AND RATIONALE OF THE STUDY

Although many studies have been carried out on the wear of enamel and porcelain, they did not cover all available systems and did not test the materials at low pH that can occur intraorally. Further research is required on newer materials to provide an insight into their mechanical and clinical performance.

To date our knowledge and past research confirms that dental wear in current populations occurs predominantly as a result of tooth grinding and mastication, although in comparison to pre-contemporary (in particular hunter/gatherer) populations, wear is much less significant because of our relatively refined, processed diets. However, current populations are exposed to the “modern day” condition of erosion, where acid from the diet (in particular drinks) and intrinsic causes (ie. acids from the gastro intestinal tract) cause damage to teeth by demineralization. In particular, intrinsic acids reach the mouth during sleep which is also a time when people grind their teeth.

The aims of this study were to compare the wear between a number of currently used ceramic materials and tooth enamel at both a “neutral” and low pH. The low pH (=1.2) was selected to mimic acid reflux and also to provide insight on the wear of ceramic materials and enamel under extreme acid conditions.

SECTION TWO

ENAMEL/CERAMIC

WEAR

CHAPTER 5

INTRODUCTION

On the basis of the information reviewed in Section One, a series of in vitro experiments on wear between enamel and indirect restorative materials were planned as part of this research project. Although in vitro conditions can never simulate completely the complicated oral environment, they offer some advantages such as being inexpensive and not requiring a follow up on patients over an extended period of time.

Four different porcelain systems and a type III gold alloy were selected for the experiments. They range from a system used for veneering metal substructure for crowns and bridges, to those for all ceramic crowns and for a computer aided design/computer aided manufacture (CAD/CAM) system. Enamel specimens wearing against each other acted as the control groups.

The experiments were conducted under a selected load at two different pHs. The wear results were determined both quantitatively and qualitatively.

CHAPTER 6

MATERIALS AND METHODS

6.1 Description of tooth wear machine

The two electro-mechanical wear machines available in the O.F Makinson Laboratory at the School of Dentistry were used for the wear experiments. These machines were designed and fabricated by the Department of Mechanical Engineering at the University of Adelaide and have been used for studies on wear of enamel, dentine and restorative materials over the last 15 years (Kaidonis et al. 1998; Burak et al. 1999; Shabanian and Richards 2002; Ranjitkar et al. 2008).

The wear machine (Fig. 6.1) was described by Kaidonis et al. (1998) in the following way:

“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. A magnetic counter, attached to the resultant 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 specimen 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”.

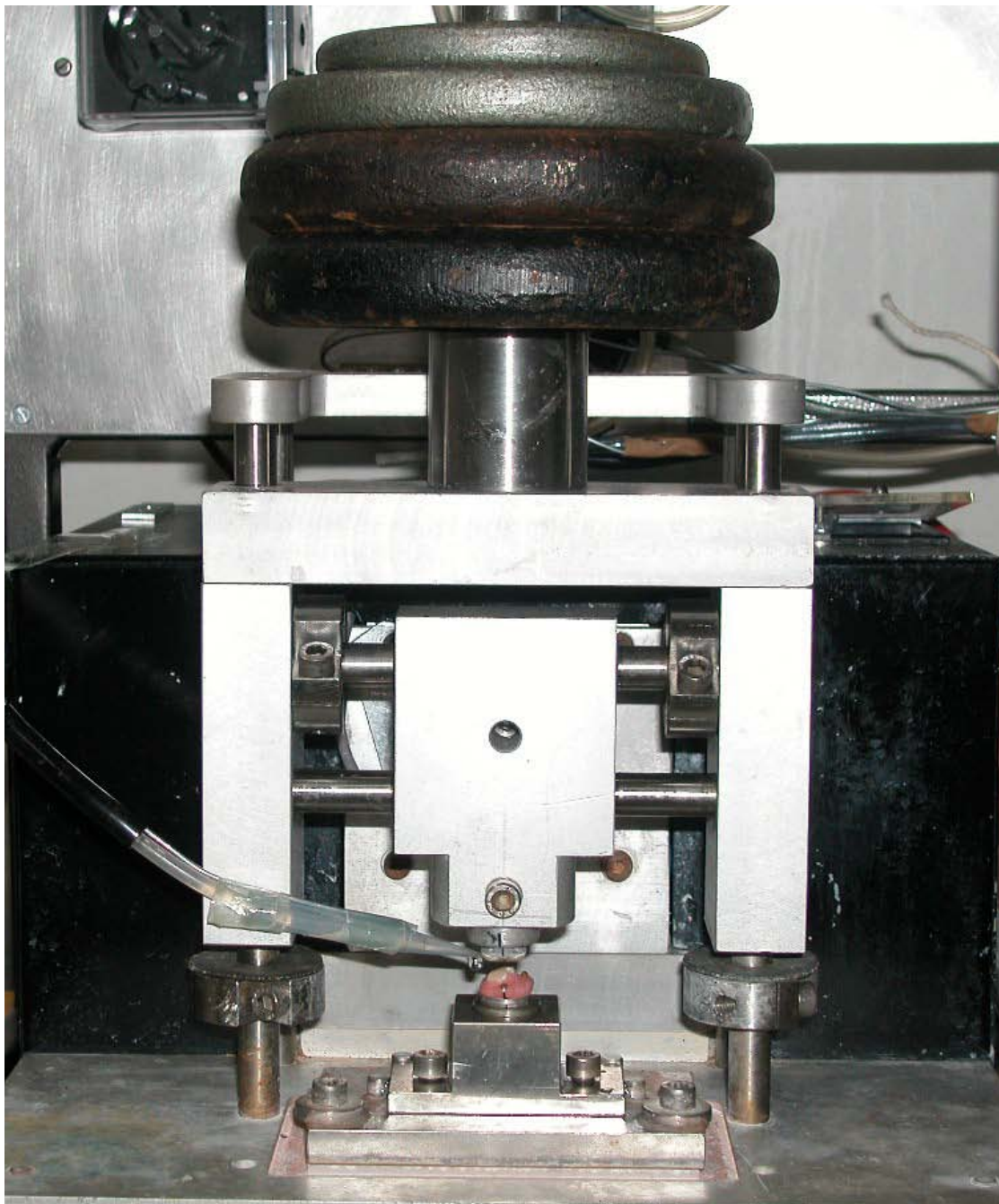


Figure 6. 1: Tooth wear machine.

The stainless steel holders were designed to accommodate the specimen cylinders and secured to the stationary base frame of the machine. The upper specimen holder was an integral part of the moveable component of the machine that was controlled by the cam. The specimens were secured by a laterally-directed screw that formed part of the specimen holders.

The upper mobile section was designed to support the weight of the applied loads to the specimens, giving an operating range of 2.5N to 162N. It can carry heavier loads but Kaidonis et al. (1998) demonstrated that under very high stresses, there was more haphazard “destructive” breakdown of enamel of a type not seen during function.

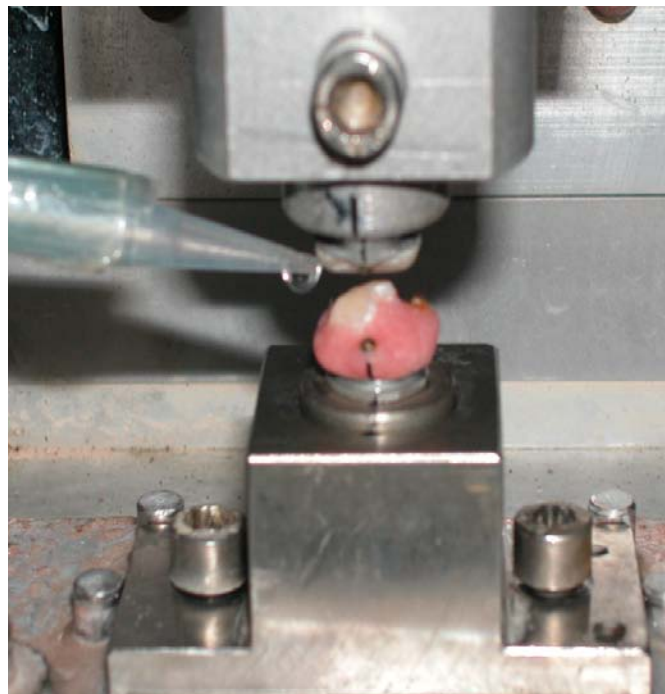


Figure 6. 2: A closer view of tooth wear machine.

6.2 Determination of experimental conditions (Preliminary experiments)

The necessity to standardize the operating conditions for the wear machine and to validate the methods required a series of preliminary studies to be carried out before the finalization of the experimental protocols.

The results were potentially affected by a range of factors including:

- cycling rates of wear machine,
- type of movement,
- specimens orientation (upper or lower),
- area of contact,
- duration of experiments,
- load,
- water uptake and measurement errors.

6.2.1 Effects of cycling rates

Previous studies by Kaidonis (1998) have demonstrated that there were no significant differences in the mean enamel loss when the wear machine was run at either 80 cycles/min or 160 cycles/min. He conducted the experiments of enamel wear under a load of 32N for two groups (each of four pairs); one group was run at a rate of 80

cycles/min and the other at 160 cycles/min. Average weight loss of enamel was calculated and is presented in Table 6.1:

Table 6. 1: Comparison of average loss of enamel (mg) at 80 cycles/min and 160 cycles/min of machine speed.

Cycles	80 cycles/min			160 cycles/min			t-value	P
	n=8			n=8				
	Wt (mg)	S.D.	S.E.	Wt (mg)	S.D.	S.E.		
89000	5.80	1.64	0.58	7.30	1.39	0.49	1.97	0.066

Although the mean enamel loss at the rate 160 cycles/min exceeded that of 80 cycles/min when the machine was run for 89,000 cycles, the difference was not statistically significant, reflecting the relatively large standard errors associated with the mean values.

In addition, a speed of over 160 cycles/min caused the upper specimen to impact the lower specimen with greater force during the return movement, causing an increase in surface breakdown. At 80 cycles/min, the return movement was controlled. These data and observations were re-evaluated during the preliminary stage of this study and the assumptions made by Kaidonis (1998) were adopted for this study.

6.2.2 Effects of type of movement

The setting on the wear machine can be adjusted to perform either uni-directional or bi-directional movements. In 1998 Kaidonis established that the average enamel wear rates (by weight) were not significantly different between the two types of movements. The conclusion was drawn after conducting wear experiments on enamel under 32N and 99.5N with similarly controlled duration of contact and number of cycles but different movement directions. These data are presented in Table 6.2.

Table 6. 2: Comparison of mean rate of tooth wear for uni-directional and bi-directional movements for various loads (N).

Load (N)	Uni-directional			Bi-directional			t-value
	n	Mean (mg/10 ³)	SE	n	Mean (mg/10 ³)	SE	
32	28	0.06	0.01	6	0.04	0.01	1.55
99.5	18	0.13	0.02	6	0.15	0.03	0.44

Shabanian (2000) again confirmed this finding by testing the wear between enamel and composite resin (Z100, 3M Co, St Paul, MN, USA). The results showed no significant difference in average mean wear rates (by height) between the two movements. This experiment was performed at pH=7, and under loads of 32, 67 and 99.5N (Table 6.3).

Table 6. 3: Mean wear rate ($\mu\text{m}/10^3$) of composite resin for uni-directional and bi-directional movements at pH=7.0 and under three different loads (N) after 80,000 cycles.

Load (N)	Uni-directional			Bi-directional			p-value
	n	Mean ($\mu\text{m}/10^3$)	SD	n	Mean ($\mu\text{m}/10^3$)	SD	
32	7	0.43	0.11	6	0.68	0.31	0.08
67	9	0.80	0.23	6	0.76	0.23	0.8
99.5	7	1.53	0.63	6	1.41	0.31	0.54

6.2.3 Specimen orientation

It could be hypothesized that the observed wear rates were different between the upper movable specimen and the lower fixed specimen. However, this has been shown not to be the case in studies by both Kaidonis (1995) and Shabaniyan (2000). The first author conducted a wear experiment on eight pairs of teeth at 32N for a total of 89,000 cycles. The mean wear rates of enamel (by weight) are presented in Table 6.4.

Table 6. 4: Loss of enamel (mg) for both upper and lower specimens over a total of 89,000 cycles.

Cycles	Upper		Lower		t-value
	Mean ($\text{mg}/10^3$)	SE	Mean ($\text{mg}/10^3$)	SE	
89000	13.55	0.56	12.60	0.64	1.120

The second author carried out experiments on three samples (each of nine pairs) at loads of 32, 67 and 99.5N for 80,000 cycles in water (pH=7.0). The results (mean enamel wear rates in height) are shown in Table 6.5. Unpaired t-tests showed no significant differences in mean enamel wear rates of upper and lower specimens under different loads.

Table 6. 5: Mean wear rates ($\mu\text{m}/10^3$) of enamel for upper and lower specimens under different loads and after 80,000 cycles.

Load (N)	Upper			Lower			p-value
	n	Mean ($\mu\text{m}/10^3$)	SD	n	Mean ($\mu\text{m}/10^3$)	SD	
32	7	0.46	0.12	8	0.45	0.16	0.9
67	9	0.64	0.22	9	0.66	0.23	0.8
99.5	6	1.11	0.32	6	1.08	0.60	0.9

6.2.4 Effects of specimen contact

The shape and size of natural teeth vary and as a result facet areas vary between specimens. Therefore, it is necessary to determine whether variation in these characteristics of the specimen teeth affected wear rates.

A study conducted by Kaidonis (1998) described the pattern of change in facet area during the wear process. He used four specimens which were worn against each other under controlled conditions until the secondary wear phase was reached. The teeth were then worn for between 5000 (period 1) and 10000 cycles (period 2) and the facet areas were calculated initially and after each period by making positive replicas from silicone impressions of the facets. The result showed that the increase in facet areas linearly related to the number of cycles (Fig. 6.3), a phenomenon that has also been described by other researchers (Ratledge et al. 1994).

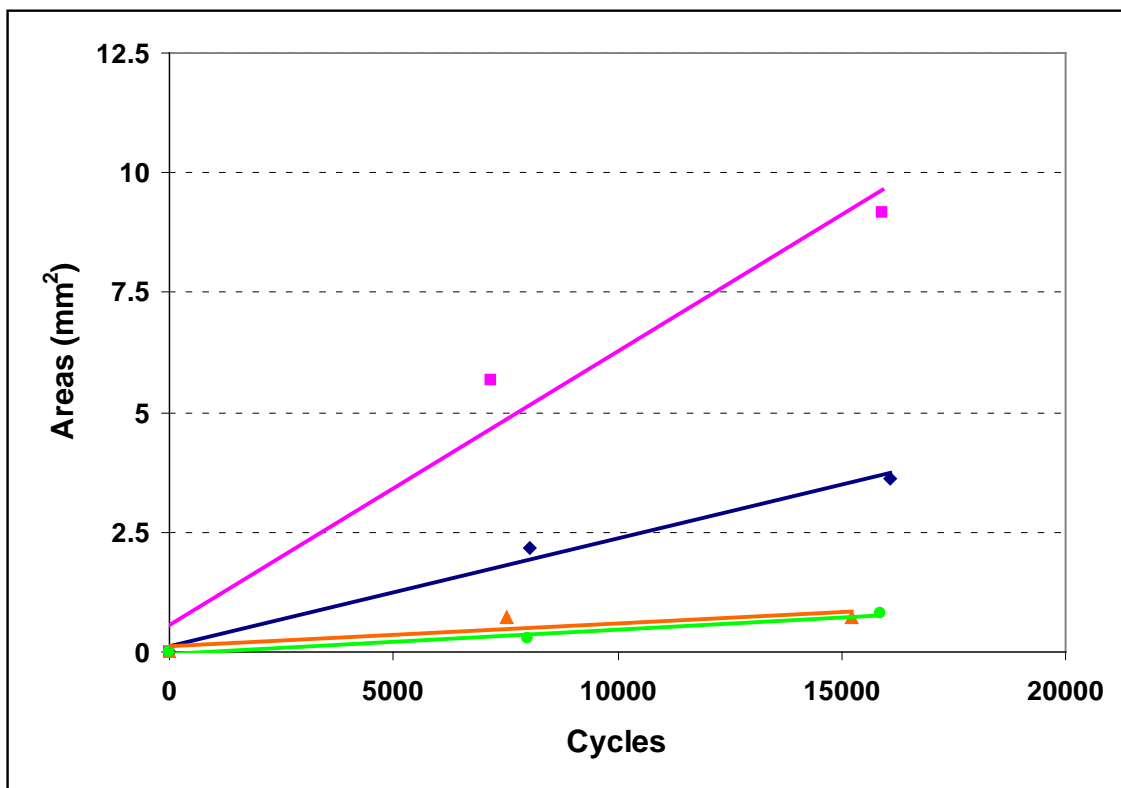


Figure 6.3: Trends of increasing facet area over two consecutive periods for each of four specimens.

Shabaniyan (2000) tested the effect of the facet area on the rate of wear by wearing 10 specimens with water lubricant at a load of 99.5N. Wear facet areas for each specimen were measured from scanning electron microscope images after establishing an early facet by 5,000 cycles and again after 40,000 cycles. In addition, the change in height of the centre of the facet between the two states was measured using a modified light microscope. The results showed a low correlation between the initial area and the change in height ($r=0.37$), which indicated no significant relationship between facet area and wear rate (Table 6.6 and Fig. 6.4).

Table 6. 6: Change in facet height and facet areas after 5000 cycles (S1) and 40,000 cycles (S2) of wear.

Specimen	Facet height change (μm)	Facet area at S1 (μm^2)	Facet area at S2 (μm^2)
1	151	1.65	2.99
2	95	1.60	2.50
3	80	0.81	1.76
4	100	1.04	1.63
5	90	1.65	2.97
6	50	0.36	0.43
7	20	1.47	2.79
8	60	0.81	1.78
9	70	0.61	1.06
10	80	0.31	0.88

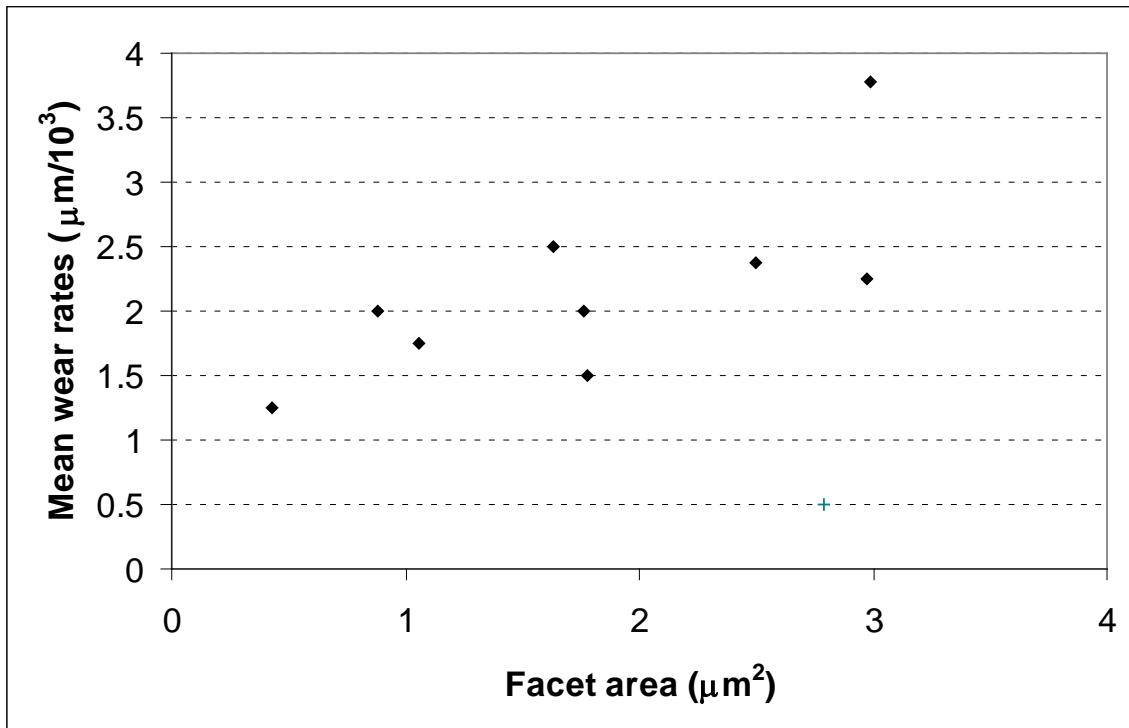


Figure 6. 4: The relationship between the facet area and mean wear rates ($\mu\text{m}/10^3$) of enamel.

Again, these data and observations were also re-evaluated during the preliminary stage of this study and the assumptions made by Kaidonis (1998) and Shabanian (2000) were adopted for this study. The potential contribution of facet area to wear rate (13 percent of the total observed variation) was considered to be small and likely to be even less significant in the present study where porcelain specimens were constructed as flat discs.

6.2.5 Duration of experiments (number of cycles)

Kaidonis (1998) used eight pairs of teeth worn at 32N for a total of 89,000 cycles at a machine speed of 80 cycles/min with data collected at 10000, 20000, 30000, 40000, 50000, 70700 and 89000 cycles. It became apparent that enamel wear rate showed two

distinct phases. The first (primary) phase was of short duration (20,000 cycles) and faster rate than the second (secondary) phase. For both phases the relationship between number of cycles and enamel wear was linear (Table 6.7 and Fig. 6.5). It was therefore decided to conduct the wear experiments on the ceramics during the secondary wear phase.

Table 6. 7: Cumulative loss of enamel (mg) for upper (n=8) specimens over a total of 89,000 cycles.

Cycles	Mean (mg)	SE
10000	2.75	0.22
20000	4.98	0.40
30000	6.63	0.31
40000	8.08	0.35
50000	9.10	0.33
70700	11.58	0.40
89000	13.55	0.56

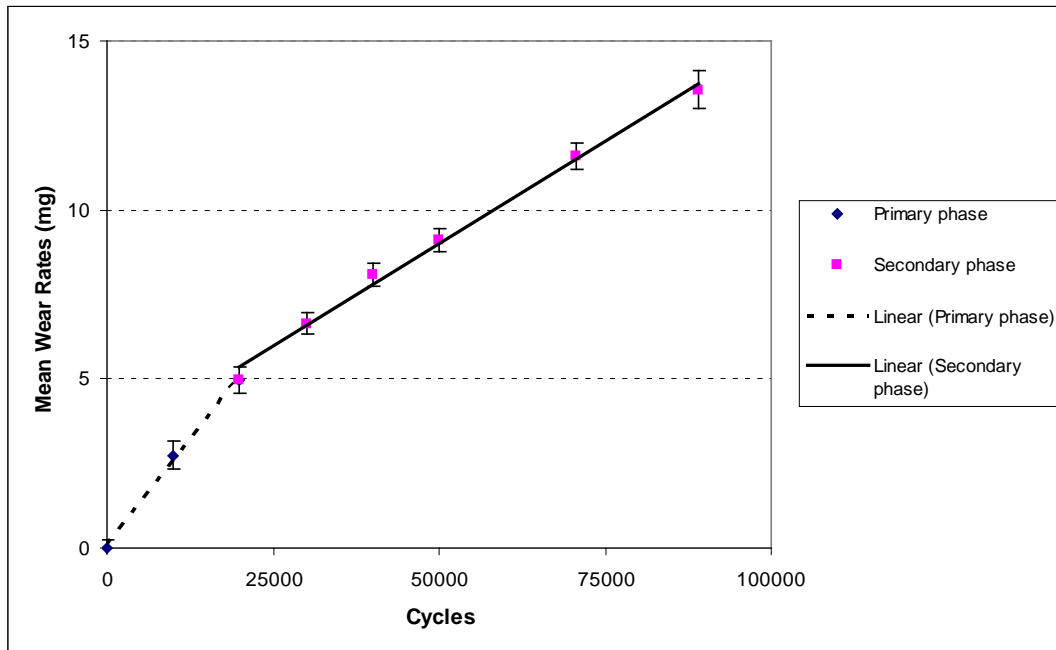


Figure 6. 5: The two phases of enamel wear (with standard error bars) for upper specimens at a fixed load of 32N.

Subsequently, Kaidonis (1998) carried out experiments on 55 pairs of teeth during the primary phase and 33 pairs of teeth during the secondary phase under three different loads of 32, 99.5 and 162N and in dry condition. The results from Kaidonis's study (1998) confirmed that the wear rates of each phase, the wear rates by load, and the trend in wear rate between primary and secondary phases under increasing loads were significantly different.

6.2.6 Load

It has been reported in the literature that the loads during mastication and swallowing range from 5N to 364N (Kelly 1999). The nocturnal bite forces in humans can range

from 11 to 423N (Nishigawa et al. 2001) and the maximum voluntary bite forces can be as high as 1270N (Gibbs et al. 1981). There is little consensus on the selection of loads in previous in vitro studies on wear between enamel and restorative materials (Li et al. 2007). In various studies researchers have applied different loads in the range 2N to 162N (Eisenburger and Addy 2002a;2002b). A load of 100N has been used in previous studies (Kaidonis et al. 1998; Burak et al. 1999; Shabaniyan and Richards 2002; Ranjitkar et al. 2008) and this falls in the operating range of the available wear machines.

6.2.7 Effect of water uptake and measurement errors

Dental tissues and materials can take up or lose water depending on the conditions and the materials involved. This in turn results in dimensional changes. In order to determine water uptake effects on quantitative measurements, a preliminary analysis of water uptake was conducted by the author.

Twenty one selected specimens including three specimens of each group for seven different groups of materials (enamel, a veneering porcelain for porcelain-bonded-to-metal restorations (PBM-veneering) (Duceram®Kiss, Degussa, Germany), a leucite-reinforced veneering ceramic (LR-veneering) (Authentic®Veneering, Ceramay, Germany), a leucite-reinforced pressable ceramic (LR-pressable) (Authentic®Pressable, Ceramay, Germany), a machinable ceramic (Vita®Mark II, Vita Zahnfabrik, Germany), a type III gold alloy (Argenco 2, Argen Corp, USA) and a zirconia (Zerion™, Straumann GmbH, Germany) were scanned twice with a 3D profilometer described in

section 6.6 and the volume of enamel, ceramic or gold above the reference plane was calculated. The second scanning was done after storing the specimens in deionised water for seven consecutive days at room temperature. No significant differences were found between consecutive scans in either enamel or any indirect restorative materials when paired t-test was performed. This indicates there were no systematic differences between different scans and no significant effects of water uptake on the calculated volume (Table 6.8).

The error variance (Se^2) and the standard deviation of a single measure (Ss) between the first and second measurements were calculated according to the Dahlberg's method (Dalberg 1940):

$$Se^2 = (\text{sum } (x_1 - x_2)^2) / 2n$$

$$Ss = \sqrt{(\text{sum } (x_1 - x_2)^2) / 2n}$$

The standard deviation of a single measure was used to determine the extent to which the variability due to experimental error affected the observed variance of the population. The observed variance (So^2) can be considered to be the sum of the error variance (Se^2) and the true sample variance (St^2):

$$So^2 = St^2 + Se^2$$

Errors were estimated to contribute 11 to 12 % to the total observed variation.

This information indicated that the volume assessment method was sufficiently reproducible for the purposes of this study.

Table 6. 8: Two different calculations of volume of enamel and different restorative materials specimens.

Material	Specimen	1st calculation (mm³)	2nd calculation (mm³)
Enamel	1	70.49	70.71
	2	85.41	84.97
	3	24.27	24.23
PBM- veneering	1	26.21	26.12
	2	32.64	32.82
	3	28.87	28.49
LR- veneering	1	33.91	33.72
	2	31.41	31.97
	3	34.13	34.06
LR- Pressable	1	41.85	41.67
	2	47.42	47.84
	3	26.90	26.70
Machinable ceramic	1	47.89	47.30
	2	35.95	36.23
	3	42.0	41.71
Gold	1	34.07	34.49
	2	40.17	40.42
	3	37.78	38.55
Zirconia	1	10.34	10.00
	2	23.67	23.23
	3	20.55	20.79

6.2.8 Conclusion

The final experimental protocols determined from the results of the preliminary experiments were as follows:

- experiments were run at 80 cycles/min to maintain consistency and to minimize the effect of impact stress
- the wear machine was conducted with uni-directional movement
- the specimens were mounted in a consistent way with the different indirect restorative materials attached to the upper component of the machine while the lower component held the enamel specimens
- all experiments were conducted during the secondary wear phase, and
- a load of 100N was applied.

6.3 Preparation of teeth and test materials

Freshly intact third molars extracted as part of routine dental treatment were collected from private practices and stored in thymol solution at 4°C for use. They were examined visually and only those with intact and “normal” buccal and lingual surfaces were selected. The roots were then sectioned off along the cementum-enamel junction using a diamond disc attached to a high speed cutting machine under copious water coolant.

This machine was fabricated at the Department of Mechanical Engineering, the University of Adelaide in 1990 (Fig. 6.6).



Figure 6. 6: Tooth-sectioning machine.

The dental pulp was removed with a spoon excavator and the crowns were then sectioned again mesio-distally into buccal and lingual halves (Fig. 6.7). The teeth were then cleaned in water using finger pressure while wearing latex gloves until any attached soft tissue was removed.

