WEAR OF HUMAN ENAMEL OPPOSING MONOLITHIC ZIRCONIA, GLASS CERAMIC AND RESIN COMPOSITE

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การสึกของเคลือบฟันมนุษย์เมื่อสบกับโมโนลิธิคเซอร์โคเนีย กลาสส์เซรามิก และเรซินคอมโพสิต

นางสาวจีรภา ศรีเพ็ชรดานนท์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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Field of Study	Esthetic Restorative and Implant Dentistry
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จีรภา ศรีเพ็ชรดานนท์ : การสึกของเคลือบฟันมนุษย์เมื่อสบกับโมโนลิธิคเซอร์โคเนีย กลาสส์เซรามิก และเรซินคอมโพสิต. (WEAR OF HUMAN ENAMEL OPPOSING MONOLITHIC ZIRCONIA, GLASS CERAMIC AND RESIN COMPOSITE) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ทพ. เฉลิมพล ลี้ไวโรจน์, 46 หน้า.

วัตถุประสงค์ เพื่อศึกษาการสึกของเคลือบฟันมนุษย์เมื่อทำการสบกันกับเซรามิกทางทันตกรรม (โมโนลิธิคเซอร์โคเนีย และ กลาสส์เซรามิก) และเรซินคอมโพสิต วิธีการทดลอง ชิ้นงานทดสอบจำนวนทั้งหมด 24 ชิ้นงาน ได้แก่ โมโนลิธิคเซอร์โคเนีย กลาสส์เซรามิก เรซินคอมโพสิต และเคลือบฟันมนุษย์ เตรียมเป็นรูปทรงกระบอก ชนิดละ 6 ชิ้นงาน เพื่อเป็นคู่สบกับชิ้นงานเคลือบฟันจำนวน 24 ชิ้นงานซึ่งเตรียมจากฟันกรามแท้มนุษย์ นำมาทดสอบการสึกกับคู่สบแต่ละชนิดโดยใช้เครื่องมือศึกษาการสึกกร่อนแบบพินออนดิสก์ที่น้ำหนักกดคงที่ 25 นิวตัน ความเร็ว 20 รอบต่อนาที เป็นจำนวน 4,800 รอบ วัดความลึกของการสึกสูงสุด ความลึกของการสึกเฉลี่ย และความหยาบผิวเฉลี่ยของชิ้นงานเคลือบฟันโดยใช้เครื่องโปรไฟโลมิเตอร์ ผลการทดสอบที่ได้นำมาวิเคราะห์ทางสถิติด้วยการวิเคราะห์ความแปรปรวนแบบทางเดียว และการวิเคราะห์แบบตูเกร์ ที่ระดับนัยสำคัญ 0.05 และใช้การวิเคราะห์ด้วยสถิติการทดสอบที สำหรับกลุ่มตัวอย่างสองกลุ่มที่สัมพันธ์กันเพื่อใช้เปรียบเทียบความหยาบผิวเฉลี่ยของเคลือบฟัน ก่อนและหลังการทดสอบ ประเมินลักษณะการสึกในเชิงคุณภาพของผิวเคลือบฟันและผิวคู่สบด้วยภาพถ่ายจากกล้องจุลทรรศน์อิเลคตรอนชนิดส่องกราด ผลการทดลอง ไม่พบความแตกต่างอย่างมีนัยสำคัญของความลึกในการสึกของผิวเคลือบฟัน (ค่าสูงสุด และค่าเฉลี่ย) ระหว่างกลุ่มโมโนลิธิคเซอร์โคเนีย (2.17±0.80 และ 1.83±0.75 ไมโครเมตร) กับกลุ่มเรซินคอมโพสิต (1.70±0.92 และ 1.37±0.81 ไมโครเมตร) และระหว่างกลุ่มกลาสส์เซรามิก (8.54±2.31 และ 7.32±2.06 ไมโครเมตร) กับกลุ่มเคลือบฟันมนุษย์ (10.72±6.31 และ 8.81±5.16 ไมโครเมตร) ความแตกต่างอย่างมีนัยสำคัญทางสถิติของความลึกในการสึกของผิวเคลือบฟันพบเมื่อกลุ่มโมโนลิธิคเซอร์โคเนียและกลุ่มเรซินคอมโพสิตเปรียบเทียบกับกลุ่มกลาสส์เซรามิกและกลุ่มเคลือบฟันมนุษย์ ($P < 0.001$) และพบว่าความหยาบผิวเฉลี่ยของชิ้นงานเคลือบฟันที่ทำการทดสอบกับโมโนลิธิคเซอร์โคเนีย กลาสส์เซรามิก และเคลือบฟัน มีค่าเพิ่มขึ้นหลังการทดสอบอย่างมีนัยสำคัญทางสถิติ ($P < 0.05$) แต่ไม่พบความแตกต่างของความหยาบผิวเฉลี่ยระหว่างกลุ่มดังกล่าว สรุป ภายใต้เงื่อนไขและข้อจำกัดของการศึกษานี้ โมโนลิธิคเซอร์โคเนีย และเรซินคอมโพสิตทำให้เกิดการสึกบนเคลือบฟันน้อยกว่ากลาสส์เซรามิก และเคลือบฟัน และความหยาบผิวเคลือบฟันที่เกิดขึ้นหลังการทดสอบการสึกมีค่าเพิ่มขึ้นในวัสดุบูรณะทุกกลุ่ม ยกเว้นกลุ่มเรซินคอมโพสิต

สาขาวิชา ทันตกรรมบูรณะเพื่อความสวยงามและทันตกรรมรากเทียม ลายมือชื่อนิสิต .....

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5376143332 : MAJOR ESTHETIC RESTORATIVE AND IMPLANT DENTISTRY
KEYWORDS: ABRASIVENESS / ABRASIVE WEAR / CERAMIC / DENTAL CERAMIC / ENAMEL WEAR / GLASS CERAMIC / IPS EMAX PRESS / LAVA ALL ZIRCONIA / LITHIUM DISILICATE GLASS CERAMIC / MONOLITHIC ZIRCONIA / PIN ON DISC / RESIN COMPOSITE / WEAR

JEERAPA SRIPETCHDANOND: WEAR OF HUMAN ENAMEL OPPOSING MONOLITHIC ZIRCONIA, GLASS CERAMIC AND RESIN COMPOSITE. ADVISOR: ASSOC. PROF. CHALERMPOL LEEVAILOJ, 46 pp.

Objective The purpose of this study was to investigate wear of human enamel when opposed to dental ceramics (monolithic zirconia, glass ceramic) and resin composite. Materials and methods Twenty-four test specimens (antagonists) – 6 each of monolithic zirconia, glass-ceramic, resin composite, and enamel – were prepared into cylindrical rods. Enamel specimens were prepared from 24 extracted human permanent molars. Using a pin-on-disc wear tester, enamel specimens were abraded against each type of antagonist under a constant load of 25 N, at 20 rpm for 4,800 cycles. Maximum depth of wear (Dmax), mean depth of wear (Da), and mean surface roughness (Ra) of enamel specimens were measured with a profilometer. All data were statistically analyzed using one-way ANOVA, followed by Tukey's test (α = 0.05). A paired t-test was used to compare Ra of enamel at baseline and after testing. SEM pictures were used for evaluating wear qualitatively of both enamel and antagonists. Results There were no significant differences in enamel wear depth (Dmax, Da) between monolithic zirconia (2.17 ± 0.80, 1.83 ± 0.75 μm) and resin composite (1.70 ± 0.92, 1.37 ± 0.81 μm), and between glass-ceramic (8.54 ± 2.31, 7.32 ± 2.06 μm) and enamel (10.72 ± 6.31, 8.81 ± 5.16 μm). Significant differences were found when enamel wear depth by monolithic zirconia and resin composite were compared with those by glass-ceramic and enamel ($P < 0.001$). Ra of enamel specimens increased significantly after wear tests with monolithic zirconia, glass-ceramic and enamel ($P < 0.05$), however no difference was found among these materials. Conclusions. Within the limitations of this study, monolithic zirconia and resin composite caused less wear depth to human enamel compared to glass-ceramic and enamel. All test materials except resin composite similarly increased enamel surface roughness after wear testing.

Field of Study: Esthetic Restorative and Implant Dentistry
Student's Signature .....
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CHAPTER I

INTRODUCTION

Rationale and Significance of the problem

Nowadays, all-ceramic materials and resin composite are commonly used for posterior tooth-colored restorations. Their utilization has increased following the demand for non-metallic dental prostheses. The superiority of ceramic substrate is renowned for its high biocompatibility, strength and, especially, excellent esthetics as it could naturally mimic the characteristic of human tooth structure [1, 2]. However, the abrasiveness of these materials against enamel antagonist is still a clinical concern. Several investigators have demonstrated that, in general, ceramic material cause greater enamel wear compared with any other restorative materials or enamel [2-5].

Since wear of a material is influenced by numerous factors, include contact geometry, surface roughness, microstructural features, grain size, fracture toughness, speed, load, temperature, duration, environment and lubrication [6], enamel wear by ceramic or composite is also the multi-factorial condition. In many decades, there were a lot of studies trying to find out which factors affect wear of human enamel by these materials [2, 7].

Wear of ceramics and enamel antagonist

Surface condition (rough, polished and glazed surfaces), hardness and fracture toughness are some of the contributing factors that determine enamel wear by ceramic [7]. In recent years, many studies have indicated that polished surface of ceramic has shown to cause less enamel wear than the glazed surface [8-12]. This information could be implied that polishing alone is acceptable after chairside adjustment [7].

Wear ability of ceramic is different from that of metal and composite. To some extent, ceramic (as well as enamel) wears through the microfracture mechanism, while metal and composite wear through adhesion [7]. Fischer et al

stated that “For most materials, metal in particular, the wear resistance is believed to be directly proportional to the hardness” [13]. However, for abrasion of most ceramic, hardness and wear are probably not well-associated with each other [14-16]. According to several studies, it was noticed that enamel wear by ceramic is more related to surface roughness and fracture toughness than hardness values [13, 17, 18].

Wear of resin composite and enamel antagonist

Posterior resin composite can abrade human enamel differently due to the size, hardness, and content of the filler particles [19, 20]. Modern composite materials are high resistant to wear from opposing dentition because of the improvement in filler composition and quality of resin matrix. It was informed that the most wear-resistant composites are composed with fillers, which are small in size (1 μm or less), high in concentration, and well bonded to the matrix [7].

