Canada

EVALUATION OF DIFFERENT DENTAL MATERIALS
USING A SLUGGING FLUIDIZED BED

by

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ABSTRACT

Because of the large number of commercial dental restorative materials that continue to be introduced, there is an obvious need for testing dental materials on their wear resistance. A simple but reliable special method was developed for *in vitro* evaluation of the wear characteristics of dental restorative materials in a gas-solid fluidized bed under slugging operation regime.

A preliminary test on the wear of the dental materials was carried out in a room temperature fluidized column with a cross-section of 76 mm and a height of 0.91m. A stainless beam was fixed horizontally crossing the axis of the column at 0.39 m above the air distributor. The packed bed height was kept constant at 0.34 m for all tests to ensure comparability. Due to bed expansion, the specimens were covered by the particles when fluidized. The parameters tested were particle size (200 μm to 700 μm), different particles, superficial air velocity (0.25 m/s to 0.992 m/s) and different dental restorative materials.

The additional experiment was carried out in the same column under the base operation condition (superficial air velocity = 0.464 m/s; particle size = 400 μm) with more different types of dental materials. The hardness of each material was tested to find out if the wear of the material can be simply predicted by the hardness value.

Keywords: Dental materials, slugging fluidized bed, wear resistance, particle velocity

CO-AUTHORSHIP

Title: Evaluation of Different Dental Materials Using a Slugging Fluidized Bed
(Chapter 4)

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Title: Mimicking the Wear of Dental Materials in a Novel Fluidized Bed Test Apparatus
(Chapter 5)

Authors: J. Li, S. H. Kofman, A. S. Bassi and J. Zhu

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CHAPTER 1: INTRODUCTION AND OBJECTIVES

1.1 Introduction

The wear of human enamel and of the restorative material is often a critical concern when selecting a restorative material for any given clinical restorative treatment (Ramp *et al.* 1997). It is the desire of the restorative dentist to provide a material that has the function and appearance of the enamel that it replaces. But the goal has been elusive because the materials which function most like enamel do not resemble it esthetically, and materials which resemble enamel do not necessarily function like enamel. In an *in vivo* investigation, Lambrechts *et al.* (1989) reported vertical wear of enamel to be 20 μm and 40 μm per year for the observation enamel in the premolar and molar regions, respectively. It is important that the restorative materials have a similar or greater wear resistance than the tooth enamel.

In tribology the fundamental wear processes are: abrasive wear, adhesive wear, wear due to fatigue, fretting wear, erosive wear and corrosive wear (Pugh 1973). These mechanisms occur in various combinations to cause surface loss (van Noort 1994). The wear mechanisms for dental materials have recently been reviewed (Mair, 1992), and two main mechanisms have been identified for dental composites: abrasion and attrition. When two surfaces rub together, the harder of the two materials may indent, produce grooves in, or cut away material from the other surface. This direct contact wear is known

as two-body abrasion, and occurs in the mouth whenever there is direct tooth-to-tooth contact. in what most dentists would call attrition. Abrasive wear may also occur when there is an abrasive slurry interposed between two surfaces such that the two solid surfaces are not actually in contact. This is called three-body abrasion, and occurs in the mouth during mastication. with food acting as the abrasive agent. Tooth pastes act as abrasive slurries between the toothbrush and the tooth as well (van Noort 1994). The combined action of these two mechanisms is mitigated by the changing morphology of the antagonist as wear progresses. Surface texture and surface hardness have each been investigated as possible determinants of wear rate. However, surface hardness has been shown to be a poor indicator of wear rate (Ramp *et al.* 1997). The complexity of the process makes it fairly difficult to model *in vitro* wear (Condon and Ferracane 1996).

Factors influence the wear of the tooth or the restorative materials include:

1. Those arising from masticatory movement. i.e..

    - Speed of movement of the mandible

    - Rate of chewing

    - Forces developed in mastication (Bates *et al.* 1975).

2. Different dietary in-taking.

3. Others: such as medical sources and chewing habits.

The wear rate of the enamel and dental materials can be tested in two ways: *in vivo* and *in vitro*. *In vivo* is the clinical method which operates in the oral environment while *in vitro* is the laboratory method. The main concern for the longevity of posterior

composite restorations continues to be the resistance to intraoral wear (Bayne *et al.* 1994). However, *in vivo* investigations are time-consuming, as a period of at least 6 to 12 months is usually required to produce a measurable amount of wear. In addition, the complexity of the oral environment make it seems to be impossible to establish a standard *in vivo* measurement.

Since it has been nearly impossible to make controlled measurements of gross abrasion in the mouths of humans, numerous *in vitro* tests have attempted to overcome this problem by accelerating the wear rate in a simulated oral environment (Ratledge *et al.* 1994), and to try to predict a material's clinical performance (Wassell *et al.* 1994).

The *in vitro* tests differ widely in the time and effort required for making the measurements, in the cost of equipment and labor, and in their ability to generate quantitative wear values. Two of the most common test methods *in vitro* include the toothbrush/dentifrice abrasion test and the pin and plate method. The former is a three-body wear test that uses a slurry of abrasive particles. The pin and plate method is essentially a two-body abrasion test that compares pairs of materials against one another. Test specimens are shaped to form a pin of known size and dimensions. The pin is loaded against another material, which is usually a rotating disk. Wear assessment can be made by measuring the weight loss of the specimens, the pin height, or the dimensions of the wear scar (Ratledge *et al.* 1994).

In the literature, numerous results from *in vitro* abrasion tests have been

published. The methods of abrading the sample of materials and of quantifying the wear are varied. Unfortunately, this has resulted in an appreciable scatter in the experimental results which at best are characterized as inconclusive. The data are often in qualitative disagreement with clinical studies (Lambrechts *et al.* 1984). The time for a set of tests is usually no more than 24 hours which seems too short compared with the natural wear process.

In general, the unreliable nature of much of the data obtained can generally be attributed to the four factors listed below:

1. The test conditions did not adequately simulate the *in vivo* wear environment.

2. Wear was not accurately measured.

3. The tests were too short to establish the long-term wear properties of the material.

4. Conclusions were based on the results of tests with a single specimen of each material (McKellop *et al.* 1978).

So, a reasonably convenient, economical and reliable procedure with a minimum of special apparatus is the goal for the *in vitro* study.

A novel test method which uses the gas-solid fluidized bed is proposed in this study. In a fluidized bed, fine particulate materials of several micrometers to several millimeters are suspended by an up-flowing gas. The gas-solids suspension thus formed behaves like a fluid (thus the name fluidized bed). Solids particles move freely in the fluidized bed due to the movement of gas bubbles or slugs. Gas flow and the movement

of solids have been studied for many years, so that the flow mechanism is well characterized (Davidson *et al.* 1985). Models based on the behavior of bubbles and slugs in beds of fine particles have been developed to predict the solids flow behavior inside the beds. Therefore, choosing a fluidized bed for the wear test provides a well controlled environment.

When the fluidized bed is operated under low gas velocity, wear caused by the solid particles to objects in the bed is the combination of different wear mechanisms, including abrasion, erosion, attrition, etc. similar to those encountered in human mouth. So, the principal mechanisms in dental wear can be very well simulated in the fluidized bed. The time for undertaking one set of wear tests is about a week, which is between the conventional *in vitro* test and the *in vivo* test. This provides a more reasonable duration for mimicking the long process of dental wear.

1.2 Objectives

The main purposes for this study were:

1. To develop a simple but reliable *in vitro* wear test apparatus/method that would simulate the main clinical wear mechanisms using a fluidized bed operated under slugging flow regime.

2. To evaluate the wear rates of several different composites and amalgam, and to obtain a relative wear rate comparable with enamel, so as to provide useful information to the dentist in selecting materials for the occlusal surfaces of dental

restorations.

3.   To study how the abrasive particle properties and the fluidizing velocity influence

the specimen wear rate and to provide some scientific insight, where possible, into

the observed wear phenomena.

The experiments are carried out in a gas-solid fluidized bed. The major parameters

varied are particle properties, dental material properties and gas superficial velocity.

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CHAPTER 2: THE TOOTH AND THE DENTAL MATERIALS

2.1 The Tooth

Each tooth has a crown and root portion. The crown is covered with enamel, and the root portion is covered with cementum. The crown and root joint at the cementoenamel junction. This junction, also called the cervical line, is plainly visible on a specimen tooth. The main bulk of the tooth is composed of dentine, which is clearly shown in a cross section of the tooth. This cross section displays a pulp chamber and pulp canals, which normally contain the pulp tissue. The pulp chamber is mainly in the crown portion, and the pulp canal is in the root. The spaces are continuous with each other and are spoken of collectively as the pulp cavity. The anatomical relations of the dental tissues are shown diagrammatically in Fig. 2. 1.
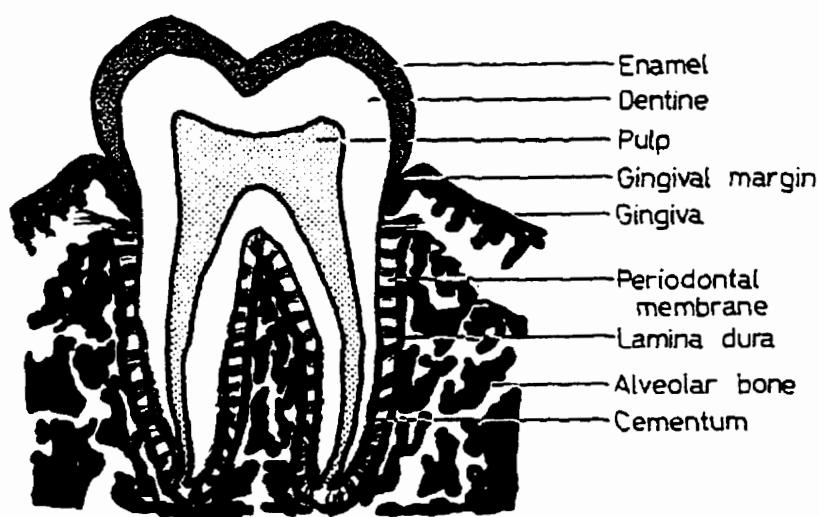
Figure 2.1 Structure of the tooth (Jenkins 1978)

The four tooth tissues are enamel, cementum, dentine, and pulp. The first three are known as hard tissue, the last as soft tissue. The cementum and dentine have a similar composition and the proportion of cementum present in the normal tooth is relatively small. But the composition of enamel and dentine are very different. Table 2.1 shows the different compositions of the dental hard tissues. The enamel is the hardest tissue in the human body: it has the highest mineral content and least organic composition. The dentine is next to the enamel in hardness and mineral content, and then, the cementum.

Table 2.1 Properties of the tooth components (von der Fehr 1967, Weidmann *et al.* 1967, Jenkins 1978).

	Vickers Hardness (Kg/mm^2)	Density (g/ml)	Inorganic (%wt)	Organic (%wt)
Enamel	360-400	2.84-3.01	96%	<1%
Dentine	68	2.14	70%	19-21%
Cementum	Less than Dentine	2.03	45-50%	50-55%

2.2 Dental Materials

The materials available for restoring teeth include (Smith *et al.* 1994):

1. Amalgam

2. Composite

3. Glass-ionomer cement

4. Other cement materials

5. Cast gold or other metal

Most of these materials simply provide a plug to fill the hole in the tooth. It is hoped that the development of restorative materials will lead to restorations which not only replace the lost tissue but also duplicate the properties of the lost dentine and enamel as well as being cariostatic.

2.2.1 Dental amalgam

Amalgam is the most widely used dental restorative material. It is an alloy produced by mixing liquid mercury with solid particles of silver, tin, copper, and sometimes zinc, palladium, indium, and selenium. The reaction between mercury and alloy which follows mixing is termed as amalgamation reaction. It results in the formation of a hard restorative material of silvery-grey appearance (McCabe and Walls 1998). The microstructure of the set material consists of the unreacted centers of the alloy particles bonded together by a matrix composed of the intermetallic compounds (γ1 phase and γ2 phase) formed in the reaction. The setting mechanism can be summarized as:

$$Ag_3Sn + Hg = Ag_3Sn + Ag_2Hg_3 + Sn_{7-8}Hg$$
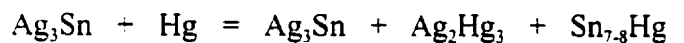
(γ phase)   (γ phase)   (γ1 phase)   (γ2 phase)

Amalgam can be easily placed and is relatively inexpensive. It has very strong mechanical properties compared with tooth substances (See Table 2.2).

Table 2.2 Mechanical properties of a lathe-cut amalgam compared with tooth substance (McCabe and Walls 1998).

Property	Enamel	Dentine	Amalgam
Modulus of elasticity (GPa)	50	12	30
Compressive strength at 7 days (MPa)	250	280	350
Tensile strength at 7 days (Mpa)	35	40-260	60
Vickers hardness (Kg/mm^2)	350	60	100

It is not uncommon to observe amalgam restorations still functioning after 30-40 years of service in the mouth. However, this material has several deficiencies. Amalgam dose not adhere to enamel or dentine and there is no artificial bonding mechanism available. This means that the preparation of the cavity has to include mechanically retentive features, and this may involve cutting away more sound tooth tissue (including overhanging enamel and weakened cusps) than is desirable. The main types of amalgam material failure are marginal breakdown and fracture. Additional deficiencies of dental amalgam restorations are: unnatural appearance, tarnish and corrosion, mercury toxicity, metallic taste and galvanic shock, marginal leakage, discoloration of tooth structure, and high rate of secondary caries (Willianm et al. 1985).

Mercury is one of the main concerns to limit the use of the material. The main source of exposure has been identified as mercury vapor, with dental personnel having a higher risk than patients. Mercury levels in the blood and urine can be affected by the amalgam restoration, and mercury can pass from amalgam fillings to the body organ, and from mother to fetus. However, the mercury level for patients is considered well within safe limits, there does not appear to be any evidence linking amalgam fillings with

neurological function, kidney dysfunction, reduced immunocompetence, effects on oral and intestinal bacteria, birth defects and general health. The present evidence does not appear to demonstrate that amalgam restorations are hazardous to the health of the general population. The only adverse effect documented is the rare occurrence of hypersensitivity to mercury in a very tiny proportion of the population.

2.2.2 Composite restorative materials

A composite is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally. In addition, the constituent phases must be chemically dissimilar and separated by a distinct interface (Callister, Jr. 2000). Composite restorative materials used in dentistry are most resin based composite, called composite resin. Composite restorative materials are used for esthetic restoration of carious lesions, enamel defects and traumatized anterior teeth. The constituents of composite resins are shown in Table 2.3.

Table 2.3 Constituents of composite resins (Braden *et al.* 1997)

Resin matrix phase	(i) Bis-GMA + diluents, or (ii) urethane dimethacrylate + diluents
Filler phase	(iii) macro e.g. quartz, glasses, or (iv) micro e.g. fumed (pyrolytic) silica
Coupling agent	Silane
Curing system	(i) chemical e.g. benzoyl peroxide + amine, or (ii) photo e.g. visible or UV
Other additives	inhibitors, UV stabilisers, optical brighteners, pigments

Composite materials generally consist of four general components, the most obvious being the polymer matrix and reinforcing particle filler phase. The third component is the chemical bonding between the polymer and particle phase, and the fourth component consists of the active chemical additives which serve as initiators of the polymerization process and which provide chemical stability to the materials.

The classification of dental composites is based on filler type, consisting of three fundamental systems from which all other varieties could be formed. These types were traditional (conventional, large) macrofillers, microfillers (microfine) and microfiller complexes (hybrid), see Figure 2.2.

| CONVENTIONAL | MICROFILLED | HYBRID |
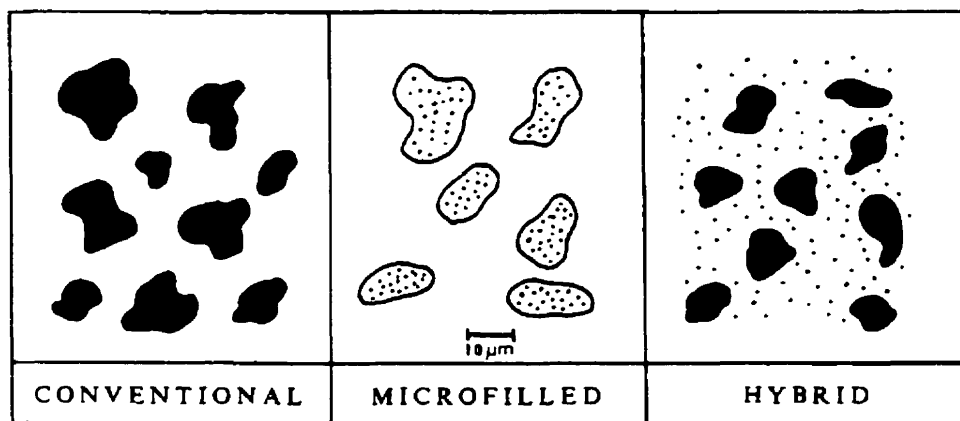
Figure 2.2 Type of the composite (McCabe and Walls 1998).

The conventional macrofilled composites typically contain 60-80%, by weight, of quartz or glass in the particle size range of 1-50 μm. They provide for satisfactory optical appearance, reasonable abrasion resistance and physical properties and, possibly,

radiopacity; but they lack polishability and have surface roughness leading to plaque accumulation and staining. The microfilled composites contain silica particles in the range 0.01-0.1 µm with a typical mean diameter of 0.04 µm. The microfine composites polish well, but tend to absorb water and sometimes discolor. They have lower abrasion resistance and are not radio-opaque. Hybrid materials combine the advantages of both conventional and microfine composites. In recent years there has been an emphasis on the development of hybrid materials with graded particle sizes, and also particles with rounded shapes, which improve particle packing within a limited amount of resin material. The mechanical properties of composite resins are shown in Table 2.4.

There is a growing tendency to consider composite resin for use as alternatives to amalgam in posterior cavities. However, the pulpal irritation, marginal staining, non-radiopaque, and the limited clinical durability have kept the materials from reaching their full potential. Techniques for using these materials more effectively are still being developed.

Table 2.4 Mechanical properties of composite resins (McCabe and Walls 1998).

	Typical conventional composite	Typical microfilled composite	Typical hybrid composite
Compressive strength (MPa)	260	260	300
Yield stress (Mpa)	260	160	300
Tensile strength (Mpa)	45	40	50
Flexural strength (MPa)	110	80	150
Modulus of elasticity (GPa)	12	6	14
Vickers Hardness (Kg/mm^2)	60	30	90

2.2.3 Glass-ionomer Cements

Glass-ionomer cement restorative materials may be supplied as a powder and liquid or as a powder mixed with water. The composition is outlined in Table 2.5.