Sixty molars were sectioned and randomly assigned into the groups shown in Fig. 6.8.

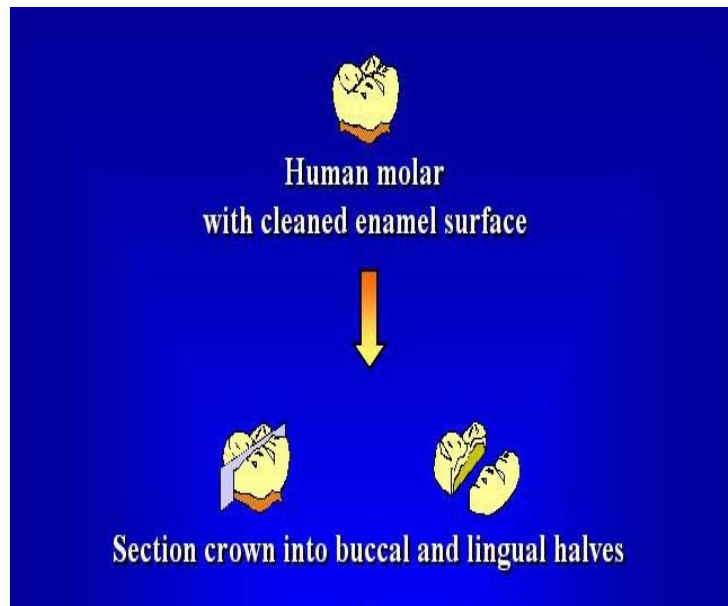


Figure 6. 7: Flow chart showing sequence of tooth sectioning.

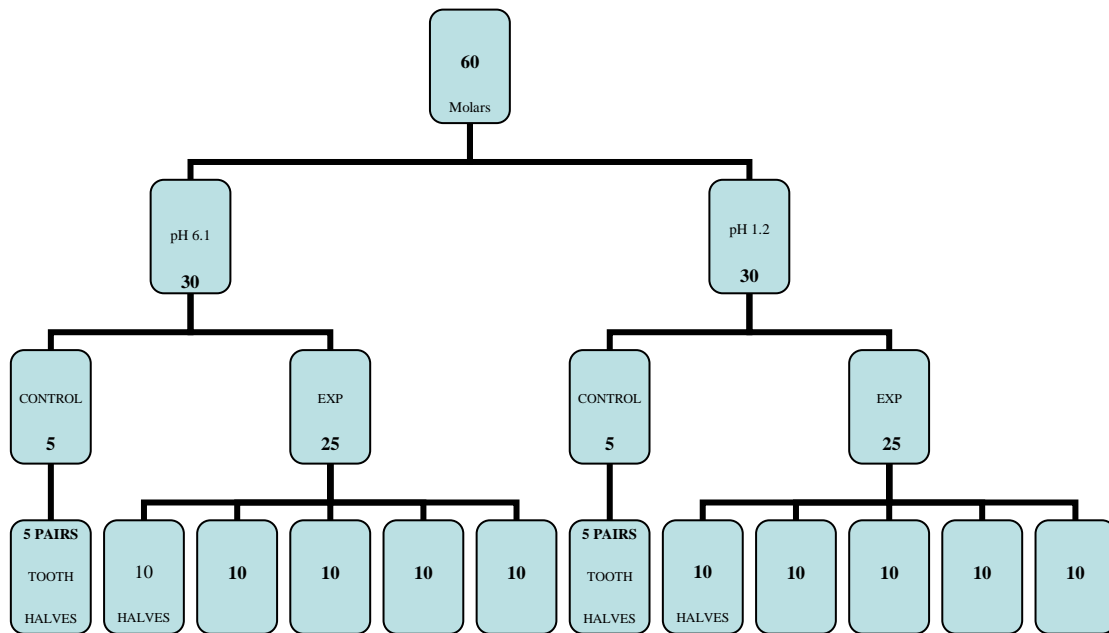


Figure 6. 8: Diagram showing tooth grouping for experiments.

Four different porcelain systems and a type III gold alloy were employed in the experiment. They consisted of:

- a veneering porcelain used over metal substructure where the porcelain is bonded to metal restorations (PBM-veneering) (Duceram[®] Kiss, Degussa, Germany)
- a leucite-reinforced glass ceramic for veneering (LR-veneering) (Authentic[®], Ceramay, Germany)
- a pressable leucite reinforced glass ceramic (LR-pressable) (Authentic[®], Ceramay, Germany)
- a machinable ceramic (Vita[®]Mark II, Vita Zahnfabrik, Germany) preformed by manufacturer for CAD-CAM system
- a type III gold alloy (gold) (Argenco 2, Argen Corp, USA).

Information on the products from the manufacturers is included in Appendix 2.

The firing temperatures required during manufacture are presented for each ceramic (Table 6.9).

Table 6. 9: Firing temperatures of porcelain systems.

Porcelain systems	Firing temperature (°C)
PBM-veneering	910
LR-veneering	750
LR-pressable	940

The composition of the gold alloy is as follows (Table 6.10):

Table 6. 10: Composition of type III gold.

Au	Pt	Pd	Ag	Cu	Others
74.1%	1.6%	2.3%	13.4%	7.2%	Ir, Sn

Twenty discs of PBM-veneering and 20 of LR-veneering, each of 10mm diameter and 1mm thick, were fabricated in the laboratory by a specialist dental ceramist. The porcelain powder was mixed with the liquid, condensed, shaped and fired in Programat P300 porcelain furnace (Ivoclar, Vivadent) with a set-up program of suitable temperature and time for each type of porcelain following the manufacturer's recommendations as presented in Table 6.11.

Table 6. 11: Set up temperature and time for porcelain firing.

Material	Starting temperature	Vacuum start	Final temperature	Holding time
	°C	°C	°C	Min
PBM-veneering	400	400	910	1
LR-veneering	400	400	750	1

For LR-pressable ceramic, the buttons which were sectioned from the castings were collected from the Adelaide Dental Hospital laboratory and used for the study (Fig. 6.9). They were sectioned to provide 20 discs of 1mm thickness with a high speed diamond disc under water coolant and only the newly cut surfaces were used as the facet for wearing in the experiment.

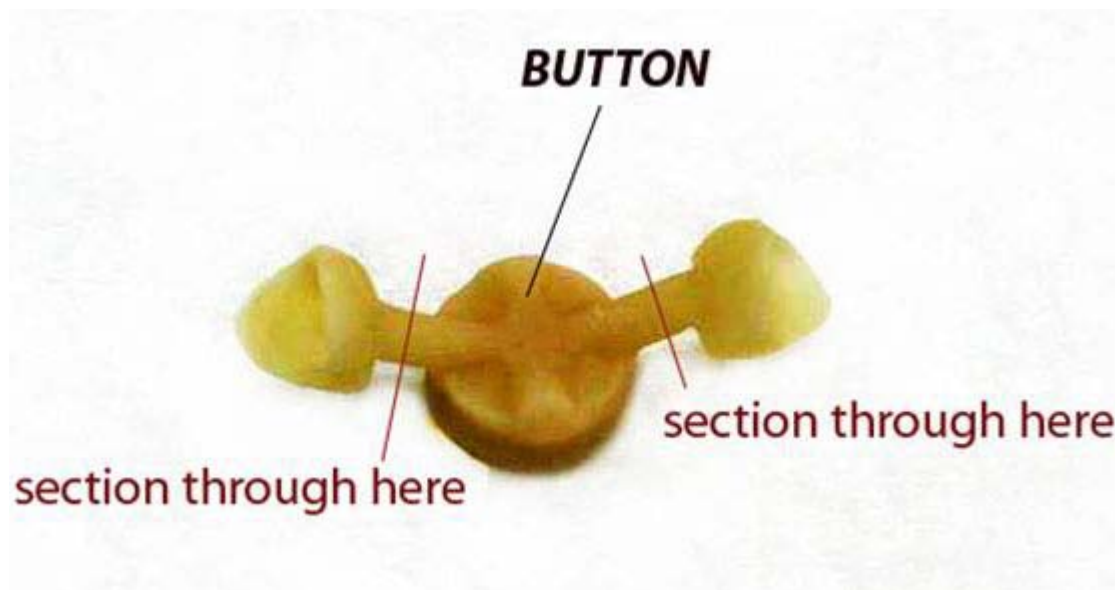


Figure 6. 9: LR-pressable ceramic- crowns attached to the button after casting.

Twenty discs of machinable ceramic were also obtained by sectioning the ingots in the same way as for the LR-pressable blocs.

Surfaces of porcelain discs were subjected to different treatment procedures, depending on the material. Porcelain discs of PBM-veneering, LR-veneering and LR-pressable ceramic were treated in the following way. They were initially ground relatively flat

with a green stone (Edenta, Switzerland). Then both surfaces of each disc were first polished flat with silicone carbide papers of 180 grit (Eagle Brand, Kovax®, Tokyo, Japan). On examination, only the smoother and more even surface of each disc was then subjected to further polishing with silicone carbide paper of finer grits in order to obtain a suitable surface for the wear experiment. These surfaces were polished gradually with abrasive paper of 320, 800 and 1000 grit. After each polishing, the surface was observed under a magnification ($\times 2.5$) to ensure that the procedure had been performed adequately before proceeding onto the next step. The porcelain surfaces were expected to be very smooth on finishing with the paper of 1000 grit. They then were autoglazed in the furnace to be ready for the experiment.

Porcelain discs of the machinable ceramic were only hand polished due to the fact that this material is used chairside by the operator and does not involve any laboratory work. This was done using a porcelain polishing kit (Shofu, Japan) and finally finished with a felt wheel and diamond paste (Henry Schein Inc., USA).

Gold was provided in square ingots of 1m thickness from the manufacturer. The smooth surfaces of the discs were polished with a gold polishing kit (Shofu, Japan) and paste.

6.4 Mounting specimens of Scanning Electron Microscopy (SEM) studs

The surface of SEM studs and the rougher surfaces of porcelain discs and gold alloy were sandblasted with aluminum powder $125\mu\text{m}$ (Argibond Australia) under pressure (2

bars) using a sandblasting unit (Renfert GmbH, Germany). The porcelain and gold discs were cemented to the SEM studs, using a chemically-cured resin cement (Panavia F, Kuraray, Japan). The base and catalyst component of the cement was mixed with the ratio of 1:1, and applied to the roughened surface of the discs and they were attached to the sandblasted surface of the studs and light cured for 40 seconds with a light cure unit (Visilux 2, 3MTMESPETM, Australia).

On each specimen, three metal balls of 2mm diameter were attached to the side of the stud with cold-cured resin (Vertex self curing, Vertex Dental BV, the Netherlands) to act as reference points for the subsequent scanning process.

Tooth halves were mounted onto the sandblasted SEM studs in a different way. They were secured in cold-cured acrylic resin (Vertex self curing, Vertex Dental BV, the Netherlands), which covered one half of the tooth specimen. Care was taken to ensure that the most prominent part of the enamel was positioned in the middle of the studs. The three reference metal balls were also partially embedded in resin, leaving the top part exposed. It was important that all the metal balls were equally spaced and were below the highest point of the enamel, porcelain or gold discs when the stud surfaces were horizontally positioned. This played an important role in the interpretation of the scanning images and for calculating the object volume above the reference plane formed by the metal balls (Figs. 6.10, 6.11 and 6.12).



Figure 6. 10: Enamel specimen on SEM stud with three reference metal balls.



Figure 6. 11: Porcelain specimen on SEM stud with three reference metal balls.



Figure 6. 12: Gold specimen on SEM stud with three reference metal balls.

6.5 Method of lubrication

The lubrication system was set up in a similar way to that used by previous researchers (Kaidonis et al. 1998; Burak et al. 1999; Shabaniyan and Richards 2002; Ranjitkar et al. 2008). It consists of a plastic reservoir containing lubricant, which was connected by plastic tubing to a small pipette tip. The reservoir was placed on a stand that was 0.5m higher than the tip. A device was attached to the tubing to allow the adjustment of the lubricant flow. The end of the tubing was clamped to an adjustable stand, the position of which could be changed to direct the lubricant onto the crown without impinging on the movement of the upper and lower specimens. The flow rate was fixed at 0.5ml/min (Shabaniyan 2000) and the lubricant collected and discarded after a single passage through the system.

6.6 Methods of assessment

6.6.1 Three Dimensional Scanning (3-D) and quantitative results

A 3D scanner (PIX-4, Roland DG, Tokyo, Japan) (Fig. 6.13) with an active piezo sensor that detects the contact between its stylus and the scanned surface was used to scan the surface of each specimen at various stages. This scanner was interfaced with a personal computer to record the mesh points of the scanned object. The “Dr PICZA” software (Roland DG, Tokyo, Japan) was provided with the scanner to define the scanning area and set the upper and lower scan limits to scan to optimize the data set and shorten the scanner’s calibration and scanning times. The resolution of the scanner can be set

according to the user's preference and purpose. The finer the resolution, the longer the scanning time was. In this experiment, the resolution was set at the highest resolution, that is $50\mu\text{m}$ for the X and Y coordinates and $25\mu\text{m}$ for the Z axis. With this resolution, it took roughly four hours for one object to be scanned. The software also allows the manipulation and visualization of the data and has the facility to export data in a range of formats for further analysis.



Figure 6. 13: PIX-4 3D scanner.

A purpose-written software package especially developed using MATLAB 6.5 (The Mathworks Inc, Natick MA, USA) was used to analyze the data. This package accepts data from “Dr PICZA”, which have been converted to a regular mesh of grid for optimum use. The package allows the researcher to view the graphic data in 3D. It also enables the defining of a reference plane, which, in this case, was determined by the highest point of the three metal balls (Figs 6.14 and 6.15). The volume of the scanned

object above the reference plane then can be analyzed and calculated by the software. The volume was determined as the mean result after each data set was evaluated three times.

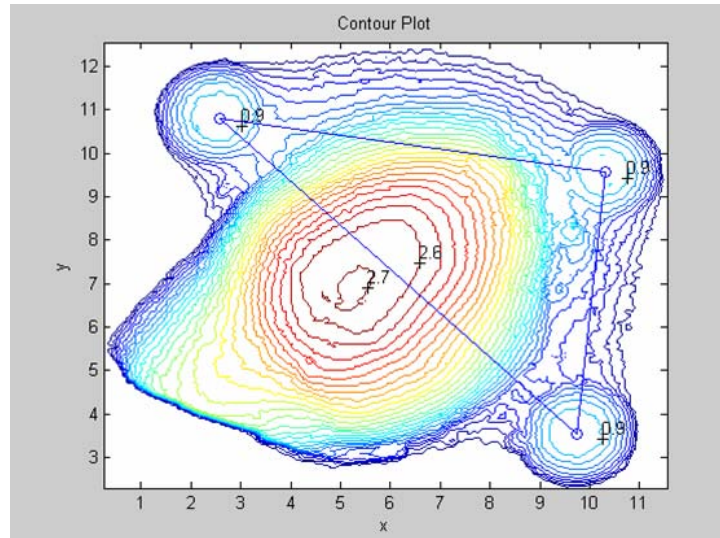


Figure 6. 14: The graphic data in 3D.

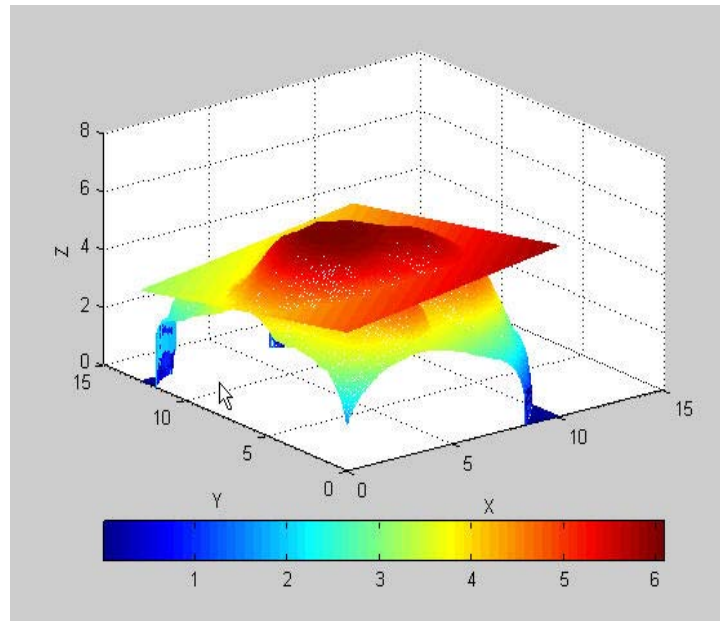


Figure 6. 15: Reference plane defined for volume calculation with Matlab.

The validity and reliability of the scanning system have been confirmed in experiments reported by Liu et al. (2004), who showed that the difference in the scanning volume compared to the true volume when scanning a relatively simple object was between 3.7 percent and 8.5 percent, depending on the size and geometry of the specimen. This study also confirmed that there were no significant differences between observers and repeated scans.

Baseline scanning was performed for enamel, porcelain and gold specimens after the primary wear phase when the initial wear facets were obtained. The volume of tooth or material above the reference plane formed by the three steel balls was calculated.

After wearing specimens during the second wear phase, they were subjected to the same scanning and calculating process to determine the volume of the specimens above the reference plane. Wear volume in cubic millimeter was defined as the difference between the two sets of data collected using the software Excel 2003 (Microsoft Corporation).

6.6.2 Scanning Electron Microscopy (SEM) and qualitative results

6.6.2.1 Fabrication of specimen replicas

For each specimen, two resin replicas were fabricated, the first after the initial wear facet was created and the second after being subjected to the number of wear cycles required as part of each experimental protocol. Polyvinylsiloxane (Imprint™, Quick Step,

Regular body 3M™ ESPE™) was used to make an impression of the specimens and the replicas were constructed in epoxy die material (Adelaide Epoxy supplies, South Australia) according to the manufacturer's instructions. The replicas were mounted on standard SEM studs. These were subjected to subsequent scanning electron microscope examination.

6.6.2.2 Scanning Electron Microscopy (SEM) observations

Scanning electron microscopy was used to examine the surface characteristics of enamel, porcelain and gold. Epoxy resin replicas on SEM studs were coated in gold-carbon for SEM analysis with the Phillips 20XL Scanning Electron Microscope. Observations of the overall surface were completed first under low magnification (20 times) and subsequent details of the wear facet were closely observed under higher magnification (100 and 200 times). Images were stored and written to CD for subsequent comparison and analysis.

6.7 Statistical analysis

For each experiment, data were summarized in tabular form with sample size, mean wear rates and their corresponding standard deviation. Mean wear rates of enamel and the opposing materials were analyzed by Analysis of Variance and post hoc Bonferroni test. A t-test was used to compare the mean wear rates of enamel and antagonists within groups.

Statistical analyses were performed using Stata (StataCorp Lp, version 8). Statistical significance was set at the 0.05 probability level.

6.8 Experiments at pH 6.1 and pH 1.2

Two series of experiments at pH 6.1 and 1.2 were conducted to investigate the interaction between enamel, the four selected porcelain systems and a type III gold alloy under a load of 100N with the two lubricants. The pH 6.1 simulates the condition of a near neutral oral environment and pH 1.2 simulates the conditions occurring in patients with acid regurgitation (Holbrook et al. 2009). Deionised water was used because it is one of the standard lubricants in tribological studies of dental materials (ISO/TS 14569-2-2001).

The selected load under which the wear experiments were conducted was 100N. This load has been used in previous studies on wear of enamel, dentine and restorative materials (Kaidonis et al. 1998; Shabaniyan and Richards 2002; Ranjitkar et al. 2008). Lower loads would require longer time to yield measurable results when two hard surfaces such as enamel and porcelain wear against each other, and this is not practical, considering that the experimental procedure was extremely time-consuming. For example each specimen being investigated at pH 6.1 required 30 hours for wearing and scanning to give measurable results.

Each experiment included six groups of specimens. In each group, there were ten enamel specimens paired with material specimens (Figs 6.16 and 6.17). The controls had five pairs of enamel specimens wearing against each other.

All the enamel, porcelain and gold specimens were stored in deionised water at room temperature for seven days before being subjected to wear. The pH of deionised water was measured in triplicate (3 aliquots per solution) with an electrode (Activon digital pH/mV meter, Activon Scientific products, Australia) and the mean pH was 6.1 (SE = \pm 0.11) (Ranjitkar et al. 2008).

In this study specimens were subjected to 20,000 cycles of wear during the initial primary phase before the actual experiment commenced (Kaidonis, 1998). In the experiment conducted at pH 6.1, specimens were then worn under a load of 100N in deionised water for 120,000 cycles at a wear rate of 80 cycles per minute over a period of 25 hours of continuous operation of the machine.

The enamel and porcelain specimens used for the experiment at pH 1.2 were painted with nail varnish (Rimmer, London), leaving a window of 2×3mm in order to protect the surfaces from the eroding effect of HCl (Fig. 6.18). Hydrochloric acid was prepared in the biomaterial laboratory at a pH=1.2 (titratable acidity = 0.063M). Because enamel wear occurred quickly in this acidic environment, specimens were subjected to 10, 000 cycles of wear rate 80 cycles per minute.

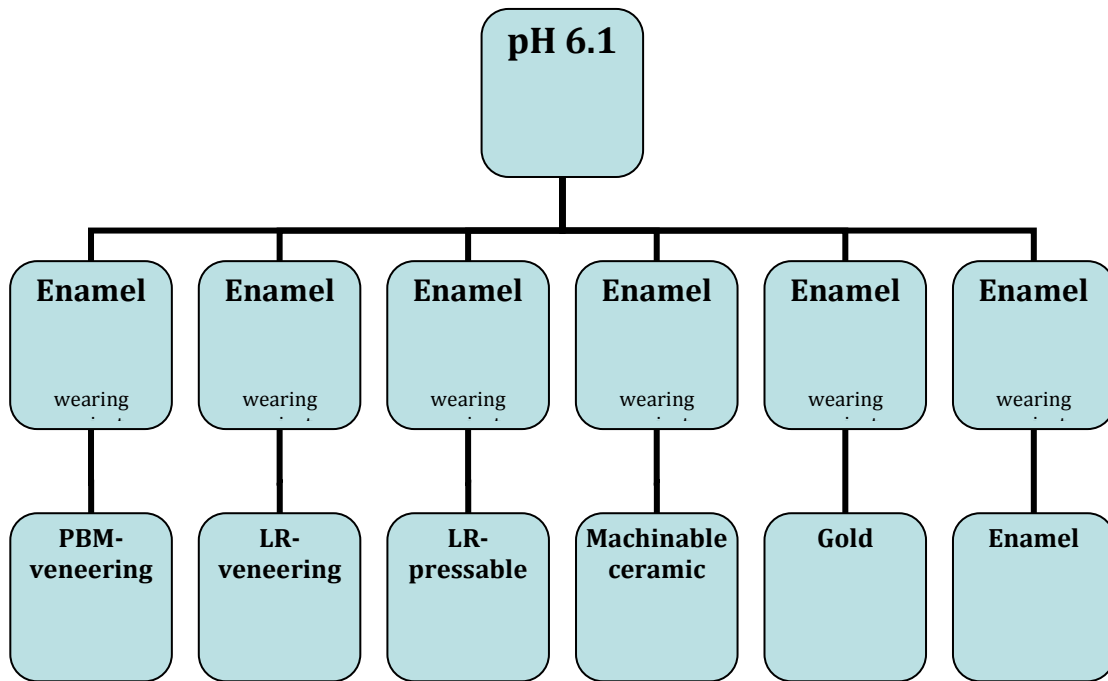


Figure 6. 16: Experimental design at pH 6.1

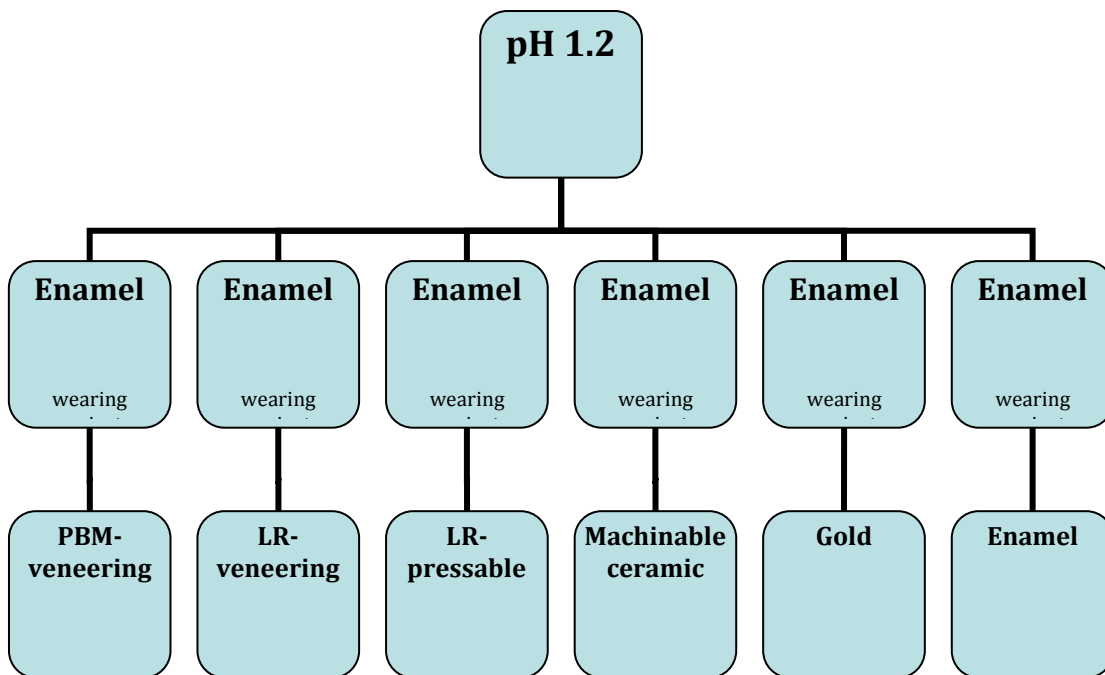


Figure 6. 17: Experimental design at pH 1.2



Figure 6. 18: Specimens covered with nail varnish.

CHAPTER 7

RESULTS

7.1 Introduction

In this chapter the quantitative and qualitative wear results for experiments conducted at pH 6.1 and 1.2 are presented for the selected materials, the details of which were outlined in detail in section 6.3 and are summarized in Table 7.1.

Table 7. 1: Types of materials and their abbreviations.

Materials	Brand name	Abbreviation
Veneering ceramic for porcelain bonded to metal (PBM) restorations	Duceram® Kiss	PBM-veneering
Leucite reinforced veneering ceramic	Authentic™ Veneering	LR-veneering
Leucite reinforced pressable ceramic	Authentic™ Pressable	LR-pressable
Machinable ceramic	Vita® Mark II	Machinable ceramic
Type III gold alloy	Argenco 2	Gold

Although no specimens failed during the experiments, those with the mean wear rates outside of the range “mean \pm 2 standard deviation” were eliminated to make sure the results were not skewed by outliers. This explained why the numbers of specimens were not consistent in all experimental groups.

7.2 Experimental results at pH 6.1

7.2.1 Quantitative assessment at pH 6.1

Wear rates of enamel and opposing indirect restorative materials in deionised water (pH=6.1) were obtained under a load of 100N over 120,000 cycles. The mean wear rate of enamel obtained from the control group, in which buccal and lingual tooth halves of the same tooth wore against each other, was at $0.09 \pm 0.05 \text{ mm}^3$ per 1,000 cycles. In addition, the **mean wear rates of enamel** wearing against different restorative materials are summarized and descriptive statistics are presented in Table 7.2. Fig. 7.1 shows mean wear rates with the corresponding standard error bars for **enamel** worn against each material.

One-way analysis of variance showed significant differences between the wear rates of enamel between groups ($p < 0.01$). Bonferroni test was used to compare the mean wear rate of enamel of one group to the others. While the mean wear rate of enamel opposed by LR-pressable was significantly higher than that of the enamel control specimens ($p < 0.01$), the wear rates of enamel in other groups were comparable to that of the

controls. In addition, enamel wear rates caused by LR-veneering and LR-pressable were also found to be significantly different to each other. This also applied to the wear rates of enamel opposed by gold in comparison with that opposed by either PBM-veneering or LR-pressable (Table 7.3).

Table 7. 2: Mean wear rates of enamel opposed by different restorative materials under a load of 100N and at pH 6.1.

