

VYSOKÉ UČENÍ TECHNICKÉ V BRNĚ

BRNO UNIVERSITY OF TECHNOLOGY

FAKULTA CHEMICKÁ
ÚSTAV CHEMIE MATERIÁLŮ

FACULTY OF CHEMISTRY
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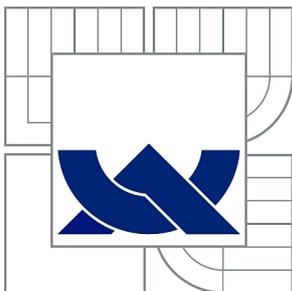
EFFECT OF INTERLAYER COMPOSITION ON BOND STRENGTH
BETWEEN FRC
FRAMEWORK AND COMPOSITE VENEER

DIPLOMOVÁ PRÁCE
MASTER'S THESIS

AUTOR PRÁCE
AUTHOR

Bc. JONATANH JOSÉ IBARRA

BRNO 2010



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VLIV SLOŽENÍ MEZIVRSTVY NA PEVNOST ADHEZNÍHO SPOJE MEZI VLÁKNOVÝM A ČÁSTICOVÝM KOMPOZITEM

DIPLOMOVÁ PRÁCE

MASTER'S THESIS

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BRNO 2010



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Master's thesis Assignment

Number of master's thesis: **FCH-DIP0367/2009** Academic year: **2009/2010**
Institute: Institute of Materials Science
Student: **Bc. Jonatanh José Ibarra**
Study programme: Chemistry, Technology and Properties of Materials (N2820)
Study field: Chemistry, Technology and Properties of Materials (2808T016)
Head of thesis: **Ing. Petr Poláček, Ph.D.**
Supervisors:

Title of master's thesis:

Effect of Interlayer Composition on Bond Strength between FRC Framework and Composite Veneer

Master's thesis assignment:

Literature review

Current state of problem of adhesion between composite materials

Preparing of particulate filled and fiber reinforced composites with dimethacrylate matrix

Preparing of samples with different thickness of adhesive interlayer

Determination of strength of adhesive joint

Deadline for master's thesis delivery: 14.5.2010

Master's thesis is necessary to deliver to a secretary of institute in three copies and in an electronic way to a head of master's thesis. This assignment is enclosure of master's thesis.

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In Brno, 1.12.2009

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ABSTRACT

The aim of the investigation is to study the current state of adhesion problems between composite materials and determine the influence of interlayer composition in the bond strength of the adhesive joint between FRC frameworks and composite veneer (PFC) for dental applications.

Samples consisting in FRC rods made with commercial dimethacrylate and S-glass fibers and PFC made of a resin mixture (bis-GMA and PEGDMA) with barium glass powder as the filler were constructed. A total of 84 samples were prepared and measured. The samples were divided into five different groups. The first group was made of series of samples without any interlayer. The other four groups were divided according the composition of the interlayer (thickness and resins used). PFC of the specimens differed in the filler content (0, 10, 40 wt. %). The FRC rod was identical for all samples. The universal testing machine ZWICK Z010 was used to determine the shear strength of adhesive joint. A Scanning Electron Microscope (SEM) was used to study the loci of failure.

Results show that the addition of an interlayer between the fiber-reinforced and particle filled composite, resulted in an improvement on the shear strength bond. This is very important, since the use of these materials in dentistry has been increasing in the last years and adhesion still reminds one of the biggest problems in clinical application.

KEY WORDS

Dental Composites, FRC, Fiber-Reinforced Composite, PFC, Particle Filled Composite, Adhesion, Shear Bond Strength, Interlayer Composition.

ABSTRAKT

Hlavním cílem této práce je studium aktuálního problému adheze mezi kompozitními materiály, a určení vlivu složení mezivrstvy v pevnosti adhezivního spoje mezi vláknové (FRC) a částicové (PFC) kompozity, používané ve stomatologii.

FRC tyčinky byly vytvořené na bázi komerční dimetakrylatové pryskyřice a skleněných *S* vláken. PFC byl vytvořen ze směsi pryskyřic (bis-GMA a PEGDMA), plněných drceným barnatým sklem. Celkově bylo připraveno a vyzkoušeno 84 vzorků. Vzorky byly rozdělené do pěti hlavních skupin. První skupina byla vytvořena ze série vzorků bez mezivrstvy. Zbylé čtyři skupiny byly rozděleny dle složení mezivrstvy (tloušťky a druhu pryskyřic). Částicové kompozity vzorků se lišily obsahem plniva (0, 10, 40 hm %). FRC tyčinky byly stejné pro všechny vzorky. Univerzální testovací přístroj ZWICK Z010 byl použit k zjištění smykové pevnosti adhezivního spoje všech vzorků. Rastrovací elektronový mikroskop byl použit k pozorování místa porušení.

Ze získaných výsledků vyplývá, že s přidáváním mezivrstvy mezi vláknovým a částicovým narůstá smyková pevnost spoje. Důležitost těchto výsledků je způsobená tím, že v posledních letech použití těchto materiálů ve stomatologii narůstá a adheze zůstává jeden z hlavních problémů při klinické praxi.

KLÍČOVÁ SLOVA

Dentální kompozity, FRC, vláknové kompozity, PFC, částicové kompozity, adheze, smyková pevnost, složení mezivrstvy.

IBARRA, J. *Effect of interlayer composition on bond strength between FRC frameworks and composite veneer*. Brno: Brno University of Technology, Faculty of Chemistry, 2010. 51 p. Supervisor: Ing. Petr Poláček PhD.

DECLARATION

I declare that my master's thesis was worked out independently and that the used references are quoted correctly and fully. The content of the above mentioned thesis is considered a property of BUT, Faculty of Chemistry and can be used for commercial purposes only with the supervisor's and dean's consents.

In Brno May 14th 2010

.....

Author's signature

PROHLÁŠENÍ

Prohlašuji, že diplomová práce byla vypracována samostatně, a že všechny použité literární zdroje jsou správně a úplně citovány. Tato práce je z hlediska obsahu majetkem Fakulty chemické Vysokého učení technického v Brně a může být využita ke komerčním účelům jen se souhlasem vedoucího diplomové práce a děkana FCH VUT.

V Brně 14. Května 2010

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Podpis autora

Acknowledgments:

I would like to thank in first place to my supervisor Ing. Petr Poláček PhD., which during the whole year has been helping me with this investigation, giving me all kind of technical and theoretical advices. Of course to my nearest family for all the support, they gave to me during my entire career. And last but not least to Helena Ambrožová, who had stood by me in the good and the bad times during the last two years.

