

**COMPARISON OF THE FRACTURE TOUGHNESS, FLEXURAL  
STRENGTH, AND FLEXURAL MODULUS OF NINE  
RESTORATIVE MATERIALS OVER FIVE TIME INTERVALS**

**By**

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Title of thesis: COMPARISON OF THE FRACTURE TOUGHNESS, FLEXURAL STRENGTH,  
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## Abstract

The aims of this study were: 1) to determine the effect of time on the fracture toughness ( $K_{IC}$ ), flexural strength ( $\sigma_f$ ), and flexural modulus ( $E_f$ ) of six groups of direct restorative materials including amalgam; and 2) to compare the selected properties of the materials at each time. The null hypotheses tested were: 1) there is no difference between the 1-hour, 1-day, 7-day, 30-day, and 90-day values of each of the  $K_{IC}$ ,  $\sigma_f$ , and  $E_f$  for each of the selected materials, and 2) there is no difference between the selected materials with respect to each of their mechanical properties tested at each time. The materials selected for this study were KetacFil (KF; ESPE) – a conventional glass-ionomer cement (GIC); PhotacFil (PF; ESPE) – a resin-modified glass ionomer cement (RMGIC); Dyract (Dentsply) and F2000 (3M) – polyacid-modified composite resins (PAMCR) or "compomers"; Z100 (3M) – a hybrid composite resin; Heliomolar (H; Vivadent) – a microfil composite resin; SureFil (SF; Dentsply) and Prodigy Condensable (PC; Kerr) "packable" composite resins; and Valiant PHD XT (V; Ivoclar) – a phase dispersed unicompositional amalgam. Twelve notchless triangular prisms (NTP) (6x6x6x12 mm) and twelve rectangular bars (2x2x25 mm) were made for each test at each time following manufacturers' instructions, and stored in distilled water at 37° C for the duration of the five time intervals before testing. For the  $K_{IC}$  test, the samples were placed in a specimen holder and loaded in tension at a crosshead speed of 0.1 mm/min. The maximum load recorded before crack arrest or complete failure was used to calculate  $K_{IC}$  in MPa•m<sup>1/2</sup>. The bars were loaded in a three point bending mode and the maximum load recorded at failure was used to calculate  $\sigma_f$ . The modulus was determined from the straight portion of the stress/strain curve. The results were analyzed using a one way ANOVA followed by Scheffe multiple means comparisons ( $\alpha=0.05$ ). Based on the limitations of this study and representative materials the results indicate that GICs are the weakest materials and are inferior to RMGIC especially in the short term. RMGICs and PAMCRs are comparable, while both are inferior to CRs and PCR. PCR do not show any significant improvement over conventional CRs. Amalgam has the best intrinsic properties over time. The intrinsic properties,  $K_{IC}$  and  $E_f$ , appear to correlate better with the clinical success of materials than their  $\sigma_f$  does.

Keywords: Fracture toughness, flexural strength, flexural modulus, dental material

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## Chapter I. Introduction

The quest for an ideal dental restorative material has become the greatest challenge for the science of dental materials. An ideal dental restorative material should bond permanently to the tooth structure, aesthetically match tooth structure, exhibit properties similar to those of enamel and dentin, and initiate tissue repair. The adverse conditions of the oral environment, however, pose a challenge to the concept of a “*universal restorative material*”.

The restoration of primary dentition compared to that of permanent dentition poses an even greater challenge in that its structure is weaker, the clinical crowns are much smaller than that of their permanent successors, the coronal pulp occupies a much larger volume of the clinical crown, and due to the differences in their enamel and dentin structure, bonding to primary teeth is not as effective as to permanent teeth [Croll 1990; Pinkham 1999]. Providing the most conservative restoration, therefore, becomes essential in the long-term function and maintenance of the primary dentition. In addition, conservative restorations may allow for the completion of restorative treatment with minimal or no need for local anaesthesia. In certain clinical scenarios, patient's developmental disability may not allow completion of definitive restorative treatment. The material of choice in these situations is one that is capable of bonding to the tooth structure under salivary contamination, exhibiting physical and mechanical properties similar to those of enamel and dentin, and acting as a pulpal sedative [Croll 1990].

The conventional approach to restoring primary teeth has been via the use of silver amalgam alloy and stainless steel crowns [Qvist et al. 1997]. The teachings of Black in the design of cavity preparations for the placement of amalgam alloys continue to persist and are widely accepted. Due to the concept of ‘extension for prevention’ in the placement of amalgam alloys and recent controversy with regard to their possible toxicity, concomitant with improvements in materials that are capable of bonding to tooth structure, the continued use of silver amalgam alloy has been challenged [Croll 1990; Lloyd et al. 1997; Manhart et al. 2000b; Price 2003]. Several groups of materials have been advocated as alternatives to amalgam for use in the restoration of posterior primary teeth, ranging from glass-ionomer cements to composite resin restorative materials.

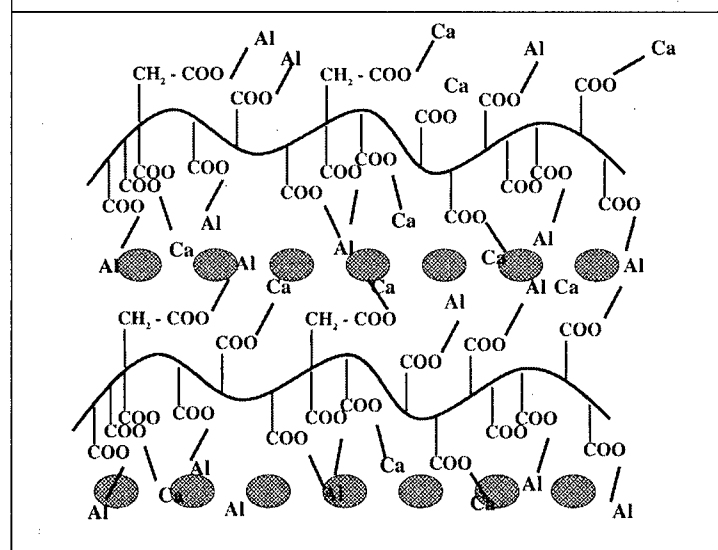
### 1.1 Glass-Ionomer Cements

Glass-ionomer cements (GIC) have been the focus of a considerable amount of research since their introduction in the early 1970's. By combining the biocompatibility and adhesion characteristics of a polyacrylic acid liquid with the strength, rigidity, and fluoride release properties of a silicate glass powder, Wilson and Kent developed the glass-ionomer cements

[Wilson and Kent 1972]. GIC have a range of applicability in primary dentition. They are used as luting cements for cementing stainless-steel crowns, and also as restorative materials for conservative Class I and II preparations [Croll 1990]. In uncooperative children with exposed caries, GIC serve as temporary means of controlling the caries process and restoring function [Croll 1990].

GIC set via an acid base reaction, in which an alkenoic acid polymer forms the acidic component, with a ratio of 1.5:2 between the carboxylic groups and the backbone carbon atoms, while the base is an ion-leachable fluoroaluminosilicate glass (Figure 2) [Ruse 1999]. GIC are composed of an interpenetrating network of inorganic and organic components in a hydrated silicon matrix, with glass particles that are sheathed with a thin layer of silicon gel on their surface [Wasson and Nicholson 1993]. In essence, set GIC are composed of unreacted glass particles as fillers in a matrix of calcium aluminum cross-linked polyalkenoic chains (Figure 1) [Ruse 1999].

Figure 1: Schematic Representation of Set GIC (Ruse, 1999)



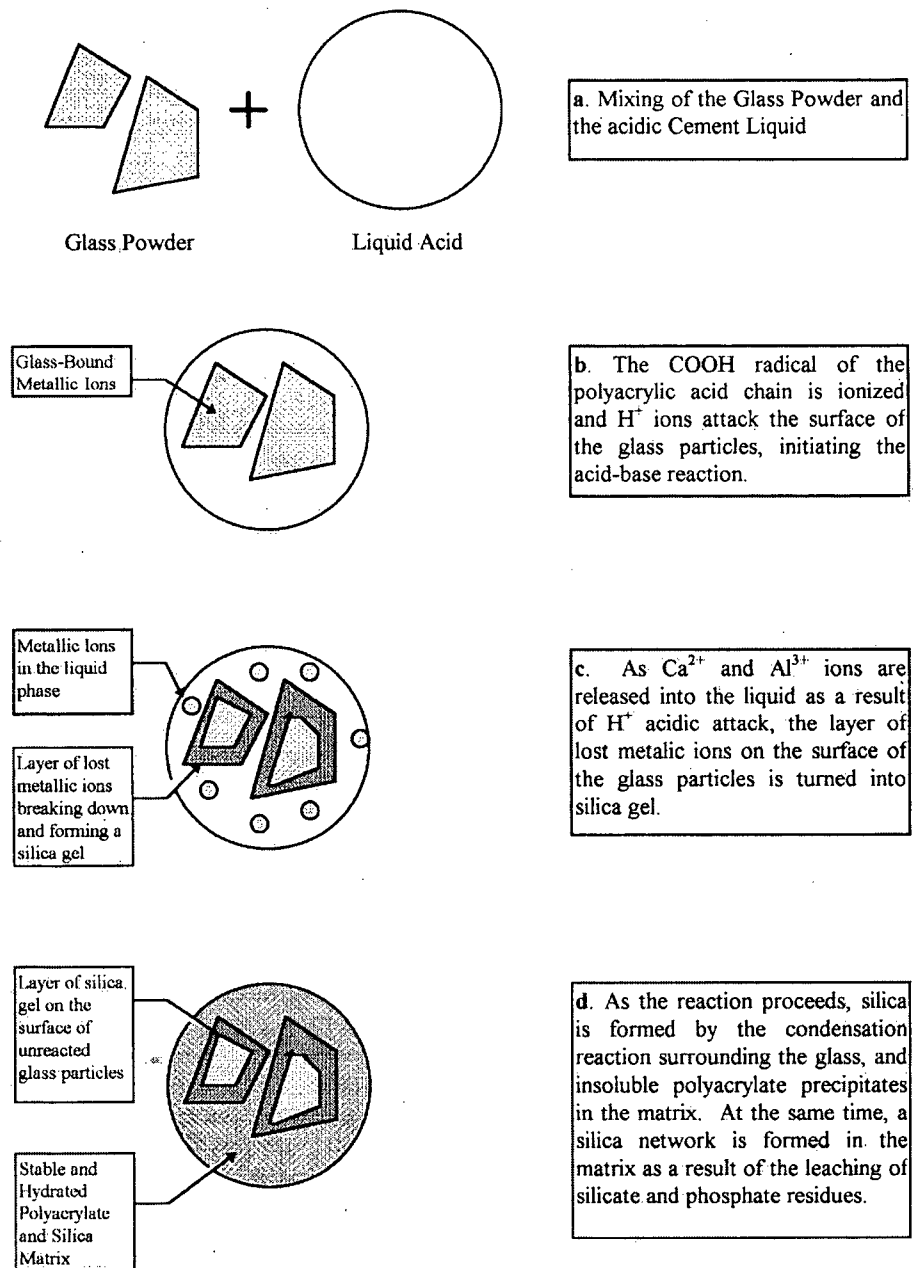
It has been claimed that this group of materials possesses excellent biocompatibility, moderate adhesion to tooth structure, and a clinically significant fluoride release [Wilson and McLean 1988]. The short working time and long setting of the conventional glass-ionomer cements, however, made their clinical application as restorative and aesthetic materials difficult. The properties of the newly placed cement were less than desirable to withstand the hostile environment of the oral cavity. In addition, the mechanical properties of GIC appear to be the weak link in their use as restorative materials in stress-bearing areas [Wilson and McLean 1988]. A cement with better handling properties was desired.



Figure 2: Acid-Base Reaction in Setting of Glass-Ionomer Cements

**Diagrammatic representation of the coagulation and hardening processes involved in the setting reaction of glass-ionomer cements.**

(Katsuyama et al., 1993; Nicholson, 1996)



## 1.2 Resin-Modified Glass-Ionomer Cements

One modification to GIC, was the development of dual-cure GIC, better known as resin-modified glass-ionomer cements (RMGIC). RMGIC are formed by a combination of the conventional acid-base components and the addition of resin monomers such as Bis-GMA and hydroxyethyl methacrylate (HEMA) at different ratios. In place of some of the water, HEMA is incorporated into the RMGIC composition [Antonucci 1988]. This provides for second setting mechanism via light-activated addition polymerization (Figure 3) [Mitra 1991].

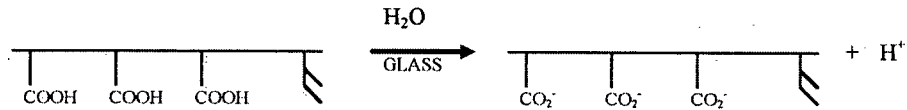
A typical RMGIC, therefore, is composed of (1) a polyacrylic acid or a modified polyacrylic acid with a photo-curable side chain of HEMA grafted onto the polymer backbone, (2) photo-curable monomers such as HEMA or Bis-GMA, (3) an ion-leachable glass, and (4) water [Mathis and Ferracane 1989; Nicholson et al. 1992; Wilson 1990]. RMGIC still possess the beneficial properties of GICs, i.e. adhesion to dental tissue and release of fluoride, while improving handling, setting characteristics, and mechanical properties [Mount 1994].

Figure 3: Setting of Resin-Modified Glass-Ionomer Cements

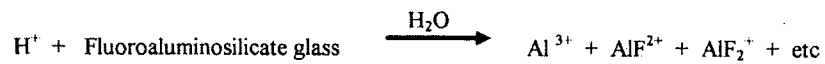
### Steps in the setting reaction of Vitrabond, a RMGIC

(Mitra, 1991-a)

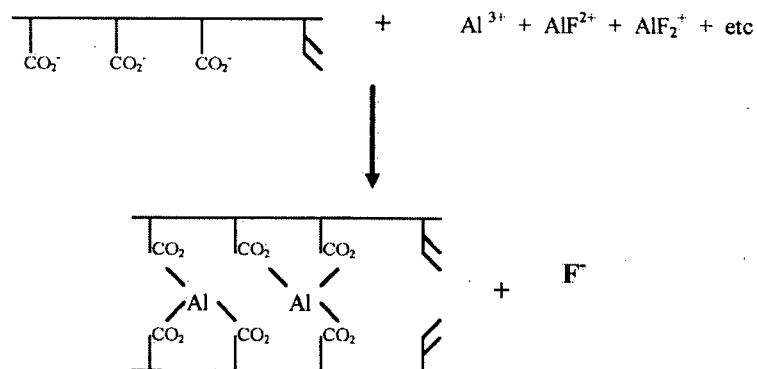
**Step 1:** Ionization of polycarboxylic acid by water in the presence of the fluoroaluminosilicate glass



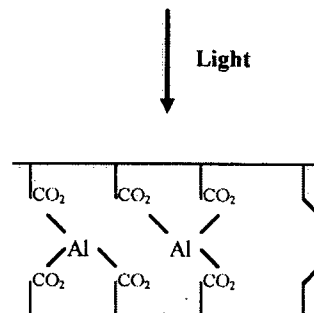
**Step 2:** Release of positive ions as a result of acidic reaction between the liberated protons and the basic glass



**Step 3:** Formation of an ionically cross-linked network between the metal ions and the polycarboxylate polymer



**Step 4:** Visible light polymerization through the pendent methacryloxy groups of the polycarboxylate molecular chains



### 1.3 Polyacid-Modified Composite Resins

It is important to differentiate between true RMGIC and cements which are marketed as such but do not demonstrate the acid-base setting reaction. This latter group is marketed under the name “compomers” or “resinomers”. Compomers have fillers similar to the ion-leachable glass in GICs, and a matrix comprised of dimethacrylate monomers with two carboxylic groups present in their structure [Ruse 1999]. The ratio of carboxylic groups to backbone carbon atoms is approximately 1:8, hence reducing matrix-filler bonding during their free-radical polymerization reaction [Ruse 1999].

Compomers show minimal if any acid-base reaction, and their chemistry is almost identical to that of composite resins [Guggenberger et al. 1998]. All of these products are one component, light-curing materials, with glass particles as fillers, and at least two different resins for the matrix, including a light-curable monomer like UDMA or Bis-GMA.


Many researchers, pioneers in the field of GIC, have urged the scientific community to adopt a more concise generic name in referring to these materials [McLean et al. 1994; Mount 1994]. The term “GIC” has been suggested for cements that consist of a basic glass and an acidic polymer that set only by an acid-base reaction between these components. The term RMGIC should be used to refer only to that group of hybrid cements that set by both an acid-base reaction and a photochemical polymerization [Antonucci 1988]. Thus, RMGIC have sufficient acid and base to allow setting within a reasonable time, even if kept in the dark.

The photopolymerizable group of restorative materials that have the correct ingredients of acid-decomposable glass and some polymeric acid, but in insufficient amounts to promote cure in the dark, should be referred to as polyacid-modified composite resins (PAMCR) rather than the terms “compomer” or “resinomer”. PAMCR, therefore, are not true GIC [McLean et al. 1994]. In consideration of the low volume fraction filler and the incomplete silanization of the filler, one may refer to PAMCR as weaker composites [Ruse 1999]. This would negate placing GIC, RMGIC, PAMCR, and Composite Resins on the same “continuum” as suggested by some clinicians (Figure 4).

**CONTINUUM OF TOOTH-COLOURED RESTORATIVE MATERIALS**

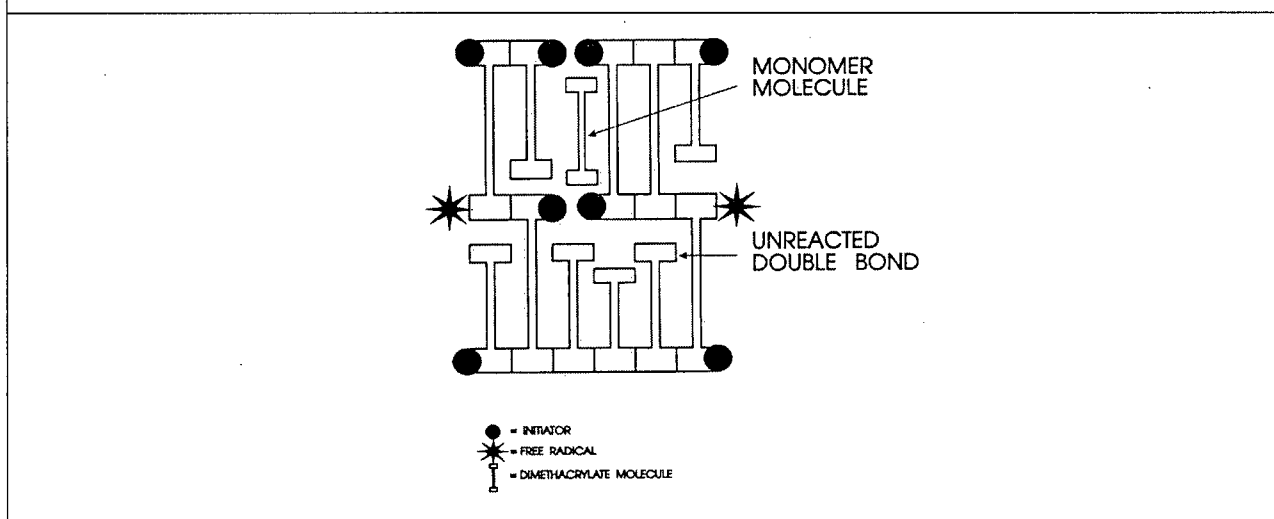
A continuum of materials between conventional glass-ionomer cements and composite resins could be defined with glass-ionomer cements at one end, followed by resin-modified glass-ionomer cements and polyacid-modified composite resins, and the true composite resins at the other end ( McLean et al., 1994).

**GIC                      RMGIC                      PAMCR                      CR**

A horizontal double-headed arrow is positioned below the material names, spanning the width of the continuum from GIC to CR.

Composite resins (CR) are heterogeneous materials comprised of an organic matrix and inorganic filler. The early formulations of CR were very weak due to the lack of a strong bond between the fillers and the surrounding matrix. Bowen's resin, namely bis-phenol A-glycidyl methacrylate (Bis-GMA) revolutionized the science of composite resins by enhancing the bond between the matrix and the filler [Bowen 1962]. The resin matrix is generally made up of monomers such as Bis-GMA, UEDMA, and TEGDMA. The dimethacrylate allows for extensive cross-linking during the addition polymerization. This has a direct effect on the degree of conversion of unreacted methacrylate groups after polymerization. Degree of conversion, "C",  $[C = (1-R) \times 100 \ %]$  is a measure of the consumed carbon double bonds, and is normally in the range of 50-70%. This does not imply that 30-50% of monomer remains unreacted, as one end of the dimethacrylate molecule may be covalently bonded to the polymer chain or to the filler (Figure 5) [Soderholm 1996].

Figure 5: Degree of Conversion in Setting of Composite Resins (Soderholm, 1996)



The inorganic, silanated filler particles in different sizes and shapes are mainly responsible for the enhancement of the mechanical and physical properties of the composites. Fillers contribute to a reduction in polymerization shrinkage and water sorption, and allow for inclusion of radiopaque metals. Silane coupling agents, light sensitive activators, inhibitors for a controlled polymerization, and color pigments are also present in CR. Volume fraction filler ( $V_f$ ), filler size, size distribution, index of refraction, radiopacity, and hardness are important determinants of composite resin properties. The size of filler particles may be used to classify composite resins (Table 1).

Table 1: Classification of Composite Resins based on filler size	
Category of CR	Average filler size
Macrofillers/ Traditional CR	8 to 12 $\mu\text{m}$ (as large as 50 $\mu\text{m}$ )
Small-particle-filler CR	1 to 5 $\mu\text{m}$ (clinically similar to Macrofillers)
Hybrid CR	0.6 to 1.0 $\mu\text{m}$ (fillers and microfillers)
Microfilled CR	0.04 to 0.4 $\mu\text{m}$

Traditional CR, also known as macrofills, have fillers made up of ground quartz particles with a 8-12  $\mu\text{m}$  range size. Small particle-filled CR have inorganic fillers of 1-5  $\mu\text{m}$  in size. Their broad particle size allows for higher filler loading. The primary filler consists of silane-coated ground particles of either quartz or glasses that contain heavy metals. Microfilled CR contain filler sizes of 0.04- 0.4  $\mu\text{m}$ . In an effort to reduce the agglomeration of the microfillers, ground cured-CR with sizes larger than the quartz fillers in the traditional CR are added in. As a

result, if one were to count the CR particles as filler particles, the filler content would be closer to 80% weight (~ 60% volume) [Soderholm 1996].

Hybrid CR contain fillers with average size of 0.6- 1.0  $\mu\text{m}$ . Most hybrid CR contain both colloidal silica fillers and ground particles of glasses containing heavy metals, reaching a filler content of ~ 75-80% weight. As  $V_f$  plays an important role in the mechanical properties of composites, composite resins may be classified based on their  $V_f$  [Willems et al. 1992].

### **1.5 Packable Composite Resins**

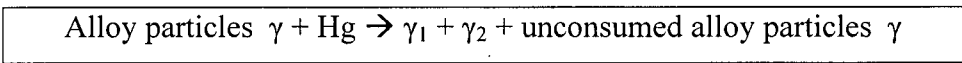
More recent CR formulations have lead to the introduction of the so-called “packable” composite resins (PCR). PCR have been developed to mimic the handling characteristics of amalgams, thereby avoiding common clinical problems with hybrid and microfilled composite resins in large and multisurface posterior restorations. Manufacturers of PCR claim that packability they was achieved through modifications in the filler of each system. Surefil, for instance, employs a patented “interlocking particle technology”, which is a blend of different sized particles (Caulk/ Dentsply, 2003). Manufacturers claim alleviation of open proximal contacts, flat contact areas and voids, inadequate finishing, post-operative sensitivity, and polymerization shrinkage with the use of PCR [Cobb et al. 2000; Leinfelder et al. 1998; Manhart et al. 2000a]. Filler size/length range anywhere from 0.04  $\mu\text{m}$  to 60  $\mu\text{m}$ . PCR are undergoing continuous testing in order to ascertain their potential handling and mechanical improvements to find out if they could be the true answer for the replacement of amalgam.

### **1.6 Dental Amalgam Alloy**

Amalgam has served the dental profession well since its introduction to dentistry over 100 years ago. Amalgams are metallic alloys of mercury (Hg). As mercury exists in liquid form at room temperature, it may be mixed with a dental amalgam alloy, consisting principally of silver, copper, and tin, to form a plastic mass that may be condensed into a cavity preparation in a tooth. Dental amalgam will finally harden as the alloying reaction proceeds. Historically, amalgam alloys contained 65% weight silver, 29% weight tin, and less than 6% weight copper. This formulation has been replaced since the introduction of the superior high copper alloys, with 6-30% weight copper, in the 1970s. During the trituration process, mercury dissolves the surface of the alloy particles and forms a new phase with melting points far above temperatures experienced in the mouth (Table 2) [Marshall et al. 1996].

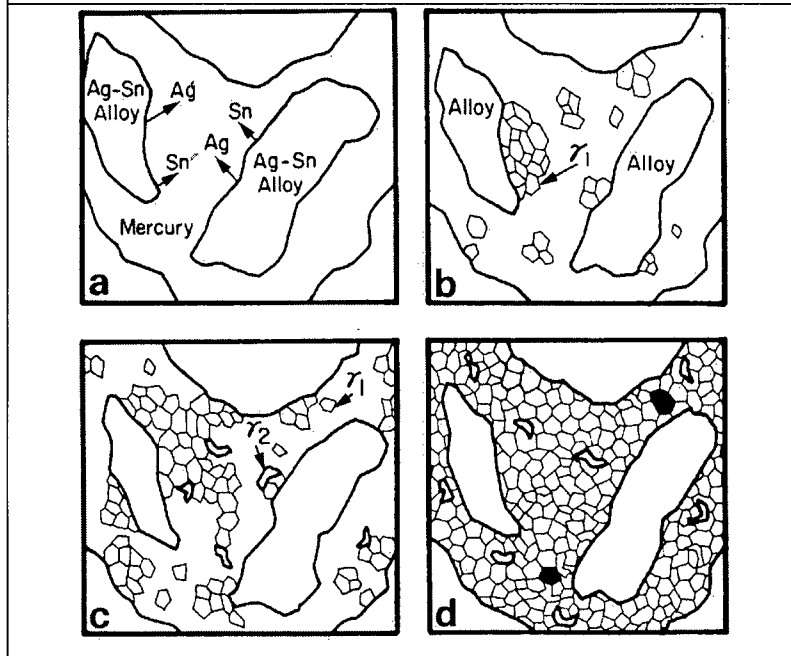
Table 2: Phases involved in Setting of Dental Amalgam	
Phase	Formula
$\gamma$ (gamma)	$\text{Ag}_3\text{Sn}$
$\gamma_1$	$\text{Ag}_2\text{Hg}_3$
$\gamma_2$	$\text{Sn}_{7-8}\text{Hg}$
$\epsilon$ (epsilon)	$\text{Cu}_3\text{Sn}$
$\eta$ (eta)	$\text{Cu}_6\text{Sn}_5$
Silver-copper eutactic	$\text{Ag-Cu}$

For early generation low copper amalgam, the setting reaction is as follows:



The  $\gamma_2$  phase is the weakest component and is also the least stable in a corrosive environment (Figure 6). The more unconsumed Ag-Sn phase that is retained, the stronger the amalgam.

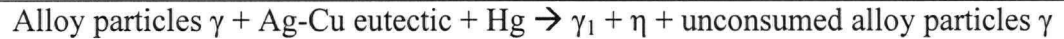
Figure 6: Low-Copper alloy particles mixed with mercury. a) dissolution of silver and tin into mercury, b) precipitation of  $\gamma_1$  crystals, c) consumption of remaining mercury by  $\gamma_1$  and  $\gamma_2$  phases, d) final set amalgam. (Anusavice, 1996)



High-copper alloys (> 6% copper) that have become the materials of choice are available in two different types: admixed alloy powder and single composition-alloy powder [Marshall et al.



1996]. The admixed alloy powder consists of spherical silver-copper (Ag-Sn) eutectic alloy and lathe-cut low-copper amalgam alloy particles. The admixed alloy reacts with Hg as follows, in which the  $\gamma_2$  phase is eliminated:



The copper content in various single-composition alloys ranges from 13- 30% by weight. The single-composition alloy powder reacts with Hg as follows, in which the  $\gamma_2$  phase has been eliminated (Figure 7):

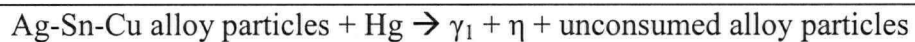
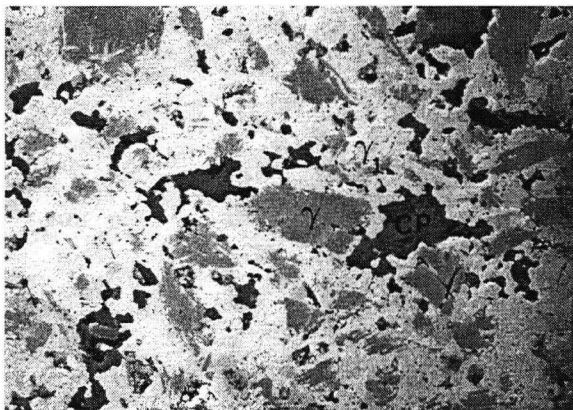
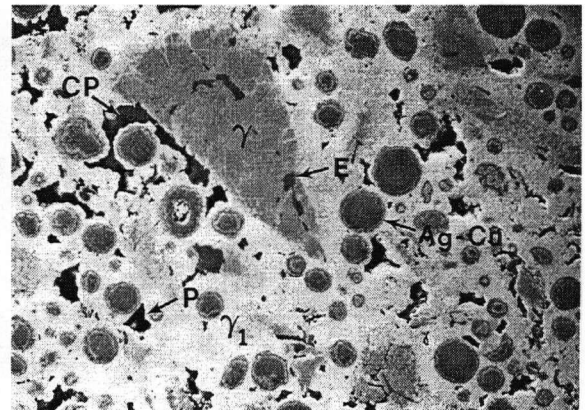


Figure 7: Microstructural differences between set low-copper and high-copper amalgam (Anusavice, 1996)

a. aged low-copper amalgam, in which  $\gamma_2$  phase is replaced with corrosive products



b. aged high-copper amalgam with few corrosive products



Despite the improvements in amalgam formulations, many other factors influence the success of amalgam restorations. In a controlled state, however, conservative amalgam restorations serve the patient well.

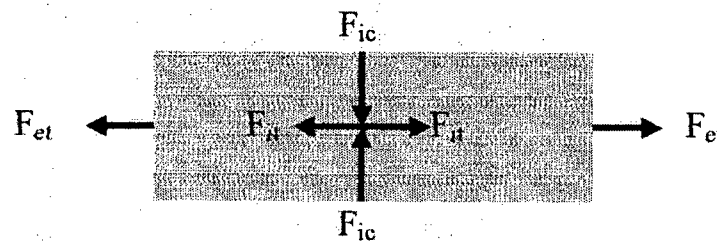
False advertisements by dental manufacturers have led to a significant degree of misunderstanding amongst practitioners as to the true nature of the physical and mechanical properties of dental materials. Academicians and clinicians need to base their teachings and practices on sound scientific findings as to the properties of these materials and the indications for their use.

## 1.7 Testing for Mechanical Properties of Biomaterials

As lives become more complex due to our dependency on different types of machines that surround us, from a wristwatch with its delicately rotating hands to crane elevators capable of lifting tonnes of steel, the durability and reliable performance of the materials that surround us gain more importance. Perhaps the greatest benefit of science to humanity has been the affordability of power of prediction. It is with this power that mankind has been able to fabricate materials and machines with a highly predictable performance. For any machine to perform at its maximum potential it is essential that every component of it withstand the forces applied to it within the limitations of its design.

Dental prostheses, from a simple “filling” to a complex implant prosthesis must withstand high forces of mastication over repeated cycles for many years in the very hostile environment of the oral cavity. Understanding the principles of materials science is imperative in the design and success of dental materials. Material properties that are affected by external or internal forces applied to a sample of material are referred to as the *mechanical properties* of that material [Anusavice 1996]. *External forces* are those applied by sources external to the surface of the material. *Internal forces*, on the other hand, are a result of molecular or atomic interactions within the material. All forces could be defined on the basis of two principal components: *compressive forces* are those that tend to compress or shorten the material, and *tensile forces* are those that stretch or elongate the material (Figure 8). Each of these principal forces affects the internal dynamics of the material. External tensile forces, for example, produce not only internal tensile forces, but also internal compressive forces that are perpendicular to the direction of the pull (Figure 8).