Table 2.5 Composition of glass ionomer cements (McCabe and Walls 1998).

1. Powder/liquid materials	
Powder	sodium aluminosilicate glass with about 20% CaF and other minor additives
Liquid	aqueous solution of acrylic acid/itaconic or aqueous solution of maleic acid polymer and tartaric acid in some products to control setting characteristics
2. Powder/water materials	
Powder	Glass (as above) + vacuum-dried polyacid (acrylic, maleic or copolymers)
Liquid	manufacturers supply a dropper bottle which the operator fills with water or the manufacturer supplies a dilute aqueous solution of tartaric acid

The glass ionomer cement has the advantage of adhering to dentine and enamel and of leaching fluoride, which is effective in reducing recurrent caries. The anhydrous version, provided as a powder mixed with water, has a low solubility compared with the earlier versions of the material and a better appearance. It is useful in restoring Class V cervical lesions and has been tried in posterior teeth as a base which is then veneered with a more abrasion-resistant composite or metal restoration. However, cement is not tooth colored, and the surface of glass-ionomer restorations should be protected with a varnish to overcome its initial solubility.

The most popular used dental materials were compared in the Table 2.6.

Table 2.6 Comparison of the main dental materials (McCabe and Walls 1998)

	Advantage	*Disadvantage*
Amalgam	1. Strong mechanical properties 2. Relatively easy to use 3. Relatively inexpensive 4. Radio-opaque	1. Lack of adhesion 2. Poor aesthetics 3. High thermal conductivity 4. Mercury toxicity 5. Galvanic effects 6. Discoloration of tooth structure
Composite	1. Tooth-colored materials 2. Reasonable strength 3. Available bonding agent	1. Loss of substance through wear 2. Color changes 3. Pulpal irritation 4. Marginal staining 5. Recurrent caries 6. Shrinkage
Glass-ionomer	1. Adhering to the tooth tissue 2. Fluoride release 3. Aesthetics	1. None-radio-opaque 2. Water solubility 3. Poor abrasion resistant

2.2.4 Other cement materials

More recently, glass-ionomer/composite resin hybrid materials have been introduced into the market. These materials set partly via an acid-base reaction and partly via a photochemical polymerization. Materials that may contain either or both of the essential components of a glass-ionomer cement but insufficient to promote the acid-base cure reaction in the dark should be referred to as polyacid-modified composite resins or

compomer (McLean 1994). These type of materials are newly developed and there are no sufficient reports about their qualities.

2.3 Wear in Dentistry

Wear is in fact somewhat difficult to define. It is generally accepted as a "progressive loss of substance from the surface of a body brought about by mechanical action" (Jones et al. 1978).

Wear is a natural process that occurs whenever two or more surfaces move in contact with one another (Zum-Gahr 1987). It is not a single process, but the overall result of at least five underlying processes which seldom act in isolation (Pugh 1973). As the teeth, together with any restorations, move in contact with one another wear is inevitable. Its progress will depend upon the structure of the surfaces, the contact stress, the activity of any lubricating layer, the temperature, and the duration of contact (Sarkar 1980a). It is important that the restorative materials have a similar or greater wear resistance than the teeth. Therefore it can be seen that tooth wear and restoration wear need to be studied in relation to one another. The terms abrasion, attrition and erosion have slightly different meanings when used by dental material scientists to describe restoration wear.

In tribology the fundamental wear processes are: abrasive wear, adhesive wear, wear due to fatigue, fretting wear, erosive wear and corrosive wear (Pugh 1973). These occur in various combinations to cause surface loss.

2.3.1 Fundamental wear mechanism

Abrasive Wear describes the cutting away of a surface by abrasive asperities or particles. This kind of wear may occurs in two types: two-body abrasion occurs when the cutting asperities are fixed to one or both surfaces (e.g. sandpaper) and three-body abrasion occurs when hard abrasive grains are present between two sliding surfaces (e.g. polishing paste). With two-body abrasion the shape of the harder or sharper surface is imposed into the softer surface. With three-body abrasion the slurry may 'hollow out' the softer areas in a heterogeneous surface.

Adhesive Wear results from friction between the moving surfaces which causes local cold welding between asperities. Further movement of the surfaces fracture these welds, but the line of separation is not necessarily coincident with the original weld (Zum-Gahr 1987). The overall result is the transfer of material from one surface to another. As a result of this transfer, plates of material may build up on one surface which may subsequently break away and contribute to the three-body abrasive slurry.

Fatigue Wear occurs as a result of the formation and propagation of subsurface microcracks when two surfaces move under dynamic load. Strictly speaking the term

fatigue should be reserved for situations where there is rolling rather than sliding of the surfaces as in gears and bearings (Pugh 1973, Sarkar 1980b). However, the related process of delimitation has been described by Suh (1973 1977). According to this theory, when two moving surfaces are in contact, stress is applied to the asperities of the softer surface. This deforms the asperities in the direction of movement resulting in the accumulation of plastic deformation energy in the subsurface. Dissipation of this energy nucleates cracks which eventually spread laterally to the surface. Eventually a small area of the surface material becomes surrounded by a network of linked cracks and the fragment is subsequently displaced. In filled materials the subsurface cracks may propagate either through the filler particles or around the interface (Jahanmir and Suh 1977). There is an interaction between fatigue and adhesive wear because fatigue may weaken the subsurface allowing adhesive forces to pluck out the surface fragments.

Erosive Wear results from the impact of particles or fluid under pressure (Pugh 1973, Tilly 1979). Sand-blasting and the wear of rocks under waterfalls are examples. The essential feature of erosion is that the wear medium (e.g. sand and water) forms the second surface. This can therefore be distinguished from three-body abrasion where the particles are compressed between two separate surfaces.

Corrosive Wear results form the interaction of chemical degradation and movement of the surfaces. The surface is weakened by chemical degradation and then removed by rubbing against an opposing surface. Some modern classifications use the

term 'tribochemical wear' to distinguish this form of surface loss from static corrosion which occur in the absence of movement (Zum-Gahr 1987).

Fretting Wear occurs as a result of prolonged slow slipping between surfaces under load. These conditions do not occur in the mouth therefore this process does not feature in dental wear.

2.4 Wear Tests in Dentistry

The wear rate of an ideal restorative material should approximate that of enamel. In an *in vivo* investigation Lambrechts *et al.* (1989) reported vertical wear of enamel to be 20 μm and 40 μm per year when opposing enamel in the premolar and molar regions, respectively. Surface texture and surface hardness have each been investigated as possible determinants of wear rate. However, surface hardness has been shown to be a poor indicator of wear rate (Ramp 1997).

2.4.1 Types of tests

The wear rate of the dental materials can be tested in two ways: *in vivo* and *in vitro*. *In vivo* is the clinical method while *in vitro* is the laboratory method. *In vitro* wear tests can be further divided into two main directories: two-body wear test and three-body wear test.

1. *In vivo*:

Wear of restorations. quantified as vertical loss of substance (μm). is material-specific and most obvious in the occlusal contact point areas (OCA) (Lutz *et al.* 1984. Braem *et al.* 1986. Roulet 1987). OCA-wear curves of restorative materials are characterized by a steep initial ascent followed by a gradual leveling off with increasing time (Lambrechts 1983. Roulet 1987. Krejci *et al.* 1990. Krejci and Lutz 1990). However. *in vivo* wear tests which cover OCA and enamel wear of the antagonistic cusps are technically complex. expensive and time-consuming. Furthermore. the standard deviations of *in vivo* wear data are always large (Lambrechts 1983. Lutz *et al.* 1984). The clinical findings indicate that the chewing pressure defined by the applied chewing force and the size of the occluding contact area is an important factor influencing the wear of both composite and antagonistic enamel (Bailey *et al.* 1981. Bailey and Rice 1981. Chapman and Nathanson 1983. Harrison and Moores 1985. Sarrett *et al.* 1991). However. the chewing pressure cannot be standardized *in vivo*. The reason for that can come from:

(1). Different masticatory function

- Speed of movement of the mandible: The speed of the masticatory movement is the speed developed by the mandible as it approaches or moves away from the maxilla.

- Rate of chewing: The rate of chewing is usually expressed as the time taken per chew or as the number of chews carried out in a known period of time --- usually 1 min.

- Forces developed in mastication: The forces developed between the teeth are those produced in the normal chewing of foods and those that can be applied when a maximum biting load is applied without food being present (Bates et al. 1975).

(2). Different dietary in-taking

(3). Others such as different chewing habits

Therefore, the discriminative power of *in vivo* wear test is limited. With respect to the research economy, newly developed composites have to be optimized in regard to wear prior to large-scale clinical testing.

2. *In vitro*:

a) Two-body wear tests

Test specimens are shaped to form a pin of known size and dimensions. The pin is loaded against another material, which is usually a rotating disk. Wear assessment can then be made by measuring the weight loss of the specimens, the pin height, and the dimensions of the wear scar (Ratledge et al. 1994).

b) Three-body wear tests

Three-body wear tests, best represented by the 'Amsterdam' wear machine, are valuable instruments for wear evaluation. The main difference between two-body and three-body test is that there is a food mimic slurry between the pin and the rotating disk acting like the third body. However, considerable shortcomings cannot be denied:

- Wear mechanisms characteristic of the OCA cannot be simulated

- Wear rates strongly depend on the abrasion medium and on the slip

- The computation of material-specific wear factors for correlation with *in vivo* results is questionable (DeGee *et al.* 1986, Pallav *et al.* 1988)

2.4.2 Limitations of the traditional wear tests

The unreliable nature of much of the data obtained can generally be attributed to four factors:

(a) The test conditions did not adequately simulate the *in vivo* wear environment.

(b) Wear was not accurately measured.

(c) The tests were too short to establish the long-term wear properties of the material.

(d) Conclusions were based on the results of tests with a single specimen of each material (Mckellop et al. 1978).

As discussed in Chapter one. The test conditions can not completely simulate the *in vivo* wear. due to the complexity of the oral environment. such as the speed of the mandible movement. the rate of chewing, and the forces developed in chewing etc. The *in vitro* tests differ widely in the time and effort required for making the measurements. in the cost of equipment and labor, and in their ability to generate quantitative wear values. The wear rate is too small for the material or the tooth tissue (normally the units for the measurement are in the micro level), so that it is hard to measure the wear precisely. Most

of the *in vitro* tests are undertaken under a heavy load condition to achieve a short test period (usually less than 24 hours for a single test). This kind of result seems too short to be convincible, compared with the natural process which takes a year to reach 20-40 μm vertical loss for enamel.

2.4.3. The proposed fluidized bed *in vitro* tester

Considering the limitations of the conventional wear testers, a new testing method which combines the fluidization technology and the dental wear mechanism is proposed as the solution. This new test can mimic the principal wear mechanism by the fluidized particles, and its test period is relatively longer (about a week). The principle for this method is to put the material samples in a gas-solid fluidized column, where solids are fluidized like a liquid and travel with the blowing air to create wear to the samples inside the column (see Chapter 3 for details). The wear mechanisms in this case are the combinations of abrasion, erosion, adhesive wear and fatigue. The measuring technique for testing the wear rate was weighing technique using a digital balance with accuracy of 0.05 mg. The specimens were mounted on a supporting bar and eroded by the solid particles. The specimens were carefully weighed before and after the wear test. The weight loss was the difference between the two measurements. To minimize disturbances, the balance was located on a special heavily loaded table to reduce vibration, and was enclosed in a box to prevent air currents. A special stainless steel screw were always kept aside and used as standard weights to calibrate the balance every time it was used. These

special efforts allowed the balance to reach its maximum available accuracy and helped give good reproducibility. The whole testing time for a single run is about four days to a week. This interval of time is quite reasonable for testing slow progressive wear with very tiny amounts of material loss, which is very close to the natural process of dental wear. Furthermore. since the existing testing systems in the dental field are mainly limited in the pin and plate test with or without a slurry. The proposed test here represents a new attempt at material testing methods and break the traditional dental material testing concepts.
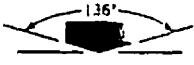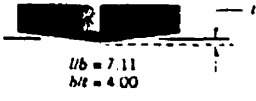
2.5. Hardness Tests

In engineering, hardness is most commonly defined as the resistance of a material to *indentation*. Indentation is the pressing of a hard round ball or point against the material sample with a known force. so that a depression is made. The depression. or indentation. results from plastic deformation beneath the indenter. Some specific characteristic of the indentation. such as its size or depth. is then taken as a measure of hardness. The softer the material. the larger and deeper the indentation. and the lower the hardness index number. Measured hardnesses are only relative (rather than absolute), and care should be exercised when comparing values determined by different techniques.

Hardness tests are performed more frequently than any other mechanical test for several reasons:

1. They are simple and inexpensive–ordinarily no special specimen needs to be prepared. and the testing apparatus is relatively inexpensive.

2. The test is nondestructive–the specimen is neither fractured nor excessively deformed: a small indentation is the only deformation.

3. Other mechanical properties often may be estimated from hardness data.

Table 2.7 Hardness testing technique (Callister. Jr. 2000).

Test	Indenter	Shape of Indentation		Load	Formula for Hardness Number*
		Side View	Top View		
Brinell	10-mm sphere of steel or tungsten carbide			P	$HB = \dfrac{2P}{\pi D[D - \sqrt{D^2 - d^2}]}$
Vickers microhardness	Diamond pyramid	136°	d_1 d_1	P	$HV = 1.854 P/d_1^2$
Knoop microhardness	Diamond pyramid	$l/b = 7.11$ $b/t = 4.00$	b l	P	$HK = 14.2 P/l^2$
Rockwell and Superficial Rockwell	Diamond cone $\tfrac{1}{16}, \tfrac{1}{8}, \tfrac{1}{4}, \tfrac{1}{2}$ in. diameter steel spheres	120°		60 kg 100 kg 150 kg } Rockwell 15 kg 30 kg 45 kg } Superficial Rockwell	

* For the hardness formulas given, P (the applied load) is in kg, while D, d, d_1, and l are all in mm.

Source: Adapted from H. W. Hayden, W. G. Moffatt, and J. Wulff. *The Structure and Properties of Materials*. Vol. III. *Mechanical Behavior*. Copyright © 1965 by John Wiley & Sons, New York. Reprinted by permission of John Wiley & Sons, Inc.

There are many hardness-testing techniques that are frequently employed (see Table 2.7). Vickers test is commonly used for engineering purposes. A very small diamond indenter having pyramidal geometry is forced into the surface of the specimen. Applied loads are much smaller than for Rockwell and Brinell. ranging between 1 and 1000g. The resulting impression is observed under a microscope and measured: this

measurement is then converted into a hardness number (Figure 2.3). Careful specimen

surface preparation (grinding and polishing) may be necessary to ensure a well-defined

indentation that may be accurately measured. It is well suited for measuring the hardness

of small, selected specimen regions (Callister, Jr. 2000).
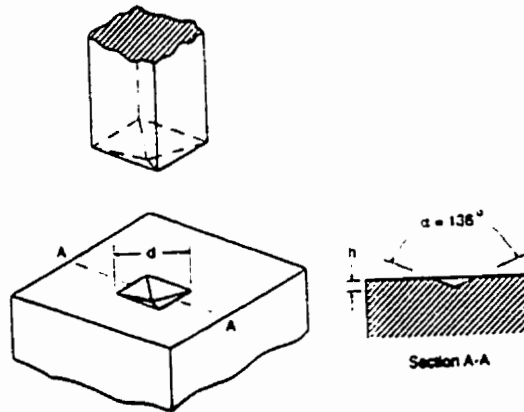
Figure 2.3 Vickers hardness indentation.

At one time. it was thought that the hardness would provide a good indicator of

the wear resistance of a composite. and this is true up to a point. The original acrylic

resins were very soft materials. but their hardness and wear resistance were much

improved by the addition of a filler. Measurement of the hardness initially gave some

indication of the wear resistance, but this relationship unfortunately breaks down at the

high filler loadings used in the current generation of composites (van Noort 1994). Tooth

material hardness may not be a major factor influencing wear rate. This conclusion

confirmed the finding of Ramp *et al.* (1997) that surface hardness has been shown to be a

poor indicator of wear rate.

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CHAPTER 3: HYDRODYNAMIC BEHAVIOR IN CONVENTIONAL FLUIDIZED BEDS

Conventional gas fluidized beds operate mainly in the bubbling or slug flow regime. With an increase in gas flow rate beyond that required for minimum fluidization (minimum bubbling for group A particles), gas voids form at or near the gas distributor and grow in size. mostly by coalescence, as they rise. These gas voids are often called bubbles because of the analogies between them and large bubbles in real liquids. If the bubbles grow large enough compare to the column cross-section. they become slugs. It is the bubbles or slugs that are responsible for most of the features that differentiate a packed or moving bed from a fluidized bed. They also determine the particle movement which is the key element for the wear phenomena in this experiment.

The following sections consider first the hydrodynamics behavior of the conventional bubbling fluidized bed and the hydrodynamics behavior of the slugging fluidized bed, and the formulae for calculating the important parameters in each operating system (the main operating regime used in this experiment was slugging flow). Then the particle impact wear and the wear in the fluidized bed are reviewed individually.

3.1 Freely Bubbling Beds

Bubbles rising in fluidized beds are commonly represented as spherically-capped voids with concave indentations at their bases (Davidson and Harrison 1963), although

the true shape is often closer to an ellipsoidal cap (Clift *et al.* 1978). In a bubbling

fluidized bed there are regions of very low solids density defined as bubble phase and

regions of higher solid density called emulsion or particulate phase. As a first

approximation, all gas in excess of that needed to just fluidize the bed passes through the

bed as bubbles, while the emulsion phase remains at minimum fluidizing conditions with

a voidage of ε_{mf} and interstitial velocity u_{mf}/ε_{mf}.

The average bubble size is found to increase rapidly with height and with an

increase in gas flow rate, mainly as a result of coalescence, until a maximum bubble size

has been reached. A number of correlations have been proposed to estimate the mean

bubble size at a certain level, the best known being those of Geldart (1970, 1972), Mori

and Wen (1975), Rowe (1976), Darton *et al.* (1977), Werther (1978), Bar-Cohen *et al.*

(1981) and Horio *et al.* (1987). In all of these correlations, the average bubble diameter is

a function of the gas flow rate and the height above the gas distributor. In some, the effect

of bed scale, characteristics of the gas distributor and the powder properties are also

considered.