TABLE OF CONTENTS

1.	INTRODUCTION	8
2.	GOAL OF THE WORK	10
3.	THEORETICAL PART	11
3.1.	Dental Composite Materials	11
3.1.1.	Dental Resins	11
3.1.2.	Fiber Reinforcements	15
3.1.3.	Filler Particles	17
3.1.4.	Main Clinical Applications	18
3.2.	Adhesion	19
3.2.1.	Theory of Diffusion	20
3.2.2.	Theory of Mechanical Adhesion	20
3.2.3.	Theory of Adsorption	21
3.3.	Surface and Interface Interactions	23
3.3.1.	Boundary Layer Theory	23
3.3.2.	Interphase region	24
3.3.3.	Influence of the Interface on the Mechanical Properties of Composites	25
3.4.	Fracture of Adhesive Bond	26
3.4.1.	Griffith Fracture Criterion	26
3.4.2.	Stress at a Crack Tip	27
3.4.3.	Locus of Failure	28
4.	EXPERIMENTAL PART	29
4.1.	Materials and Devices	29
4.1.1.	Resins and Photo-initiation Complex	29
4.1.2.	Reinforcements	30
4.1.3.	Devices	30
4.2.	Resin Mixture Preparation	30
4.3.	PFC Preparation	30
4.4.	FRC Preparation	31
4.5.	Interlayer Preparation	32
4.6.	Sample Preparation	32
4.7.	Measurement Techniques	34

4.8. Equations and Calculations	35
5. RESULTS AND DISCUSSIONS	36
6. CONCLUSION	43
7. REFERENCES	44
8. LIST OF USED SYMBOLS AND ABBREVIATIONS	47
9. APENDIX.....	48

1. INTRODUCTION

During the last few decades the development of adhesive dentistry has changed the concepts of restorative dentistry and directed considerable attention to minimally invasive, tissue-saving treatment alternatives. There is an increasing number of procedures in current dentistry using adhesive bonding as means of attachment of prosthetic and orthodontic devices directly to the tooth structure, i.e., to enamel or dentin [1][2].

Most of the composites used to fill the cavities, veneers used to sculpture artificial teeth in prostheses and some adhesives used in current dentistry are based on a mixture of dimethacrylate monomers filled with particulate fillers and can be cured in situ using visible light. Particulate filler composites (PFC) provide successful, durable intra-coronal restorations; however, PFCs are reported not to have enough flexural strength to replace a missing tooth. In recent years, various types of fiber-reinforced composites (FRC) posts have been introduced in order to provide the dental profession with an alternative to cast or prefabricated metal posts for the restoration of endodontically closer to that of dentin than that of metal posts. The combination of PFC with FRC for intraoral use has shown to provide significant benefits in terms of mechanical properties. Additionally, the possibility of direct chair side application and the ability to bond to tooth structure make FRC an attractive candidate for dental applications [1][3].

The phenomenon of adhesion has revolutionized dentistry in recent decades. However, despite major technological advances in the area of dental materials, an entirely predictable adhesive restoration technique has not been found yet; today's adhesive techniques are sensitive in each of clinical stages, therefore it is important to know and use a set of variables to optimize clinical outcomes [4]. The adhesion between a substrate and an adhesive is usually considered to be dependent on two types of characteristics: the surface properties of both the materials, and the textural characteristics of the substrate [5].

Strong bonding is often demanded for composite materials under loading. However, a strong interfacial bonding increases the strength of multiphase materials at the expense of the material toughness. Functional interlayers are successfully used in fiber-reinforced composites to eliminate this conflict of material parameters, where the interlayer has to enable the stress transfer from the matrix to the fiber [6]. The interface adjusts the composite's mechanical resistance: here, concentration of mechanical stress occurs, as a result of the differences between the matrix's coefficient of thermal expansion and the coefficient of the filler or reinforcing agent, due to contraction at cross-linking and to crystallization. At the same time, it acts as a site for the production of chemical reactions [7].

The interface between the filler or reinforcing material and the polymeric matrix is essential in polymeric composites and involves challenging scientific and technological problems, and the complex issue of interface design and properties has a direct repercussion on the behavior and properties of the composite. Despite significant improvements in both sealing and bonding capabilities of dental adhesives systems, the bonded interface remains the weakest area of the adhesive joint [8][9].

In all applications of FRC, there is a need to cover the FRC framework with a PFC. In contrast to the in vitro studies showing good adhesion between FRC framework and PFC, the clinical failures reported in the literature were mostly delaminating or debonding of veneering

PFC from the underlying FRC framework. Therefore, adequate bonding of the PFC to the FRC substrate is critical for the longevity of the restoration. The traditional laboratory testing methodology used to characterize the adhesion between materials is based on shear bond strength measurements.

Shear bond strength tests have been shown to be sensitive to many parameters. Consequently, the results achieved between different test centers and testing devices are not always comparable. The specimen design and specimen preparation variables such as the height of the adherend material, the thickness of the intermediate resin layer, the polymerization conditions and the cross-sectional bonding area have been reported to influence the shear bond strength values achieved. Despite these documented limitations, the shear bond strength test has remained the standard method routinely used to evaluate the adhesion properties of dental materials to tooth substrate. Some studies have concluded that the tooth substrate anisotropy, specifically the orientation of the enamel prisms to the applied load direction, influences the bond strength values. This finding raises the hypothesis that, like enamel, the anisotropic nature of FRC as a substrate may result in differences in the bond strength.

Even though, a large amount of literature exists on adhesion of filling composites to dentin and enamel, one of the fundamental problems in the field of adhering restorations, concerns the control of the degree of adhesion between the various dental materials used in manufacturing the dental devices [2][8]. As a result, the aim of the current study was to determine the influence of interlayer composition in the bond strength between FRC frameworks and composite veneer for dental applications were studied.

2. GOAL OF THE WORK

The aim of the study is to study the current state of problem of adhesion between composite materials and determine the influence of interlayer composition in the bond strength of the adhesive joint between FRC frameworks and composite veneer (PFC) for dental applications.

For determination of strength of adhesive joint, the universal testing machine ZWICK Z010 will be used to determine the strength of adhesive joint of prepared samples consisting on FRCs posts and PFCs ring with different interlayer's composition. Scanning Electron Microscope (SEM) was used to study the loci of failure.

3. THEORETICAL PART

3.1. Dental Composite Materials

Dental materials are among the most used substitutes for biological tissue in the human body and half of the population in industrial countries receives new dental restorations annually. Many persons receive the materials in childhood and they will stay in the body for a large part of the person's life. Due to their common use and longevity in the body, dental materials should have a low toxicity [10]. In addition to biocompatibility, dental application needs the materials to meet very demanding requirements, like aesthetic and high mechanical performance. Physical requirements include low thermal conductivity and low density [11][12].

Some of the strong factors influencing the usage of fibrous composite in dentistry are; *Aesthetics*, with a variety of ceramic fiber and transparent matrix systems, almost all type of dental shades can be obtained. *Non-corrosive*, the polymer matrix composites are less susceptible to corrosion compared to the metal alloys. *Toughness*, the polymer matrix composites are not brittle. Hence the problem of abrasion and fracture is reduced. *Metal allergy* many people exhibit an allergic reaction to the presence of metallic devices in the body. The devices made of polymer composites eliminate such allergic reaction. *Chairside handling* many of the restoration and fabrication of dental devices need to be done in-situ.

Composite materials used in dental restoration consist of a continuous polymeric matrix, usually involving the visible-light-curing bisphenol- α -glycidyl methacrylate (bis-GMA) diluted with triethyleneglycol dimethacrylate (TEGDMA) reinforced with a dispersed phase (barium or zinc glasses, quartz, zirconia, silica, etc) [13].

The excellent handling properties of the fibers and matrix systems, capability of the composite to adapt to any shape makes FRC highly suitable for a wide range of dental applications like removable dentures, root canal posts, provisional restorations and fixed partial dentures (FPDs) [11][14].

3.1.1. Dental Resins

Many of the resins used in dentistry are made from 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropyl)phenyl]propane, commonly known as bis-GMA. The main components have been identified as a linear bis-GMA and a branched bis-GMA.