Wear of enamel by composite was occurred through hard filler protruding from the abraded resin matrix and that the amount of enamel wear directly correlated with composite’s hardness value [19, 20]. Thus, hardness might be a reliable factor to predict enamel wear by resin composite.

Recently, monolithic zirconia (so-called “full zirconia”) has been used for posterior fixed partial dentures in order to eliminate the problem from chipping of veneering porcelain [12]. Because of its high fracture resistance and ability to withstand high force by only 0.5 mm occlusal thickness, the monolithic zirconia was suggested to use with patient with limited inter-occlusal space [21]. These advantages make the full zirconia become a promising substitute of metal, apart from the predominance in esthetics.

Research Question

How is the wear of human enamel when opposed to monolithic zirconia, glass ceramic and resin composite?

Objective of the Study

The purpose of this study was to investigate wear of enamel when opposed to dental ceramics (monolithic zirconia, glass ceramic) and resin composite.

Statement of Hypothesis

Null hypothesis:

1. There is no significant difference in enamel wear depth and surface roughness among monolithic zirconia, glass ceramic and resin composite.
2. For each material tested, there is no significant difference in enamel surface roughness compared to baseline.

Alternative hypothesis:

1. There are significant differences in enamel wear depth and surface roughness among monolithic zirconia, glass ceramic and resin composite.
2. For each material tested, there is a significant difference in enamel surface roughness compared to baseline.

Scope of the Study

This is an experimental research for evaluation of the wear of human enamel when opposed to different types of restorative material by means of the pin-on-disc wear tester (Model TE 79, Plint&Partners LTD., Berkshire, England). This study utilized occlusal enamel of human molar teeth as a representative of the posterior teeth. The restorative materials included in this study are monolithic zirconia (Lava All Zirconia, 3M ESPE, Seefeld, Germany), lithium-disilicate glass-ceramic (IPS e.max Press, Ivoclar vivadent, Amherst, NY) and resin composite (Premise, Kerr, Orange, CA), which are commonly used to restore teeth in the posterior region of the mouth. The control parameters of the wear test (load, speed, duration) were determined from the pilot study together with manufacturer's recommendations for this wear tester

following ASTM G 99 (Standard test method for wear testing with a pin-on-disc apparatus).

Basis Assumption

1. All procedures were performed under well-controlled conditions and prepared by one operator and evaluated by one examiner.
2. The well-known ceramic systems in Thailand with reliable fabrication procedures were chosen to be included in this study (Lava All Zirconia, 3M ESPE and IPS e.max press, Ivoclar Vivadent).
3. One of the favorite posterior resin composites in Thailand was chosen to be included in this study (Premise, Kerr).
4. The ceramic specimens were fabricated according to the recommendations of the respective manufacturers by one technician from each of the laboratories (Trinity Dental Lab and Dental Art Laboratory, Thailand).
5. The authors report no financial or other conflict of interest relevant to the subject of this study.

Study Limitation

1. Due to a limited budget in this study, all brands cannot be evaluated. Thus, two ceramic systems and one resin composite commonly used in posterior teeth were chosen to be tested in this study.
2. Due to the structural variation of natural teeth, the thickness of enamel layer cannot be controlled to be equal in every unit of tooth specimens. Therefore, some areas with thin enamel layer may be exposed to the dentin layer after test. However, these areas will not be included into the measurement process since the aim of the study was to investigate merely wear on enamel.
3. For the wear test in this study; load, speed of rotation and duration were limited by the resistance of test specimen and the wear-testing machine. Therefore, the optimal values of these control variables were from the pilot study of this research together with manufacturer's recommendations for this wear tester following ASTM G 99. Further study

may increase duration of the test to compare the result at longer period of abrasive wear process.

4. There is no comparison about surface roughness and material loss of the test specimens before and after testing, since this study was not designed to investigate wear on the antagonists. Further study may include these aspects to gain more informative data.

Keywords

Abrasiveness/ Abrasive wear/ Ceramic/ Dental ceramic/ Enamel wear/ Glass ceramic/ IPS emax press/ Lava All zirconia/ Lithium disilicate glass ceramic/ Monolithic zirconia/ Pin on disc/ Resin composite/ Wear

The Expected Benefits

The expected benefits of this study are: to be another informative data regarding abrasiveness of restorative material to human enamel, to be a useful knowledge for material selection in restorative dentistry, and to be the foundation for further study in the aspect of material and method for tooth wear testing.

CHAPTER II
REVIEW OF LITERATURES

In general, wear of a material is influenced by several factors, include contact geometry, surface roughness, microstructural features, grain size, fracture toughness, speed, load, temperature, duration, environment and lubrication [6]. In dentistry, there have been attempts to figure out which factor(s) affects amount of enamel wear occurred by ceramic substrate or resin composite as these types of restorative materials are more frequently selected for rehabilitation of posterior teeth [2-5].

Abrasiveness of dental ceramics against tooth enamel

Ceramics are generally considered the most biocompatible, durable, and esthetic materials available for rehabilitation of teeth, occlusal function, and appearance. In spite of their overall excellence in meeting the ideal requirements of a prosthetic material, dental ceramics have one major drawback. These materials can cause catastrophic wear of opposing tooth structure under certain conditions.

Abrasive wear mechanisms for ceramics and tooth enamel include microfracture, which results from gouging, asperities, impact, and contact stress that cause cracks and localized fracture and the subsequent damage that a roughened ceramic surface can cause to tooth enamel surfaces. The wear of either material depends on the ease with which crack can propagate through the structure. If microscopic cracks are forced to pass around the crystal particles rather than through them, the material will usually be more fracture- and abrasion-resistant unless residual stresses enhance the propagation of the cracks through the glass phase, the particles are less fracture-resistant than the glass matrix, or excessive voids or other defects exist along the pathway [7].

There are several investigations about the effect of surface condition (rough, polished and glazed surfaces) of ceramic on enamel wear [8-12]. Krejci et al (1993) reported that the polished surface of glass ceramic caused significantly less wear than the glazed surface and also stated that wear rate of enamel depends on the

hardness, texture, and surface finish of the opposing restoration [9]. Elmaria and colleagues (2006) evaluated the 2 surface conditions –polished and glazed- of 3 ceramic substrates and found that polished Finesse and polished All-Ceram caused the least enamel wear, while glazed IPS Empress caused the most wear [11]. Kadokawa et al (2006) evaluated mutual wear rate between dental porcelain (rough and smooth surface) and opposing materials (gold, composite resin, and enamel). They found that wear rates of enamel when opposed to smooth porcelain surface were significantly lower than those opposed to rough porcelain surface [22]. Recently, a study about wear of monolithic zirconia and their corresponding enamel antagonists revealed that polished monolithic zirconia showed lower wear rate on enamel antagonists compared to veneered and glazed zirconia [12]. Another study stated that glaze layers have shown to be worn after 6 months under clinical conditions [23]. The wear of glazed layer might be the reason why glazed ceramic made more enamel roughness after wear testing in several literatures.

Hardness and fracture toughness are some of the factors that relate to abrasive ability of ceramics. However, hardness and wear tend to be poorly correlated in in vitro study, as demonstrated by Seghi et al (1991) and Magne et al (1999), suggesting the presence of more complex relationship [15, 16]. Fischer et al (1989) state that ceramic wear predominantly occurs by fracture [13]. In this paradigm, it stands to reason that the hardness plays a much smaller role and that the wear resistance is in fact ruled by its fracture toughness.

Abrasive wear may be described as 2-body abrasive wear, for example the action of a cusp on an opposing restoration or as 3-body abrasive wear when an intermediate abrasive medium comes between the two contacting surfaces [24]. Kadokawa et al (2006) reported that wear of porcelain opposing enamel in 2-body condition were significantly greater than those in the 3-body condition (this study used PMMA slurry to simulate a food bolus), regardless of the surface condition of the porcelain. It was suggested that wear by a 3-body mechanism varies with the nature of the abrasive particle used to form the paste slurry [22]. Various characteristics of abrasive medium might affect wear by 3-body mechanism [25]. Therefore, ceramic debris that exfoliate during abrasion of enamel-ceramic couple might act as a third-body particle and may have influenced the enamel wear rate or

pattern of wear [26].

In abrasion process, speed, pressure, and lubrication are also the factors affecting the rate of abrasion. From *Dental materials and their selection* by William J. O'Brien, The greater the speed at which the abrasive travels across the surface of the substrate, the greater the rate of abrasion. The greater the pressure applied, the more rapid the abrasion. Lubricants, such as silicone grease, water, and glycerol, are used to reduce heat buildup and to wash away debris to prevent clogging of the abrasive instrument. However, too much lubrication can reduce the abrasion rate because it may prevent some of the abrasive from coming in contact with the substrate [27].

Abrasiveness of resin composite against tooth enamel

Resin composite produces different amount of enamel wear depending on the different characteristics of the filler particle, in term of filler size, shape, hardness, and content [19, 20].

Abrasive wear mechanism for composite and tooth enamel occurs through adhesion [7]. Adhesion means that localized bonding of two surfaces occurs, resulting in pullout, and transfer of matter from one surface to the other. Sulong and Aziz (1990) described that adhesion or adhesive wear occurs when one solid material slides over the surface of another material or is pressed against it, causing the removal of small particle from the rubbing surfaces [28].

Mechanism of human enamel wear by resin composite was described by Shimane et al (2010) as follows: after the initial abrasion of resin composites, filler particles protruded from the abraded resin matrices, thereby resulting in increased surface roughness. Consequently, the rough surfaces and protruding hard filler particles induced enamel wear although the average hardness values of the composite resins were much lower than that of human enamel. The authors found that significant enamel wear was induced by composite resins with large protruding filler particles and that enamel wear became reduced with decrease in protruding filler size. Moreover, enamel wear was found to increase with increasing hardness of

the composite resins [20].

Methods for wear test

Since there has been no standardization of wear-related literature [16], different means of wear test might lead to various result of literatures. Therefore, comparison between studies might be difficult to summarize. Table I presented methods for wear test of enamel and their antagonists from some of the literatures from 1989 to 2013.

Table I. Methods for enamel wear test from some of the literatures from 1989 to 2013.