Opposing material	n	Mean	SD	Range	SE
(Enamel Wear)					
(mm ³ /1000 cycles)					
PBM-veneering	10	0.12	0.04	0.07 to 0.18	0.01
LR-veneering	9	0.09	0.05	0.03 to 0.17	0.02
LR-pressable	9	0.21	0.12	0.04 to 0.38	0.04
Machinable ceramic	8	0.09	0.08	-0.04 to 0.20	0.03
Gold	9	0.00	0.04	-0.06 to 0.08	0.01
Enamel control specimens	10	0.09	0.05	0.01 to 0.19	0.02

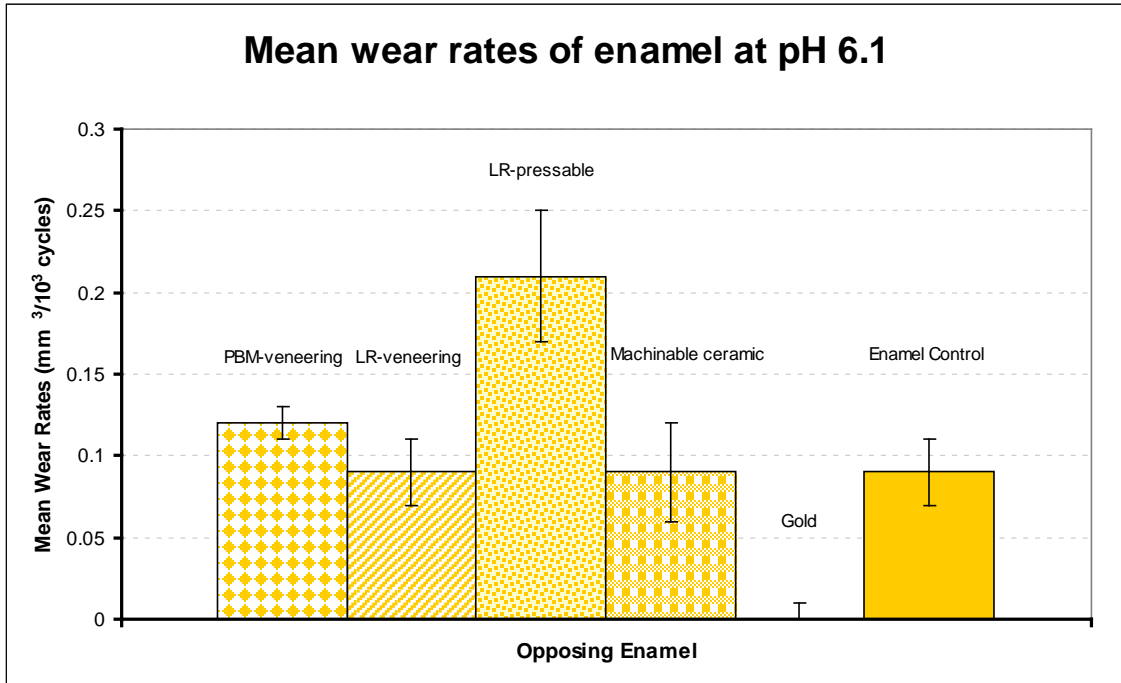


Figure 7.1: Mean wear rates of enamel (with standard error bars) wearing against different indirect restorative materials under a load of 100N at pH 6.1.

Table 7. 3: Pair-wise comparison of enamel wear rates between groups by Bonferroni test (load 100N, pH 6.1).

Opposing material	PBM-veneering	LR-veneering	LR-pressable	Machinable ceramic	Gold
LR-veneering					
LR-pressable		p<0.01			
Machinable ceramic			p<0.05		
Gold	p<0.01		p<0.01		
Enamel control			p<0.01		

The descriptive statistics of the **mean wear rates of different indirect restorative materials** wearing against the enamel is summarized and presented in Table 7.4. The highest wear rate of the material was found with LR-veneering ($0.21 \pm 0.12 \text{ mm}^3$), while the least wear remained with gold ($0.03 \pm 0.07 \text{ mm}^3$). Fig. 7.2 shows mean wear rates with the corresponding standard error bars for each material.

Table 7. 4: Mean wear rates of different indirect restorative materials under a load of 100N at pH 6.1.

Material	n	Mean	SD	Range	SE
		(mm ³ /1000 cycles)			
PBM-veneering	10	0.12	0.05	0.05 to 0.21	0.02
LR-veneering	9	0.21	0.12	0.05 to 0.37	0.04
LR-pressable	9	0.10	0.05	0.03 to 0.17	0.04
Machinable ceramic	8	0.07	0.04	0.01 to 0.13	0.03
Gold	9	0.03	0.07	-0.05 to 0.18	0.02

Analysis of variance also showed significant differences in wear rates between materials ($p < 0.01$). Pair-wise comparison by Bonferroni test found significant differences between wear rate of LR-veneering in comparison to that of other materials such as LR-pressable ($p < 0.05$), machinable ceramic ($p < 0.01$) and gold $p < 0.01$) (Table 7.5).

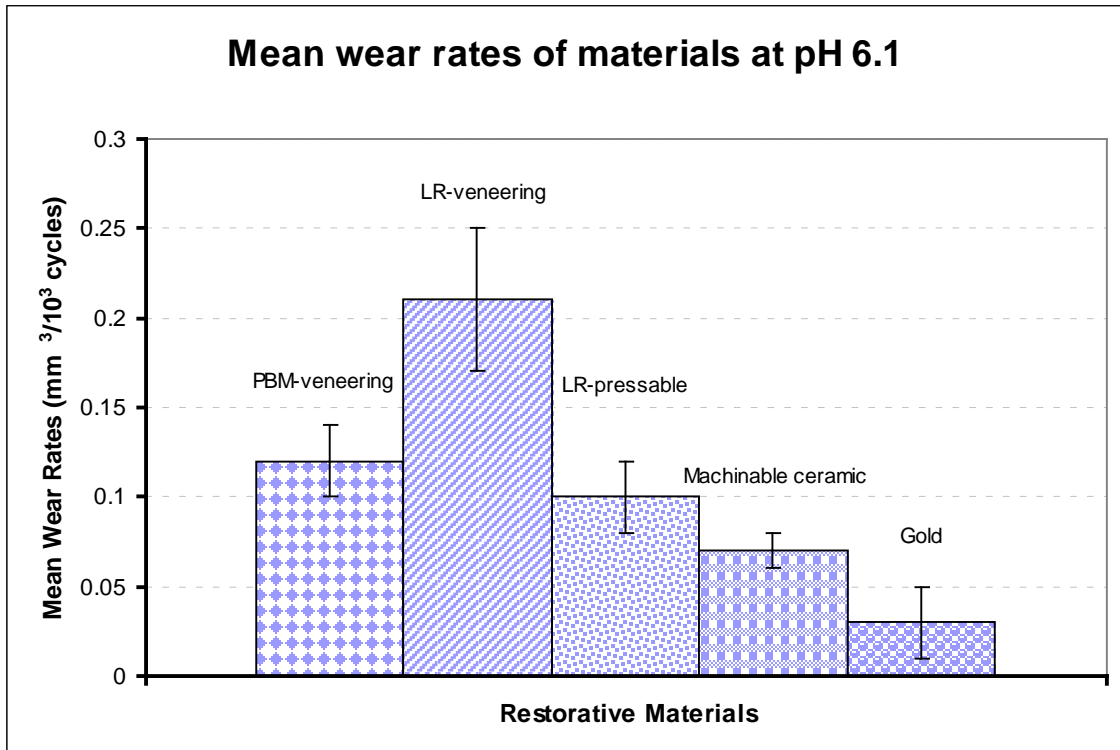


Figure 7. 2: Mean wear rates of different indirect materials (with standard error bars) opposing enamel under a load of 100N at pH 6.1.

Table 7. 5: Pair-wise comparison of material wear rates between groups by Bonferroni test (load 100N, pH 6.1).

Material	PBM-veneering	LR-veneering	LR-pressable	Machinable ceramic
LR-veneering				
LR-pressable		p<0.05		
Machinable ceramic		p<0.01		
Gold		p<0.01		

The mean wear rates of enamel and the opposing material within the same group were compared using t-test (Table 7.6). Statistically significant differences were found in the mean wear rates of enamel and LR-veneering combination ($p<0.05$), in which enamel wear rate was approximately half of that of LR-veneering. The mean wear rate of enamel and was also found to be significantly different to that of LR-pressable ($p<0.05$), the enamel wear rate was twice the material wear rate. However, no statistically significant differences were found between the wear rates of enamel and the opposing PBM-veneering, machinable ceramic and gold antagonists.

Table 7. 6: Comparison of mean wear rates of enamel and opposing materials within groups (load 100N, pH 6.1).

		Materials	t-test
Enamel	<i>versus</i>	PBM-veneering	NS*
		LR-veneering	p<0.05
		LR-pressable	p<0.05
		Machinable ceramic	NS
		Gold	NS

* NS: no significant difference

Mean wear rates of enamel and opposing materials in all groups are present in Fig. 7.3 to give a complete picture of the experiment at pH 6.1.

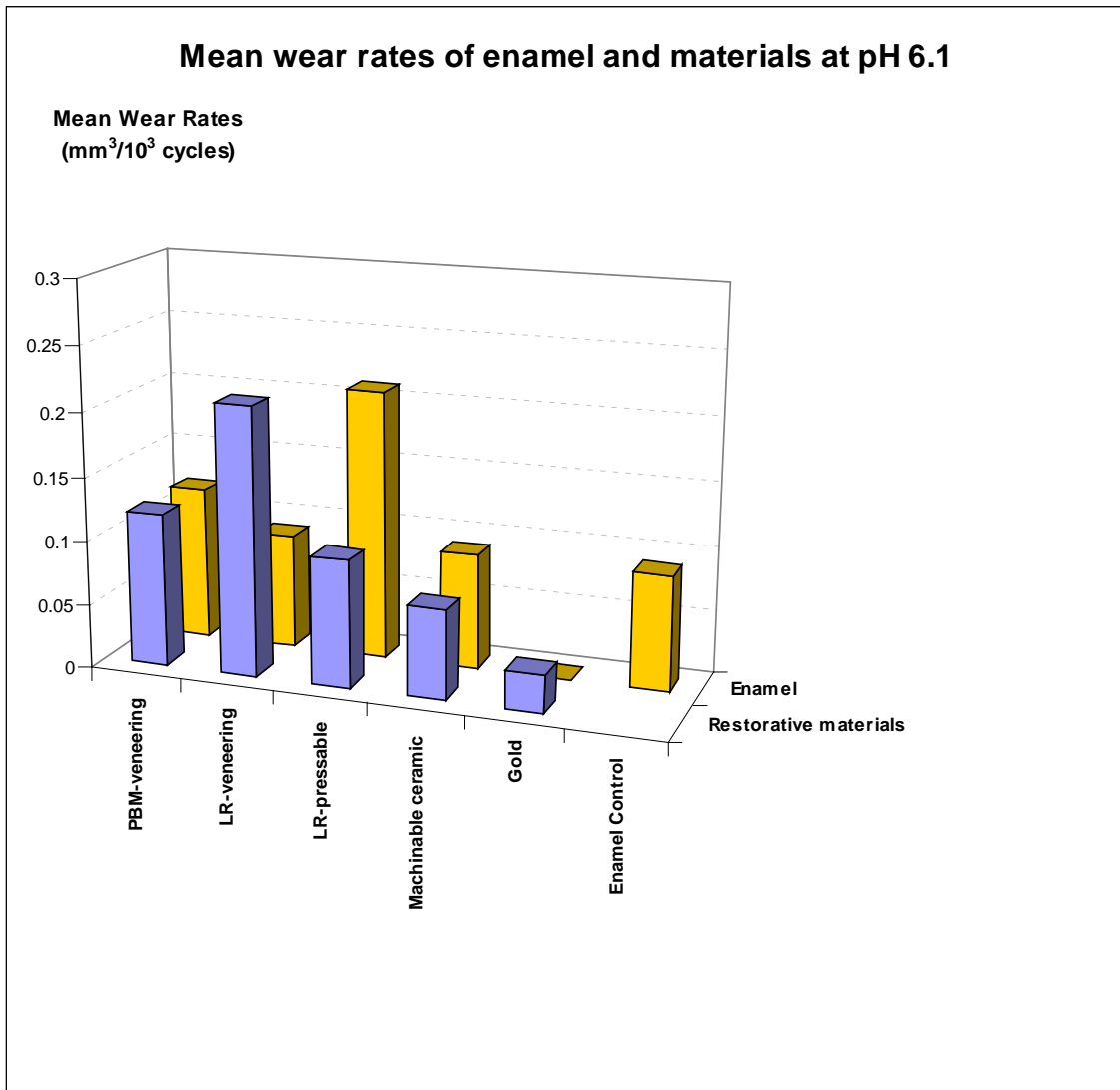


Figure 7. 3: Mean wear rates of enamel and different indirect restorative materials under a load of 100N at pH 6.1.

7.2.2 Qualitative assessment at pH 6.1

SEM examinations were conducted to study the surface morphology of enamel and the opposing restorative materials after wear. The surface observations were made in paired samples of the enamel and the material that wore against each other. Five random sites on each wear facet were analyzed to provide the total picture of the surface micro-morphology of the enamel and opposing materials.

SEM observation of the specimens after wearing under a load of 100N and pH 6.1 revealed that the surfaces of enamel control specimens, in which enamel specimens were worn against each other, were roughened. No clear pattern was noticed in this group.

For the groups of enamel wearing against PBM-veneering, LR-veneering, LR-pressable and machinable ceramic, similar patterns were found for the enamel surfaces and the opposing restorative materials. They were composed of prominent striations running parallel to the direction of wear movement and enamel or material chips present on the surface.

With the group in which enamel was worn against gold, a smear layer was observed on the enamel surface while a light and smooth wear pattern were found on the surface of gold.

These observations are presented in Figs. 7.4 to 7.15.

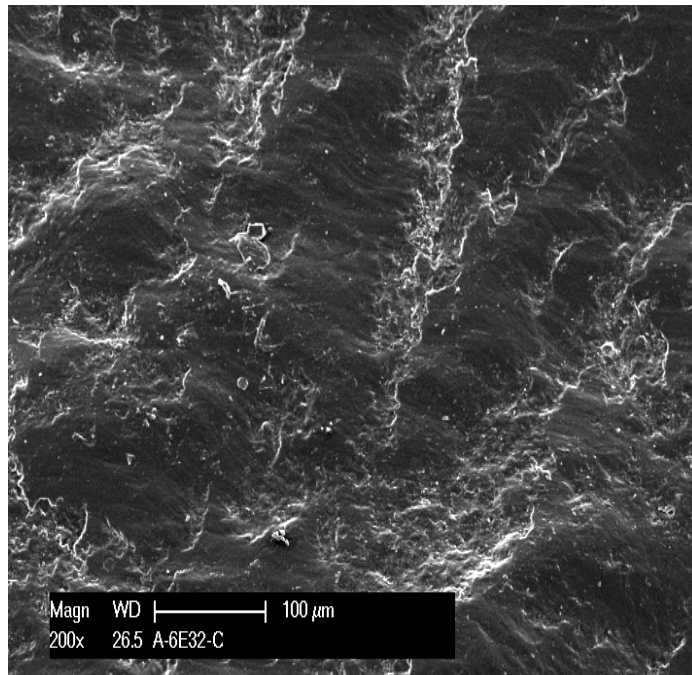


Figure 7. 4: Buccal enamel surface. The control group (x 200).

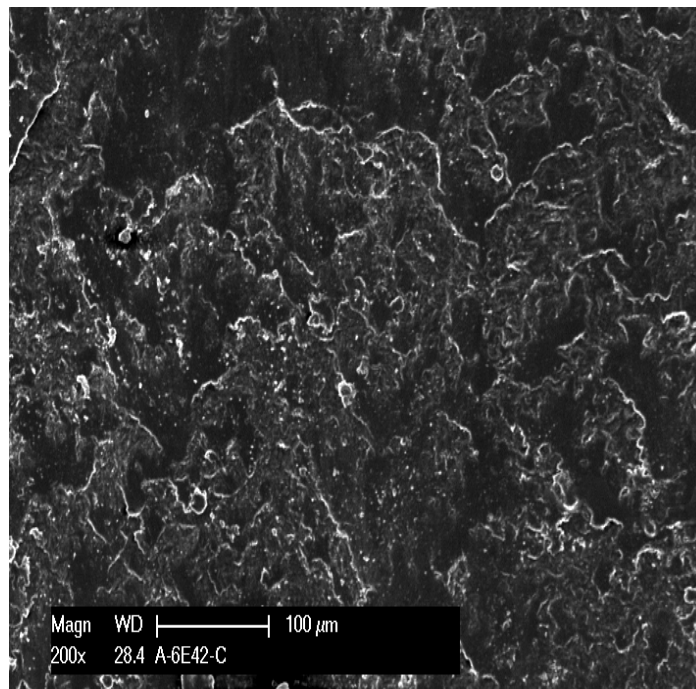


Figure 7. 5: Lingual enamel surface. The control group (x 200).

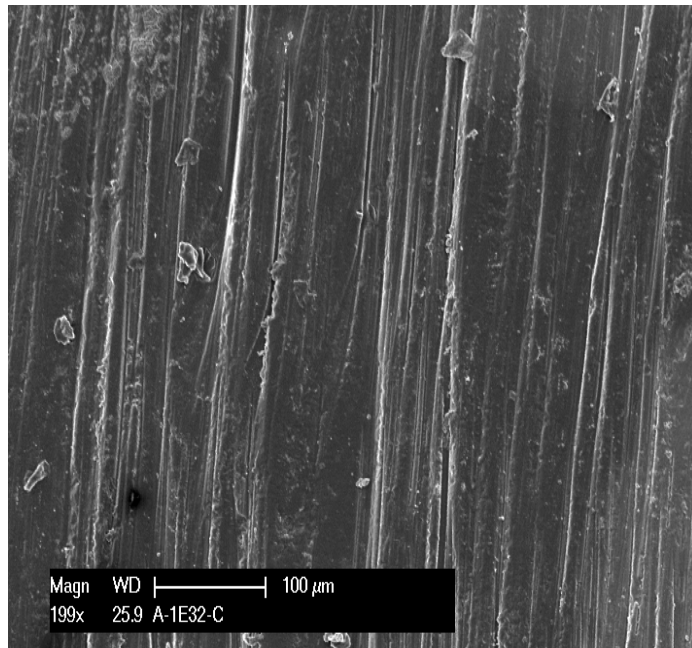


Figure 7. 6: Surface of enamel wearing against PBM-veneering (x 200).

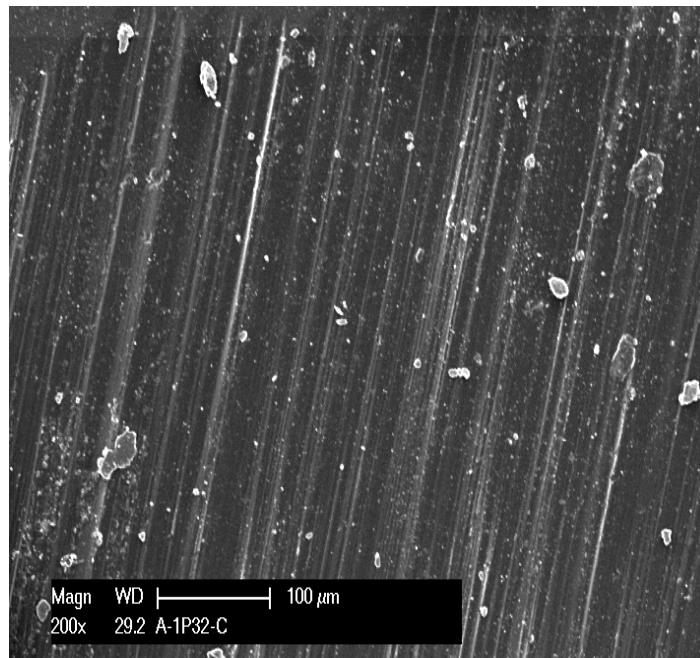


Figure 7. 7: Surface of PBM-veneering (x 200).

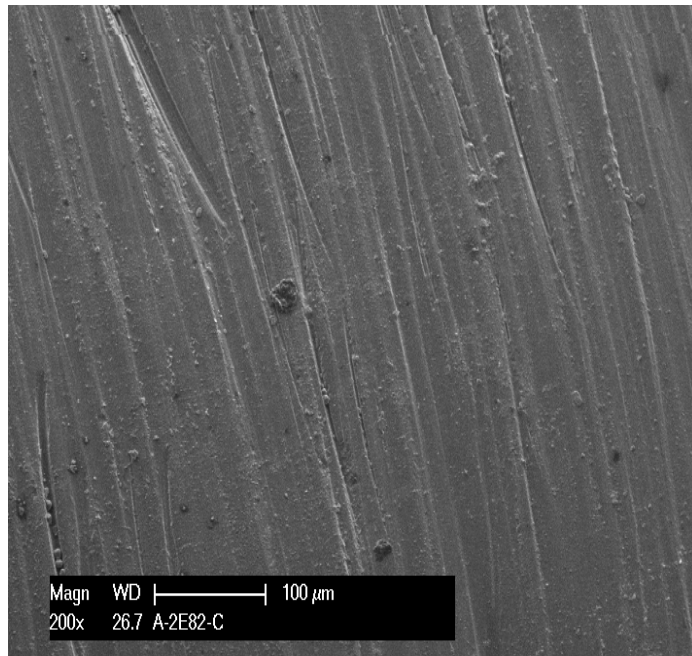


Figure 7. 8: Surface of enamel wearing against LR-veneering (x 200).

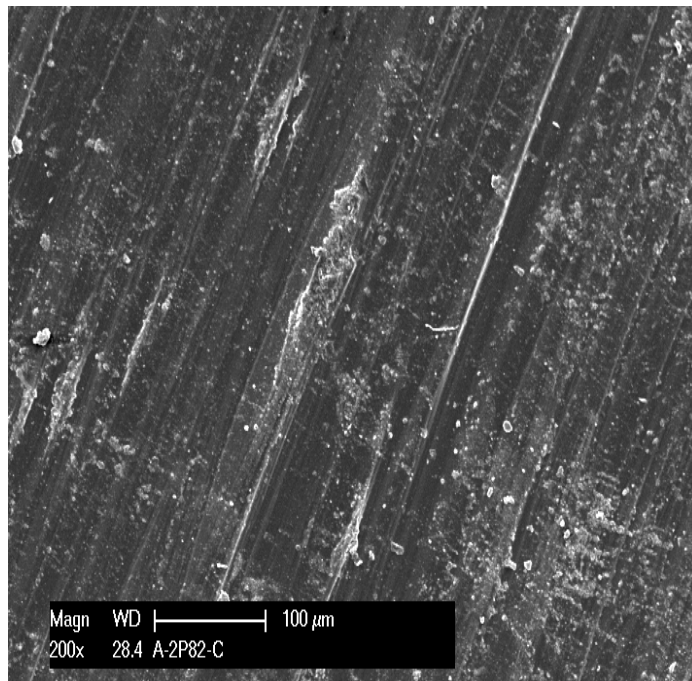


Figure 7. 9: Surface of LR-veneering (x 200).

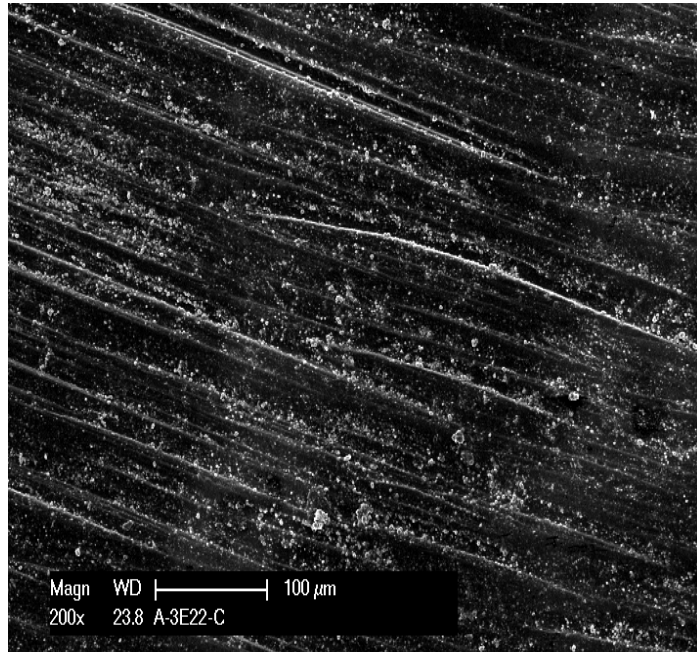


Figure 7. 10: Surface of enamel wearing against LR-pressable (x 200).

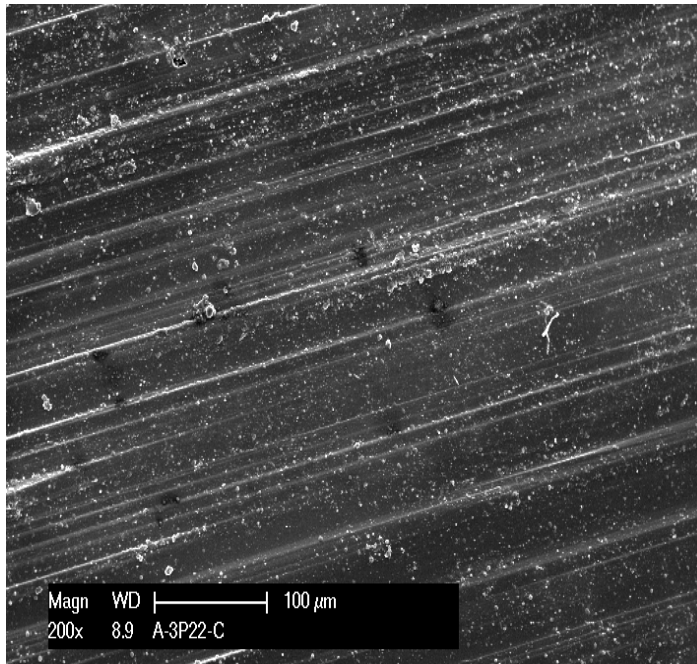


Figure 7. 11: Surface of LR-pressable (x 200).

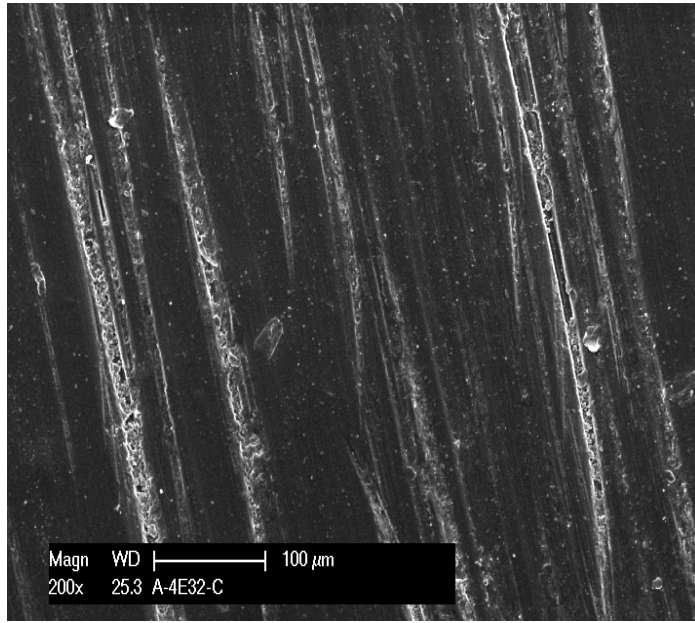


Figure 7. 12: Surface of enamel wearing against machinable ceramic (x 200).

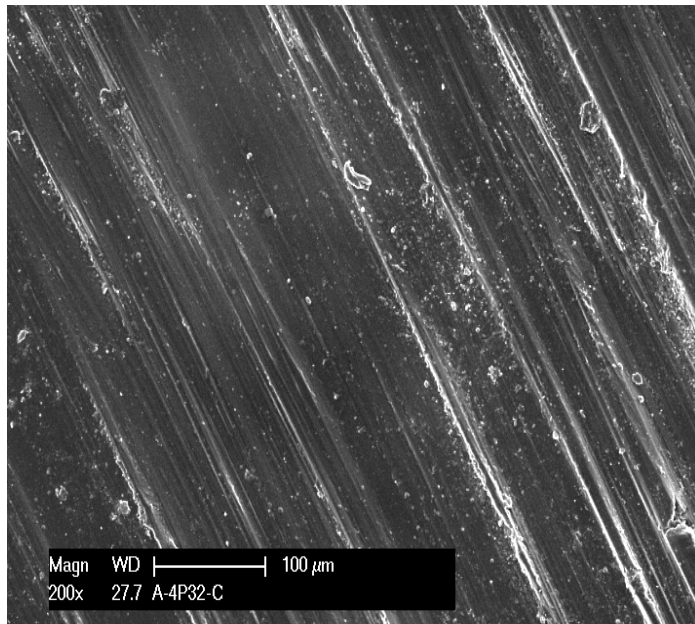


Figure 7. 13: Surface of machinable ceramic (x 200).

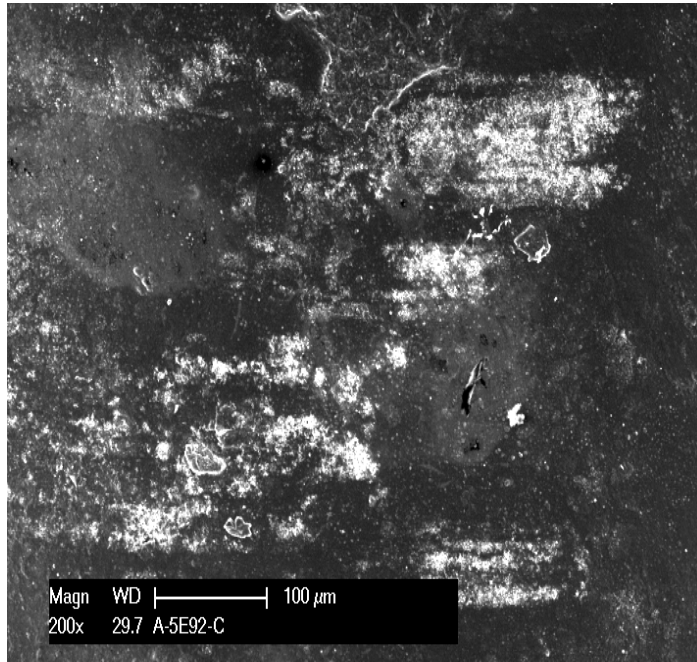


Figure 7. 14: Surface of enamel wearing against gold (x 200).