The correlation of Darton *et al.* (1977) is a semi-empirical correlation based on

lateral bubble coalescence. The proposed equation is:

$$D_e = 0.54(u - u_{mf})^{0.4}(x + 4A_D^{1/2})^{0.8} / g^{0.2} \tag{3.1}$$

where A_D is the area of distributor plate per orifice and x is the height above the

distributor. The above equation agrees well with most literature data, providing that

neither a maximum stable bubble size nor slugging is achieved. Bar-Cohen *et al.* (1981) modified the constants in the semi-empirical correlation of Darton *et al.* (1977) to offer better agreement with the data of other workers. The modified equation is:

$$D_e = 0.45(u - u_{mf})^{0.4}(x + 4.63A_D^{1/2})^{0.8} / g^{0.2}$$
(3.2)

The correlation of Mori and Wen (1975) includes an estimate for the mean bubble size, $D_{e,0}$, formed at the distributor:

$$D_{e,0} = 1.38g^{-0.2}[A_D(u - u_{mf})]^{0.4} \quad \text{for perforated plates}$$
(3.3)

$$D_{e,0} = 0.376(u - u_{mf})^2 \qquad \text{for porous plates}$$
(3.4)

It also includes an estimate for the maximum bubble size, $D_{e,x}$, attainable by coalescence.

$$D_{e,x} = 1.49\{D^2(u - u_{mf})\}^{0.4}$$
(3.5)

where D is the bed mean diameter. Then the bubble size is estimated by:

$$D_e = D_{e,x} - (D_{e,x} - D_{e,0})\exp(-0.3x / D)$$
(3.6)

The pattern of the solids motion with respect to the rising bubble is analogous to potential flow past a sphere (Reuter 1966). Particles continuously stream downwards around the sides of the rising bubble. Behind the bubble there is a wake region in which particles are carried upward at the bubble velocity (Rowe 1971). The wake fraction. defined as the wake volume per unit bubble volume. is about 0.1-0.4 depending on solid properties. Small and rounded particles give larger wakes than coarse or angular particles.

The absolute rise velocity of bubbles at a particular height in freely bubbling beds. u_b, can be estimated (Davidson and Harrison 1963) from

$$u_b = u_{b,\infty} + u - u_{mf} \qquad (3.7)$$

or

$$u_b = 0.71(gD_e)^{1/2} + u - u_{mf} \qquad (3.8)$$

where $u_{b,\infty}$ is the velocity of the bubble in isolation, estimated from Equation (3.2). A more accurate equation were given by Weimer and Clough (1983):

$$u_b = 0.71[gD_e(1-\varepsilon_b)]^{1/2} + C_o G_b / A \qquad (3.9)$$

where G_b is the visible bubble flow rate. A is the cross-sectional area of the bed and C_o is a coefficient depending on the distribution of bubbles across the bed (Clift and Grace 1985). This equation may be compared with Equation (3.8). The value of u_b is reduced by a factor $(1-\varepsilon_b)^{0.5}$, which is normally close to unity. Since $C_o > 1$ and $G_b / A < (u - u_{mf})$ (Clift and Grace 1985), the value of $C_o G_b / A$ is frequently close to $(u - u_{mf})$. Hence Equation 3.8 often provides a reasonable approximation for u_b.

The above two equations both imply that the mean bubble velocity increases with distance from the distributor and with the gas velocity. Particle movement and solids circulation in gas fluidized beds are caused primarily by the motion and disturbance of gas bubbles passing through the bed. Rising bubbles cause transport of solids by two mechanisms, firstly by carrying solids in their wakes and pushing solids in their caps, and secondly by drawing up the solids in a drift profile behind the bubble. Outside the bubble

paths. solids move downwards in the bed to replace the particles brought to the surface. The non-uniform spatial distribution of the rising bubbles tends to enhance particle movement and to establish solids circulation patterns depending on the bed depth.

The previous discussion applies when the vessel dimensions greatly exceed the bubble size. If the size of the rising bubble approaches the bed diameter. the bubble tends to elongate. the rise velocity is retarded by the containing wall. and the wake is smaller. For the ratio $D_e / D < 0.125$. the bubble rise velocity is considered not affected by the wall (Clift and Grace 1985). For $0.125 \leq D_e / D < 0.6$. the retardation may be estimated (Wallis 1969) by:

$$u_b = 1.13 u_{b,\infty} \exp(-D_e / D) + u - u_{mf} \qquad (3.10)$$

when D_e is about $D/2$ or larger. the slug flow regime has been achieved.

3.2 Slug Flow

There are two types of behaviors which have been termed as "slug flow" (Stewart and Davidson 1967). and they are represented in Figure 3.1.
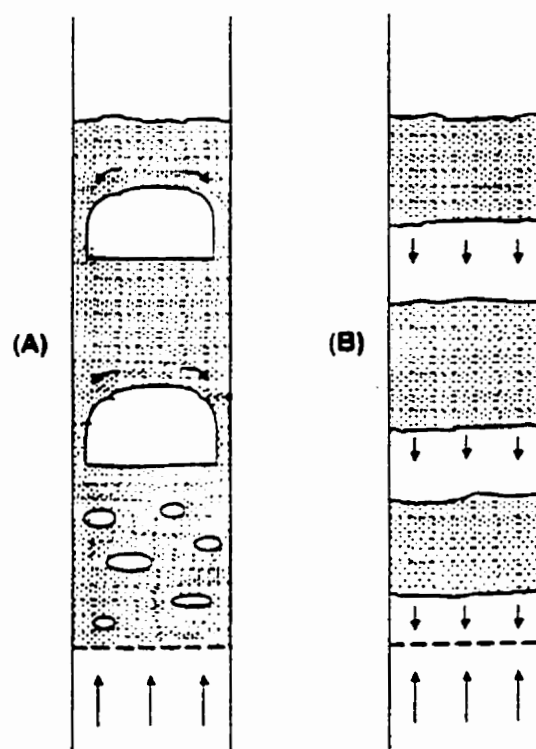
Figure 3.1 Alternative slug flow regimes in a fluidized bed (Stewart and Davidson 1967)

In type A. round-nosed slugs rise through the bed of particles which rain down on either sides or both of the void to allow its upward motion. With slug flow of type B. the fluidized bed also contains successive regions of dense and disperse phase. but the upward movement of the interfaces is slow and is largely caused by particles raining down uniformly through the disperse regions (Stewart and Davidson 1967). This type of slug flow is very common in tubes up to 0.05 m diameter. However, slug flow of type A is of more general interest. Figure 3.2. shows that a laboratory sized fluidized bed will usually operate in the slug flow region when $(u-u_{mf})$ is greater than about 0.10 m/s and $H/D > 1$ (Hovmand and Davidson 1971).
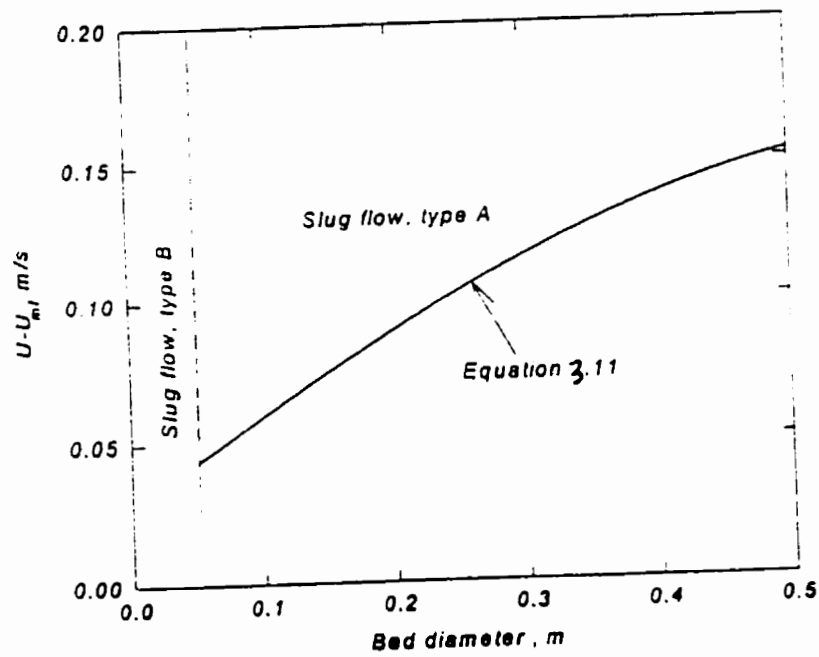
Figure 3.2 Slug flow regime in laboratory fluidized bed ($H/D > 1$), according to criterion of Stewart (1965).

3.2.1 Onset of slugging regime and the rise velocity of a slug

For the bed to achieve the slugging regime, the superficial gas velocity must exceed a minimum slugging velocity (Clift and Grace 1985):

$$u_{ms} = u_{mf} + 0.07(gD)^{1/2} \tag{3.11}$$

and must be below the value at which turbulent or fast fluidization occurs. The bed also must be sufficiently deep for coalescing bubbles to attain the size of slugs. Baeyens and Geldart (1974) concluded that Equation (3.11) is only applicable if $H_{mf} > 1.3D^{0.175}$ (SI units). Otherwise. the minimum slugging condition is given by:

$$u_{ms} = u_{mf} + 0.07(gD)^{1\ 2} + 0.16(1.3D^{0.175} - H_{mf})^2 \tag{3.12}$$

The rise velocity of a single slug is in analogy with the slug in liquid (Hovmand and Davidson 1971):

$$u_{s,\infty} = 0.35(gD)^{1\ 2} \tag{3.13}$$

The rise velocity of a slug in a freely slugging bed is then approximated by:

$$u_s = u_{s,\infty} + u - u_{mf} \tag{3.14}$$

3.2.2 Absolute rising velocity of slug, u_s

A comparison of 48 sets of observed u_s values with the equations introduced by references is shown in Table 3.1.

It can be seen that the Ormiston' s equation agrees better with the observed value. So. the Ormiston' s equation was used for calculations in this project:

$$u_s = (u - u_{mf}) + 0.35(gD)^{1/2} \tag{3.15}$$

Table 3.1 Comparison of equations for absolute rising slug velocity (Shichun *et al.* 1985)

Source	Ormiston et al. (1965)	Birknoff and Carter (1957)	Nakamura (1976)	Shichun et al. (1985)
Equation	$u_s = (u-u_{mf}) + 0.35(gD)^{1/2}$	$u_s = (u-u_{mf}) + 0.35(2gD)^{1/2}$	$u_s = 0.28(gL_s)^{1/2}$	$u_s = 0.7613(u-u_{mf}) + 0.35(1.19gD)^{1/2}$
MRD	0.73%	23.10%	30.52%	9.27%
SRRD	5.64%	8.48%	13.88%	5.76%
MARD	21.06%	-39.4%	56.37%	-21.29%

*MRD, mean-relative deviation.
*SRRD, standard error of relative deviation.
*MARD, Maximum-relative deviation.

3.2.3 Expansion of slugging fluidized bed

From continuity:

$$NV_s u_s = u - u_{mf}$$ (3.16)

where in unit bed volume there are N slugs each of volume V_s whose absolute velocity. u_s is given by Equation (3.15). N and V_s are not necessarily constant throughout the bed. because of coalescence: but from Equation (3.15). u_s is constant and therefore NV_s must be constant and because the coalescence occurs in a regular manner. it is reasonable to assume constant N and V_s for a given height in the bed. Since the increase in bed height from H_{mf} at incipient fluidization to height H is caused by the slug volume. one has:

$$NV_s H = H - H_{mf}$$ (3.17)

and with Equations (3.15) and (3.16) (Hovmand and Davidson 1971):

$$(H - H_{mf}) / H_{mf} = (u - u_{mf}) / 0.35(gD)^{1/2}$$ (3.18)

Therefore. bed expansion can be estimated with given superficial gas velocity.

3.2.4 Length of stable slugs

The slug volume V_s is obtained by the following procedure (Nicklin *et al.* 1962):

$$m = 4V_s / D^3\pi = L_s / D - 0.495(L_s / D)^{1/2} + 0.061 \qquad (3.19)$$

where m is the shape factor for slug volume. Assuming that two successive slugs will not coalesce when the vertical distance between them is more than $2D$ (Hovmand and Davidson 1971), the slug length L_S can be calculated, if this spacing of slugs holds throughout a tall fluidized bed. Since the expansion from height H_{mf} to height H is entirely due to slugs, then

$$(H - H_{mf}) / H = 4V_s / \pi D^2 (L_s + 2D) \qquad (3.20)$$

which, with Equations (3.17) and (3.18) and eliminating V_s and $(H-H_{mf})/H_{mf}$ gives (Hovmand and Davidson 1971):

$$\frac{L_s}{D} - 0.495(\frac{L_s}{D})^{1/2}\left[1 - \frac{U - U_{mf}}{0.35(gD)^{1/2}}\right] + 0.061 - \frac{1.939(U - U_{mf})}{0.35(gD)^{1/2}} = 0 \qquad (3.21)$$

This quadratic in $(L_S/D)^{1/2}$ gives the slug length L_S uniquely in terms of fluidizing velocity.

3.2.5 Slug frequencies

An equation for slug frequency, f_s, and slug length, L_s, can be derived from considerations of bed expansion and a material balance on the slugs and slug wake W_s.

which is assumed to be proportional to the slug diameter, d_s, and therefore to the bed diameter D. Considering the solids and gas present between the noses of consecutive slugs yields (Baeyens and Geldart 1974):

$$\varepsilon_B = \frac{L_s}{W_s + L_s} = \frac{H - H_{mf}}{H} = \frac{u - u_{mf}}{u_s} \tag{3.22}$$

Now the slug frequency

$$f_s = \frac{u_s}{L_s + W_s} \tag{3.23}$$

And, substituting for u_s from Equation (3.22) gives:

$$f_s = \frac{u - u_{mf}}{L_s} \tag{3.24}$$

Now it has been shown that

$$\frac{H - H_{mf}}{H_{mf}} = \frac{u - u_{mf}}{u_{s,\infty}} \tag{3.25}$$

The total height of all the wakes of n slugs $= nW_s = H_{mf}$ and the total height of all the slugs $= nL_s = H - H_{mf}$. Substituting in (3.25) gives

$$\frac{L_s}{W_s} = \frac{u - u_{mf}}{u_{s,\infty}} \tag{3.26}$$

Assuming $W_s = kD$, and $u_{s,\infty} = 0.35(gD)^{1/2}$, the slug length is given by (Matsen et al. 1970):

$$L_s = \frac{(u - u_{mf})kD^{1/2}}{0.35g^{1/2}} \tag{3.27}$$

The slug frequency is given by (Matsen et al. 1970):

$$f_s = \frac{0.35g^{1/2}}{kD^{1/2}} \tag{3.28}$$

This means that the slug frequency is independent of the gas velocity, as is often observed. The constant k, the only unknown parameter in the equation, must be experimentally determined.

And there is also some other correlation in the references. One of the equations which is better agreed with the observed values is (Shichun et al. 1985):

$$f_s = 0.533(u - u_{mf})^{-0.152} \tag{3.29}$$

3.3 Solids Impact Wear

The mechanism of solids impact wear has been studied in detail by a number of researchers (e.g. Finnie 1958 and 1960, Finnie et al. 1967, Finnie 1972, Ives et al. 1976, Finnie and McFadden 1978, Ruff 1979, Finnie 1979, Finnie et al. 1979, Hutchings 1979a and 1979b, Tilly 1979, Ruff & Wiederhorn 1979, Hutchings 1980, Bellman and Levy 1981, Hutchings 1981, Levy 1982, Cousens and Hutchings 1983, Levy 1983, Rao and Buckley 1983, Sundararajan 1983, Sundararajan and Shewmon 1983, Hutchings 1987, Zhu et al. 1990, 1991). This work is commonly associated with wear of aircraft and turbines at very high particle impact velocities (100-300 m/s). Tests are usually conducted by causing particles to impinge individually or continuously on a target plate.

Erosive wear involves the eroding surface, the particles causing the wear, and the fluid flow conditions which bring the particles into contact with the surface. Factors which may influence wear include:

Flow properties	Particle velocity
	Impact angle
	Particle rotation
	Temperature
	Number of particles striking
	Corrosive environment
Particle properties	Size
	Density
	Hardness
	Strength or friability
Material properties	Shape
	Hardness
	Elasticity
	Other mechanical and material properties

In summary, the solid impact wear is mainly related to these three factors: the solid flow properties, the particle properties, and the material properties. Each of these factors plays a very important role in the wear rate and form. The following section will discuss how they give the influence individually.

3.3.1 Flow properties

The velocity of the fluid flow influences the wear rate, though indirectly, since it affects the velocity with which individual particles strike the object. Fundamental studies have examined the dependence of wear rate on the particle impact velocity, while more applied investigations have studied the variation of wear rate with the overall flow velocity in specific systems. Both types of work reveal that velocity has a strong effect on wear rate. Basic studies have shown that erosive wear, measured as the mass removed from the surface per unit mass of impinging particles, varies with the impact velocity raised to some power. This velocity exponent lies typically in the range from 2 to 4, for wear both by gas-borne particles (Ruff and Wiederhorn 1979) and by slurries (de Bree *et al.* 1982, Levy and Yau 1983).

The wear resulting from particle impact depends on the angle at which the particles strike the surface. Metals tend to suffer most severe wear at impact angles of 20° to 30° (measured from the plane of the surface), while ceramic materials often suffer peak wear for normal incidence (see Figure 3.3).
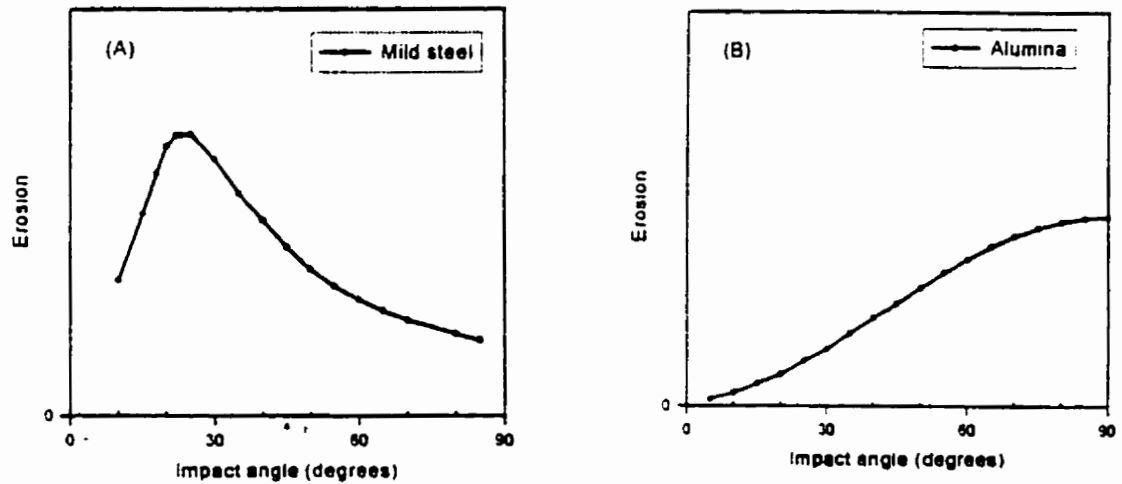
Figure 3.3 Dependence of wear on impact angle (a) for a typical ductile metal (de Bree *et al.* 1982) and (b) for a brittle ceramic (Finnie *et al.* 1967)

Figure 3.3a shows by way of example the variation with impingement angle of wear of a low-carbon steel by silica/water slurry. Similar results are found for gas-borne particles. In contrast. Figure 3.3b shows the behavior of an alumina ceramic. eroded by silicon carbide particles.