Literature	Wear tester	Enamel specimen or its substitute	Measuring instrument (required outcome)		Load	Speed	Cycle or duration	Sample size per group
			Enamel specimen	Material specimen				
DeLong et al, 1989	Artificial mouth	Mesiolingual cusp of maxillary molar	Computer graphic (depth, area, volume of wear) Microscope, SEM	Computer graphic (max depth and volume of wear) Microscope, SEM	13.35 N	4 Hz	300,000 cycles	5
Jacobi et al, 1991	Abrasion machine	Cusp tip of canine	A scale (weight before-after testing)	A scale (weight before-after testing)	40 N	58 rpm	8,000 cycles	20
Ratledge et al, 1994	Stress cycling machine	Semicircular section of premolar	A computer image analysis program (area loss)	Profilometer (depth)	40 N	70 stroke/min	25,000 cycles	6
Hudson et al, 1995	Wear machine (back-and forth stroke)	Cusp of molar	Optical comparator (vertical height loss, area loss)	Profilometer (roughness)	178.35 g	n.i.	10,000 cycles	10
Jagger et al, 1995	Follow Harrison&Lewis 1975 (stud and plate)	Buccal surface of incisors, canines and premolars (plate specimen)	Profilometer (depth)	Bench micrometer (length loss)	395 g (0.4MPa)	n.i.	62 hours	5
Al-Hiyasat et al, 1998	Dental wear machine (lower rotating disc)	Buccal cusp of lower premolar	A reflex microscope (cusp height reduction)	Profilometer (depth, roughness)	40 N	80 rpm	25,000 cycles	10
Metzler et al, 1999	Pin-on-plate wear model	Cusp of premolars and molar	A digital micrometer (volume loss)	Profilometer (depth, roughness) SEM (wear patterns)	600 g	60 rpm	6, 12, 24, 48 hours	12
Magne et al, 1999	Artificial mouth follow DeLong 1983	Mesiolingual cusp of maxillary third molar	Computer graphic (mean depth, volume of wear) SEM (wear patterns)	Computer graphic (mean depth, volume of wear) SEM (wear patterns)	13.5 N	4 Hz	300,000 cycles	5
Clelland et al, 2001	Oral wear simulator	Cusp of third molar	Optical scanning method (area of wear facet) SEM (wear patterns)	SEM (wear patterns)	20 N	1 Hz	50,000 cycles	10
Elmaria et al, 2006	Custom-constructed wear machine	Palatal cusp of maxillary molar Buccal cusp of mandibular molar	Profile projector (cusp height loss)	Profilometer (roughness) SEM (wear patterns)	180 g (1.8 N)	n.i.	10,000 cycles	10
Preis et al, 2011	A pin-on-block design	1) Steatite ball (MgSilicate) 2) Cusp of human molars	Optical 3D profilometer (vertical loss, area loss) SEM (wear patterns)	Optical 3D profilometer (vertical loss) SEM (wear patterns) Profilometer (roughness)	50 N	1.6 Hz	1.2×10^5 cycles	16
Stawarczyk et al, 2013	Chewing simulator	Mesiobuccal cusp of maxillary molar	3D Profilometer (vertical loss) SEM (wear patterns)	3D Profilometer (vertical loss) SEM (wear patterns)	49 N	1.7 Hz	1.2, 2.4, 6.4, 12 $\times 10^5$ cycles	6

ni = no information available

CHAPTER III

MATERIALS AND METHODS

Research Design

Experimental research

Sample Description

1. The population of this study was occlusal enamel of human permanent molars.

2. Sample size estimation was calculated from this formula;

$$N = \frac{4\sigma^2(Z_{crit} + Z_{pwr})^2}{D^2}$$

Where: N represents the required sample size per group

σ^2 represents the variance of the variable as estimated by the data from pilot study (Estimated standard deviation = 2.5)

D represents Minimum expected difference (6)

Z represents the Z value (Z_{crit}= 1.96, Z_{pwr}= 0.84)

At 95% confident interval and 80% power of test, the result from sample size estimation was 5.45. Therefore, the number of specimens per group in this study should be 6.

Materials

1. Human permanent molars. *(Ethic approval no. 042/2012)*
2. Two systems of all-ceramic antagonist
   a. Monolithic zirconia (Lava All Zirconia, 3M ESPE, Seefeld, Germany)
   b. Lithium-disilicate glass-ceramic (IPS e.max Press, Ivoclar vivadent, Amherst, NY)
3. Resin composite (Premise, Kerr, Orange, CA)
4. Apoxy resin (Huntsman, Huntsman Advanced Materials Americas Inc., Houston, Texas)
5. Transparent silicone (TempSpan Clear, Kerr)
6. Silicon carbide abrasive paper (400, 800, 1200 grit)
7. Cylindrical tube with 5-mm screw tap (diameter: 20 mm, height: 10 mm)
8. Cylindrical brass rod (diameter 3 mm)
9. Resin cement (Super-bond C&B, Sun medical, Japan)

Methods of Data collection

Preparation of test specimens (antagonists)

Material used in this study are presented in Table II. 24 test specimens separated into 4 types of test material with 6 pieces for each group were fabricated into cylindrical rods (3 mm in diameter and 10 mm in length). A flat circular surface of any test material was finished with a polishing kit (Jota All Ceramic Kit 1369, Jota AG, Rüthi SG, Switzerland). A mean radius of 1.5 mm was selected for the test due to the pin-on-disc wear tester used in this study (Model TE 79; Plint & Partners Ltd., Berkshire, England) (Figure 1) could accept this size of material, following ASTM G99 (Standard test method for wear testing with a pin-on-disc apparatus).

Table II. List of materials selected for wear tests and some of their properties.

Materials	Product	Fracture toughness (MPa.m1/2)	Vicker hardness (GPa)	Elastic modulus (GPa)	Manufacturer
Monolithic zirconia	Lava All Zirconia	8-10.3[a]	8.8-11.8[a]	210[a]	3M ESPE, Seefeld, Germany
Lithium-disilicate glass-ceramic	IPS e.max Press	2.2-3.3[a]	6.3[a]	95-103[a]	Ivoclar Vivadent, Amherst, NY
Resin composite	Premise	1.32[b]	0.55-0.58[b]	11-15[a]	Kerr, Orange, CA
Human occlusal enamel	-	0.77[a]	3.23-3.62[a]	84[a]	-

[a]From Anusavice KJ. Phillips' science of dental materials. 12th ed. St Louis: Elsevier; 2013. p66, 284, 453

[b]From manufacturer's data

1. The zirconia pin was milled from a monolithic block (Lava All Zirconia, 3M ESPE, Seefeld, Germany) by one lab technician at the Trinity dental lab, Bangkok, Thailand.

2. The fabrication of the lithium-disilicate glass-ceramic pin was undertaken in accordance with the manufacturer's recommendations (IPS e.max Press, Ivoclar Vivadent, Amherst, NY). A cylindrical rod with a constant diameter of 3 mm was waxed, after which each of the wax patterns was finally checked by an individual investigator. All of the wax patterns were invested and pressed by one lab technician at the Dental Art Laboratory, Bangkok, Thailand.

3. The resin composite pin was formed by a mold made of transparent silicone material (TempSpan Clear, Kerr, Orange, CA). After loading proper amount of resin composite (Premise, Kerr) into the mold, the resin composite was light-cured with the LED light curing unit (450-470 nm, Demi, Kerr).

4. The human enamel pin was derived from the occlusal surface of permanent third molar and was prepared by one operator. To make a 10-mm length of enamel pin, a piece of 3 mm-diameter occlusal enamel was attached to a tip of brass rod by resin cement (Super-Bond C&B, Sun Medical, Moriyama, Japan)

Preparation of enamel specimens

Inclusion criteria: freshly extracted unrestored non-carious human permanent molars. Occlusal dimension is large enough to obtain flat circular area of entirely enamel with diameter at least 8 mm.

Exclusion criteria: human permanent molars with enamel defects, hypomineralized enamel.

24 human permanent molars were cleaned with ultrasonic scaler and stored in 0.1% thymol solution. All teeth were randomly divided into each group of antagonists.

To prepare enamel specimen, occlusal surface was ground down using rotary cutting instrument in the presence of water until obtaining flat circular area entirely of enamel with a diameter of at least 8 mm in order to enable it to undergo wear testing by pin-on-disc apparatus (Figure 2). The enamel surface was confirmed by viewing through a stereomicroscope (ML 9300, Meiji Techno, Saitama, Japan).

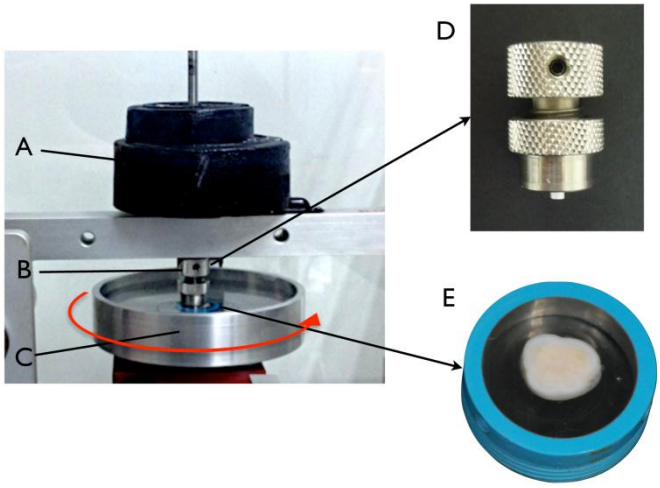

Figure 1. A pin-on-disc wear tester: (A) constant load; (B) upper specimen holder; (C) lower specimen holder; (D) ceramic pin inserted into the upper specimen holder; (E) Enamel specimen.

The enamel specimen was embedded in the middle of a cylindrical tube using epoxy resin (Huntsman, Woodlands, Texas) (Figure 1E). Only wide pit(s) or fossa(e) presenting on the occlusal surface after flattening were filled with flowable composite (Premise Flowable; Kerr) and light-cured with a LED light curing unit (450–470 nm) in order to avoid errors from macroscopic roughness during the test. Afterward, all prepared enamel specimens were finished with silicon carbide abrasive papers (400, 800 and 1,200 grit, respectively) under running water for 2 min each with a revolving polishing machine (Nano 2000 grinder-polisher; Pace Technologies, Tucson AZ).

Intervention

Wear tests were conducted using a pin-on-disc wear tester. The test specimen (antagonist) was inserted into the upper specimen holder. A screw inside the slot

could be used to adjust the specimen vertically. The test specimen was controlled to project at 5 mm length from the opening of the holder (Figure 1D). The upper specimen holder could be inserted and tightened to the lever arm of the device. The enamel specimen was also attached to the lower specimen holder, which could be run in rotational movement (counter-clockwise direction). The wear machine was connected to the electrical supply through a control system, by which the rate of cycling and duration of the test could be set and monitored through a digital counter device.