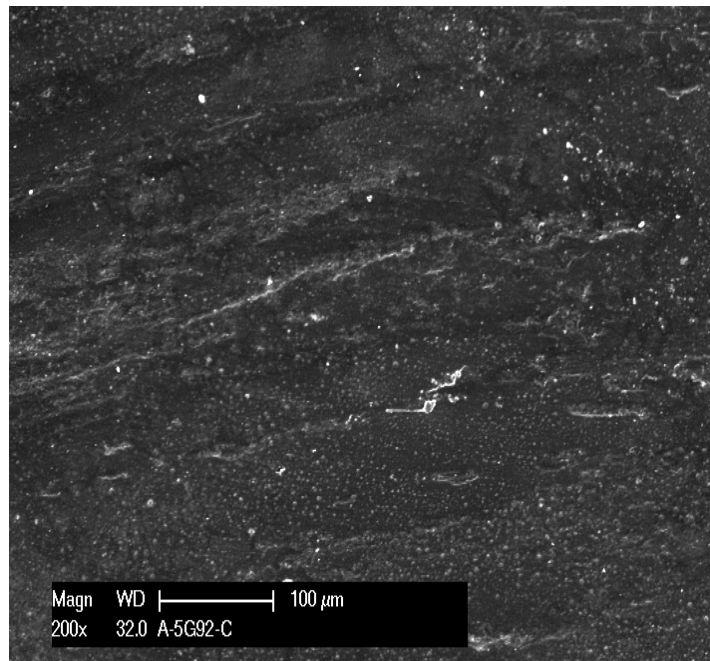


Figure 7. 15: Surface of gold (x 200).

7.3 Experimental results at pH 1.2

7.3.1 Quantitative assessment at pH 1.2

This experiment explored the wear rates of enamel and different indirect materials in a low pH environment (pH=1.2) under a load of 100N in 10,000 cycles.

The mean wear rate of enamel in the control group in which the buccal and lingual half of the same tooth wearing against each other was at 0.35 mm³ per 1,000 cycles. Table 7.7 presents descriptive statistics of the **mean wear rates of enamel** opposed by different indirect materials in low pH, and Fig. 7.16 shows the mean wear rates with the corresponding error bars.

Table 7. 7: Mean wear rates of enamel opposed by different restorative materials under a load of 100N at pH 1.2.

Opposing material	n	Mean	SD	Range	SE
		(Enamel Wear)			
		(mm³/1000cycles)			
PBM-veneering	9	0.60	0.16	0.43 to 0.96	0.05
LR-veneering	10	0.52	0.14	0.25 to 0.73	0.04
LR-pressable	8	0.90	0.31	0.44 to 1.37	0.11
Machinable ceramic	8	0.80	0.19	0.44 to 0.99	0.07
Gold	10	0.35	0.15	0.19 to 0.56	0.05
Enamel control specimens	10	0.36	0.12	0.20 to 0.56	0.04

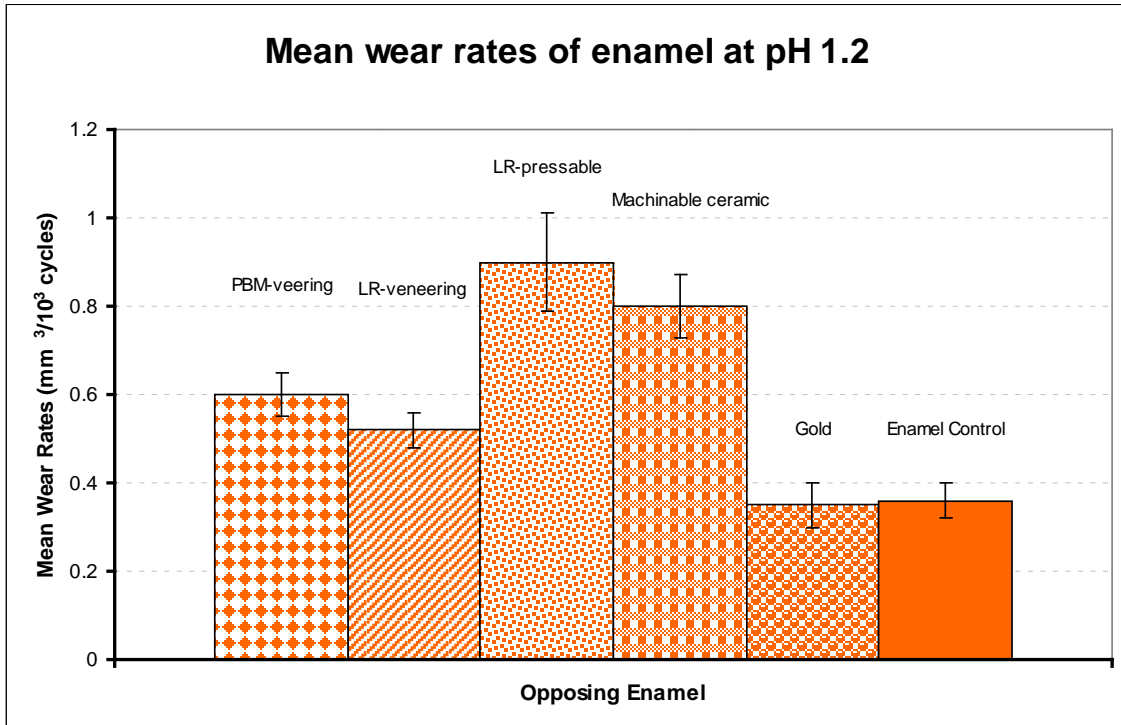


Figure 7. 16: Mean wear rates of enamel (with standard error bars) wearing against different indirect restorative materials under a load of 100N at pH 1.2.

One way analysis of variance showed significant differences between the mean wear rates of enamel between groups ($p < 0.01$). The Bonferroni test was used to compare the mean wear rate of enamel of one group to the others. The result showed that while the mean wear rates of enamel worn by LR-pressable and machinable ceramic were statistically significantly different from the control group, enamel specimens worn by PBM-veneering, LR-veneering and gold were comparable to the controls. This is presented in Table 7.8.

Table 7. 8: Pair-wise comparison of mean wear rates of enamel worn by different indirect materials by Bonferroni test (load 100N, pH 1.2).

Opposing material	PBM-veneering	LR-veneering	LR-pressable	Machinable ceramic	Gold
LR-veneering					
LR-pressable	$p < 0.05$	$p < 0.01$			
Machinable ceramic		$p < 0.05$			
Gold			$p < 0.01$	$p < 0.01$	
Enamel control			$p < 0.01$	$p < 0.01$	

The descriptive statistics of the mean **wear rates of different indirect materials** wearing against enamel are summarized and presented in Table 7.9. Fig. 7.17 shows mean wear rates with the corresponding standard error bars for each material. It was found that at low pH material wear was minimal. The highest mean wear rate was for Duceram Kiss ($0.04 \pm 0.05 \text{ mm}^3$), whereas there was almost no wear for the remaining materials.

Table 7. 9: Mean wear rates of different indirect restorative materials under a load of 100N at pH 1.2.

Material	n	Mean (mm ³ /1000 cycles)	SD	Range	SE
PBM-veneering	9	0.04	0.05	-0.01 to 0.12	0.02
LR-veneering	10	0.01	0.05	-0.09 to 0.11	0.02
LR-pressable	8	-0.03	0.06	-0.11 to 0.07	0.02
Machinable ceramic	8	-0.01	0.16	-0.33 to 0.17	0.06
Gold	10	-0.02	0.05	-0.09 to 0.04	0.01

Analysis of variance showed no significant differences of the mean wear rates between materials at pH 1.2

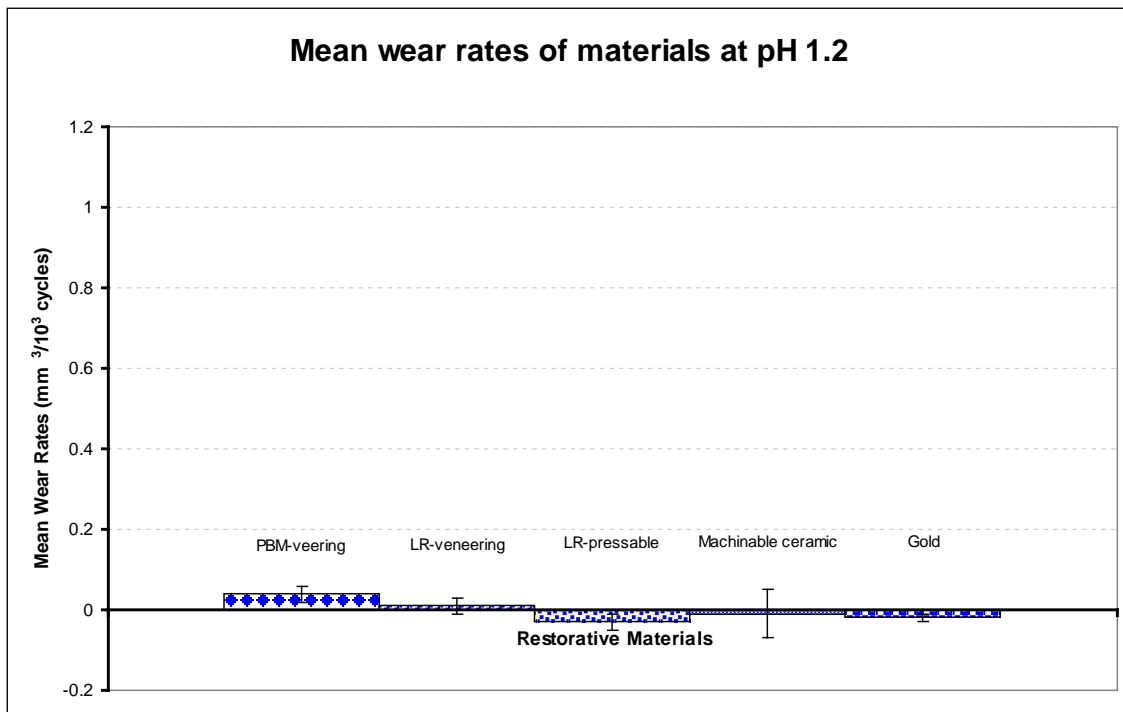


Figure 7. 17: Mean wear rates of different indirect materials (with standard error bars) opposing enamel under a load of 100N at pH 1.2.

NB*: the reported negative mean wear rates of LR-pressable, machinable ceramic and gold indicated that they are so small and below the resolution of the scanner

A t-test was used to compare the mean wear rates of enamel and the opposing material within the same group at pH 1.2. The data are presented in Table 7.10. It was found that the mean wear rates of enamel were statistically higher than those of the opposing materials in all groups. The largest difference was for enamel wearing against LR-pressable, and the smallest was for gold.

Table 7. 10: Comparison of the mean wear rates of enamel and the opposing material within groups (load 100N, pH 1.2).

		Materials	t-test
Enamel	<i>versus</i>	PBM-veneering	p<0.01
		LR-veneering	p<0.01
		LR-pressable	p<0.01
		Machinable ceramic	p<0.01
		Gold	p<0.01

The mean wear rates of enamel and the opposing materials in all groups are presented in Fig. 7.18 to give a total picture of the results of the experiment at pH 1.2.

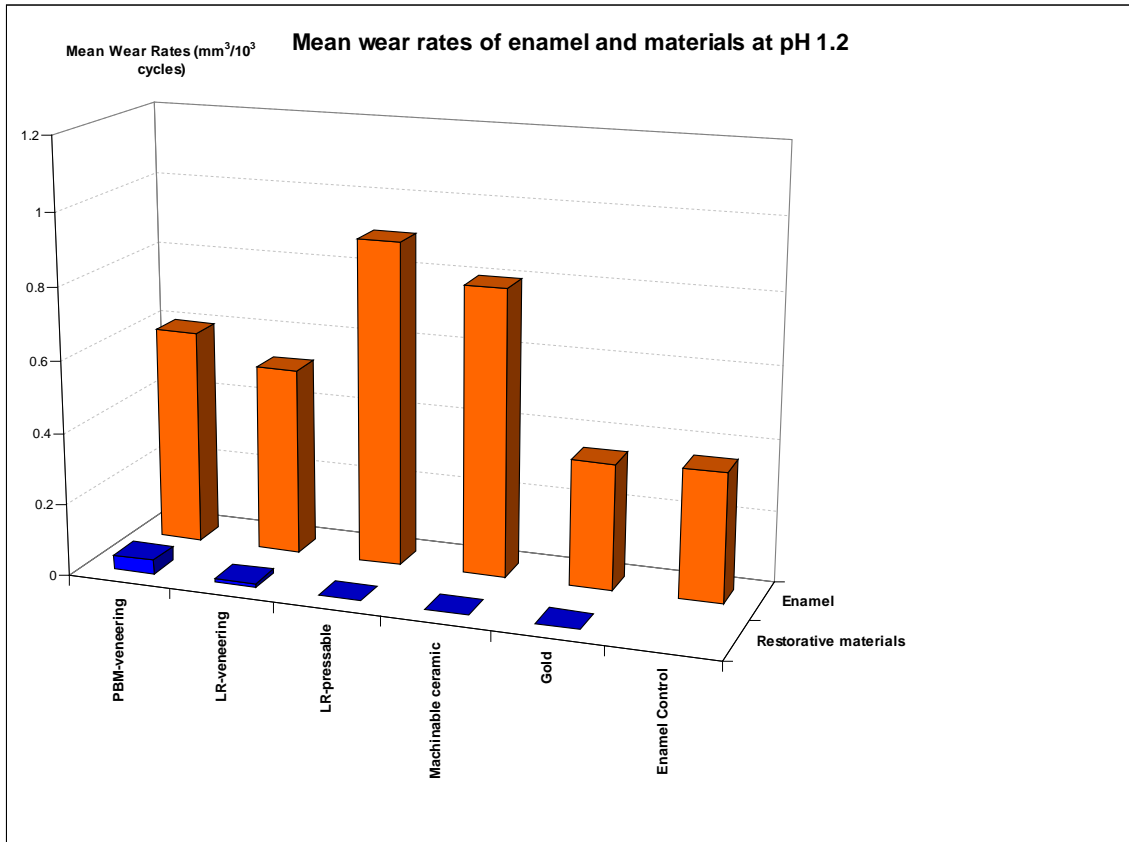


Figure 7. 18: The mean wear rates of enamel and different indirect restorative materials under a load of 100N at pH 1.2.

7.3.2 Qualitative assessment at pH 1.2

Micro-surface morphology of the specimens in the wear experiments at pH 1.2 showed different characteristics compared to specimens worn at pH 6.1.

It was observed that the enamel specimens of all groups showed smooth, wavy surfaces characteristics of erosion. No clear wear pattern was present on any of the specimens. At higher magnification, acid etching and the superimposed wear revealed the prism structure of the enamel and the ends of the exposed rods showing evidence of flattening associated with the concurrent wear.

Gold presented with relatively smooth surfaces, which was similar to that observed at pH 6.1. Porcelain including PBM-veneering, LR-veneering and LR-pressable showed surfaces with light striations running parallel to the movement of the wear machine. Surface debris were much less than what was observed in the experiment with pH 6.1.

The surfaces of machinable ceramic were not similar to other selected porcelain systems. Besides the light wear striations, the surfaces showed a large amount of surface porosities. These were consistently spread over the surfaces that were worn in the low pH environment.

The observations are presented in Figs 7.19 to 7.30.

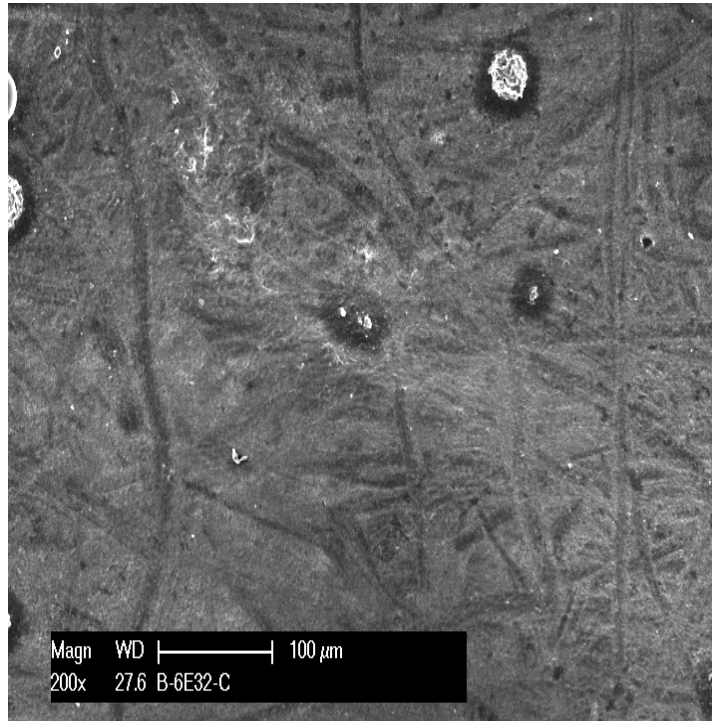


Figure 7. 19: Buccal enamel surface. The control group (x 200).

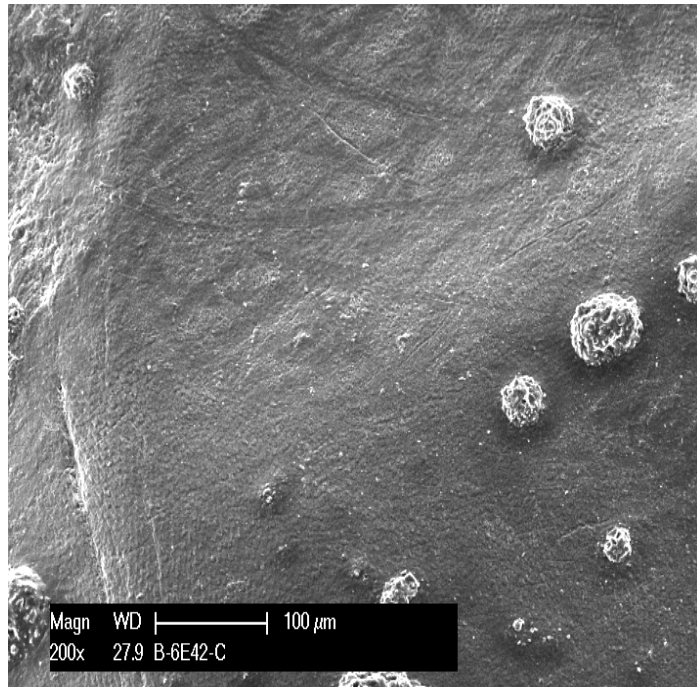


Figure 7. 20: Lingual enamel surface. The control group (x 200).

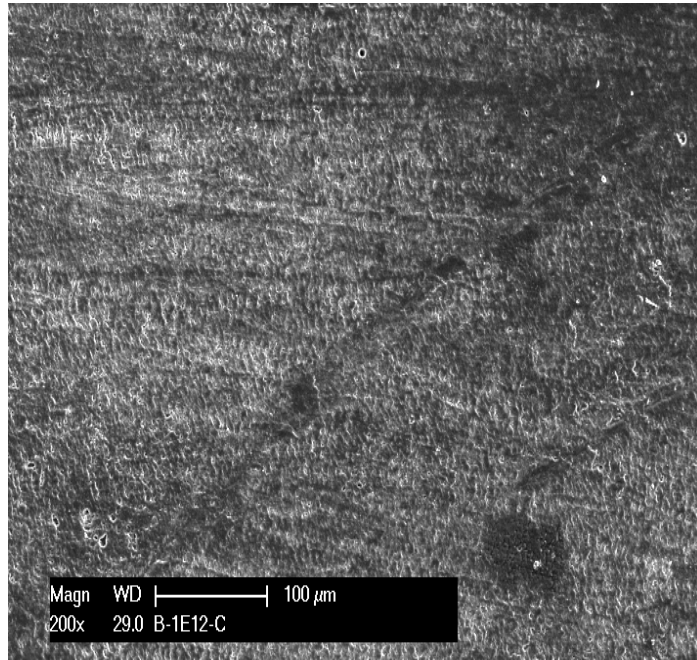


Figure 7. 21: Surface of enamel wearing against PBM-veneering (x 200).

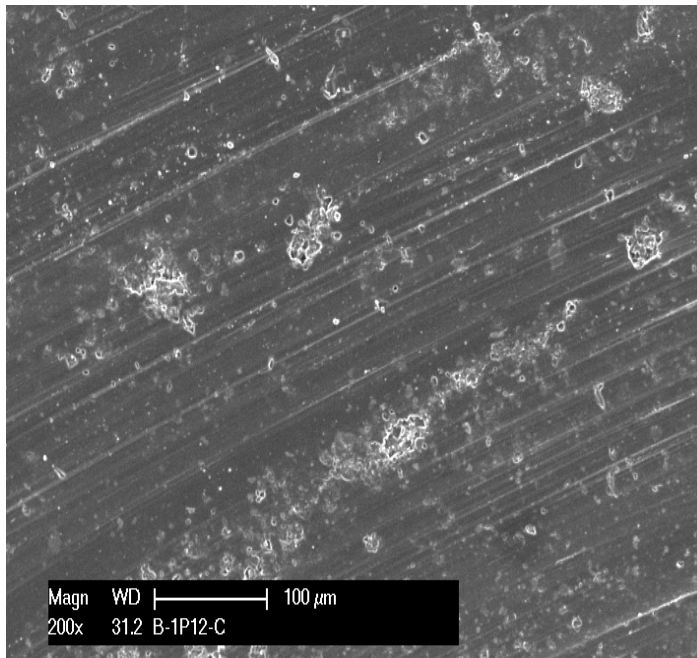


Figure 7. 22: Surface of PBM-veneering (x 200).

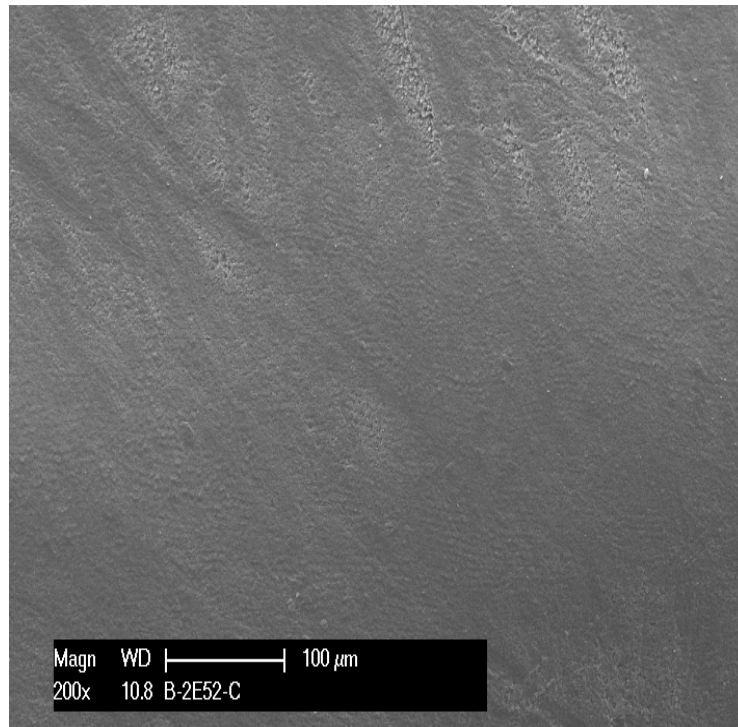


Figure 7. 23: Surface of enamel wearing against LR-veneering (x 200).

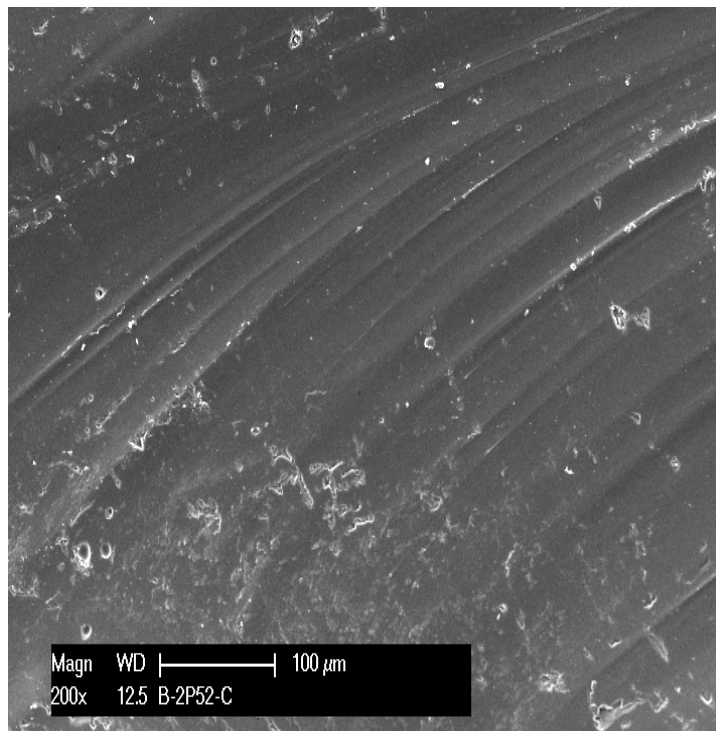


Figure 7. 24: Surface of LR-veneering (x 200).

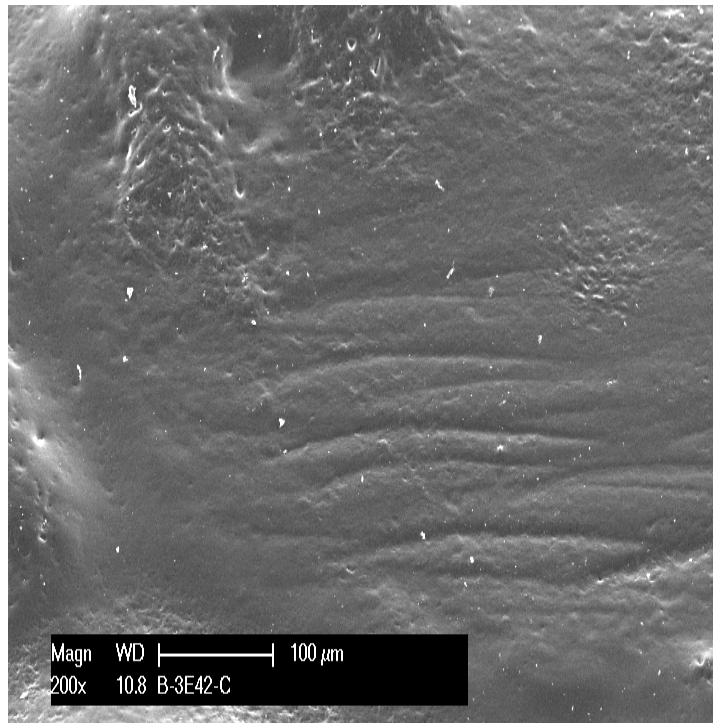


Figure 7. 25: Surface of enamel wearing against LR-pressable (x 200).

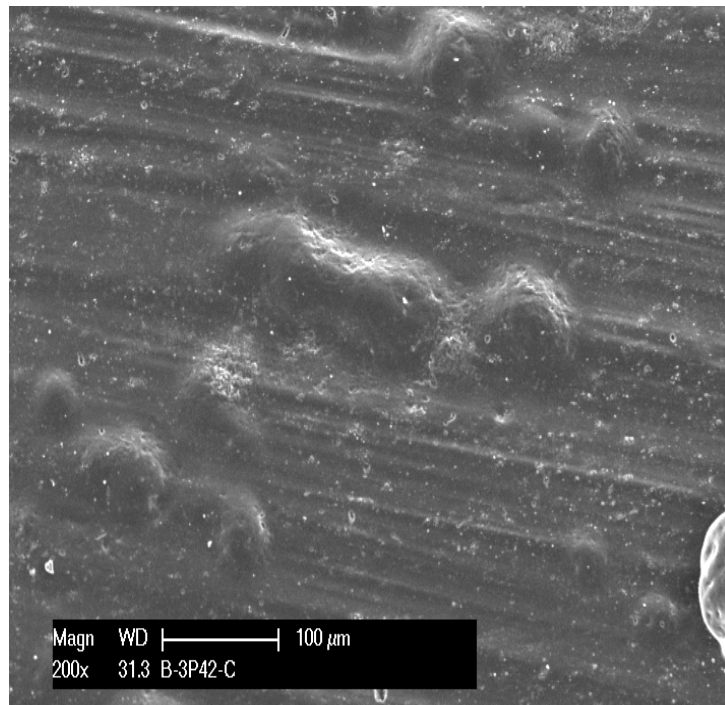


Figure 7. 26: Surface of LR-pressable (x 200).