Other flow conditions can influence the wear in different ways. i.e. the rotated particles and more particles striking on the object gives more wear. temperature and corrosive environment also influence the wear in some cases.

3.3.2 Particle properties

The nature of the particles can profoundly affect the rate of wear. The mechanical properties of the particles, especially hardness, are important: if the hardness is greater than about 1.2 times that of the surface, the particle will be able to scratch the surface. For lower particle hardness, scratching will not occur and the wear rate will be much lower. When the particles are appreciably harder than the material, their hardness becomes less important and there is little or no dependence of wear rate on particle hardness.

The shape of the particles also affects the wear rate, although it is difficult to define a quantity. It is generally true that the sharper and more angular the particles are, the greater the wear rate will be. The difference in wear rate between angular and rounded particles may be greater than a factor of ten.

Wear rates are found to depend on particle size too. There is a genuine size effect in abrasion and erosion which leads to decreasing wear rate with diminishing particle size (Misra and Finnie 1981).

3.3.3 Materials properties

Materials differ in their susceptibility to erosive wear, and also in the mechanisms by which such wear occurs. In most metals, local plastic flow occurs at the points where hard particles strike the surface, and wear proceeds by the formation and detachment of

plastically deformed fragments. Wear fragments may be formed and detached by the action of a single particle, but more commonly several successive stages of deformation from subsequent impacts are needed to remove material. More brittle materials such as ceramics, on the other hand, may suffer local brittle fracture leading to the removal of material by the propagation and intersection of cracks. With small enough abrasive particles, the volume of material under stress may be too small for brittle fracture to occur, and material may then be removed by plastic processes. When this occurs the dependence of wear on angle is like that of ductile metal rather than that of a brittle material.

Generally, materials harder than the erosive particles still experience wear, but considerably more slowly than softer materials: more particle impacts are needed to remove each fragment of debris, and from what is known of the mechanisms it is evident that fatigue processes are involved.

3.4 Wear in Fluidized Beds

The previous section reviewed the impact wear by solid particles at relatively high impingement velocity. However, the wear in the fluidized beds is different than the impact wear. As pointed out by Stringer (1988), dense-phase particles striking the surface of the tubes are not independent of the particles which follow so that the mechanism for wear in the fluidized beds may involve corrosion, erosion, abrasion, fatigue, etc.

Internal objects such as tubes can suffer serious wear in the fluidized bed, and in some applications this wear may be so severe as to limit the life of a component or tube Zhu *et al.* (1990, 1991) conducted detailed study on the wear in fluidized beds. They found that the wear of the tubes in the fluidized beds may be influenced by many variables, including gas velocity, particle size, shape and hardness, tube diameter, and tube material properties. Zhu *et al.* (1988) pointed out that wear is most likely to occur when particles in the wakes behind bubbles and in the caps at the top of bubbles strike the underside of tubes. The average particle velocities are closely related to average void velocities, indicating that particle motion in the vicinity of a tube is mainly caused by voids or bubbles. Bubble coalescence immediately below a tube can lead to abnormally large impact velocities which may be of considerable importance in the wear process since wear is approximately proportional to the 2.3 power of impact velocity.

The particle properties influence the wear rate in the fluidized beds. The wear rate increased with particle size. Increasing the particle hardness augmented the wear rate of ferrous metals, but did not appreciably affect the wear of non-ferrous metals. Angular particles caused much faster wear than rounded ones (Zhu *et al.* 1990).

The wear rate in the fluidized beds is also influenced by the tube dimensions, location, configuration, and the material properties. The wear rate increased slightly with a reduction in the tube diameter. Wear was higher in the middle of the bed than close to the walls due to the increased frequency of voids in the bed interior. Young' s modulus appeared to be the major tube material property that influences the wear rate, a higher

Young's modulus producing higher resistance to wear. The wear rate was relatively

insensitive to changes in material hardness, yield strength and tensile strength (Zhu *et al.*

1990).

In low velocity gas fluidized beds, particle impacts occur when the wakes of

bubbles slam into the underside of the tube as well as when the leading edge of bubbles

impinge on the tube. The number of impinging particles is therefore related to the void

frequency, f_v, while the particle velocity is related to the void velocity, u_v. These two

quantities can be estimated by standard hydrodynamic relationships for bubbling and

slugging fluidized beds (Clift and Grace 1985).

Zhu *et al.* (1991) established a simple model based on the following assumptions:

- Material loss occurs by surface fatigue

- Local plastic deformation, which precedes material loss by surface fatigue, is

  related to the degree of elastic deformation. Material loss is assumed to be

  proportional to the volume of target material which undergoes substantial

  elastic deformation due to particle impacts.

- The wear rate is proportional to h^3, where h is the maximum deformed depth.

  This effectively assumes that volumes of target material, stressed enough to be

  eventually removed by fatigue-crack initiation and growth, are geometrically

  similar.

- All particles striking the surface transfer the same fraction of their kinetic

  energy to the target surface regardless of their size, density or velocity.

With the above assumptions, a predictive equation was derived by Zhu *et al.* (1991) for wear in bubbling and slugging fluidized beds:

$$E = C f_v \rho_p d_p^{1.2} u_v^{2.1} (1.1 - \Phi_p)$$ (3.30)

where E is the wear rate, which is a strong function of impact velocity u_v, particle size d_p, particle shape Φ_p, and void (bubble or slug) frequency f_v. C is a function of material properties and its values for some materials are given in Table 3.2.

Table 3.2 C values of various metals with silica sand as the particulate materials (Zhu *et al.* 1991).

Material	$C(\dfrac{\mu m / h \cdot s^{3.1}}{kg \cdot m^{0.3}})$
Brass	0.115
Aluminum 2011	0.119
Copper	0.0683
Stainless steel 304	0.0101
Carbon steel 1050	0.0199

The predictions by this empirical equation are consistent with the experimental results. The equation can be applied to metal tubes under low velocity (1-5 m/s) particle impacts. It can be used to predict wear rates for horizontal tubes in bubbling and slugging fluidized beds operated at low temperature, with a single parameter C, which is a function of tube and particle material properties. Wear increases as the Young' s modulus decreases and as the particle hardness increases.

Nomenclature

ε_B Fraction of total bed volume occupied by bubbles.

ε_b Voidage.

ε_{mf} Voidage in the minimum fluidizing conditions.

ρ_p Particle density (kg/m^3).

Φ_p Particle shape factor = (particle projected area/area of smallest circumscribing circle)$^{0.5}$.

A Cross-sectional area of the bed (m^2).

A_D Area of distributor plate per orifice (m^2).

b   Empirical constant in Equation (3.30).

C_0 Coefficient depending on the distribution of bubbles across the bed.

D Bed diameter or distance between walls (m).

D_e Average bubble size in Equation (3.10) (m).

D_e Diameter of a sphere having the same volume as the bubble (m).

$D_{e,x}$ Maximum bubble size attainable by coalescence (m).

$D_{e,0}$ Mean bubble size formed at the distributor (m).

d_p Mean particle diameter (m).

d_p Surface-to-volume (Sauter) mean particle diameter (m).

E Circumferential averaged wear rate .

f_b Mean bubble frequency at any level.

f_s Slug frequency.

f_v Void (bubble or slug) frequency.

g Acceleration of gravity.

G_b Visible bubble flow rate.

h Maximum deformed depth (m).

H Height of fluidized bed (m).

H_{mf} Height of incipiently fluidized bed (m).

k Coefficient for slug wake.

L_s Slug length (m).

m Shape factor for slug volume defined by (3.19).

m,n Constant exponents in Equation (3.30).

N Number of slugs per unit volume.

u Interstitial fluid velocity (m/s).

$u_{b,x}$ Rising velocity of a single bubble (m/s).

u_{mf} Interstitial gas velocity at incipient fluidization (m/s).

u_{mf} Minimum fluidized velocity (m/s).

u_{ms} Minimum slugging velocity (m/s).

u_s Rise velocity of slug in a freely slugging bed (m/s).

$u_{s,x}$ Rise velocity of single slug (m/s).

u_T Terminal settling velocity of spherical particles (m/s).

u_v Void (bubble or slug) rise velocity (m/s).

V_s Slug volume (m³).

W_s Height of slug wake (m).

X Height above the distributor (m).

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CHAPTER 4: EVALUATION OF DIFFERENT DENTAL MATERIALS USING A SLUGGING FLUIDIZED BED

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4.1 Introduction

Wear is generally accepted as a "progressive loss of substance from the surface of a body brought about by mechanical action" (Jones *et al.* 1978). Wear of the tooth or its restorations is not a single process, but the overall result of at least six underlying processes which seldom act in isolation. In tribology, the fundamental wear processes in dentistry are: abrasive wear, adhesive wear, wear due to fatigue, fretting wear, erosive wear and corrosive wear (Pugh 1973). These six processes occur in various combinations to cause surface loss of tooth or dental restorations. In an *in vivo* investigation, Lambrechts *et al.* (1989) reported vertical wear of enamel to be 20 μm and 40 μm per year in the premolar and molar regions, respectively. It is important that the restorative materials have a similar or greater wear resistance than the tooth enamel. Surface texture and surface hardness have each been investigated as possible determinants of wear rate. However, surface hardness has been shown to be a poor indicator of wear rate (Ramp *et al.* 1997).

Many previous research works have been carried out on comparing the wear resistance of different dental materials *in vivo* and *in vitro*. The *in vivo* test is based on the teeth or the restorations in the mouth, so the measurement is relatively more difficult to take, and it requires more complicated and expensive equipment. Wear in dentistry is also a long-term process. The time for an observable wear usually takes more than 6 months. Furthermore, speed of movement of the mandible, rate of chewing, and forces developed in chewing are different for different individuals, and they also vary with the types of food, the age groups, the chewing habits, etc. Very few quantitative *in vivo* wear investigations have been published up to now due to the above difficulties.

In the literature, numerous results from *in vitro* wear tests have been published. Unfortunately, there has not been an accurate way to assess the wear resistance till today. The *in vitro* tests differ widely in the time and effort required for taking the measurements, the cost of equipment and labor, and in their ability to generate quantitative wear values (Bayne et al. 1994). Two of the most common test methods *in vitro* include the toothbrush/dentifrice abrasion test and the pin and plate method. The former is a three-body wear test that uses a slurry of abrasive particles. The pin and plate method is essentially a two-body abrasion test that compares pairs of materials against one another (Ratledge *et al.* 1994). All these methods are still in use today, but they all have many limitations. First of all, the test conditions are far from completely simulating the *in vivo* wear due to the complexity of the oral environment. Secondly, the units for the *in vitro* wear measurement are normally in the micro level so that it is hard to precisely

quantify the wear rate. Third, most of the *in vitro* tests in use today operate under a relatively heavy load condition in a very short test period, and most of them take less than 24 hours for a single test. Compared with 20-40 µm vertical loss a year for enamel, how well such a process can mimic the mechanisms in the natural process remains to be a question.

The widespread development of the new composite restorative materials creates the need for more reliable and simpler technique to assess restoration wear resistance. Considering the limitations of the conventional wear tests, the gas-solid fluidized bed which is commonly used in the chemical process industry was proposed in this work as an excellent choice for testing the wear of dental materials.

In a fluidized bed, fine particulate materials of several micrometers to several millimeters are suspended by up-flowing gas. The gas-solids suspension thus formed behaves like a fluid (so that the name fluidized bed). Solids particles move freely in the fluidized bed due to the movement of gas bubbles or slugs. Gas flow, heat transfer, and the movements of solids inside a fluidized bed have been studied for many years. Models based on the behavior of bubbles and slugs in beds of fine particles have been suggested to predict the solids flow behavior inside the beds. Zhu *et al.* (1990; 1991) have carefully studied the mechanism of wear of tubes in fluidized beds and found that the wear rate of a horizontal tube increases with particle velocity, particle size, density and sharpness. They also concluded that when the fluidized bed is operated in the bubbling or slugging regime, wear caused by the solid particles to the object in the column is the result of a

few different wear mechanisms, including abrasive wear, adhesive wear, erosive wear, etc. So, the principal mechanisms in dental wear can be well simulated in the fluidized bed. The time for taking one set of wear test is about a week, which is between the conventional *in vitro* test and the *in vivo* test. This is a more reasonable time period for mimicking the long dental wear process *in vitro*.

This study is an attempt to grade the dental restorative materials in order of their resistance to wear using a simple test technique. The experiments were carried out in a gas-solid slugging fluidized bed where the periodical passage of slugs provides very stable and well predicted particle flow patterns (Hovmand and Davidson, 1971). The major parameters varied were particle properties, dental material properties and gas superficial velocity. Its main advantage lies in its simplicity, the speed with which it can be performed, and good simulation of the *in vivo* environment.

The main purposes for this study were (1) to develop a simple but reliable *in vitro* wear test apparatus/method that would simulate the main clinical wear mechanisms using a fluidized bed operated under slugging flow regime, (2) to study how increasing the size of the abrasive particles and/or the particle velocities influence the specimen wear rate for better understanding the wear phenomena, and (3) to evaluate the wear rates of several different dental materials to provide useful information to dentists in selecting materials for dental restorations.

4.2 Apparatus and Materials

4.2.1. Apparatus

The experiments were carried out in a three-dimensional fluidization column at room temperature to measure the specimen' s wear rate under different operating conditions. Figure 4.1 shows the set-up of the fluidization apparatus used in this experiment.

The three-dimensional fluidization column itself has a diameter of 76 mm and a height of 0.91m. A stainless steal beam was fixed horizontally crossing the axis of the column at 0.39 m above the air distributor. Test specimens can be fixed below the beam to face the upflowing particles brought up by slugs. Two specimens can be tested each time. placed symmetrically near the center of the column and 15 mm apart from each other. Two mounting ports were installed at the two ends of the beam to allow the quick change of the specimens. The packed bed height was kept constant at 0.343 m for all tests to ensure comparability. Due to bed expansion. the specimens were covered by the particles when fluidized.

The column was constructed entirely from plexiglas with a thickness of 6.35 mm to achieve wall rigidity and resistance to wear. On the top of the column, there is an expansion section with a diameter of 127 mm and a height of 0.46 m. This expansion section is designed for slowing down the particle velocity through increased cross-section

Figure 4.1 Experiment set-up (units in mm)

area. so that the particle entrainment can be efficiently reduced within a relatively short column length. The air distributor was a multi-orifice plate with an opening area of 1.5%.

The orifice plate was covered with a fine steel wire screen to prevent solid particles from dropping through the holes. Air was from the building compressor which has a gauge pressure of 180 kPa and can provide a maximum superficial velocity of 3.0 m/s in the fluidization column. The air flowrate was controlled by adjusting the two valves: Valve-I and Valve-II and monitored by a rotameter. The pressure at the pressure gauge was kept at 70 kPa constantly.

4.2.2. Specimen Preparation

The specimens made of different dental materials were fabricated into a molar crown shape by using the replica technique. The technique is borrowed from an earlier metal crown fabrication method and relies on the precise reproduction of tooth surfaces by the impression material used. A well selected human third molar was used as the model. The root of this tooth was removed at the cementoenamel junction (1 in Figure 4.2). An impression was first taken out of this selected molar crown by using the dental alginate impression material. Then. a dental limestone mold was duplicated out of the impression immediately after setting of the alginate. Now. this limestone mold has the same shape of the selected molar crown (2 in Figure 4.2). Using the limestone mold. a final lead three-division impression was made with a special tool shown in Figure 4.2 (3). The lead impression can be split in three and repositioned in one (4). This special

property ensures the material teeth made out of this impression to be easily removed. Otherwise the undercut of the tooth crown will make the material tooth impossible to be removed out of the lead impression. Then. different dental restorative materials were filled in this impression and cured layer by layer (5). Finally, a screw with known weight was set into the material on the side opposite to the enamel side during curing (6). This screw provides a mounting handle for fastening to the beam in the column. The whole procedure was illustrated in Figure 4.2 and the final specimens all have the uniform shape and size. same as the selected molar crown portion. The surface area of the tooth crown was approximately $2.9 \ cm^{2}$ and the shape is shown in Figure 4.3 (a) and (b).
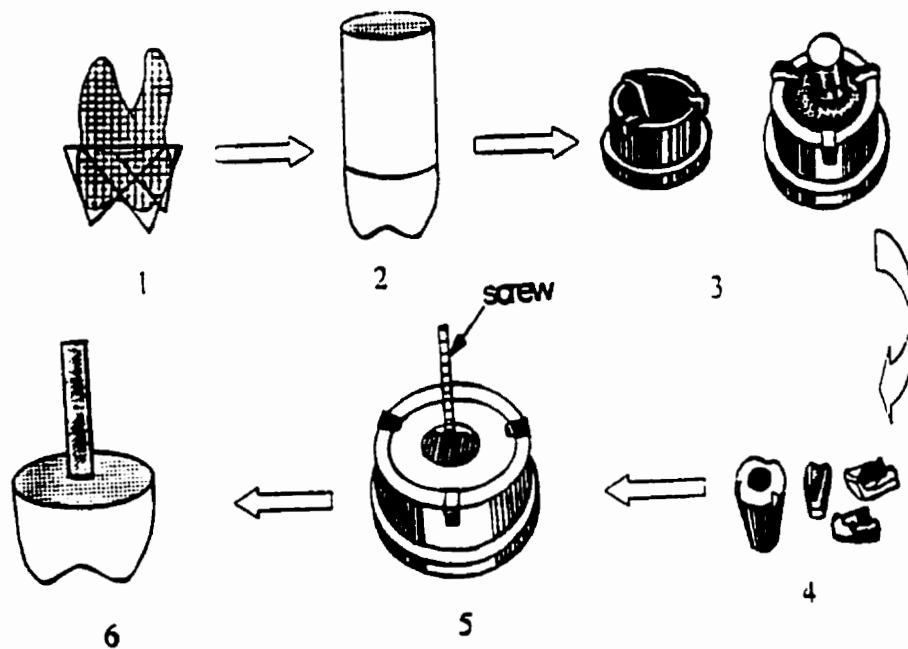
Figure 4.2 Specimen preparation