Wear tests were performed with a load of 25 N, 20 cycles/min for 240 min (4,800 cycles). These control parameters were determined from the pilot study of this research together with the manufacturer's recommendations for the wear tester according to ASTM G99. The center of the upper specimen surface was set at 2 mm from the center of rotation (x-position: 2 mm) (Figure 2). The samples were tested in distilled water, which was renewed after each test.

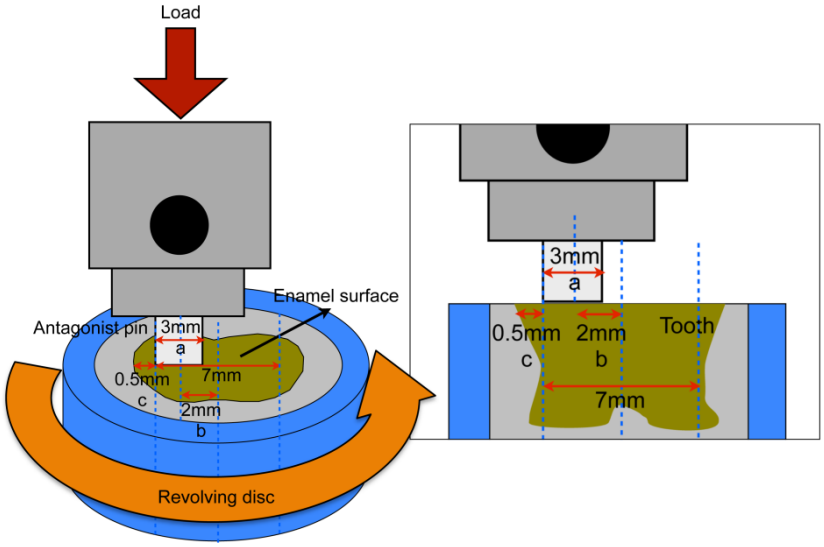

Figure 2. Illustration of rationale for the occlusal enamel diameter: (a) diameter of antagonist pin, (b) x-position from center of rotation, (c) minimal distance from the interface between tooth and epoxy resin.

Data collection

Maximum depth of wear (D_{max}) and mean depth of wear (D_a) of human enamel specimens were evaluated using a profilometer (Talyscan 150; Taylor Hobson, Leicester, England). Five measurements of wear track depth were made on each specimen (speed = 1,500 μm/s, spacing = 1 μm). Each measurement was at least 15° of angulation away from each other (Figure 3). Errors from depths of pit(s) and/or groove(s) were excluded.

Mean surface roughness (R_a) before (baseline) and after testing of the enamel specimens were determined using the same profilometer with a 0.008 mm Gaussian filter. The transverse length was set at 1 mm. Five measurements per specimen were made for each R_a value. Baseline measurements were made on unworn portions of enamel adjacent to the worn areas [17].

For the qualitative characterization of wear patterns, all test materials and enamel specimens were evaluated under scanning electron microscopy (SEM) (JSM - 5410 LV; JEOL, Tokyo, Japan). The surfaces were examined at a magnification of 50-350 at 15 keV.

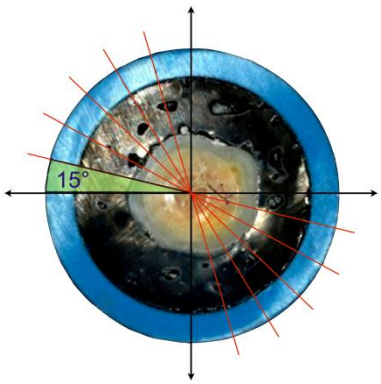

Figure 3. Linear measurements of enamel wear depths.

Statistical Analysis

The effect of the materials tested on enamel wear depth and R_a was evaluated using one-way analysis of variance, followed by a Tukey's test to compare all possible pairs of means at a 95% confidence interval. Paired-sample *t*-tests were used to compare R_a of enamel specimens between baseline and after testing for all types of materials tested. Results with a *P*-value < 0.05 were considered statistically significant.

CHAPTER IV
RESULTS

The results of enamel wear depth (D_{max}, D_a) are recorded in Tables III and IV. For both D_{max} and D_a, no statistically significant differences were found between those of resin composite and monolithic zirconia (subset 1) and those of lithium-disilicate glass-ceramic and human enamel (subset 2); however, a significant difference was revealed between these two subsets ($P < 0.001$).

Table III. Distribution of maximum depth of wear (D_{max}) of enamel for all test specimens.

Enamel wear depth	Test specimens				Significance (one-way ANOVA)
	Monolithic zirconia	Lithium-disilicate glass-ceramic	Resin composite	Human enamel	
D_{max} (μm) (mean ± sd)	2.17 ± 0.80[a]	8.54 ± 2.31[b]	1.70 ± 0.92[a]	10.72 ± 6.31[b]	<0.001

Values with the same lowercase letter are not significantly different at $P < 0.05$

Table IV. Distribution of mean depth of wear (D_a) of enamel for all test specimens.

Enamel wear depth	Test specimens				Significance (one-way ANOVA)
	Monolithic zirconia	Lithium-disilicate glass-ceramic	Resin composite	Human enamel	
D_a (μm) (mean ± sd)	1.83 ± 0.75[a]	7.32 ± 2.06[b]	1.37 ± 0.81[a]	8.81 ± 5.16[b]	<0.001

Values with the same lowercase letter are not significantly different at $P < 0.05$

To compare R_a of enamel in each material group before and after wear testing, paired t-tests were conducted; the results are shown in Table V. R_a of enamel specimens increased significantly after wear tests with monolithic zirconia (P = 0.005), glass-ceramic (P = 0.001) and enamel (P < 0.001); however, no difference was found among these materials (Table VI). Resin composite was the only material that produced no significant difference in R_a of the enamel specimen before and after wear testing (P = 0.354).

Table V. Comparison of mean surface roughness (R_a) of enamel between baseline and after wear testing for all test specimens.

Test specimen	R_a (nm) (mean ± sd)		Significance (two-tailed)
	At baseline	After test	
Monolithic zirconia	1.64 ± 0.98	3.02 ± 0.68	0.005
Lithium-disilicate glass-ceramic	1.63 ± 0.08	3.19 ± 0.56	0.001
Resin composite	1.66 ± 0.15	1.51 ± 0.22	0.354
Human enamel	1.64 ± 0.15	3.38 ± 0.54	<0.001

Table VI. Distribution of mean surface roughness (R_a) of enamel after abrasion against test specimens.

Enamel surface roughness	Test specimens				Significance (one-way ANOVA)
	Monolithic zirconia	Lithium-disilicate glass-ceramic	Resin composite	Human enamel	
R_a (nm) (mean ± sd)	3.02 ± 0.68[a]	3.19 ± 0.56[a]	1.51 ± 0.22[b]	3.38 ± 0.54[a]	<0.001

Values with the same lowercase letter are not significantly different at P < 0.05

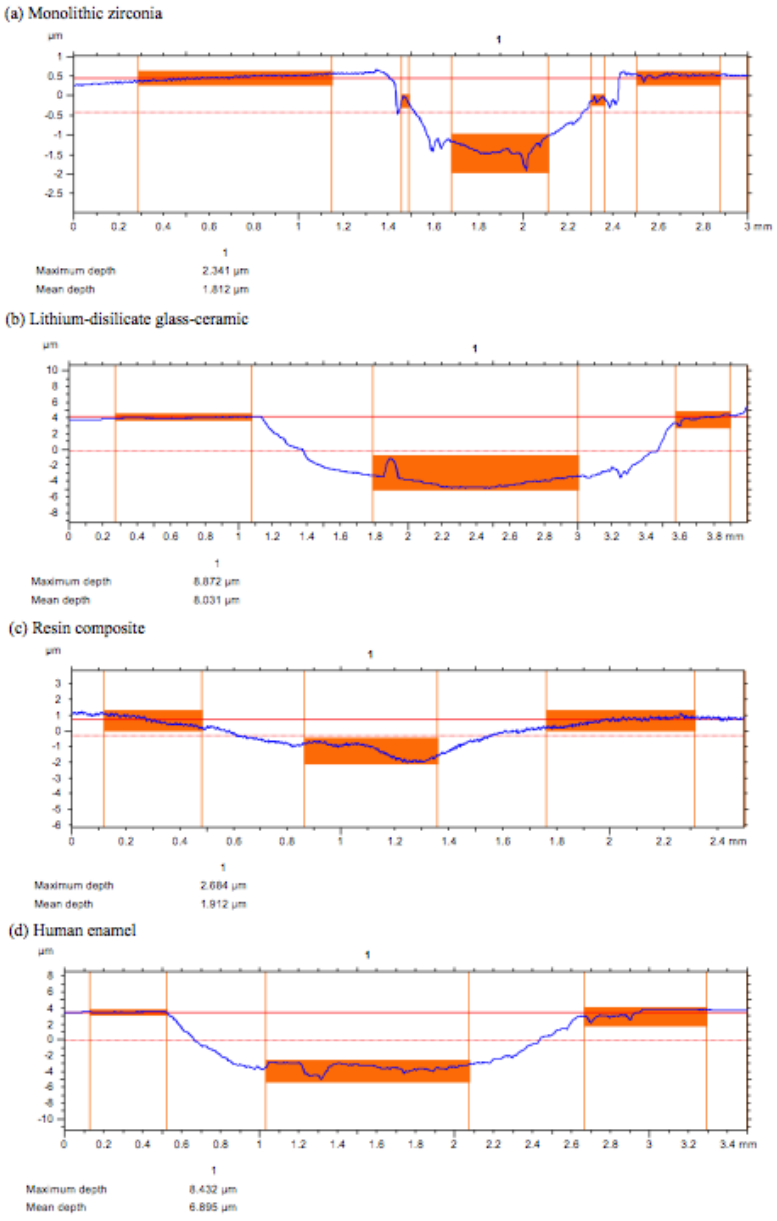

Figure 4. Examples of enamel wear profiles of each type of antagonists obtained after wear testing.