Figure 8: Formation of internal forces as a result of external forces



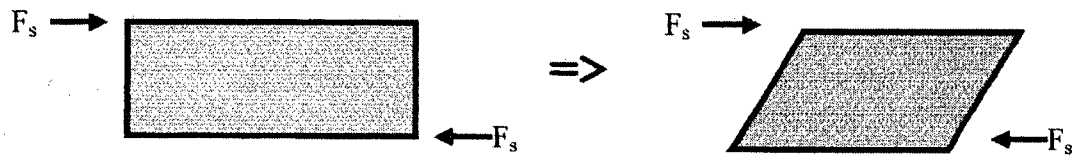
**An External Tensile force Produces Internal Compressive and Tensile Forces**

$F_{et}$ : external tensile force ;  $F_{it}$ : internal tensile force;  $F_{ic}$ : internal compressive force

Tensile forces tend to elongate the material; Compressive forces tend to shorten the material

A *shearing force* is an external force that results in the deformation of the material around a central axis. It is the formation of internal tensile and compressive forces that lead to deformation of the material (Figure 9).

Figure 9: Formation of Shearing Forces



An External Shearing Force Results in the Deformation of A Material Around A Central Axis ( $F_s$  : Shearing force)

*Stress* ( $\sigma$ ) is defined as the force per unit area acting on millions of atoms or molecules in a given plane of material [Anusavice 1996]. A tensile force produces *tensile stress*, a compressive force produces *compressive stress*, and a shear force produces *shear stress*. Any stress within a structure could be defined in terms of its tensile, compressive, and shear components [Anusavice 1996]. A *flexural force* can produce compressive, tensile, and shear stresses.

Stress may be further subdivided on the basis of its effect on the shape of the material. *Elastic stresses* cause temporary deformation of a material that is capable of changing shape as a result of applied forces. *Plastic stresses*, on the other hand, cause permanent deformation and may be high enough to produce a fracture. For brittle materials that exhibit only elastic deformation and no plastic deformation, stresses at or beyond the maximum elastic stress result in fracture [Anusavice 1996]. *Strain* ( $\epsilon$ ) is the deformation that is produced in a body of material whenever a stress is present. Tensile and compressive stresses produce tensile and compressive strains in a body of material respectively. Strain, by definition, is the change in length,  $\Delta l$ , per original length,  $l_o$ .

$$\text{Stress } (\sigma) = \frac{\text{Force (F)}}{\text{Area (A)}} \quad \begin{array}{l} \text{units: N/m}^2 = \text{Pa} \\ \text{Thus: 1 MPa} = 10^6 \text{ N/m}^2 \end{array}$$

$$\text{Strain } (\epsilon) = \frac{\Delta l}{l_o} \quad \text{units: m/m} = \text{relative change expressed in \%}$$

*Elastic strain* refers to the type of deformation that is reversible when the force is removed, whereas *plastic strain* represents a permanent deformation of a material that never recovers to its original shape when the force is removed [Anusavice 1996].

In general it can be said that a tensile stress is caused by a load that tends to stretch or elongate a material and is always accompanied by tensile strain, while compressive stress refers to the internal resistance to a load that tends to compress or shorten a material and is associated with compressive strain. *Shear stress* tends to resist the sliding of one portion of a body over another and can also be produced by a twisting or torsional action on a material. Shear stress is calculated by dividing the force by the area parallel to the force direction [Anusavice 1996].

The relationship between stress and strain of a material could be plotted on a graph, the slope of which is defined as the *elastic modulus*,  $E$ , also known as the *Young's modulus of elasticity* (Figure 10). The elastic modulus is an indication of the relative stiffness or rigidity of a material. The *strength* of a material is given by the value of the ultimate stress. It should be noted that strength is a collective measure of the interatomic forces of a body of material and not a measure of individual atom-to-atom attraction or repulsion [Anusavice 1996]. Depending on the intended application of a material, one or more of the following properties can be important: (1) *Proportional limit* (PL), which is the stress above which stress is no longer proportional to strain; (2) *elastic limit*, which is the maximum stress a material can withstand before it becomes plastically deformed; (3) *yield strength* ( $Y_s$ ), which is the stress required to produce a given amount of plastic strain (0.2%); and (4) *ultimate tensile/compressive/shear/flexural strength* (UTS) [Anusavice 1996].

*Resilience* can be defined as the amount of energy absorbed by a structure when it is stressed to its proportional limit and is represented by the area under the elastic region of its stress-strain plot. Resilience is associated with “springiness”; a material with higher resilience has a greater potential to act as a spring. *Toughness* is measured by the total area under the stress-strain curve. This is a measure of the total elastic and plastic deformation energy a material absorbs before it fractures. Toughness, therefore, is a measure of the resistance of a material to fracture [Anusavice 1996].

Figure: 10; Stress-Strain Relationship

PL: proportional limit represents the point up to which the material deforms elastically with  $\sigma$  and  $\epsilon$  being proportional to each other

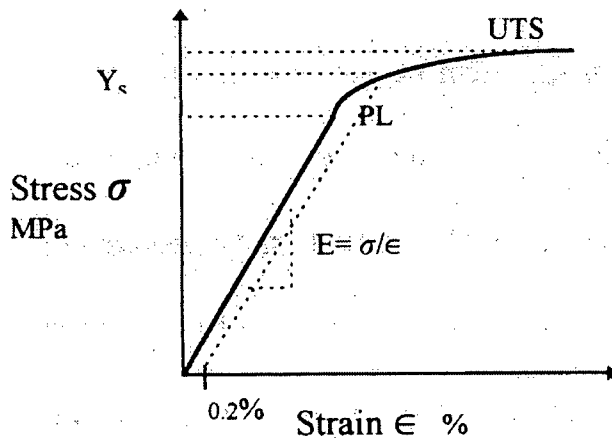
Ys: Yield strength; after this point there is no proportionality between  $\sigma$  and  $\epsilon$  and the material will undergo permanent deformation

UTS: Ultimate tensile strength is the highest stress on the  $\sigma/\epsilon$  curve

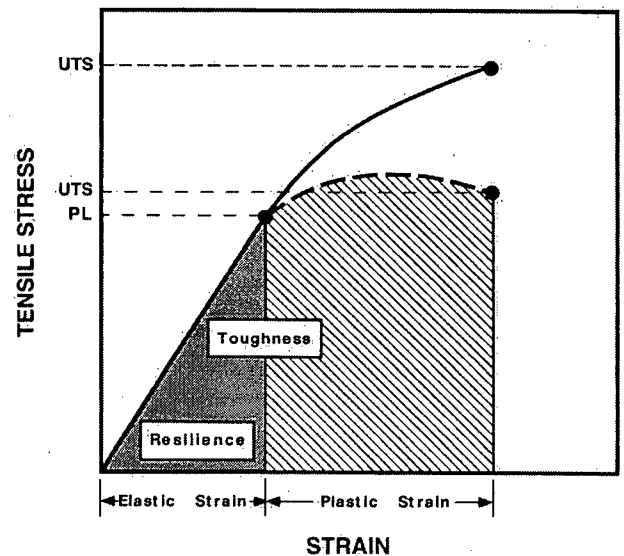
Resilience: the energy absorbed by the material up to the PL

Toughness: total area under the curve represents the total amount of energy a material absorbs prior to fracture

a. Stress-Strain Relationship

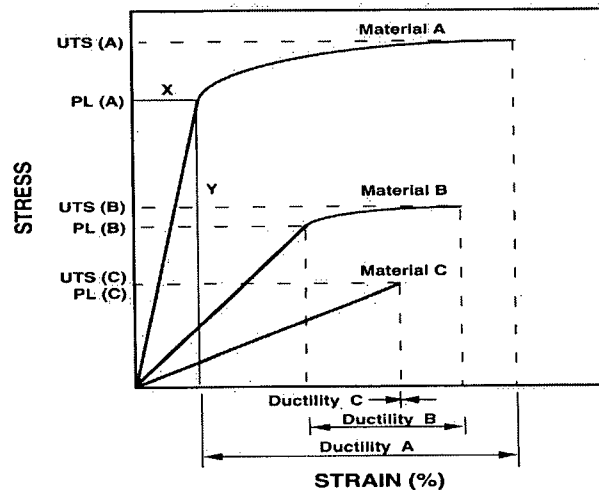


b. Resilience and Toughness



*Brittleness* is the relative inability of a material to undergo plastic deformation before it fractures. Both GIC and RMGIC are examples of brittle materials at oral temperatures ( $5^\circ$  to  $55^\circ$  C). They undergo little or no plastic strain before they fracture near their proportional limit (Figure 11).

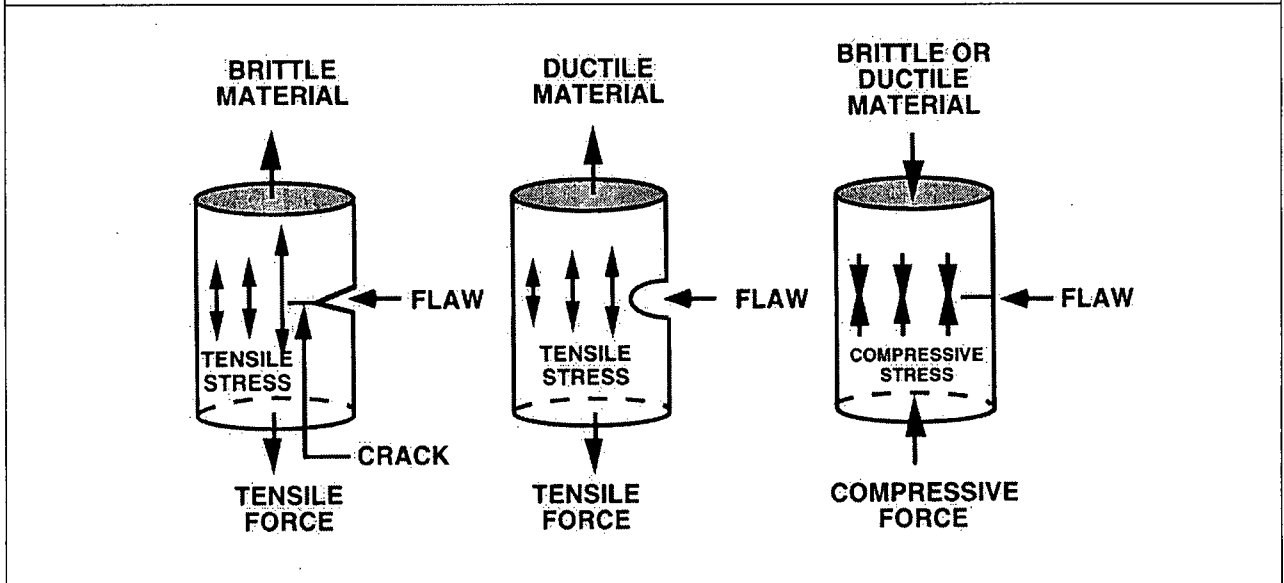
Figure 11: Strain-Stress plots of three materials with different mechanical properties



Small *microscopic flaws* or *microstructural defects* on the surface or within the internal structure of a material also affect its mechanical properties (Figure 12) [Anusavice 1996]. Brittle materials are especially affected by these structural defects. A microcrack on the surface of a brittle material, for example, has a greater tendency to grow deeper into the material than a microcrack on the surface of a ductile material. A ductile material has the ability to deform plastically and reduce the high stress concentrations at the tip of the crack, whereas a brittle material is devoid of such plastic accommodation [Anusavice 1996]. These structural flaws render a brittle material especially prone to failure when tensile forces are applied to it. On the other hand, compressive forces that are perpendicular to the direction of surface cracks tend to close the gap in a microcrack and prevent their propagation.

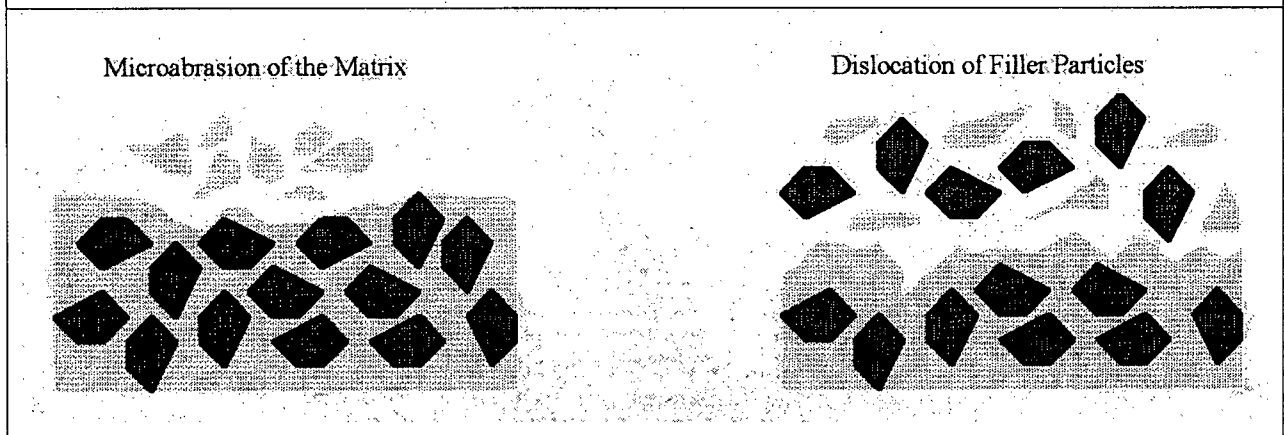
Reduction of surface flaws serves an important function in increasing the clinical longevity of restorative materials, particularly for brittle materials. Two important aspects of these surface flaws are: (1) the stress intensity increases with the size and length of the flaw, and (2) surface flaws are associated with higher stresses than internal flaws. With GIC, for instance, it has also been shown that different wear mechanisms exist on different surfaces of a restoration [Anusavice 1996].

Figure 12: Effect of Material Ductility and Brittleness on Flaw Propagation [Anusavice 1996]



Wear in a contact-free area involves the microabrasion and dissolution of the matrix (Figure 13) [Bauer et al. 1996]. Following matrix dissolution, filler particles lose their bond with the remainder of the restoration and are displaced from the surface. *In vitro* experiments using a thermal cycling test in lactic acid have also supported this concept of matrix dissolution in the contact free surfaces of GICs/RMGICs [Hotta et al. 1995; Watson and Banerjee 1993].

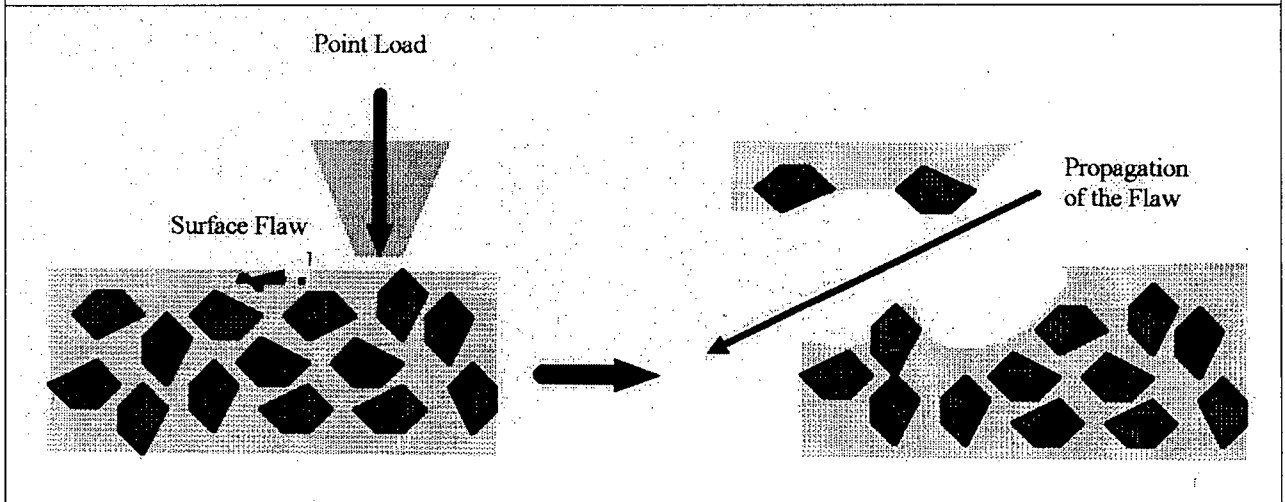
Figure 13: 2-body wear for Contact-free Areas: matrix dissolution and subsequent loss of filler particles (based on work by Bauer et al. 1996)



Wear at occlusal and proximal contact areas, however, is affected more by the filler system than by the intervening matrix (Figure 14) [Bauer et al. 1996; Oilo 1992]. Deformation of the surface in the contact areas results in the coalescence of surface microcracks and their

subsequent propagation parallel to the surface of the material. This results in the loss of the surface layer of the material that is in contact with the adjacent or opposing tooth.

Figure 14: 3-body wear for Contact Areas: Propagation of surface flaws and loss of material (matrix and filler) (based on work by Bauer et al. 1996)



The existence of different wear mechanisms in different areas of a restoration indicates the importance of applying a surface protecting material that not only seals the surface and protects the matrix, but also penetrates surface defects and forms a strong bond to the surface of the material. It should be emphasized, however, that both of these wear mechanisms act simultaneously, but to different degrees, on different surfaces of the restoration. Consequently most dental restorations should have their surface protected following placement [Watson et al. 1991]; [Wilson and Paddon 1993]. The surface protecting material should not impede the initial setting reaction, the matrix should be protected from early dissolution and water loss, and the surface microcracks should be sealed and bonded in order to enhance surface toughness of the restoration. Surface finishing of brittle materials is therefore, important in areas subjected to tensile stress. Experiments with the '*glazing technique*' in which the surface of the finished restoration is etched for a short time and a low viscosity glazing agent is applied and light-cured have also been shown to be effective [Cho et al. 1995; Hotta and Hirukawa 1994; Hotta et al. 1995]. This technique allows the overlying glazing agent to bond to the conditioned restoration surface, and thus takes advantage of the superior surface finish, wear resistance, and aesthetics of the glazing agent. One such light-cured glazing agent for use over GIC restorations, Bellfeel Brightener (Kanebo; Tokyo, Japan), has produced a significantly harder surface, which is not affected by thermal cycling in a lactic acid solution, than other surface protecting materials in



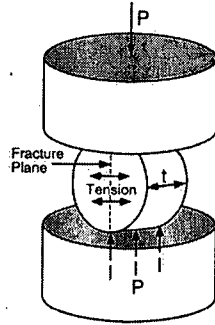
use [Hotta and Hirukawa 1994; Hotta et al. 1995]. Bellfeel-hardened GIC had a significant reduction in wear and abrasion when compared with GIC restorations protected by means of light-cured bonding agents. As a result, the '*glazing technique*' with a low viscosity glazing agent that is capable of bonding to the surface of the conditioned restoration and developing a strong surface toughness upon light curing, is the most effective means of both protecting restorative materials during their initial setting and enhancing their wear and abrasion resistance.

Sharp changes in shape, such as the sharp internal angle at the pulpal-axial line angle of a tooth preparation also introduce areas of high stress concentration. Differences between the elastic moduli or the coefficient of thermal expansion/contraction of the restorative material and the tooth structure also result in the development of stress concentrations at their interface [Anusavice 1996]. A load applied at a point (*Hertzian point load*) to the surface of a brittle material also produces an area of high stress concentration. The average maximum sustainable biting force has been estimated to be 756 N, and varies from one area of the mouth to another. The compressive stress that results from the application of this force through a cusp tip is estimated to be 193 MPa (Anusavice, 1996).

As a result, several clinical modifications may be suggested to help reduce the development of high areas of stress concentration in the tooth structure, the restorative material, and at their interface: the surfaces should be finely polished, glazed, or sealed to reduce the depth of the surface flaws; the quality of the restorative material should be improved to reduce the formation of structural flaws; the cavosurface design of the cavity preparation and its restoration should have a gradual flow; angles at the periphery of the cavity outline and at the pulpal-axial line angle should be rounded; the elastic modulus and the coefficient of thermal expansion/contraction of the tooth structure and the restorative material should be closely matched; and the cusp tip of an opposing tooth should be well rounded such that occlusal contact areas in the brittle material are larger [Anusavice 1996].

The *tensile strength* of a brittle material is determined by subjecting it to a *diametral compression test* and is known as the *diametral tensile strength* ( Figure 15). In this method, a short cylindrical specimen of the material is placed under compression. As a result of the vertical compressive forces on the side of the disk, a tensile stress that is perpendicular to the direction of the compressive force is produced. In a properly carried out test, fracture occurs along the vertical plane. The tensile stress is then calculated.

Figure 15: Diametral Compression Test for a Brittle Material



$t$ = thickness

$D$ =diameter

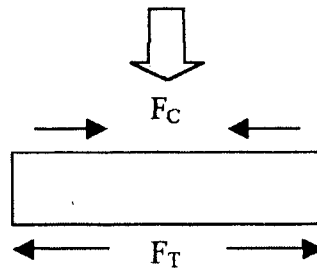
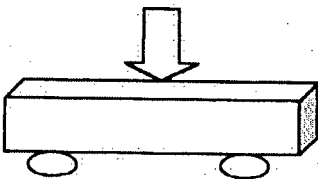
$F$ =compressive force

$$\sigma_T = \frac{2F}{\pi Dt}$$

The diametral compression test should be used only for materials that exhibit predominantly elastic deformation and little or no plastic deformation. It should be noted that most dental restorative materials present with some plastic qualities, especially during initial set [Wilson and McLean 1988].

Brittle materials have tensile strengths that are far lower than their compressive strengths. This is due to their susceptibility to surface flaws and internal defects when tensile stresses are present, and their inability to undergo plastic deformation to reduce stress concentrations [Anusavice 1996]. The clinician should, therefore, be aware of the unreliability of some of the reported values for mechanical properties of brittle materials in the dental literature. The tensile strength values reported for restorative materials represent the average stress values below which 50% of the test specimens have fractured and above which only 50% have survived. The range of measured values, therefore, should be known. Tensile strength of brittle materials is also derived from their *flexural strength*. Flexural strength is essentially a strength test of a bar supported at either end under a static load (Figure 16).

Figure 16: Measurement of Flexural Strength



$$\sigma_f = \frac{3Pl}{2bd^2}$$

$P$ : maximum load at the point of fracture

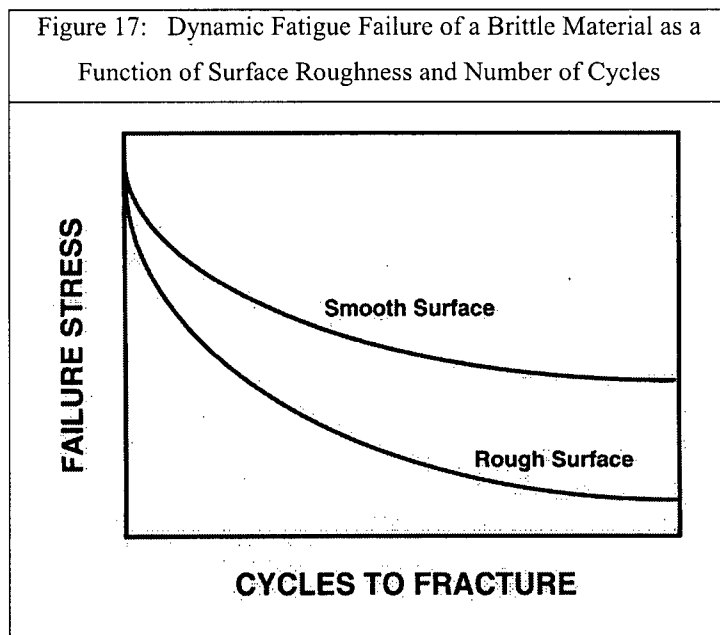
$l$ : distance between the supports

$b$ : width of the bar

$d$ : depth or thickness of the bar

As the load is applied on top of the bar, compressive forces result in compressive strain, while at the bottom of the bar tensile forces result in tensile strain and eventual failure. The point at which the upper compressive forces and the lower tensile forces balance is referred to as the neutral axis. It is believed that flexural strength is a more important and clinically relevant parameter than either compressive or diametral tensile strengths [Wilson and McLean 1988]. Flexural strength testing is believed to be a collective measurement of tensile, compressive, and shear stresses simultaneously [Burke et al. 2002].

The forces of mastication result in repeated production of tensile stresses in a brittle restorative material and lead to the propagation of the microscopic flaws. As a result, the material fractures at stress values far below its ultimate tensile strength. This phenomenon is known as *fatigue failure*, and is determined by subjecting the material to a cyclic stress of a maximum known value and determining the number of cycles that are required to produce failure [Anusavice 1996]. *Endurance limit* refers to the maximum stress that can be maintained over an infinite number of cycles without causing failure of the material. Static fatigue failure refers to failure of a material under static but repeated load, while dynamic fatigue failure refers to periodic and varying cyclic loads that result in failure (Figure 17).



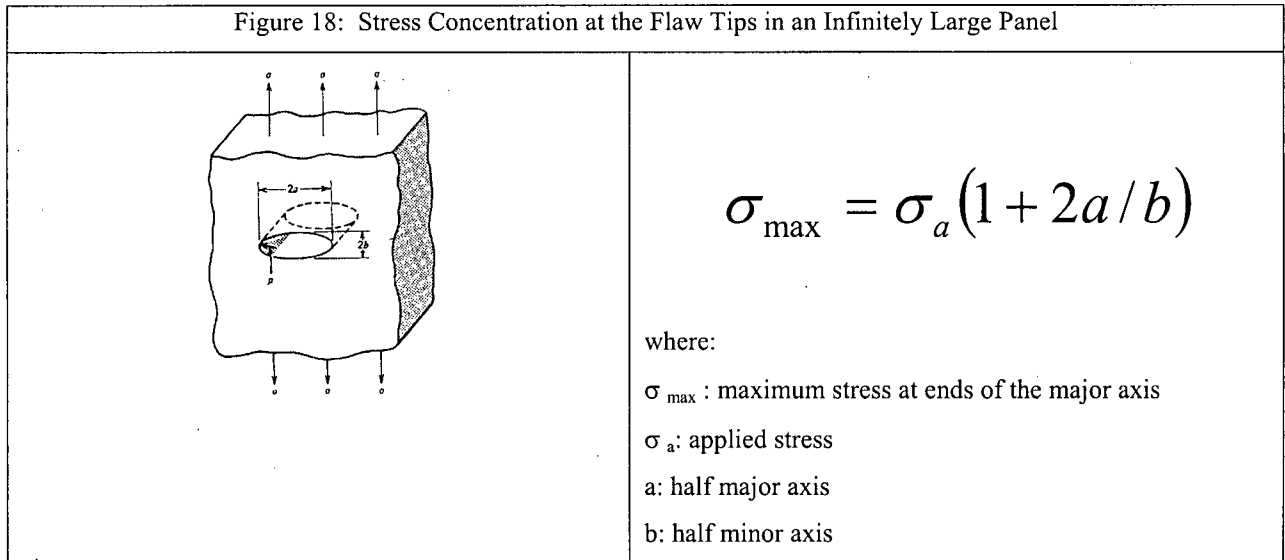
Dental restorative materials can exhibit static and dynamic fatigue failure depending on the nature of the loading situation. In both situations, it is the propagation of flaws within the material that ultimately leads to failure. A brittle material with a polished surface performs better and exhibits a higher endurance limit than the same material with a rough surface. Small

defects that are randomly distributed throughout the structure of a brittle material cause a significant variation in the strength values of otherwise identical specimens. As a result, *fracture toughness*, which is a mechanical property that describes the resistance of brittle materials to the catastrophic propagation of flaws under an applied stress is a more precise measure of the fracture resistance of a brittle material than is tensile strength [Anusavice 1996]. Toughness is the amount of elastic and plastic deformation required to fracture a material, and is dependent on strength and ductility. Fracture toughness ( $K_{IC}$ ) is given in units of stress times the square root of crack length, that is:  $\text{MPa}\cdot\text{m}^{1/2}$  or  $\text{MN}\cdot\text{m}^{-3/2}$ .

## 1.8 Principles of Fracture Mechanics

In an ideal world one would be able to predict the exact longevity of any restorative material based on its chemical constituents and the developing bonds amongst them. Experimental and clinical behaviour of materials varies significantly from theoretical predictions. The discrepancy is due to the presence of defects and flaws, and the process by which cyclic use of materials leads to the exacerbation of such flaws and their catastrophic propagation that leads eventually to their premature failure. Failure of restorative materials under load as a result of fracture initiation and propagation has been the Achilles' heel of restorative dentistry. *Fracture mechanics* aims to study and quantify the influence of factors such as stress level, presence of extrinsic and/or intrinsic flaws, inherent material properties, and mechanisms of catastrophic propagation of a flaw to failure, in order to determine the fracture behaviour of a material in aiding future designs [Hertzberg 1996].

One may attribute the elementary steps in analysis and quantification of crack propagation to work by Inglis, nearly a century ago [Inglis 1913]. Considering a plate with an elliptical hole or flaw, Inglis was able to demonstrate that the applied stress was magnified at either end of the major axis of the ellipse (Figure 18):



The radius of the curvature  $\rho$  at the end of the ellipse is given by  $\rho = b^2/a$ ,

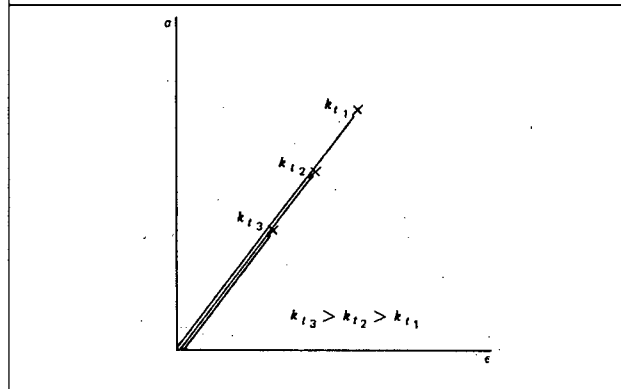
$$\sigma_{\max} = \sigma_a \left(1 + 2\sqrt{a/\rho}\right)$$

For a very sharp crack,  $a$  is much larger than  $\rho$ , then  $\sigma_a$  is much smaller than  $2\sigma_a\sqrt{a/\rho}$ ; therefore:

$$\sigma_{\max} \approx 2\sigma_a\sqrt{a/\rho} \quad , \quad \sigma_{\max} \approx \sigma_a k_t$$

The term  $2\sqrt{a/\rho}$  is referred to as the *stress-concentration factor*;  $k_t$ , and describes the effect of crack geometry (length and tip radius) on the local crack tip stress level. This means that  $k_t$  increases with increasing crack length ( $2a$ ) and decreasing crack radius ( $\rho$ ) or increasing sharpness of crack tip. To reduce crack propagation, either the crack length must be reduced or the crack tip radius increased by drilling a round hole at the tip of the crack. It should also be noted that the maximum stress and strain level that any component may support decreases with increasing  $k_t$  (Figure 19). Hence, the sharper and longer the crack, the easier the material fractures.

Figure 19: Effect of Stress Concentration Factor,  $K_t$ , on allowable Stress and Strain in completely elastic materials [Hertzberg 1996]

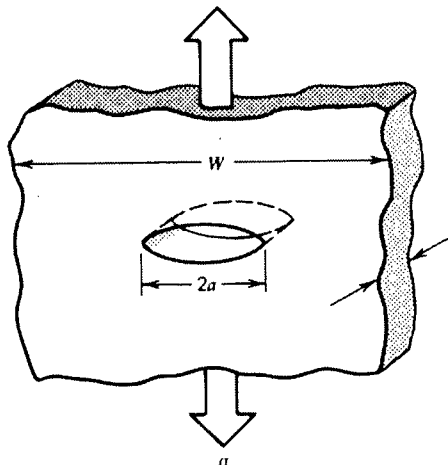


Fortunately in most materials the plastic deformation at the crack tip prevents a catastrophic crack propagation to failure. Furthermore as long as the applied stress is sufficiently low,  $\sigma_a k_t$  values will remain comfortably below the local stress level necessary for fracture.

A.A. Griffith in 1920 was the first to offer quantitative relations in determining the fracture of cracked solids based on Inglis' work [Griffith 1920; Inglis 1913]. Griffith's crack theory is based on the fact that when a crack is introduced into a stable body of a material, the

reduction in the potential energy of that material must be balanced by the increase in surface energy. The crack would then grow when additional necessary energy is supplied. He then calculated the change in surface energy of an elastic plate as a result of the introduction of a crack as shown in Figure 20:

Figure 20: Change in Surface Energy of an Elastic Plate as a result of introducing a crack

	$U - U_0 = -\frac{\pi\sigma^2 a^2 t}{E} + 4at\gamma_s$ <p>U: potential energy of body with crack; U<sub>0</sub>: potential energy without crack; <math>\sigma</math>: applied stress; a is half crack length; t: thickness of body; E: modulus of elasticity; <math>\gamma_s</math> specific surface energy</p>
---	---

Differentiating the potential energies by crack length and when the body is at equilibrium, we will have:

$$0 = -2\frac{\pi\sigma^2 a t}{E} + 4t\gamma_s \quad \rightarrow \quad 2\gamma_s = \frac{\pi\sigma^2 a}{E}$$

Griffith rewrote this as:

$\sigma = \sqrt{\frac{2E\gamma_s}{\pi a}}$	<p>For the case of Plane Stress (biaxial stress condition)</p>
$\sigma = \sqrt{\frac{2E\gamma_s}{\pi a(1-\nu^2)}}$	<p>For the case of Plane Strain (triaxial stress condition)</p>

As Poisson's ratio,  $\nu$ , is 0.25 to 0.33, there is not much difference between the two equations. Griffith's formula was derived for elastic materials containing a very sharp crack and does not apply to materials capable of plastic deformation, such as metals and polymers.