(a)   (b)
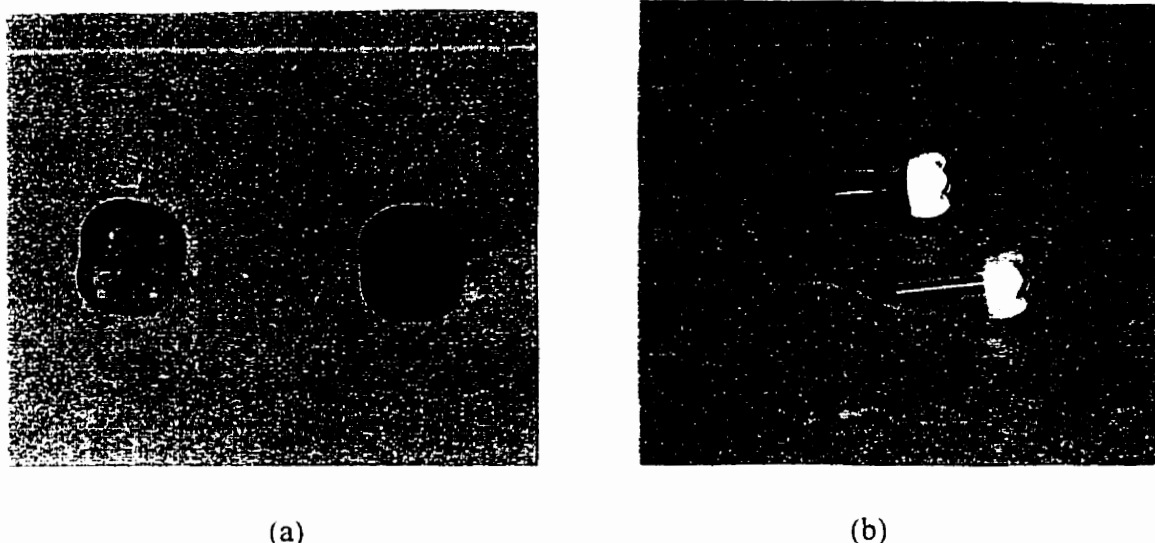
Figure 4.3 Final samples (a) top view (left is the final specimen, right is the selected real tooth crown) and (b) side view (top is the real tooth crown, bottom is the final specimen)

4.2.3. Measurement method

The wear rates were obtained from the weight loss of the specimen over time. The weighing method was very simple. The specimens were mounted onto the supporting beam and worn by the solid particles in the fluidized bed. The specimens were carefully weighed before and after the test. Each time before weighing, the specimens were put into an ultrasonic cleaner for 5 minutes to get rid of the fine particles which stick on the surface. The weight loss was the difference between the two measurements before and after a certain time interval. The material teeth were usually weighed every 40-60 hours. The wear rate was calculated from weight loss of the samples over the time consumed. The balance was a digital balance with an accuracy of 0.01 mg. To minimize disturbances, the balance was located on a heavily loaded table to reduce vibration, and

was enclosed in a box to prevent air currents. A special stainless steel screw was always kept aside and used as a standard weight to calibrate the balance every time it was used. These special efforts allowed the balance to reach its maximum available accuracy and helped give good reproducibility.

4.2.4. Materials

Four different types of particles (three grades of silica sand and one grade of glass beads) were used as the fluidized bed materials in the tests in order to test how the particle properties influence the wear resistance of the specimen. Table 4.1 listed the properties of these particles.

Table 4.1 Particle properties

Particle Name	Mean Diameter (μm)	Density (kg/m³)	Sphericity	Minimum Fluidization Velocity (m/s)	Terminal Velocity (m/s)	Hardness
Silica Sand #1	700	2530	0.86	0.3020	5.14	350
Silica Sand #2	400	2530	0.86	0.1199	2.97	350
Silica Sand #3	200	2530	0.86	0.0313	1.38	350
Glass Beads	355	2500	1.0	0.0092	2.61	340

Four typical types of dental materials were studied: compomer F-2000, composite Z-100, glass ionomer cement Vitremer and amalgam. Their composition and key properties were listed in Table 4.2.

Table 4.2 Dental materials

Material Name	Composition	Manufacturer	Batch number
F2000	Compomer	3M company	44-0019-9175-9
Z100	Composite	3M company	44-0017-8308-1
3M Vitremer	Glass ionomer cement	3M company	44-0017-8331-1
Amalgam	Dental Amalgam		

4.3. Results and Discussion

The operating conditions for the fluidized bed are provided in Table 4.3. The independent variables were:

- Particle size and sphericity

- Gas superficial velocity

- Tooth materials

Calculations based on literature correlations (Stewart and Davidson 1967):

$$u_{ms} = u_{mf} + 0.07(gD)^{1/2}$$ (5.1)

and

$$D_e/D > 1/2$$ (5.2)

confirm that the bed is in the slugging regime for all test conditions. The base condition was set at a gas superficial velocity of 0.464 m/s with 400 μm silica sand.

Table 4.3 Operating conditions

	Material name	d_p (μm)	u (m/s)	u-u_{mf} (m/s)
Base condition	F-2000	400	0.464	0.772
Slug velocity vs. wear rate for 400 μm particles	F-2000	400	0.25	0.558
	F-2000	400	0.464	0.772
	F-2000	400	0.65	0.958
	F-2000	400	0.8	1.108
Slug velocity vs. wear rate for 700 μm particles	F-2000	700	0.442	0.558
	F-2000	700	0.656	0.772
	F-2000	700	0.842	0.958
	F-2000	700	0.992	1.108
Wear rate for different materials	F-2000	400	0.464	0.772
	Z-100	400	0.464	0.772
	Amalgam	400	0.464	0.772
	Vitrimer	400	0.464	0.772
Wear for different particle size	F-2000	700	0.464	0.772
	F-2000	400	0.464	0.772
	F-2000	200	0.464	0.772
Wear rate vs. particle Sphericity (glass beads)	F-2000	355	0.464	0.772

4.3.1. Incubation, initial finding

For fresh materials, there may exist an incubation period during which the wear rates differ from the wear rates for previously eroded materials. To test the incubation behavior, fresh-made samples of 3M-F2000 were first tested over an extended period. The weight losses of the materials were plot against operating duration in Figure 4.4 where the slopes of the curve present the wear rate of the dental materials. For the F-2000 compomer, the slopes of the curves are not constant but fluctuate at the beginning of the

wear process and then became quite constant after 50-60 hours of wear. This suggested

the existence of an incubation period.
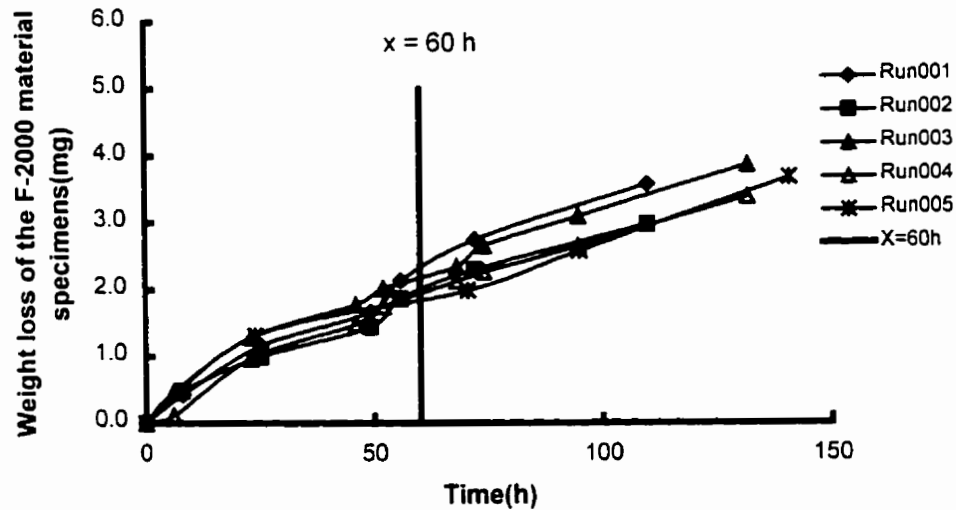
Figure 4.4 Incubation period for material F-2000

This phenomenon can be more clearly observed by plotting the variation of the

wear rate with time. Figure 4.5 shows that the average wear rate of material F-2000 was

around 45 µg/h in the first 20 hours of wear, and then dropped to about 20 µg/h from 20

to 50 hours. After 60 hours, the wear rates started to stabilize around 25 µg/h. Therefore,

wear in the first 60 hours was considered as in the incubation period. Additional

experiments proved that all materials tested have such an incubation period and this

period is always less than 60 hours. The mechanism of the incubation was not further

investigated since the incubation period was found to be short and therefore of limited

practical interest.
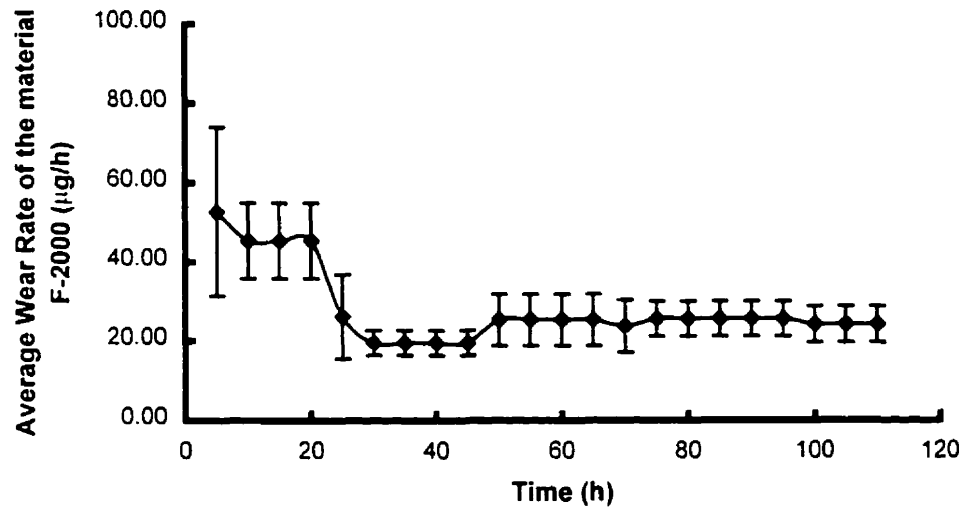
Figure 4.5 Average wear rate of F-2000 vs. Time under 0.464 m/s
superficial air velocity using 400 μm particles

Because of the existence of the incubation period, all new specimens were worn

in the column for about 60-80 hours before taking any measurements in order to exclude

the initial high wear rate in the incubation period. After the initial incubation period,

specimens were weighed at each 40-60 hours interval.

There is also a similar "incubation" effect on the particles used. Since the sand

particles are initially somewhat angular and gradually loose sharp edges as the wear

proceeds, the wear rate of a specific specimen would initially be higher if fresh sands are

used. To avoid the uncertainties caused by the initial change of particle sharpness, all particles were fluidized over an extended period of time before they were used for any wear tests.

4.3.2. The influence of superficial air velocity on the wear

The superficial air velocity is a very important parameter that influences the wear rates. In this experiment, four different superficial air velocities were chosen for each of the two grades of sand particles tested. The material teeth were all made from composite 3M-F-2000. The abrasive particles were silica sands of 400 μm and 700 μm in diameter. The only changing parameter is the air velocity.

In a slugging fluidized bed, particle movements are mainly associated with the passage of slugs. As illustrated by Zhu *et al.* (1989), particles around the specimens are relatively stagnant when a slug is not in the vicinity. As a slug is approaching the specimen, particles below the specimens begin to move upwards, causing mainly abrasive wear. When the specimens are enveloped by the slug, there is almost no wear since there are few particles inside the slug. As the slug passes, particles behind the slug are brought up to impact on the specimens, leading to more wear. Thus, those particles which directly cause wear are passing the specimens at very similar velocity as the slugs.

Changing the superficial air velocity changes the slug velocity, which in term changes the particle impact velocity and therefore changes the wear rate. For meaningful

comparison. the slug velocity was kept the same for the two particle sizes in each pair, as shown in Table 4.3. The Ormiston's equation (Ormiston *et al.* 1965) was used for calculate the slug velocity:

$$u_s = (u - u_{mf}) + 0.35(gD)^{1/2} \tag{5.3}$$

Experimental results under different slug velocities are shown in Figure 4.6. Generally. the wear rate for 700 micron particle test is higher than 400 micron particles under the same slug velocity. A statistic analysis (F-test) shows that the differences are significant. In Figure 4.6a, the wear rate is seen to increase with the slug velocity until the slug velocity reaches 0.958 m/s for the 400 micron particles. A similar trend can be seen in Figure 4.6b for the 700 micron particles. As the slug velocity increases. the velocity of the wear-causing particles also increase. leading to high wear. The reduction of wear at further higher slug velocity can be explained by the variations of the bed density or the particle holdup with the air velocity in the slugging bed. The wear of the material tooth is proportional to the particle velocity and the number of particles which strike on the surface. For a higher slug velocity. the particle velocity increases. but at the same time. the bed expansion also increases which reduces the number of particles per unit volume. At very high velocity. the bed density is significantly reduced so that the combined effects of these two factors give the results shown in Figure 4.6.

The above point is further illustrated on Figure 4.7. where wear rates at various slug velocities shown in Figure 4.6 are normalized by the bed density with $u_s = 0.772$ m/s as the base condition. With this correction, the wear rate is clearly shown to increase with

slug velocity. In addition, Figure 4.7 further reveals that the wear rate is proportional to the 1.7-2.0 power of slug velocity, consistent with the finding of Zhu *et al.* (1990), who reported a power of 2.1.

The upflowing gas velocity creates the driving force for wear by the particles. It functions similarly to the biting force in the mouth. The particle density may be imagined as the food particle density. So, if the biting force is high and there are sufficient food particles, the tooth or the dental material suffers high wear rate. When any of these parameters changes, the result will be different.
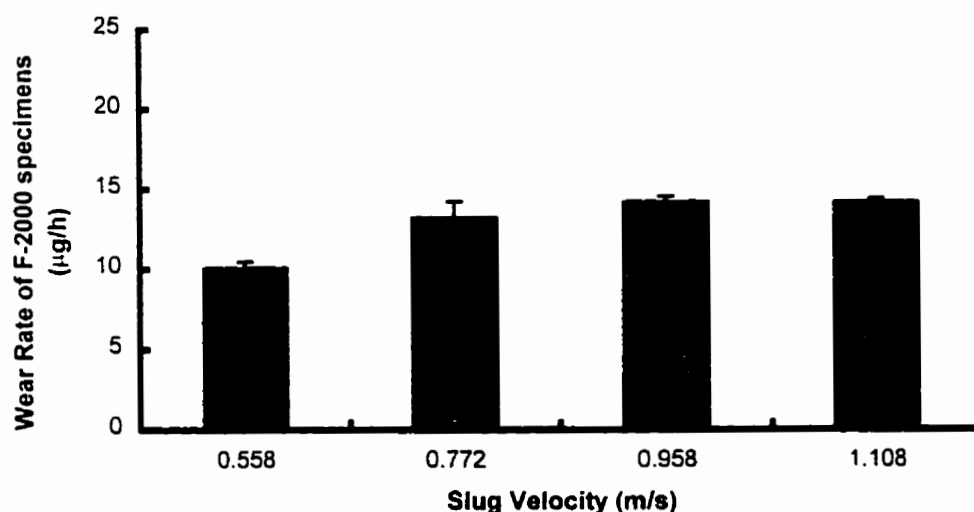
Figure 4.6a Wear rates of material F-2000 vs. different slug velocities
with 400 µm particles
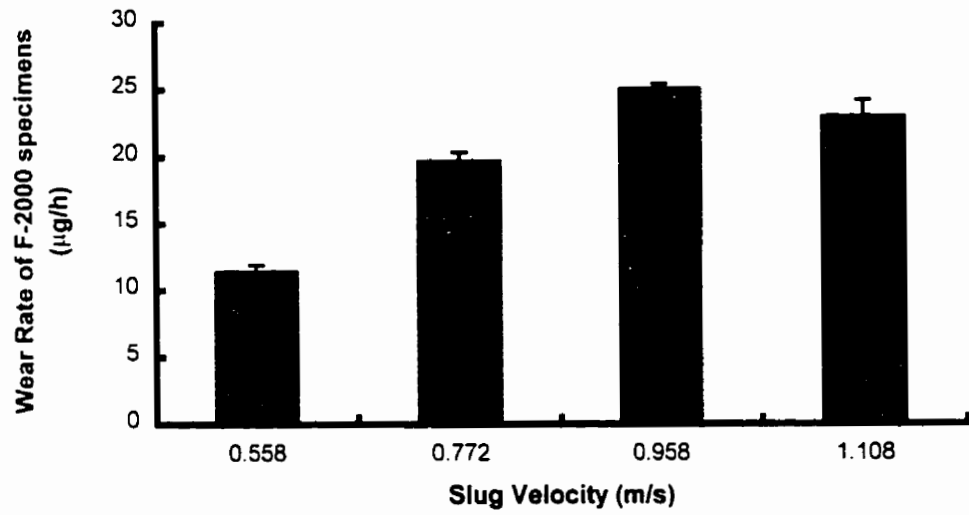
Figure 4.6b Wear rates of material F-2000 vs. different slug velocities with 700 μm particles
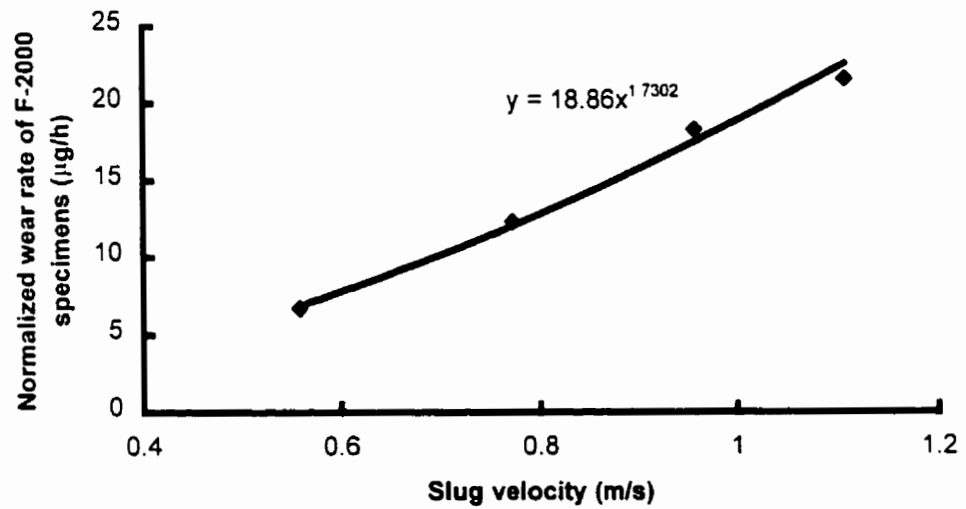
Figure 4.7a Normalized wear rate vs. slug velocity for F-2000 using 400 μm particles
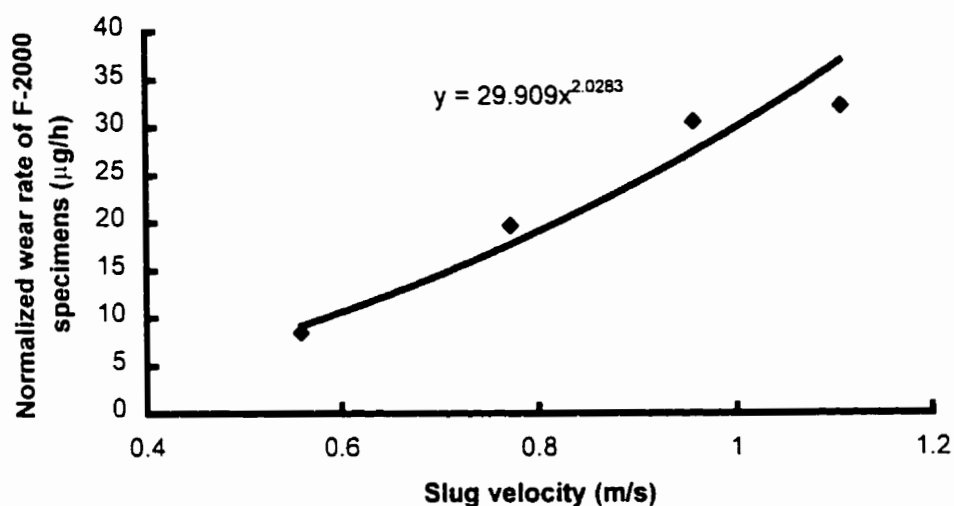
Figure 4.7b Normalized wear rate vs. slug velocity for F-2000 using 700 μm particles