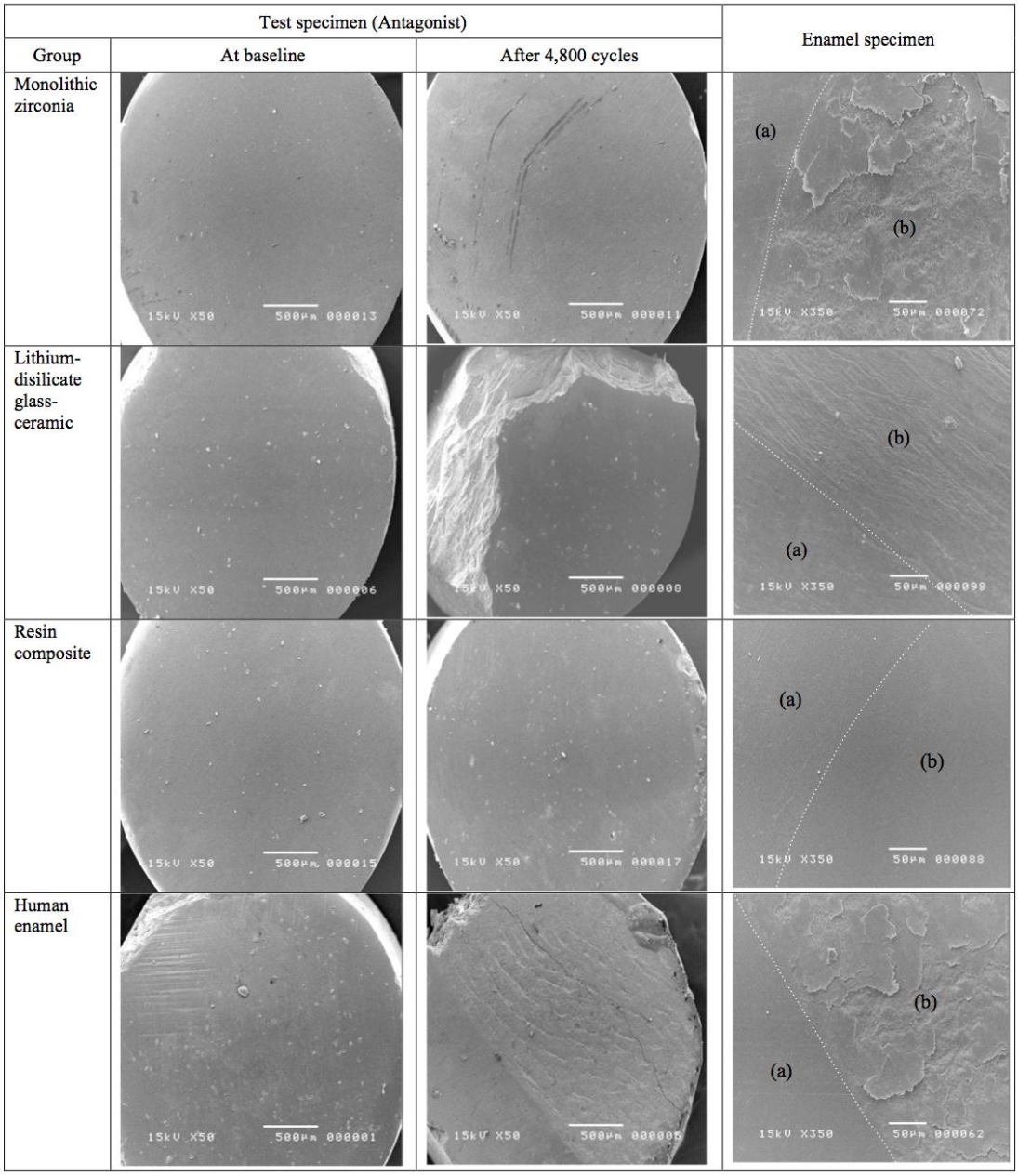

Figure 5. SEM pictures of surfaces of test specimens at baseline and after 4,800 cycles of wear testing and their enamel surfaces at unworn (a) and worn (b) areas.

The qualitative characterizations of wear of all test specimens and their enamel specimens are illustrated in Figure 5. After the wear test, monolithic zirconia showed some scratches on its abraded surface, lithium-disilicate glass-ceramic

showed some cracks and chipping, resin composite showed slightly different surface compared to its beginning, and human enamel showed rough plowed surface with craze line. It was noticed that enamel specimen of resin composite group showed little to no wear on the worn zone.

CHAPTER V

DISCUSSION

The outcomes of this study revealed significant differences in enamel wear depth (D_{max}, D_a) and R_a of enamel among all materials after wear testing. Therefore, the first hypothesis was rejected. Within the limitations of this study, the results showed that the least enamel wear depth was produced by resin composite and monolithic zirconia. In addition, a comparison between enamel R_a before and after testing showed that resin composite was the only material that caused similar R_a values and was supported by its SEM image (Figure 5). Thus, the second hypothesis of this study was accepted only for the resin composite group. Since the wear mechanisms for dental restorative materials and tooth enamel differ depending on type of material, the rational explanation of these findings should be considered separately – wear of ceramic (as well as enamel) occurs due to a microfracture mechanism, while metal and composite wear is due to adhesion [7].

Concerning enamel wear by ceramics, an interesting outcome is that monolithic zirconia does not cause greater wear of human enamel compared with lithium-disilicate glass-ceramic. This observation about the small amount of antagonist wear by zirconia has some connections with the investigation by Preis et al [29]. They reported lower wear of steatite antagonist against zirconia compared to veneering porcelain. Their SEM images exhibited a comparable range of steatite- and enamel-wear areas, and also showed that enamel was polished when opposed to zirconia but ground when opposed to veneering porcelain. In our study, the possible explanation of enamel wear between zirconia and glass-ceramic is that zirconia is less susceptible to the microfracture mechanism than glass-ceramic due to the much higher fracture resistance of zirconia (Table II). Fracture toughness of the material is a key to the prevention of cracking [13]. Besides, the microfracture mechanism is considered to be the dominant mechanism responsible for surface breakdown of ceramic and the subsequent damage that a roughened ceramic surface can cause to enamel surfaces [7]. Consequently, under the same condition of wear process, the

microcrack is probably more difficult to propagate through the crystalline structure of zirconia. Hence, the zirconia surface remains smoother because fewer microfractures occur during abrasive wear. The smoother surface of zirconia throughout the test, as shown by its SEM image (Figure 5), leads to the lower wear depth of opposing enamel (Figure 4). On the contrary, the roughened surface of glass-ceramic causes more depth of enamel wear due to the increased development of microfractures along the surface (Figure 5).

Surface roughness of the ceramic surface is taken into account. An in vitro study by Kadokawa, Suzuki and Tanaka showed that the wear rate of enamel when opposed to a smooth porcelain surface was significantly lower than when opposed to a rough porcelain surface [22]. In this study, rough ceramic surfaces or asperities originated during the period of the abrasive wear process (Figure 5). This might relate to the clinical situation when polished restorations are in daily function and then start to develop roughness on the contact surfaces. Moreover, differences in wear rates of mutual opposing teeth and/or restorations might alter an individual's occlusal relationship [30]. Thus it should be kept in mind that periodically checking on the occlusion and maintaining the smoothness of restoration surfaces might be necessary [31].

Another possibility of higher enamel wear by glass-ceramic might arise from the formation of wear debris. Glass particles that come off during the wear process might behave as an abrasive medium and lead to a three-body wear mechanism [3]. These abrasive particles might emphasize the consequences of enamel wear. Although this wear test was run under distilled water, which would help lubricate the contact surface, flush out debris and reduce heat generation from abrasion, some wear debris may still remain in the wear track and influence the contact stresses and wear [18].

Modern resin composites are widely used as posterior resin composites due to the improvement of their physical and mechanical properties, particularly in filler composition, size and morphology [20]. There has been an attempt to develop a composite that is resistant to wear from the opposing dentition, and also does not

cause excessive wear of human enamel. Unlike the case of ceramics, hardness is suggested to be a reliable predictor of enamel wear by resin composite, as the wear of enamel occurs through hard filler protruding from the abraded resin matrix, and the amount of enamel wear is directly correlated with the composite's hardness value [19, 20]. The resin composite used in this study (Premise; Kerr) has the least hardness value compared with other test materials (Table II). According to the general knowledge about wear between two contacting materials, softer material is abraded more easily than harder material [20]. Thus, the amount of enamel wear produced by composite would be less, and this supposition was supported by the results of this study. However, as the wear behavior of a composite is different from that of a brittle substrate (ceramic or enamel), hardness could not be used as a wear predictor for other test materials.

Although both resin composite and monolithic zirconia produced the least enamel wear depth, the SEM image of enamel specimen abraded by monolithic zirconia showed some wear and cracks, while enamel specimen abraded by resin composite showed no wear or even smoother surface compared to baseline (Figure 5). This SEM investigation conformed with the results of enamel roughness after wear testing obtained by profilometry (Table V). The reason why resin composite was the only material that made similar R_a values might also be answered by its hardness value.

It was noted that enamel wear by enamel made a significant depth of wear, together with high standard deviation. Similar findings were obtained by Ratledge et al (1994) who suggested that three-body wear occurred because of chipped hydroxyapatite particles acting as an abrasive medium [3]. Regarding the high variation of the results, one possible supposition is the lack of homogeneity in natural enamel [29, 32]. Not only variations between teeth have an influence on this, but also variations within individual teeth: that is, the different position of enamel on the tooth results in different properties of the enamel [7]. This test group consisted of human occlusal enamel in both upper and lower members. Thus, a high scattering of the result was anticipated.

Regarding the methods of wear testing, the amount and duration of load, as well as speed, are some of the factors that influence the amount of enamel wear [27]; The greater the speed at which the abrasive moves along the surface of the substrate, the greater the rate of abrasion; also, the greater the pressure applied, the more rapid the abrasion. The lack of standardization is a problem found in wear-related literature [16, 33]. Dissimilarities in the testing method may lead to a different outcome in any individual study, so it is difficult to directly compare the present result with various prior investigations. Moreover, the pin-on-disc wear tester used in this study was not invented for simulation of human masticatory function; therefore it is difficult to directly imply the study's result to use in clinical practice. However, the constant contact of the specimens thorough the wear process might resemble characteristics of grinding or clenching habits.

Conclusions

This in vitro study investigated wear of human enamel by examining the enamel wear depth and surface roughness when opposed to dental ceramics (monolithic zirconia, glass ceramic) and resin composite.

Within the limitations of this in vitro study, the following conclusions can be drawn:

The depth of enamel wear by monolithic zirconia and resin composite was significantly lower than those by glass-ceramic and enamel.

Surface roughness of enamel specimens worn by glass-ceramic, monolithic zirconia and enamel increased significantly after wear testing, but no significant difference was found among these materials. For the resin composite group, surface roughness of enamel specimen before and after wear tests was not significantly different.

Implication of the result of this study

With regard to enamel wear by restorative materials in this study, resin composite seems to be the least abrasive material to be used in the posterior region. However, enamel wears by ceramics are comparable with those by enamel, suggesting the feasibility to use these kinds of ceramic material.