For metals and polymers capable of plastic deformation, the fracture energy is several times greater than the surface energy of a material. [Orowan 1950]. Orowan suggested a modification to Griffith's equation to include the energy of plastic deformation in the fracture process.

$$\sigma = \sqrt{\frac{2E(\gamma_s + \gamma_p)}{\pi a}} = \sqrt{\frac{2E\gamma_s}{\pi a} \left(1 + \frac{\gamma_p}{\gamma_s}\right)}$$

Where  $\gamma_p$  is the plastic deformation energy, and  $\gamma_p \gg \gamma_s$ , meaning:

$$\sigma \approx \sqrt{\frac{2E\gamma_s}{\pi a} \left(\frac{\gamma_p}{\gamma_s}\right)}$$

As opposed to Orowan who used the energy sink terms,  $\gamma_s$  or  $\gamma_s + \gamma_p$ , Irwin used the energy source term [Irwin 1957]; i.e. the elastic energy per unit crack-length increment:

$$\frac{\delta U}{\delta a} = G \rightarrow \sigma = \sqrt{\frac{EG}{\pi a}}$$

This is one of the most important relations in the field of fracture mechanics. Irwin deduced that:

$$G = 2(\gamma_s + \gamma_p)$$

This means that at the point of instability, the elastic energy release rate,  $G$ , also referred to as the crack driving force, reaches a critical value,  $G_c$ , at which point fracture occurs. This critical

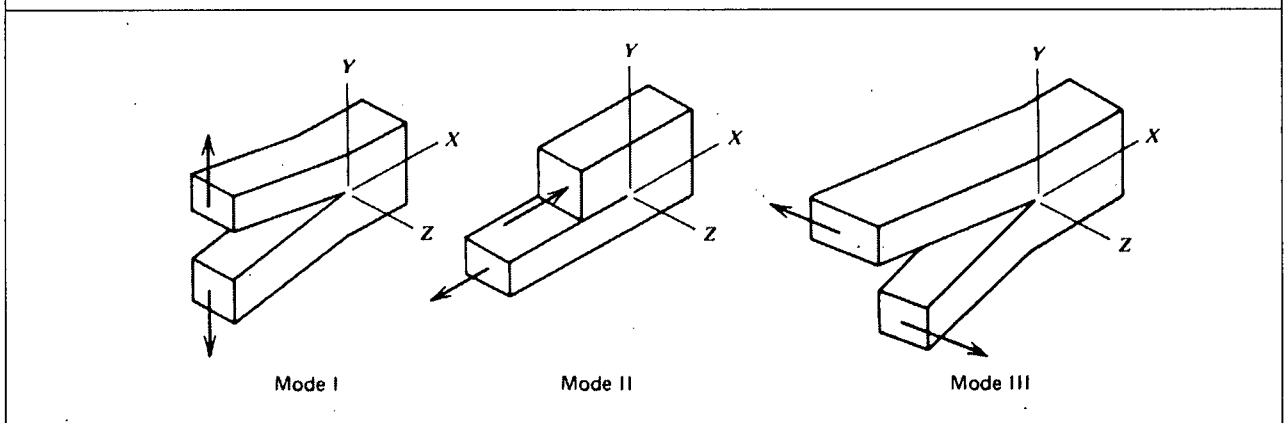


elastic energy release rate is a material property that can be measured in the laboratory with sharply notched test specimens [Irwin 1957].

The fracture of flawed components may also be analyzed using stress analysis [Hertzberg 1996]. Irwin also suggested solutions for crack-tip stress distribution in the three major modes of loading, namely:

1. Mode I: opening or tensile mode, where the crack surfaces move directly apart (Figure 21)
2. Mode II: sliding or in plane shear mode, where the crack surfaces slide over one another in a direction perpendicular to the leading edge of the crack (Figure 21)
3. Mode III: Tearing or antiplane shear mode, where the crack surfaces move parallel to one another and parallel to the leading edge of the crack (Figure 21)

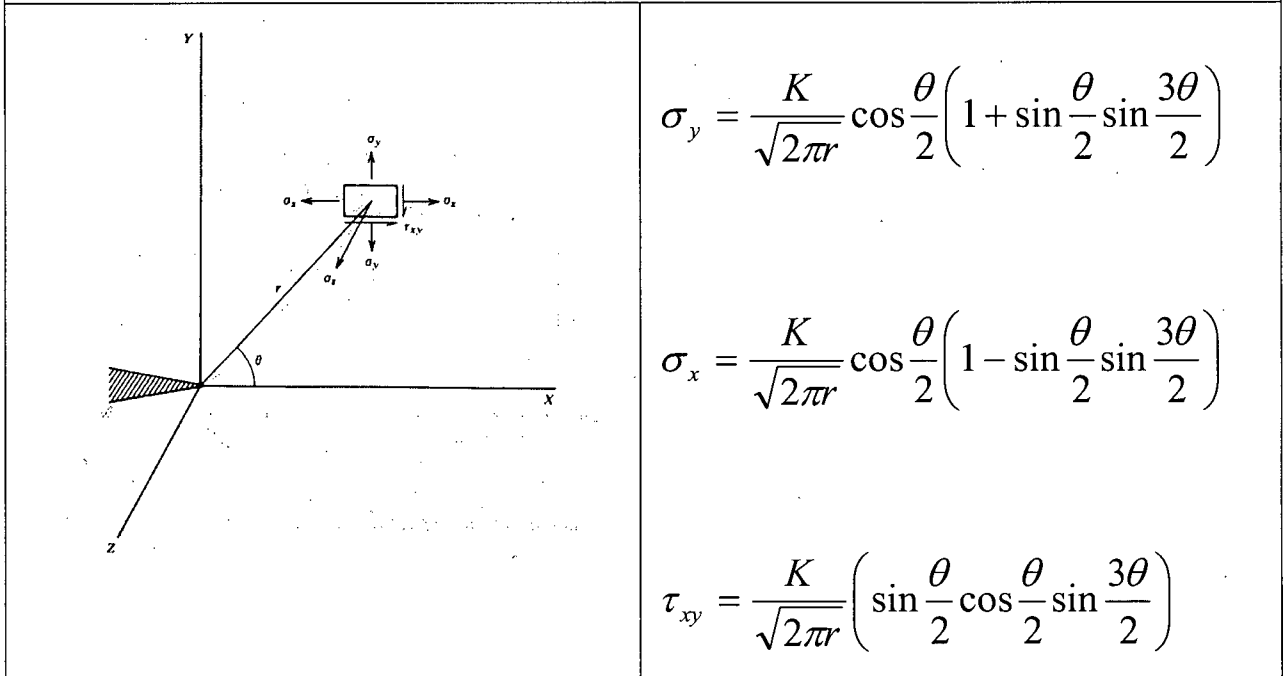
Figure 21: Different Modes of loading: Mode I, Mode II, Mode III (Hertzberg, 1996)



Because Mode I loading is the most commonly encountered, quantification of Mode I stress-crack-length relation deserves considerable attention.

The crack tip stresses at any point in the polar coordinates initiating at the crack tip, may be presented as (Figure 22):

Figure 22: Distribution of Stresses in the vicinity of crack tip (Hertzberg, 1996)



Clearly, as  $r$  approaches zero, stresses could rise to extremely high levels. This relationship therefore indicates that the stress distribution around any crack in a structure is similar and depends only on the parameters  $r$  and  $\theta$ . The difference between one cracked component and another lies in the magnitude of the stress field parameter  $K$ , defined as the stress-intensity factor. As Irwin stated, the functionality depends on the configuration of the cracked component and the manner in which the loads are applied.

$$K = f(\sigma, a)$$

Note that in a thin plate, the  $z$ -axis is eliminated, leading to a biaxial stress condition known as plane-stress. In a thick plate, however, the triaxial condition limits straining in the  $z$ -dimension, leading to development of plane-strain.

The interaction of materials properties, such as the fracture toughness, with the design stress and crack size controls the conditions for fracture in a component. The fracture condition for an infinitely large cracked plate would be:

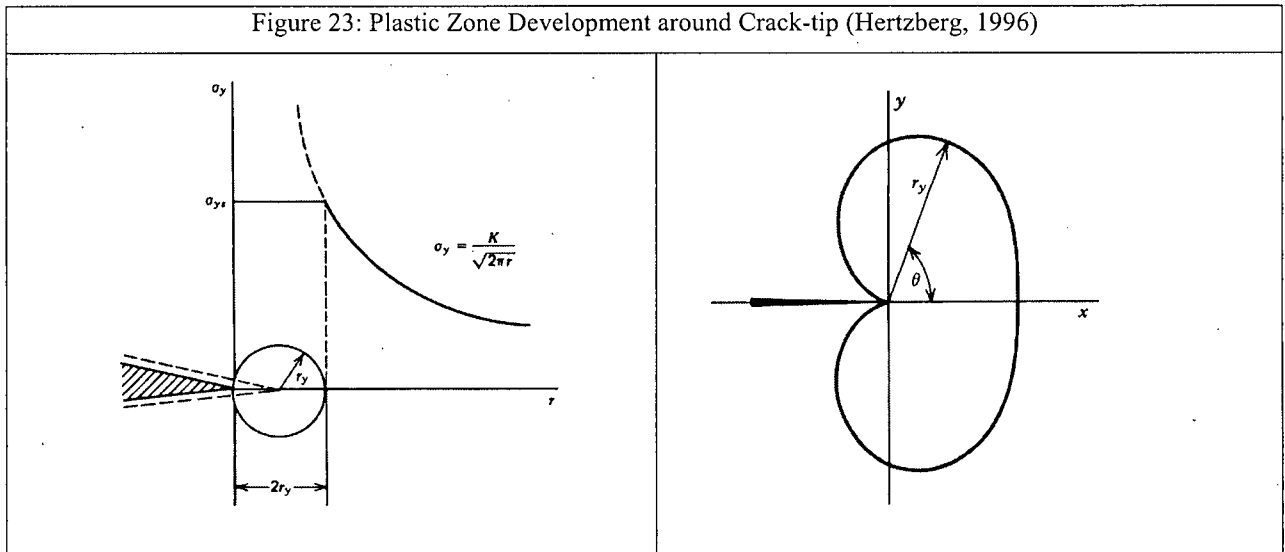
$$K = K_c = \sigma \sqrt{\pi a}$$

$K_c$ : material selection

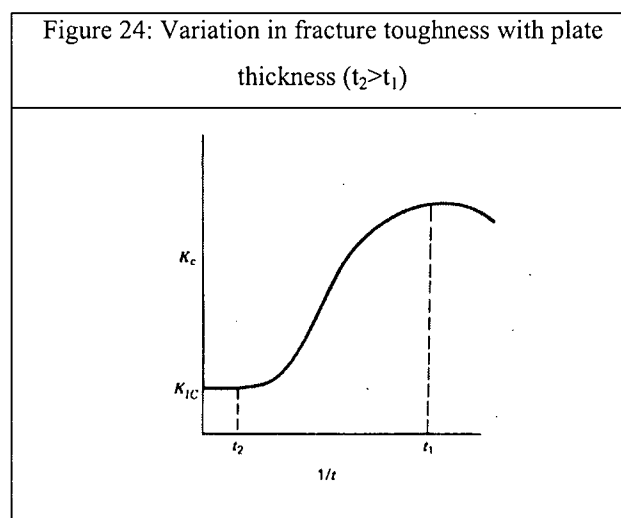
$\sigma$ : design stress

$a$ : allowable flaw size or selection

This allows one to design materials for a specific purpose, depending on the relative importance of material properties, the design stress level, or the flaw size to the overall needs for that material. For any ductile material; that is one capable of any plastic deformation, a plastic zone develops around the crack tip (Figure 23) [Hertzberg 1996].



The plastic-zone size is dependent on the stress-intensity factor and the state of the stress acting on the crack tip. The stress-intensity factor itself is dependent on the thickness of the material parallel to the direction of the crack. The thickness of the material controls the ability of the material to undergo plastic deformation due to available material. Figure 24 demonstrates this relationship between stress intensity factor, fracture toughness, and thickness of the material.



When the sample is thin, the plastic constraint acting at the crack tip is minimal, thereby resulting in plane-stress conditions prevailing with the material exhibiting its maximum toughness. However, when thickness is increased, the plastic constraint and plane-strain conditions increase at the crack tip, resulting in a sharp drop in toughness to a minimum level, known as the *plane-strain fracture toughness*,  $K_{IC}$ . This means that  $K_{IC}$  does not decrease further with increasing thickness, making it the lower limit of toughness in any engineering application. Henceforth, once  $K_{IC}$  is determined for a sample at least as thick as  $t_2$ , any component thicker than  $t_2$  should exhibit the same toughness.

In summary, the plane-stress fracture toughness,  $K_C$ , is related to both material properties and specimen geometry, while the plane-strain fracture toughness,  $K_{IC}$ , depends only on material properties. This means that  $K_{IC}$  allows for the best way to compare intrinsic fracture toughness levels of materials of different thickness, since the effect of geometry is factored out.

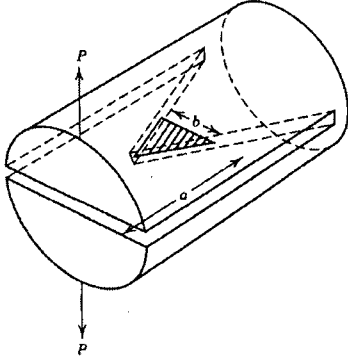
### 1.9 Methods for Measuring Fracture Toughness

Plane-strain fracture toughness,  $K_{IC}$ , is an important material property in characterizing the behaviour of materials under stress.  $K_{IC}$  may be determined for any size and shape of a material specimen so long as the stress intensity calibration is known. The American Society for Testing and Materials (ASTM) has stated acceptable test methodology for measuring plane-strain fracture toughness,  $K_{IC}$ . Some commonly used tests are:

1. Three-point bending bar sample (ASTM Standard E399, 1983) also referred to as single-edge notched (SEN), in which a load is applied to a notched bar supported on two points
2. Compact Tension Sample (ASTM Standard E399, 1983), in which opposing tensile loads are applied perpendicular to the pre-notched crack direction
3. C-shaped/ arc-shaped specimen sample (ASTM Standard E399-81), in which opposing tensile loads are applied at the tail ends and perpendicular to the concavity of the "C"
4. Disk-shaped sample (ASTM Standard E399-81))
5. Chevron-notched Short Rod (CNSR) and Chevron-notched Short Bar (CNSB), (ASTM Standard E1304, 1989), consisting of cylindrical or rectangular specimens in which a deep, machine-cut notch with Chevron configuration is placed (Figure 25). In this technique, wedge loading causes a pre-crack to form before the test is conducted.

Barker detailed testing for  $K_{IC}$  with the use of cylindrical or rectangular specimens that contained a deep, machined-notch with a chevron configuration (Figure 25) [Barker 1977]. Both CNSR and CNSB specimens allow for small specimen sizes and avoid the use of fatigue pre-cracking procedure. Bubsey and co-workers determined the stress intensity factor for a short rod specimen with a straight-through crack [Bubsey et al. 1982].

Figure 25: Chevron-notched Short Rod Specimen

	$K_I = \frac{P}{D\sqrt{W}} Y$ <p>D: specimen diameter W: specimen length Y: dimensionless stress intensity factor coefficient</p>
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For a chevron-notched specimen, however, the crack proceeds with a trapezoidal front, and the stress intensity factor is expressed as:

$$K_I = \frac{P}{D\sqrt{W}} Y^*$$

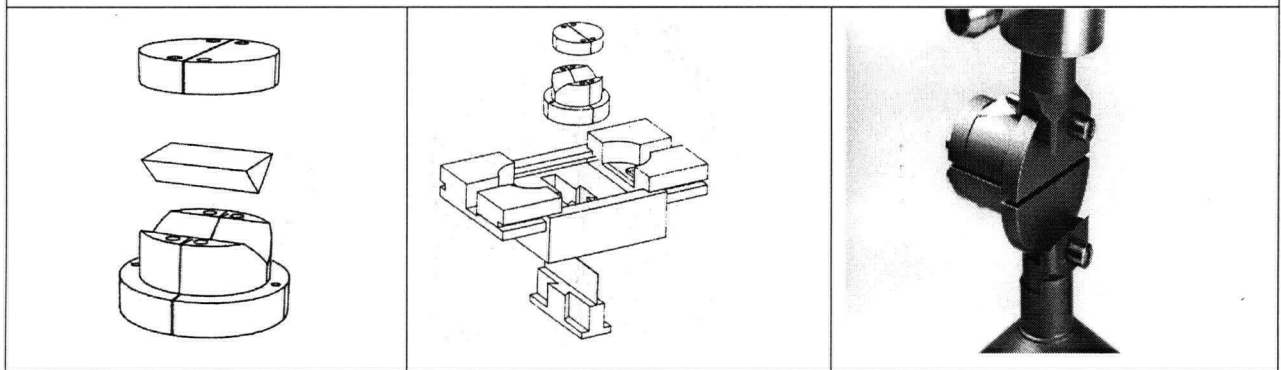
Bubsey and co-workers performed compliance calibrations to determine values for  $Y^*$  [Bubsey et al. 1982]. For a sufficiently brittle material, maximum load occurs at a minimum  $Y$ , and  $K_{IC}$  is the critical stress intensity factor, or fracture toughness, at which point catastrophic failure occurs:

$$K_{IC} = \frac{P_{\max}}{D\sqrt{W}} Y_M^*$$

More recently, Ruse and co-workers introduced a simplified method, namely the notchless triangular prism (NTP) specimen  $K_{IC}$  test [Ruse et al. 1996]. In this method, a

triangular prism of 6x6x12 mm is held in a multi-segmental jig (Figure 26). The pre-test apparatus then resembles the CNSR specimen, and therefore, is governed by the same relations in calculating  $K_{IC}$ . Finite element analysis has been used to confirm the validity of NTP test by showing that maximum stresses concentrated at a point corresponding to the tip of the chevron notch. The presence of crack arrest in most tested specimens is further indication that the test takes place in a stable and reliable manner. By linear extrapolation based on Bubsey's previous work in compliance calibration of CNSR specimen, Ruse et al. found the value of  $Y_{min}^*$  to be 28 [Bubsey et al. 1982; Ruse et al. 1996]. The accuracy of the NTP test was demonstrated by the excellent correlation between the  $K_{IC}$  values measured using the NTP test and the ones reported in the literature [Ruse et al. 1996].

Figure 26 : Notchless-Triangular Prism Specimen for the Measurement of Fracture Toughness [Ruse 1998]



The NTP test offers the advantages of: 1. ease of specimen fabrication by moulding, grinding, or milling; 2. use of small enough specimens to approximate actual clinical situations; 3. avoidance of cutting chevron notches into samples, thereby eliminating the introduction of surface flaws in the process and also enabling testing of very brittle materials with very small  $K_{IC}$ ; 4. avoidance of embedding of tooth tissues for interfacial adhesive tests; and 5. reproducibility of test conditions by means of a specimen holder [Ruse 1998]. It is also claimed that the NTP test is the only available testing methodology for materials with a fracture toughness less than  $1 \text{ MPa.m}^{1/2}$  [Ruse et al. 1996].

From the preceding discussion, one may conclude that fracture mechanics aim to study and quantify crack initiation and propagation. The larger the flaw, the lower the stress needed to cause fracture. A structure is as durable as its weakest aspect because once a crack goes through a catastrophic propagation at a maximum stress value, it will take less stress to cause further failure of the material. Fracture toughness,  $K_{IC}$ , is a material constant at fracture, and is

independent of loading conditions, crack size, and geometry. For the same flaw size, a material with a lower  $K_{IC}$  value fractures at a lower stress [Lloyd and Adamson 1987].

Fracture toughness,  $K_{IC}$ , is as much a material parameter as elastic modulus,  $E$ , is.  $K_{IC}$  gives the gradient of stress rise as the flaw is approached, measures the strain absorption capacity of a material, and is related to the level of tensile strength that must be exceeded at the crack tip before failure occurs. Fracture toughness, therefore, gives a greater understanding of the potential resistance of a material to stress than is given by the tensile or compressive strength [Lloyd 1982].

### **1.10 Clinical Significance of Mechanical Tests**

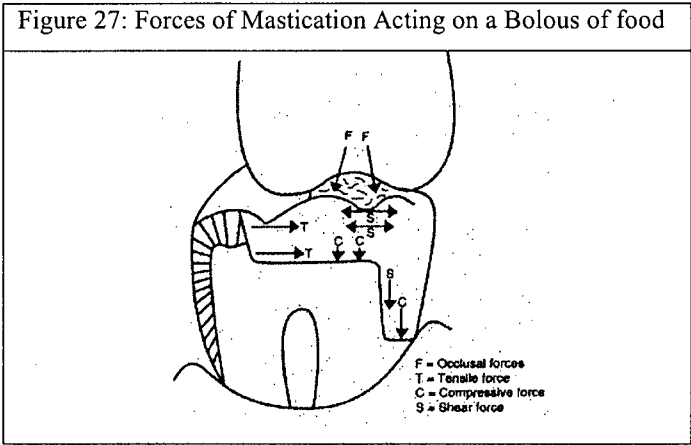
“Anecdotal evidence” may play too great a role in the decisions made by dental clinicians. To reduce the subjectivity of the clinical decision-making process, a clinician must rely on sound scientific evidence. Recent literature surrounding practice guidelines and standards in dentistry suggest that we are in the midst of an “evidence-based era”, which aims to base our daily practice of dentistry on sound and relevant scientific findings. Perhaps in the foreseeable future computer software will dictate the appropriate treatment taking into consideration scientific evidence and diagnostic findings. One should be able to assume that the options provided to patients have taken into account the relevant scientific background.

While mechanical testing aims to study and quantify the behaviour of a material in response to applied forces in order to facilitate improvements in design, a clinician is primarily concerned with failure of restorative materials, from marginal breakdown to resultant pulpal pathology. Failure of restorative materials may be either due to mechanical or biological factors [McLean 1990].

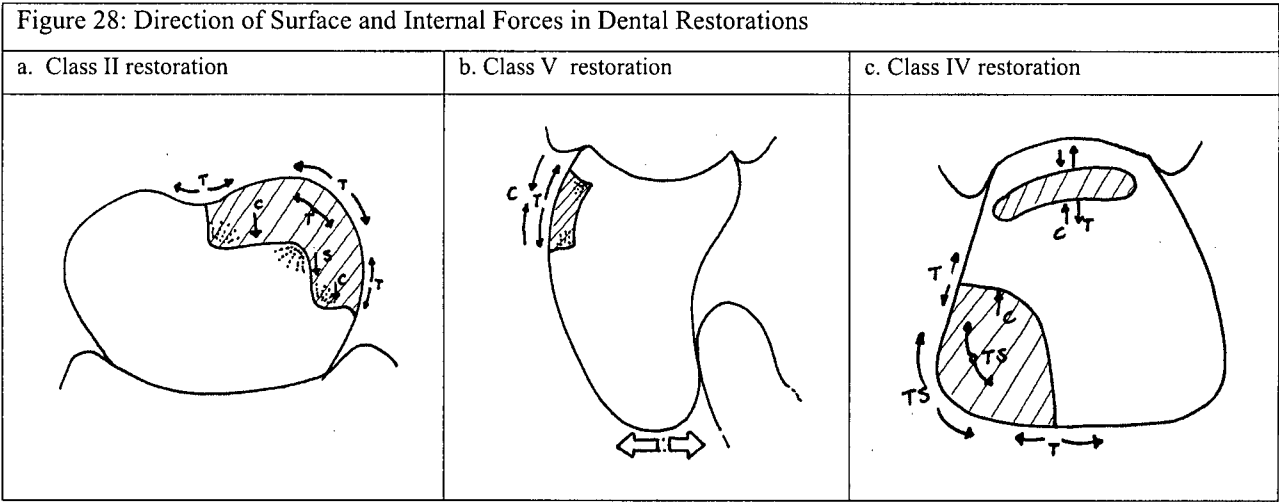
Mechanical failure may be associated with incorrect use of a material and/ or ignorance of stress-analysis when preparing teeth. Biological failure, on the other hand, relates to the body's response to the restoration. This biological response may range from contact dermatitis, when the material lacks biocompatibility, to pulpal inflammation and pathology due to recurrent thermal stimuli or bacterial ingress as a result of microleakage. It may be that biological failures are, at least in part, indirectly due to clinician's lack of understanding of the mechanical principles of materials.

Different methodologies are used to test each mechanical property of a material, each with its limited relevance to the clinical performance of that material. As a bolus of food is

crushed and chewed between opposing teeth, the primary forces of mastication are subdivided into infinitely smaller components, each acting on different surfaces of the teeth, the restoration, and the interface between the two (Figure 27). Concentration of forces is primarily at the corners and curved surfaces. One may assume that any one point of this complex is under a combination of forces.



The direction and magnitude of forces in a restoration is also dependent on the positional relationship of a restoration to the long axis of the tooth (Figure 28). It is important to determine the anticipated direction of loading of a restoration before assessing the mechanical properties of interest [Burke et al. 2002].



In Class V restorations, for instance, the material is placed under tensile and compressive forces as the tooth flexes away or onto the restoration respectively. There will be less need for wear resistance in Class V as opposed to Class II restorations, in which wear resistance is necessary to withstand the compressive and tensile forces of occlusal contacts and food products. Class II

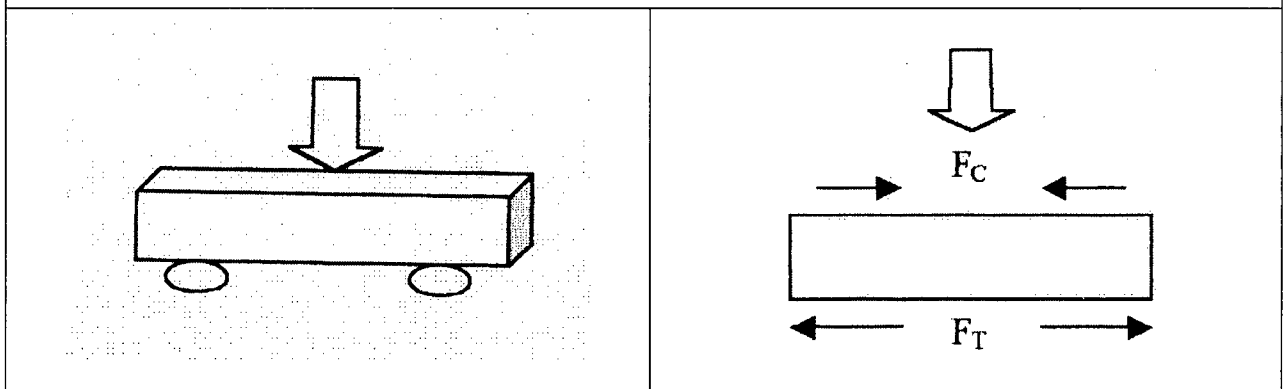


restorations should also withstand such elementary forces without cracking or fracturing. Compressive testing is applied to materials that are expected to withstand high levels of direct loading. Testing for compressive strength of a material, however, has limited value as most restorative materials possess high compressive strength, generally above the levels applied by masticatory forces [Burke et al. 2002].

Tensile strength measurements are more relevant as masticatory forces are frequently applied obliquely and tend to create tensile stress. Diametral tensile strength (DTS), for instance, represents the minimal stress that a body can withstand without rupture when tensile loads are applied [Burke et al. 2002]. As was discussed earlier, any force applied to any specimen produces a combination of compressive, tensile, and shear forces that cannot be considered in isolation.

Flexural strength testing with a bar supported on two points, known as the three-point bend test, combines elements of tensile, compressive, and shear testing (Figure 29). On the upper surface of the bar where the load is applied, compressive forces result in compressive strain of the material, whereas on the lower surface tensile forces that form result in tensile strain, or elongation of the material (Figure 29).

Figure 29: Distribution of Compressive and Tensile forces in Three-point bending Flexural Test



Dispersion of intrinsic and extrinsic flaws, however, leads to a range of strength values. Strength results may be affected by many practical aspects of testing, such as stressing rates, stress concentration, specimen size, elastic limitations, and failure mode [Kelly 1995]. Strength results by themselves cannot explain all clinical findings either. In ceramic crowns, for instance, it has been shown that failure initiates from the cementation surface, and not the occlusal surface which is under the highest tensile forces [Kelly 1995].

Griffith also showed that strength is sensitive to microcrack-like defects, and therefore, is not an intrinsic material property [Griffith 1920]. Proper extrapolation of strength data for predicting the clinical behaviour of a material requires that: 1. critical flaws in test specimens replicate those involved with clinical failure, 2. environmental factors are replicated in the lab, 3. failure parameters regarding flaw size distribution and crack growth rates are known, and 4. stress distributions in clinical situations are well characterized [Kelly 1995].

In most strength tests, the crack or flaw size at the fracture origin is not controlled or measured [Mecholsky 1995]. Since strength is dependant on crack size, handling and finishing procedures lead to randomly dispersed intrinsic and extrinsic flaws that introduce discrepancies in measurements of strength. Strength alone does not provide sufficient information to decide whether or not a treatment process has enhanced the resistance to fast fracture [Mecholsky 1995]. In a clinical situation, a material with low flexural strength will end up with tooth-restorative margin and surface defects due to Mode I and Mode II wear under high tensile forces, such as the case for Class II restorations. For a Class V restoration, which experiences minimal tensile forces on its surface, a material with low flexural strength may be adequate ( e.g. GICs).

Measurement of the elastic modulus of a material is important in relation to anticipated longevity of a restoration [Burke et al. 2002]. Elastic modulus measures stiffness of a material and represents intrinsic properties of that material. It is important for restorative materials to have moduli similar to those of the tooth tissues that they replace [Burke et al. 2002]. A material with a low elastic modulus undergoes a significant deformation, and could fail at low stresses, leading to marginal gaps at the restoration-tooth interface and increased rate of surface dissolution, thus causing eventual microleakage and premature failure [Tyas 1990]. In restorations where different layers of materials are used on dentin, such as liners, bases, and “flowable” composite resins prior to placement of the restoration, a significant difference between the stiffness of the layers could lead to stripping of the layers and eventual failure of the restoration. A material with a very low modulus placed adjacent to a pulp horn may induce a “hydraulic pump” action as a result of its elastic deformation, leading to hydraulic pressure on dentinal tubules and eventual pulpal pathology. For a Class V restoration, however, a material with low modulus may perform satisfactorily as it can elastically deform to compensate for tooth flexures under masticatory loading.

The fracture toughness, which measures the resistance of a material to the propagation of a crack, is also an intrinsic property of a material [Burke et al. 2002]. As mentioned earlier,

toughness can be measured by either the critical stress intensity factor,  $K_{IC}$ , fracture energy,  $\gamma_C$ , or critical strain energy release rate,  $G_C$ .  $K_{IC}$  is measured in units of stress-(distance)<sup>1/2</sup>, or MPa.m<sup>1/2</sup>, while  $\gamma_C$  and  $G_C$  are measured in energy per unit area, or J/m<sup>2</sup>. In a clinical situation, sharp cavity line angles, such as the pulpal-axial line angle, lead to increased stress intensity in the region leading to crack initiation and propagation. Surface wear, chipping, and marginal breakdown have shown a strong negative correlation with fracture toughness [Tyas 1990]. This appears reasonable as fracture toughness is a measure of the material's brittleness, and at high stresses materials with higher fracture toughness perform best.

Ferracane and Condon also showed an excellent inverse correlation ( $r^2=0.914$ ) between marginal breakdown and  $K_{IC}$  of tested composites [Ferracane and Condon 1999]. This means that 91% of the variation in marginal breakdown is explained by  $K_{IC}$ . Fracture toughness measurements may also be used to explain the fatigue crack propagation path across dentin, dentino-enamel junction, and enamel [Dong and Ruse 2003]. This has important implications on the role of the bonding or luting agents used.

Although each one the aforementioned tests by itself may explain the clinical behaviour of a material to a certain extent, an overall consideration of all the measurements is essential. By using the three-point bend (TPB) test, De Groot and colleagues showed that the combination of elastic modulus, flexural strength, and fracture toughness provided the best prediction for deflection and load at failure as opposed to each property by itself [De Groot et al. 1988]. Lewis also showed that the best prediction of wear requires consideration of all values for fracture toughness, flexural strength, modulus of elasticity, and resilience [Lewis 1993].

This review of the clinical relevance of some mechanical tests will allow for the correlation of the findings of this study with the clinical situation. Inherent differences in test methodology, however, make it impractical to draw direct comparisons between various reports of mechanical properties of dental restorative materials. In addition, many commercial products that have been developed for identical clinical purposes, behave quite differently with respect to their mechanical properties [Cattani-Lorente et al. 1993; Sidhu and Watson 1995]. The clinician is, therefore, advised to await the results of long-term clinical trials prior to employing newly marketed dental materials.