Bis-GMA is used because of its large molecular size, which has the effect of reducing the proportion of the molecule that undergoes polymerization, hence ensuring a low overall polymerization shrinkage of about 2%. However, bis-GMA has a high viscosity monomer and hence low viscosity reactive co-monomers, such as triethyleneglycol dimethacrylate (TEGDMA) and polyethyleneglycol dimethacrylate (PEGDMA) are commonly added as diluents [15].

The selection of the monomers strongly influences the reactivity, viscosity and polymerization shrinkage of the composite paste, as well as the mechanical properties, water uptake, and swelling by water uptake of the cured composite [16]. TEGDMA and PEGDMA are cross-linking agents. By cross-linking physical and mechanical properties of the polymer are improved, because the molecular movement of the single chains is restricted [15].

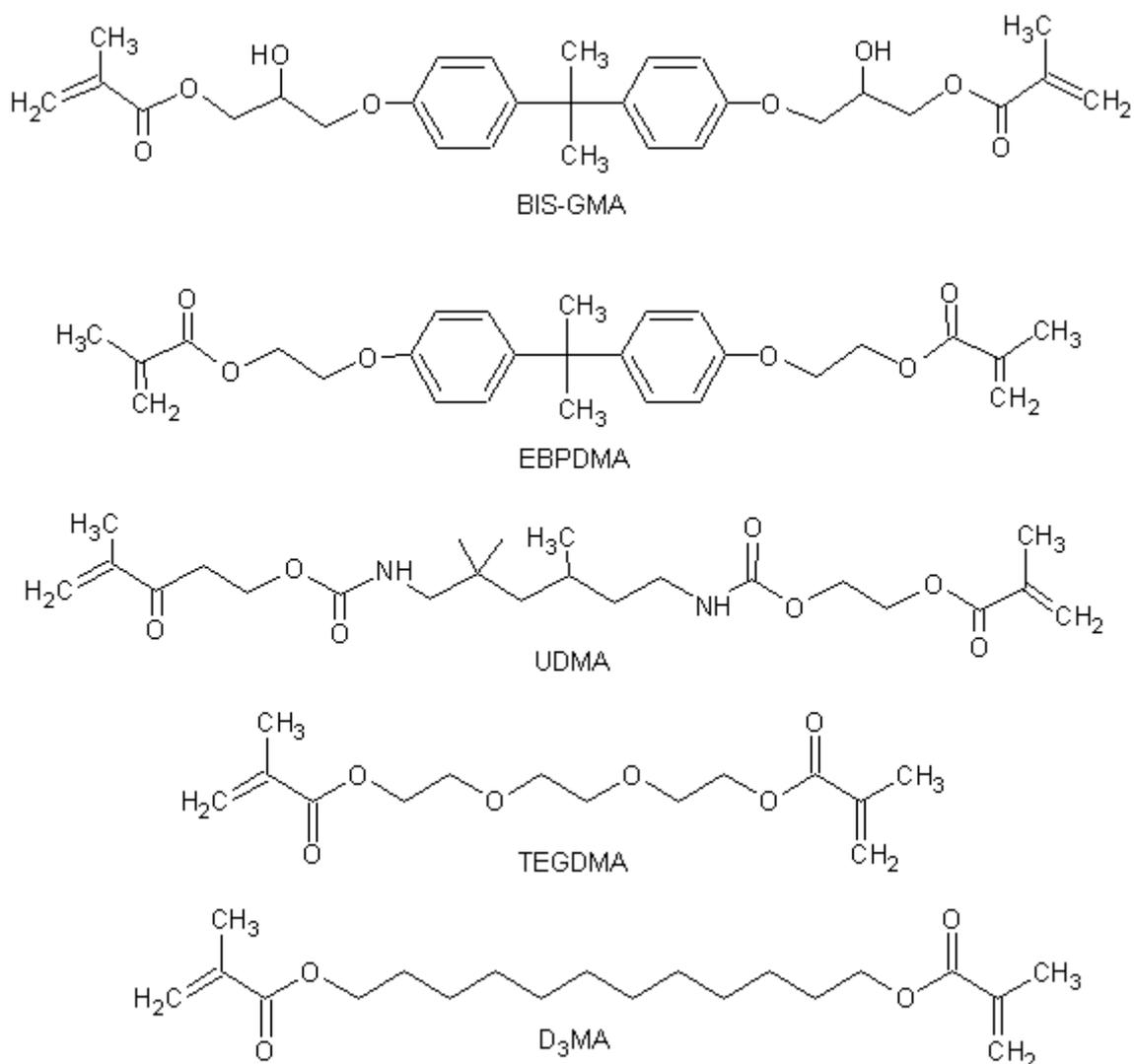


Fig. 1 Dimethacrylates mostly used in dentistry [16].

3.1.1.1. Methyl Methacrylate

Methyl methacrylate is a clear, transparent liquid at a room temperature with a characteristic sweet odor. It exhibits a high vapor pressure and is an excellent organic solvent. Although ultraviolet light, visible light, or heat can initiate the polymerization of methyl methacrylate, it is commonly polymerized in dentistry by the use of a chemical initiator [17][18].

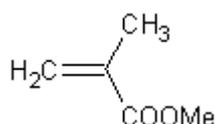


Fig. 2 Methyl methacrylate molecule

In dentistry, the liquid monomer methyl methacrylate is mixed with the polymer, which is in the powder form. The monomer partially dissolves the polymer to form plastic dough. This dough is packed into the mold, and the monomer is polymerized [18].

3.1.1.2. Polymerization of PPMA

Dental resins solidify when they polymerize. Polymerization occurs through a series of chemical reactions by which the macromolecule, or the polymer, is formed from large numbers of molecules known as monomers. Most dental resins are polymerized by addition polymerization. The macromolecules are formed from smaller units, or monomers, without change in composition, because the monomer and polymer have the same empirical formulas. In other words, the structure of the monomer is repeated many times in the polymer.

The addition polymerization process occurs in four stages: induction, propagation, termination and chain transfer [18]. The reaction may be accelerated by heat, UV light, peroxide, etc. In any case, the reaction must be initiated by free radical which may be created by heat, chemical or photochemical reactions.

In the **initiation** stage the free radical is produced and reacts with a molecule of monomer by opening the unsaturated double bond and transferring the free electron to the end of the chain being formed. The monomer molecule is activated and able to bond another monomer molecule by again transferring the free electron to the end of the growing chain. This addition of monomer molecules to the already existing structure is called the stage of **propagation**. Propagation continues until all free radicals have been joined or until no adjacent linkages are available. At this point, the reactions stop, this stage is called **termination** [15].

Two types of activation of polymerization are used in dental adhesives. The first method involves free-radical initiation. The material is supplied as a two-paste system, one containing benzoyl peroxide (BPO) as a free radical initiator, the other containing an accelerator, typically a tertiary amine such as N,N-bis(2-hydroxyethyl)-p-toluidine, which on mixing the two components, stimulates the fragmentation of the BPO into free radicals at room temperature, thus initiating polymerization of the unsaturated methacrylate groups.

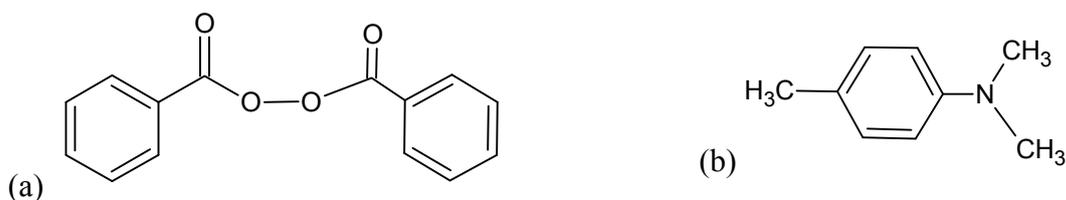


Fig. 3 (a) Benzoyl peroxide and (b) N,N-dimethyl-p-toluidine.