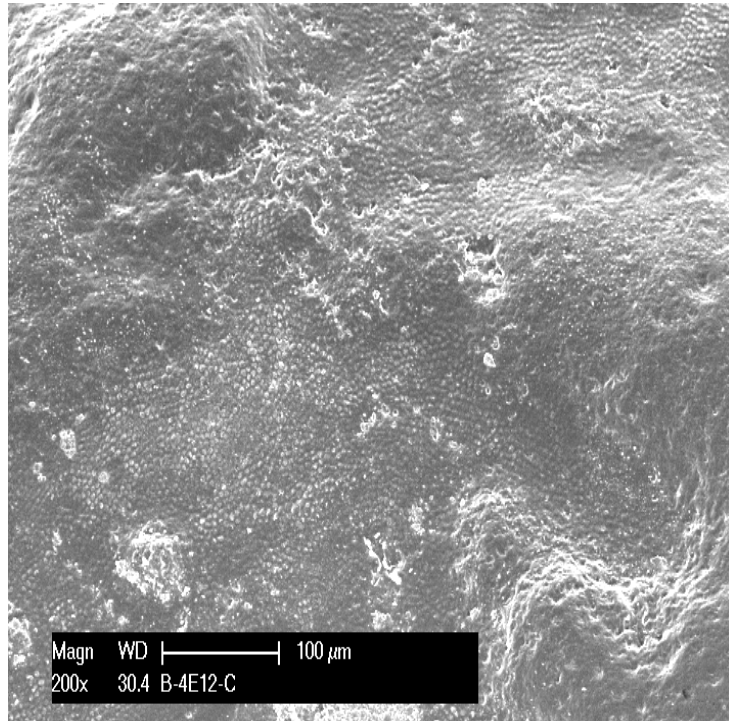


Figure 7. 27: Surface of enamel wearing against machinable ceramic (x 200).

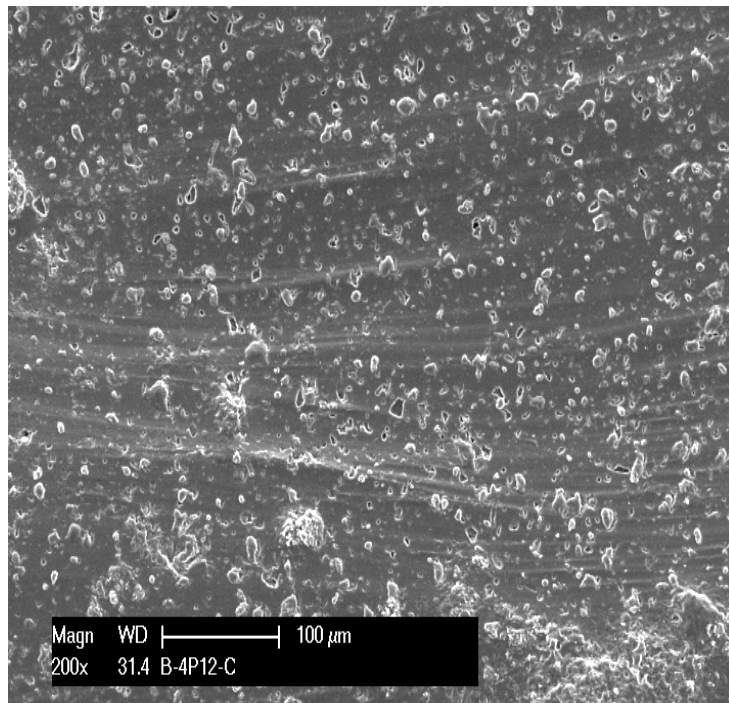


Figure 7. 28: Surface of machinable ceramic (x 200).

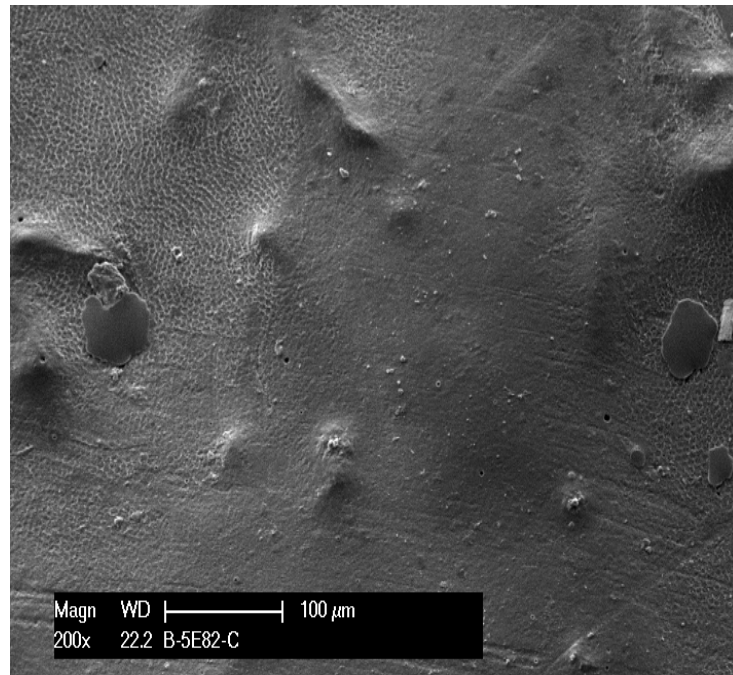


Figure 7. 29: Surface of enamel wearing against gold (x 200).

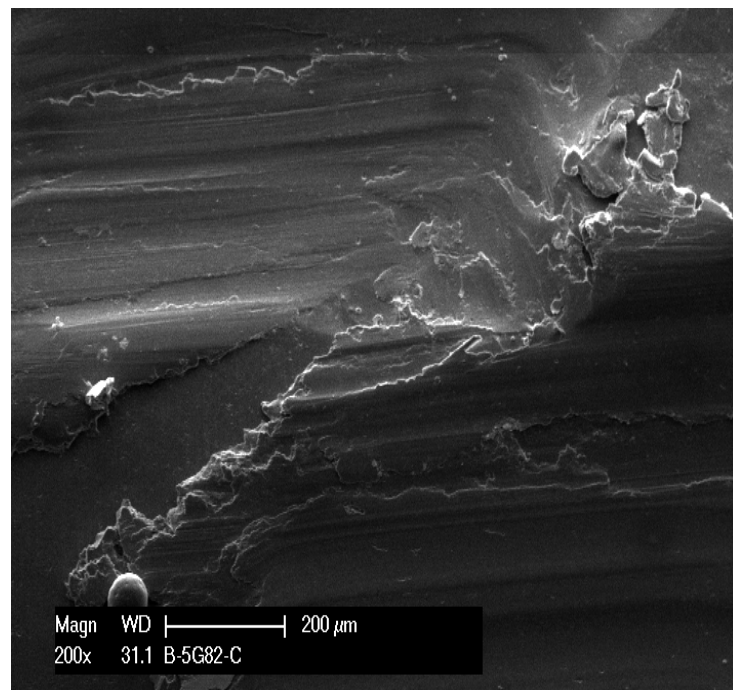


Figure 7. 30: Surface of gold (x 200).

7.4 Summary of results

A comparison of the mean wear rates of enamel and various indirect restorative materials in different environments is provided in Figs. 7.31 and 7.32. A similar summary of mean wear rates, sample sizes and standard deviations is given in Table 7.11.

Table 7. 11: Summary of both experiments including materials, sample sizes (n), mean wear rates (x) and standard deviation (SD).

Materials		pH = 6.1			pH = 1.2		
		n	x	SD	n	x	SD
PBM-veneering	Enamel	10	0.12	0.04	9	0.60	0.16
	Material	10	0.12	0.05	9	0.04	0.05
LR-veneering	Enamel	9	0.09	0.05	10	0.52	0.14
	Material	9	0.21	0.12	10	0.01	0.05
LR-pressable	Enamel	9	0.21	0.12	8	0.90	0.31
	Material	9	0.10	0.05	8	-0.03	0.06
Machinable ceramic	Enamel	8	0.09	0.08	8	0.80	0.19
	Material	8	0.07	0.04	8	-0.01	0.16
Gold	Enamel	9	0.00	0.04	10	0.35	0.15
	Material	9	0.03	0.07	10	-0.02	0.04
	Enamel Control	10	0.09	0.05	10	0.36	0.12

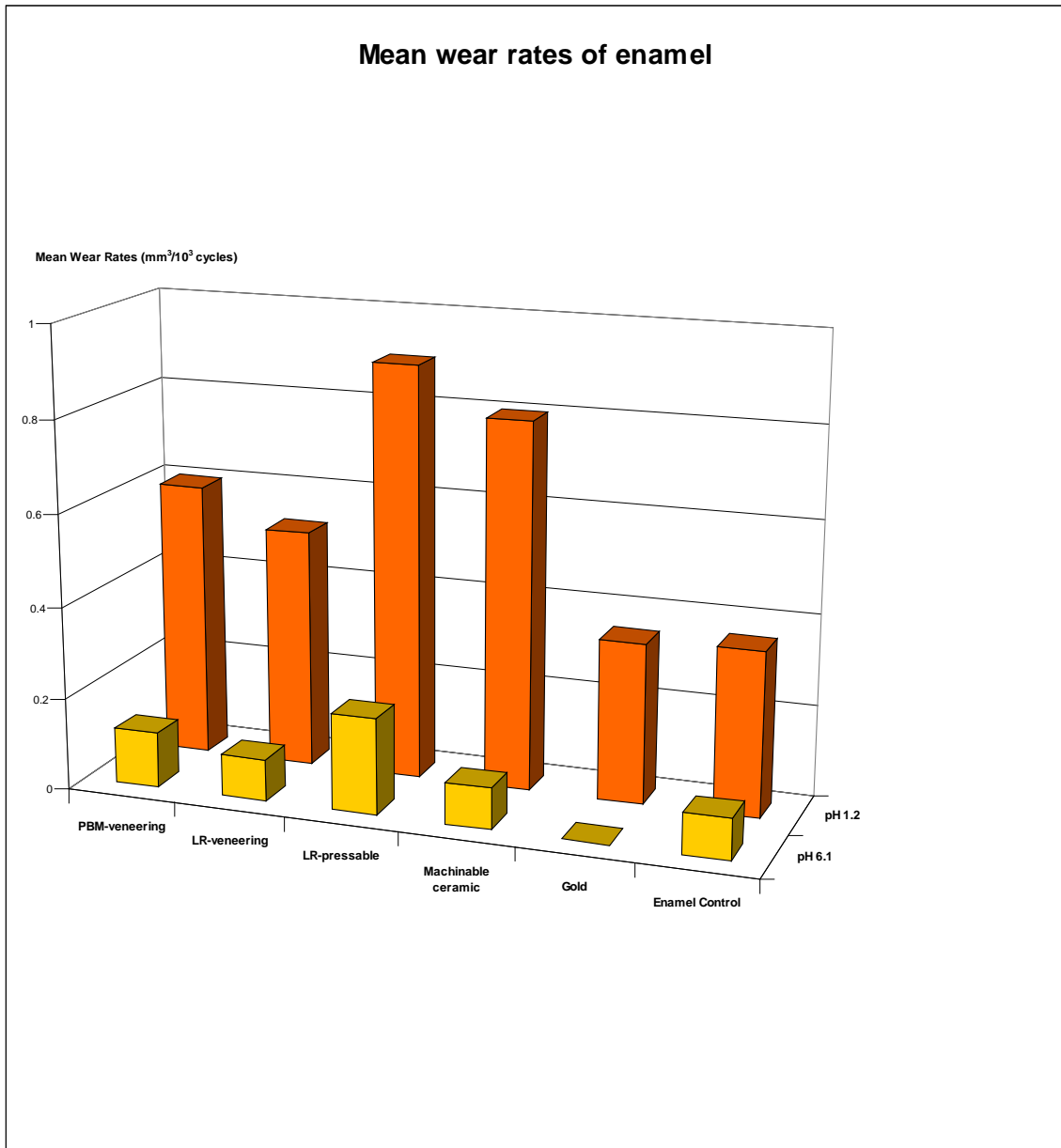


Figure 7. 31: Mean wear rates of enamel worn by different indirect materials in two acidic environments.

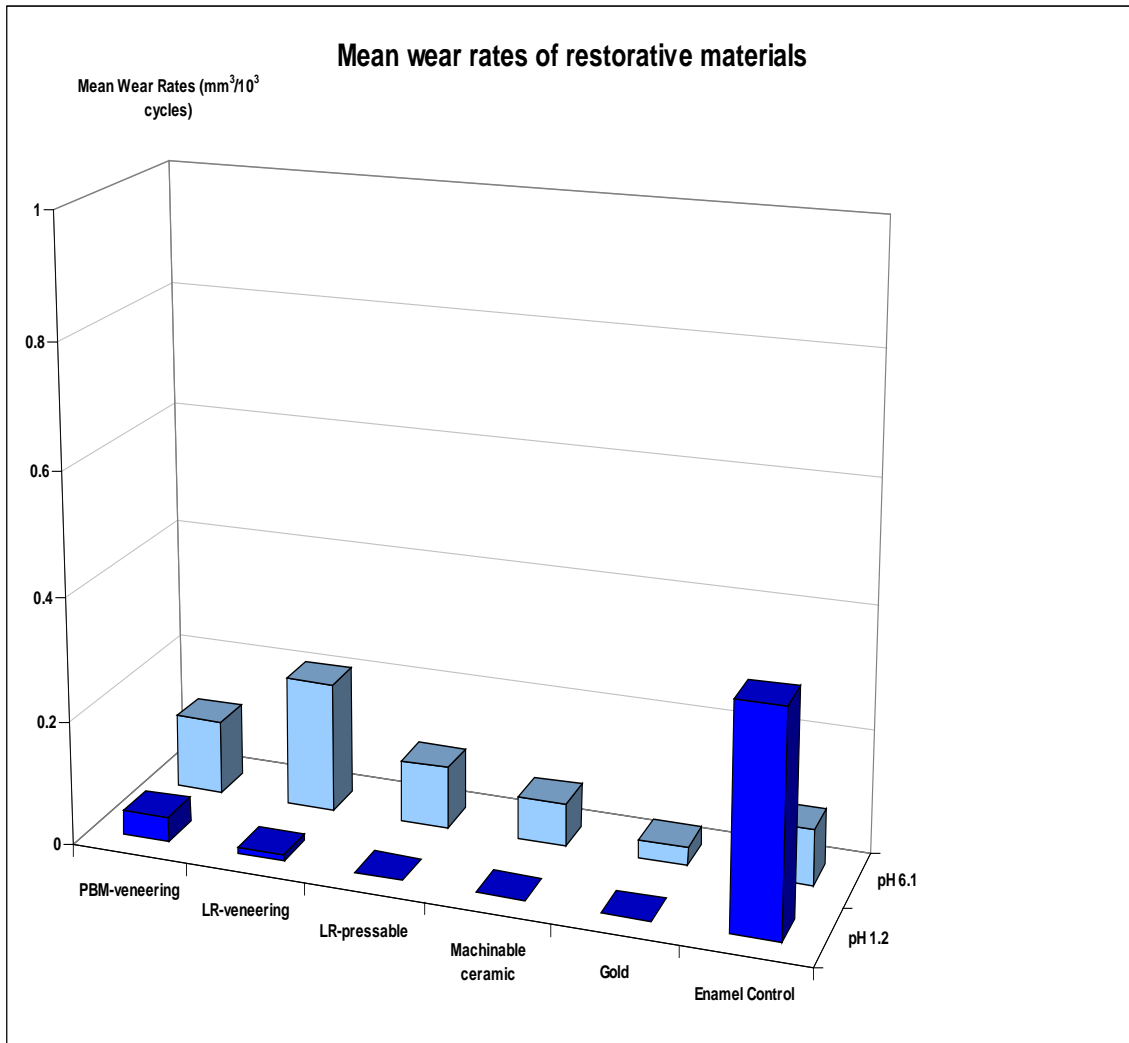


Figure 7. 32: Mean wear rates of different indirect materials opposing enamel in two acidic environments.

A t-test was used to compare the mean wear rates of enamel and the materials at different pHs. There were significant differences in the mean wear rates of enamel at near neutral and acidic pH ($p < 0.01$), regardless of the opposing materials. For the indirect restorative materials, statistically significant differences were only found with PBM-veneering, LR-veneering and LR-pressable. The results are summarized and presented in Table 7.12.

Table 7. 12: A t-test comparing the wear rates of enamel and different indirect restorative materials at pH 6.1 and 1.2.

Materials	Significant differences
Enamel Control	pH = 6.1: Sig. Diff. to pH = 1.2
Enamel (wearing against) PBM-veneering	pH = 6.1: Sig. Diff. to pH = 1.2
Enamel (wearing against) LR-veneering	pH = 6.1: Sig. Diff. to pH = 1.2
Enamel (wearing against) LR-pressable	pH = 6.1: Sig. Diff. to pH = 1.2
Enamel (wearing against) machinable ceramic	pH = 6.1: Sig. Diff. to pH = 1.2
Enamel (wearing against) gold	pH = 6.1: Sig. Diff. to pH = 1.2
PBM-veneering	pH = 6.1: Sig. Diff. to pH = 1.2
LR-veneering	pH = 6.1: Sig. Diff. to pH = 1.2
LR-pressable	pH = 6.1: Sig. Diff. to pH = 1.2
Machinable ceramic	Not Sig. Diff. between various pHs
Gold	Not Sig. Diff. between various pHs

In general, the trend showed that the wear rates of enamel increased when the pH reduced from 6.1 to 1.2 and those of opposing materials decreased when the lubricant became more acidic.

CHAPTER 8

DISCUSSION

This study simulated wear, in which the natural enamel and selected restorative materials were worn against each other without intervening exogenous materials. Strictly speaking, some microfine pieces of tooth and materials produced during the wear process would inevitably be incorporated between opposing surfaces thereby incorporating a third body. The degree to which this has occurred is not known.

The project's aims were to examine the mean wear rates of tooth enamel worn against four different porcelain systems and a gold alloy at various pH conditions compared to those of the control enamel specimens. At pH 6.1, the **mean wear rates of enamel** opposed by PBM-veneering ($0.12 \pm 0.01 \text{mm}^3$), LR-veneering ($0.09 \pm 0.02 \text{mm}^3$), machinable ceramic ($0.09 \pm 0.03 \text{mm}^3$) and gold ($0 \pm 0.01 \text{mm}^3$) were not statistically different to those of the control specimens ($0.09 \pm 0.02 \text{mm}^3$) (Fig 8.1). These results indicated that the selected restorative materials are “enamel-friendly” at a near neutral pH, which was different from the reported destructive effects of ceramic on enamel in some other studies (Metzler et al. 1999; Elmaria et al. 2006; Ghazal et al. 2008). The reduction in abrasiveness of porcelains on the natural enamel was probably the result of

improvements in the microstructure, chemical composition and physical properties of the currently used ceramic products tested in our study.

The lowest wear rates of enamel found in our experiments when it was opposing to gold was in line with a number of previous studies. Monasky and Taylor (1971) reported no significant difference in the wear rates of enamel opposing a type III gold and enamel opposing enamel. Furthermore there was reported less enamel wear caused by gold compared to that by other materials such as conventional feldspathic porcelain systems Ceramco or VMK 68 (Jacobi et al. 1991; Jagger and Harrison 1995; Hacker et al. 1996), or Vita Mark II, a machinable feldspathic porcelain (Ramp et al. 1997; 1999).

On the other end, LR-pressable ceramic seems to be an abrasive material at pH 6.1. The wear rates of the natural enamel opposed by LR-pressable ($0.21 \pm 0.04 \text{mm}^3$) was twice those of the control specimens ($0.09 \pm 0.02 \text{mm}^3$) (Fig. 8.1). This material is recommended by the manufacturer to be used as a core which is covered with veneering porcelain or as a pressed crown when fabricating an all-porcelain crown. Based on this study it can be recommended that instead of producing a crown entirely of the material, the technique of veneering a LR-pressable core with a porcelain that is not abrasive to enamel will be of benefit to the natural dentition. Furthermore care must be taken to prevent the exposure of the LR-pressable core material during occlusal adjustment, which could result in excessive enamel wear.

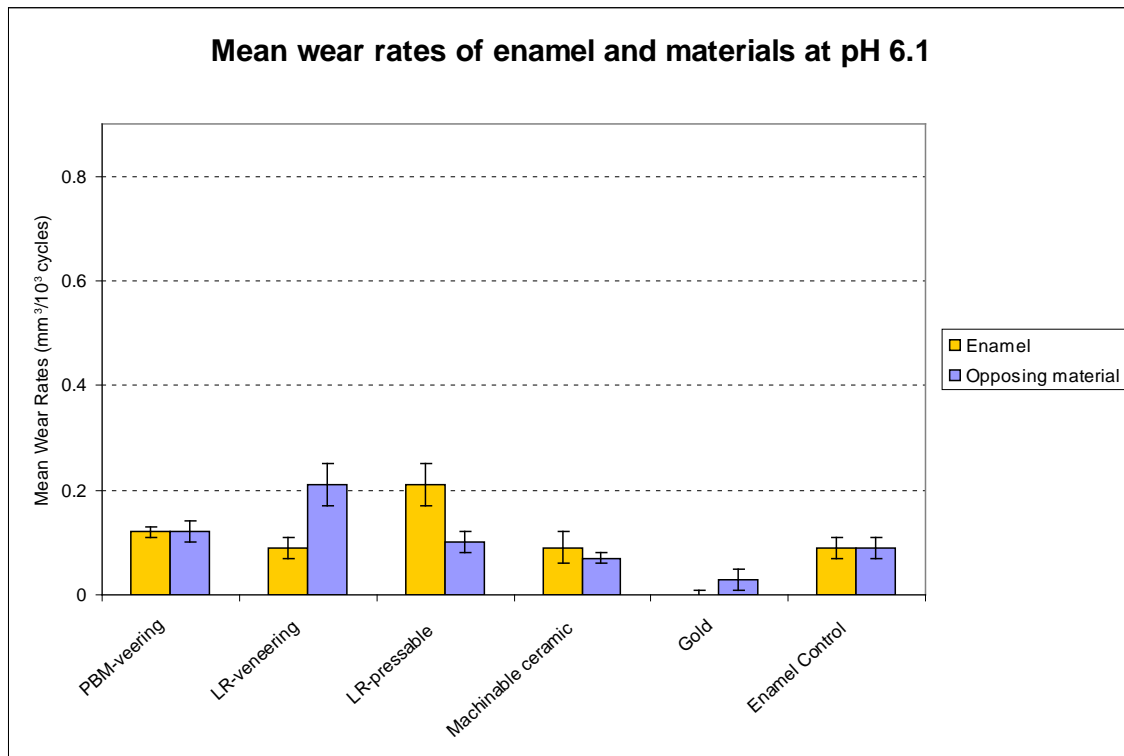


Figure 8. 1: Mean wear rates of enamel and opposing materials at pH 6.1 (previously presented in Fig. 7.3).

The selected **materials** wore at very different rates compared to the enamel antagonists after 120,000 cycles in deionised water (pH 6.1). The lowest wear rates were with gold ($0.03 \pm 0.02 \text{ mm}^3$) and the highest ones with LR-veneering ($0.21 \pm 0.04 \text{ mm}^3$). While the wear rates of LR-veneering were not significantly different to those of PBM-veneering ($0.12 \pm 0.02 \text{ mm}^3$), they were significantly higher than those of LR-pressable, machinable ceramic and gold. They doubled the wear rates of LR-pressable ($0.10 \pm 0.04 \text{ mm}^3$), tripled those of machinable ceramic ($0.07 \pm 0.03 \text{ mm}^3$) and quadrupled those of gold ($0.03 \pm 0.02 \text{ mm}^3$) (Fig. 8.1). Thus, gold demonstrated the best wear resistance and LR-veneering the least among the investigated materials.

A comparison of the mean wear rates of enamel and the opposing antagonists revealed that those of enamel and their PBM-veneering antagonists were not significantly different. The same was also true for machinable ceramic and gold groups. When the opposing restorative materials and their antagonists wear similarly, it can be expected that the occlusion can maintain its stability over the time (Ohlmann et al. 2007). However, this was not the case for the groups of LR-veneering and LR-pressable. It is very interesting that while the enamel wear rates were twice those of LR-pressable antagonists, they were only a half of those of LR-veneering material. This again indicated that LR-pressable was abrasive to the enamel in a near neutral pH and LR-veneering was low in wear resistance. These properties are not desirable because research has shown that two of the important requirements that render a restorative material successful are good occlusal wear resistance and not being abrasive to the natural dentition (Ghazal et al. 2008).

When the experiment was conducted at low pH, the **wear rates of enamel** increased dramatically. The 1.2 pH was chosen because it simulated the acidic oral environment of patients with regurgitation. Gastric reflux is strongly associated with tooth erosion and the acidity in the stomach may be below pH 1 (Jarvinen et al. 1988). With a change of the environment from a near neutral (pH 6.1) to acidic (pH 1.2), the volume loss of the enamel increased four-fold for the enamel controls (from 0.09 to 0.36mm³ per 1,000 cycles), or nine-fold for enamel opposing the machinable ceramic material (0.09mm³ at pH 6.1 to 0.80mm³ at pH 1.2 per 1,000 cycles). Nevertheless the highest enamel wear rates were caused by LR-pressable material (0.90mm³ per 1,000 cycles) (Table 8.2).

The results showed that un-veneered LR-pressable ceramic is abrasive to the enamel both in neutral and acidic environments.

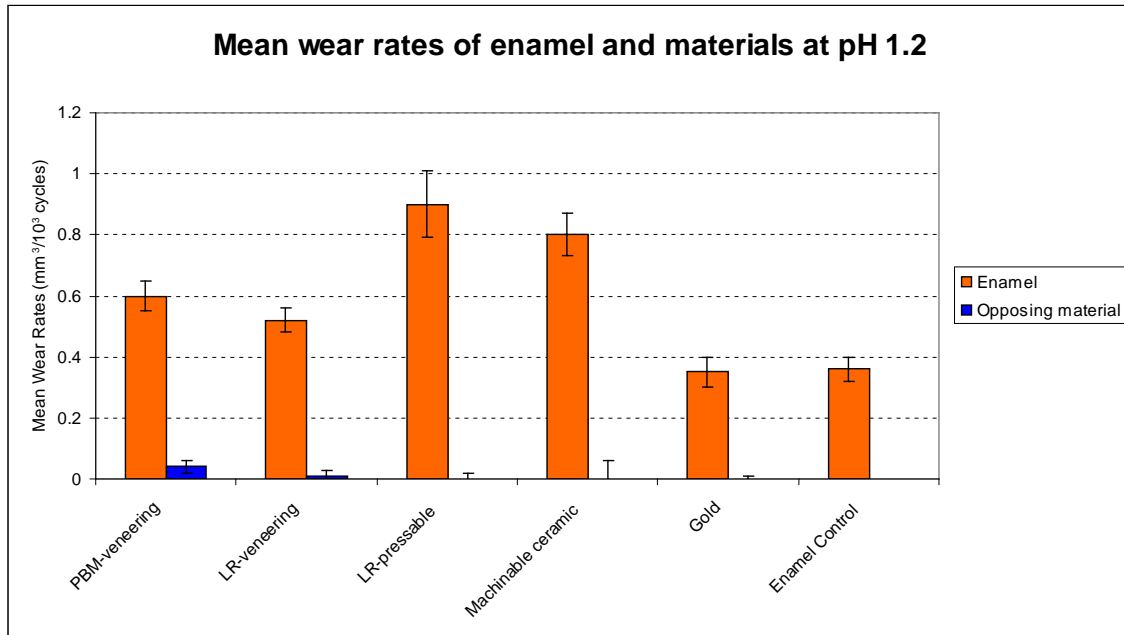


Figure 8. 2: Mean wear rates of enamel and opposing materials at pH 1.2 (previously presented in Fig. 7.18).

Although the rates of enamel wear associated with gold in an acidic environment were much higher than those observed at a near neutral pH, they were not significantly different to those of the enamel controls. This again confirms that gold is the best of the tested materials for occlusal restoration in terms of wear behaviour, even in an acidic mouth, and supports previous studies advocating that type III gold should be the indirect restorative material of choice because it is “enamel-friendly” and has good wear resistance characteristics (Ramp et al. 1997).

The ductility of gold alloy and the smooth surface that cast metals appear to have could explain why gold demonstrated good wear resistance compared to porcelain systems (Mair et al. 1996; Kadokawa et al. 2006). According to Kadokawa et al. (2006), the lower wear of gold could be due to an inherent ductility provided by metal bonds in the structure. SEM observation revealed that gold alloy was pushed to both sides of the wear tracks rather than wearing and breaking away (Fig. 8.3). Moreover, it was observed that there was a grayish-golden deposit covering the wear facets of tooth specimens after wearing for a period of time, which was also noticed in the study by al-Hiyasat et al. (1998) and Hacker et al. (1996). This was similar to the wear between amalgam and enamel where a smear of amalgam was seen on the enamel specimens (Wassell et al. 1994). The transfer of a gold layer to the opposing antagonist indicates that the wear mechanism between enamel and gold includes a component of adhesive wear. The volume of the transferred material was reported proportional to the contact area and the sliding distance (Mair et al. 1996). This layer may act as a lubricant or protective layer to reduce the abrasive effect of the material antagonist on the enamel surface (Ramp et al. 1997).