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APPENDICES

Appendix A. Copy of study protocol and consent form approval *(ethic approval no. 042/2012)*

No. 042/2012

Study Protocol and Consent Form Approval

The Human Research Ethics Committee of the Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand has approved the following study to be carried out according to the protocol and patient/participant information sheet dated and/or amended as follows in compliance with the **ICH/GCP.**

Study Title	: Wear of human enamel opposing monolithic zirconia, glass-ceramics and resin composite
Study Code	: HREC-DCU **2012-044**
Study Center	: Chulalongkorn University
Principle Investigator	: Ms. Jeerapa Sripetchadanond
Protocol Date	: August 1, 2012
Date of Approval	: September 4, 2012
Date of Expiration	: September 3, 2014

S. Amatyakul

(Associate Professor Dr. Supathra Amatyakul)
Chairman of Ethics Committee

(Assistant Professor Dr. Suchit Poolthong)
Associate Dean for Research and International Affairs

*A list of the Ethics Committee members (names and positions) present at the Ethics Committee meeting on the date of approval of this study has been attached (upon requested). This Study Protocol Approval Form will be forwarded to the Principal Investigator.

Approval is granted subject to the following conditions: (see back of the approval)

Appendix B. Descriptive statistic of maximum depth of wear (D_{max}) for all test groups (n = 6)

Test materials	Mean	SD	SE	95% Confidence interval for Mean		Minimum	Maximum
				Lower bound	Upper bound		
Monolithic zirconia	2.169547	.7965046	.3251717	1.333666	3.005427	1.1510	3.3600
Lithium-disilicate glass-ceramic	8.539100	2.3058304	.9413513	6.119279	10.958921	5.8484	11.4734
Resin composite	1.697005	.9240332	.3772350	.727292	2.666718	.7058	3.2878
Human occlusal enamel	10.721487	6.3143099	2.5778062	4.095025	17.347949	4.8818	19.3048

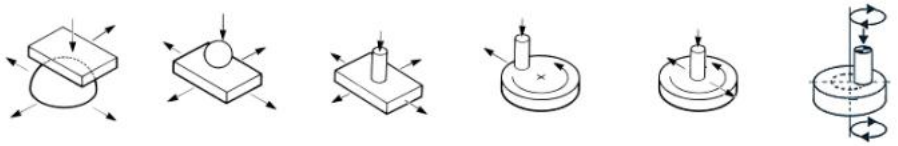
Appendix C. Descriptive statistics of mean depth of wear (D_a) for all test groups (n = 6)

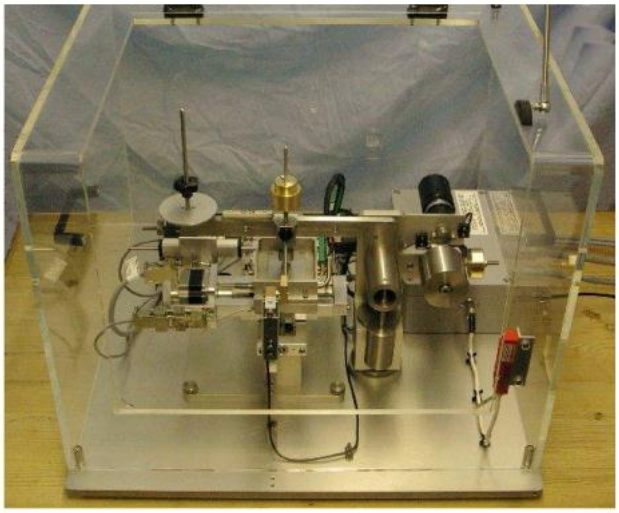
Test materials	Mean	SD	SE	95% Confidence interval for Mean		Minimum	Maximum
				Lower bound	Upper bound		
Monolithic zirconia	1.831857	.7519774	.3069935	1.042705	2.621009	.8676	3.0082
Lithium-disilicate glass-ceramic	7.319160	2.0648238	.8429608	5.152260	9.486060	5.1158	10.1374
Resin composite	1.371128	.8145260	.3325288	.516336	2.225921	.5526	2.8094
Human occlusal enamel	8.805827	5.1648524	2.1085422	3.385646	14.226007	4.2386	15.9064

Appendix D. Specification of the wear tester (TE 79 multi-axis tribometer)

PLINT Tribology Products
from Phoenix Tribology Ltd

TE 79 MULTI-AXIS TRIBOMETER





Description:

The TE 79 Multi-Axis Tribometer is for friction and wear testing of materials under low loads in pin or ball on disc or reciprocating plate configurations. In pin on disc mode the machine can perform tests according to ASTM G 99 and DIN 50 324 and provides a Class 1 contact configuration (pin or ball loaded vertically downwards onto a horizontally rotating disc). In both pin on disc and pin on plate modes, the indexing capability allows tests to be performed in accordance ASTM G132 Standard Test Method for Pin Abrasion Testing, which requires indexation of the pin so that it is always presented with a fresh abrasive surface. The Tribometer is modular, with two possible configurations, each used in conjunction with the TE 79 Base Unit.

TE 79 Base Unit:

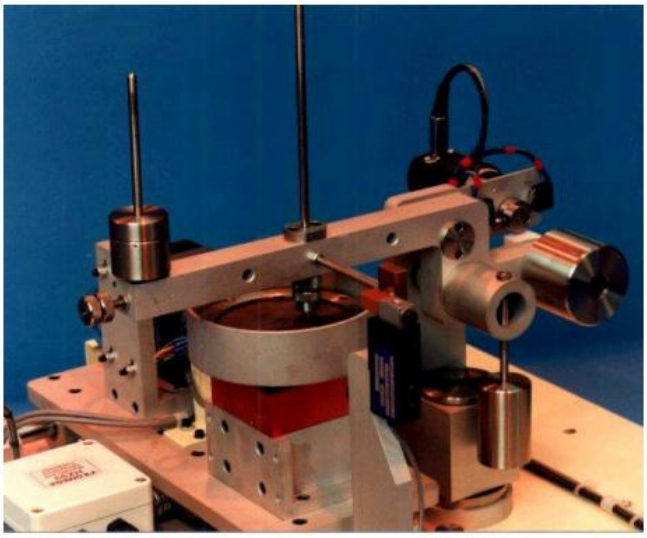
This comprises the loading and friction force measurement system mounted on a base plate, control hardware with PLINT SLIM 2000 serial interface unit and control software. The machine is bench-top mounted and includes a transparent enclosure and ambient humidity and temperature sensor. The enclosure is also used as a safety cover for the machine and incorporates a magnetic proximity switch. The machine will not run if the enclosure is removed.

The fixed pin or ball sample is carried on a trunnion and gimble mounted loading beam. This is counterbalanced both to give a neutral balance and to bring the centre of gravity onto the contact plane. Load is applied by dead weights in a range from 0.1 N to 50 N.

The loading beam is restrained by a strain gauge force transducer in a sliding link. This link ensures that only the tangential component of force in the contact (the friction force) is measured even with the large deflections associated with elastomeric test pieces. As the lower specimen surface moves the friction force on the ball or pin sample is measured.

The load beam lift/lower is servo controlled so that the load can be applied at a specific point in the test. The program can also introduce a dwell between load application and movement. This dwell period is an important parameter in determining the start-up friction in elastomeric contacts.

TE 79/P Indexing Pin on Disc Module:



This Module comprises a rotating disc assembly mounted on a cross slide, thus allowing the pin sample to follow a spiral track on the disc, if required. Rotary and translatory motions are driven by stepper motors. The module locates on the base plate of the TE 79 Base Unit and is fixed in place with locating screws.

The disc specimen is mounted in a reservoir to retain lubricating fluid. The reservoir is mounted on a vertical drive shaft assembly. This is mounted on a traversing slide, which permits the radius to be changed during a test. The control software may be set to run with a constant rpm or constant velocity during a traverse.

TE 79/R Indexing Reciprocating Module:

The Module locates on the base plate of the TE 79 Base Unit and is fixed in place with locating screws. It provides X/Y axis movement with linear positional feedback. Tangential (friction) force measurement is in the X direction. The axes are formed by cross-axis linear slides with 1 mm pitch lead screws and are driven by stepper motors.

The fixture for the lower (moving) specimen includes an electrical resistance heater and two thermocouples for temperature measurement and control above ambient conditions.

A programmable motion controller is used to coordinate movement of the two axes. Numerous motions are possible including:

Simple reciprocating along one track in the X direction.
Reciprocating in the X direction with indexing in the Y direction at stroke end, so that the wear track resembles a square wave.
Reciprocating in the X direction with indexing in opposite Y directions at stroke end, so that the wear track is rectangular.
Simultaneous indexing on both the X and Y axes so that the pin follows a circular or elliptical track with an orbiting (rotating friction vector) motion.

Test Environment:

The TE 79 Base Unit is provided with a plastic safety cover, which also acts as a chamber for the user to run under controlled humidity conditions. An ambient temperature and humidity sensor is mounted on the machine base inside the chamber.

TE 79/R/C Peltier Cooler:

This test assembly replaces the standard fixed specimen heater block on the TE 79/R Indexing Reciprocating Module with a Peltier cooler pad. With water-cooling of the hot side of the Peltier devices temperatures from -15°C to ambient may be achieved.

Used in conjunction with the RE 79/R/C Laboratory Chiller unit with water/glycol mixture as the coolant, temperatures from -35°C to ambient may be achieved. To avoid ice formation, this adapter is best used in conjunction with a simple desiccant dehumidifier system used in conjunction with a controlled air supply.

Control and Data Acquisition:

The TE 79 has PC based sequence programmable control and data acquisition. This is provided by an integrated Serial Link Interface Module and COMPEND 2000 software running on a host PC, operating under Windows. Data is stored to hard disc in standard spread sheet compatible file formats (.csv or .tsv).

Tests are defined by a sequence of steps, each step containing set-point, data recording rates and alarm level information. Set-points may be adjusted by step change or ramp. The test sequence is followed unless interrupted by the operator or an alarm. Set-points may also be adjusted manually using on screen toggles.