The second method is cure by visible light or by external energy in the form of ultraviolet light (UV). An excited state is then induced by the absorption of light quanta. The polymerization is induced by the photolytic dissociation of initiators sensitive to light or UV radiation. A typical photo-initiator, which reacts with UV radiation with a wavelength of 365 nm, is benzoin methyl ether [15]. However, because of concerns about the effect of UV light on the retina, an un-pigmented oral tissue, the limited penetration depth, and the reduction in intensity of ultraviolet light source over time, systems activated by visible light were developed [18]. For systems using visible light, a diketone such as camphoroquinone (approximately 0.2 per cent) is used in combination with an organic amine such as N,N-dimethylaminoethylmethacrylate. The diketone absorbs light in the 420 to 450 nm range, and an excited triplet state is produced which, together with the amine, results in ion radicals to initiate the polymerization [19].

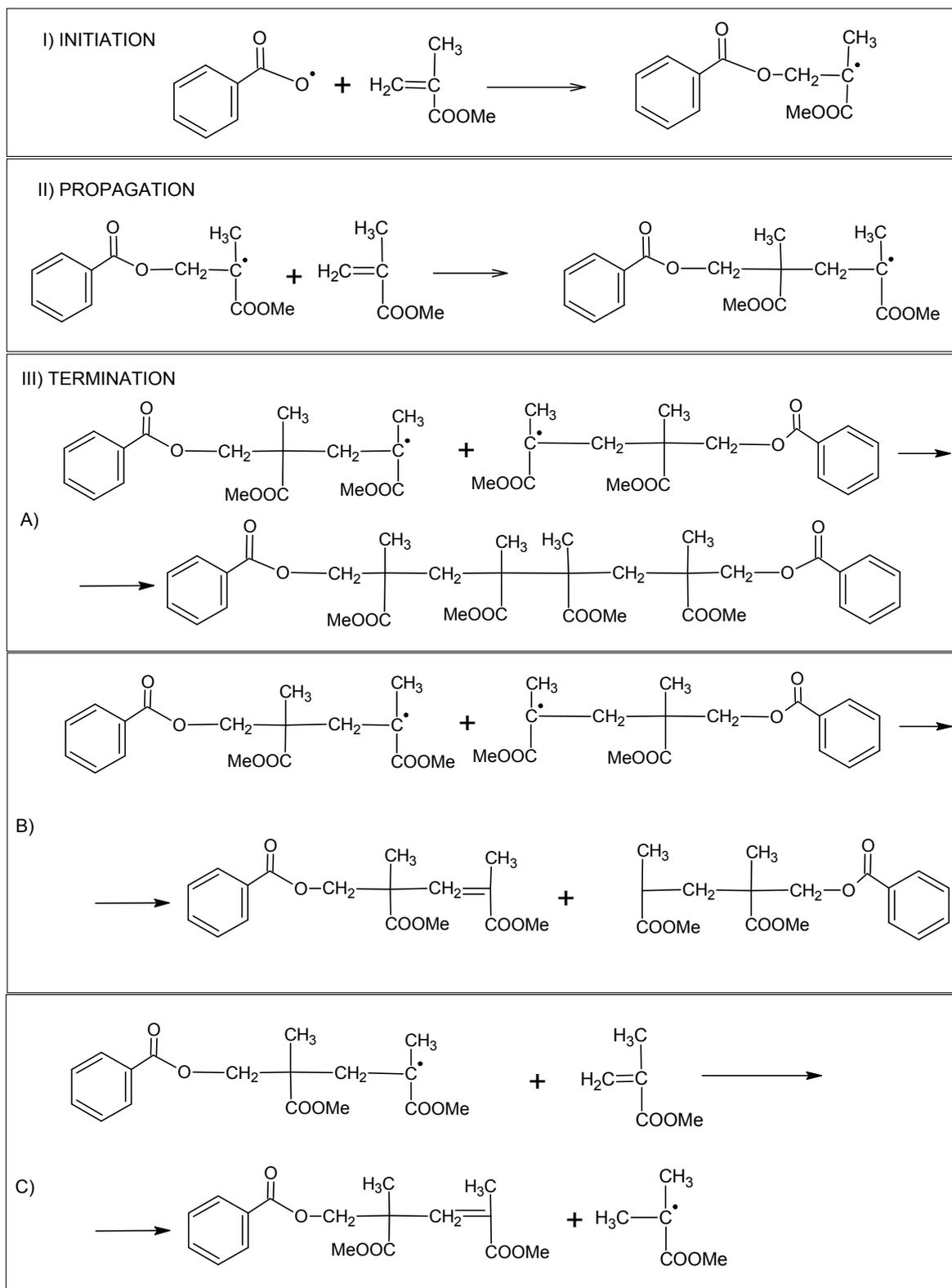


Fig. 4 Addition polymerization of PMMA – I) Initiation stage, II) Propagation stage III-A) Termination stage, III-B) Chain termination of two free radicals, III-C) Chain transfer between a free radical and an inactivated molecule.

3.1.2. Fiber Reinforcements

The usage of fiber composite materials has been increasing in volume and applications. Initially, fibrous composites found application in weight critical aerospace components. Later, the domain enlarged to infrastructure applications with additional performance requirements like, environmental stability, mold-ability, damage resistance, etc. Nowadays, with biocompatible fibers and matrix systems, fiber composites have found application as biomaterial. A number of fiber composite implants and devices for dental applications have been developed [11].

Non-reinforced resin can have a low density and relatively good stability against environment and chemicals. However, their stiffness and elastic modulus are low, which result is a relatively bad creep performance. On the other side, the fiber has good stiffness and elastic modulus but they do not have a good environmental and chemical resistance. When it is made as a Fiber Reinforced Composite, a new material is obtained that attains a value of the given physical property, not attainable by the single components [20].

The properties of FRCs that make them well suited for their application in dentistry include strength, desirable esthetic characteristics (translucency), simple of use, adaptability to various shapes; potential for direct bonding to tooth structure, lower thermal coefficient of expansion and higher resistance to abrasion thereby improving clinical performance of dental materials [12].

3.1.2.1. Principal Fiber Materials

The most used reinforcements in FRC are **glass fibers** based on silica (SiO_2) with the additions of oxides, of calcium, boron, sodium, iron and aluminum. The glass fibers can be divided by the type of glass used to make it, in E-glass, S-glass and C-glass. The most commonly fibers used are E-glass (E- for electrical), these fibers has good strength, stiffness, electrical and weathering properties. The S-glass fibers (S- for strength) have higher strength, Young's modulus and temperature resistance than E-glass fibers. The C-glass fibers (C for corrosion) are chemical more resistant, but have lower mechanical properties then E-glass [12].

Carbon fiber is a polymer which is a form of graphite. Graphite is a form of pure carbon. In graphite the carbon atoms are arranged into big sheets of hexagonal aromatic rings. The atoms in these basal planes are held together by strong covalent bonds, with only weak van der Waals forces between them. Carbon fibers are the most common high strength and high modulus reinforcing fibers used in composites for high performance applications, in dentistry are used because of favorable mechanical properties in screw-retained implant-supported fixed prostheses [21].