### **1.11 Aims of Study**

The aims of this study were: 1) to determine the effect of time on the fracture toughness ( $K_{IC}$ ), flexural strength ( $\sigma_f$ ), and flexural modulus ( $E_f$ ) of nine direct restorative

materials; and 2) to compare the selected properties of the material at each time. The null hypotheses tested were:

- 1) **Null Hypothesis 1**,  $H_0$ : there is no difference between the 1-hour, 1-day, 7-day, 30-day, and 90-day values of each of the  $K_{IC}$ ,  $\sigma_f$ , and  $E_f$  of the selected materials, and
- 2) **Null Hypothesis 2**,  $H_0$ : there is no difference between the selected materials with respect to their mechanical properties tested at each time.

## Chapter II. Materials

### 2.1 Selection of Materials

The materials of interest were those representing different groups of materials used commonly in paediatric restorative dentistry. The materials selected for this study were, therefore, KetacFil (KF, ESPE) – a conventional GIC; PhotacFil (PF; ESPE) - a RMGIC; Dyract Aplicap (DAP) (3M) and F2000 (3M) - PAMCR or "compomers"; Z100 (3M) -a hybrid composite; Heliomolar (HM; Vivadent) – a microfil composite; SureFil (SF; Dentsply) and Prodigy Condensable (PC; Kerr) "packable" composites; and Valiant PHD XT (VPHD; Ivoclar) - a phase dispersed unicompositional amalgam (Table 3, and Figure 30).

TABLE 3: Experimental Materials

Material	Abv.	Class	Manufacturer	Lot (Kic)	Lot ( $\sigma_f$ , $E_f$ )
Ketac-Fil	KF	GIC	ESPE	FW0045611-2000-04	FW0045611-2000-04
Photac-Fil	PF	RMGIC	ESPE	FW0046394	FW0048539, FW0046394
Dyract AP	DAP	PAMCR	Dentsply	9707000947, 9707000946, 9708000343, 9708000782, 9809000144	9809000144, 9812000362
F2000	F2000	PAMCR	3M	7AM-1999-09	7AM-1999-09
Z100	Z100	Hybrid CR	3M	8UR-2000-11	8UR-2000-11
Heliomolar	HM	Microfill CR	Vivadent	(1hr) D3-825793-2001-12 (1d) A4-809738-2001-05 (7d) D3-825793-2001-12 (30d) A3-905056-2002-02 (90d) A3-905056-2002-02	822577-2001-10, 905056-2002-02
SureFil	SF	"Packable"	Dentsply	(1h) 980910 (1d) 980616, 880616 (7d) 980910 (30d) 880616, 980616 (90d) 980616	980910-2001-09
Prodigy C	PC	"Packable"	KERR	810679-2001-10, 811014- 2001-10, 810859-2001-10, 810876-2001-10, 810370- 2001-10	810370-2001-10, 810679-2001-10, 810859-2001-10, 811000-2001-10, 810681-2001-10, 81082-2001-10, 811682-2001-10

Valiant PHD XT	VPH D	Amalgam	Ivoclar	7959462XA1110Z**+\$	**+\$7859277,C0814C*
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## 2.2 Chemistry of Selected Materials

In order to be able to understand the contribution of structural differences between the materials on the observed mechanical properties and the effect of ageing on such properties, it is imperative to have a sound knowledge of the structural components and setting characteristics of the selected materials. Table 4 summarizes the basic constituents, setting characteristics, clinical characteristics, and clinical applications of the experimental materials [Quiroz 1986; Ruse 1999; Titley and Kulkarni 2003].

Figure 30: Groups and Representative Materials Tested

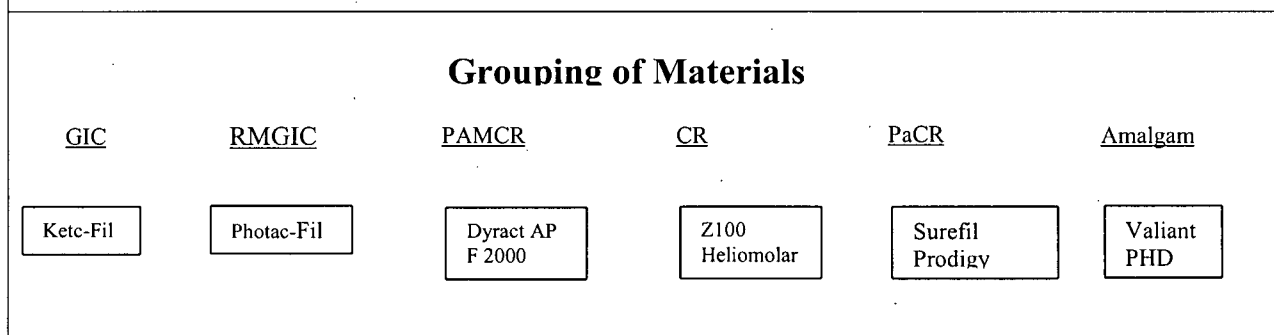


Table 4: Material constituents, setting characteristics, clinical characteristics, and clinical applications				
Material	Basic Constituents	Setting Characteristics	Clinical Characteristics	Clinical Applications
Ketac-Fil (ESPE)	Encapsulated Glass powder and liquid acid, polysalt gel matrix, calcium aluminum fluorosilicate glass, and acrylic and maleic acid copolymer, 3.91 micron particle size, 40-50 micron powder particle size	Acid base reaction	Biocompatibility, aesthetic, adhesion to tooth structure, fluoride leaching, anti-cariogenic, same coefficient of thermal expansion as the tooth, minimal tooth removal, may be placed in a moist environment, minimal microleakage, brittle and sensitive to dehydration	Class I and low stress bearing areas, Class V lesions, Atraumatic Restorative Treatment technique
Photac-Fil (ESPE)	Encapsulated Glass powder and light-sensitive liquid acid, calcium aluminum fluorosilicate glass, photo-initiators, acrylic and maleic acid copolymer, hydrophilic monomers (MA, HEMA), 3.81 microns particle size	Photopolymerization, Acid base reaction, addition polymerization	Similar to GICs, "command-set", improved physical properties, reduced sensitivity, improved esthetics, less water sensitive than conventional GICs, more sensitive to moisture during setting, polymer shrinkage of resin component, potential for microleakage	Class I and low stress bearing areas, Class II and III in primary dentition, luting, Atraumatic Restorative Treatment of anterior teeth
Dyract AP (Dentsply)	one component, dimethacrylate monomers, filler glass partially silanized, strontium-fluoro-silicate glass, strontium fluoride, photo-initiators and stabilizers, 1.89 microns particle size	Photopolymerization, and questionable acid-base reaction	similar properties to composites, considered a weak composite, mechanical bond to tooth structure, wear at the same rate as enamel in primary dentition, some fluoride release, anticariogenic, technique sensitive, polymerization shrinkage, potential for microleakage, reduced bond to tooth structure	Class I, II , III and low stress bearing areas, promoted strongly for proximal restorations in primary teeth
F2000 (3M)	one component; filler is made up of colloidal silica and fluorosalluminosilicate glass of 84% weight; matrix of citric dimethacrylate oligomer, glycerol dimethacrylate, hydrophilic monomer, and camphorquinone-amine photoinitiator; particle size of 3 microns	Photopolymerization and questionable acid-base reaction	similar properties to composites, considered a weak composite, mechanical bond to tooth structure, wear at the same rate as enamel in primary dentition, some fluoride release, anticariogenic, technique sensitive, polymerization shrinkage, potential for microleakage, reduced bond to tooth structure	Class I, II , III and low stress bearing areas, promoted strongly for proximal restorations in primary teeth
Z100 (3M)	one component, zirconia/silica filler, Bis-GMA and TEGDMA monomers, 0.8 microns particle size (0.01-3.5 microns), Vf by vol% 66, Wt% 84.5	photopolymerization	Aesthetic, bonds to tooth structure, minimal removal of sound tooth structure, very technique sensitive, very moisture sensitive, no anticariogenic properties, polymerization shrinkage, polymerization conversion of 80% or less, prone to microleakage, unsuitable for large caries and multisurface lesions	Versatile, used for both anterior and posterior dentition

Material	Basic Constituents	Setting Characteristics	Clinical Characteristics	Clinical Applications
Heliomolar (Vivadent)	one component, colloidal pyrogenic silica and ytterbium trifluoride fillers, Bis-GMA, UDMA, and DCDMA matrix, 0.04 microns particle size, 79% Vf by weight, 49.1 to 68.5% Vf by volume	photopolymerization	Aesthetic, bonds to tooth structure, minimal removal of sound tooth structure, very technique sensitive, very moisture sensitive, no anticariogenic properties, polymerization shrinkage, polymerization conversion of 80% or less, prone to microleakage, unsuitable for large caries and multisurface lesions	Versatile, used for both anterior and posterior dentition
SureFil (Dentstply)	one component, fillers are amorphous fumed silica and barium boron fluoroalumino silicate; matrix is urethane modified Bis-GMA dimethacrylate and polymerizable dimethacrylate resin, Vf vol% 60-68%, Wt% 82, particle size diverse 0.04 to 10 microns	photopolymerization	Same as Z100 and Heliomoar, but also increased polymerization shrinkage compared to conventional composites, higher viscosity renders them more prone to voids	Recommended for multisurface posterior restorations, box in Class II
Prodigy C (Kerr)	one component, silica fillers 80% by weight, and 62% by volume, 0.06 microns particle size, 2.2% volume shrinkage, 1.8% axial shrinkage, matrix of dimethacrylate resin	photopolymerization	Same as Z100 and Heliomoar, but also increased polymerization shrinkage compared to conventional composites, higher viscosity renders them more prone to voids	Recommended for multisurface posterior restorations, box in Class II
Valiant PHD (Ivoclar)	A phase-dispersed unicompositional alloy; two component: Ag-Sn-Cu-Zn powder filler, and Hg liquid and matrix; 50% Vf by weight, Hg matrix; set amalgam for low copper alloy has $\gamma_1$ (Ag <sub>2</sub> Hg <sub>3</sub> ) and $\gamma_2$ (Sn <sub>7</sub> -8Hg), and unconsumed powder particles; for low copper amalgam, $\gamma_1$ , $\eta$ (Cu <sub>6</sub> Sn <sub>5</sub> ), and unconsumed alloy particles	For low copper: $(\beta+\gamma) + \text{Hg} \rightarrow \gamma_1 + \gamma_2 + (\beta+\gamma)$ For high copper admix: $(\beta+\gamma) + \text{Ag-Cu eutactic} + \text{Hg} \rightarrow \gamma_1 + \eta + \text{unconsumed alloy of both types of particles}$ For high copper unicompositional: Ag-Sn-Cu alloy particles + $\text{Hg} \rightarrow \gamma_1 + \eta + \text{unconsumed alloy particles}$	Least technique sensitive, self sealing, initial microleakage with low copper alloys, economical, durable, may be placed in a wet environment, unesthetic, discolours dentin, no anticariogenic properties, no bond to tooth structure	Posterior dentition with sufficient dental tissue for mechanical retention

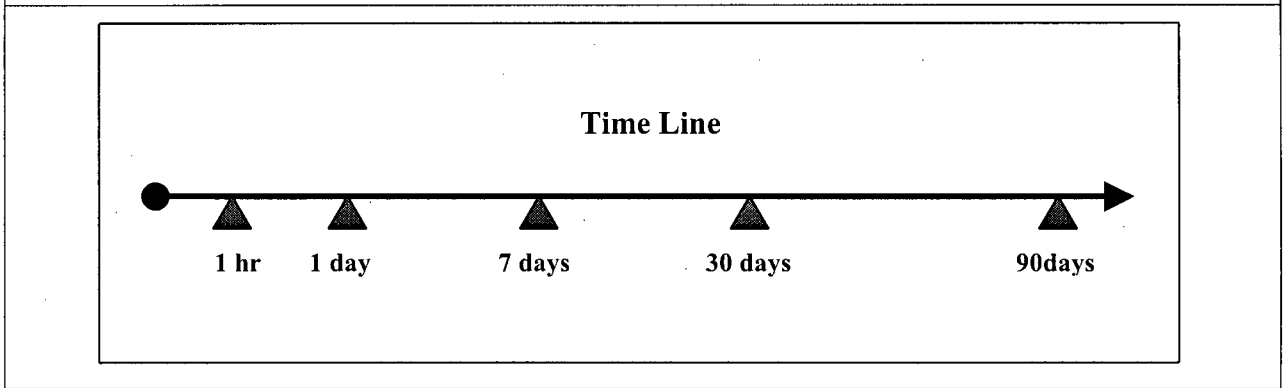


## Chapter III. Methods

### 3.1 Sample Preparation

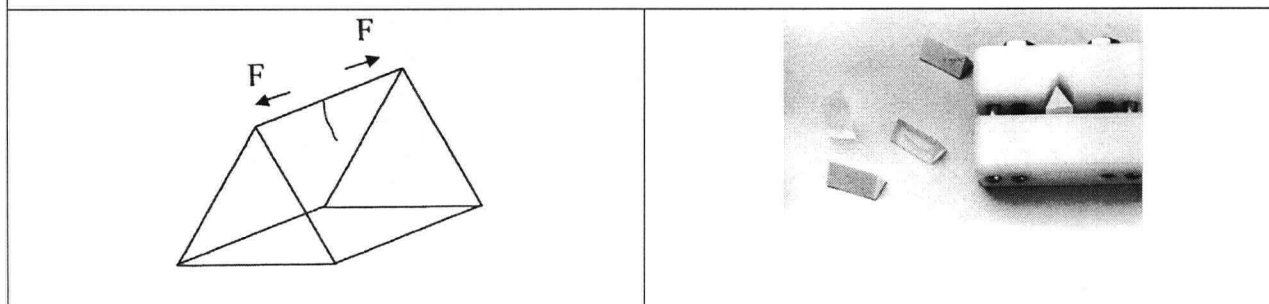
Twelve samples were fabricated for each test and for each of the five time intervals selected (1-hour, 1-day, 7-day, 30-day, and 90-day) (Figure 31). After fabrication, the samples were stored in distilled water at 37° C until testing. For Valiant PHD, only 1-day and 90-day samples were fabricated as a reference for short term and long term comparison. The selected time line was indicative of immediate placement, 1-hour, short term aging when a restoration is expected to function fully, 1-day, short term aging, 7-day, and long term aging, 30-day and 90-day.

Figure 31: Time Line for Testing of Materials



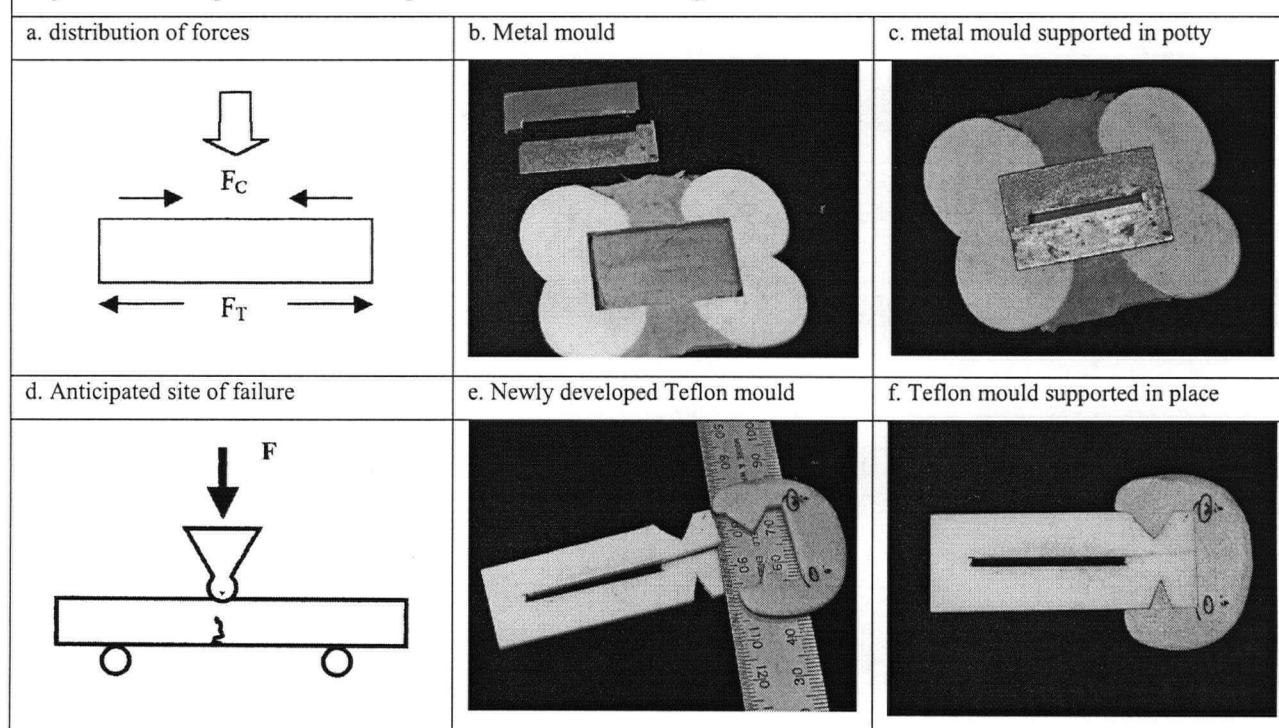
The fracture toughness was determined using the NTP specimen  $K_{IC}$  test [Ruse et al. 1996]. Briefly, the restorative materials were loaded in Teflon moulds to obtain 6x6x6x12 mm NTP specimens (Figure 32). For the photopolymerizable materials, the specimen were light-cured for 40-seconds from either end prior to removal from the mould, upon which each of its plane surfaces were cured for 40 seconds. Bulk curing was used as it was not anticipated that there is any significant difference in  $K_{IC}$  of composite resins between multi-layer curing and bulk-curing [Kovarik and Ergle 1993].

Figure 32: Fabrication of NTP Specimen for Fracture Toughness Test



Short bars for the TPB test were prepared (Figure 33). Special Teflon moulds were developed for this test, in order to minimize forces associated with the removal of specimens from the metallic moulds and to avoid using lubricants, which had the potential of interfering with the setting reaction of selected materials. The restorative materials were loaded in Teflon moulds to obtain 2x2x25 mm rectangular bars.

Figure 33: Three-point Bend Test Specimen for Flexural Strength Measurement



The Teflon mould was supported from both sides by glass slabs covered with a layer of Mylar sheet. For Ketac-Fil, samples were allowed to set for 10 minutes before removal from the mould and transfer to 37° C distilled water. For Valiant PHD, samples were removed from the mould 30 minutes after trituration and transferred to 37° C distilled water. For all other materials,

including Photac-Fil, the specimens were transferred to 37° C distilled water immediately after the completion of light curing.

### 3.2 Mechanical Tests

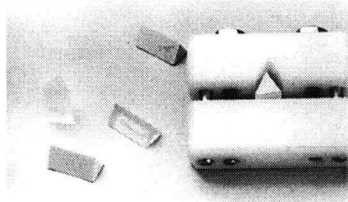
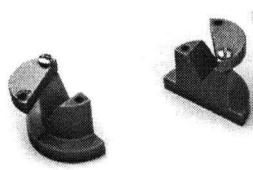
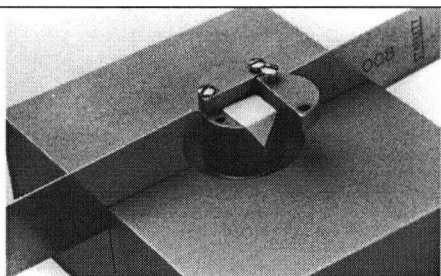
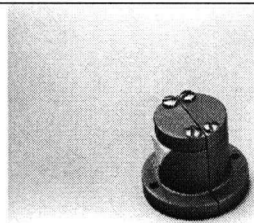
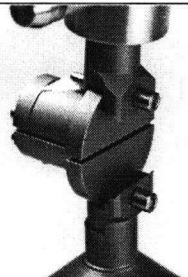
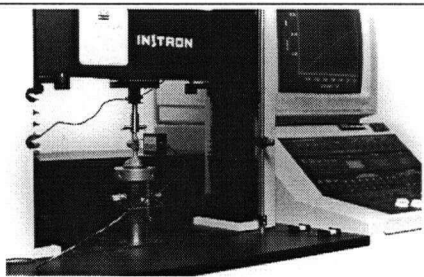
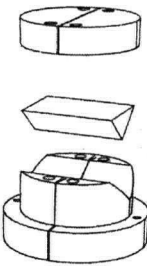
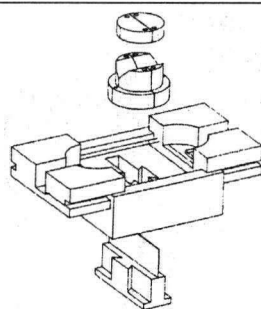
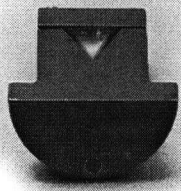
#### 3.2.1 Fracture Toughness Test

Prior to mounting samples for testing, a small crack initiate (~ 100 µm deep) was introduced on one of the edges of the triangular prism using a Bard Parker blade, under magnification. The samples were then placed in a specimen holder with a 200 µm separation between jig components centred around the initiate. The specimen-holder complex was then mounted onto an Instron 4301 Universal Testing machine (Instron Canada, Burlington, ON) and loaded in tension at a crosshead speed of 0.1 mm/min (Figure 34). The maximum load recorded before crack arrest or complete failure was used to calculate  $K_{IC}$  in  $\text{MPa}\cdot\text{m}^{1/2}$  using the formula:

$$K_{IC} = Y_{\min}^* \frac{P_{\max}}{DW^{1/2}}$$

where:  $P_{\max}$  = maximum load recorded during testing;  $D$  = specimen diameter (= 12 mm);  $W$  = specimen length (= 10.5 mm);  $Y_{\min}^*$  = the dimensionless stress intensity factor coefficient minimum (equal to 28 for the NTP test) [Ruse et al. 1996]. Samples that did not satisfy plane-strain conditions by having the crack propagate in a longitudinal direction, forming two equal halves, were eliminated [Mueller 1990].

Figure 34 a-i: Notchless-Triangular Prism Specimen for the Measurement of Fracture Toughness

a. Teflon Mould	b. Specimen Holder	c. Specimen Holder Separation
		
d. Specimen Holder Closed	e. Mounted Specimen Holder for testing	f. Instron Machine for testing
		
g. Diagrammatic Assembly	h. Specimen Holder Jig Complex	i. Fractured Specimen after Testing
		

### 3.2.2 Three-Point Bending Flexural Test

The flexural strength and modulus were determined using a TPB test (ISO 9917-1, 1999). For testing, the samples were placed on two 1.5 mm diameter cylindrical supports separated by a span of 20 mm and loaded in compression at a crosshead speed of 1 mm/min (Figure 33-d). The maximum load recorded at failure was used to calculate  $\sigma_f$  in MPa using the formula:

$$\sigma_f = \frac{3PL}{2bt^2}$$

where P = maximum load; L = span between supports; b = specimen width; t = specimen thickness.

The slope ( $\delta P/\delta d$ ) of the straight portion of the load-displacement curve obtained during the TPB test was used to calculate the flexural modulus in MPa using the formula:

$$E_f = \frac{L^3}{4bt^3} \frac{\delta P}{\delta d}$$

where L, B, and t are as defined previously.

### 3.3 Statistical Analysis

Data associated with samples that did not follow the crack initiate for the  $K_{IC}$  test, or did not exhibit a linear portion on the plot for the flexural TPB test, were excluded from analysis as they were deemed to not satisfy the validity conditions of the tests.

The results were subjected to statistical analysis using SPSS Version 10.0 for Windows. The results were analyzed using one-way analysis of variance (ANOVA) followed by Scheffe multiple means comparisons ( $\alpha=0.05$ ) to identify significant differences.

## Chapter IV. Results

The means and associated standard deviations of all the results are summarized in Table 9. Tables-5 and Table-6 summarize the effect of time on each of the mechanical properties, i.e. fracture toughness, flexural strength, and flexural modulus, for each material, as box plots, along with the results of the statistical analysis.

### 4.1 Effect of Time on the Mechanical Properties of Each Material (Null Hypothesis 1)

#### 4.1.1 Effect of Time on Fracture Toughness.

The analysis of the  $K_{IC}$  results has shown that:

- For Ketac-Fil, 1hr = 1d < 1d = 7d = 30d = 90d. The lowest fracture toughness for Ketac-Fil was recorded at 1-hour while the highest value was recorded at 7d. Time had an enhancing effect on  $K_{IC}$  of KF for 1-d, which stabilized thereafter.
- For Photac-Fil, 1d = 7d = 30d < 1hr = 7d = 30d = 90d. There was a decrease in  $K_{IC}$  at 1d compared to the 1-hour value. There were, however, no differences between the 1d, 7d and 30d values, and between the 1-hour, 7d, 30d, and 90d values. Time resulted in an initial decrease in  $K_{IC}$  to the 1d level, and thereafter an increase to the original value at 1 hour.
- For Dyract AP, 1d = 90d = 30d < 90d = 30d = 7d < 30d = 7d = 1hour. At 1-hour  $K_{IC}$  was the highest. Time had an overall deleterious effect on  $K_{IC}$ .
- For F2000, 30d = 7d = 1d = 90d < 90d = 1hour. At 1-hour  $K_{IC}$  was the highest. Time had a deleterious effect on  $K_{IC}$ , until 90d when it was equal to the 1hour value.
- For Z100, 1hour = 1d = 7d = 30d = 90d. There were no significant differences for  $K_{IC}$  of Z100 over time.
- For Heliomolar, 90d = 1hour < 30d = 1d = 7d. The 90-day and 1-hour results were significantly lower than all other groups, which were not significantly different from one another. Time resulted in an increase in  $K_{IC}$ , which subsided to the 1-hr value after 90d.
- For SureFil, 1hr = 1d = 7d = 30d = 90d. Time had no significant effect on  $K_{IC}$  of SureFil.
- For Prodigy C, 1hour = 90d = 30d < 30d = 7d = 1d. There were no differences between neither the 1-hour, 30-days, and 90-days results, nor the 1-day, 7-days, and 30-days results. Time resulted in an initial increase in  $K_{IC}$ , which subsided over 30-day to 90-day.
- For Valiant PHD XT, the 1-day results were significantly lower than the 90-day ones. Time resulted in an increase in  $K_{IC}$  of Valiant PHD.

#### 4.1.2 Effect of Time on Flexural strength.

The analysis of the Flexural Strength ( $\sigma_f$ ) results has shown that:

- For Ketac-Fil, 1hour = 30d < 30d = 1d < 1d = 90d < 90d = 7d. The lowest flexural strength for Ketac-Fil was recorded at 1-hour, while the highest value was recorded at 7-day and 90-day. Time had a positive effect on  $\sigma_f$  of Ketac-Fil.
- For Photac-Fil, 1hr < 90 d = 30d = 1d < 30d = 1d = 7d. The lowest flexural strength for Photac-Fil was recorded at 1-hour. Overall, Time resulted in an initial increase in  $\sigma_f$  of Photac-Fil, which remained the same over time.
- For Dyract AP, 90d = 1hour = 30d < 1hr = 30d = 7d < 7d = 1d. The lowest value was at 90d = 1hour = 30d. 1d and 7d had the highest values. After an initial increase, time resulted in an overall reduction of  $\sigma_f$  of Dyract-AP to its 1hr value.
- For F2000, 90d = 1hour = 7d < 1hr = 7d = 30d < 30d = 1d. After an initial increase, over time the values of  $\sigma_f$  returned back to the initial values at 1hour.
- For Z100, 90d = 1hr = 30dr < 1hr = 30d = 1d < 30d = 1d = 7d. The 7-day values were the highest and the 90-day ones the lowest. After causing an initial increase, time resulted in the return of  $\sigma_f$  to the initial value at 1-hour.
- For Heliomoar, 1hour < 1d = 7d = 30d = 90d. The 1hour value was significantly lower than all other values, which were statistically the same. Time resulted in an initial increase on the  $\sigma_f$  of Heliomolar, which remained the same after 1d.
- For SureFil, 1hour < 1d = 7d = 30d = 90d. The 1hour value was significantly lower than all other values, which were statistically the same. Time had an enhancing effect by 1d, and thereafter a stable effect on the  $\sigma_f$  of SureFil.
- For Prodigy, 1hr = 30d < 30d = 90d = 7d < 90d = 7d = 1d. The highest values were recorded for 1d, 7d, and 90d. Time resulted in an initial increase in and then somewhat stable  $\sigma_f$  for Prodigy.
- For Valiant PHD XT, there was no difference between the 1-day and 90-days  $\sigma_f$  results.

#### 4.1.3 Effect of Time on Flexural modulus.

The analysis of the  $E_f$  results has shown that:

- For Ketac-Fil, 1hr = 1d < 1d = 90d < 90d = 7d = 30d. Time resulted in an increase in  $E_f$  of Ketac-Fil, which dropped slightly after reaching the 7d maximum value.

- For Photac-Fil, 1hr < 1d < 7d = 30d = 90d. Time resulted in an initial increase, which stabilized after 7d.
- For Dyract AP, 1hr < 7d = 30d < 30d = 1d = 90d. The lowest value was at 1hr, while the highest value was at 90d. Time resulted in an initial increase and thereafter a somewhat stable  $E_f$ .
- For F2000, 1hr < 1d = 7d < 7d = 90d < 90d = 30d. The lowest value was for 1hr. There was an initial increase and thereafter a somewhat stable  $E_f$ .
- For Z100, 1hr = 1d = 90 d < 1d = 90d = 7d = 30d, 1hr < 7d = 30d. The lowest value was for 1hr. Time resulted in a slight increase by 7d, and no significant change till after 90d, where the 90d value dropped to the 1hr and 1 day values.
- For Heliomolar, 1hr < 1d = 90d < 90d = 7d = 30d. The lowest value for  $E_f$  was for 1hr. Time resulted in a slight increase by 1d, and no significant change till after 90d, where the 90d value dropped to the 1d value. Overall, Time had an enhancing effect on  $E_f$  of Heliomolar.
- For SureFil, 1hr < 1d = 7d = 90d < 7d = 90d = 30d. Time resulted in an initial increase in  $E_f$ , which was stable thereafter.
- For Prodigy C, 1hr < 1d = 30d = 7d < 90d. Time resulted in an initial increase, by 1d, which was stable till 90d, when a further increase was significant. Overall, Time had an enhancing effect on  $E_f$  of Prodigy C.
- For Valiant PHD XT, 1d = 90d. Time had no significant effect on  $E_f$  of Valiant PHD between 1d and 90d.