Technical Specifications:

Normal Load:	0.1 to 50 N
Friction Force Range:	0 to 50 N
Humidity Sensor:	10 to 90% RH
Interface:	SLIM 2000 Serial Link Interface Module
Software	COMPEND 2000

Controlled Parameters

X Position (TE 79/R)
RPM (TE 79/P)
X Axis Speed (TE 79/R)
Y Position (TE 79/P and TE 79/R)
Y Axis Speed (TE 79/P and TE 79/R)
Temperature (TE 79/R)
Dwell Period
Test Duration

Recorded Parameters

X Position (TE 79/R)
Y Position (TE 79/P and TE 79/R)
Humidity
Ambient Temperature
Temperature (TE 79/R)
Friction
Friction Coefficient

TE 79/P Indexing Pin on Disc Module:

Contact Configurations:	Ball on Disc Pin on Disc
Disc Diameter:	100 mm
Track Radius:	0 to 40 mm
Y Traverse Speed:	10 mm/min
Rotation Speed:	0 to 250 rpm
Sliding Speed:	up to 1 m/s

TE 79/R Indexing Reciprocating Module:

Contact Configurations:	Ball on Plate Plate on Plate Plate on Hemisphere
Maximum X Axis Speed:	10 mm/s
Maximum X Stroke:	50 mm
Maximum Y Axis Speed:	10 mm/s
Maximum Y Stroke:	30 mm
Temperature Range:	ambient to 100°C
Dwell (time delay):	User selected in seconds up to 8 hours
Temperature Sensor:	J-type thermocouple
Heating Power:	150 W

TE 79/R/C Peltier Cooler:

Minimum Temperature:	-15°C (ambient water cooled)
Minimum Temperature:	-35°C (chiller water/glycol cooled)

RE 79/R/C Laboratory Chiller

Working Fluid:	50:50 Water/Glycol
Minimum Fluid Temperature:	-30°C

Services:

Electricity:	220/240 V, single phase, 50 Hz, 720 W 110/120 V, single phase, 60 Hz, 720 W

Installation:

Bench-mounting machine:	570 mm x 600 mm x 600 mm high, 40 kg
Bench-mounting controller:	530 mm x 530 mm x 240 mm high, 20 kg
Packing Specifications:	0.59 m^3, GW 120 kg, NW 70 kg

Order As:

TE 79	Multi-Axis Tribometer Base Unit
TE 79/P	Indexing Pin on Disc Module
TE 79/R	Indexing Reciprocating Module
TE 79/R/C	Peltier Cooler
RE 79/R/C	Laboratory Chiller

Appendix E. Copy of standard test method for wear testing with a pin-on-disc apparatus (ASTM G99)

Designation: G99 – 05 (Reapproved 2010)

Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus[1]

This standard is issued under the fixed designation G99; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a laboratory procedure for determining the wear of materials during sliding using a pin-on-disk apparatus. Materials are tested in pairs under nominally non-abrasive conditions. The principal areas of experimental attention in using this type of apparatus to measure wear are described. The coefficient of friction may also be determined.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E178 Practice for Dealing With Outlying Observations

G40 Terminology Relating to Wear and Erosion

G117 Guide for Calculating and Reporting Measures of Precision Using Data from Interlaboratory Wear or Erosion Tests

2.2 *DIN Standard:*[3]

DIN 50324 Testing of Friction and Wear

3. Summary of Test Method

3.1 For the pin-on-disk wear test, two specimens are required. One, a pin with a radiused tip, is positioned perpendicular to the other, usually a flat circular disk. A ball, rigidly held, is often used as the pin specimen. The test machine causes either the disk specimen or the pin specimen to revolve about the disk center. In either case, the sliding path is a circle on the disk surface. The plane of the disk may be oriented either horizontally or vertically.

NOTE 1—Wear results may differ for different orientations.

3.1.1 The pin specimen is pressed against the disk at a specified load usually by means of an arm or lever and attached weights. Other loading methods have been used, such as hydraulic or pneumatic.

NOTE 2—Wear results may differ for different loading methods.

3.2 Wear results are reported as volume loss in cubic millimetres for the pin and the disk separately. When two different materials are tested, it is recommended that each material be tested in both the pin and disk positions.

3.3 The amount of wear is determined by measuring appropriate linear dimensions of both specimens before and after the test, or by weighing both specimens before and after the test. If linear measures of wear are used, the length change or shape change of the pin, and the depth or shape change of the disk wear track (in millimetres) are determined by any suitable metrological technique, such as electronic distance gaging or stylus profiling. Linear measures of wear are converted to wear volume (in cubic millimetres) by using appropriate geometric relations. Linear measures of wear are used frequently in practice since mass loss is often too small to measure precisely. If loss of mass is measured, the mass loss value is converted to volume loss (in cubic millimetres) using an appropriate value for the specimen density.

3.4 Wear results are usually obtained by conducting a test for a selected sliding distance and for selected values of load and speed. One set of test conditions that was used in an interlaboratory measurement series is given in Table 1 and Table 2 as a guide. Other test conditions may be selected depending on the purpose of the test.

3.5 Wear results may in some cases be reported as plots of wear volume versus sliding distance using different specimens for different distances. Such plots may display non-linear relationships between wear volume and distance over certain portions of the total sliding distance, and linear relationships

[1] This test method is under the jurisdiction of ASTM Committee G02 on Wear and Erosion and is the direct responsibility of Subcommittee G02.40 on Non-Abrasive Wear.

Current edition approved April 1, 2010. Published April 2010. Originally approved in 1990. Last previous edition approved in 2005 as G99–05. DOI: 10.1520/G0099-05R10.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Beuth Verlag GmbH (DIN-- DIN Deutsches Institut fur Normung e.V.), Burggrafenstrasse 6, 10787, Berlin, Germany, http://www.en.din.de.

TABLE 1 Characteristics of the Interlaboratory Wear Test Specimens

NOTE—See Note 4 for information.

	Composition (weight%)	Microstructure	Hardness (HV 10)	Roughness[A]	
				R_z(mean) (μm)	R_a(mean) (μm)
Steel ball (100 Cr6) (AISI 52 100)[B] Diameter 10 mm	1.35 to 1.65 Cr ← 0.95 to 1.10 C 0.15 to 0.35 Si 0.25 to 0.45 Mn	martensitic with minor carbides and austenite	838 ± 21	0.100	0.010
Steel disc (100 Cr6) (AISI 52 100)[C] Diameter 40 mm	← <0.030 P <0.030 S	martensitic with minor carbides and austenite	852 ± 14	0.952	0.113
Alumina ball, diameter = 10 mm[D]	← 95 % Al_2O_3 (with additives of TiO_2,	equi-granular alpha alumina with very minor secondary phases	1610 ± 101 (HV 0.2)	1.369	0.123
Alumina disc, diameter = 40.6 mm[D]	← MgO, and ZnO)		1599 ± 144 (HV 0.2)	0.968	0.041

[A] Measured by stylus profilometry. R_z is maximum peak-to-valley roughness. R_a is arithmetic average roughness.
[B] Standard ball-bearing balls (SKF).
[C] Standard spacers for thrust bearings (INA).
[D] Manufactured by Compagnie Industrielle des Ceramiques Electroniques, France.

TABLE 2 Results of the Interlaboratory Tests[A]

NOTE 1— See Note 4.
NOTE 2—Numbers in parentheses refer to all data received in the tests. In accordance with Practice E178, outlier data values were identified in some cases and discarded, resulting in the numbers without parentheses. The differences are seen to be small.
NOTE 3—Values preceded by ± are one standard deviation.
NOTE 4—Data were provided by 28 laboratories.
NOTE 5—Calculated quantities (for example, wear volume) are given as mean values only.
NOTE 6—Values labeled "NM" were found to be smaller than the reproducible limit of measurement.
NOTE 7—A similar compilation of test data is given in DIN 50324.

Results (ball) (disk)	Specimen Pairs			
	Steel-steel	Alumina-steel	Steel-alumina	Alumina-alumina
Ball wear scar diameter (mm)	2.11 ± 0.27 (2.11 ± 0.27)	NM	2.08 ± 0.35 (2.03 ± 0.41)	0.3± 0.06 (0.3 ± 0.06)
Ball wear volume (10^{-3} mm^3)	198 (198)	...	186 (169)	0.08 (0.08)
Number of values	102 (102)	...	60 (64)	56 (59)
Disk wear scar width (mm)	NM	0.64 ± 0.12 (0.64 ± 0.12)	NM	NM
Disk wear volume (10^{-3} mm^3)	...	480 (480)	...	...
Number of values	...	60 (60)	...	...
Friction coefficient	0.60 ± 0.11	0.76 ± 0.14	0.60 ± 0.12	0.41 ± 0.08
Number of values	109	75	64	76

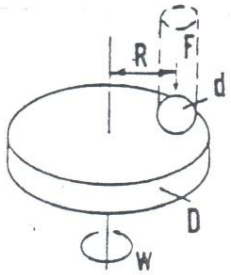
[A] Test conditions: F = 10 N; v = 0.1 ms^{-1}, T = 23°C; relative humidity range 12 to 78 %; laboratory air; sliding distance 1000 m; wear track (nominal) diameter = 32 mm; materials: steel = AISI 52 100; and alumina = α-Al_2O_3.

over other portions. Causes for such differing relationships include initial "break-in" processes, transitions between regions of different dominant wear mechanisms, and so forth. The extent of such non-linear periods depends on the details of the test system, materials, and test conditions.

3.6 It is not recommended that continuous wear depth data obtained from position-sensing gages be used because of the complicated effects of wear debris and transfer films present in the contact gap, and interferences from thermal expansion or contraction.

4. Significance and Use

4.1 The amount of wear in any system will, in general, depend upon the number of system factors such as the applied load, machine characteristics, sliding speed, sliding distance, the environment, and the material properties. The value of any wear test method lies in predicting the relative ranking of material combinations. Since the pin-on-disk test method does not attempt to duplicate all the conditions that may be experienced in service (for example; lubrication, load, pressure, contact geometry, removal of wear debris, and presence



NOTE—F is the normal force on the pin, d is the pin or ball diameter, D is the disk diameter, R is the wear track radius, and w is the rotation velocity of the disk.

FIG. 1 Schematic of Pin-on-Disk Wear Test System

of corrosive environment), there is no insurance that the test will predict the wear rate of a given material under conditions differing from those in the test.

5. Apparatus

5.1 *General Description*—Fig. 1 shows a schematic drawing of a typical pin-on-disk wear test system.[4] One type of typical system consists of a driven spindle and chuck for holding the revolving disk, a lever-arm device to hold the pin, and attachments to allow the pin specimen to be forced against the revolving disk specimen with a controlled load. Another type of system loads a pin revolving about the disk center against a stationary disk. In any case the wear track on the disk is a circle, involving multiple wear passes on the same track. The system may have a friction force measuring system, for example, a load cell, that allows the coefficient of friction to be determined.