The crystal structure of metals is considered to have a very regular atomic pattern. However, this atomic arrangement can be interrupted to create defects (Crawford and Slifkin 1975). The point and line effects plus the easy movement of atoms are often seen with metals having malleability characteristics. Perhaps under the influence of stress caused by external loads, the gold atoms move and interact and absorb the force, hence reducing the amount of wear on both enamel and material (Ramp et al. 1997).

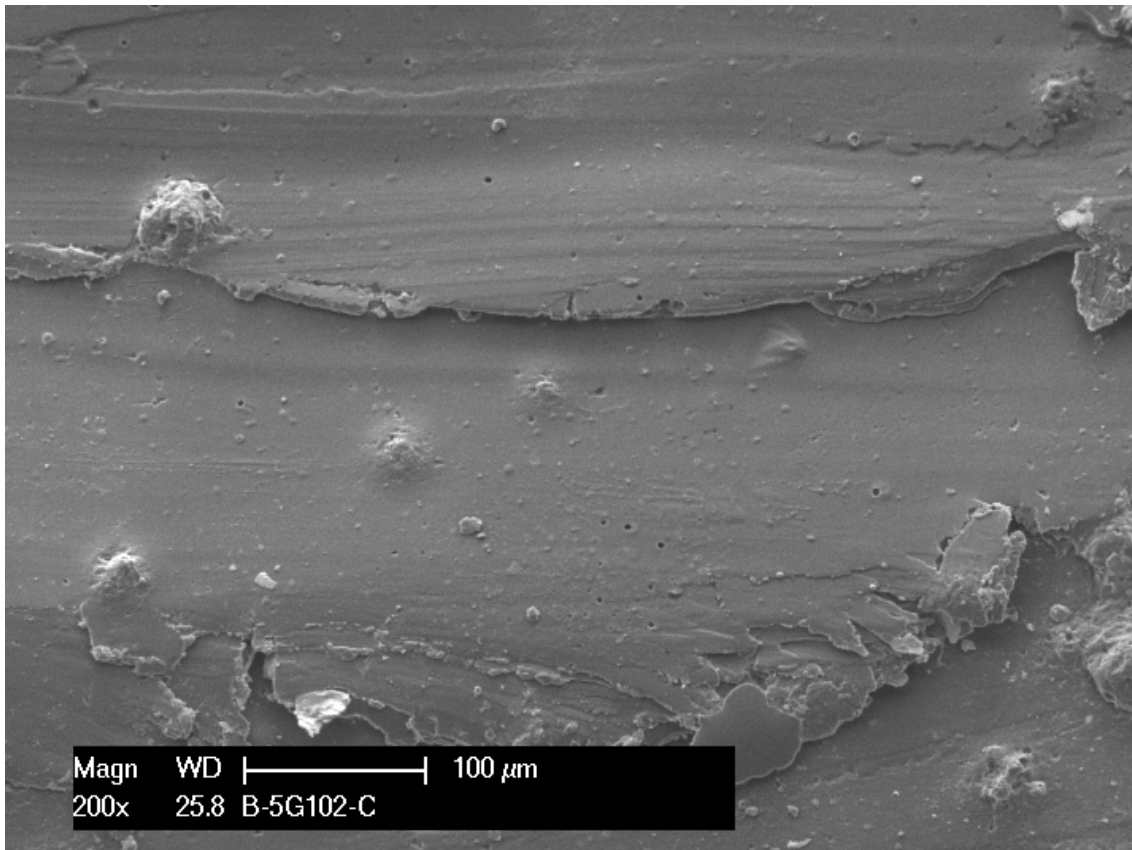


Figure 8. 3: Gold surface after wearing against enamel ($\times 200$).

The increase in wear of enamel at low pH could be expected because acidic conditions affect the solubility of the dental hard tissue. Larsen (1990) stated that the solubility could increase up to 7 or 8 times when the pH of the oral environment decreases from 6.5 to 5.5, a critical pH for enamel demineralization (Jarvinen et al. 1988; Larsen 1990). Acid dissolves apatite crystals, leading to severe or total destruction of the tooth structure (al-Hiyasat et al. 1998a). The demineralized and softened outer layer of enamel will be worn away more easily during the wear process, exposing the sound underlying enamel to be further affected directly by the acid in the environment (Eisenburger et al. 2003). This was shown on micrographs as smooth and wavy surfaces

of enamel at pH 1.2. The process repeats and as a result, a considerable amount of tooth structure can be removed. This confirms the reports in the literature that enamel wears more quickly on exposure to acid (Ratledge et al. 1994; Kaidonis et al. 1998; al-Hiyasat et al. 1998a; Shabaniyan and Richards 2002) and an acid-etched enamel is more susceptible to the effect of mechanical force (Eccles 1982).

The wear behaviour of the **material** antagonists at pH 1.2 was also different to that at pH 6.1. The volume loss of the materials was minimal for PBM-veneering and LR-veneering, and nearly no wear was found for LR-pressable, machinable ceramic and gold. This was probably because the softened demineralized enamel layer was too soft to wear the opposing antagonists. In addition, due to the accelerating wear of enamel, this experiment was conducted only for 10,000 cycles, which may not be long enough to cause any measurable wear on porcelain. In a preliminary study conducted by the author, the wear experiment had to be carried out for at least 120,000 cycles in pH 6.1 to allow the total wear to be measurable on the porcelain specimens. Furthermore, surface micromorphology observations suggested that the machinable ceramic behaves differently in a highly acidic environment and this would be further investigated in the next section.

The selected porcelain systems used in this study showed a difference in abrasive characteristics and wear resistance. Variables that influence the abrasive potential of ceramic wear include fracture toughness, porosities, crystal size, content, type and morphology and its distribution in the glass matrix, surface finish and the structure of

each phase of the materials (Powers and Craig 1972; Rice 1985; Wu and Rice 1985; Wu et al. 1985; Cho et al. 1989; Kon et al. 1994; Derand and Vereby 1999; Metzler et al. 1999; Imai et al. 2000; Della Bona and Anusavice 2002; Oh et al. 2002; Esquivel-Upshaw et al. 2006). On the other hand, both the enamel structure and hardness and the pH of the oral environment have their effects on the enamel wear by the porcelain counterpart (Kaidonis et al. 1998).

In regards to crystal size, it has been hypothesized that the porcelain containing larger particles would result in more irregularities on its surface under load and consequently higher abrasiveness. Our results however did not totally support this notion. Namely, LR-veneering and machinable ceramic which have crystal size in the 4 μ m range caused no more wear to the opposing enamel than the enamel controls, but LR-pressable ceramic also having similar crystal size induced a twice higher wear rate. Thus, factors other than crystal size may act alone or in combination with the crystal size to influence the porcelain abrasive characteristics.

The leucite particles were generally distributed heterogeneously, being clustered into grains (Cesar et al. 2008). It is thought that the more crystals a porcelain incorporates, the harder and more abrasive it is (Oh et al. 2002). It was however noticed in this study that the wear rates of enamel caused by PBM-veneering, LR-veneering and machinable ceramic were not significantly different while their leucite content was different from each other. Metal-ceramic restorations contain roughly 15 to 20 vol% leucite (Oh et al.

2002) as compared to the 30 volume % for the machinable ceramic. Thus the amount of crystals alone in a porcelain was also not indicative of its abrasiveness.

The hardness of porcelains is another parameter that may potentially contribute to its abrasiveness. Data from the literature suggests that the hardness alone is not directly related to the abrasive effect of the materials (Seghi et al. 1991; Dahl and Øilo 1994). With a brittle material like porcelain, wear does not occur by plastic deformation but by fracture (DeLong et al. 1986; Oh et al. 2002; Kadokawa et al. 2006). Repetitive loading on the porcelain surface cause subsurface cracks which propagate to the surface and eventually the piece of porcelain surrounded by the cracks dislodge, leading to surface irregularities (Imai et al. 2000; Kadokawa et al. 2006). In a study by Mair et al. (1996), sliding motion concurrent with load increased 10 times the chance for the formation of cracks in porcelain compared with that caused by load only. The fractured porcelain then forms debris as a third body that accelerates wear (Imai et al. 2000; Kadokawa et al. 2006). This process is called fatigue and abrasive wear (Mair et al. 1996). In addition, the enamel antagonists also produce hard debris which have the potential to cause abrasive wear on the porcelain (Kadokawa et al. 2006). Micrographs of porcelain surfaces before and after the wear process have supported this contention with the images of relatively smooth surfaces after the polishing procedure became rough and irregular after the wear process (Eisenburger and Addy 2002a) (Fig. 8.4).

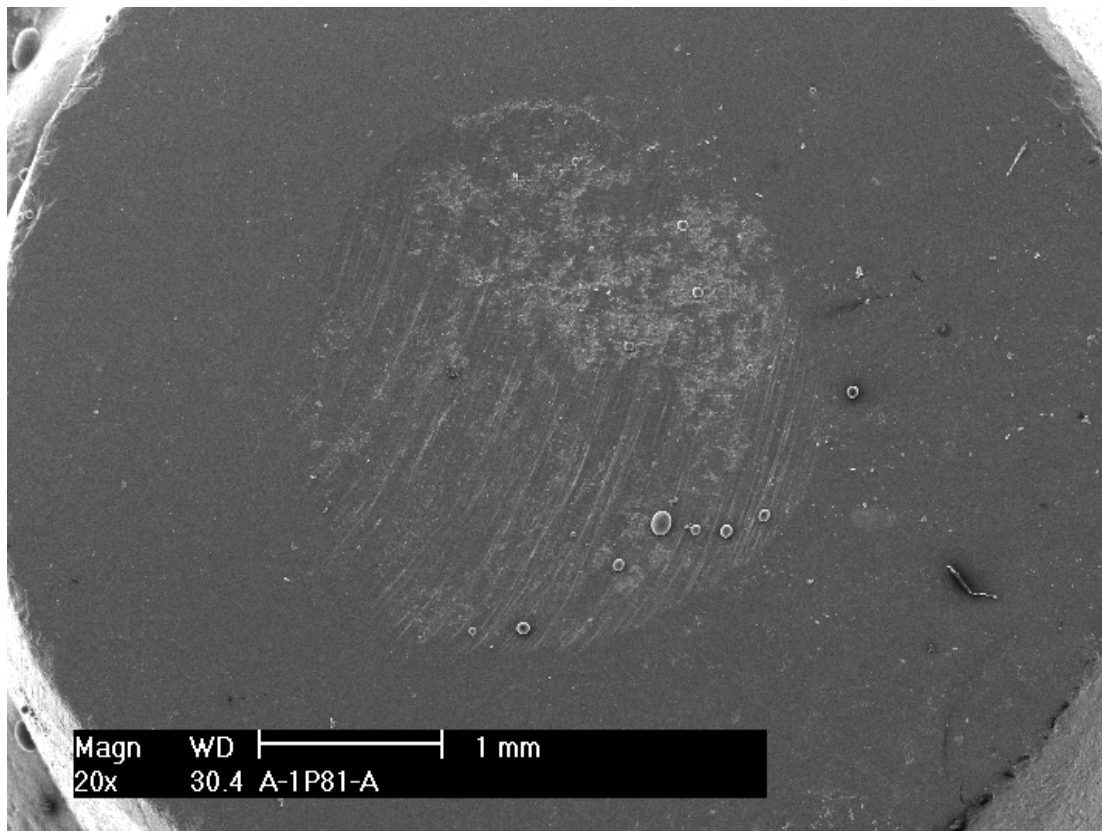


Figure 8. 4: The rough surface of porcelain after wear (in the middle) compared to the smooth surface after polishing ($\times 20$).

The wear rates measured in this study showed considerable variability which could be probably due to the source of tooth specimens. A variability can be present in tooth structure, morphology, physical characteristics, hardness and resistance to abrasion of the individual teeth (DeLong et al. 1989; Wassell et al. 1994). Importantly, the variability in teeth from different patients exists and should be taken into account (DeLong et al. 1989). Attempts to standardize enamel specimens to reduce the variation in the wear rate measurement have not always been successful (Krejci et al. 1992; al-Hiyasat et al. 1998b). Krejci et al (1992) reported that standardization not only failed to result in less variation compared to non-standardization enamel antagonists but also

yielded significantly different results both in the enamel and the opposing restorative materials.

Although sampling criteria were applied to include in our study only caries-free third molars with visibly “normal” crowns and no developmental defects, samples had to be collected from different patients with unknown history. To reduce the variability, the crowns were sectioned mesio-distally to give two specimens from each tooth, thus reducing the number of teeth required and minimizing the variation between specimens. Furthermore, all assessments of wear rates were made in the less variable secondary phase of wear, following removal of the outer surface layer that tends to vary in hardness between individuals. (Macpherson et al. 1991; Kaidonis et al. 1998).

In addition to the differences in the enamel specimens, porcelain discs could also vary. Except for the machinable ceramic which was produced by the manufacturer with quality control, all of the discs of the other selected porcelain systems were produced by a well-trained technician according to the recommendations of the manufacturer. However, air bubbles can easily be incorporated in the surface of PBM-veneering, LR-veneering and LR-pressable. Such variations in structural defects would contribute in part to the variation of wear rates of the opposing enamel.

The wear machines used in this study have been employed by other researchers to investigate the wear rates of enamel and materials under different loads and pH

conditions (Kaidonis et al. 1998; Burak et al. 1999; Shabani and Richards 2002; Ranjitkar et al. 2008). They were designed with a movable upper and a stabilized lower component. The upper component provided a vertical movement which was followed by the sliding motion. The action of impact caused by the vertical movement probably contributes in part to the wear in vitro. However, it was likely to be minimal and not have a marked contribution clinically because of the significant deceleration of the mandible before the teeth make contact (Bates et al. 1975; Wassell et al. 1994). Furthermore, the periodontium in the natural dentition would provide considerable stress relief and shock absorption which cannot be simulated on the wear machine (Eisenburger and Addy 2002a). Thus, it can be expected that results obtained with wear simulation might be an overestimation of the clinical wear.

It has been reported by Bates et al. (1975) that a rate of 80 cycles per minute is a reasonable estimation for the chewing cycle rate. This rate has also been used in previous wear studies (Kaidonis et al. 1998; Burak et al. 1999; Shabani and Richards 2002; Ranjitkar et al. 2008).

If assuming that 250,000 wear cycles in an artificial oral environment simulates one year of clinical wear in normal intraoral conditions (DeLong et al. 1986; 1992), the 120,000 cycles of wear in deionised water in this study would approximate 5.76 months which means just less than half a year, and the 10,000 cycles of wear in HCl lubricant at pH 1.2 would be 0.48 month or roughly two weeks. Although wear simulation might be an overestimation of the clinical conditions, the results of this study show how destructive

it could be for the natural dentition opposed by a porcelain prosthesis in a highly acidic mouth such as in the case of regurgitation.

The wear rates obtained in vitro could be different to what can be expected in the mouth because the role of saliva in the oral environment was not taken into account (West et al. 1998; 1999). Natural saliva has lubricating effects, although not all researchers have agreed on this (Douglas et al. 1985; Eisenburger and Addy 2002a). Its buffering and remineralizing effects are thought to have an important role in protecting the enamel from erosion, though these could all be lessened in an acidic condition (al-Hiyasat et al. 1998a; Hannig and Joiner 2006). There needs to be sufficient time (several hours) after an acid challenge for the remineralization or rehardening of the enamel to occur as this is a slow process (Eisenburger et al. 2001). If the abrasion removes the soften layer of enamel immediately after it has been demineralized, then remineralization has decreased chance to take effect (Eisenburger et al. 2000; 2001; 2003). In that case, severe wear can be expected.

In vivo wear studies are expensive and time consuming (Wassell et al. 1994; Ghazal et al. 2008). In addition, the variation between the oral environments of patients makes the results difficult to compare. One person may vary from the others in terms of diet, parafunctional habits etc (Ghazal et al. 2008). This has contributed in part to the over 100% standard deviation in clinical studies (Lambrechts et al. 1989; Ohlmann et al. 2007). Therefore, in vitro studies have been conducted to understand wear mechanisms, rank restorative materials by wear resistance, predict the clinical performance of a

material and compare new materials over a shorter time period (Wassell et al. 1994; al-Hiyasat et al. 1999; Ghazal et al. 2008). However, in vitro results should be extrapolated with care because wear experiments in artificial conditions can never completely simulate what happens in the natural mouth (de Gee and Pallav 1994; Mair et al. 1996; Eisenburger and Addy 2002b; Ghazal et al. 2008). In general, it seems that the wear results obtained from in vitro studies are more severe than the actual wear occurring on patients (Eisenburger and Addy 2002b).

SECTION THREE

EFFECT OF ACID ON MACHINABLE CERAMIC

CHAPTER 9

INTRODUCTION

The investigation of wear rates and facet micro-morphology revealed that the machinable ceramic material was involved in relatively rapid wear rates at pH 1.2 and that the specimens became porous at the lower pH.

As a result a second series of experiments were conducted to assess the effects of acid alone on this material and to determine whether the effect of acid was restricted to very low pHs or involved other less acidic conditions.

The aim of this investigation was to obtain information which might inform decisions about the use of this material under a range of erosive conditions.

CHAPTER 10

MATERIALS AND METHODS

10.1 Erosion testing

10.1.1 Preparation of acidic solutions

Hydrochloric acid (HCl) solutions of different pH were prepared from freshly made HCl 1 mol/l. Deionised water was added to make up 100ml solution of HCl pH 1.2 (titratable acidity = 0.063M), pH 2 (titratable acidity = 0.01M), pH 3 (titratable acidity = 0.001M), pH 4 (titratable acidity = 0.0001M), and pH 5 (titratable acidity = 0.00001M).

10.1.2 Sample preparation and erosion testing

Machinable ceramic ingots were sectioned into slices of 1mm thickness by high speed diamond bur under copious water coolant as described in the previous section. Only one of the two surfaces of the discs was ground flat by green stone, then finished with a diamond-impregnated silicone wheel (Shofu, Japan) from coarse to very fine. The final polishing was done with a felt wheel and diamond polishing paste (Henry Schein, USA).

Specimens were randomly divided into six groups, and based on power studies eight specimens were randomly allocated to each group (Fig. 10.1). Specimens were immersed in the experimental solution for two hours. On completion of the experiment, specimens were cleaned in running tap water for two minutes. They then were mounted on SEM studs and coated in gold/carbon for SEM observation with the Philips XL 20 Eletron Microscopy.

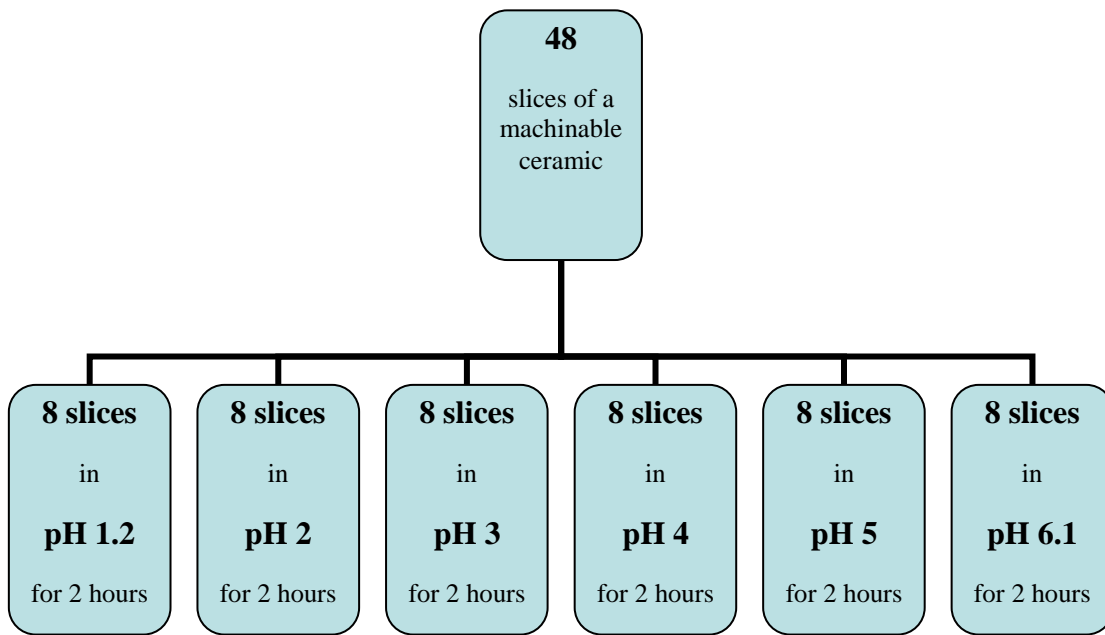


Figure 10. 1: Diagram showing the assignment of specimens for erosion test.

10.2 Porosity assessment

10.2.1 Scanning Electron Microscopy observations

Each specimen was first observed at 20 times magnification to give an overall picture of the porcelain surface. These images were divided into nine equal cells and three cells of

nine on each image were carefully observed under higher magnifications of 100, 200, 500 and 1,000 times. The 1000-times magnification image of each cell was selected for analysis. Because each group contained eight specimens, a total of 24 images in each group were subjected to porosity assessment.

To randomly select the cells on the 20 times magnification images of the 48 specimens of six experimental groups for higher magnification observation, 48 sets of three random numbers were randomly generated using the Research Randomizer by the Social Psychology network (<http://www.randomizer.org/form.htm>) (Copyright ©1997-2008 by Geoffrey C. Urbaniak and Scott Plous | [Site Statistics](#)). Each set was randomly assigned to each image, and the sites that corresponded with the three numbers in the set were selected.

10.2.2 Validation of the method for porosity assessment

The software Adobe®Photoshop® (version 7.0) and Image J (version 1.41n. Developed by Wayne Rasband. National Institutes of Health, USA. Website: <http://rsb.info.nih.gov>) were used for porosity assessment on images of 1000-times magnification. To test the reproducibility and validity of the method, five images containing areas of arbitrary shapes of predetermined area proportion were created manually (Fig. 10.2). The method was used to calculate the area proportion of the dark areas on each image and compared to the known area proportion which was 33.3%.

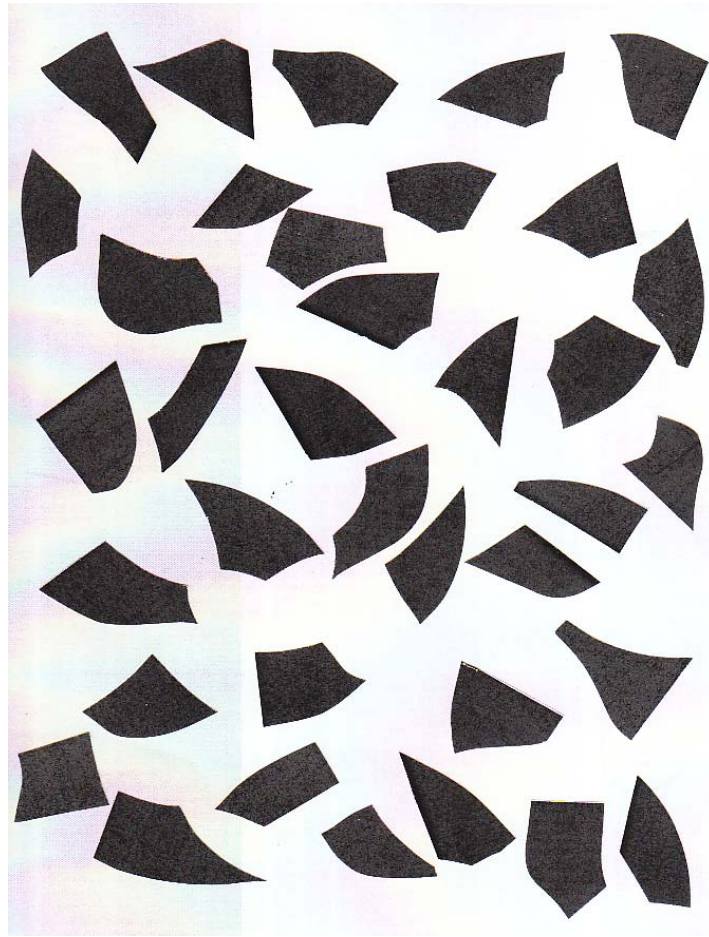


Figure 10. 2: Test image created with known surface area percentage of dark areas.

First of all, each image was scanned four times with a rotation of 90^0 each time to obtain digital images, making a total of 20 images for analysis. The images were adjusted with the software Adobe®Photoshop® (version 7.0) to select the areas of interest and to maximize the contrast between these areas and the background. The adjusted images were subjected to analysis by “ImageJ” for calculation of the area proportion of the selected areas. The threshold was set in a way that only the selected areas appeared in red (Fig. 10.3). The calculation tool was employed to calculate the area proportion of

these red areas. Each of the digital images was analyzed and calculation performed on two occasions, two weeks apart.

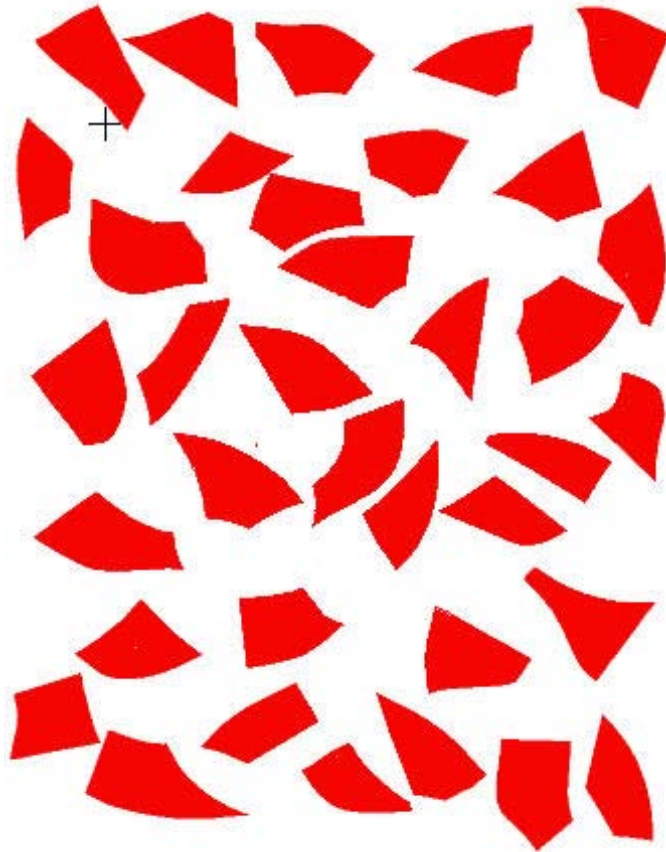


Figure 10. 3: Test image after modification.

The mean area proportion of the selected areas of each scanned image was presented in table 10.1.

Table 10. 1: Mean surface area proportion of digital images

Picture	Digital Image	Mean surface area (%) - 1st calculation	Mean surface area (%) - 2nd calculation
1	1a	32.89	33.05
	1b	32.91	33.60
	1c	32.56	33.22
	1d	33.14	33.05
2	2a	33.43	33.43
	2b	34.34	33.67
	2c	32.95	33.27
	2d	33.07	33.33
3	3a	32.97	33.07
	3b	32.91	33.13
	3c	32.90	32.61
	3d	33.00	33.05
4	4a	33.13	32.96
	4b	33.52	33.45
	4c	33.04	33.22
	4d	32.73	32.58
5	5a	33.32	32.53
	5b	32.29	32.34
	5c	33.09	32.94
	5d	32.67	32.95

Paired t-test found no significant differences between the first and the second determination ($p>0.05$). This provided an indication of no consistent differences between first and second measurements which might have arisen from inconsistencies in selecting the areas or measurement technique of the software.

Compared with the known area percentage (33.3%), measured areas ranged between 32.29% and 33.52% (ie. from 96.33% to 100.66% of the actual area).

To test the reproducibility of the measurement method, twelve random specimens were subjected to analysis and the surface area proportion calculated. The results are presented in Table 10.2.

No significant differences were found between the two determinations as analyzed by paired t-test ($p>0.05$).

The error variance and standard deviation of a single measurement were also calculated according to the method of Dalberg (1940). Error was estimated to contribute about 1.6% to the total observed variation.

Table 10. 2: Double determination of 12 random samples.

	1st determination (%)	2nd determination (%)
1	0.63	0.63
2	1.00	0.78
3	0	0
4	0.37	0.56
5	2.14	2.30
6	1.13	1.16
7	2.54	2.85
8	3.71	4.04
9	2.33	2.20
10	2.14	2.02
11	1.21	1.26
12	1.25	1.32

10.2.3 Porosity assessment

Images of 1000 magnification of each site of observation were analyzed for surface porosities (Fig. 10.3). They were first viewed with Adobe®Photoshop® program (version 7.0). Pores were defined as the areas with lower intensity covered by the edge of much higher intensity. Porous areas were selected by “magic wand”, a tool that is

designed to select pixels based on their colour similarities, and the image were adjusted to the degree of lightness and contrast so that only selected areas were clearly shown on a fairly bright background (Fig. 10.4). Adjusted images were then analyzed with the software “Image J”.