Aramid fibers are fibers in which the fiber-forming substance is a long-chain synthetic polyamide in which at least 85% of the amide (-CO-NH-) linkages are attached directly between two aromatic rings. Aramid Fibers held the distinction of having the highest strength-to-weight ratio of any commercially available reinforcement fiber at the time [22].

Ultra-high-molecular-weight-polyethylene (UHMWPE) fibers, produced by gel spinning with an ultra-high draw ratio, have a high degree of crystallinity and low density, resulting in improved physical characteristics such as ultra-high strength and modulus and,

good impact resistance and so on. Therefore, these fibers meet all the physical requirements for the fibers used in reinforced composite materials, except for the chemical inertness of UHMWPE, which implies that an effective fiber-matrix adhesion should not be expected [23][24]. Table 2 shows mechanical properties of the most used fibers.

Table 1 Properties of different fibers [12].

Fibers	σ [GPa]	E_L [GPa]	Elongation [%]	ρ [g.cm ⁻³]
E-glass	3.4	72	4.9	2.62
S-glass	4.5	85	5.7	2.50
Carbon	2.4 - 3.3	230-390	0.6-1.4	1.70-1.90
Aramid	3.6 - 4.1	62-130	2.80-4.0	1.44
UHMWPE	2.6	117	3.5	0.97

3.1.2.2. Orientation of Fibers

Most traditional dental materials are isotropic, showing similar physical and mechanical behavior in all directions. Dentine and enamel are, however, naturally anisotropic materials.^{9,10} Similarly, FRCs have properties that change from isotropic to anisotropic depending on the fiber orientation in the matrix. Some reports in the dental literature are available concerning anisotropic behavior of FRCs regarding flexural strength, modulus of elasticity, and thermal expansion [1].

Fibers consist in thousands of filaments, each filament having a diameter of between 5 and 15 μm . In FRC, the role of the components can vary depending on the properties of the individual materials and their architecture. The orientation of the fibers has a big importance since it has a direct influence on the mechanical properties of the composite. Composites with **unidirectional oriented** fibers behave as anisotropic materials, therefore unidirectional FRCs are used when there is only one direction of the highest stress and this direction is known, hence this materials are used in to make periodontal splints or pontics of fixed or removable partial dentures.

The yarns can be woven in such way that fibers are oriented in two or three directions and they give so-called quasi-orthotropic mechanical properties to the FRC and reinforce FRC structure to some extent in all directions. Thence weaves with **multidirectional orientation** of fibers, are useful when it is difficult to predict the direction of highest stress in the prosthesis.. The weaves may have various textile structures, such as linen, twill, or satin weave. Multidirectional oriented composites are used in the fabrication of crowns and some types of removable dentures, such as over dentures, and surface-retained periodontal splints.

In recent years, fiber reinforcements in the form of ribbons have been introduced to address these deficiencies. By etching and bonding to tooth structure with composite resins embedded with woven fibers adapter to the contours of teeth periodontal splints, endodontic posts, anterior and posterior fixed partial dentures, orthodontic retainers and reinforcement of single tooth restorations can be accomplished [25].

Fiber reinforcement with continuous fibers in **random orientation** is called a fiber mat whereas reinforcement with randomly oriented short fibers is called a chopped fiber mat.

These give isotropic mechanical properties for the fiber reinforced composites in plane. Fiber mats are not commonly used in dental FRC constructions at the moment. [19]

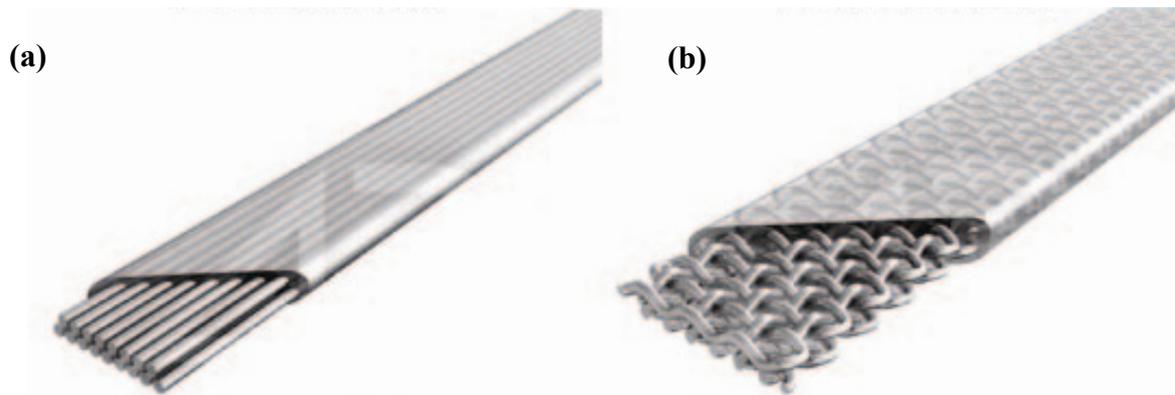


Fig. 5 FRC with (a) unidirectional and (b) multidirectional oriented fibers [26].

3.1.3. Filler Particles

Resin-based composites are made of an organic matrix which is formed by the polymerization of free radicals of dimethacrylates. These are non-toxic and capable of rapid in situ polymerization in the tooth cavity in the presence of oxygen and water. When this matrix without filler is used to restore teeth, it shows poor wear resistance, which can be improved by the inclusion of inorganic particulate fillers. Inorganic filler particles are also very important for improving the clinical performance of restorations, as they enhance resin properties and characteristics, such as polymerization shrinkage, surface staining, resistance, conversion degree, elastic modulus, and others [27].

PFCs are now being used instead of amalgam. Due to the characteristic polymerization shrinkage of resin-based composites, clinical success with composite restorative materials is fundamentally dependent on effective and durable adhesion to enamel and dentin [28][29].

The most common reinforcing materials are quartz, borosilicate glasses, zinc glasses, colloidal silica, and glass ceramics. The glasses and ceramics may be lithium aluminum, barium aluminum, or strontium aluminum silicates. Quartz is the hardest of the group, provides good resistance to wear, and has a refractive index in the correct range to provide good esthetics. The barium or other heavy metal glasses provide radiopacity and may be mixed with other reinforcing fillers for X-ray opacity of the fillings. Experimental composites consisting of zirconium and/or tin alumino-silicates have been shown to have adequate opacity on radiography.

The inorganic phase is treated with an organo-functional silane before being mixed with the unreacted low molecular weight monomer mixture. The organo-functional silane contains functional groups, such as methoxy, which hydrolyze and react with the inorganic filler as well as unsaturated organic groups that react with the monomer during polymerization. These organosilanes are called coupling agents because they form a bond between the inorganic and organic phases of the composite [19]. Besides silicate glass particles, chopped glass fibers and porous networks of fibers have also been incorporated as fillers into dental composites. These improvements have not been sufficient to merit recommendation that direct-filling resin composites be used for large stress-bearing posterior restorations, especially those that involve cusp replacements where large stresses can be generated during mastication.

Recently, ceramic single crystal whiskers were described as fillers to reinforce dental resin composites. Silica particles were fused onto the individual whiskers to facilitate whisker silanization, to minimize whisker entanglement by facilitating their dispersion, and to enhance whisker retention in the matrix by providing roughness on the whisker surfaces. The ceramic whiskers are single crystals possessing a high degree of structural perfection and, hence, superior strength and toughness values [30].