5.2 *Motor Drive*—A variable speed motor, capable of maintaining constant speed (±1 % of rated full load motor speed) under load is required. The motor should be mounted in such a manner that its vibration does not affect the test. Rotating speeds are typically in the range 0.3 to 3 rad/s (60 to 600 r/min).

5.3 *Revolution Counter*—The machine shall be equipped with a revolution counter or its equivalent that will record the number of disk revolutions, and preferably have the ability to shut off the machine after a pre-selected number of revolutions.

5.4 *Pin Specimen Holder and Lever Arm*—In one typical system, the stationary specimen holder is attached to a lever arm that has a pivot. Adding weights, as one option of loading, produces a test force proportional to the mass of the weights applied. Ideally, the pivot of the arm should be located in the plane of the wearing contact to avoid extraneous loading forces due to the sliding friction. The pin holder and arm must be of substantial construction to reduce vibrational motion during the test.

[4] A number of other reported designs for pin-on-disk systems are given in "A Catalog of Friction and Wear Devices," American Society of Lubrication Engineers (1973). Three commercially-built pin-on-disk machines were either involved in the interlaboratory testing for this standard or submitted test data that compared adequately to the interlaboratory test data. Further information on these machines can be found in Research Report RR:G02-1008.

5.5 *Wear Measuring Systems*—Instruments to obtain linear measures of wear should have a sensitivity of 2.5 μm or better. Any balance used to measure the mass loss of the test specimen shall have a sensitivity of 0.1 mg or better; in low wear situations greater sensitivity may be needed.

6. Test Specimens and Sample Preparation

6.1 *Materials*—This test method may be applied to a variety of materials. The only requirement is that specimens having the specified dimensions can be prepared and that they will withstand the stresses imposed during the test without failure or excessive flexure. The materials being tested shall be described by dimensions, surface finish, material type, form, composition, microstructure, processing treatments, and indentation hardness (if appropriate).

6.2 *Test Specimens*—The typical pin specimen is cylindrical or spherical in shape. Typical cylindrical or spherical pin specimen diameters range from 2 to 10 mm. The typical disk specimen diameters range from 30 to 100 mm and have a thickness in the range of 2 to 10 mm. Specimen dimensions used in an interlaboratory test with pin-on-disk systems are given in Table 1.

6.3 *Surface Finish*—A ground surface roughness of 0.8 μm (32 μin.) arithmetic average or less is usually recommended.

NOTE 3—Rough surfaces make wear scar measurement difficult.

6.3.1 Care must be taken in surface preparation to avoid subsurface damage that alters the material significantly. Special surface preparation may be appropriate for some test programs. State the type of surface and surface preparation in the report.

7. Test Parameters

7.1 *Load*—Values of the force in Newtons at the wearing contact.

7.2 *Speed*—The relative sliding speed between the contacting surfaces in metres per second.

7.3 *Distance*—The accumulated sliding distance in meters.

7.4 *Temperature*—The temperature of one or both specimens at locations close to the wearing contact.

7.5 *Atmosphere*—The atmosphere (laboratory air, relative humidity, argon, lubricant, and so forth.) surrounding the wearing contact.

8. Procedure

8.1 Immediately prior to testing, and prior to measuring or weighing, clean and dry the specimens. Take care to remove all dirt and foreign matter from the specimens. Use non-chlorinated, non-film-forming cleaning agents and solvents. Dry materials with open grains to remove all traces of the cleaning fluids that may be entrapped in the material. Steel (ferromagnetic) specimens having residual magnetism should be demagnetized. Report the methods used for cleaning.

8.2 Measure appropriate specimen dimensions to the nearest 2.5 μm or weigh the specimens to the nearest 0.0001 g.

8.3 Insert the disk securely in the holding device so that the disk is fixed perpendicular (±1°) to the axis of the resolution.

8.4 Insert the pin specimen securely in its holder and, if necessary, adjust so that the specimen is perpendicular (±1°) to the disk surface when in contact, in order to maintain the necessary contact conditions.

8.5 Add the proper mass to the system lever or bale to develop the selected force pressing the pin against the disk.

8.6 Start the motor and adjust the speed to the desired value while holding the pin specimen out of contact with the disk. Stop the motor.

8.7 Set the revolution counter (or equivalent) to the desired number of revolutions.

8.8 Begin the test with the specimens in contact under load. The test is stopped when the desired number of revolutions is achieved. Tests should not be interrupted or restarted.

8.9 Remove the specimens and clean off any loose wear debris. Note the existence of features on or near the wear scar such as: protrusions, displaced metal, discoloration, microcracking, or spotting.

8.10 Remeasure the specimen dimensions to the nearest 2.5 μm or reweigh the specimens to the nearest 0.0001 g, as appropriate.

8.11 Repeat the test with additional specimens to obtain sufficient data for statistically significant results.

9. Calculation and Reporting

9.1 The wear measurements should be reported as the volume loss in cubic millimetres for the pin and disk, separately.

9.1.1 Use the following equations for calculating volume losses when the pin has initially a spherical end shape of radius R and the disk is initially flat, under the conditions that only one of the two members wears significantly:

$$\text{pin (spherical end) volume loss, mm}^3 = \frac{\pi\,(\text{wear scar diameter, mm})^4}{64\,(\text{sphere radius, mm})} \quad (1)$$

assuming that there is *no significant disk wear*. This is an approximate geometric relation that is correct to 1 % for (wear scar diameter/sphere radius) <0.3, and is correct to 5 % for (wear scar diameter/sphere radius) <0.7. The exact equation is given in Appendix X1.

$$\text{disk volume loss, mm}^3 = \frac{\pi\,(\text{wear track radius, mm})(\text{track width, mm})^3}{6\,(\text{sphere radius, mm})} \quad (2)$$

assuming that there is *no significant pin wear*. This is an approximate geometric relation that is correct to 1 % for (wear track width/sphere radius) <0.3, and is correct to 5 % for (wear track width/sphere radius) <0.8. The exact equation is given in Appendix X1.

9.1.2 Calculation of wear volumes for pin shapes of other geometries use the appropriate geometric relations, recognizing that assumptions regarding wear of each member may be required to justify the assumed final geometry.

9.1.3 Wear scar measurements should be done at least at two representative locations on the pin surfaces and disk surfaces, and the final results averaged.

9.1.4 In situations where both the pin and the disk wear significantly, it will be necessary to measure the wear depth profile on both members. A suitable method uses stylus profiling. Profiling is the only approach to determine the exact final shape of the wear surfaces and thereby to calculate the volume of material lost due to wear. In the case of disk wear, the average wear track profile can be integrated to obtain the track cross-section area, and multiplied by the average track length to obtain disk wear volume. In the case of pin wear, the wear scar profile can be measured in two orthogonal directions, the profile results averaged, and used in a figure-of-revolution calculated for pin wear volume.

9.1.5 While mass loss results may be used internally in laboratories to compare materials of equivalent densities, this test method reports wear as volume loss so that there is no confusion caused by variations in density. Take care to use and report the best available density value for the materials tested when calculating volume loss from measured mass loss.

9.1.6 Use the following equation for conversion of mass loss to volume loss.

$$\text{volume loss, mm}^3 = \frac{\text{mass loss, g}}{\text{density, g/cm}^3} \times 1000. \quad (3)$$

9.2 If the materials being tested exhibit considerable transfer between specimens without loss from the system, volume loss may not adequately reflect the actual amount or severity of wear. In these cases, this test method for reporting wear should not be used.

9.3 Friction coefficient (defined in Terminology G40) should be reported when available. Describe the conditions associated with the friction measurements, for example, initial, steady-state, and so forth.

9.4 Adequate specification of the materials tested is important. As a minimum, the report should specify material type, form, processing treatments, surface finish, and specimen preparation procedures. If appropriate, indentation hardness should be reported.

10. Precision and Bias [5]

10.1 *Statement of Precision*:

10.1.1 The precision of the measurements obtained with this test method will depend upon the test parameters chosen. The reproducibility of repeated tests on the same material will depend upon material homogeneity, machine and material interaction, and careful adherence to the specified procedure by the machine operator. Normal variations in the wear test procedure will tend to reduce the precision of the test method as compared to the precision of such material property tests as hardness or density.

10.1.2 Table 2 contains wear data obtained from interlaboratory tests. Mean and standard deviation values are given for all measured quantities.

10.1.3 Statistical analysis (using Guide G117) of the steel vs. steel ball wear scar diameter results for 24 laboratories leads to a mean and standard deviation of 2.14 and 0.29 mm,

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G02-1008.

respectively. The 95 % repeatability limit (within-lab) was 0.37 mm, and the 95 % reproducibility limit (between-labs) was 0.81 mm. Statistical analysis of the steel vs. steel ball friction results for 25 laboratories leads to a mean and standard deviation of 0.60 and 0.11, respectively. The 95 % repeatability limit (within-lab) was 0.19, and the 95 % reproducibility limit (between-labs) was 0.32.

10.2 *Statement of Bias*—No bias can be assigned to these results since there are no absolute accepted values for wear.

10.3 *General Considerations*—Participants in the interlaboratory testing that led to the statements of precision and bias given above involved 28 laboratories, 2 different materials (4 material pairs), 1 test condition, and 3 to 5 replicate measurements each (see Note 4). Subsequent to this testing, data were received from another laboratory that utilized a commercial test machine. These data were found consistent with the results in the interlaboratory study.

NOTE 4—The interlaboratory data given in Table 1 and Table 2 resulted through the cooperation of thirty one institutions in seven countries with the help of national representatives within the Versailles Advanced Materials and Standards (VAMAS) working party on wear test methods.[6]

11. Keywords

11.1 ceramic wear; friction; metal wear; non-abrasive; pin-on-disk; wear

[6] Czichos, H., Becker, S., and Lexow, J., *Wear*, Vol 114, 1987, pp. 109–130 and *Wear*, Vol 118, 1987, pp. 379–380.

APPENDIX

(Nonmandatory Information)

X1. EQUATIONS

X1.1 Exact equations for determining wear volume loss are as follows for:

X1.1.1 A spherical ended pin:

$$\text{pin volume loss} = (\pi h/6)[3d^2/4 + h^2] \quad \text{(X1.1)}$$

where:

h = $r - [r^2 - d^2/4]^{1/2}$

d = wear scar diameter, and

r = pin end radius.

Assuming no significant disk wear.

X1.1.2 A disk:

$$\text{disk volume loss} = 2\pi R\,[r^2 \sin^{-1}(d/2r) - (d/4)(4r^2 - d^2)^{1/2}] \quad \text{(X1.2)}$$

where:

R = wear track radius, and

d = wear track width.

Assuming no significant pin wear.

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