“Image J” operated on the basis that it can measure the selected areas and calculate the area as a percentage. The threshold tool allows the user to interactively set lower and upper threshold values, which can range from 0 to 255. Pixels with brightness value falling within this range are shown in red and the images will then be displayed with clearly distinguishable features of interest and background (Fig. 10.5). In this study, the threshold was set at the limit of 0 for the lower and 155 for the upper end. The tool “measure” was used to measure the aggregate of the selected areas.

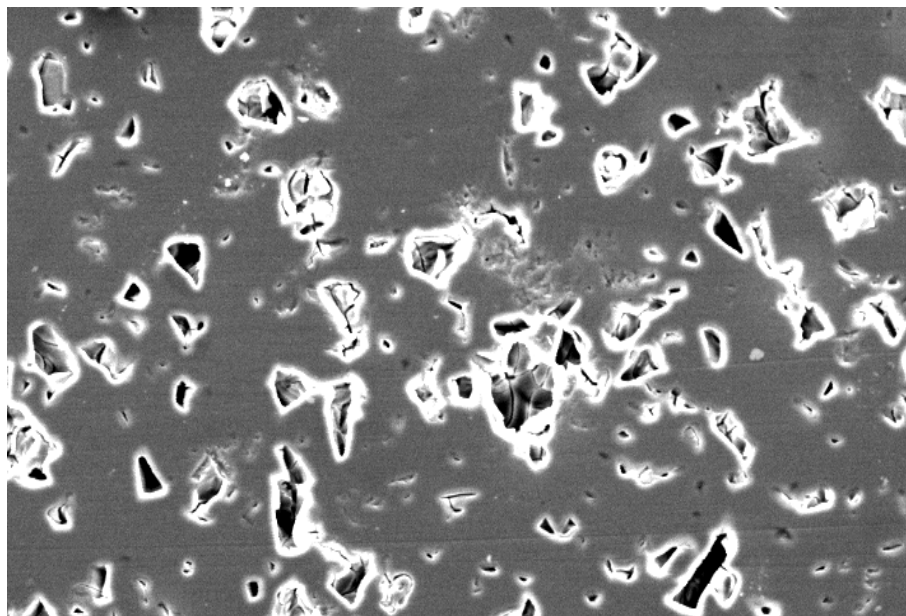


Figure 10. 4: SEM image under 1000 times of magnification.

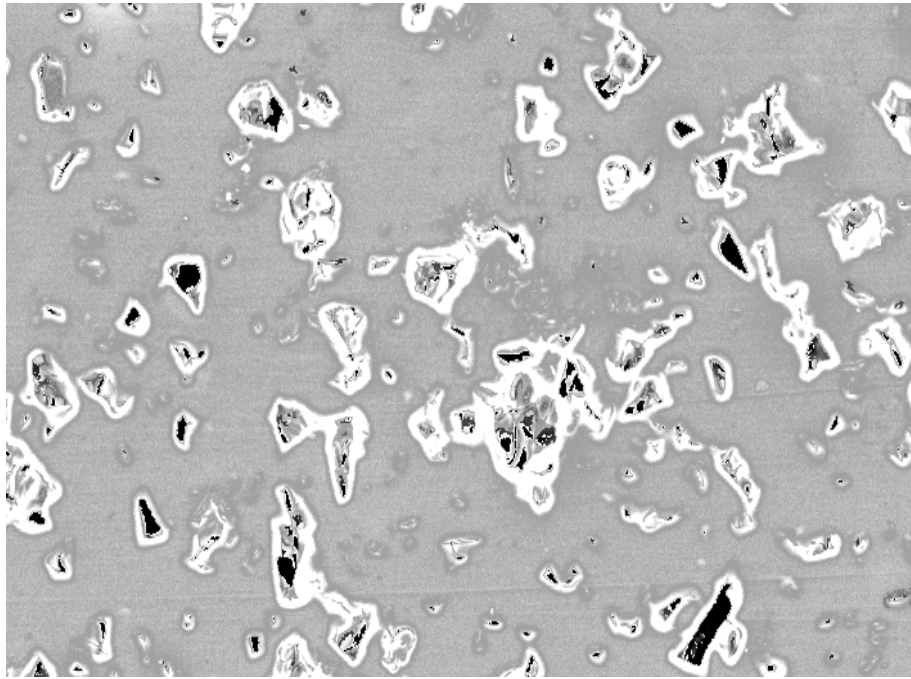


Figure 10. 5: SEM image after being adjusted with Adobe®Photoshop®.

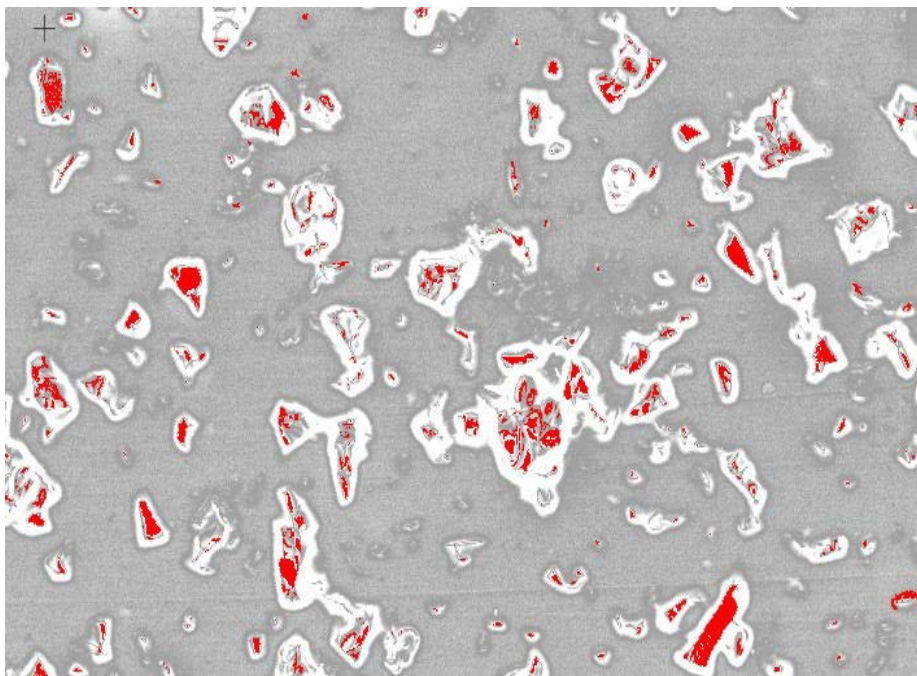


Figure 10. 6: SEM image analyzed with “Image J”.

10.3 Statistical analysis

Statistical analyses were performed using Stata (StataCorp Lp, version 8). One-way analysis of variance and post hoc Bonferroni test were used to find out any statistical significant difference of the surface porosities between groups exposed to different pH. The probability was set at 95% level.

CHAPTER 11

RESULTS

11.1 Introduction

From the results of the analysis of the surface micro-morphology of machinable ceramic in a highly acidic environment, a series of experiments were performed to investigate the effects of acidic solution at various pHs on the material.

Specimens of machinable ceramic were stored in HCl solutions with pH 1.2, 2, 3, 4 and 5 and in deionised water (pH 6.1) for two continuous hours. In each solution, there were eight specimens.

Each specimen was first observed under the magnification of 20 times to give the initial image. Three randomly selected areas on the image were then subjected to further investigation under higher magnifications. Images of magnification 1000 times were used for quantitative analysis.

11.2 Quantitative results

SEM investigation revealed that while specimens in the groups submitted to HCl solutions at pH 1.2, 2, 3 and 4 presented with porosities, specimens stored in HCl solution pH 5 and in deionised water (pH 6.1) had smooth surfaces.

The prevalence of surface alterations for specimens exposed to different pH solutions are presented in Fig. 11.1 and Table 11.1.

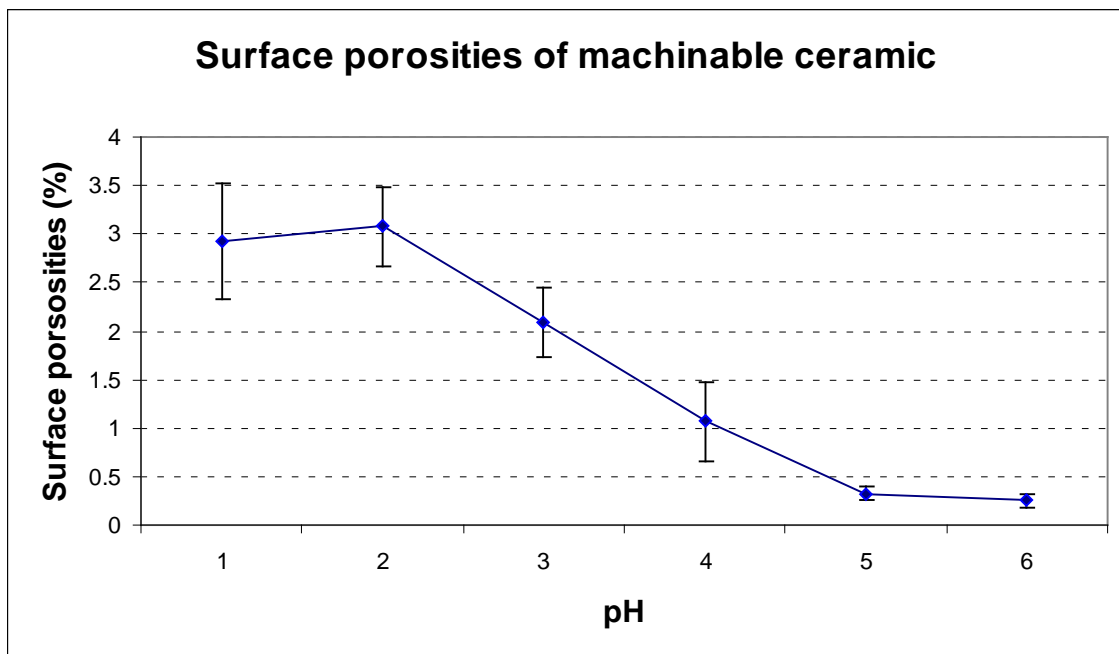


Figure 11. 1: The relative percentages of surface porosities of Vita Mark II treated at various pHs (with standard error bars)

Table 11. 1: The relative percentage of surface porosities of control specimens and specimens exposed to HCl solutions of pH 1.2, 2, 3, 4, 5 and deionised water at pH 6.1.

Specimen number	Specimens exposed to solution					
	pH =1.2	pH = 2	pH = 3	pH = 4	pH = 5	pH=6.1
	(%)	(%)	(%)	(%)	(%)	(%)
1	1.98	3.65	3.19	1.96	0.67	0.12
2	2.44	4.51	3.12	3.17	0.13	0.43
3	1.30	3.8	0.91	0.68	0.36	0.49
4	0.80	0.87	1.05	0.31	0.52	0.50
5	5.58	3.55	2.50	1.10	0	0
6	4.43	2.79	1.13	0.67	0.20	0.20
7	2.43	2.90	2.24	0.42	0.20	0.19
8	3.69	2.63	2.57	0.24	0.15	0.12
Mean	2.93	3.08	2.09	1.07	0.32	0.25
SD	1.71	1.16	0.98	1.17	0.19	0.19
SE	0.6	0.41	0.35	0.41	0.07	0.07

Specimens subjected to pH 5 and 6.1 showed a very low incidence of surface porosities (0-0.5% and 0-0.67%) with an average of 0.25% and 0.32% respectively, of the surfaces showing porosities. In contrast all surfaces exposed to acidic environment at pH 4 and below showed extensive surface change with the highest mean value record ($3.08 \pm 1.16\%$) for the specimen immersed in HCl solution at pH 2 and the lowest value ($1.07 \pm 1.17\%$) for the specimen treated with HCl solution at pH 4.

Interestingly, the prevalence of surface change was very variable in each of the specimen groups with standard deviation in the range 0.98-1.71%. For example, for the specimens exposed to HCl solution at pH 1.2, the prevalence of surface changes ranged from 0.8-5.58%.

Analysis of variance found that there were significant differences between groups ($p < 0.01$). Bonferroni test was used to compare pair-wise groups for any significant differences and the results are presented in Table 11.2.

Table 11. 2: Pair-wise comparison of the prevalence of surface porosities by Bonferroni Test.

	pH 1.2	pH 2	pH 3	pH 4	pH 5
pH 2					
pH 3					
pH 4	p<0.05	p<0.05			
pH 5	p<0.01	p<0.01	p<0.05		
pH 6.1	p<0.01	p<0.01	p<0.01		

There were no significant differences in the prevalence of surface alterations between the groups treated with pH 4, 5 and 6.1. However, there were significant differences between the specimens exposed to deionised water and specimens submitted to HCl solution at pH 1.2, 2 and 3 ($p < 0.01$).

11.3 Qualitative results

While SEM observations revealed a relatively smooth surface for machinable ceramic for the group treated with HCl solution at pH 5 and in deionised water at pH 6.1, the specimens in other groups exhibited partial etching of the porcelain with an increase in surface roughness and porosities. The porosities were of variable shapes with a range of depths and sizes and relatively even in distribution all over the surface.

The following images (Figs 11.2 to 11.7) demonstrate the micro- surface morphology of the specimens after exposure to HCl solution at pH 1.2, 2, 3, 4 and 5 or being stored in deionised water for 2 hours (original magnification 500×).

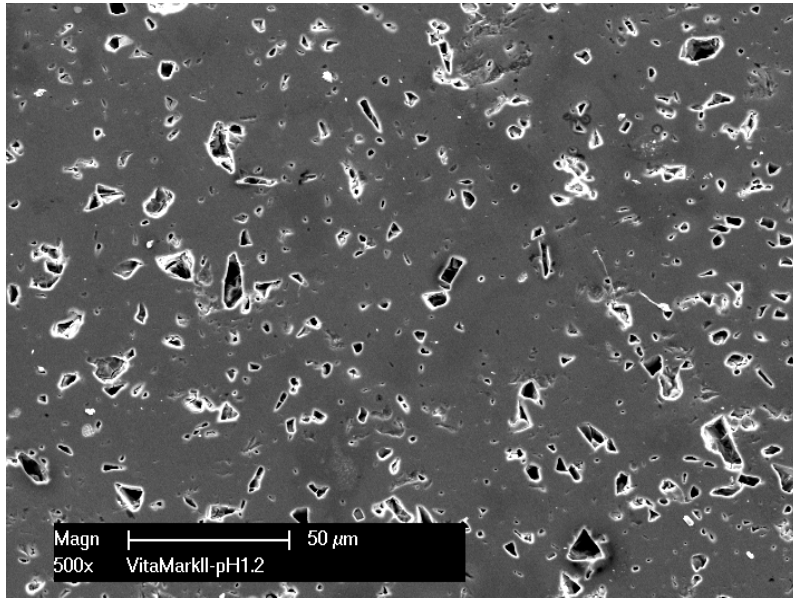


Figure 11. 2: Machinable ceramic. Surface treated with HCl pH 1.2 for 2 hours (×500).

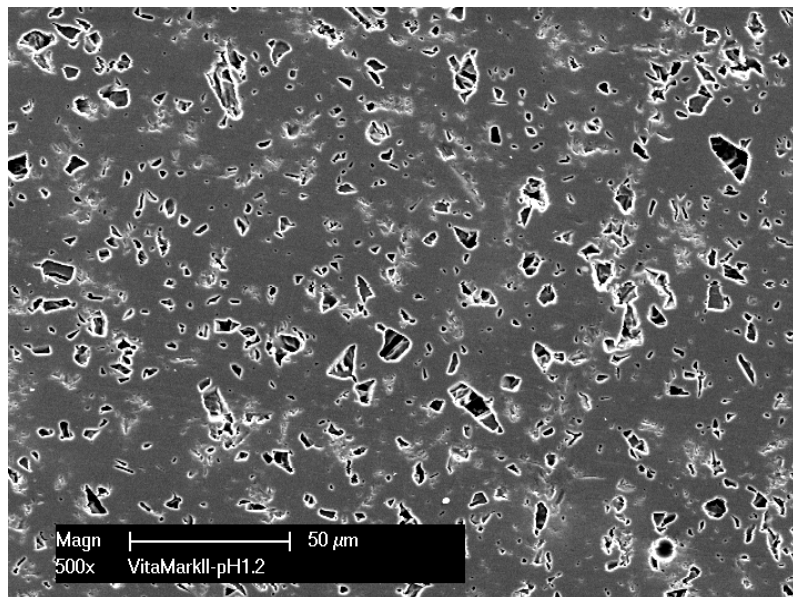


Figure 11. 3: Machinable ceramic. Surface treated with HCl pH 2 for 2 hours (×500).

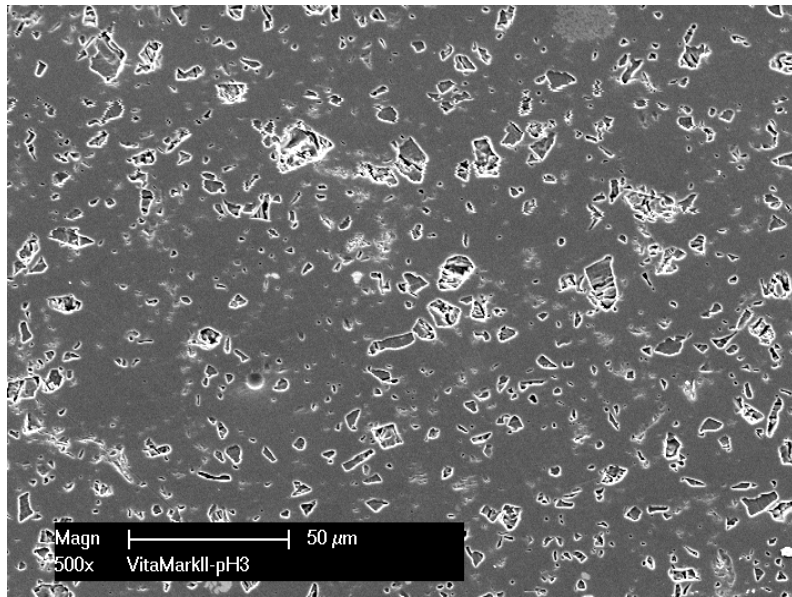


Figure 11. 4: Machinable ceramic. Surface treated with HCl pH 3 for 2 hours (×500).

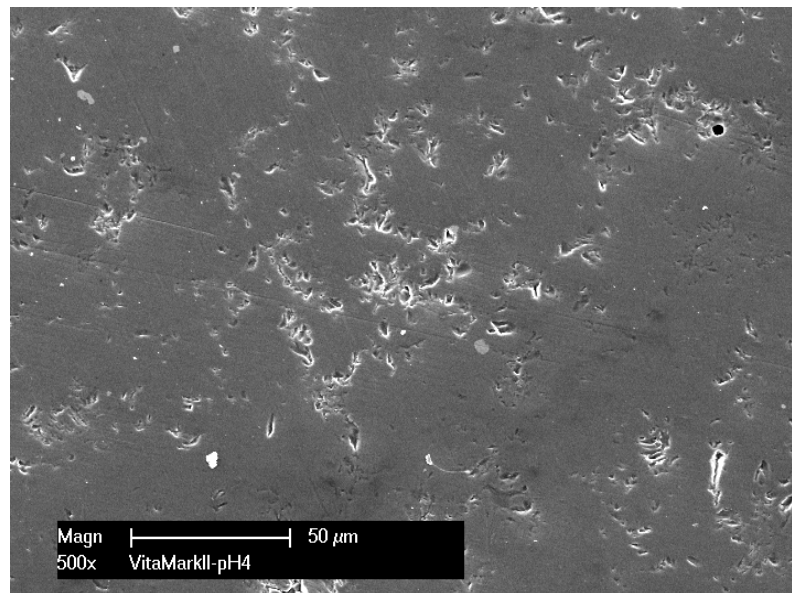


Figure 11. 5: Machinable ceramic. Surface treated with HCl pH 4 for 2 hours (×500).

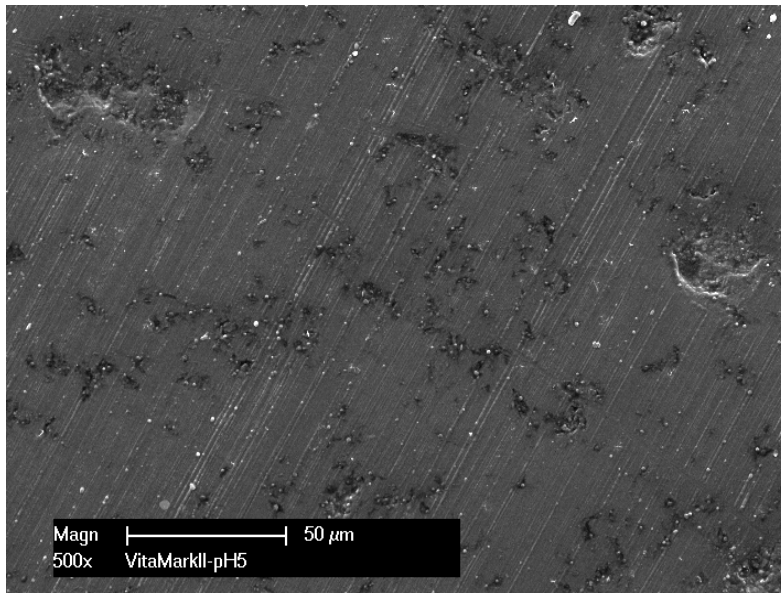


Figure 11. 6: Machinable ceramic. Surface treated with HCl pH 5 for 2 hours (×500).

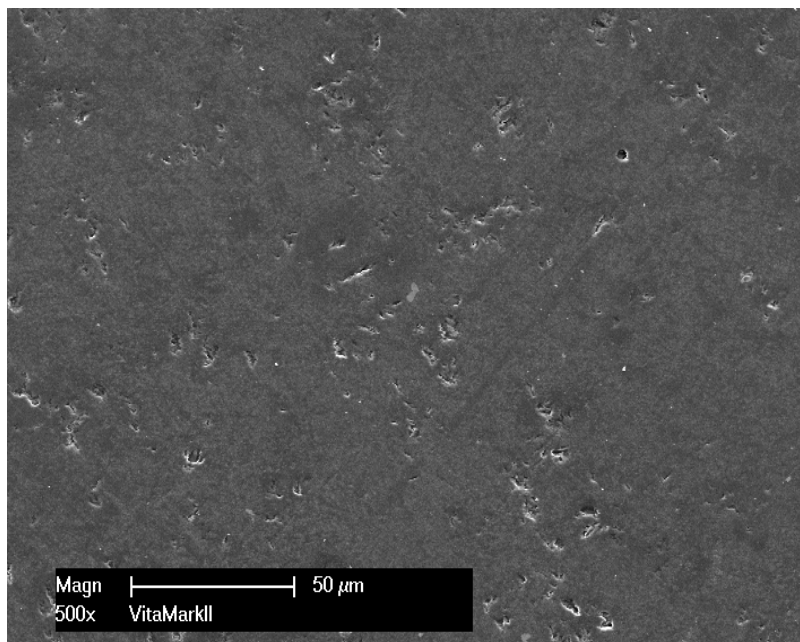


Figure 11. 7: Machinable ceramic. Surface treated with deionised water pH 6.1 for 2 hours (×500).

CHAPTER 12

DISCUSSION

Dental ceramics are often considered chemically inert. However, the finding that the surface of machinable ceramic became porous after being exposed to acid suggested that there were some chemical reactions leading to dissolution of either the glass matrix, the crystal particles or both. Previous studies have demonstrated that a wash out of elements, especially of potassium, aluminium and silicon from ceramics after their exposure of ceramics to acidic agents (Milleding et al. 2002; 2003; Kukiattrakoon et al. 2009; 2010). In addition, a change in microhardness after immersion in acid was also documented (Kukiattrakoon et al. 2009). The porcelain degradation has been reported to be dependent on the acidic agent, the exposure time, and the temperature (Milleding et al. 2002).

Interestingly, low level degradation of dental porcelain occurs even in neutral aqueous solutions. Thus, in exchange to a diffusion of the hydrogen ions (H^+) and hydronium ions (H_3O^+) from an aqueous solution into the glass of porcelain, there is selective leaching of alkaline ions such as K^+ , Na^+ , Ca^{2+} , Al^{3+} and Si^{2+} into the immersing

solutions (Charles 1958; Anusavice 1992; Kukiattrakoon et al. 2009; 2010). A study by Ernsberger (1980) suggested water molecules react with non-bridging oxygen atoms to form hydroxyl ions that diffuse out with the alkaline ions. This produces an alkali-depleted layer on the surface of ceramics (Milleding et al. 2002). Leaching of silicon ions occurs less than for other oxides but may result in the breakdown of Si-O-Si bonds that adversely affects the porcelain structure. It was also hypothesized that the leaching of the ions creates pores and channels in the glassy matrix, which then enhance the diffusion of molecular water and the localized breakage of Si-O-Si bond within the structure. The degradation process is assumed to take place in different ceramics having the glassy matrix in their structure at varying severity and leaching patterns, depending on the chemical composition, microstructure and the local corrosion conditions (Milleding et al. 2002). A more important factor is the properties of the glassy phase which plays a critical role in the corrosion of the porcelain (Milleding et al. 2002).

The corrosion of porcelain in acidic conditions was found to affect the glass phase more than the crystalline phase because alkali metal ions are much less stable in the former than the latter (Anusavice 1992; Milleding et al. 2002). Though the crystalline phase may increase the porcelain corrosion resistance, the grain boundaries between phases are weak points for acidic attack (Milleding et al. 2003). Selective leaching of alkali ions and dissolution of the glass network are the dominant mechanisms responsible for corrosion of the porcelain, and inadvertently promotes crack propagation in it (Anusavice 1992; Milleding et al. 2002; Kukiattrakoon et al. 2010). The surface dissolution was found to be dependent on the composition of porcelains (Milleding et al.

2002). Those made with a high percentage of soda (Na_2O) showed twice better chemical durability compared to the ones high in K_2O , probably because of the difference in ionic size (El-Shamy 1973; Anusavice 1992; Milleding et al. 2002).

The mismatch in thermal expansion coefficients between the crystallites and the glass phase also contribute to the corrosion properties of the porcelain (Milleding et al. 2002). A large thermal contraction upon cooling creates a mismatch between leucite (22 to $25 \times 10^{-6}/^\circ\text{C}$) and the glass matrix ($8 \times 10^{-6}/^\circ\text{C}$) in feldspathic porcelain. This creates micro-cracks in the glass around leucite crystals upon cooling, where the ion leakage may be enhanced (Deany 1996; Milleding et al. 2002).

The chemical durability of dental ceramics plays an important part in their clinical success because they function in the oral environment with frequent changes of pH and other conditions (Dundar et al. 2003). To optimize the durability, the glass ceramic should contain at least 0.5wt% Al_2O_3 and 2wt% ZrO_2 (Adair 1984). It is expected that the more durable a ceramic is, the less the silica network dissolves due to leaching and etching of the material (Dundar et al. 2003).

The porous surfaces of machinable ceramic appearing upon exposure to acid could explain in part its relative abrasiveness to the opposing enamel at pH 1.2, compared to other porcelains in this research project. The increase in abrasiveness has been stated as one of the possible side effects of ceramic degradation (Anusavice 1992; Anusavice and

Zhang 1997). In addition, surface porosities may increase plaque and staining accumulation, reduce aesthetics, roughen the surface, and increase susceptibility to further chemical attacks.

The relationship between the strength of a porcelain and porosity in it is an inverse one. For example, a porcelain containing 10% porosity has half the strength of porcelain with no porosity (Koseyan and Biswas 1976). A possible explanation for this is the concept of stress concentration, according to which the actual area that is stressed is reduced in porous porcelains, thus increasing the possibility of their fracture (Oh et al. 2002).

The surface porosity of polished specimens of machinable ceramic was found to increase from 0.25% in the polished specimens to roughly 3.0% after being immersed for two hours in HCl at pH 1.2 and 2, and to about 2% at pH 3. If the inverse relationship between porosity and the strength is linear, we can expect a reduction of porcelain strength by 17% and 7% respectively. This indicates that machinable ceramic is not a material of choice for patients having highly acidic mouth or consuming lots of acidic foods and drinks.

Why the porosities were only seen with machinable ceramic but not with other selected materials at low pH remained unanswered. In difference to the other tested ceramics, the machinable ceramic was a manufacturer's porcelain which is supposed to have superior physical properties because of better quality control and avoidance of hand

mixing that minimizes the possibility of air incorporation during the sintering process. A possible explanation for the higher acid sensitivity of the machinable ceramic could be hidden in its chemical composition, properties and/or microstructures, which require further investigations to find out.

The results of this study suggest that the selection of a porcelain material for a specific case is important and should be of benefit to the patients' dentitions in the long term. In addition, it should be taken into account that the same porcelain material could behave differently in different acidity of oral environments.