3.1.4. Main Clinical Applications

Composite materials offer unsurpassed esthetics, superior clinical reliability, minimal or non-invasive solutions, simple and fast procedure. Therefore it is generally accepted that the unique properties of particulate filled composites (PFC) and fiber reinforced composites (FRC) are beneficial in a wide range of prosthetic, periodontic, endodontic, orthodontic, traumatology, and pediatric dentistry [2][26].

In particular, FRC materials are being increasingly used as load bearing substructures in various passive dental appliances, such as frameworks for fixed partial dentures, orthodontic retainers periodontal and traumatic splints and as posts in endodontic treatments. PFC are commonly used as dental fillings, as veneer composites used to sculpture artificial teeth and crowns in fixed partial dentures. In most of the dental appliances, FRC can replace metal alloys as the load bearing component and can be beneficially combined with PFC in order to achieve excellent esthetics and biomechanics similar to the natural dental tissue.

The FRC/PFC combines the strength and rigidity of the FRC with esthetics of the PFC to provide an alternative to porcelain-fused-to-metal restorations, crowns, posts, splints and dentures. In addition to meeting the performance criteria, the FRC/PFC appliances possess an advantage over the metal–ceramic ones by not excessively wearing the opposing teeth or previous restorations [1].

With increasing aesthetic demands and fear of mercury toxicity associated with amalgam, the use of dental composites has increased exponentially in restorative dentistry during the last decade. In the formulation and development of dental composite restoratives, it is of paramount importance to understand the elastic properties of the material in order to achieve the best clinical results. Ideally, the elastic properties of the restorative materials must be closely matched to that of enamel and/or dentin. This would then allow a more uniform masticatory stress distribution across the restorations-enamel/dentin interface. An imperfect match of the elastic values between the materials and the surrounding hard tissues will lead to marginal adaptation and fracture problems [31].

The development of the mechanical properties of fiber-reinforced composites suggests that FRCs can potentially be used in structure of dental appliances such as crowns and bridges. The use of novel FRC technology has allowed more tooth-saving prosthodontic treatment with fixed partial dentures (FPDs). In FPDs, the high-strength FRC framework is veneered with particulate filler composite, and the strength of the appliance is also dependent on the adhesion between the veneering composite and the FRC framework. When composite resin luting cements with different adhesives are adhered to FRC frameworks, the adequate adhesion of the cement to the FRC framework is also essential [32].

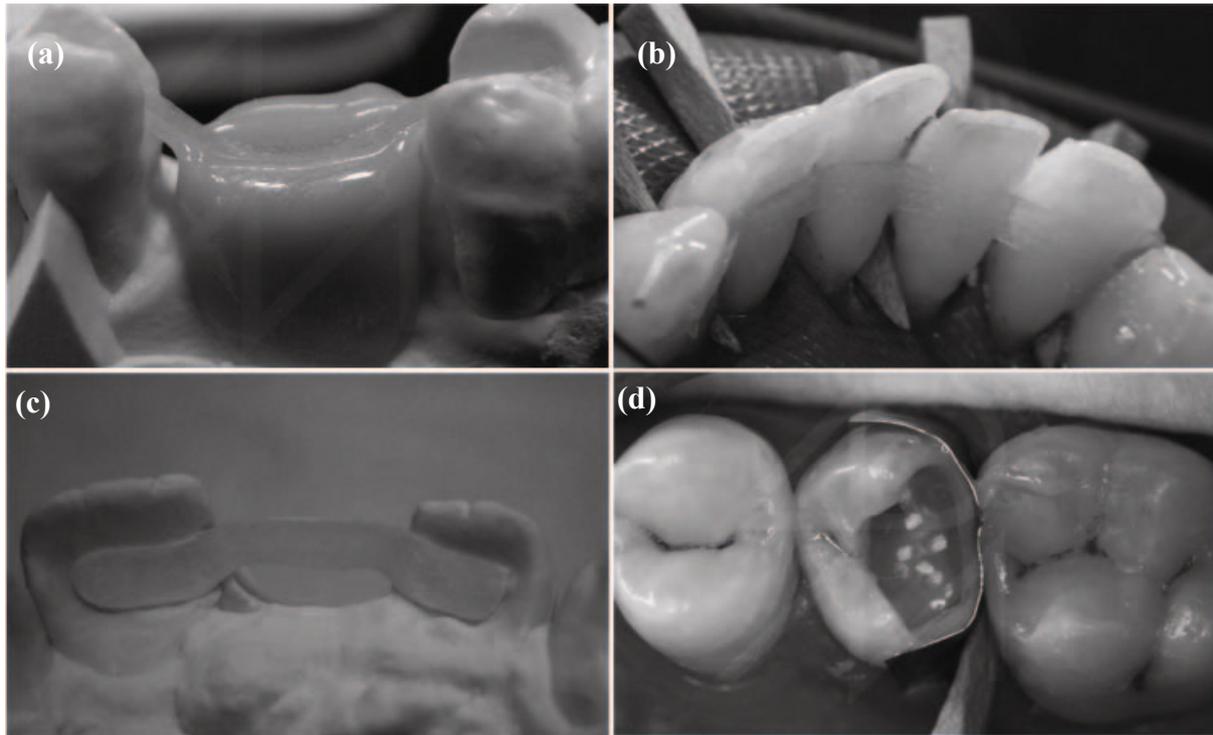


Fig. 6 Composite materials in clinical applications. (a) Inlay bridge, (b) oral splint (c) FRC Framework used for the fabrication of anterior bridges Maryland, (d) Pin posts [26].

3.2. Adhesion

Adhesion is defined as the molecular attraction exerted between the surfaces of bodies in contact, or the attraction between molecules at an interface. This definition, proposed by the American Society for Testing and Materials, appears to be accepted by the majority of materials scientists, although some would include mechanical interlocking as well as interfacial bonding in their understanding of adhesion [33].

The actual mechanism of adhesive attachment is not explicitly defined. Several theories attempt to describe adhesion. No single theory explains adhesion in a general, comprehensive way. Some theories are more applicable for certain substrates and applications; other theories are more appropriate for different circumstances. Each theory has been subjected to much study, question, and controversy. However, each contains certain concepts and information that are useful in understanding the basic requirements for a good bond.

The adhesion force between the solid polymers and other substances, along with other parameters, such as the contact surface and the diffusion distance, are strongly influenced by the type and magnitude of the intermolecular forces manifested between adhesion parameters. The adhesion forces may be characterised by the application of three main types of methods, as follows: direct measurement of the force, spectral determination of the chemical composition of the solid surfaces and physico-chemical determination of the energetic interactions. The theories discussing the increase of the composite's mechanical properties are based on the hypothetical existence of a perfect adhesion between phases.

The most common theories of adhesion are based on adsorption, simple mechanical interlocking, diffusion, electrostatic interaction, and weak-boundary layers [34].

3.2.1. Theory of Diffusion

Initially, the theory known as ‘self-adhesion’, has been proposed by Voyutski, with a view to explaining the adhesion between two identical polymers. When two polymeric materials are in intimate contact and are thermodynamically compatible, the molecular motion (at not too low temperatures) makes possible interdiffusion of the two species. A mutual interdiffusion zone, or an interphase, which is the only one responsible for adhesion, thus results. It has been determined that adhesion of polyisobutylene with another polymer results from an interdiffusion that depends on the contact time, temperature and pressure, the development of such an interdiffusion [7].