<b>Table 5: Summary of Comparative Analysis for Fracture Toughness, Flexural Strength, and Flexural Modulus of Each Material Over Time</b>				
<b>No.</b>	<b>Material</b>	<b><math>K_{IC}</math></b>	<b><math>\sigma_f</math></b>	<b><math>E_f</math></b>
<b>1</b>	<b>Ketac-Fil (KF)</b>	1hr=1d< 1d=7d=30d=9d	1hr=30d< 30d=1d< 1d=90d< 90d=7d	1hr=1d< 1d=90d< 90d=7d=30d
<b>2</b>	<b>Photac-Fil (PF)</b>	1d=30d=7d< 30d=7d=1hr=90d	1hr< 90d=30d=1d< 30d=1d=7d	1hr< 1d< 7d=30d=90d
<b>3</b>	<b>Dyract AP (DyAP)</b>	1d=90d=30d< 90d=30d=7d< 30=7d=1hr	90d=1hr=30d< 1hr=30d=7d< 7d=1d	1hr< 7d=30d< 30d=1d=90d
<b>4</b>	<b>F2000</b>	30d=7d=1d=90d< 90d=1hr	90d=1hr=7d< 1hr=1d=30d< 30d=1d	1hr< 1d=7d< 7d=90d< 90d=30d
<b>5</b>	<b>Z100</b>	30d=90d=1d=7d=1hr	90d=1hr=30d< 1hr=30d=1d< 30d=1d=7d	1hr=1d=90d< 1d=90d=7d=30d
<b>6</b>	<b>Heliomolar (HM)</b>	1hr=90d< 30d=1d=7d	1hr< 30d=1d=90d=7d	1hr< 1d=90d< 90d=7d=30d
<b>7</b>	<b>SureFil (SF)</b>	90d=1d=30d=7d=1hr	1hr< 1d=7d=30d=90d	1hr< 1d=7d=90d< 7d=90d=30d
<b>8</b>	<b>Prodigy C (PrC)</b>	1hr=90d=30d< 30d=7d=1d	1hr=30d< 30d=90d=7d< 90d=7d=1d	1hr< 1d=30d=7d< 90d
<b>9</b>	<b>Valiant PHD XT (ValPHD)</b>	1d< 90d	1d=90d	1d=90d

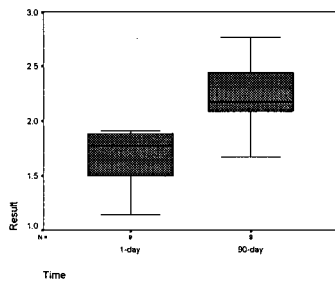
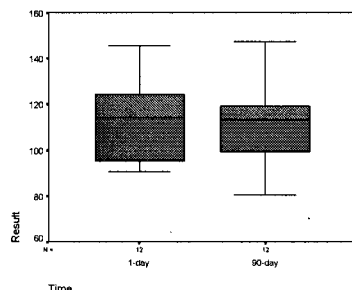
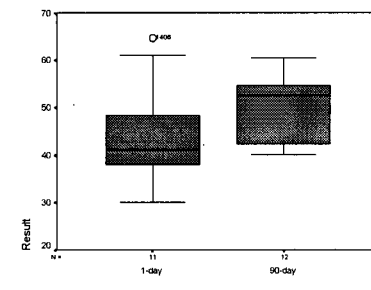
**Table 6: Effect of Time on Mechanical Properties of Materials**

Time	KIC	$\sigma_f$	$E_f$																																																																																																																								
KF																																																																																																																											
ANOVA	<p>ANOVA-KIC of KetacFil at 5 Times</p> <table><tr><th>Result</th><th>Sum of Squares</th><th>df</th><th>Mean Square</th><th>F</th><th>Sig.</th></tr><tr><td>Between Groups</td><td>300</td><td>4</td><td>7.508E-02</td><td>10.069</td><td>.000</td></tr><tr><td>Within Groups</td><td>298</td><td>40</td><td>7.457E-03</td><td></td><td></td></tr><tr><td>Total</td><td>599</td><td>44</td><td></td><td></td><td></td></tr></table>	Result	Sum of Squares	df	Mean Square	F	Sig.	Between Groups	300	4	7.508E-02	10.069	.000	Within Groups	298	40	7.457E-03			Total	599	44				<p>ANOVA-FlexStr of KetacFil at 5 Times</p> <table><tr><th>Result</th><th>Sum of Squares</th><th>df</th><th>Mean Square</th><th>F</th><th>Sig.</th></tr><tr><td>Between Groups</td><td>2810.032</td><td>4</td><td>702.508</td><td>22.897</td><td>.000</td></tr><tr><td>Within Groups</td><td>1687.504</td><td>55</td><td>30.682</td><td></td><td></td></tr><tr><td>Total</td><td>4497.536</td><td>59</td><td></td><td></td><td></td></tr></table>	Result	Sum of Squares	df	Mean Square	F	Sig.	Between Groups	2810.032	4	702.508	22.897	.000	Within Groups	1687.504	55	30.682			Total	4497.536	59				<p>ANOVA-FlexMod of KetacFil at 5 Times</p> <table><tr><th>Result</th><th>Sum of Squares</th><th>df</th><th>Mean Square</th><th>F</th><th>Sig.</th></tr><tr><td>Between Groups</td><td>557.164</td><td>4</td><td>139.291</td><td>13.087</td><td>.000</td></tr><tr><td>Within Groups</td><td>585.375</td><td>55</td><td>10.643</td><td></td><td></td></tr><tr><td>Total</td><td>1142.538</td><td>59</td><td></td><td></td><td></td></tr></table>	Result	Sum of Squares	df	Mean Square	F	Sig.	Between Groups	557.164	4	139.291	13.087	.000	Within Groups	585.375	55	10.643			Total	1142.538	59																																																			
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Scheffe Multiple means Comp.	<p>Result-KIC of KetacFil at 5 Times</p> <p>Scheffe<sup>a,b</sup></p> <table><tr><th>Time</th><th>N</th><th colspan="2">Subset for alpha = .05</th></tr><tr><td></td><td></td><td>1</td><td>2</td></tr><tr><td>1-hour</td><td>8</td><td>.179673</td><td></td></tr><tr><td>1-day</td><td>7</td><td>.262731</td><td>.262731</td></tr><tr><td>30-day</td><td>10</td><td></td><td>.377910</td></tr><tr><td>90-day</td><td>11</td><td></td><td>.386873</td></tr><tr><td>7-day</td><td>9</td><td></td><td>.387156</td></tr><tr><td>Sig.</td><td></td><td>.411</td><td>.078</td></tr></table> <p>Means for groups in homogeneous subsets are displayed a. Uses Harmonic Mean Sample Size = 8.774. b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.</p>	Time	N	Subset for alpha = .05				1	2	1-hour	8	.179673		1-day	7	.262731	.262731	30-day	10		.377910	90-day	11		.386873	7-day	9		.387156	Sig.		.411	.078	<p>Result-FlexStr of KetacFil at 5 Times</p> <p>Scheffe<sup>a</sup></p> <table><tr><th>Time</th><th>N</th><th colspan="4">Subset for alpha = .05</th></tr><tr><td></td><td></td><td>1</td><td>2</td><td>3</td><td>4</td></tr><tr><td>1-hour</td><td>12</td><td>21.530833</td><td></td><td></td><td></td></tr><tr><td>30-day</td><td>12</td><td>25.355000</td><td>25.355000</td><td></td><td></td></tr><tr><td>1-day</td><td>12</td><td></td><td>31.056667</td><td>31.056667</td><td></td></tr><tr><td>90-day</td><td>12</td><td></td><td>37.937500</td><td>37.937500</td><td></td></tr><tr><td>7-day</td><td>12</td><td></td><td>39.031667</td><td>39.031667</td><td></td></tr><tr><td>Sig.</td><td></td><td>.585</td><td>.190</td><td>.069</td><td>.993</td></tr></table> <p>Means for groups in homogeneous subsets are displayed. a. Uses Harmonic Mean Sample Size = 12.000.</p>	Time	N	Subset for alpha = .05						1	2	3	4	1-hour	12	21.530833				30-day	12	25.355000	25.355000			1-day	12		31.056667	31.056667		90-day	12		37.937500	37.937500		7-day	12		39.031667	39.031667		Sig.		.585	.190	.069	.993	<p>Result-FlexMod of KetacFil at 5 Times</p> <p>Scheffe<sup>a</sup></p> <table><tr><th>Time</th><th>N</th><th colspan="3">Subset for alpha = .05</th></tr><tr><td></td><td></td><td>1</td><td>2</td><td>3</td></tr><tr><td>1-hour</td><td>12</td><td>11.793750</td><td></td><td></td></tr><tr><td>1-day</td><td>12</td><td>12.844750</td><td>12.844750</td><td></td></tr><tr><td>90-day</td><td>12</td><td></td><td>16.127333</td><td>16.127333</td></tr><tr><td>7-day</td><td>12</td><td></td><td>18.630833</td><td>18.630833</td></tr><tr><td>30-day</td><td>12</td><td></td><td>19.315000</td><td>19.315000</td></tr><tr><td>Sig.</td><td></td><td>.960</td><td>.209</td><td>.236</td></tr></table> <p>Means for groups in homogeneous subsets are displayed. a. Uses Harmonic Mean Sample Size = 12.000.</p>	Time	N	Subset for alpha = .05					1	2	3	1-hour	12	11.793750			1-day	12	12.844750	12.844750		90-day	12		16.127333	16.127333	7-day	12		18.630833	18.630833	30-day	12		19.315000	19.315000	Sig.		.960	.209	.236
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## 4.2 Comparison of the Mechanical Properties of the Selected Materials at Each Time (Null Hypothesis number 2)

Tables 7 and 8 summarize the comparison between mechanical properties of materials at each time.

### 4.2.1 Comparison of Fracture Toughness of Materials

- At 1-hour: Valiant PHD was not tested at this time.  $KF < PF = F2000 = \text{Prodigy} = \text{HM} = \text{Z100} < \text{Prodigy} = \text{HM} = \text{Z100} = \text{Dyract} < \text{Dyract} = \text{SF}$ . Compared to other materials tested at 1-hour, KF had the lowest  $K_{IC}$  value, while SF and Dyract had the highest values. For inter- and intra-group comparisons,  $KF < PF$ ,  $PF = F2000 < \text{Dyract}$ ,  $\text{HM} = \text{Z100} = \text{Dyract}$ ,  $\text{Prodigy} < \text{SF}$ ,  $\text{HM} = \text{Z100} = \text{Prodigy} < \text{SF}$ .
- At 1-day:  $KF = PF = F2000 = \text{Dyract} < PF = F2000 = \text{Dyract} = \text{Z100} < \text{Z100} = \text{SF} < \text{SF} = \text{Valiant} = \text{Prodigy} = \text{HM}$ . For inter- and intra-group comparisons,  $KF = PF = F2000 = \text{Dyract}$ ,  $PF = F2000 = \text{Dyract} = \text{Z100} < \text{HM}$ , and  $\text{Z100} = \text{SF} < \text{SF} = \text{Valiant} = \text{Prodigy} = \text{HM}$ .
- At 7-day: Valiant PHD was not tested at this time.  $KF = F2000 = PF < F2000 = PF = \text{Z100} < \text{Z100} = \text{Dyract} < \text{Dyract} = \text{Prodigy} = \text{SF} < \text{Prodigy} = \text{SF} = \text{HM}$ . For inter- and intra-group comparisons,  $KF = PF = F2000 < \text{Dyract}$ ,  $F2000 = \text{Z100} < \text{Z100} = \text{Dyract} < \text{HM}$ , and  $\text{Z100} < \text{Prodigy} = \text{SF} = \text{HM}$ .
- At 30-day: Valiant PHD was not tested at this time.  $KF = F2000 = PF = \text{Z100} < F2000 = PF = \text{Z100} = \text{Dyract} < \text{Z100} = \text{Dyract} = \text{Prodigy} < \text{Dyract} = \text{Prodigy} = \text{SF} <$

Prodigy=SF=HM. For inter- and intra-group comparisons,  $KF=PF=F2000 < PF=F2000=Dyract, F2000=Dyract=Z100 < HM, Z100=Prodigy < Prodigy=SF=HM$ .

- At 90-day: Compared to other materials tested at 90-day, Valiant PHD had significantly the highest value of  $K_{IC}$ .  $KF=F2000 < F2000=PF=Z100=Dyract=HM < PF=Z100=Dyract=HM=Prodigy=SF < Valiant$ . For inter- and intra-group comparisons,  $KF < PF, KF=2000 < F2000 = PF = Dyract, F2000=Dyract=Z100= HM, Z100=HM=Prodigy=SF < Valiant PHD$ .

#### 4.2.2 Comparison of Flexural Strength of Materials

- At 1-hour: Valiant PHD was not tested at this time.  $KF < PF=HM < F2000=Prodigy=Dyract < Prodigy=Dyract=SF < Z100$ . For the materials tested at 1-hr, KF had the lowest value, while Z100 had the highest value for flexural strength. For inter- and intra-group comparisons,  $KF < PF < F2000=Dyract, HM < F2000=Dyract < Z100, HM < Prodigy=SF < Z100$ .
- At 1-day: Compared to other materials tested at 1-day, KF had the lowest value for flexural strength.  $KF < PF=Valiant < Valiant=HM=F2000 < SF=Prodigy=Dyract=Z100$ . For inter- and intra-group comparisons,  $KF < PF < F2000 < Dyract, F2000=HM < Dyract=Z100, HM < Z100=Prodigy=SF, Valiant < Dyract=Z100=Prodigy=SF$ .
- At 7-day: Valiant PHD was not tested at this time.  $KF < PF=F2000=HM < HM=Dyract=Prodigy=SF < Z100$ . In comparison to other materials tested at 7-day, KF had the lowest value, while Z100 had the highest value for flexural strength. For inter- and intra-group comparisons,  $KF < PF=F2000 < Dyract, F2000=HM < HM=Dyract < Z100, and HM=Prodigy=SF < Z100$ .
- At 30-day: Valiant PHD was not tested at this time.  $KF < PF=F2000=HM < F2000=HM=Dyract=Prodigy < SF=Z100$ . Compared to other materials, KF had the lowest value, while Z100 and SF had the highest values for flexural strength at 30-day. For inter- and intra-group comparisons,  $KF < PF=F2000 < F2000=Dyract, F2000=Dyract=HM < Z100, HM=Prodigy < SF=Z100$ .
- At 90-day: Amongst the materials tested, Valiant PHD had significantly the highest value for flexural strength at 90-day.  $KF=F2000 < F2000=PF=Z100=Dyract=HM < PF=Z100=Dyract= HM= Prodigy=SF < Valiant PHD$ . For inter- and intra-group comparisons,  $KF < PF, KF=F2000 < F2000=PF=Dyract, F2000=Dyract=Z100=HM, Z100=HM=Prodigy=SF < Valiant$ .

#### 4.2.3 Comparison of Flexural Modulus of Materials

- At 1-hour: Valiant PHD was not tested for this time.  $HM < PF = Dyract < Dyract = Prodigy < Prodigy = KF = SF = F2000 < Z100$ . Compared to other materials tested at 1-hour, HM had the lowest value, while Z100 had the highest value for flexural modulus. For inter- and intra-group comparisons,  $PF < KF$ ,  $Dyract = PF < KF = F2000$ ,  $HM < Dyract < F2000 < Z100$ , and  $HM < Prodigy = SF < Z100$ .
- At 1-day: In comparison to other materials tested at 1-day, Valiant PHD had the highest value of flexural modulus.  $HM = PF = Prodigy < PF = Prodigy = KF = Dyract = F2000 = SF < Prodigy = KF = Dyract = F2000 = SF = Z100 < Valiant PHD$ . For inter- and intra-group comparisons,  $KF = PF = F2000 = Dyract$ ,  $HM < Dyract = F2000 = Z100$ , and  $HM = Prodigy < Prodigy = SF = Z100$ .
- At 7-day: In comparison to other materials tested at 7-day HM had the lowest value for flexural modulus.  $HM < Dyract = Prodigy < PF = F2000 = SF = KF < SF = KF = Z100$ . For inter- and intra-group comparisons,  $KF = PF$ ,  $Dyract < F2000 = KF = PF$ ,  $HM < Dyract < F2000 < Z100$ , and  $HM < Prodigy < SF = Z100$ .
- At 30-day: Valiant PHD was not tested at this time. Amongst all the materials tested, HM had the lowest value for flexural modulus at 30-day.  $HM < Dyract = Prodigy = PF < KF = SF = Z100 = F2000$ . For inter- and intra-group comparisons,  $PF < KF$ ,  $PF = Dyract < F2000$ ,  $HM < Dyract < F2000 = Z100$ , and  $HM < Prodigy < SF = Z100$ .
- At 90-day: Compared to all the materials tested at 90-day, Valiant PHD had significantly the highest value, while HM had the lowest value for flexural modulus.  $HM < Dyract = Prodigy = PF = KF = SF = Z100 < Prodigy = PF = KF = SF = Z100 = F2000 < Valiant PHD$ . For inter- and intra-group comparisons,  $Dyract = KF = PF < KF = PF = F2000$ ,  $HM < Dyract = Z100 < Z100 = F2000$ ,  $HM < Prodigy = SF = Z100 < Valiant PHD$ .



**Table 7: Summary of Inter-group and Intra-group Comparative Analysis for Fracture Toughness, Flexural Strength, and Flexural Modulus at Each of Five Times**

Time	Group	$K_{IC}$	$\sigma_f$	$E_f$
<b>1-hour</b>	GIC-RMGIC KF vs. PF	KF < PF	KF<PF	PF<KF
	RMGIC-PAMCR PF vs. (F2000 vs. Dyract)	PF=F2000 < Dyract	PF<F2000=Dyract	Dyract=PF<KF=F2000
	PAMCR-HCR-MFCR (F2000 vs. Dyract) vs. (Z100 vs. HM)	F2000=HM=Z100 < HM=Z100=Dyract	HM<F2000=Dyract<Z100	HM<Dyract<F2000<Z100
	HCR-MFCR-PCR (Z100 vs. HM) vs. (SF vs. Prodigy)	HM=Z100=Prodigy<SF	HM<Prodigy=SF<Z100	HM<Prodigy=SF<Z100
<b>1-day</b>	GIC-RMGIC KF vs. PF	KF=PF	KF<PF	KF=PF
	RMGIC-PAMCR PF vs. (F2000 vs. Dyract)	PF=F2000=Dyract	PF<F2000<Dyract	PF=Dyract=F2000
	PAMCR-HCR-MFCR (F2000 vs. Dyract) vs. (Z100 vs. HM)	F2000=Dyract=Z100<HM	F2000=HM<Dyract=Z100	HM<Dyract=F2000=Z100
	HCR-MFCR-PCR (Z100 vs. HM) vs. (SF vs. Prodigy)	Z100=SF=Prodigy=HM	HM<SF=Prodigy=Z100	HM=Prodigy<Prodigy=SF=Z100
	All-Allogy All vs. Valiant PHD	SF=Prodigy=HM=Valiant, Val among the highest grp	Valiant<SF=Prodigy=Dyract=Z100	Valiant PHD has the highest value. All < Valiant
<b>7-day</b>	GIC-RMGIC KF vs. PF	KF=PF	KF<PF	KF=PF
	RMGIC-PAMCR PF vs. (F2000 vs. Dyract)	F2000=PF<Dyract	PF=F2000<Dyract	Dyract<PF=F2000
	PAMCR-HCR-MFCR (F2000 vs. Dyract) vs. (Z100 vs. HM)	F2000=Z100<Z100=Dyract < HM	F2000=HM<HM=Dyract<Z100	HM<Dyract<F2000<Z100
	HCR-MFCR-PCR (Z100 vs. HM) vs. (SF vs. Prodigy)	Z100<Prodigy=SF=HM	HM=Prodigy=SF<Z100	HM<Prodigy<SF=Z100
<b>30-day</b>	GIC-RMGIC KF vs. PF	KF=PF	KF<PF	PF<KF
	RMGIC-PAMCR PF vs. (F2000 vs. Dyract)	PF=F2000=Dyract	PF=F2000<F2000=Dyract	PF=Dyract<F2000
	PAMCR-HCR-MFCR (F2000 vs. Dyract) vs. (Z100 vs. HM)	F2000=Dyract=Z100<HM	F2000=Dyract=HM<Z100	HM<Dyract<F2000=Z100

	HCR-MFCR-PCR (Z100 vs. HM) vs. (SF vs. Prodigy)	Z100=Prodigy<Prodigy=SF=HM	HM=Prodigy<SF=Z100	HM<Prodigy<SF=Z100
<b>90-day</b>	GIC-RMGIC KF vs. PF	KF<PF	KF<PF	KF=PF
	RMGIC-PAMCR PF vs. (F2000 vs. Dyract)	PF=F2000=Dyract	PF=F2000=Dyract	PF=Dyract<F2000
	PAMCR-HCR-MFCR (F2000 vs. Dyract) vs. (Z100 vs. HM)	F2000=Dyract=Z100=HM	F2000=Dyract=HM<HM=Z100	HM<Dyract=Z100<Z100=F2000
	HCR-MFCR-PCR (Z100 vs. HM) vs. (SF vs. Prodigy)	Z100=HM=Prodigy=SF	HM=Z100=Prodigy<Z100=Prodigy=SF	HM<Prodigy=SF=Z100
	All-Allogy All vs. Valiant PHD	Valiant has significantly the highest value of all. All<Val.	PF=F2000=Dyract=Valiant, Valiant=HM< HM=Z100=Prodigy<Z100=Prodigy=SF	Valiant has significantly the highest value of all. All<Val.

**Table 8: Mechanical Properties of Materials at Each Time**

Time	KIC	$\sigma_f$	$E_f$																																																																																																																																																																																																																																														
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ANOVA	<p>ANOVA-KIC of all materials at 1-hour</p> <table><tr><th>Result</th><th>Sum of Squares</th><th>df</th><th>Mean Square</th><th>F</th><th>Sig.</th></tr><tr><td>Between Groups</td><td>17.095</td><td>7</td><td>2.442</td><td>26.804</td><td>.000</td></tr><tr><td>Within Groups</td><td>6.687</td><td>70</td><td>.0956</td><td></td><td></td></tr><tr><td>Total</td><td>23.782</td><td>77</td><td></td><td></td><td></td></tr></table>	Result	Sum of Squares	df	Mean Square	F	Sig.	Between Groups	17.095	7	2.442	26.804	.000	Within Groups	6.687	70	.0956			Total	23.782	77				<p>ANOVA-FlexSt of Materials at 1-hour</p> <table><tr><th>Result</th><th>Sum of Squares</th><th>df</th><th>Mean Square</th><th>F</th><th>Sig.</th></tr><tr><td>Between Groups</td><td>152569.0</td><td>7</td><td>21795.566</td><td>124.169</td><td>.000</td></tr><tr><td>Within Groups</td><td>15271.222</td><td>87</td><td>175.531</td><td></td><td></td></tr><tr><td>Total</td><td>167840.2</td><td>94</td><td></td><td></td><td></td></tr></table>	Result	Sum of Squares	df	Mean Square	F	Sig.	Between Groups	152569.0	7	21795.566	124.169	.000	Within Groups	15271.222	87	175.531			Total	167840.2	94				<p>ANOVA-FlexMod of Materials at 1-hour</p> <table><tr><th>Result</th><th>Sum of Squares</th><th>df</th><th>Mean Square</th><th>F</th><th>Sig.</th></tr><tr><td>Between Groups</td><td>1203.082</td><td>7</td><td>171.869</td><td>79.424</td><td>.000</td></tr><tr><td>Within Groups</td><td>190.427</td><td>88</td><td>2.164</td><td></td><td></td></tr><tr><td>Total</td><td>1393.509</td><td>95</td><td></td><td></td><td></td></tr></table>	Result	Sum of Squares	df	Mean Square	F	Sig.	Between Groups	1203.082	7	171.869	79.424	.000	Within Groups	190.427	88	2.164			Total	1393.509	95																																																																																																																																																																									
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Within Groups	32884.045	97	340.042																																																																																																																																																																																																																																														
Total	276788.7	105																																																																																																																																																																																																																																															
Result	Sum of Squares	df	Mean Square	F	Sig.																																																																																																																																																																																																																																												
Between Groups	10716.801	8	1339.600	92.055	.000																																																																																																																																																																																																																																												
Within Groups	1426.116	96	14.552																																																																																																																																																																																																																																														
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Scheffe Multiple Means	<p>Result</p> <table><tr><th>Scheffe<sup>a,b</sup></th><th>Material</th><th>N</th><th>1</th><th>2</th><th>3</th><th>4</th></tr><tr><td></td><td>Ketac-Fil</td><td>7</td><td>.282731</td><td></td><td></td><td></td></tr><tr><td></td><td>Photac-Fil</td><td>12</td><td></td><td>.591917</td><td></td><td></td></tr><tr><td></td><td>F2000</td><td>9</td><td></td><td>.721933</td><td></td><td></td></tr><tr><td></td><td>Dyract AP</td><td>11</td><td></td><td>.723636</td><td></td><td></td></tr><tr><td></td><td>Z100</td><td>11</td><td></td><td></td><td>1.062200</td><td></td></tr><tr><td></td><td>SureFil</td><td>10</td><td></td><td></td><td></td><td>1.382780</td></tr><tr><td></td><td>Valiant PHD XT</td><td>9</td><td></td><td></td><td></td><td>1.658333</td></tr><tr><td></td><td>Prodigy C</td><td>11</td><td></td><td></td><td></td><td>1.804636</td></tr><tr><td></td><td>Heliomolar</td><td>11</td><td></td><td></td><td></td><td>1.820000</td></tr><tr><td></td><td>Sig.</td><td></td><td>.192</td><td>.170</td><td>.694</td><td>.257</td></tr></table> <p>Means for groups in homogeneous subsets are displayed. <sup>a</sup> Uses Harmonic Mean Sample Size = 9.868. <sup>b</sup> The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.</p>	Scheffe <sup>a,b</sup>	Material	N	1	2	3	4		Ketac-Fil	7	.282731					Photac-Fil	12		.591917				F2000	9		.721933				Dyract AP	11		.723636				Z100	11			1.062200			SureFil	10				1.382780		Valiant PHD XT	9				1.658333		Prodigy C	11				1.804636		Heliomolar	11				1.820000		Sig.		.192	.170	.694	.257	<p>Result-FlexSt of 9mats at 1day</p> <table><tr><th>Scheffe<sup>a,b</sup></th><th>Material</th><th>N</th><th>1</th><th>2</th><th>3</th><th>4</th></tr><tr><td></td><td>Ketac-Fil</td><td>12</td><td>31.056667</td><td></td><td></td><td></td></tr><tr><td></td><td>Photac-Fil</td><td>12</td><td></td><td>90.325833</td><td></td><td></td></tr><tr><td></td><td>Valiant PHD XT</td><td>12</td><td></td><td>113.0642</td><td></td><td></td></tr><tr><td></td><td>Heliomolar</td><td>12</td><td></td><td></td><td>122.5792</td><td></td></tr><tr><td></td><td>F2000</td><td>12</td><td></td><td></td><td>129.0333</td><td></td></tr><tr><td></td><td>SureFil</td><td>12</td><td></td><td></td><td></td><td>167.8917</td></tr><tr><td></td><td>Prodigy C</td><td>12</td><td></td><td></td><td></td><td>172.9667</td></tr><tr><td></td><td>Dyract AP</td><td>12</td><td></td><td></td><td></td><td>175.5417</td></tr><tr><td></td><td>Z100</td><td>10</td><td></td><td></td><td></td><td>182.8100</td></tr><tr><td></td><td>Sig.</td><td></td><td>1.000</td><td>.360</td><td>.816</td><td>.233</td></tr></table> <p>Means for groups in homogeneous subsets are displayed. <sup>a</sup> Uses Harmonic Mean Sample Size = 11.739. <sup>b</sup> The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.</p>	Scheffe <sup>a,b</sup>	Material	N	1	2	3	4		Ketac-Fil	12	31.056667					Photac-Fil	12		90.325833				Valiant PHD XT	12		113.0642				Heliomolar	12			122.5792			F2000	12			129.0333			SureFil	12				167.8917		Prodigy C	12				172.9667		Dyract AP	12				175.5417		Z100	10				182.8100		Sig.		1.000	.360	.816	.233	<p>Result-FlexMod of 9mats at 1day</p> <table><tr><th>Scheffe<sup>a,b</sup></th><th>Material</th><th>N</th><th>1</th><th>2</th><th>3</th><th>4</th></tr><tr><td></td><td>Ketac-Fil</td><td>12</td><td>6.014333</td><td></td><td></td><td></td></tr><tr><td></td><td>Heliomolar</td><td>12</td><td></td><td>11.864833</td><td></td><td></td></tr><tr><td></td><td>Photac-Fil</td><td>12</td><td></td><td>12.249167</td><td></td><td></td></tr><tr><td></td><td>Prodigy C</td><td>12</td><td></td><td></td><td>12.249167</td><td></td></tr><tr><td></td><td>Ketac-Fil</td><td>12</td><td></td><td></td><td></td><td>12.844750</td></tr><tr><td></td><td>Dyract AP</td><td>12</td><td></td><td></td><td></td><td>13.483333</td></tr><tr><td></td><td>F2000</td><td>12</td><td></td><td></td><td></td><td>16.216667</td></tr><tr><td></td><td>SureFil</td><td>12</td><td></td><td></td><td></td><td>17.631667</td></tr><tr><td></td><td>Z100</td><td>12</td><td></td><td></td><td></td><td>18.382500</td></tr><tr><td></td><td>Valiant PHD XT</td><td>11</td><td></td><td></td><td></td><td>44.483636</td></tr><tr><td></td><td>Sig.</td><td></td><td>.056</td><td>.109</td><td>.085</td><td>1.000</td></tr></table> <p>Means for groups in homogeneous subsets are displayed. <sup>a</sup> Uses Harmonic Mean Sample Size = 11.880. <sup>b</sup> The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.</p>	Scheffe <sup>a,b</sup>	Material	N	1	2	3	4		Ketac-Fil	12	6.014333					Heliomolar	12		11.864833				Photac-Fil	12		12.249167				Prodigy C	12			12.249167			Ketac-Fil	12				12.844750		Dyract AP	12				13.483333		F2000	12				16.216667		SureFil	12				17.631667		Z100	12				18.382500		Valiant PHD XT	11				44.483636		Sig.		.056	.109	.085	1.000
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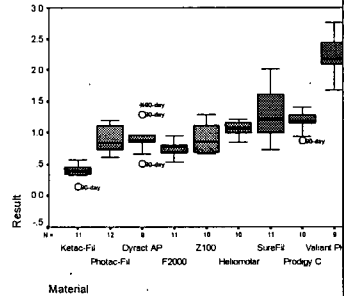
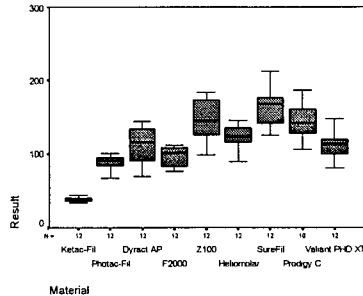
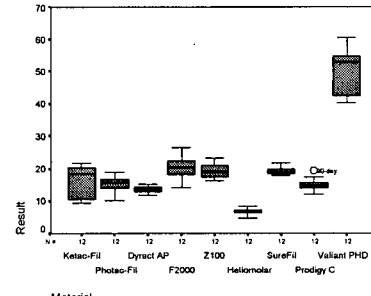
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Scheffe Multiple Means	<p>Result-KIC of 8mats at 90-day</p> <table><tr><th>Scheffe<sup>a,b</sup></th><th>Material</th><th>N</th><th>1</th><th>2</th><th>3</th><th>4</th></tr><tr><td></td><td>Ketac-Fil</td><td>11</td><td>.388873</td><td>.744800</td><td>.883800</td><td></td></tr><tr><td></td><td>F2000</td><td>12</td><td>.744800</td><td>.883800</td><td>.909900</td><td></td></tr><tr><td></td><td>Photac-Fil</td><td>12</td><td>.883800</td><td>.909900</td><td>.928422</td><td></td></tr><tr><td></td><td>Z100</td><td>10</td><td>.909900</td><td>.928422</td><td>1.048560</td><td></td></tr><tr><td></td><td>Dyract AP</td><td>9</td><td>.928422</td><td>1.048560</td><td>1.176030</td><td></td></tr><tr><td></td><td>Heliomolar</td><td>10</td><td>1.048560</td><td>1.176030</td><td>1.283109</td><td></td></tr><tr><td></td><td>Prodigy C</td><td>10</td><td>1.176030</td><td>1.283109</td><td>2.225889</td><td></td></tr><tr><td></td><td>SureFil</td><td>11</td><td>1.283109</td><td>2.225889</td><td></td><td></td></tr><tr><td></td><td>Valiant PHD XT</td><td>9</td><td>2.225889</td><td></td><td></td><td></td></tr><tr><td></td><td>Sig.</td><td></td><td>.200</td><td>.426</td><td>.078</td><td>1.000</td></tr></table> <p>Means for groups in homogeneous subsets are displayed.</p> <p>a. Uses Harmonic Mean Sample Size = 10.247.</p> <p>b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.</p>	Scheffe <sup>a,b</sup>	Material	N	1	2	3	4		Ketac-Fil	11	.388873	.744800	.883800			F2000	12	.744800	.883800	.909900			Photac-Fil	12	.883800	.909900	.928422			Z100	10	.909900	.928422	1.048560			Dyract AP	9	.928422	1.048560	1.176030			Heliomolar	10	1.048560	1.176030	1.283109			Prodigy C	10	1.176030	1.283109	2.225889			SureFil	11	1.283109	2.225889				Valiant PHD XT	9	2.225889					Sig.		.200	.426	.078	1.000	<p>Result-FlexSt of 8mats at 90day</p> <table><tr><th>Scheffe<sup>a,b</sup></th><th>Material</th><th>N</th><th>1</th><th>2</th><th>3</th><th>4</th></tr><tr><td></td><td>Ketac-Fil</td><td>12</td><td>37.937500</td><td>88.025000</td><td>96.653333</td><td></td></tr><tr><td></td><td>Photac-Fil</td><td>12</td><td>88.025000</td><td>96.653333</td><td>110.4242</td><td></td></tr><tr><td></td><td>F2000</td><td>12</td><td>96.653333</td><td>110.4242</td><td>111.8825</td><td></td></tr><tr><td></td><td>Dyract AP</td><td>12</td><td>110.4242</td><td>111.8825</td><td>123.5917</td><td></td></tr><tr><td></td><td>Valiant PHD XT</td><td>12</td><td>111.8825</td><td>123.5917</td><td>145.5217</td><td></td></tr><tr><td></td><td>Heliomolar</td><td>12</td><td>123.5917</td><td>145.5217</td><td>145.7400</td><td></td></tr><tr><td></td><td>Z100</td><td>12</td><td>145.5217</td><td>145.7400</td><td></td><td></td></tr><tr><td></td><td>Prodigy C</td><td>10</td><td>145.7400</td><td></td><td></td><td></td></tr><tr><td></td><td>SureFil</td><td>12</td><td></td><td></td><td></td><td></td></tr><tr><td></td><td>Sig.</td><td></td><td>1.000</td><td>.403</td><td>.222</td><td>.502</td></tr></table> <p>Means for groups in homogeneous subsets are displayed.</p> <p>a. Uses Harmonic Mean Sample Size = 11.739.</p> <p>b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.</p>	Scheffe <sup>a,b</sup>	Material	N	1	2	3	4		Ketac-Fil	12	37.937500	88.025000	96.653333			Photac-Fil	12	88.025000	96.653333	110.4242			F2000	12	96.653333	110.4242	111.8825			Dyract AP	12	110.4242	111.8825	123.5917			Valiant PHD XT	12	111.8825	123.5917	145.5217			Heliomolar	12	123.5917	145.5217	145.7400			Z100	12	145.5217	145.7400				Prodigy C	10	145.7400					SureFil	12						Sig.		1.000	.403	.222	.502	<p>Result-FlexMod of 8mats at 90day</p> <table><tr><th>Scheffe<sup>a</sup></th><th>Material</th><th>N</th><th>1</th><th>2</th><th>3</th><th>4</th></tr><tr><td></td><td>Heliomolar</td><td>12</td><td>6.701917</td><td>13.616667</td><td>14.991667</td><td></td></tr><tr><td></td><td>Dyract AP</td><td>12</td><td>13.616667</td><td>14.991667</td><td>15.269167</td><td></td></tr><tr><td></td><td>Prodigy C</td><td>12</td><td>14.991667</td><td>15.269167</td><td>16.127333</td><td></td></tr><tr><td></td><td>Photac-Fil</td><td>12</td><td>15.269167</td><td>16.127333</td><td>19.102500</td><td></td></tr><tr><td></td><td>Ketac-Fil</td><td>12</td><td>16.127333</td><td>19.102500</td><td>19.135000</td><td></td></tr><tr><td></td><td>SureFil</td><td>12</td><td>19.102500</td><td>19.135000</td><td>20.112500</td><td></td></tr><tr><td></td><td>Z100</td><td>12</td><td>19.135000</td><td>20.112500</td><td></td><td></td></tr><tr><td></td><td>F2000</td><td>12</td><td>20.112500</td><td></td><td></td><td></td></tr><tr><td></td><td>Valiant PHD XT</td><td>12</td><td></td><td></td><td></td><td></td></tr><tr><td></td><td>Sig.</td><td></td><td>1.000</td><td>.067</td><td>.123</td><td>49.8900</td></tr></table> <p>Means for groups in homogeneous subsets are displayed.</p> <p>a. Uses Harmonic Mean Sample Size = 12.000.</p>	Scheffe <sup>a</sup>	Material	N	1	2	3	4		Heliomolar	12	6.701917	13.616667	14.991667			Dyract AP	12	13.616667	14.991667	15.269167			Prodigy C	12	14.991667	15.269167	16.127333			Photac-Fil	12	15.269167	16.127333	19.102500			Ketac-Fil	12	16.127333	19.102500	19.135000			SureFil	12	19.102500	19.135000	20.112500			Z100	12	19.135000	20.112500				F2000	12	20.112500					Valiant PHD XT	12						Sig.		1.000	.067	.123	49.8900
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Table 9: Results (Mean  $\pm$  S.D.)