4.3.3. The influence of particle properties

Particle properties are other factors influencing the wear rate. Since changes in particle properties also affect the slug behavior, determination of the effects of particle size and sphericity are carried out under the same slug velocity. Three grades of silica sands with different sizes were used to test the effect of particle size. The slug velocity was kept the same for the different particles by adjusting the superficial gas velocity. Figure 4.8 shows how the wear rate changes under different particle sizes.
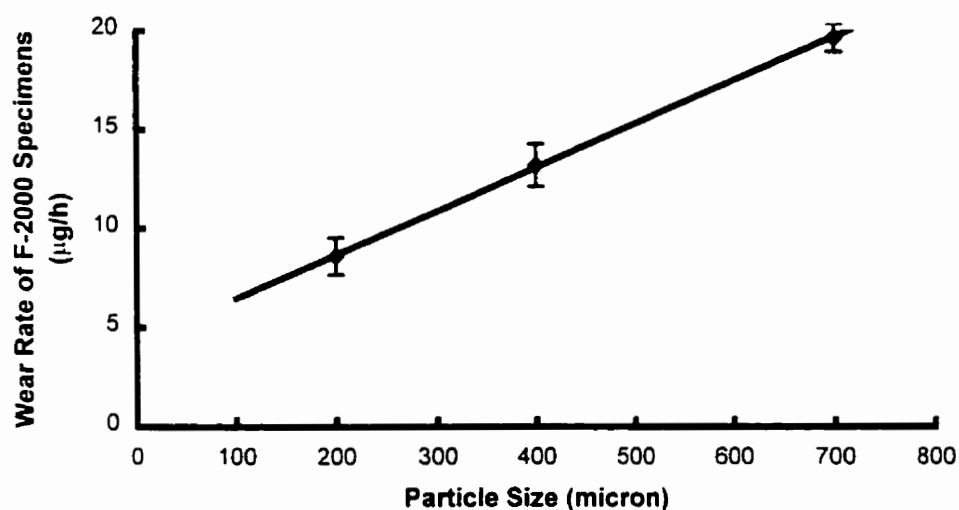
Figure 4.8 Wear rate of material F-2000 vs. different particle size under 0.772 m/s slug velocity

From the Figure 4.8 above, it is very obvious that the size of the particles does affect the wear rate of the dental materials. Increasing particle size increases the wear rate almost linearly when the slug velocities are constant. This is consistent with the findings of Zhu *et al.* (1990) that wear rate is approximately proportional to the 1.2 power of the particle size. Considering that the size of the particles represents that of the food particles in the mouth, it is reasonable to see that bigger particles give higher wear rate if the biting force is constant in mastication.

Table 4.4 shows the wear rates of two types of 355 μm particles: silica sands and glass beads. To facilitate direct comparison, the wear rate of 400 μm sand was corrected to that of 355 μm through Figure 4.8. The wear rates on the F-2000 material are different for the two types of particles. The wear created by the glass beads was less than by silica

sands. This difference is shown to be significant by a statistical analysis (T-test). This is reasonable because the glass beads are less sharp and have similar hardness than silica sands. Given the small difference in particle hardness. it is easy to conclude that particle sphericity has a significant effect on the wear rate.

Table 4.4 Wear rates of F-2000 by two different particles of 355 μm

	Wear rate (μm)	Standard deviation	Hardness	Sphericity	Material type
Silica sands	24.9	0.750	350	0.86	F-2000
Glass beads	10.2	0.566	340	1.0	F-2000

4.3.4. The influence of the materials properties

Under the same operating conditions. different materials suffer different wear rates. with some materials being eroded up to 5 times faster than others. The wear rate of different dental materials under the base condition (400 μm sand. at u_g =0.464 m/s) was shown in Figure 4.9. The resin based material Z-100 proved to be the most wear resistant material. while the glass ionomer cement has the lest resistant to wear.
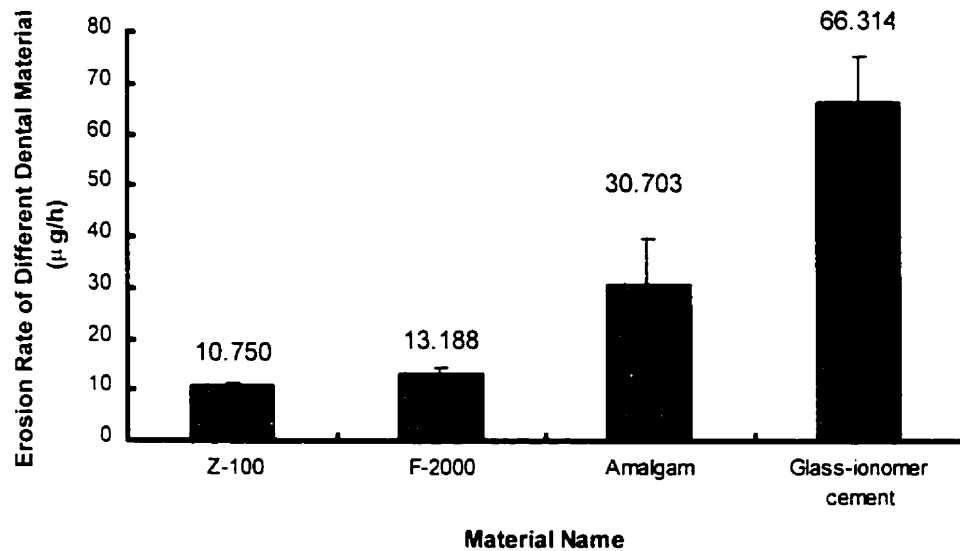
Figure 4.9 Wear rate of different dental materials under 0.772 slug velocity with 400 μm wear particles

4.4. Conclusions

The following conclusions can be drawn from this test:

1. There exists an incubation period at the beginning of the wear test due to the surface texture and/or the different initial wear mechanism.

2. The wear rate of the dental materials is proportional to the superficial gas velocity, the abrasive particle size and sharpness in this operating system. It presents that the wear rate of the dental materials is proportional to the biting force, hardness of the food particles and the size of the food particles.

3. The material properties changes the wear rate. Z-100 proved to be the most wear resistant materials in this experiment.

4. The fluidized bed is a simple and reliable tool to test wear rate of different dental materials.

Nomenclature

D Bed diameter or distance between walls (m)

D_e Equivalent bubble diameter (m)

g Acceleration of gravity (m/s^2)

u Interstitial fluid velocity (m/s)

u_{mf} Minimum fluidized velocity (m/s)

u_{ms} Minimum slugging velocity (m/s)

u_s Rise velocity of slug in a freely slugging bed (m/s)

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CHAPTER 5: MIMICKING THE WEAR OF DENTAL MATERIALS IN A NOVEL FLUIDIZED BED TEST APPARATUS

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5.1. Introduction

Human tooth wear occurs at a rate that annual decreases in molar cusp height rarely exceed 50 μm in most human groups (Xhonga *et al.* 1972, Roulet *et al.* 1980, Molnar *et al.* 1983b, lambrechts *et al.* 1989, Teaford *et al.* 1991). The main purpose of the restorative dentistry is to replace diseased or lost tooth structure with materials that have the same function and appearance as the enamel. Dental materials manufacturers have marketed many new types of composite resins to meet this purpose in restoration. However, it is difficult to develop a material that has both the enamel-like finish and the function of teeth. Because of the large number of commercial composites that have been and continue to be developed, there is always the need for appropriate *in vitro* tests on the material wear resistance in order to evaluate and improve the quality of the materials. In many practical situations, the wear process has involved a combination of more than two wear mechanisms. The design of laboratory tests to assess wear resistance should be based upon the correct combination of wear mechanisms similar to that in the clinical situation (McCabe *et al.* 1981).

In the literature, numerous results from *in vitro* wear tests have been published. Unfortunately, there has not been an accurate way to assess the wear resistance till today. The *in vitro* tests differ widely in the time and effort required for taking the measurements, the cost of equipment and labor, and in their ability to generate quantitative wear values (Bayne et al. 1994). Two of the most common test methods *in vitro* include the toothbrush/dentifrice abrasion test and the pin and plate method. The former is a three-body wear test that uses a slurry of abrasive particles. The pin and plate method is essentially a two-body abrasion test that compares pairs of materials against one another (Ratledge *et al.* 1994). All these methods are still in use today, but they all have many limitations. First of all, the test conditions are far from completely simulating the *in vivo* wear due to the complexity of the oral environment. Secondly, the units for the *in vitro* wear measurement are normally in the micro level so that it is hard to precisely quantify the wear rate. Third, most of the *in vitro* tests in use today operate under a relative heavy load condition in a very short test period, and most of them take less than 24 hours for a single test. Compared with 20-40 μm vertical loss a year for enamel, how well such a process can mimic the mechanisms in the natural process remains to be a question.

A fluidized bed has been proposed here as a better choice for measuring the wear rate of the dental restorative materials. In a fluidized bed, fine particulate materials of several micrometers to several millimeters are suspended by up-flowing gas. The gas-solids suspension thus firmed behaves like a fluid (so that the name fluidized bed). Solids

particles move freely in the fluidized bed due to the movement of gas bubbles or slugs. When the fluidized bed is operated under low gas velocity, wear caused by the solid particles to the object in the column are the results of a few different wear mechanisms. including abrasive wear. adhesive wear. erosive wear. etc. Its main advantage lies in its simplicity and good simulation of the *in vivo* environment.

A preliminary investigation (Li *et al.* 2000) on the test method and equipment reported that:

1. The fluidized bed is a simple and reliable tool to test wear rate of different dental materials.

2. There exists an incubation period at the beginning of the wear test due to the surface texture and/or the different initial wear mechanism.

3. The wear rate of the dental materials is proportional to the superficial gas velocity. the abrasive particle size and hardness in this operating system.

4. The material properties change the wear rate.

The main purposes for this study were to evaluate the wear rates of several different dental materials compared with the wear rate of tooth to provide useful information to dentists in selecting materials for dental restorations. and find how the hardness of the material influence the specimen wear rate for better understanding the wear phenomena.

5.2. Materials and Methods

In this study, the experiment set up and method is the same as the preliminary investigation, the difference lie in more dental materials involved and the hardness test.

5.2.1 Dental materials used in the study

Eight dental materials were studied in the three-dimensional fluidized bed test apparatus. They were F-2000, Z-100, Vitremer, Z-250, Amelogen Universal, Permafalo, Vitalescence and Amalgam. Their composition and some properties were listed in Table 5.1.

5.2.2. Specimen Preparation

The specimens made of different dental materials were fabricated into a molar crown shape by using the replica technique. The technique is borrowed from an earlier metal crown fabrication method and relies on the precise reproduction of tooth surfaces by the impression material used. A well selected human third molar was used as the model. The root of this tooth was removed at the cementoenamel junction (1 in Figure 5.1). An impression was first taken out of this selected molar crown by using the dental alginate impression material. Then, a dental limestone mold was duplicated out of the impression immediately after setting of the alginate. Now, this limestone mold has the same shape of the selected molar crown (2 in Figure 5.1). Using the limestone mold, a

Table 5.1 Dental materials

	Filler type	Filler content	Resin type	Curing method	Manufacturer
F-2000	Compomer restorative system	NA	methacrylate	Visible-light Activated	3M Company
Z-100	Zirconia/Silica 3.5-0.01 μm	66% filled by volume	BIS-GMA and TEGDMA resin	Visible-light Activated	3M Company
Vitremer	Fluoroalumino silicate glass	NA	Light sensitive, aqueous solution of a modified polyalkenoic acid	Visible-light cure Mixing the powder and liquid	3M Company
Amelogen universal	0.7 μm average particle size	72% filled by weight; 60% by volume	BIS-GMA	Visible-light Activated	Ultradent Products, Inc.
Permaflo	0.7 μm average particle size	68% filled by weight	Methacrylate monomer 28% Alkylamino methacrylate 1% Camphorquinone 1%	Visible-light Activated	Ultradent Products, Inc.
Vitalescence	1 μm average particle, micro-hybrid	75% filled by weight	BIS-GMA based	Visible-light Activated	Ultradent Products, Inc.
Z-250	Zirconia/silica 0.01-3.5 μm particle size	60% filled by volume	BIS-GMA, UDMA, BIS-EMA	Visible-light Activated	3M Company

final lead three-division impression was made with a special tool shown in Figure 5.1 (3). The lead impression can be split in three and repositioned in one (4). This special property ensures the material teeth made out of this impression to be easily removed. Otherwise the undercut of the tooth crown will make the material tooth impossible to be

removed out of the lead impression. Then, different dental restorative materials were filled in this impression and cured layer by layer (5). Finally, a screw with known weight was set into the material on the side opposite to the enamel side during curing (6). This screw provides a mounting handle for fastening to the beam in the column. The whole procedure was illustrated in Figure 5.1 and the final specimens all have the uniform shape and size, same as the selected molar crown portion. The surface area of the tooth crown was approximately 2.9 cm^2 and the shape is shown in Figure 5.2 (a) and (b).
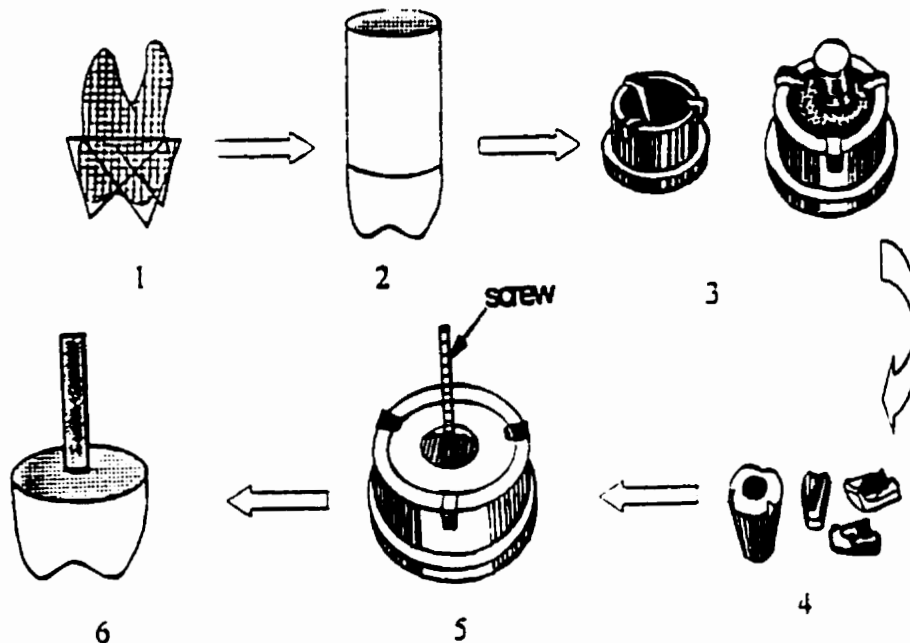
Figure 5.1 Specimens preparation
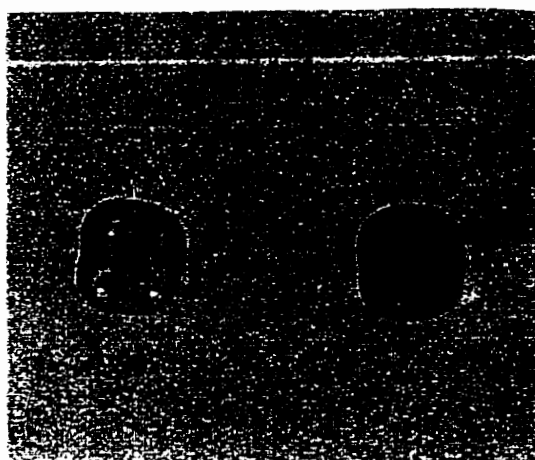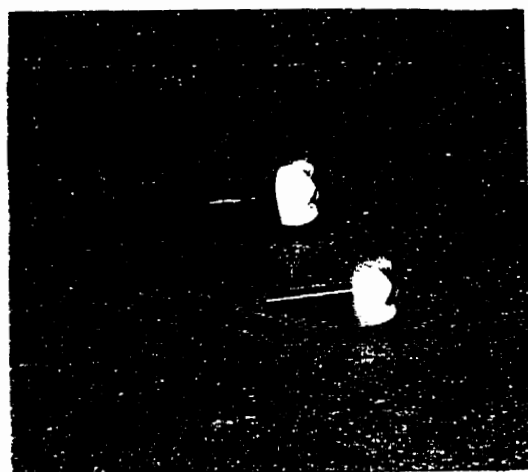
(a)   (b)

Figure 5.2 Final samples (a) top view (left is the final specimen, right is the selected real tooth crown) and (b) side view (top is the real tooth crown, bottom is the final specimen)

5.2.3. Measurement method

The wear rates were obtained from the weight loss of the specimen over the time. The weighing method was very simple. The specimens were mounted onto the supporting beam and worn by the solid particles in the fluidized bed. The specimens were carefully weighed before and after the test. Each time before weighing, the specimens were put into an ultrasonic cleaner for 5 minutes to get rid of the fine particles which stick on the surface. The weight loss was the difference between the two measurements before and after a certain time interval. The material teeth were usually weighed every 40-60 hours. The wear rate was calculated from weight loss of the samples over the time consumed. The balance was a digital balance with an accuracy of 0.01 mg. To minimize disturbances, the balance was located on a heavily loaded table to reduce vibration, and

was enclosed in a box to prevent air currents. A special stainless steel screw was always kept aside and used as a standard weight to calibrate the balance every time it was used. These special efforts allowed the balance to reach its maximum available accuracy and helped to give good reproducibility.