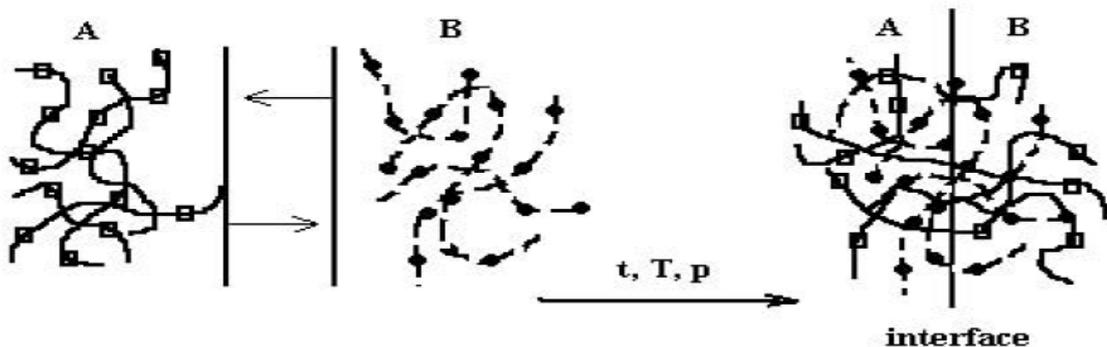


Fig. 7 Process of adhesion by diffusion [7]

3.2.2. Theory of Mechanical Adhesion

The surface of a solid material is never truly smooth but consists of a maze of peaks and valleys. Mechanical adhesion is adhesion between surfaces in which the adhesive by a penetration into pores in the adherend (substrate) holds the parts together by the action of mechanical interlocking. Although at one time, adhesion was believed to involve mainly a mechanical attachment, nowadays this theory is certainly regarded as of secondary importance in general adhesive technology when compared with the specific adhesion due to physical and chemical phenomena, even when bonding porous surfaces. It is probable that in practice both mechanisms contribute to the strength of a bonded joint, but one or the other predominates [15][33].

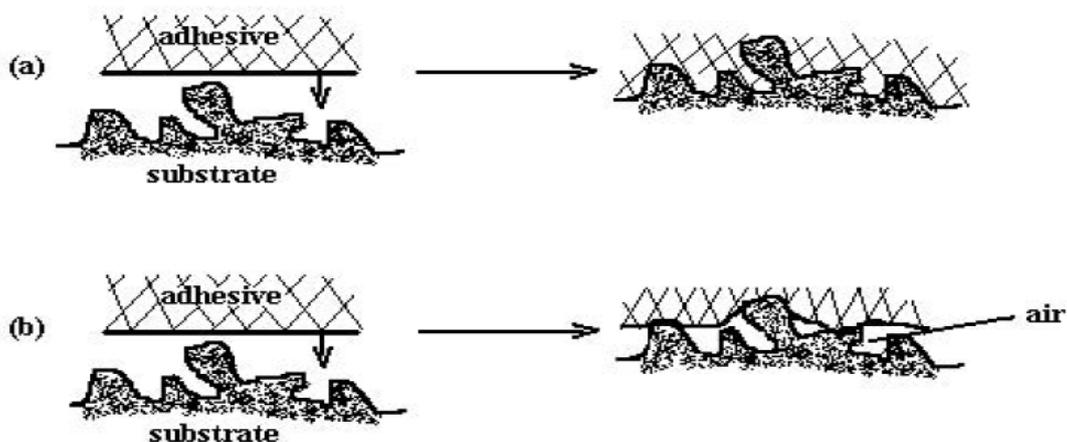


Fig. 8 Model of mechanical adhesion (a) good wetting of surface and (b) moderate wetting of surface [7]

3.2.3. Theory of Adsorption

The adsorption theory states that adhesion results from molecular contact between two materials and the surface forces that develop. Adhesion results from the adsorption of adhesive molecules onto the substrate and the resulting attractive forces, usually designated as secondary or van der Waals forces. For these forces to develop, the respective surfaces must not be separated more than five angstroms in distance. Therefore, the adhesive must make intimate, molecular contact with the substrate surface [34]. It is for this reason that all normal adhesives are, at one stage in their use, mobile liquids which can wet and flow across the adherend, penetrating all the irregularities and roughness of the surface so that the very closest and most intimate contact is achieved [35].

It is generally considered unnecessary to develop hydrogen or primary chemical bonds for high joint strength, van der Waal's physical forces being more than sufficient to form joints which may in effect be stronger than the cohesive strength of the adhesive or adherend. However in certain circumstances, such as when technical requirements are severe, especially with regard to temperature and humidity, chemical bonding at the interface becomes important. When chemical bonding does occur, it will certainly provide greater resistance to heat, water and chemicals within the bonded assembly while also providing increased bonding, which may not be required in the presence of adequate secondary forces of attraction [33].

The forces involved in holding adhesives to their substrates or in holding adhesives and together as a bulk material arise from the same origins. These same forces are all around us in nature. Although there are many kinds of forces, it is mainly those of a physical and chemical nature that are important in understanding the development of adhesive joints [34].

3.2.3.1. Work of Adhesion and Cohesion

Bond strength is not only the result of adhesion forces. Other forces contribute to the strength of a joint. For example, molasses may have good adhesion, but it is a poor adhesive. Its failure is usually cohesive. Cohesive strength of an adhesive or sealant is at least as important as its adhesive strength. Like a weak link in a chain, the bond will fail at the place where the intermolecular forces are the weakest. Adhesive forces hold two materials together at their surfaces. Cohesive forces hold adjacent molecules of a single material together. Adhesive joints may fail either adhesively or cohesively.

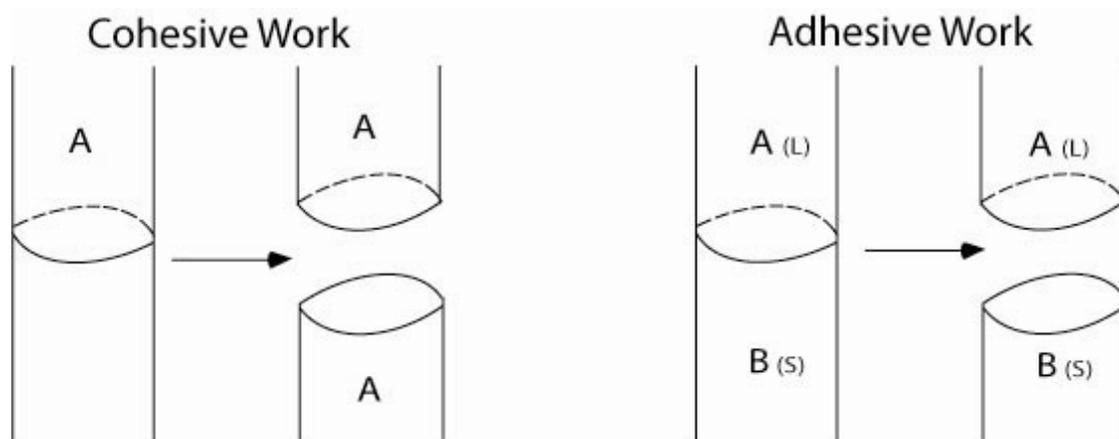


Fig. 9 Work of adhesion and cohesion

Adhesive failure is failure at the interface between adherend and the adhesive. An example would be the peeling of cellophane tape from a glass surface if the adhesive film separates cleanly from the glass. Cohesive failure is failure within the adhesive or one of the adherends [35].