Material	Time	$K_{IC}$ (MPa $\cdot$ m <sup>1/2</sup> )		$\sigma_f$ (MPa)		$E_f$ (MPa)	
Ketac Fil	1 hr	.1797	.0181	21.5308	2.1955	11.7938	2.0244
	1 d	.2627	.0809	31.0567	6.8266	12.8448	2.0712
	7 d	.3872	.0989	39.0317	7.9559	18.8308	2.4474
	30 d	.3779	.0782	25.3550	5.2517	19.3150	3.7871
	90 d	.3869	.1111	37.9375	3.3332	16.1273	4.9494
Photac Fil	1 hr	.7912	.1194	69.0850	9.4531	7.1128	.7406
	1 d	.5919	.0803	90.3258	5.8370	11.8648	1.8584
	7 d	.7096	.1564	99.5975	6.4786	16.7167	1.8553
	30 d	.7004	.1181	89.2825	11.2702	15.0775	2.1719
	90 d	.8838	.2088	88.0250	9.7022	15.2692	2.3878
Dyract AP	1 hr	1.4943	.4960	118.7325	15.5109	9.1225	.4754
	1 d	.7238	.2381	175.5417	17.3976	13.4833	2.0891
	7 d	1.3541	.5151	146.5083	33.6542	11.3870	1.7349
	30 d	1.0103	.3458	127.6217	21.6093	11.9680	1.4669
	90 d	.9284	.2880	110.4242	24.6159	13.6167	.9783
F2000	1 hr	1.0012	.3534	99.4733	11.0886	11.8565	1.2632
	1 d	.7219	.1273	129.0333	13.7157	16.2167	2.0155
	7 d	.6894	.0758	103.5318	17.1324	17.1991	2.1800
	30 d	.6778	.1116	115.7940	13.9885	21.2933	2.7533
	90 d	.7448	.1289	96.6533	13.1913	20.1125	3.5292
Z100	1 hr	1.1929	.3530	163.0182	15.0307	16.3867	2.8681
	1 d	1.0622	.4323	192.8100	25.6895	18.3825	2.2575
	7 d	1.1243	.4250	214.5667	37.5018	20.1442	1.8838
	30 d	.8840	.2160	179.5300	35.7961	20.5675	2.2609
	90 d	.9099	.2219	145.5217	29.1927	19.1350	2.2392
Heliomolar	1 hr	1.1793	.0959	69.7783	13.2773	3.6501	.6742
	1 d	1.8200	.2170	122.5792	15.3009	6.0143	.4987
	7 d	1.9729	.2455	133.5000	9.2256	7.2441	.6372
	30 d	1.6324	.4333	119.8325	20.8191	7.4298	.7509
	90 d	1.0486	.1254	123.5917	16.2079	6.7019	.9366

<b>SureFil</b>	1 hr	1.9460	.2732	123.1333	17.7390	11.8450	1.1044
	1 d	1.3828	.4548	167.8917	30.6373	17.6317	1.0954
	7 d	1.9082	.4778	169.4000	31.2912	18.3750	2.1473
	30 d	1.5358	.6548	168.3667	23.4522	19.7208	1.1875
	90 d	1.2931	.4048	162.0917	24.3130	19.1025	1.0825
<b>Prodigy C</b>	1 hr	1.1759	.2262	115.4892	15.3044	9.5616	.9710
	1 d	1.8046	.4260	172.9667	19.9946	12.2492	1.3618
	7 d	1.7646	.2439	157.2667	26.8917	12.6910	1.4300
	30 d	1.3787	.4199	133.7200	16.3139	12.2787	1.3837
	90 d	1.1760	.1718	145.7400	25.3536	14.9917	1.8633
<b>Valiant PHD XT</b>	1 d	1.6583	.2827	113.0642	17.7362	44.4636	10.7510
	90 d	2.2259	.3418	111.6825	18.0011	49.8900	7.3295

## Chapter V. Discussion

We are surrounded by a world that in every aspect is mysterious and unknown to us. For the most part we take the elements of our immediate environment, from the people around us to the tools and materials that we use routinely, for granted. It is only when we pause and put the questions that roam our imagination to scientific test that we develop a better understanding and appreciation, though nominal, for the world around us. This research has attempted to answer some questions with regard to the mechanical properties, and hence the limitations of, an array of dental restorative materials used routinely for intra-coronal restorations in primary dentition.

### 5.1 Materials

Dental amalgam alloy has been the most historic and widely used material for intra-coronal restorations in primary posterior teeth. The advances in dental materials over the last half century have allowed for use of tooth coloured restorations, initially in the form of silicate and GICs, and more recently as composite resins. Packable composite resins have been developed more recently with the intention to improve the handling and mechanical properties of traditional composite resins. The mechanical properties of representative restorative materials were determined in this project in order to provide the means to discuss their suitability for a specific clinical application in paediatric dentistry.

Ketac-Fil (ESPE) represented conventional GICs, which set via an acid-base reaction. Photac-Fil (ESPE) was used to represent the RMGICs, which set via a dual-setting mechanism by means of an initial light-activated polymerization and an acid-base reaction similar to the conventional GICs. The group of PAMCR materials, referred to as “compomers” by some dental manufacturers, was represented by Dyract AP (Dentsply) and F2000 (3M). Composite-resins were represented by Heliomolar (Vivadent) and Z100 (3M), a microfill and a hybrid composite resin respectively. The more recent modification of composite resins, namely the “packable composite resins”, were represented by SureFil (Dentsply) and Prodigy Condensable (KERR). Valiant PhD, a unicompositional high copper amalgam, was used as the standard against which newer direct restorative materials are most often compared.

### 5.2 Methods

#### 5.2.1 Tests and Clinical Significance of Tests: TPB, NTP

The three-point bend test was used to measure the flexural strength and the modulus of elasticity of these materials. The flexural strength ( $\sigma_f$ ) is an important and clinically relevant parameter of a material, as it combines the elements of tensile, compressive, and shear stresses



simultaneously. As Griffith had shown, however, flexural strength is sensitive to microcrack-like defects, and therefore, is not an intrinsic material property [Griffith 1920]. In a clinical situation a material with low flexural strength that is subjected to high tensile forces will result in tooth-restoration marginal breakdown and surface defects. Elastic modulus,  $E$ , on the other hand, measures stiffness of a material and represents intrinsic properties of that material. The elastic modulus of a material is important in relation to the anticipated longevity of a restoration [Burke et al. 2002]. In a clinical situation, a material with low elastic modulus undergoes a significant deformation and fails at low stresses, leading to microleakage and increased surface dissolution [Tyas 1990].

The NTP test was used to determine the  $K_{IC}$ , the critical stress intensity factor in Mode I loading that represents the resistance of a material to the propagation of a crack.  $K_{IC}$  is a measure of the material's brittleness and at high stresses, materials with higher  $K_{IC}$  perform best [Tyas 1990]. Surface wear, chipping, and marginal breakdown have shown a strong negative correlation with  $K_{IC}$  [Tyas 1990].

### **5.2.2 Resolution, Precision, Accuracy, and Validity of the NTP $K_{IC}$ Test**

Since its introduction by Ruse and colleagues in 1996, the NTP test has yet to be recognized as a standard test for the measurement of  $K_{IC}$  by ASTM. For drawing conclusions from this test it is important to evaluate its reliability. Ruse and colleagues delineate the approach taken in ensuring the precision, accuracy, and validity of the NTP  $K_{IC}$  test [Ruse et al. 1996].

Validity refers to the ability of a test to measure what it is intended to measure [Brunette 1996]. Ruse and colleagues employed finite element analysis to confirm the validity of NTP test by showing that maximum stresses concentrated at a point on the NTP specimen corresponding to the tip of the chevron notch in the CNSR  $K_{IC}$  test [Ruse et al. 1996]. The presence of crack arrest in most tested specimens is further indication that the test was taking place in a stable and reliable manner. This is also indicative of a stable crack growth and planar crack propagation satisfying plain strain conditions [Hertzberg 1996]. Based on this criteria specimen that demonstrated unstable crack growth resulting in multiple fracture sites or oblique fracture lines were excluded in our study, as they did not satisfy the plain strain conditions. Examining the validity of NTP specimen  $K_{IC}$  test by correlating its results with those from standard methods and the literature is also helpful. Ruse and colleagues fabricated Polymethyl methacrylate (PMMA) specimen for both CNSR and NTP specimen  $K_{IC}$  tests [Ruse et al. 1996]. Their results

are summarized in Table 10. As these results indicate, the NTP specimen and CNSR specimen tests are in excellent agreement. This further proves the validity of the NTP test.

Table 10: $K_{IC}$ of PMMA determined by NTP and CNSR Mean $\pm$ S.D. in $\text{MPa}\cdot\text{m}^{1/2}$			
Sample	W/D	$\alpha_0$	$K_{IC}$
CNSR	1.23	0.270	$1.15 \pm 0.15$
CNSR	1.63	0.518	$1.08 \pm 0.13$
CNSR	1.75	0.552	$1.15 \pm 0.12$
NTP	0.88	0.500	$1.03 \pm 0.15$

Ruse and colleagues also tested other dental materials and compared their results with those available from other reliable sources with standard techniques from the literature [Ruse et al. 1996]. Specifically they tested Durelon (ESPE), Vitremer (3M), Panavia, and Z100 (3M) and found excellent correlation with the existing literature. Table-11 is a summary of their comparisons.

Table 11: $K_{IC}$ ( $\text{MPa}\cdot\text{m}^{1/2}$ ) of selected materials by Ruse et al. 1996			
Material	NTP test	Literature value	Literature reference
Durelon	$0.35 \pm 0.03$	$0.37 \pm 0.03$	Jacobs et al. 95
Vitremer	$0.79 \pm 0.10$	$0.79 \pm 0.07$	Jacobs et al. 95
Panavia	$0.88 \pm 0.15$	0.98	Not listed
Z100	$1.21 \pm 0.25$	$1.03 \pm 0.06$	Ferracane et al. 93

In a similar approach, based on the results of this study for KF, the following tables draw some comparison between the values attained from our study and those reported in the literature [Beatty and Pidaparti 1993; Bonilla et al. 2000; Goldman 1985; Kovarik and Muncy 1995; Lloyd and Mitchell 1984; Miyazaki et al. 1996a; Pidaparti and Beatty 1995]. Data available from representative GIC materials are also included (Tables 12). The 1-day values for  $K_{IC}$  of Ketac-Fil from two studies match our 1-day value rather closely [Beatty and Pidaparti 1993; Pidaparti and Beatty 1995]. In one of these studies the authors had also used 2-dimensional and 3-dimensional finite element analysis to confirm the validity of their results [Pidaparti and Beatty 1995]. The 7-day value for  $K_{IC}$  of Ketac-Fil from our study also is not significantly different from that of another study [Goldman 1985]. By inference these correlations confirm the validity of NTP specimen for measuring  $K_{IC}$  of Ketac-fil in our experiment.

Table 12: $K_{IC}$ of GIC Materials in MPa.m <sup>1/2</sup>								
Study	Nouri, MR; unpublished	Pidapartti & Beatty 95	Lloyd & Mitchell 84	Goldman 85	Goldman 85	Beatty & Pidapartti 93	Bonilla et al. 00	Miyazaki et al. 96
Material	<b>KF</b>	<b>KF</b>	Fuji II	<b>KF</b>	Fuji II	<b>KF</b>	KM	Fuji II
Aging/ Medium	1h, 1d, 7d, 30d, 90d, 37 H2O	1d, 37 H2O	7d, 37 H2O	7d, 37 H2O	7d, 37 H2O	1d, 37 H2O	1d, Air	1h, 1d, 7d, 30d, 180d, 37 H2O
Method	NTP	FEA 2D, 3D; TPB	SEN	DT	DT	TPB	SEN	SEN
Results	1h: 0.18 (0.02) 1d: 0.26 (0.08) 7d: 0.39 (0.10) 30d: 0.38 (0.08) 90d: 0.39 (0.11)	All same <b>1d: 0.34 (0.05)</b>	7d: 0.26 (0.03)	<b>7d: 0.55 (0.05)</b>	7d: 0.51 (0.03)	<b>1d: 0.35 (0.05)</b>	1d: 0.72 (0.07)	1h: 0.35 (0.02) 1d: 0.49 (0.04) 7d: 0.49 (0.04) 30d: 0.47 (0.04) 180d: 0.52 (0.06)
Notes: Ketac-molar (ESPE), Fuji II (GC), Ketac-Fil (ESPE), TPB (Three-point Bend), SEN (Single edge notched), FEA (finite element analysis 2-D and 3-D), DT(Double torsion)								

The accuracy of a test is also an important aspect of any test. Accuracy refers to the closeness of the test results to the actual value of a measurement [Brunette 1996]. The basis for this assertion is the absence of bias. In experiments measuring mechanical properties of materials, however, the accuracy of any value may be ascertained by comparing the results with the reported values in the literature using substantiated and standardized techniques. As mentioned earlier, the excellent correlation between the  $K_{IC}$  values of PMMA by using NTP and CNSR specimen is also an indication of the accuracy of the former test [Ruse et al. 1996]. The results of  $K_{IC}$  of dental materials from Ruse et al. and those from our study are also comparable to those reported in the literature using standardized techniques (Tables 12). It is important to note that systematic error may be a contributing factor in any study that involves calculations based on assumption of accuracy of some preliminary measurements. In our study, for instance, in order to calculate the  $K_{IC}$  of the NTP specimen the formula that governs the measurements of CNSR specimen was used. By measuring the maximum load at failure the value of  $K_{IC}$  was calculated [Ruse et al. 1996].

$$K_{IC} = Y_{min}^* \frac{P_{max}}{DW^{1/2}}$$

where:  $P_{max}$  = maximum load recorded during testing;  $D$  = specimen diameter (= 12 mm);  $W$  = specimen length (= 10.5 mm);  $Y_{min}^*$  = the dimensionless stress intensity factor coefficient minimum (equal to 28 for the NTP test) [Ruse et al. 1996]. We did not confirm that the

specimen tested in our study presented with the exact dimensions listed above. Furthermore, the value of  $Y_{\min}^*$  had been extrapolated from results of compliance calibration work by other researchers [Bubsey et al. 1982].  $Y_{\min}^*$  is a constant, which is not dependent on material property, but changes based on specimen geometry. Although such systematic error is noted in our measurements, it is important to note that all values for  $K_{IC}$  are affected to the same degree. This implies that although the  $K_{IC}$  values based on the NTP specimen test may not be 100% accurate, they may still be used to draw comparisons between different materials and time-lines.

To draw such comparisons between different experimental conditions it is important for the test to be as precise as possible. Precision refers to degree of closeness of repeated measurements to each other and may be indicated by stating standard deviation, variance, mean deviation, the range, and confidence intervals [Brunette 1996]. Coefficient of variation (C.V.), which eliminates the units of measurement and allows a relative comparison between the standard deviation in relation to the mean, may also be used to discuss the precision of results. Comparative results for the measurement of  $K_{IC}$  of PMMA using both NTP and CNSR specimen indicated an excellent precision with a coefficient of variation of 13-15% [Ruse et al. 1996]. The measurements for other materials, however, were not as precise.  $K_{IC}$  of Z100, for instance, showed a coefficient of variation of about 20% [Ruse et al. 1996]. Variations less than 20% appear to be the norm for both NTP and CNSR techniques. In our experiments, however, for  $K_{IC}$  of KF the coefficient of variation ranged from 10-30%. This is an indication that most likely there were more systematic and random errors present in our study. Variations in materials formulations affecting their setting characteristics and surface hardness are examples of random errors affecting the precision of our experimental measurements. Variations in surface hardness, for instance, affect the precision of placing a crack initiate prior to testing. This is an aspect of the NTP specimen  $K_{IC}$  test that needs to be re-evaluated and modified in order to reduce systematic and random errors in the measurements. This should not reflect negatively on the NTP specimen  $K_{IC}$  test, however, as its precision has been well documented by Ruse and colleagues by comparing it with the CNSR specimen  $K_{IC}$  test using PMMA [Ruse et al. 1996].

Resolution of a test refers to how fine a detail can be measured [Brunette 1996]. The maximum load measured for the calculation of  $K_{IC}$  in our experiment was reported to two decimal points. The results in Table-2 are reported with four significant figures, as the  $K_{IC}$  values for KF are relatively low. This allows for an acceptable resolution in identifying differences amongst materials and different aging treatments.

From the preceding discussion one may conclude that despite its limitations, the NTP specimen  $K_{IC}$  test is a valid test that offers a reliable degree of accuracy and precision for measuring and comparing the fracture resistance of different restorative materials. Extreme care should be exercised, however, to reduce any sources of systematic error.

### 5.2.3 Sample fabrication

In order to facilitate fabrication of bar specimens for very weak materials and to avoid possibly creating interference by lubricating agents, a Teflon mould was developed for the three-point bend test. It is important to note that for the fabrication of bar specimens, various specimen preparation and moulding techniques have been used in the past. These include grinding, the use of putty impression materials, metal moulds, and Teflon moulds. Grinding has a tendency to overheat the material and induce microcracks on the surface of the specimen. In order to allow for consistency in methodology, this writer suggests the development of a standard, machine fabricated, Teflon mould could support testing of materials of different consistencies and strengths. The Teflon moulds that were used for the preparation of triangular prism specimen for the fracture toughness test in this study seemed to allow for preparation of consistently identical specimens. In this experiment we only randomly measured the dimensions of specimen to ensure that the dimensions were close to the desired values. This may have introduced a significant error in our measurements, particularly for flexural strength, as the size of each specimen affects the calculated value for that mechanical property. It is recommended that the exact dimensions of each specimen be taken into account for the measurement of its mechanical properties.

The use of a Bard Parker blade for creating an initiate in the specimen prior to testing for  $K_{IC}$  may not allow for a reliable and accurate crack initiate as the surface hardness of materials tested varies significantly. In some materials, such as GIC, one may inadvertently create a crack leading to inaccurate measurements of  $K_{IC}$ . The issue of pre-crack initiates has been evaluated in the literature. It has been shown that  $K_{IC}$  values for tests without a pre-crack that has been created with cyclic loading will be higher than when an atomically sharp pre-crack has been created [Ferracane et al. 1987; Kovarik and Fairhurst 1993]. Zhao et al. also showed that  $K_{IC}$  was dependent on the depth of straight notch and independent of the depth of a V-notch [Zhao et al. 1997]. As the depth of the initiate increased, the  $K_{IC}$  value for the straight notch approached the stable value for the V-notch, indicating a closer resemblance to atomic sharp crack front. In consideration of different methodologies described in the literature, Fujishima and Ferracane evaluated different test methods for the measurement of  $K_{IC}$  [Fujishima and Ferracane 1996].

Interestingly, they found that although the actual values for  $K_{IC}$  of similar materials vary amongst tests, the relative  $K_{IC}$  were in agreement with those reported in the literature. This means that although the absolute values may not be accurate, the tests are reliable in the sense that they provide a reasonable comparison on the fracture properties of a group of materials [Fujishima and Ferracane 1996].

#### **5.2.4 Significance of storage medium and aging**

Although FDA guidelines recommend ethanol as food simulating liquid, in comparison to aging in water, however, composite resins have shown an improvement in  $K_{IC}$  up to 30-days, and thereafter a decrease up to 6-months [Pilliar et al. 1986; Pilliar et al. 1987]. Essentially, after 60-days the  $K_{IC}$  values for composite resins between water and ethanol become undifferentiated [Ferracane and Marker 1992]. In order to simulate body temperature, after preparation, the specimens were stored in 37° C distilled water for the duration of aging based on evidence that water aging provides a better representation of aging in the mouth than aging in the air [Drummond et al. 1998]. It has also been documented that thermocycling and water storage have the same effect on  $K_{IC}$  [Mair and Vowles 1989]. The effect of temperature of the test environment has also been shown to be minor [Lloyd 1982]. The specimens were only removed from water immediately prior to testing in order to minimize the effect of dehydration.

#### **5.3 Effect of Time on the mechanical properties of each material**

For Ketac-Fil (KF), the largest standard deviation was noted with the  $K_{IC}$  results. There was no significant increase in  $K_{IC}$  from 1-hour to 1-day. Thereafter, however, there was a significant increase in  $K_{IC}$  to 7-day with values that were stable to 30-days and 90-days. The values and pattern observed in our results are comparable with those reported previously [Cattani-Lorente et al. 1999b; Mitra and Kedrowski 1994]. This indicates that the intrinsic properties of KF, a conventional GIC, do not significantly improve from 1-hour to 1-day. This is a reflection of the fact that the acid-base reaction in KF continues beyond 1-day and stabilizes after 7-days. Aging from 7 to 90-days did not have a deleterious effect on the intrinsic properties of KF. The pattern observed in  $E_f$  replicates that observed in  $K_{IC}$  for KF. As noted earlier,  $E$  is also an intrinsic property of the material. Decohesive rupture and debonding of glass particles has been proposed as the primary fracture process [Mueller 1990]. For  $\sigma_f$ , however, aging does not have the same effect. Despite an initial increase from 1-hour to 1-day and 7-days, there seems to be a drop in values of  $\sigma_f$  by 30-days to the same level of the 1-day values, prior to a similar increase in values to 90-days. Because  $\sigma_f$  is not an intrinsic property of materials, one

may assume that surface flaws as a result of dehydration or faults in fabrication of the specimen may have given rise to the drop in  $\sigma_f$  values at 30-days. Despite a similar pattern of aging effect on  $\sigma_f$ , our results for KF appear to be significantly larger than those reported previously [Cattani-Lorente et al. 1993; Cattani-Lorente et al. 1994; Li et al. 1996; Xie et al. 2000]. For KF, in general, one may conclude that the mechanical properties remain the same between 1-hour and 1-day, after which there is an improvement in  $K_{IC}$ ,  $E_f$ , and  $\sigma_f$  to 7-day values, which remain unchanged thereafter up to 90-days. This clearly indicates that the acid-base reaction in KF, a typical GIC, continues beyond 1-day and stabilizes after 7-days. Null hypothesis 1 is therefore rejected because time has an enhancing effect on the mechanical properties of KF up to 7-days. Beyond 7-days, however, aging in 37° C distilled water did not have a deleterious effect on the mechanical properties of KF.

For Photac-Fil (PF), a RMGIC, the  $K_{IC}$  values were relatively the same throughout the time-line of the test. There was, however, an initial decrease to the 1-day value, and thereafter a return to the 1-hour value for 7-, 30-, and 90-days. The results and the pattern of aging observed in our experiment are supported by previous reports [Cattani-Lorente et al. 1999b]. The initial drop in  $K_{IC}$  may be attributed to initial water sorption hindering the polymerization of the resin matrix. The  $E_f$  for PF showed a gradual increase from 1-hour to 1-day to 7-days, after which it stabilized to 30- and 90-days values. The  $\sigma_f$  of PF also presented a similar pattern to  $E_f$ , in that it showed a significant increase from the 1-hour to the 1-day value, after which the values were stable. These results indicate that the resistance of PF to critical flaw propagation is primarily dependent on the resin monomer and its light polymerization, while stiffness, represented by  $E_f$ , improves beyond 1-hour to 1-day, and thereafter to a relatively stable level after 7-days. This is reflective of the continued acid-base reaction to 7-days, which enhances the intrinsic properties of the material. As  $\sigma_f$  of PF achieves an improved and stable value after 1-day, it appears that the surface acid-base reaction that continues beyond 1-hour is halted after 1-day. This allows for a scenario in which the surface acid-base reaction slows down long before the internal acid-base reaction does. This makes sense in that it has been claimed that the initial light polymerization has a protective umbrella effect for the continuing acid-base reaction in RMGICs [Mitra and Kedrowski 1994]. Although most of our values correlate well with the reported values, our results for  $\sigma_f$  of PF is significantly higher than the reported values [Li et al. 1996]. Null hypothesis 1 is also rejected for PF, as time does have an effect on its mechanical properties.

In the PAMCR group, Dyract AP and F2000 showed interesting results over the time-line of the mechanical tests. Overall, the values for F2000 showed much less standard deviation than those for Dyract AP. For Dyract,  $K_{IC}$  showed a gradual decrease from the 1-hour value to the 90-days value. Although the 1-hour, 7-day, and 30-day values did not differ significantly, aging to 90-days had a deleterious effect on the  $K_{IC}$  of Dyract AP. The sudden drop in  $K_{IC}$  at 1-day may be attributed to water sorption and its inhibitory effect on continued polymerization of the resin matrix. These results vary with that of Photac-Fil in that its  $K_{IC}$  was generally stable to 90-days, while for Dyract AP, the  $K_{IC}$  at 90-days was significantly less than the 1-hour value. As  $K_{IC}$  is an intrinsic material property, one may conclude that for Dyract AP, there is either an interference with the resin component by the acid-base constituents, or that Dyract AP goes through an internal degradation process leading to its reduce ability to resist catastrophic crack propagation.  $E_f$  for Dyract AP, however, shows a similar trend to that of Photac-Fil as it improves from 1-hour to 90-days, indicating an increase in stiffness of Dyract AP over time. The results of  $\sigma_f$  for Dyract AP are also interesting in that the values seem to peak at 1-day, and thereafter gradually drop to the original 1-hour level by 90-days. As  $\sigma_f$  is not an intrinsic property and is affected by surface flaws, one may deduce that after the initial increase in  $\sigma_f$  as a result of the reaction of acid-base constituents, the surface resin component undergoes a gradual degradation, possibly as a result of dissolution by its acid-base constituents. In comparison with the reported values, the  $\sigma_f$  of Dyract AP reported in the literature is significantly lower than our values, while the reported  $E_f$  are comparable [Cattani-Lorente et al. 1999a; Piwowarczyk et al. 2002]. For Dyract AP, Null Hypothesis 1 is rejected as time does appear to have an effect on its mechanical properties.

For F2000, the other PAMCR tested,  $K_{IC}$  shows a significant drop from the 1-hour value to the 1-day value, which remains the same throughout the time-line of the test. This again may be representative of rapid water sorption and breakdown of the resin constituents by the acid-base constituents, even at a higher rate than for Dyract AP,. The ability of F2000 to resist crack propagation, therefore, diminishes quickly from the 1-hour level to 1-day level. The stiffness of F2000, represented by its  $E_f$ , however, does show a gradual increase from the 1-hour value to the 90-days value. The  $\sigma_f$  of F2000 demonstrates an initial peak at 1-day, similar to that for Dyract AP, and thereafter a gradual decrease to the 1-hour level at 90-days. This again demonstrates that after 1-day the initial resin umbrella goes through a degradation process on the surface of the material, possibly by the acid-base constituents. Null hypothesis 1 is also rejected for F2000 as time does appear to show an enhancing effect on  $E_f$ , and a deleterious effect on its  $K_{IC}$  and  $\sigma_f$ .



The two representatives of conventional composite resins were Z100, a hybrid composite resin with particle sizes of 0.8 microns and Vf of 71%, and Heliomolar, a microfill with particle sizes of 0.04 microns and Vf of 79% by weight. For Z100 there was no significant difference in  $K_{IC}$  over the time-line of the experiment, although a large standard deviation was noted. This indicates that aging up to 90-days has no effect on the ability of Z100 to resist catastrophic crack propagation. The other intrinsic property of Z100, namely  $E_f$ , showed a similar behaviour, with only the 1-hour value being lower than the 7-day and 30-day values. This indicates that the intrinsic structure of Z100 hybrid composite achieves a stable state within 1-hour and is not affected by aging up to 90-days. The  $\sigma_f$  of Z100, however, showed a slight peak at 7-day, after which it dropped back to its 1-day and 1-hour values at 30- and 90-days. This indicates that surface exposure may allow for some degree of surface dissolution over 30-day and 90-day time-line. Null hypothesis 1 is, therefore, accepted for the  $K_{IC}$  of Z100, whereas it is rejected for its  $E_f$  and  $\sigma_f$  as time does seem to have an effect on the latter properties.

For Heliomolar,  $K_{IC}$  shows an initial increase in value from 1-hour to 1-day. It is thereafter stable over 7-days and 30-days until it drops to its 1-hour value at 90-days. It is interesting to note that the  $K_{IC}$  of Heliomolar at 1-hour and 90-days is not significantly different from that of Z100 over all time-lines. The stiffness, or  $E_f$ , of Heliomolar also shows a similar pattern to its  $K_{IC}$ , with the difference that the 90-day value remains high at the same level as that of 7-day and 30-days.  $E_f$  of Heliomolar is, however, significantly less than that of Z100. Heliomolar is, therefore, not as stiff as Z100, but its intrinsic characteristics allow it to be as strong as Z100 in resisting crack propagation over a long time. Eventual water maturation and its plasticizing effect on the resin matrix may be the reason behind the drop in its  $K_{IC}$  at 90-days. Another reason for the higher  $K_{IC}$  in HM may be attributed to the microfillers, which have a crack blunting effect causing a crack jump, and in effect, enhancing  $K_{IC}$  [Drummond et al. 1998; Mueller 1990; Sih and Berman 1980]. In comparison with reported values, our results for mechanical properties of CR are supported by some reports while they vary from others [Braem et al. 1995; Bryant and Mahler 1986; Cobb et al. 2000; Ferracane and Condon 1992; Higo et al. 1991; Kildal and Ruyter 1997; Kim et al. 1991; Li et al. 1996; Mair and Vowles 1989; Miyazaki et al. 1996a; Zhao et al. 1997]. This may be attributed to large variation in formulations of marketed materials with regards to their matrix and filler constituents and their interaction [Drummond et al. 1998]. Null hypothesis 1, therefore, is rejected for Heliomolar over 90-days as time does appear to have an effect on its mechanical properties.

For the “packable” composites, SureFil and Prodigy Condensable, based on manufacturers’ claims, one would anticipate better mechanical properties than those of conventional composite resins. This was not observed in this study. Although a high standard deviation was noted for the  $K_{IC}$  values of SureFil, there was no significant difference between the values over the five time-lines. The  $E_f$  of SureFil demonstrated an increase after 1-hour and remained relatively stable between 1- and 90-days. The  $\sigma_f$  of SureFil also showed a similar pattern, with the 1-hour value being the lowest of all. Null hypothesis 1, therefore, is accepted for the  $K_{IC}$  of SureFil, while it is rejected for the  $\sigma_f$  and  $E_f$  of SureFil, as the latter values did change over time.