SECTION FOUR

PRELIMINARY STUDY OF ZIRCONIA WEAR

CHAPTER 13

INTRODUCTION

During the time period when the experiments for this project were planned and completed, it became evident that the use of zirconia-based all-ceramic fixed prostheses by dentists is becoming more common (Christensen 2009). Therefore, in order to make this research project more complete, a few preliminary experiments have been carried out to investigate the wear behaviour of this relatively new material.

Zirconia is a material suitable to use on patients who desire all-ceramic indirect restorations. It is biocompatible, aesthetic and easy to fabricate (Christensen 2009). The structure of zirconia changes upon changing of temperature (Guazzato et al. 2005). The structure is monoclinic between room temperature and 1170⁰C, tetragonal above this point to 2370⁰C and then enters the cubic phase if the temperature continues to rise up to the melting point (Denry and Kelly 2008).

The transformation process is reversible with an associated increase or decrease in volume. A reduction in temperature such as in the cooling process will cause the

tetragonal phase to convert to monoclinic phase with an accompanying 4.5% increase in volume, inducing stress within the material (Denry and Kelly 2008). To overcome this, oxides such as CaO, MgO, Y₂O₃ or CeO₂ are added to zirconia to retain the tetragonal structure at room temperature. However, the tetragonal zirconia is metastable and it can change to the monoclinic form under the influence of stress-causing processes such as sandblasting or surface grinding (Kosmac et al. 1999). An increase in the volume associated with the phase transformation results in the formation of surface compressive stress, thus inducing higher toughness and slowing down the crack propagation. However, when the structural integrity of the material is affected, both surface integrity and strength will be compromised (Denry and Kelly 2008).

There are many unanswered questions about the use of zirconia. If zirconia is opposed by the natural enamel, how does it affect the wear rates of enamel? Will the wearing process affect the structural integrity of the material over time and then in return, how does a change in the structure phase affect the wear rates? Anecdotal evidence suggests that the wear rate of enamel opposed by un-veneered zirconia may be unacceptably high but to date this has not been systematically investigated.

This preliminary study looked at the wear rates of enamel and zirconia, using the same protocol set up for previous experiments on other indirect restorative materials. The structure and surface morphology was also investigated to determine if the cyclical loading in the wear machine resulted in a change of the phase distribution in zirconia.

The results will be used for development of protocols for further experiments in this area.

CHAPTER 14

MATERIALS AND METHODS

Zerion™ (Straumann GmbH, Germany), a zirconium oxide ceramic in presintered blanks, was selected for the study. They were carefully sectioned into 10 discs of 1mm thickness. Only one surface of the zirconia discs was thoroughly hand polished with diamond lapping films (Struers A/C, USA). The polishing procedure started with the lapping film of 30µm grit for 10 minutes, then the specimen was washed under tap water to eliminate debris on the surface. It was continued with the film of 15µm grit and finished with 3µm grit in the same procedure. The polished discs were heated in a porcelain furnace to 910°C with one minute holding time and left to cool down at room temperature. The unpolished surfaces were sandblasted with Al₂O₃ powder for mounting on SEM studs, using Panavia F (Kuraray, Japan) in the same manner as for other porcelain mounting in previous studies.

The enamel specimens were prepared from extracted third molars in a similar way to those in previous experiments. They were also mounted on SEM studs using cold-cured resin (Vertex self curing, Vertex Dental BV, the Netherlands) with three embedded

metal spheres for reference. Both zirconia and enamel specimens were stored in water for seven days before being subjected to the wear experiment.

The wear experiments between enamel and Zerion™ were performed on the wear machines under a load of 100N, at 80 cycles per minute, in deionised water (pH 6.1) for 120,000 cycles and HCl (pH 1.2) for 10,000 cycles. The investigation was conducted on five specimens for each pH and in the secondary phase of wear.

The specimens were scanned before and after the wear experiment with a profilometer and purpose-written Matlab software was used to calculate the volume loss. Impressions were taken of the specimens to produce epoxy replicas for SEM observation.

A polished and heat-treated zirconia specimen (unstressed specimen) and one subjected to 120,000 cycles of wear at pH 6.1 (stressed specimen) were used for x-ray diffraction analysis to detect any phase transformation in the material surface under stress.

CHAPTER 15

RESULTS

15.1 Quantitative results

15.1.1 Wear results

The results obtained from volume calculation showed that there was no wear on both enamel (mean wear rate = 0 mm³/1000 cycles) and zirconia (mean wear rate = 0 mm³/1000 cycles) specimens at pH 6.1. At pH 1.2, no wear was detected not for zirconia but for enamel, which was worn at a rate of 0.22 ± 0.06 mm³ per 1000 cycles. A t-test analysis showed that this is significantly lower than the mean wear rates of enamel control specimens at the same pH (0.36 ± 0.12) ($p=0$).

15.1.2 X-ray diffraction results

X-ray diffraction (XRD) traces were collected between 6 and 90°2- θ at 0.02° intervals at rate of 0.05°/minute. Co K- α X-rays (30mA, 60kV, 1.7902Å) were used with a 1/12° collimation slit. Quantitative XRD analysis was performed using SIROQUANT V3

software. The traces did not show any evidence of the monoclinic phase of Zirconia. The structural phases in weight percentage and the XRD traces of the two specimens are presented in Table 15.1 and Fig 15.1.

Table 15. 1: Summary of quantitative XDF analysis.

Specimen	Wt% of Zirconia Phase	
	Tetragonal	Cubic
Stressed specimen	90.4	9.6
Unstressed specimen	87.9	12.1

15.2 Qualitative results

SEM images of enamel and zirconia surfaces after 120,000 cycles of wear in deionised water (pH 6.1) showed a relatively smooth surface. There were no gouging or striations of the type observed on the surfaces of other selected porcelains (Figs 15.2 and 15.3).

While the enamel after wear in HCl (pH 1.2) showed a smooth surface typical of erosion, the porcelain remained as smooth as after polishing (Figs 15.4 and 15.5)

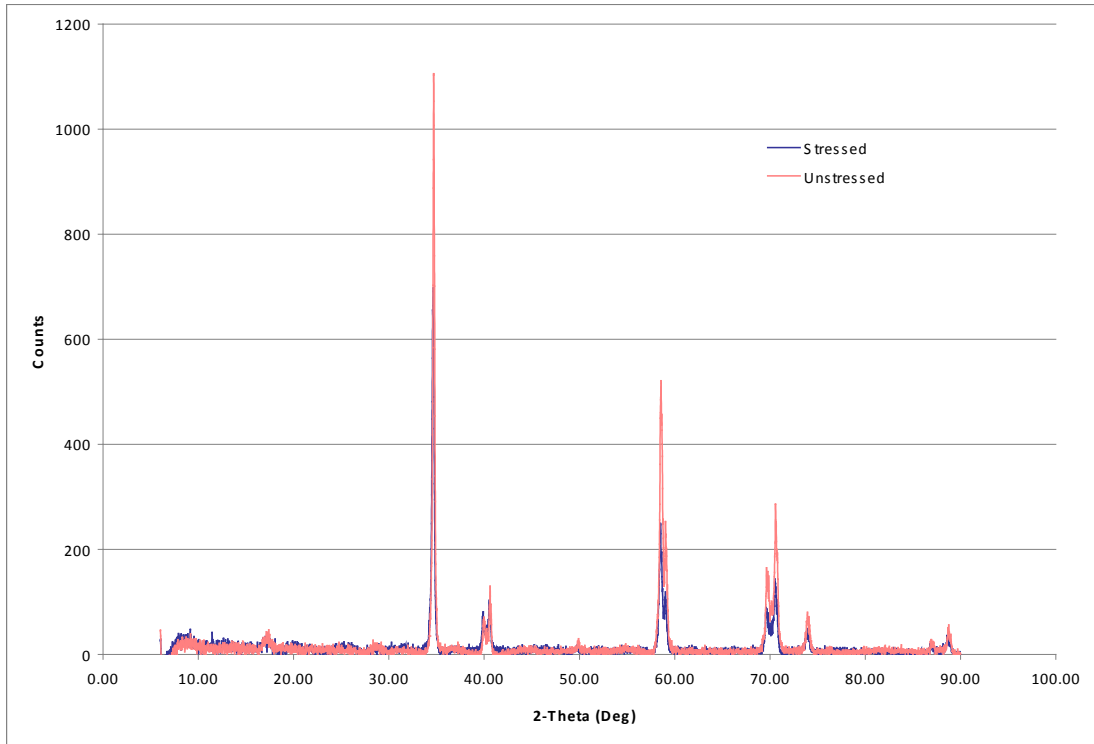


Figure 15. 1: XRD traces of specimen

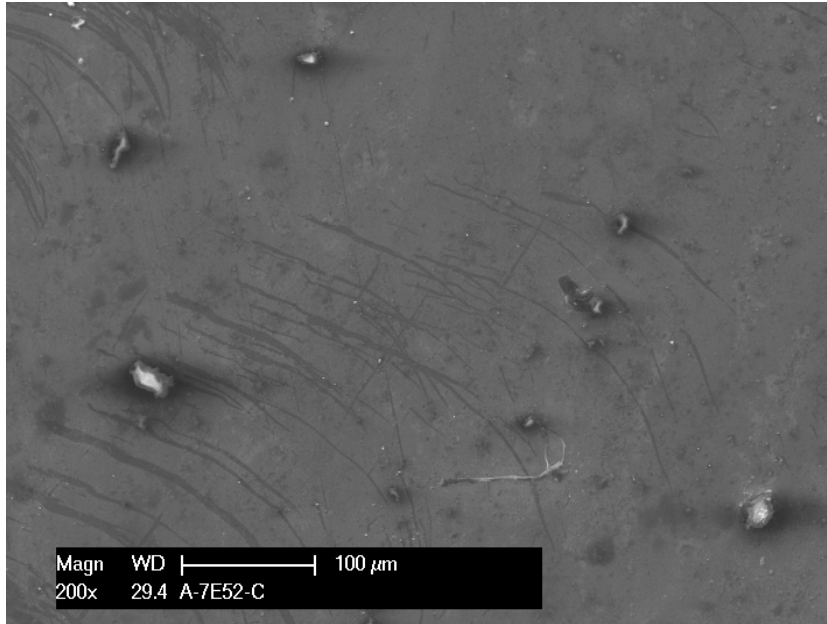


Figure 15. 2: Enamel surface after 120,000 cycles of wear in deionised water (pH 6.1) (x200).

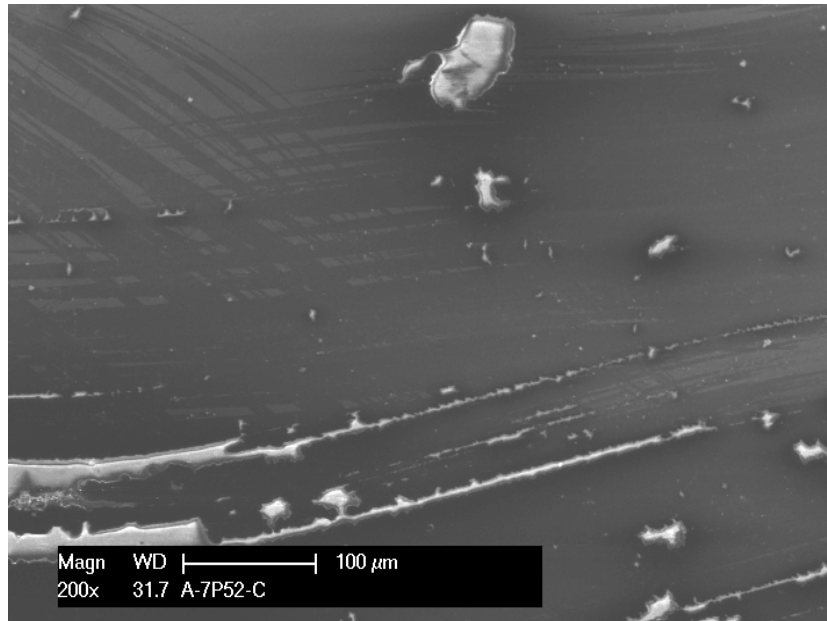


Figure 15. 3: Zirconia surface after 120,000 cycles of wear in deionised water (pH 6.1) (x200).

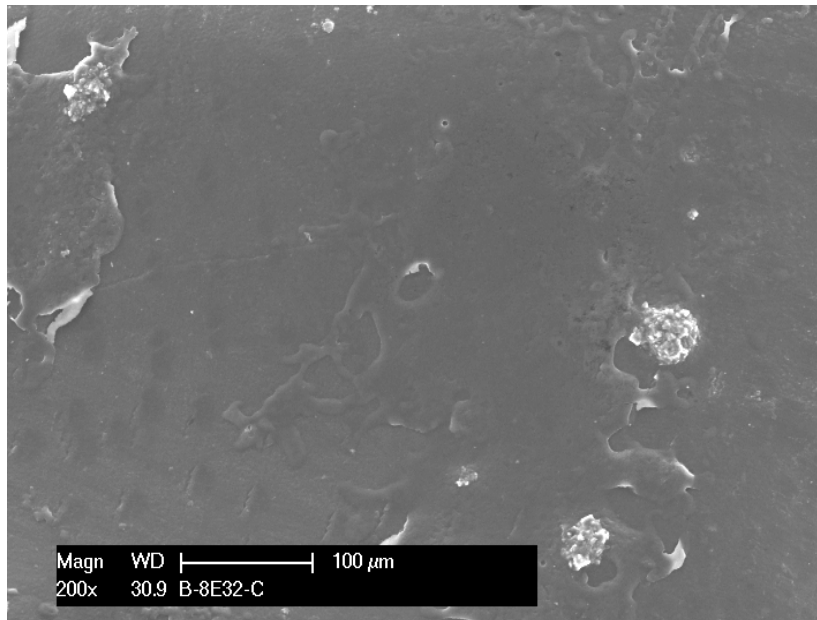


Figure 15. 4: Enamel surface after 10,000 cycles of wear in HCl (pH 1.2) (x 200).

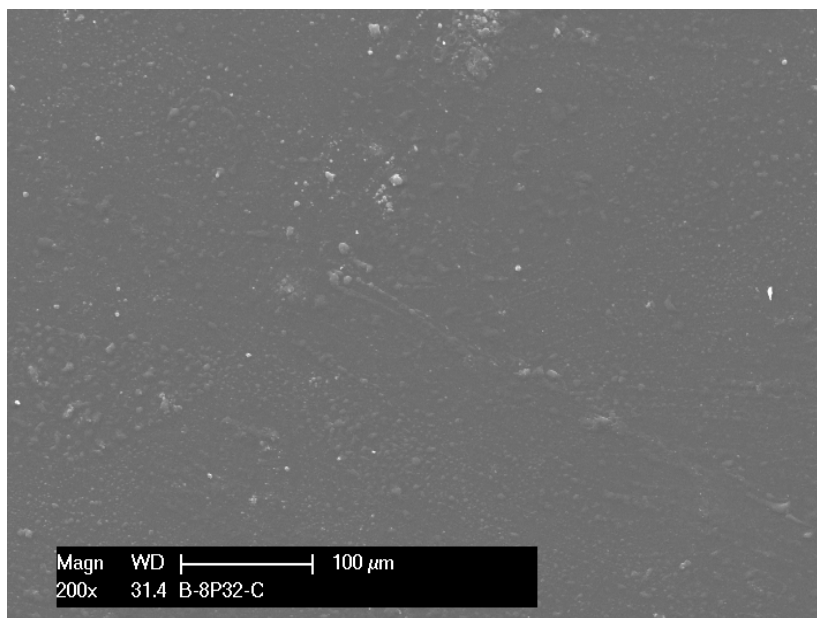


Figure 15. 5: Zirconia surface after 10,000 cycles of wear in HCl (pH 1.2) (x 200).

CHAPTER 16

DISCUSSION

The minimal mean wear rates of zirconia and enamel at pH 6.1 and 1.2 compared to other porcelains were unexpected findings to us, but could be explained. The wear between porcelain and enamel was found to be associated with the surface roughness (Elmaria et al. 2006). The final polishing of zirconia with diamond lapping film of 3 μ m grit rendered the surface very smooth, which might reduce wear. This was supported by observations under SEM, in which the zirconia and enamel seemed to polish each other. This was different when compared to other porcelain surfaces in which the wearing process, especially at pH 6.1, caused parallel striations on the surfaces of the specimens.

Heat treatment at 900⁰C following the polishing procedure of zirconia was reported to reverse the transformation from monoclinic to tetragonal (Kosmac et al. 2000). This was confirmed by the analysis of X-ray diffraction, in which no trace of monoclinic phase was found in heat-treated zirconia. Furthermore, it has been shown that tetragonal zirconia might be transformed to monoclinic under stress. However, in our experiment wearing of zirconia against enamel in deionised water for 25 hours under a load of 100N

did not cause any transformation on the zirconia surface. It might be assumed that the magnitude or the time of stressing was not enough to trigger any phase changes. As wear rates of enamel could be dramatically increased due to phase transformation at high enough stress, these preliminary experiments will need to be expanded in future studies to carefully examine the potential abrasive effects of this new material.

SECTION FIVE

CONCLUSIONS

This research project was designed to investigate the interaction of human enamel and different indirect restorative systems using tooth wear machines. The selected materials included a porcelain-bonded-to-metal ceramic (Duceram® Kiss, Degussa, Germany), a leucite-reinforced veneering ceramic (Authentic™ Veneering, Ceramay, Germany), a leucite-reinforced pressable ceramic (Authentic™ Pressable, Ceramay, Germany), and a machinable ceramic (Vita®Mark II, Vita, Zankfabrik, Germany) and a type III gold alloy (Argenco 2, Argen Corp, USA). The conditions of the wear experiments were controlled by applying a load of 100N and deionised water as lubricant at pH 6.1 or HCl as lubricant at pH 1.2. The mean wear rates of each material were quantitatively assessed by calculating the volume loss per 1000 cycles. Qualitative assessment of the wear facets was conducted by observing the specimens under SEM at different magnifications. Furthermore, the presence of porosities on the surface of the machinable ceramic after being exposed to acid at varying pH was also quantitatively and qualitatively evaluated.

It was impossible to completely simulate the complicated oral environment for in vitro studies, therefore the results should be cautiously extrapolated to in vivo conditions. However, they provide a better understanding and help with the ranking of the materials.

The results from this study showed that compared with data from older systems, the newer porcelains have improved in wear behaviour and become more “enamel-friendly”

to the natural enamel at neutral pH. However, a low pH environment greatly affects the surface characteristics and abrasiveness, especially for the machinable ceramic.

At pH 6.1:

- Enamel wear rates were not significantly different between the enamel control group and enamel opposed by porcelain-bonded-to-metal ceramic, leucite-reinforced veneering ceramic, machinable ceramic, or gold;
- Gold was the least abrasive and the most wear resistant material;
- Leucite-reinforced pressable ceramic wore enamel the fastest;
- Leucite-reinforced veneering ceramic had the least wear resistant characteristics;
- Leucite-reinforced veneering ceramic wore at twice the rate of the enamel antagonist;
- Leucite-reinforced pressable ceramic wore 50% less than the opposing enamel; and
- The wear of enamel and material antagonists were not significantly different for porcelain-bonded-to-metal ceramic, machinable ceramic and gold.

At pH 1.2:

- Enamel wear rates were 4 to 8 times greater than those at neutral pH;
- Material wear was minimal;

- Wear rates of the control enamel specimens were comparable to wear rates from enamel against porcelain-bonded-to-metal ceramic, leucite-reinforced veneering ceramic and gold; and
- Leucite-reinforced pressable and machinable ceramic were the most abrasive materials.

SEM observations revealed:

- Rough surfaces of enamel wear facets at neutral pH;
- Smooth and wavy surfaces typical of enamel erosion at pH 1.2;
- Rough surfaces in porcelain wear facets with striation patterns;
- Porosities present on machinable ceramic surfaces after 10,000 cycles wear at pH 1.2; and
- Porosities were observed on machinable ceramic specimens after exposure to pH 4 or lower in the absence of any load.

Zirconia wear:

- Minimal wear was observed for both zirconia and enamel at both “neutral” and low pH with the applied protocol.

Though these results add a novel dimension to our knowledge of wear behavior and surface micromorphology of enamel and porcelain materials, especially under acidic attack, they bring to attention a number of yet unknown issues that require further investigations. In particular, a more thorough understanding of factors influencing dental

wear rates may be obtained through future wear studies taking into consideration more complete chemical and physical characteristics of the materials.

SECTION SIX

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SECTION SEVEN

APPENDICES

APPENDIX 1

MATHEMATICAL MODELLING OF WEAR

Halling (1975) suggested a simplified model to quantitate abrasive wear. In this model, one surface consists of hard conical asperities with the same semi-angle θ , while the other surface is softer and flat (Fig. A1.1).

NOTE:

This figure is included on page 203 of the print copy of the thesis held in the University of Adelaide Library.

Figure A1.1 Abrasive wear by a conical indenter (adapted from Halling 1975).

“When a single asperity traverses a unit distance, it displaces a volume of material rd .

but $d = r \cot \theta$

therefore $\text{the volume displaced} = r^2 \cot \theta$

Assuming that the material has yielded under the normal load and therefore the asperity supports a load of $\pi r^2 p_0/2$, in which p_0 is the yield pressure of the softer material.

If there are n asperity contacts

total normal load $W = n\pi r^2 p_0/2$

and the total volume displaced in unit distance is

$$Q = nr^2 \cot \theta$$

eliminating n

$$Q = 2W \cot \theta / p_0$$

This equation does not take into account the asperity heights and shapes. It also does not account for the build up of the material in front of the asperities which alters the conditions. In addition, Young's modulus are also important in some situations. However, an equation in the form of

$Q = k_a W/H$ is found to cover a wide range of abrasive situations.

where H is the hardness of the softer material

k_a is the abrasive wear constant" (Halling 1975).

Archard (1953) suggested a simplified law of adhesive wear.

"It is assumed that the contact is made up of a number of small asperities each of radius a. The area of each contact is πa^2 and each contact supports a load of $p_0 \pi a^2$, where p_0 is the yield pressure. The surfaces will pass completely over each asperity in a sliding distance of $2a$ and we will assume

that the wear fragment produced at each asperity is hemispherical in shape and of volume $2/3 \pi a^3$.

Then the total wear volume Q per unit distance of sliding is given by

$$\begin{aligned} Q &= \sum (2/3 \pi a^3) / 2a \\ &= 1/3 \sum \pi a^2 = (\pi a^2 / 3) \times n \end{aligned}$$

Where n is the total number of contacts. But each contact supports a load of $p_0 \pi a^2$, therefore

total load $W = p_0 \pi a^2 n$

or $n \pi a^2 = W / P_{0t}$

therefore $Q = W / 3p_0$

This equation has been derived assuming that all asperity encounters produce a wear particle. If only a fraction k of all encounters produce wear particles then the equation becomes

$$Q = kW / 3p_0$$

Where k is the probability of an asperity contact producing a wear particle.

k depends on different combinations of sliding materials and conditions of rubbing.”(Archard 1953).

Knowledge of the interaction of these factors provides a basis for understanding the wear process, but a more detailed investigation was beyond the scope of this study.

APPENDIX 2

PRODUCT INFORMATION

Duceram®Kiss (Degudent, A Densply International Company)

Composition and Mechanical Properties (Source: market release and ISO 6872/9693)

Property	Unit of measure	Value
Veneering porcelain specifically intended for veneering crown and bridge frameworks made of titanium or titanium-niobium dental alloys with a CTE range 13.8-15.4 $\mu\text{m}/\text{m.K}$ (25-600 $^{\circ}\text{C}$). Produced by Densply		
Fusing temperature	$^{\circ}\text{C}$	≈ 900
Coefficient of thermal expansion dentine 25-600 $^{\circ}\text{C}$	$\mu\text{m}/\text{m.K}$	13.0
Solubility	$\mu\text{g}/\text{cm}^2$	30
TG	$^{\circ}\text{C}$	490
Flexural strength	MPa	≈ 90
Fracture toughness	MPa.m $^{0.5}$	0.9-1.2

Authentic™ Veneering (Ceramay GmbH+Co.KG, Stuttgart, Germany)

Composition and Mechanical Properties (Source: product brochure)

Property	Unit of measure	Value (ISO 6872/9693)
Leucite-reinforced glass ceramic		
Leucite crystal size	μm	<4
Low fusing temperature	°C	750
Coefficient of thermal expansion 25-500°C	10 ⁻⁶ K ⁻¹	14.7
Solubility	μg/cm ²	30
TG	°C	490
Composition:		
SiO ₂	%	≈ 60
Al ₂ O ₃		≈ 12
K ₂ O		≈ 12
Na ₂ O		≈ 8
Others		≈ 8

Authentic™ Pressable (Ceramay GmbH+Co.KG, Stuttgart, Germany)

Composition and Mechanical Properties (source: product brochure)

Property	Unit of measure	Value (ISO 6872/9693)
Leucite-reinforced glass ceramic		
Leucite crystal size	μm	<4
Pressing temperature of ingot	°C	940
Coefficient of thermal expansion 25-500°C	10 ⁻⁶ K ⁻¹	14.7
Solubility	μg/cm ²	30
TG	°C	580
Composition:		
SiO ₂	%	≈ 70
Al ₂ O ₃		≈ 13
K ₂ O		≈ 9
Na ₂ O		≈ 3
Others		≈ 5

**Vita® Mark II (VITA Zahnfabrik, H.Rauter GmbH & Co.KG, Bad
Säckingen, Germany)**

Composition and Mechanical Properties (source: www.vident.com)

Property	Unit of measure	value
Mixture of feldspathic crystalline particles embedded in a glassy matrix	Vol%	≈ 30
Average particle size	μm	4
Density	g/cm ³	2.44 ± 0.01
Refractive Index	-	1.501 ± 0.001
Expansion Coefficient α _{5000C}	10 ⁻⁶ K ⁻¹	9.4 ± 0.1
Transformation Area	°C	780-790
Knoop Hardness HK 0.2/30	-	521 ± 8
Vickers Hardness HV 0.1/15	-	640 ± 20
Flexural Strength (1.2 × 4 × 15mm surface prepared by the CEREC 2 machine 0.5mm/min)	MPa	113 ± 10
Toughness (SENB method)	MPa√m	1.7 ± 0.1
Toughness (Vickers indentation)	MPa√m	2.2 ± 0.1
Young's Modulus	GPa	63.0 ± 0.5

Argenco 2 (The Argen Corporation, San Diego, CA, USA)

Composition and mechanical properties

(Source: ww.argen.com/ENGLISH/properties.asp?ALLOY_ITEM=100134)

Property	Unit of measure	Value (ISO 6872/9693)
High gold alloy for crown and bridge Type III Colour: Yellow		
Vickers Hardness Soft		130
Vickers Hardness Hard		210
Proof Stress Soft	MPa	310
Proof Stress Hard	MPa	390
Tensile Strength Soft	MPa	410
Elongation Soft		43
Elongation Hard		26
Melting Range	°C	930-970
Casting Temp	°C	1070
Density	g/cc	16.3
Burnout Temp	°C	650-705
Composition:		
Au	Weight %	74.1
Pt		1.6
Pd		2.3
Ag		13.4
Cu		7.19
In		0
Ga		0

Zerion™ (Etkon Straumann, Germany)

Composition and mechanical properties (Source: <http://cadcam.straumann.us>)

Property	Unit of measure	Value (ISO 6872/9693)
Material: Y-TZP-A		
Average particle size (ISO 13356)	μm	≤ 0.6
Density (ISO 13356)	g/cm ³	≥ 6.0
Vickers Hardness HV		≥ 1200
Flexural strength (3-point bend) (ISO 6872)	MPa	max 1200
Compressive strength	MPa	≥ 2000
Modulus of elasticity	GPa	210
Thermal expansion coefficient CTE (20-500 ⁰ C)	K ⁻¹	10 × 10 ⁻⁶
Composition (ISO 13356):		
ZrO ₂ + Y ₂ O ₃ + HfO ₂	Weight %	≥ 99
Y ₂ O ₃		4.5 – 5.4
Al ₂ O ₃		< 0.5

APPENDIX 3

OTHER ACTIVITIES DURING PHD CANDIDATURE

Scholarships and grant

- Vietnam government scholarship: 2006-2010
- Australian Dental Research Foundation grant: 2007
- Herbert Gill-Williams scholarship for attending IADR conference: 2008

Meetings and conferences

- IADR (ANZ division): 2008. Poster presentation
- IADR (Barcelona): 2010. Poster presentation
- Research Day (School of Dentistry, The University of Adelaide): 2008 Poster presentation
- Research Day (School of Dentistry, The University of Adelaide): 2009. Oral presentation

Teaching activities

- Tutoring (BDS) 2 in simple cons: 2007, 2008 and 2009
- Tutoring BDS 4 in removable prosthodontics and complex cons: 2006-2010
- Tutoring BDS 5 in general dental practice: 2009

Courses or seminars

- weekly seminars on implantology: 2006-2009
- monthly seminars of the restorative group (School of Dentistry): 2008
- weekly seminars of the cranio-facial research unit: 2008
- the “New Technology in Dentistry” : 2007
- the “Dental Board’s Big Day Out”: 2008
- meetings of the South Australian Prosthodontic Society: 2009-2010
- monthly clinical implant clinic (School of Dentistry): 2006-2010.