Hardness test was carried out for all the tested materials. There are many hardness-testing techniques that are frequently employed. Vickers test that is commonly used for engineering purpose was chosen for this investigation. Specimens were prepared in a small column shape with upper surface paralleled with the bottom, and diameter of the section was 0.5 cm. Specimen surface were carefully prepared (grinding and polishing) to ensure a well-defined indentation that may be accurately measured. A very small diamond indenter having pyramidal geometry is forced into the surface of the specimen. Applied loads were 1000g. The resulting impression is observed under a microscope and measured; this measurement is then converted into a hardness number (Vickers number). A minimum of six indentation measurements were taken for each test material to ensure accuracy.

5.2.4. Apparatus

The experiments were carried out in a three-dimensional fluidization column at room temperature to measure the specimen' s wear rate under different operating conditions. Figure 5.3 shows the set-up of the fluidization apparatus used in this experiment.
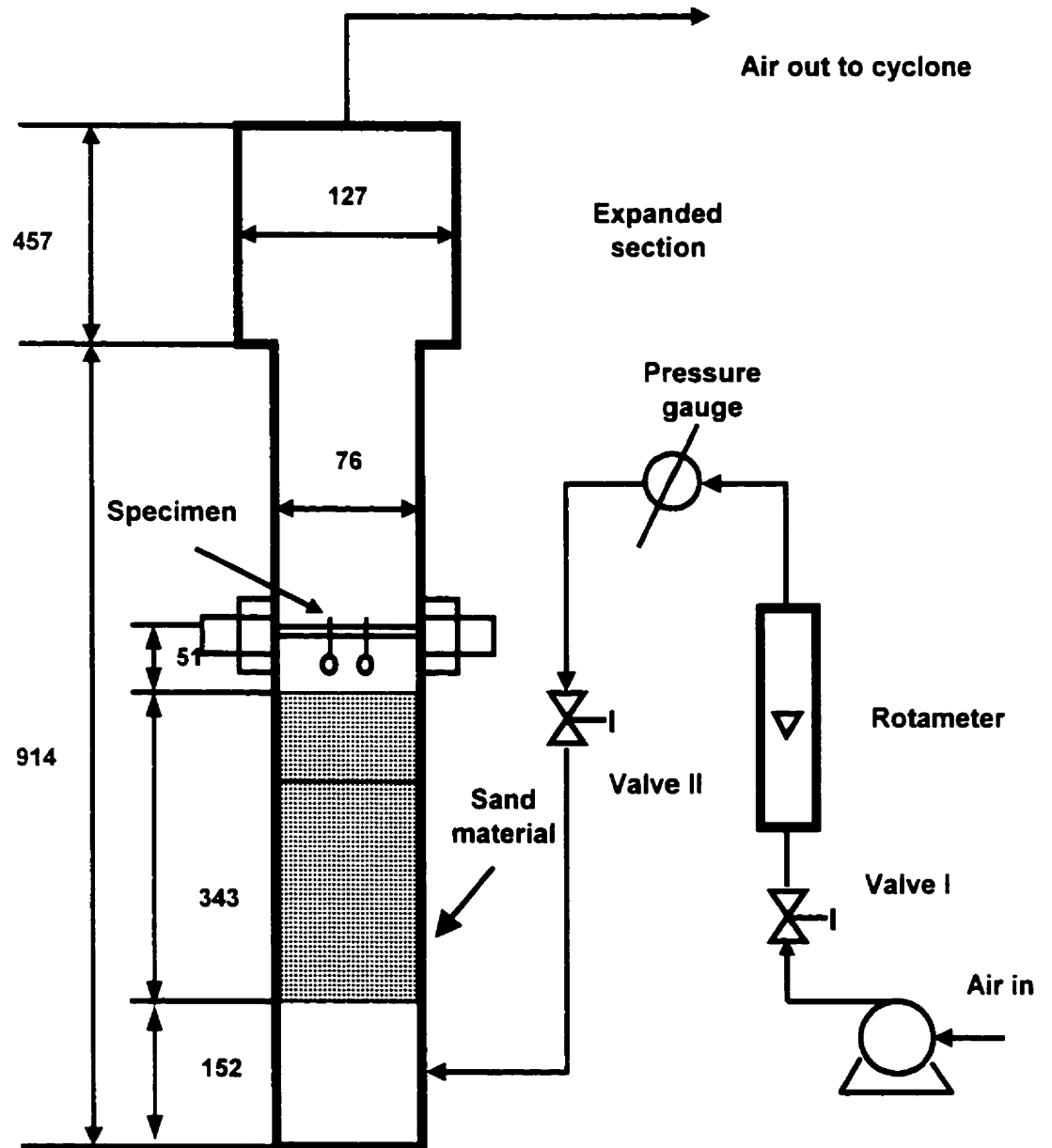
Figure 5.3 Experiment set-up (units in mm)

The three-dimensional fluidization column itself has a diameter of 76 mm and a height of 0.91m. A stainless steal beam was fixed horizontally crossing the axis of the column at 0.39 m above the air distributor. Test specimens can be fixed below the beam to face the upflowing particles brought up by slugs. Two specimens can be tested each time, placed symmetrically near the center of the column and 15 mm apart from each other. Two mounting ports were installed at the two ends of the beam to allow the quick change of the specimens. The packed bed height was kept constant at 0.343 m for all tests to ensure comparability. Due to bed expansion, the specimens were covered by the particles when fluidized.

The column was constructed entirely from plexiglas with a thickness of 6.35 mm to achieve wall rigidity and resistance to wear. On the top of the column, there is an expansion section with a diameter of 127 mm and a height of 0.46 m. This expansion section is designed for slowing down the particle velocity through increased cross-section area, so that the particle entrainment can be efficiently reduced within a relatively short column length. The air distributor was a multi-orifice plate with an opening area of 1.5%. The orifice plate was covered with a fine steel wire screen to prevent solid particles from dropping through the holes. Air was from the building compressor which has a gauge pressure of 180 kPa and can provide a maximum superficial velocity of 3.0 m/s in the fluidization column. The air flowrate was controlled by adjusting the two valves: Valve-I and Valve-II and monitored by a rotameter. The pressure at the pressure gauge was kept at 70 kPa constantly.

5.2.5. Abrasive materials

The wear was produced by the silica sand which is traveled under the air. The property and the diameter of the sand are listed in the Table 5.2.

Table 5.2 Particle properties

Particle Name	Mean Diameter (μm)	Density (Kg/m^3)	Spherity	Minimum Fluidization Velocity (m/s)	Terminal Velocity (m/s)	Hardness
Silica Sand	400	2530	0.86	0.1199	2.97	350

5.3. Results and Discussion

5.3.1. Single wear test

Before testing large amount of specimens. wear test on each material was undertaken to observe the length of the incubation period effect. Two specimens of each type of dental material were suspended in the column in pair. After a certain time interval. specimens were taken out and weighed after a 5 minutes wash in the ultrasonic cleaner. How the cumulated weight loss of the materials varied with the time is drawn in the Figure 5.4. There are two groups of data that present two types of materials in this figure: Amelogen and Z-250. Each type of material has two samples tested in the experiment. The weight loss vs. time curves for both materials follow a similar trend. They have a relative higher slope in the first 50 hours and constant and same slope

afterwards. This shows the existence of an incubation period. For all the dental materials tested in this project. the incubation period was around 50-60 hours. So, all further tests were simplified by starting the regular measurements after 60 hours of wear in the column. In each test, the specimens were first worn in the column for 60 hours, then regular weight measurements were take at each 40-50 hours interval. The weight difference over the time consumed in the column is the wear rate. However, weight losses for the first 20-25 hours were also recorded separately to study the initial wear rate of each materials.
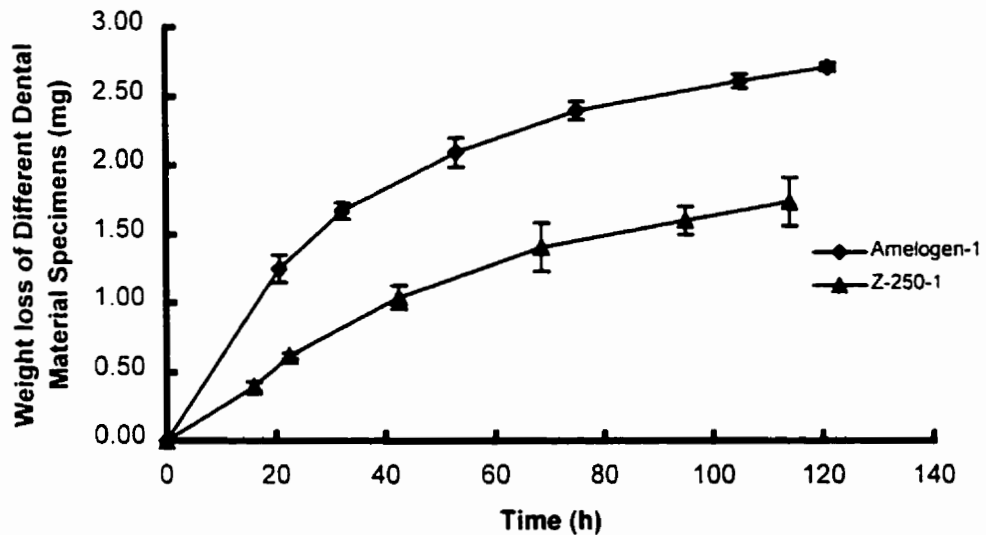

Figure 5.4 Weight loss of two different dental materials vs. time under 0.772 m/s slug velocity with 400 μm wear particles

This incubation phenomenon can also be found in the *in vivo* test. Most studies do show that loss of material due to wear is greatest in the first 24 months after placement (Lutz 1984. Braem 1986. Goldberg 1984, Boksman 1986. Leinfelder 1986. Lugassy

1988. Burgoyne 1991). In Goldberg's report (1984), the similar trend was found as in Figure 5.4. The reason for this is not so clear. It may be due to initial formation of some surface structure. Alternatively, it may be due to the rough initial surface condition of the samples, leading to bigger apparent wear rates determined from weight losses. The mechanism in the mouth is even more complicated.

5.3.2. Wear rates comparison

Dental composites are classified on the basis of the filler particle size and distribution of the inorganic filler. The earlier composites contained filler particles having diameters of 30-50 µm; these were followed by composites having filler particles with diameters of 0.04 µm. Currently most composites have fillers with average diameters of 1-3 µm (fine composites), or 0.04 µm (microfilled composites), or mixtures of particles with diameters of 1 to 3 µm and 0.04 µm (hybrids). The volume percentage of fillers is lower than the weight percentage because of the higher density of the fillers compared with the polymer matrix.

For the composite materials, material properties relate to the filler content, particle size, and distribution (Johnson et al. 1993). Figure 5.5 showed that Z-100, Vitalescence, and Permaflo have almost the same level of wear resistance as the natural tooth. Vitremer, a glass ionomer cement, showed the least wear resistance. Compomer F-2000, Amalogen, and Z-250 have the wear resistance in between. At the present time, the best wear characteristics for posterior use are generally exhibited by heavily filled

composite (more than 60% by volume) with a mean filler particle size between 1 and 3 μm (Lutz *et al.* 1984, Willems *et al.* 1993). Clinical studies have shown that the more heavily filled hybrid resin composites are more resistant to wear than are the microfilled materials (Lutz *et al.* 1984). In this project, the dental materials used almost all have the filler content more than 60% by volume. Z-100 is the one that has the highest filler content (66% by volume) and it is a hybrid resin as well. The wear test using the fluidized bed showed that it also has the best quality in wear resistance.

The wear rate varies with filler particle size and the resin type as well (Dickinson *et al.* 1993, Gerbo *et al.* 1990, Mitchem *et al.* 1982, Wendt *et al.* 1994, Willems *et al.* 1993). For the same kind of resin (Bis-GMA), Vitalescence (1.0 μm average filler particle size) showed lower wear rate than Amelogen (0.7 μm average filler particle size). It appears that the smaller the mean filler particle size the higher the wear resistance for hybrid composite materials. On the other hand, for the same filler particle size, Permaflo (methacrylate monomer *et al.*) has more wear resistance than Amelogen (Bis-GMA) because of different resin matrix.
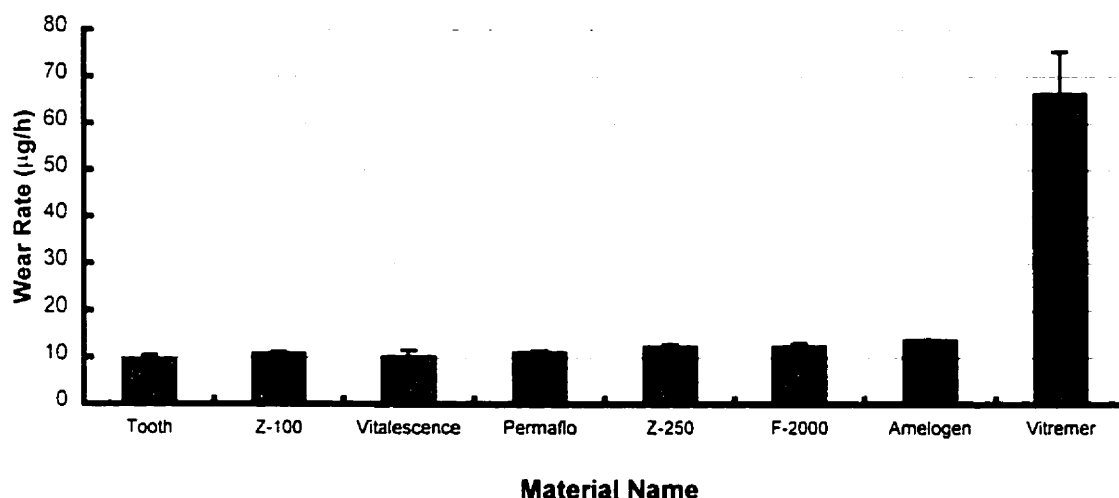
Figure 5.5 Wear rates comparison of different dental materials under 0.772 m/s slug velocity with 400 μm wear particles

Although the hardness of Vitremer is similar to the other materials, the wear rate is exceptionally high. This can be explained by the material's constituents. Vitremer is a glass ionomer cement and tend to be brittle while other materials are resin based matrix. This brittleness of Vitremer certainly contribute to wear due to chips formation and fracture. The five fold increase of wear rate results from different wear mechanisms. Given the change in wear mechanism, hardness along can not provide a good indication to the wear rate.