For Prodigy, there was an initial rise in  $K_{IC}$  from 1-hour values to 1-day and 7-day values, which reduced to the 1-hour level at 30- and 90-days. Water maturation and the breakdown of polymerized chains over time may be contributing factors in diminishing the ability of this packable composite resin to resist catastrophic propagation of flaws. Prodigy’s  $E_f$ , however, increased consistently from 1-hour to 90-day values. The  $\sigma_f$  of Prodigy also increased from 1-hour to 90-days values, but dropped slightly at 30-days. Although most of our results for SF and Prodigy were supported by other reports, similar to that for CR, some variation in the reported values may be indicative of changes in material constituents [Cobb et al. 2000; Kelsey et al. 2000; Manhart et al. 2000b]. Null hypothesis 1 is rejected for Prodigy Condensable as its mechanical properties seem to vary over time.

For Valiant PHD amalgam alloy, time had a significant effect on its  $K_{IC}$  between 1-day and 90-day values. The  $\sigma_f$  and  $E_f$  of Valiant PHD showed no significant differences between 1-day and 90-days. This indicates that although amalgam shows no surface degradation by storage in 37° C distilled water and maintains its stiffness over time, its intrinsic ability to resist catastrophic flaw propagation does continue to increase from 1- to 90-days. This implies that the only mechanical property indicative of such internal maturation beyond 1-day in amalgam is  $K_{IC}$ . The  $K_{IC}$  value for VPHG at 1-day was  $1.65 (+/- 0.28) \text{ MPa.m}^{-1/2}$ , which is in agreement with the value of  $1.39 (+/- 0.13) \text{ MPa.m}^{-1/2}$  recorded by Bapna and Mueller at 1-day aging in water [Bapna and Mueller 1993]. Another high copper alloy commonly reported in the literature is Tytin. Reported  $K_{IC}$  for Tytin range from 1.07 to  $1.52 \text{ MPa.m}^{-1/2}$  [Beatty and Pidaparti 1993; Bonilla et al. 2000; Pidaparti and Beatty 1995]. Most researchers have indicated that the  $K_{IC}$  of low copper amalgams tends to be higher than that of high copper amalgams [Bryant and Mahler 1986; Cruickshanks-Boyd and Lock 1983]. In low copper amalgams a high creep may allow for plastic flow mechanisms, such as dislocation climb and vacancy migration [Cruickshanks-Boyd

and Lock 1983]. In general, our results are in agreement with those reported in the literature [Bryant and Mahler 1986; Lloyd and Adamson 1985]. Null hypothesis 1 is rejected for  $K_{IC}$  of Valiant PHD between 1- and 90-days, while it is accepted for its  $\sigma_f$  and  $E_f$ .

## **5.4 Comparison of the mechanical properties of nine materials at each time**

### **5.4.1 Comparative analysis at 1-hour**

Amalgam alloy was not tested at 1-hour. At 1-hour,  $K_{IC}$  of Ketac-Fil, the conventional GIC, was significantly the lowest (at  $0.18 \text{ MPa m}^{1/2}$ ) amongst the materials tested. For  $K_{IC}$ , the RMGIC Photac-Fil fell in the second group along with the PAMCR F2000, the hybrid composite resin Z100, the microfil Heliomolar, and the packable composite resin Prodigy at values of 0.79 to  $1.19 \text{ MPa m}^{1/2}$ . The other PAMCR, Dyract, fell in the third grouping with the composite resins, and eventually in the fourth group along with SureFil “packable” composite resin which had the highest value of 1.95 for  $K_{IC}$  at 1-hour. These results are interesting as Dyract proved to be one of the toughest materials tested at 1-hour and faired just as well as hybrid, microfil, and packable composites.

A slightly different pattern evolved when assessing the results of  $\sigma_f$  testing at 1-hour. Ketac-Fil GIC still ranked the lowest at 21.53 MPa, while the hybrid composite resin Z100 ranked the highest with a  $\sigma_f$  of 163.02 MPa at 1-hour. The RMGIC Photac-Fil and the microfil composite Herculite comprised the second distinct group, while the PAMCRs and other composites formed the third and fourth overlapping groups. For  $\sigma_f$ , Z100 also presented with the highest stiffness of 16.39 GPa at 1-hour, while it was the microfil, Heliomolar, that presented with the lowest stiffness. The modulus was not significantly different at 1-hour between Photac-Fil and Dyract. The other PAMCR, F2000, along with the GIC Ketac-Fil, had a comparable modulus to that of the composites 1-hour. Null hypothesis 2 is therefore rejected for the mechanical tests at 1-hour.

### **5.4.2 Comparative analysis at 1-day**

With the slight increase in observed for Ketac-Fil at 1-day, the first grouping for  $K_{IC}$  at 1-day comprised the GIC Ketac-Fil, the RMGIC Photac-Fil, and the PAMCRs Dyract and F2000. The other composites ranked closely in the second and third groups. Interestingly, the amalgam Valiant PHD, the “packable composites”, and the microfil Heliomolar had statistically similar  $K_{IC}$  at 1-day. The other intrinsic property of Heliomolar and Prodigy,  $E_f$ , however, was the lowest amongst all materials. Valiant PHD had the highest stiffness at 1-day. Interestingly, the  $\sigma_f$  of Valiant PHD at 1-day was among the lower values alongside that of Photac-Fil,

Heliomolar, and F2000. Ketac-Fill presented with the lowest  $\sigma_f$  at 1-day as it was the case at 1-hour. The strongest group at 1-day was comprised of Z100, the “packable” composite resins, and Dyract AP. Overall, one can conclude that Ketac-Fill appears to have the lowest values for these mechanical properties, while Sure-Fil “packable” composite appeared to consistently have the higher values amongst the materials tested. Valiant PHD presented with better intrinsic characteristics reflected in its  $K_{IC}$  and  $E_f$  values, but failed to rank high in  $\sigma_f$ , indicating that in a clinical situation without proper support from tooth structure, amalgam is prone to failure under high tensile forces acting on its surface. Overall, Null hypothesis 2 is also rejected at 1-day as the mechanical varied among the materials tested in this research.

#### **5.4.3 Comparative analysis at 7-days**

For  $K_{IC}$  at 7-days, with the exclusion of Valiant PHD which was not tested, a pattern similar to that at 1-day developed. Ketac-Fil, Photac-Fil, and F2000 still showed the lowest values for  $K_{IC}$ , while Heliomolar and the “packable” composites, SureFil and Prodigy, ranked the highest. The pattern was also similar to that at 1-day for  $E_f$  of the materials, with Heliomolar having the lowest stiffness. The pattern for  $\sigma_f$  was also similar to that for 1-day, with Ketac-Fil having the lowest value of 39.03 MPa, while Z100 had the highest  $\sigma_f$  at 214.57 MPa. Null hypothesis 2, is therefore, rejected again as the mechanical properties of the tested materials were different at 7-days.

#### **5.4.4 Comparative analysis at 30-days**

The consistency in the observed relative  $K_{IC}$ ,  $\sigma_f$ , and  $E_f$  of the tested materials at 30-days continued similar to that for 1 and 7-days. This demonstrates that aging between 1- and 30-days did not cause a significant variation in the profiling of the materials tested. It also lends support to the consistency and reliability of the mechanical tests used. Null hypothesis 2 is once again rejected, as there is a significant difference between the mechanical properties of the materials tested at 30-days.

#### **5.4.5 Comparative analysis at 90-days**

The relative mechanical properties of the tested materials continued to show a similar profile at 90-days to that for 1, 7, and 30-days. The only exception was the  $K_{IC}$  of Valiant PHD, which was significantly the highest at 90-days. As was discussed in the previous section, the  $K_{IC}$  of Valiant PHD increased significantly between 1 and 90-days, rendering it more resistant to the propagation of critical flaws. Null hypothesis 2 is once again rejected at 90-days.

## 5.5 Comparative analysis of the inter-group and intra-group results over time

In order to facilitate the clinical application of the results, it is helpful to discuss the mechanical properties of the nine restorative materials within and between groups. Note that over the 1, 7, 30, and 90-day points, with the exception of  $K_{IC}$  for Valiant PHD, there was relative consistency in behaviour of the tested materials. This finding documents that aging from 1-hour to 90-days did not change the relative brittleness, stiffness, and strength of the tested materials, with the exception of the  $K_{IC}$  for Valiant PHD between 1 and 90-days, and for Ketac-Fil between 1-hour and 1-day. Table-7 presents a summary of inter-group and intra-group comparative analysis for  $K_{IC}$ ,  $\sigma_f$ , and  $E_f$  across the time-line of this study.

### 5.5.1 GIC vs. RMGIC

Ketac-Fil (KF) and Photac-Fil (PF) were representative materials for the conventional GICs and RMGICs respectively. At 1-hour, KF had significantly lower  $K_{IC}$  than PF. At 1, 7, and 30-days, however, there was no significant difference in their  $K_{IC}$ . At 90-days,  $K_{IC}$  of PF had, once again, achieved a significantly higher level. This indicates that the initial light polymerization of the resin monomer in PF imparts a higher resistance to crack propagation, while partial maturation of the chemical setting of KF by 1-day improves its  $K_{IC}$ . The increase in  $K_{IC}$  of PF at 90-days may be attributed to the maturation of its underlying chemical constituents [Wilson and McLean 1988]. The  $E_f$  of KF, however, is significantly higher than that of PF at 1-hour and 30-days. The difference is negligible, however, at 1, 7, and 90-days. This may be an indication that the same light polymerization that gives PF a higher resistance to flaw propagation initially, delays the underlying acid-base chemical reaction and causes a delay in developing stiffness. One may conclude that while both KF and PF contain acid-base constituents, the resin monomers present in PF do not allow the chemical reaction to proceed at the same rate as that in KF in the first hour. This may also be attributed to a possible reduction in percent weight of the chemical constituents in RMGIC as compared to GIC. The  $\sigma_f$  of PF was consistently higher than that of KF at all five times. The latter may be directly related to the umbrella effect of the resin polymerization after its initial set.

In comparing KF and PF, one may deduce that, although the addition of resin imparts a higher resistance to crack propagation in RMGIC at 1-hour after light polymerization, it makes RMGIC less stiff initially. The latter is a negligible drawback in light of the added  $\sigma_f$  that is offered throughout the time-line of this experiment, due to the umbrella effect of the resin-polymerization. The relative improvement in mechanical properties of RMGIC over GIC is also supported by other reports [Li et al. 1996; Mitra and Kedrowski 1994; Xie et al. 2000]. One may

conclude that addition of resin monomers in RMGIC has improved the mechanical properties of GIC.

### 5.5.2 RMGIC vs. PAMCR

The results of this experiment also offer a comparison between RMGIC and PAMCR, the latter class being represented by Dyract AP and F2000. As stated previously, the  $K_{IC}$  of Dyract and F2000 dropped significantly between 1-hour and 1-day, and remained relatively stable thereafter. The results indicate that, over time, there is no significant difference between the  $K_{IC}$  of RMGIC and that of PAMCR ( $p=0.05$ ). The stiffness of Photac-Fil and Dyract appear to be less than that of F2000 at 1-hour, 30-days, and 90-days. The  $\sigma_f$  of PAMCRs was higher than that of RMGIC at 1-hour, but gradually equalized by 90-days. Overall, the mechanical properties of RMGICs and PAMCRs are similar. Considering that RMGICs still possess many of the clinically advantageous properties of GICs, one may argue that, in view of their similar mechanical properties, it would be preferable to use RMGICs clinically instead of PAMCRs.

### 5.5.3 PAMCR vs. CR

The composite resins (CR) tested were Z100, a hybrid CR, and Heliomolar, a microfil CR. It is appropriate to compare PAMCRs with the CRs, since the manufacturers of "compomers" claim that they possess mechanical properties of CRs, while maintaining the biological advantages of GICs. With its higher  $K_{IC}$  at 1-hour than that of F2000, Dyract fell in the higher subgroup along with both composites. At 1, 7, and 30-days, however, the microfil Heliomolar had significantly higher  $K_{IC}$ . At 90-days, there was no significant difference between the  $K_{IC}$  PAMCRs and CRs. Within the CRs tested, the microfill CR had significantly higher  $K_{IC}$  than the hybrid CR throughout the time-line of the experiment, except at 90-days when the values were not statistically different. The  $E_f$  of Heliomolar, however, was consistently significantly less than that of PAMCRs and the hybrid CR, Z100. The  $\sigma_f$  of Heliomolar was less than that of Z100, except for 90-days when they were not statistically different. The  $\sigma_f$  of PAMCRs was significantly less than that of Z100, but not different from that of Heliomolar. These results indicate that microfillers impart a higher resistance to crack propagation and a reduction in stiffness, while hybrid-type fillers impart higher  $E_f$ . The higher  $K_{IC}$  of Heliomolar may be attributed to its smaller filler particle size of 0.04 microns as compared to the filler particle size of 0.8 microns in Z100. It is important to note that although the mechanical properties of PAMCR falls somewhere in between those of RMGIC and CR, the properties of CR are significantly superior to that of RMGIC [Cohen et al. 2001]. Overall, the mechanical

properties of PAMCR are close to CR. This is also supported in the literature [Gladys et al. 1997; Mueller 1990].

#### **5.5.4 CR vs. PCR**

SureFil (SF) and Prodigy Condensable (PC) were the two “packable” composite resins (PCR) studied in this experiment. The manufacturers of PCR claim the added advantage of ease of handling and condensation when restoring Class II cavities and placing large restorations. They also claim improved mechanical properties for PCR. At 1-hour, the  $K_{IC}$  of SF was significantly higher than that of PC, Z100, and Heliomolar (HM). The difference decreased, however, as for 1, 7, and 30-days the  $K_{IC}$  of PC, SF, and HM were about the same, and significantly higher than that of Z100. The latter relationship is also reported by others [Bae et al. 2001; Bonilla et al. 2001; Bryant and Mahler 1986; Cobb et al. 2000; Kelsey et al. 2000] At 90-days, the  $K_{IC}$  of all four composite resins were not statistically different ( $p=0.05$ ). This long term equalization of  $K_{IC}$  amongst CR and PCR may be due to equilibrium in water sorption [Kelsey et al. 2000; Mecholsky 1995; Mueller 1990] One may deduce that, over long periods, PCR do not impart an increase in resistance to flaw propagation compared to hybrid and microfil CR. For  $E_f$ , the PCR fell between the higher value of Z100 and the lower value of HM at all time intervals. This pattern was also observed for  $\sigma_f$  over all time intervals. This leads one to conclude that the mechanical properties of PCR are relatively the same as those of hybrid and microfil composite resins, although the microfil composite Heliomolar appears to be the least stiff at all times.

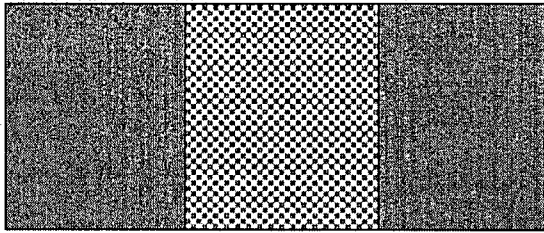
#### **5.5.5 Amalgam vs. Other Materials**

Valiant PHD was the amalgam alloy of choice tested at 1-day and 90-day time-lines. As stated before, the  $K_{IC}$  of Valiant improved significantly over time, while its  $E_f$  and  $\sigma_f$  stayed the same. Although at 1-day the  $K_{IC}$  of Valiant ranked in the same subgroup as that of PCRs SureFil and Prodigy and the microfil composite resin Heliomolar, by 90-days Valiant had the highest  $K_{IC}$  ( $2.23 \text{ MPa.m}^{-1/2}$ ). The short term comparable value of  $K_{IC}$  for amalgam with that of posterior composites has also been documented [Lloyd 1983]. The  $E_f$  of Valiant was the highest at both 1 and 90-day. The results of these two tests of the intrinsic properties of amalgam indicate that over time, Valiant has the highest stiffness and the most resistance to crack propagation [Cho and Cheng 1999]. The  $\sigma_f$  of Valiant at 1 and 90-days was similar to that of PhotacFil, Heliomolar, and F2000, while consistently less than that of hybrid Z100 and the PCRs. In light of the excellent clinical characteristics and performance of amalgam in the hostile

environment of the oral cavity, it would be fair to say that  $K_{IC}$  and  $E_f$  appear to be better indicators of such clinical superiority over time.

## 5.6 Future Directions

One of the observations made during this research had to do with the physical appearance of the fabricated NTP specimens. With some of the composite resins, a whitish, porous-looking region on the mid-surface of the prism was noted. This was only noted in those materials that set via light polymerization. The only exceptions to this were the microfil composite resin Heliomolar and Photac-Fil, which has a dual setting mechanism with the initial light activation. Ketac-Fil and Valiant PHD amalgam, which set chemically, did not show this porosity either. Table-13 demonstrates the presence and the relative length of the porous region noted in each material.

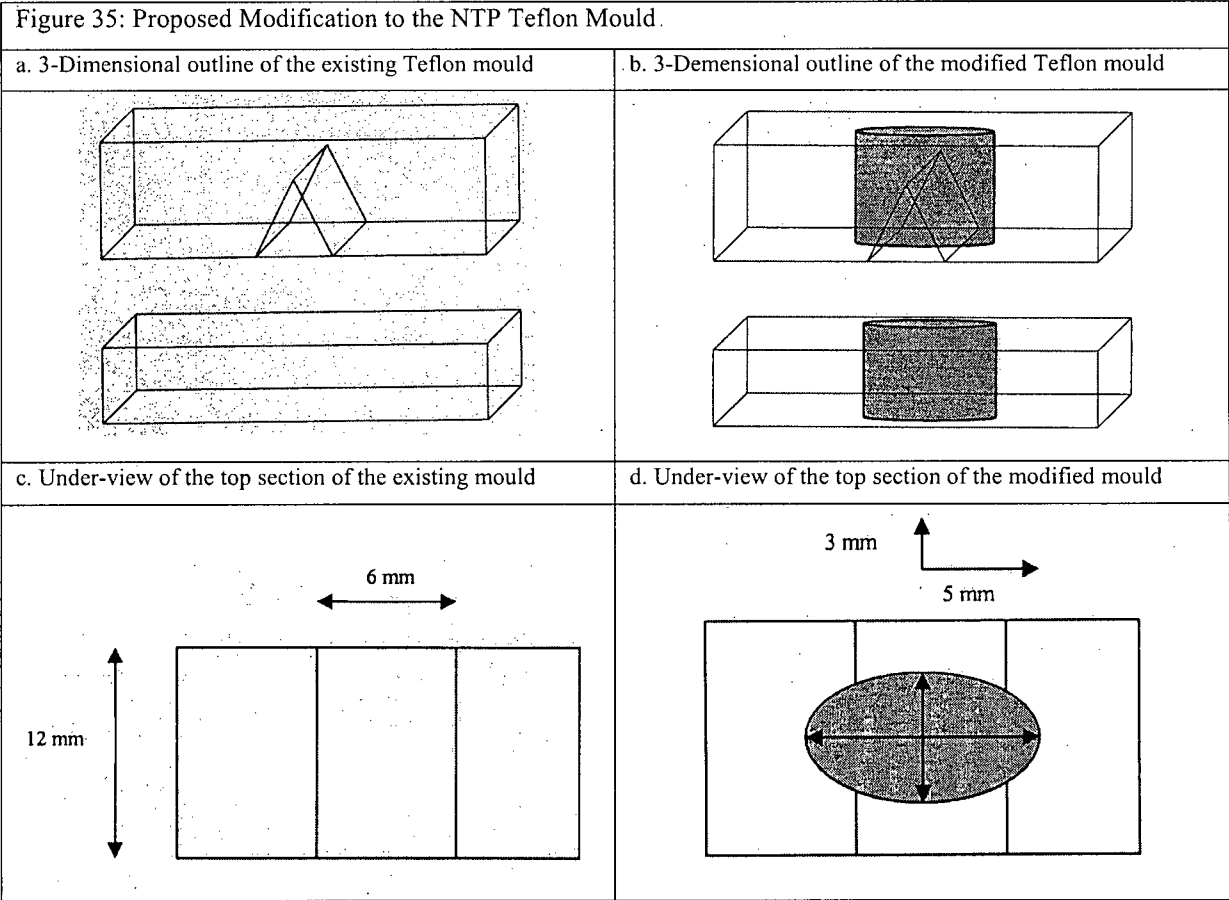
Table 13: Presence and degree of surface porosity on dried NTP samples									
Surface of Notchless-Triangular Prism Specimen									
									
Porous Surface Area									
Material	KetacFil	PhotacFil	Dyract AP	F2000	Z100	Heliomolar	SureFil	Prodigy C	Valiant PHD
Proportion of Porous Surface	None	None	50%	50%	50%	None	33%	50%	None

This may be an important phenomenon that results from the initial light polymerization from the two ends of the prism during the NTP specimen fabrication. Because light polymerization causes shrinkage of composite resins, the two ends may create a pull on the uncured mid-section of the NTP sample, rendering it porous and defective. The observations made in this study seem to support this, as the porous zone appeared to be just beyond 3mm from either end of the 12mm long triangular prism. The depth of cure is generally approximated to be within 2mm to 5mm from the composite resin surface [Unterbrink and Muessner 1995]. An increase in the presence of surface and internal flaws will certainly affect the results of all tests, and particularly those reflecting fracture resistance of the material. Because the mid-section of the NTP specimen is where the flaw initiate is placed, and thereafter, fracture progresses, this



may have serious implications in rendering the NTP test for  $K_{IC}$  invalid as the structure of the material may become compromised and vulnerable to uncontrolled fracture. This is clearly an issue that needs to be evaluated further.

The NTP mold could be modified by incorporating a cylinder of fiberoptic fibers or plexi-glass, with an ovoid-shaped end, with the dimensions of 6mm by 10mm that runs through the mould (Figure 35).



The dimensions proposed for the ovoid cylinder encompass each arm of the prism and cover 50% of the length of the specimen. By initiating the light polymerization through the translucent cylinder, one may assure that the 6mm mid-section of the NTP specimen is the first area to be cured, hence preventing the formation of the porous segment and avoidance of compromised NTP fracture toughness test results. The top and bottom sections of the mould may also be thinned to reduce the distance from the light tip to the specimen surface, in effect enhancing light polymerization. In order to examine the significance of the suggested modifications, fracture toughness of a composite resin material with validated  $K_{IC}$  may be measured by preparing samples with both moulds. It would also be useful to evaluate the surface

topography of this porous area in the specimens using Scanning Electron Microscopy (SEM), and to evaluate the structural change with the aid of other analytical methods.

Sliding of the specimen while in the specimen holder during testing may also play a role in the large standard deviations noted with the NTP  $K_{IC}$  test. Excessive tightening of the specimen in the holder may result in operator produced fracture lines in the specimen could alleviate this concern. Perhaps allowance for additional ledges at either end of the Teflon mould for the fabrication of the specimen. The presence of these ledges at the end of the specimen may help reduce any potential sliding of the specimen in the jig during testing.

As mentioned earlier, the results of this project warrant further analysis after validation using a modified NTP mould for the photo-curable composite resins. Another method of evaluation of the existing tested specimen is to perform SEM analysis on the fracture surfaces. This may allow one to delineate the reasons behind the variations between and within groups of materials in this study. With a total of 1008 samples fabricated and tested during this project an opportunity exists for developing a better understanding of the materials used in this study.

As indicated previously, there is ample data in the literature indicating a direct correlation between an increase in fraction volume filler of composite resins and a rise in their  $K_{IC}$  [Braem et al. 1989; Davis and Waters 1987; Drummond et al. 1998; Ferracane et al. 1987; Ferracane et al. 1998; Fujishima and Ferracane 1996; Htang et al. 1995; Lloyd 1982; Lloyd and Iannetta 1982; Lloyd and Mitchell 1984; Miyawaki et al. 1993; Pilliar et al. 1986; Zhao et al. 1997]. It would be helpful to investigate an optimum  $V_f$  which could give rise to a maximum  $K_{IC}$  in composite resins using the NTP  $K_{IC}$  test.

The effect of different filler shapes on the  $K_{IC}$  of composite resins should also be investigated. Different shapes of fillers could play a significant role in crack deflection and rise in  $K_{IC}$  values of composite resins. It is possible that the use of arc-shaped fillers without sharp angles and interlocking into each other could play a significant role in dissipating crack propagation. The NTP  $K_{IC}$  test may also be used to measure the  $K_{IC}$  of dental ceramics, because this is cumbersome using other methods due to significant brittleness of these materials. Use of magnetic resonance imaging (MRI) for observing the fracture dynamics and path under cyclic loading may also be entertained. Recent work in measuring the  $K_{IC}$  of human dentin provides a basis for studying the  $K_{IC}$  of the composite-dentin interface at different orientations of the dentinal tubules [Iwamoto and Ruse 2003].

## 5.7 Clinical Significance of our findings

The ultimate goal of this project is to provide an understanding of the clinical limitations of the experimental materials in different types of restorations. Ideally a restorative material should possess the same mechanical properties of the dental tissue that it replaces. The initial reports of the fracture toughness of human enamel were in the range of 0.7 to 1.27 MPa.m<sup>1/2</sup> [Hassan et al. 1981]. Such wide variation was primarily associated with the enamel rod orientation. The fracture toughness of dentin has also been measured and is reported to be in the range of 1.13 to 2.02 MPa.m<sup>1/2</sup> depending the relationship of direction of fracture to dentinal tubules [Iwamoto and Ruse 2003]. Modulus of dentin is about 18 GPa, while the modulus of enamel is considerably higher at 80 GPa [Ruse 1999].

Due to excessive wear, post-operative sensitivity, and secondary caries, the use of composite resins for restoring posterior teeth has been challenging for dental clinicians [Kelsey et al. 2000]. Recent formulations of composite resins have been promising due to a reduction in their annual wear to close to that of amalgam [Leinfelder et al. 1998]. There is strong evidence that the intrinsic properties of a materials, such as its  $K_{IC}$ , are better indications of clinical strength of a material than average stress-based tests [Goldman 1985; Kovarik and Muncy 1995; Lloyd and Mitchell 1984; Miyazaki et al. 1996b]. A strong correlation between  $K_{IC}$ ,  $\sigma_f$ , and  $E_f$  with surface chipping and bulk fracture of composite resins *in vivo* has been established [Tyas 1990]. Surface wear reduces with increased fracture toughness [Tyas 1990]. There is also a strong inverse correlation ( $r^2 = 0.914$ ) between the fracture toughness of a restorative material and marginal breakdown [Ferracane and Condon 1999].

Based on the results of this study and the preceding discussion, and in consideration of the limitations of the experimental design, one could delineate the suitability of different materials for different areas of the mouth. For the anterior region of the mouth esthetics is an important consideration. For anterior Class III and V restorations, GIC have the least desirable mechanical properties. With their low  $K_{IC}$  and  $\sigma_f$  they are bound to show significant wear and abrasion over time. Their moderate  $E_f$ , makes them more suitable for Class V posterior gingival abrasions, because they are expected to withstand the flexural stresses at the "neck" of the tooth. RMGIC, PAMCR, and microfil CR are expected to perform better than GIC, while offering the ability to achieve a fine gloss following polishing.

GIC have the least desirable properties for posterior restorations. Particularly due to their low  $K_{IC}$  and  $\sigma_f$ , GIC will end up with marginal breakdown, excessive wear, and bulk fracture when used in posterior teeth under high occlusal stresses. They may be suitable for very

conservative Class I restorations in primary dentition or as interim restorations in atraumatic restorative management of carious lesions. RMGIC provide the advantage of a fast cure by light polymerization, but would be susceptible in the short term. RMGIC, PAMCR, and microfil CR should perform better than GIC, but not significantly different from each other. If used in conservative, low stress bearing, areas in the posterior region for both primary and permanent dentition PAMCR and microfil CR should perform equally. They are, however, susceptible to surface wear and marginal breakdown in the long term.

For Class II restorations, Valiant PHD appears to possess the best intrinsic properties, although its moderate  $\sigma_f$  is an indication of its vulnerability in very large posterior restorations, particularly if its surface is not properly finished. Amalgam, hybrid CR, and PCR should perform well in low stress bearing areas.

Of all the materials tested, GIC are the weakest, while amalgam is the strongest overall materials. Hybrid CR and PCR should perform better than microfil CR if, and when, the esthetics demands for posterior restorations take precedence over their mechanical properties. Patients should be warned about the inferiority and reduced durability of multi-surface and stress-bearing posterior composite resin restorations when making decisions [Price 2003].

## Chapter VI. Conclusion

Within the limitations of this study and based on representative materials used for each group of dental materials, the following conclusions may be drawn:

1. GIC are the weakest materials tested as they have the lowest  $K_{IC}$  and  $\sigma_f$ , while their  $E_f$  is comparable to other materials, other than amalgam, which has the highest  $E_f$  amongst all materials tested. This means that GIC are prone to marginal breakdown, wear, and bulk fracture when placed in high stress bearing areas.
2. RMGIC appear to possess better mechanical properties than GIC, making their use under controlled and dry conditions much more favorable than GIC.
3. RMGIC possess similar strength properties to that of PAMCR, while both are inferior to CR and PCR. Their stiffness, however, is comparable to other light cured materials, and higher than the microfill HM.
4. The PAMCR, Dyract, has comparable  $K_{IC}$  to the hybrid CR Z100, while both are less resistant to fracture than microfill Heliomolar. F2000 presents with inferior fracture mechanics as compared to Dyract.
5. The microfil CR, Heliomolar, has a higher  $K_{IC}$  than the hybrid CR Z100, while having a lower  $\sigma_f$ . Heliomolar is also less stiff than Z100. Although the microfil Heliomolar shows higher fracture resistance, its lower  $\sigma_f$  prohibits its use in areas subject to high tensile stress. Its lower stiffness is also a detriment when used for large restorations. In conservative Class I restorations, however, microfill Heliomolar should exhibit good durability and marginal adaptation.
6. The PCR, Surefil and Prodigy had comparable  $\sigma_f$  and  $E_f$  to the hybrid CR Z100. Their  $K_{IC}$  were, however, lower than that of the microfil Heliomolar. PCR do not show any significant improvement over conventional composite resins.
7. The amalgam, Valiant PHD, had the highest stiffness amongst all materials tested. Its  $K_{IC}$ , however, was initially comparable to that of CR and PCR, but increased significantly to higher levels by 90-days. Valiant PHD, however, presented with moderate  $\sigma_f$  in the range of values from CR and PCR. This indicates that over time amalgam should have excellent wear properties, reduced marginal gap formation, and reduced bulk fracture, as long as it is not used in very large and extended restorations. Overall, amalgam appears to be the best material over time for restoring moderate sized cavities in posterior teeth.

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## Appendix I: Abbreviations

$\mu\text{m}$	meter x $10^{-6}$
$\Delta\text{pH}$	Change in pH
cm	meter x $10^{-2}$
CR	Composite Resin
CS	Compressive Strength
DMS	Diametral Tensile Strength
F	Fluoride
GIC	Glass-Ionomer Cement
HEMA	Hydroxy-ethyl methacrylate
m	meter
MIC	Minimum Inhibitory Concentration
mm	meter x $10^{-3}$
MPa	Mega Pascal
PAMCR	Polyacid-Modified Composite Resin
ppm	Parts per million
RDT	Residual Dentin Thickness
RMGIC	Resin-Modified Glass-Ionomer Cement
SS	Shear Strength
TS	Tensile Strength
VLC	Visible Light Cure
$K_{IC}$	Fracture Toughness
NTP	Notchless Triangular Prisms
$\sigma_f$	Flexural Strength
$E_f$	Flexural Modulus

## Appendix II. Some Physical Quantities and their Units [Hertzberg 1996]

Quantity	Symbol	Unit	Abbreviation
Length	$l$	inch	in
		meter	m
Wavelength	$\lambda$	meter	m
Mass	$m$	kilogram	kg
Time	$t$	second	s
Temperature	$T$	degree Celsius	°C
		degree Fahrenheit	°F
		kelvin	K
Frequency	$\nu$	hertz	Hz [s <sup>-1</sup> ]
Force	$F$	newton	N [kg·m·s <sup>-2</sup> ]
Stress:			
Tensile	$\sigma$	pascal	Pa [N·m <sup>-2</sup> ]
Shear	$\tau$	pounds per square inch	lb/in <sup>2</sup> or psi
Energy, work, quantity of heat		joule	J [N·m]
Power		watt	W [J·s <sup>-1</sup> ]
Current flow	$I$	ampere	A
Electric charge	$q$	coulomb	C [A·s]
Potential difference, electromotive force	$V, E$	volt	V
Electric resistance	$R$	ohm	$\Omega$ [V·A <sup>-1</sup> ]
Magnetic induction	$B$	tesla	T [V·s·m <sup>-2</sup> ]

### Greek Alphabet

Name	Lowercase	Capital	Name	Lowercase	Capital
Alpha	$\alpha$	A	Nu	$\nu$	N
Beta	$\beta$	B	Xi	$\xi$	$\Xi$
Gamma	$\gamma$	$\Gamma$	Omicron	$o$	O
Delta	$\delta$	$\Delta$	Pi	$\pi$	$\Pi$
Epsilon	$\epsilon$	E	Rho	$\rho$	$\rho$
Zeta	$\zeta$	Z	Sigma	$\sigma$	$\Sigma$
Eta	$\eta$	H	Tau	$\tau$	T
Theta	$\theta$	$\Theta$	Upsilon	$u$	Y
Iota	$i$	I	Phi	$\phi$	$\Phi$
Kappa	$\kappa$	K	Chi	$\chi$	X
Lambda	$\lambda$	$\Lambda$	Psi	$\psi$	$\Psi$
Mu	$\mu$	M	Omega	$\omega$	$\Omega$

### SI Unit Prefixes

Multiple	Prefix	Symbol
10 <sup>-12</sup>	pico	p
10 <sup>-9</sup>	nano	n
10 <sup>-6</sup>	micro	$\mu$
10 <sup>-3</sup>	milli	m
10 <sup>-2</sup>	centi	c
10 <sup>-1</sup>	deci	d
10 <sup>1</sup>	deca	da
10 <sup>2</sup>	hecto	h
10 <sup>3</sup>	kilo	k
10 <sup>6</sup>	mega	M
10 <sup>9</sup>	giga	G
10 <sup>12</sup>	tera	T

Example: 1 kilometer = 1 km = 10<sup>3</sup> meters.

### **Appendix III: Materials Index**

Advance (Caulk, Dentsply, DE, USA)  
ASPA IV (DeTrey Dentsply, Weybridge, England)  
Baseline (DeTrey Dentsply, Weybridge, England)  
Baseline VLC (DeTrey Dentsply, Weybridge, England)  
Bellfeel Brightener (Kanebo; Tokyo, Japan)  
Cavalite (Kerr, MI, USA)  
Chelon-Fil (ESPE GmbH, Germany)  
Chelon-Silver (ESPE GmbH, Germany)  
Chemfil II (DeTrey Dentsply, Weybridge, England)  
Chemfil II capsulated (DeTrey Dentsply, Weybridge, England)  
Chlorohexidine (Concepsis, Ultradent)  
Clearfil Photobond (Kuraray, Osaka, Japan)  
Compoglass (Vivadent, Lichtenstein)  
Copalite Resin Varnish (Cooley & Cooley, TX, USA)  
Delton (DeTrey Dentsply, Weybridge, England)  
Delton opaque (Johnson & Johnson, NJ, USA)  
Dycal VLC (DeTrey Dentsply, Weybridge, England)  
Dyract (DeTrey Dentsply, Weybridge, England)  
Dyract AP (Caulk, Dentsply, DE, USA)  
F2000 (3M, MN, USA)  
Fuji Cap II (GC Corp, Tokyo, Japan)  
Fuji I (GC Corp, Tokyo, Japan)  
Fuji II (GC Corp, Tokyo, Japan)  
Fuji II LC (GC Corp, Tokyo, Japan)  
Fuji III (GC Corp, Tokyo, Japan)  
Fuji Lining (GC Corp, Tokyo, Japan)  
Geristore (DenMat Corp, CA, USA)  
Gluma-3-step (Miles Inc.)  
Heliomolar (Vivadent, Lichtenstein)  
Herculite XRV (Kerr, Peterborough, UK)  
HiDense (Shofu, Kyoto, Japan)  
HiFi (Shofu, Kyoto, Japan)  
Instron Machine (Instron Co., MA, USA)  
Ionocap (Ionos, DMG, Hamburg, Germany))  
Ionoseal (Voco Chemie, Cuxhaven, Germany)  
Ketac bond (ESPE-Premier, PA, USA)  
Ketac Cem (ESPE GmbH, Germany)  
Ketac conditioner (ESPE, PA, USA)  
Ketac Varnish (ESPE GmbH, Germany)  
Ketac-Fil (ESPE GmbH, Germany)  
Ketac-Fil Applicap (ESPE GmbH, Germany)  
Ketac-Glaze ( ESPE, Premier Dental, PA, USA)  
Ketac-Molar(ESPE GmbH, Germany)  
Ketac-Silver (ESPE GmbH, Germany)  
Miracle Mix (GC Corp, Tokyo, Japan)  
Optinbond/XRV (Kerr, Peterborough, UK)  
Panavia21 (J. Morita, Kuraray, Osaka, Japan)

Pertac Universal Bond (ESPE GmbH, Germany)  
Pertac-Hybrid (ESPE GmbH, Germany)  
Photac-Fil (ESPE GmbH, Germany)  
Photac-Fil Applicap (ESPE, PA, USA)  
Prodigy-C (Kerr, Peterborough, UK)  
Protect Dentin Desensitizer (J.O. Butler, IL, USA)  
Scotchbond 2 (3M, MN, USA)  
Scotchbond MP (3M, MN, USA)  
Shofu Hy-bond (Shofu, Kyoto, Japan)  
Shofu II (Shofu, Kyoto, Japan)  
SureFil (Caulk, Dentsply, DE, USA)  
Timeline (Caulk, Dentsply, DE, USA)  
Tubulicid (Global)  
Valiant PHD (Ivoclar)  
Variglass (Caulk-Dentsply, Weybridge, England)  
Vitrabond (3M Co., MN, USA)  
Vitremer (3M, MN, USA)  
VLC Dycal ( L.D. Caulk, MI, USA)  
XR-Ionomer (Kerr, Peterborough, UK; MI, USA)  
Z100 (3M, MN, USA)

## **Appendix IV: Experimental Medium**

### **A4.1 IN VITRO EXPERIMENTS**

*In vitro* experiments are conducted under the controlled conditions of the laboratory (Anusavice, 1996). Modeling the unpredictable conditions of the oral cavity, however, remains an impossible task. Test specimens may be immersed in water, for example, in order to measure their release of ions. Artificial saliva, on the other hand, may be used in place of water as a milieu similar to that in the oral cavity. Extrapolation of the results of *in vitro* experiments to the multivariate environment of the oral cavity, however, is questionable.

### **A4.2 IN SITU EXPERIMENTS**

*In situ* experiments are carried out in such a way to recreate the conditions of the oral cavity (Anusavice, 1996). During an *in situ* test, specimens may be attached to a partial denture that is worn throughout the duration of the experiment, in effect subjecting the specimens to many more variables not present *in vitro*. *In situ* experiments are, therefore, more clinically relevant.

### **A4.3 IN VIVO EXPERIMENTS**

*In vivo* experiments that are carried out in the oral cavity, also have inherent experimental flaws (Anusavice, 1996). Sampling for site specific plaque, as an example, presents great challenges due to the possibility of infecting the sample during retrieval from the oral cavity. By evaluating the body of knowledge that is accumulated as a result of *in vitro*, *in situ*, and *in vivo* experiments, however, one is able to formulate a reasonable understanding of the events that take place in the clinical situation.

## Appendix V: Key to Table of Raw Data

Materials code	Tests code	Time code
1 = Ketac-Fil (P)	1 = KIC	1 = 1 hr
2 = Photac-Fil (Q)	2 = Flex	2 = 24 hr
3 = Dyract AP	3 = E	3 = 7 d
4 = F2000		4 = 30 d
5 = Z100		5 = 90 d
6 = Heliomolar		
7 = SureFil		
8 = Prodigy C		
9 = Valiant PHD XT		

It should be noted that 0.00 values have been entered for the three time intervals of 1 hour, 7 days, and 30 days for all three tests for Valiant PHD XT. In compiling data for Valiant PHD, time values of 1, 3, and 4 should not be included.

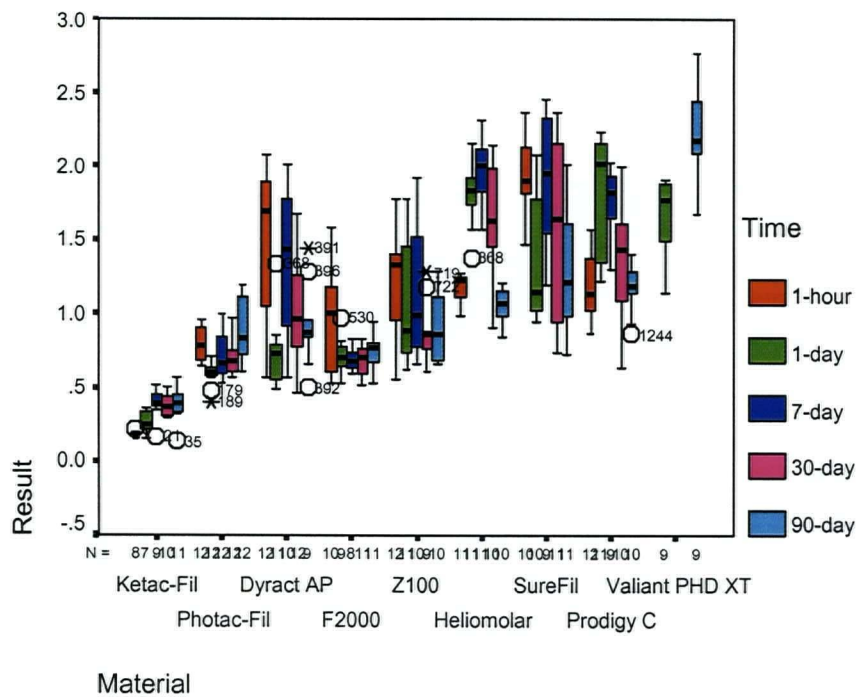
The number of data points per test could vary from 7 to 13 in some instances.

Out of range values, other than invalid data, have been entered as well for accuracy of statistical analysis.

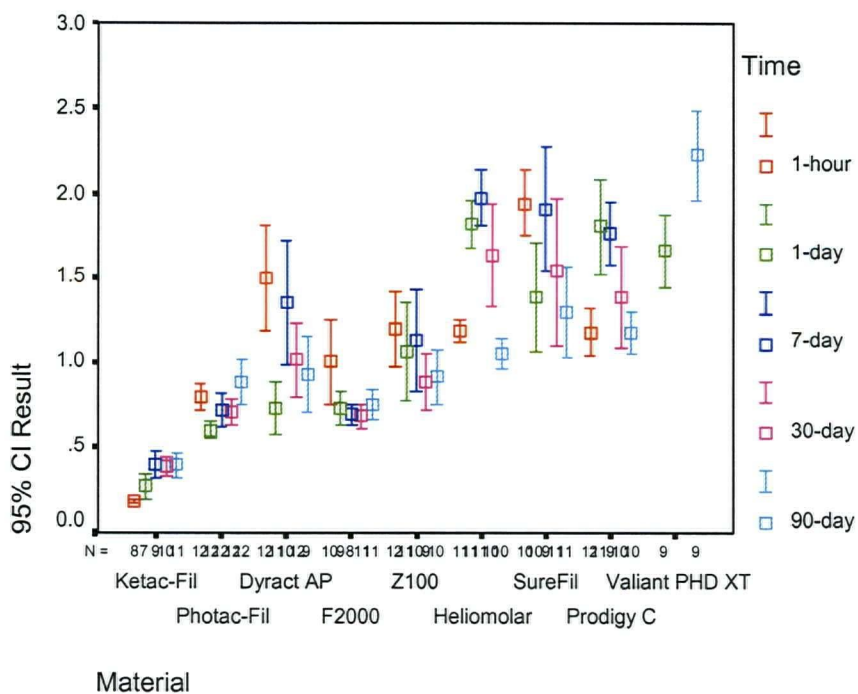


## Appendix VI: Master Plots

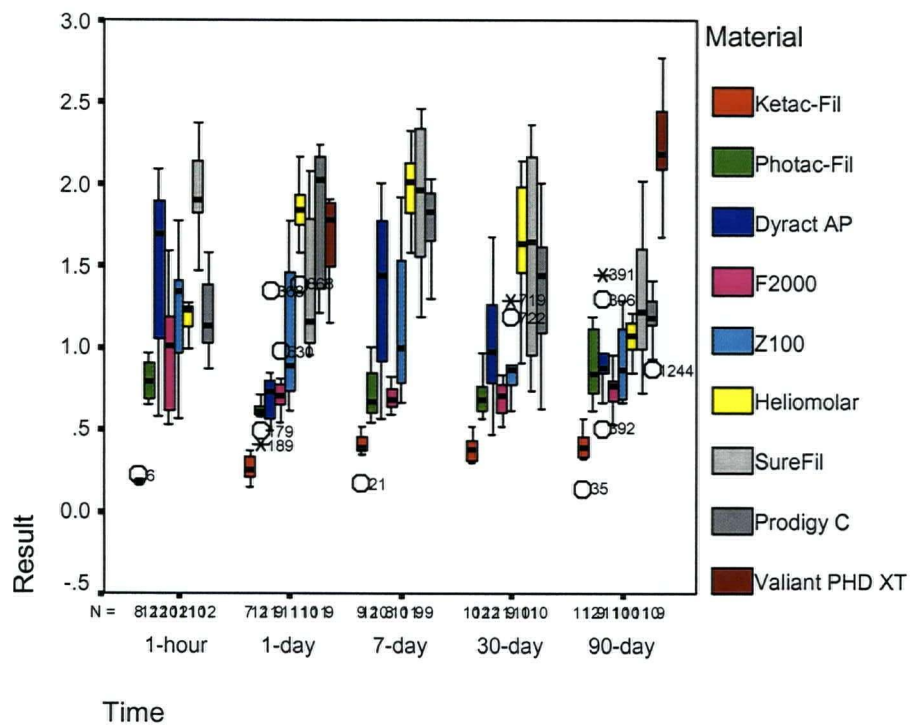
### Cluster Box Plot- KIC of all 9 Materials Over Time



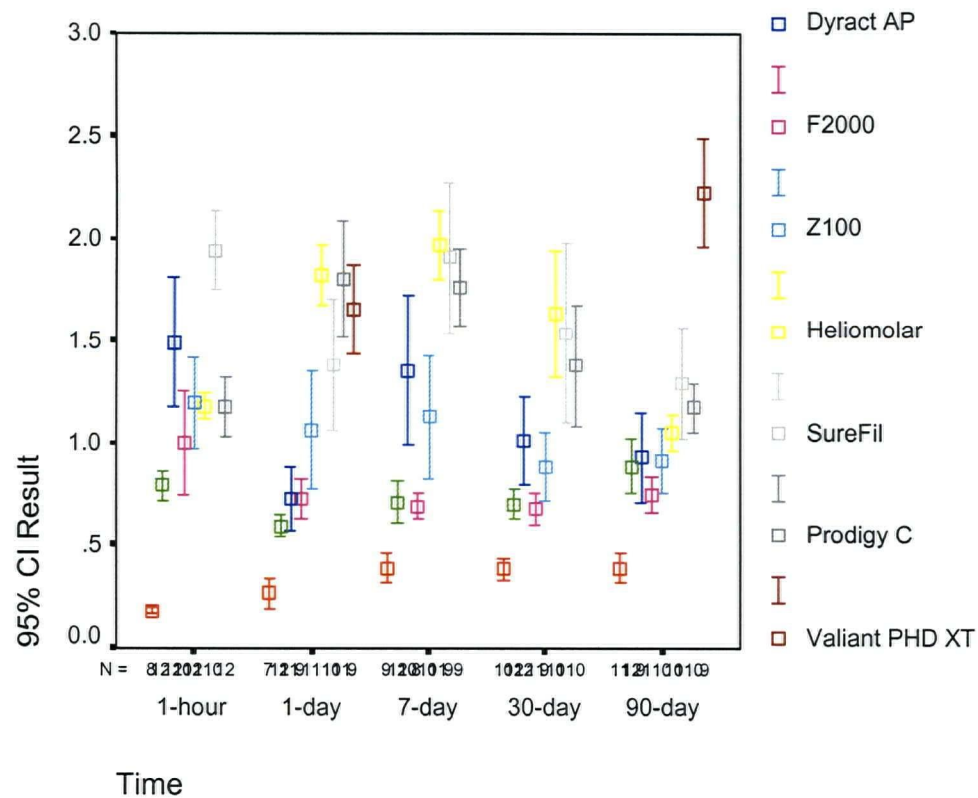
### Cluster Error Bar Plot- KIC of all 9 Materials Over Time



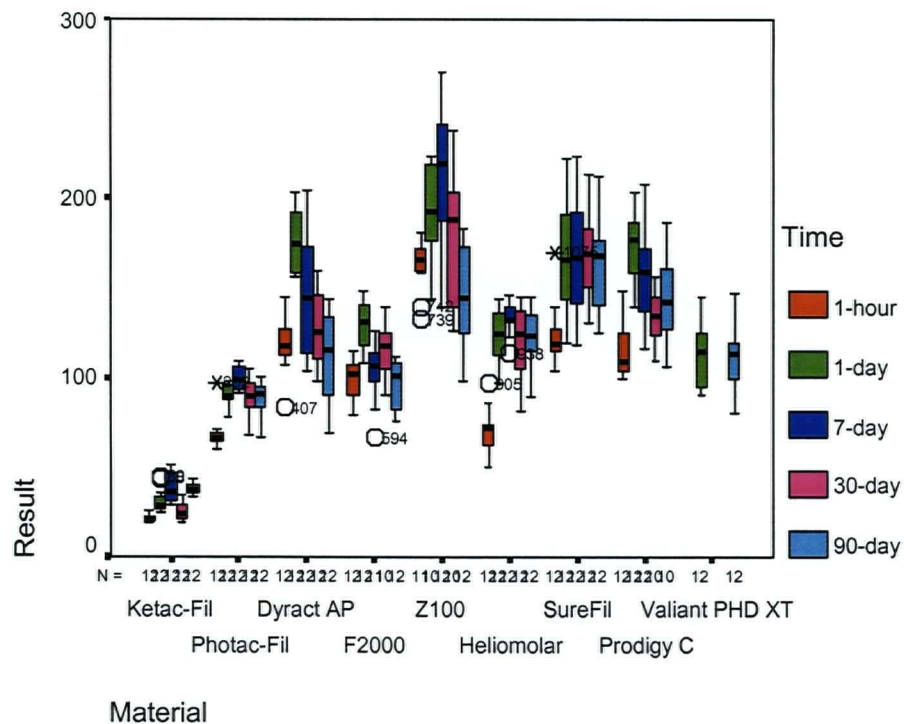
## Cluster Box Plot- KIC of all 9 Materials at Each Time



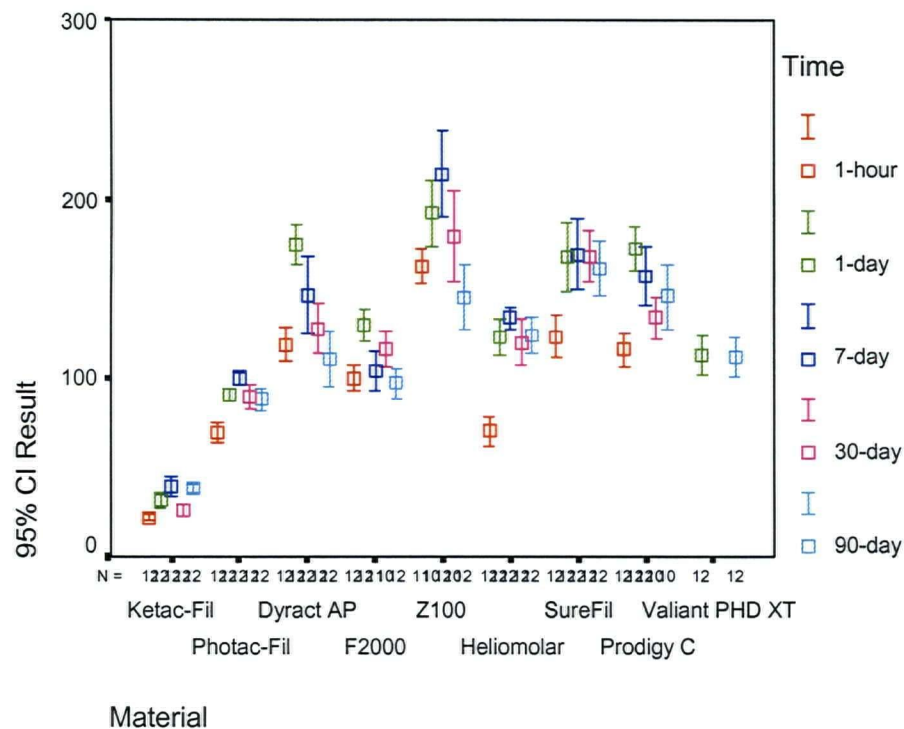
## Cluster Error Bar Plot- KIC of all 9 Materials at Each Time



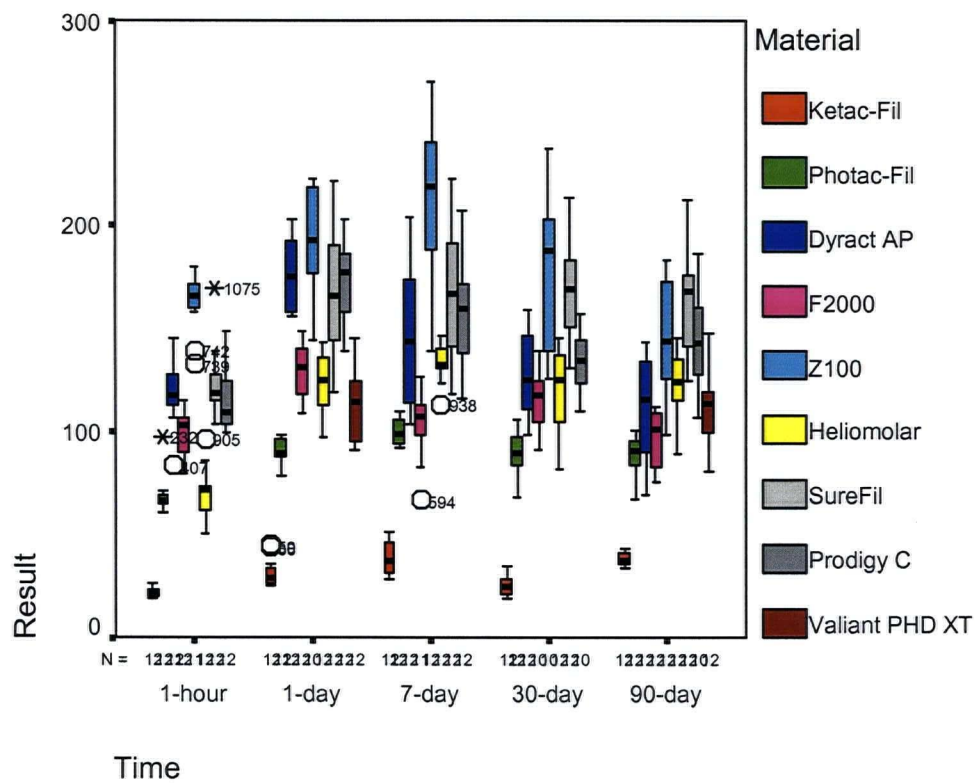
## Cluster Box Plot-Flexural Strength of all Materials over Time



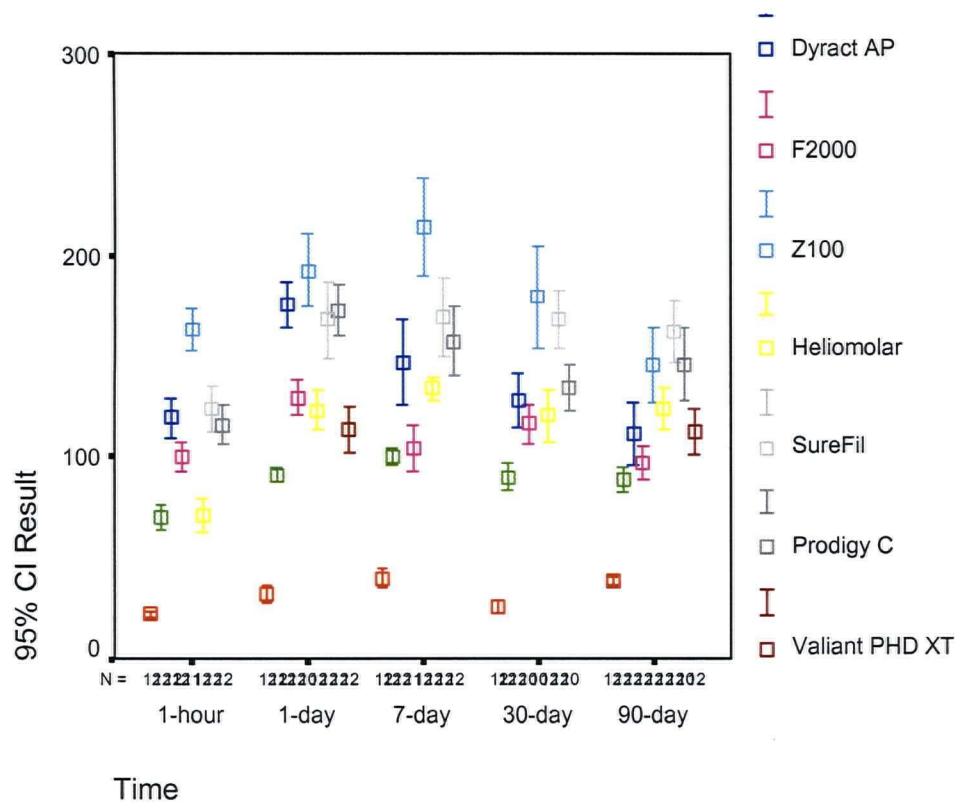
## Cluster Error Bar-Flexural Strength of all Materials over Time



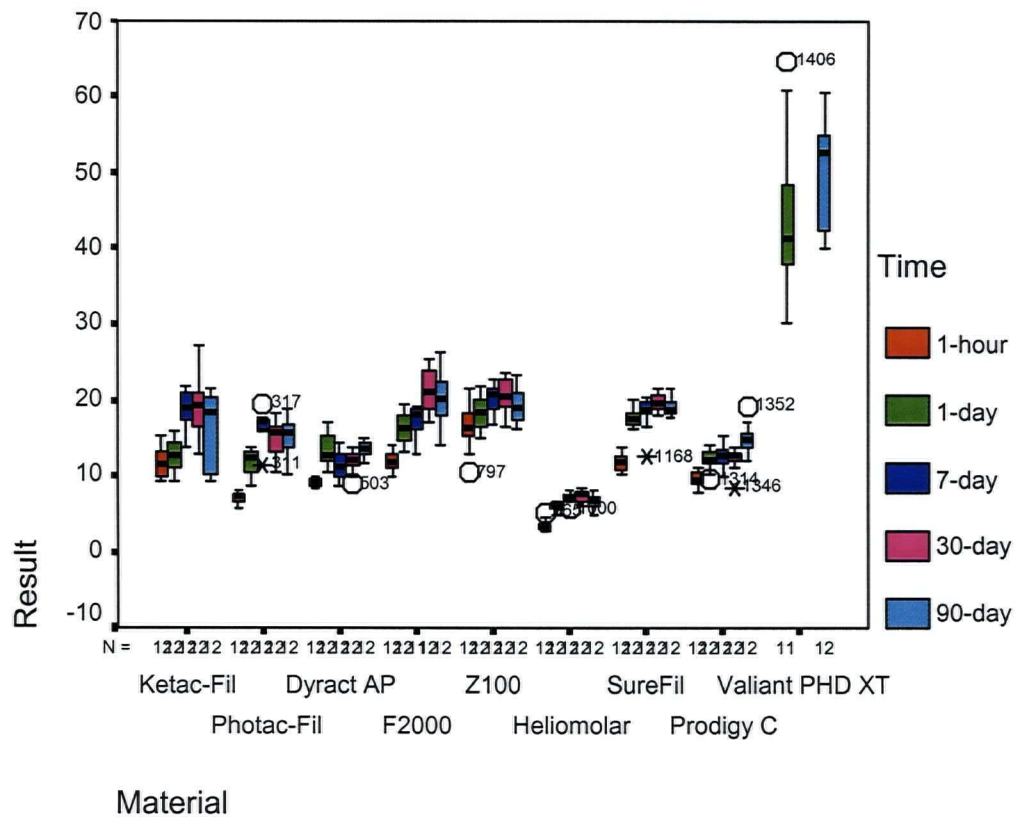
## Cluster Box Plot-Flexural Strength of all Materials at Each Time



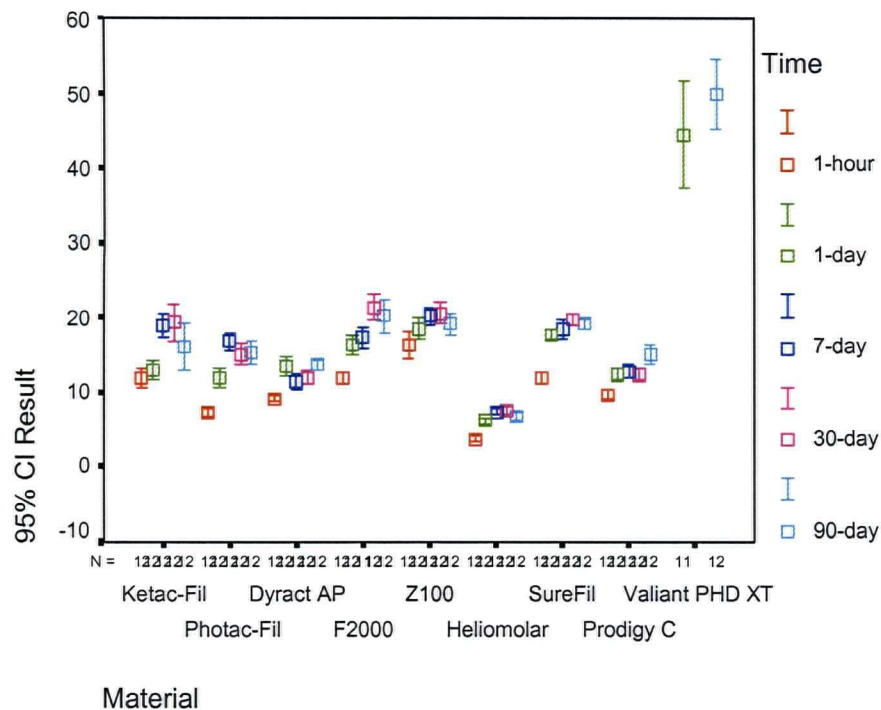
## Cluster Error Bar-Flexural Strength of all Materials at Each Time



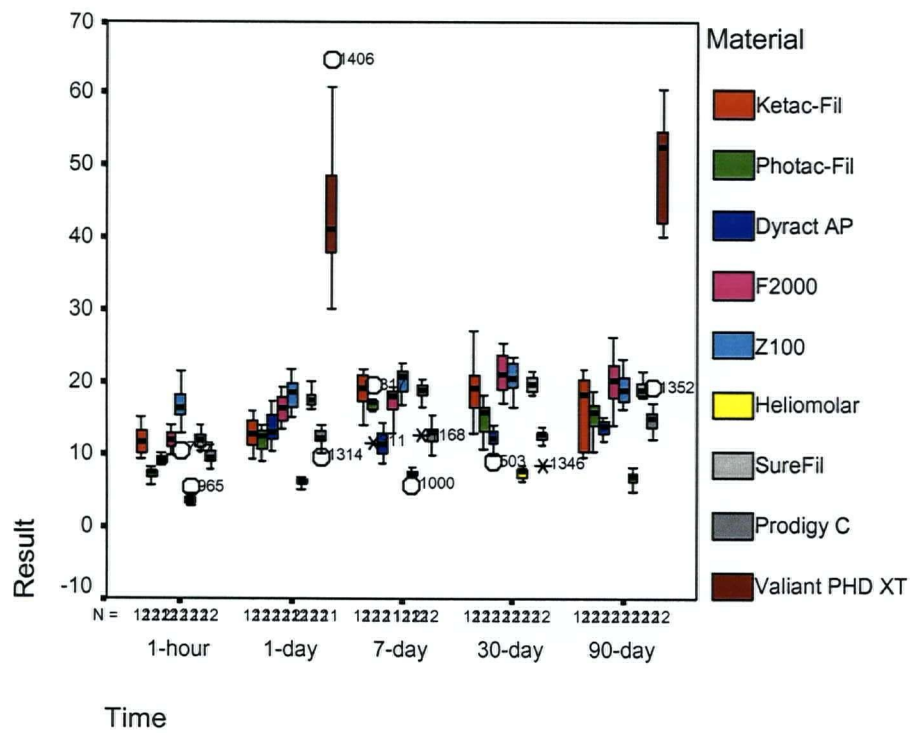
### Cluster Box Plot-Flex Mod of all Materials over time



### Cluster Error Bar-Flex Mod of all Materials over time



## Cluster Box Plot-Flex Mod of all Materials at Each time



## Cluster Error Bar-Flex Mod of all Materials at Each time