5.3.3. Initial wear rate

The initial wear rates of different dental materials was given in Figure 5.6, in comparison with the wear rate of a natural tooth. As mentioned before, the initial wear of

the material is relatively higher than the wear rate after the incubation period. This high initial wear of the dental restorative material compared with the relatively low wear of enamel may create undesirable clinical conditions. such as loss of occlusal vertical dimension (Burgoyne 1991). Thus. it is useful to compare the initial wear rate of different restorative materials. In Figure 5.6. test results for four dental materials have been shown. As can be seen from Figure 5.5. these four materials have almost the same wear rate as the tooth after the incubation period. Figure 5.6. however. shows that they all have significant higher wear rate than the tooth. and the Permaflo has the highest initial wear rate which is over ten times the wear of the tooth. Different initial wear resistance maybe contributes to the size and content of the filler particles. The higher the filler particles. the higher the initial wear resistance. and the bigger the filler particle size. the higher the wear resistance. For example. Vitalescence has the highest filler content (75% by weight). and the highest filler particle size (1 μm). so it has the lowest initial wear rate. On the opposite. Permaflo. which has the lowest filler content (68% by weigh) and particle size (0.7 μm) has the highest initial wear rate.
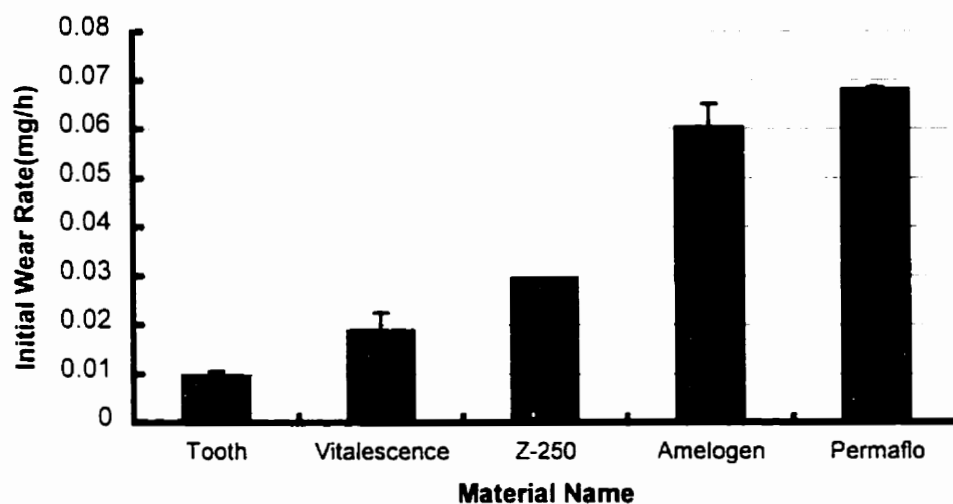
Figure 5.6 Initial wear rate of dental materials compared with tooth under 0.772 m/s slug velocity with 400 μm wear particles

5.3.4. Wear rate of the materials and the hardness

There are many material properties that may affect the wear rate. The most commonly quoted mechanical property is hardness. For each material tested. the Vickers hardness was measured. Figure 5.7 showed the results of these measurements. Tooth has the highest hardness and the Permaflo has the lowest. The hardness was also measured after the tests and it was found that the hardness of all the materials did not change appreciably due to wear.
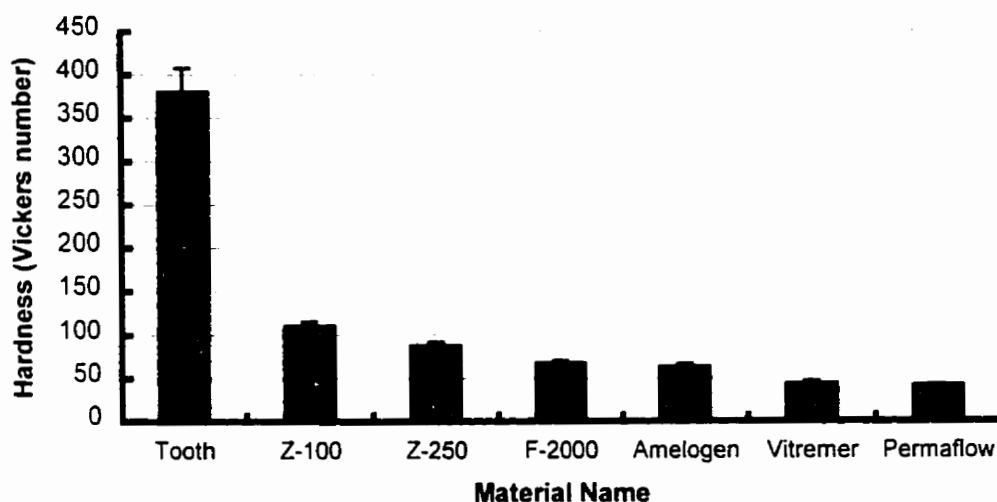
Figure 5.7 Hardness of different dental restorative materials

The average initial wear rates at the base operating conditions are plotted in Figure 5.8 against the hardness of four different materials and natural tooth. Wear appears to generally decrease with increasing hardness. However, when plot the wear against hardness for steady wear (after 60 hours) as seen in Figure 5.9, the trend is unclear, especially for the material Permaflo and the natural tooth. Permaflo has the lowest hardness but experienced high wear resistance, and the tooth which has the highest hardness but showed proximately the same wear resistance as Z-100. The above findings suggest that the wear of dental materials have two wear regimes: initial (before 24 hours) and steady wear (after 60 hours). In initial wear stage, the wear resistance of the materials decreases with decreasing hardness. In steady stage, material hardness may not be a major factor influencing wear rate. This conclusion confirmed the finding of Ramp *et al.* (1997) that surface hardness has been shown to be a poor indicator of wear rate in steady stage.
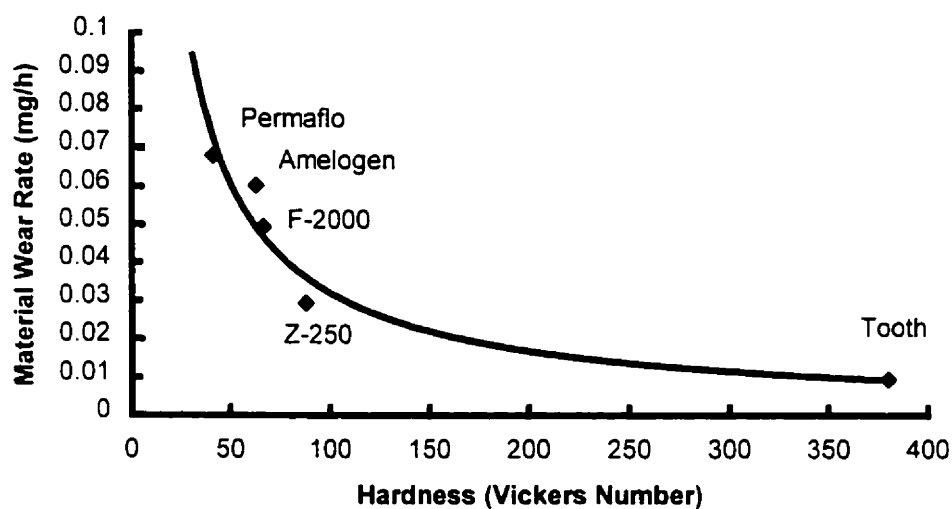
Figure 5.8 Initial wear rate of dental materials under 0.772 m/s slug velocity with 400 μm wear particles vs. their hardness
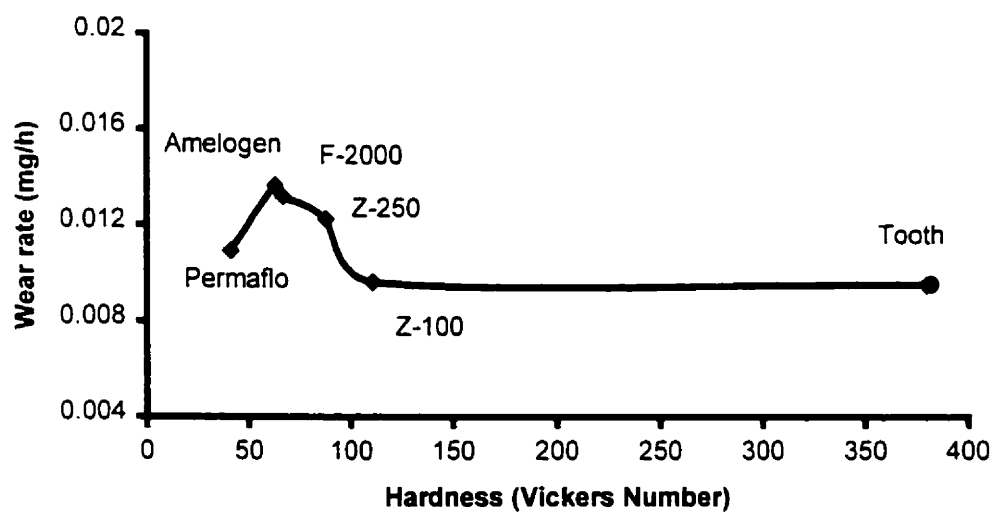
Figure 5.9 Wear rate of dental materials under 0.772 m/s slug velocity with 400 μm wear particles vs. their hardness

5.4. Conclusions

1. Fluidized bed is a simple and effective test apparatus to study the wear resistance of various dental restorative materials.

2. The most wear resistant dental material from the seven dental materials tested is the material Z-100, and the lest wear resistant one is the material Vitremer.

3. Z-100, Vitalescence and Permaflo have almost the same wear resistance as tooth, but Permaflo has a relatively high initial wear rate.

4. The hardness does not proved to be a good indicator of the wear rate for the dental materials.

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CHAPTER 6: CONCLUSIONS AND RECOMMENDATIONS

6.1 General Conclusions

Several major conclusions can be drawn based on this study:

1. The fluidized bed proved to be a simple and reliable tool to test wear resistant of different dental restorative materials.

2. There exists an incubation period during the beginning of the wear test due to the surface texture or the different initial wear mechanism.

3. The wear rate of the dental materials is proportional to the superficial gas velocity, the abrasive particle size and sharpness in this operating system. It presents that the wear rate of the dental materials is proportional to the biting force, the size and sharpness of the food particles.

4. The material properties such as filler content, filler particle size, and resin type of the composite materials change the wear rate.

5. The hardness related very well in the initial wear process, but does not proved to be a good indicator in the steady wear stage for the dental materials.

6. The most wear resistant dental material from the testing dental materials is the material Z-100, and the lest wear resistant one is the material Vitremer.

7. Z-100, Vitalescence and Permaflo have almost the same wear resistance as tooth, but Permaflo has a relatively high initial wear rate.

6.2 Recommendations

1. Further tests with more types of erosive particles and dental materials should be performed in the gas-solid fluidized bed.

2. The influence of finishing conditions on the wear rate of the dental restorative materials should be further investigated.

3. The wear rate of the dental material obtained by profilometer or graphic measurement should be used in the further study in order to give dimensional results.

4. Influence of water sorption by the natural tooth should be further tested during the tooth wear test.

5. Further test can consider testing the fillings in the tooth or a sample composed of a combination of different materials.

APPENDIX A

A.1 Incubation period for F-2000

1. Material: F-2000

2. Superficial air velocity: 0.464 m/s

3. Particle size: 400 μm

Time (h)	Weight (g)		Normalized Weight loss (mg)	
	#01	#02	#01	#02
0.00	1.91590	1.86524	0.00000	0.00000
8.00	1.91547	1.86476	0.43000	0.48000
25.25	1.91474	1.86424	1.16000	1.00000
49.25	1.91423	1.86380	1.67000	1.44000
55.75	1.91376	1.86337	2.14000	1.87000
72.00	1.91315	1.86294	2.75000	2.30000
110.00	1.91233	1.86227	3.57000	2.97000

Time (h)	Weight (g)		Normalized Weight loss (mg)	
	#03	#04	#03	#04
0.00	1.92365	1.80132	0.00000	0.00000
6.00	1.92319	1.80121	0.46000	0.11000
23.00	1.92236	1.80035	1.29000	0.97000
46.00	1.92187	1.79984	1.78000	1.48000
52.00	1.92163	1.79957	2.02000	1.75000
68.00	1.92130	1.79919	2.35000	2.13000
74.00	1.92099	1.79905	2.66000	2.27000
94.75	1.92056	1.79867	3.09000	2.65000
131.75	1.91979	1.79794	3.86000	3.38000

Time(h)	Weight (g) #05	Normalized Weight loss #05(mg)
0.00	1.79851	0.00000
24.00	1.79720	1.31000
70.50	1.79652	1.99000
95.00	1.79593	2.58000
141.00	1.79484	3.67000

A.2 Average wear rate of F-2000 vs. Time under 0.464 m/s superficial air velocity using 400 μm particles

Time(hr)	Average Wear Rate (μg/h)	Standard deviation
0	5.3E+01	2.1E+01
5	5.3E+01	2.1E+01
10	4.5E+01	9.6E+00
15	4.5E+01	9.6E+00
20	4.5E+01	9.6E+00
25	2.6E+01	1.1E+01
30	2.0E+01	3.1E+00
35	2.0E+01	3.1E+00
40	2.0E+01	3.1E+00
45	2.0E+01	3.1E+00
50	2.5E+01	6.5E+00
55	2.5E+01	6.5E+00
60	2.5E+01	6.5E+00
65	2.5E+01	6.5E+00
70	2.4E+01	6.6E+00
75	2.6E+01	4.4E+00
80	2.6E+01	4.4E+00
85	2.6E+01	4.4E+00
90	2.6E+01	4.4E+00
95	2.6E+01	4.4E+00
100	2.4E+01	4.5E+00
105	2.4E+01	4.5E+00
110	2.4E+01	4.5E+00

A.3 Wear rates of material F-2000 vs. different slug velocities with 400 μm particles

Air Velocity(m/s)	Wear Rate(g/hr)	Stdev
0.558	10.0642	0.38572
0.772	13.1878	1.0614
0.958	14.2078	0.33488
1.108	14.1751	0.22256

A.4 Wear rates of material F-2000 vs. different slug velocities with 700 μm particles

Velocity	Average wear rate (μg/h)	Stdev
0.558	11.3725	0.5361
0.772	19.65	0.70391
0.958	25.0139	0.37237
1.108	22.9683	1.19058

A.5 Normalized wear rate vs. slug velocity for F-2000 using 400 µm particles

Slug Velocity (m/s)	Air Velocity (m/s)	Average density (Kg/m³)	wear Rate (µg/hr)	Normalized wear rate (µg/hr)
0.558	0.25	0.40561	10.0642	6.73386
0.772	0.464	0.27139	12.3129	12.3129
0.958	0.65	0.21077	14.2078	18.2941
1.108	0.8	0.1786	14.1751	21.5399

A.6 Normalized wear rate vs. slug velocity for F-2000 using 700 µm particles

Slug Velocity (m/s)	Air Velocity (m/s)	Average density (Kg/m³)	wear Rate (µg/hr)	Normalized wear rate (µg/hr)
0.558	0.442	0.28095	11.3725	8.47076
0.772	0.656	0.20927	19.65	19.65
0.958	0.842	0.17128	25.0139	30.5612
1.108	0.992	0.14941	22.9683	32.1698

A.7 Wear rate of material F-2000 vs. different wear particle size under 0.772 m/s slug velocity

Material Number	Wear Rate (ug/h)	Stdev
200	8.60584	0.93619
400	13.1878	1.0614
700	19.65	0.70391

A.8 Weight of two different dental materials vs. time under 0.772 m/s slug velocity with 400 μm wear particles

| Amelogen | 1 | 2 | Z-250 | 1 | 2 |
Time(h)	Wear rate(mg/h)	Wear rate(mg/h)	Time(h)	Wear rate(mg/h)	Wear rate(mg/h)
0.00	0.00	0.00	0	0	0
20.75	1.32	1.18	16	0.37	0.42
32.25	1.71	1.63	22.5	0.6	0.63
53.00	2.17	2.02	42.5	0.98	1.1
75.00	2.44	2.35	68.5	1.28	1.53
121.00	2.73	2.69	114	1.61	1.86

A.9 Wear rates comparison of different dental materials under 0.772 m/s slug velocity with 400 μm wear particles

Material Name	Wear Rate(mg/h)	Standard deviation
Tooth	9.49627	0.94963
Z-100	10.7503	0.32347
Vitalescence	10.0406	1.52741
Permaflo	10.9825	0.41651
Z-250	12.2463	0.49601
F-2000	12.3129	0.62468
Amelogen	13.6554	0.10388
Vitremer	66.3141	9.09457

A.10 Initial wear rate of dental materials compared with tooth under 0.772 m/s slug velocity with 400 μm wear particles

Material Name	Wear Rate(mg/h)	Standard deviation
Tooth	0.0095	0.00095
Vitalescence	0.01885	0.00362
Z-250	0.02927	1.7E-05
Amelogen	0.06024	0.00477
Permaflo	0.06798	0.00043

A.11 Hardness of different dental restorative materials

Material Name	Hardness(vicker number)	Standard deviation
Tooth	380.00	28.28
Z-100	110.50	4.81
Z-250	87.71	4.21
F-2000	66.69	3.47
Amelogen	62.82	2.76
Vitremer	43.25	2.21
Permaflow	41.25	0.58

APPENDIX B

B.1 F-test for Figure 4.5a: Wear rates of material F-2000 vs. different slug velocities with 400 mm particles

Air Velocity(m/s)	Erosion Rate(mg/hr)	Standard Deviation	Sample Size
0.558	10.06415621	0.385718438	5
0.772	13.18776151	1.061399823	5
0.958	14.207806	0.334881199	5
1.108	14.17514936	0.222557808	5

Grand mean

12.90871827

Between groups mean square

19.1014585

Within groups mean square

0.359256423

F-test

53.1694279

B.2 F-test for Figure 4.5b: Wear rates of material F-2000 vs. different slug velocities with 700 mm particles

Velocity	Average wear rate(mg/h)	Standard Deviation	Sample Size
0.558	11.3725	0.536104791	5
0.772	19.64997106	0.703911662	5
0.958	25.01391304	0.37236858	5
1.108	22.96830208	1.190576311	5

Grand mean

19.75117155

Between groups mean square

180.431259

Within groups mean square

0.584757572

F-test

308.5573709

B.3 F-test for Figure 4.7: Wear rate of material F-2000 vs. different wear particle size under 0.772 m/s slug velocity

Material Number	Erosion Rate (ug/h)	Standard Deviation	Sample Size
200	8.605838146	0.936191771	5
400	13.18776151	1.061399823	5
700	19.64997106	3.940203756	5

Grand mean

13.81452357

Between groups mean square

153.939205

Within groups mean square

5.842743419

F-test

26.34707601

B.4 F-test for Figure 4.8: Wear rate of different dental materials under 0.772 slug velocity with 400 mm wear particles

Material Name	Erosion Rate(mg/hr)	Stdev	
Z-100	10.75034868	0.323474232	5
F-2000	13.18776151	1.061399823	5
Amalgam	30.70340649	8.824461794	5
Glass-ionomer cement	66.31406904	9.094565322	5

Grand mean

30.23889643

Between groups mean square

3286.96424

Within groups mean square

40.45336238

F-test

81.25317766

B.5 F-test for Figure 5.4: Wear rates comparison of different dental materials under 0.772 m/s slug velocity with 400 mm wear particles

Material Name	Wear Rate(mg/h)	Standard deviation	Sample Size
Tooth	9.496266241	0.949626624	2
Z-100	10.75034868	0.323474232	5
Vitalescence	10.04057018	1.527412674	5
Permaflo	10.98253968	0.416509362	5
Z-250	12.246337	0.496010801	5
F-2000	12.31292826	0.624682097	5
Amelogen	13.65537248	0.1038823	5
Vitremer	66.31406904	9.094565322	5

Grand mean

18.93252322

Between groups mean square

1861.583967

Within groups mean square

11.8889265

F-test

156.5813336

B.6 F-test for Figure 5.5: Initial wear rate of dental materials compared with tooth under

0.772 m/s slug velocity with 400 mm wear particles

Material Name	Wear Rate(mg/h)	Standard deviation	Sample Size
Tooth	0.009496266	0.000949627	5
Vitalescence	0.018851521	0.003622815	5
Z-250	0.029266827	1.69978E-05	5
Amelogen	0.060240964	0.004770841	5
Permaflo	0.06798048	0.000431077	5

Grand mean

0.037167212

Between groups mean square

0.003306772

Within groups mean square

7.39472E-06

F-test

447.1798508

B.7 F-test for Figure 5.6 Hardness of different dental restorative materials

Material Name	Wear Rate(mg/h)	Standard deviation	Sample Size
Tooth	380.00	28.28	2
Z-100	110.50	4.81	5
Z-250	87.71	4.21	5
F-2000	66.69	3.47	5
Amelogen	62.82	2.76	5
Vitremer	43.25	2.21	5
Permaflow	41.25	0.58	5

Grand mean

88.15920759

Between groups mean square

33240.06666

Within groups mean square

42.52852215

F-test

781.59468