The cohesive strength of any solid depends upon the various forces of attraction that exist between the ultimate atoms or molecules. These forces are of various types depending upon the particular nature of the solid but, in general, will include those regarded as of chemical origin (covalent, ionic and metallic bonding), as well as those considered as of physical origin (hydrogen bonding or van der Waal's forces including dipole interactions and the dispersion forces). In any particular instance, which of these are present and their relative significance will depend upon the chemical nature of the material, but the dispersion forces will always be present and effective to a greater or less extent [34][36].

A quantitative consideration of these forces will enable an ideal strength of the material to be calculated. However, this strength is never normally achieved. This is because of irregularities, flaws and defects both throughout the structure and more particularly in the surface [35].

If a bulk material is subjected to a sufficient tensile force, the material will break thereby creating two new surfaces. If the material is completely brittle, the work done on the sample is dissipated only in creating the new surface. Under those assumptions, if the failure is truly cohesive where both sides of the broken material are of the same composition, then

$$W_C = 2\gamma \quad (1)$$

where W_C is defined as the work of cohesion and γ surface free energy.

Now similarly consider separating an adhesive from a substrate. The energy expended should be the sum of the two surface energies. However, because the two materials were in contact, there were intermolecular forces present before the materials were split apart. This interfacial energy can be represented as γ_{SL} . The work of adhesion (W_A), may be defined by the surface energies of the adhesive and the adherend

$$W_A = \gamma_{LV} + \gamma_{SV} - \gamma_{SL} \quad (2)$$

where, the subscripts S , L and V refer to solid, liquid and vapor, respectively. This is the classical Dupre equation, which was developed in 1869.

3.2.3.2. *Wetting*

The first step in the formation of an adhesive bond is the establishment of interfacial molecular contact by wetting. The theoretical consideration of the situations involved in adhesion begins from the expression for the thermodynamic work of adhesion, described by the Dupre equation (2).

The Young equation (3), describe the relationship between surface tensions and contact angle of a solid surface by a liquid during wetting (*Fig. 10*).

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cos\theta \quad (3)$$

Wetting can affect adhesion in two ways. First, incomplete wetting will produce interfacial defects and thereby lower the adhesive bond strength. Second, better wetting can

increase the adhesive bond strength by increasing the work of adhesion, which is directly proportional to the fracture energy.

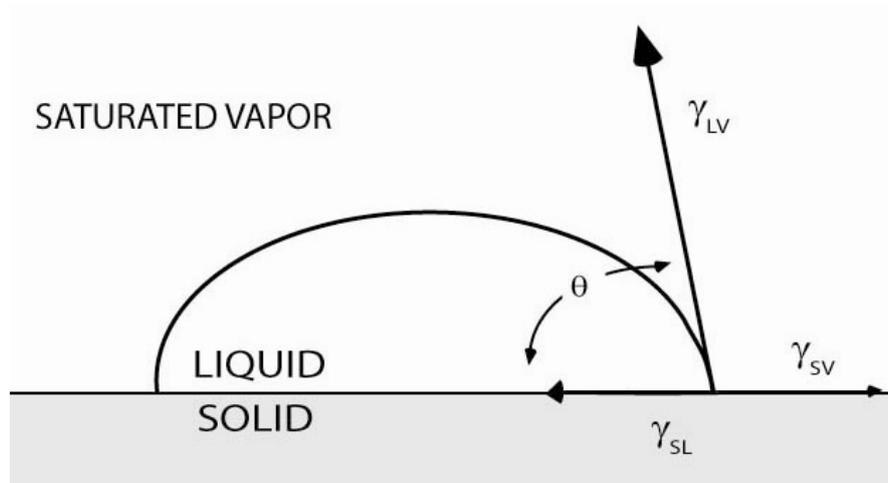


Fig. 10 Contact angle θ and surface energies γ for a liquid drop on a solid surface.

3.3. Surface and Interface Interactions

When two materials are bonded, the resultant composite has at least five elements: adherend – interface – adhesive – interface – adherend.

The force acting on a polymeric composite's matrix is transmitted to the filler or reinforcing material through the interface. Its resistance and durability are determined by several factors which govern the matrix's adhesion to the surface of the included material. With a view to increasing the interfacial resistance between the included material and the matrix, the surface of the filling or reinforcing material is usually treated by special techniques, so that to create the conditions in which the interface should assure shifting (without any discontinuities) from the properties of the matrix to those of the inclusion material [36].

The strength of the joint will be determined by the strength of the weakest member. Often the weakest member is one of the interface regions, since this is generally where weak boundary layers can occur. Examination of weak boundary layers and investigation of phenomena occurring at the interface regions of the joint provides valuable information.

3.3.1. Boundary Layer Theory

One interpretation of adhesion that has been the most useful in describing adhesion phenomenon is the boundary layer theory set forth by Bikerman. This theory proposes the existence at an interface of a finite boundary layer composed of adsorbed molecules that differ in nature from those in the bulk phases. The criterion for good adhesion is merely that the boundary layer be strong enough to withstand the effects of external stress. According to Bikerman, rupture of an adhesive joint practically always proceeds through a single material phase rather than between two materials. The rupture is initiated at a point where the local stress exceeds the local strength. When failure occurs in the boundary stratum, a teak boundary layer is present [34].

Weak boundary layers may form due to a variety of causes. Often the formation is unpredictable, and it is difficult or impossible to determine the actual composition of the boundary layer. Examples of common weak boundary layers include:

- Entrapment of air
- Impurities, or low molecular weight species that concentrate near the surface
- Cohesively weak oxide layers on the substrate surface
- Chemical deterioration of the coating, adhesive polymer, or substrate during the bonding process (e.g., catalytic air oxidation during heating)
- Chemical deterioration and/or corrosion between the adhesive and the substrate. (In some cases, for example with metals, the formation of brittle inter-metallic compounds can lead to a weak boundary layer.)

3.3.2. Interphase region

Sharpe extended Bikerman's two-dimensional weak boundary layer concept into a three-dimensional interphase concept. The interphase is an intermediate region between the fiber and the matrix and it comprises the interlayer and a part of the matrix affected by the presence of the coated fiber. Interphases are thin regions of the joint that have properties that are widely different from the bulk materials. These regions are thick enough to have properties, such as modulus, Poisson's ratio, tensile strength, etc., that will affect the final properties of the joint [6][35].

Interphase regions can be formed by solidification of certain polymers on certain high energy substrates. The interfacial structure will be characteristic of the composition and structure the polymer and the substrate, as well as the ambient conditions. Solidification preserves this organization to create an interphase with unique properties that becomes a permanent part of the joint, influencing its mechanical response. Examples include semi-crystalline polymers, such as polyethylene. When these polymers are solidified from the melt while in contact with a solid substrate, a visibly different structure in the polymer near the interface, the so called trans-crystalline structure is formed. This region has mechanical properties different from the ordinary bulk structure of the polyethylene from which it came [34].

It is clear that interphases, which are quite thin relative to the joints in which they are present, will not have much effect on small deformation properties of the joint. However, they can have remarkable effects on the ultimate properties such as the breaking stress of the joint. This is particularly important if the interphase regions are sensitive to various environments, such as temperate and moisture. It is also highly probable that interphases are not homogeneous in the sense that their composition, structure and, therefore, properties vary across their depth. Research needs to be directed at answering questions such as how to model the joint to include interphase regions, what are the appropriate properties of the interphase to measure, and how do you measure these properties [34].

In fiber reinforced composites (FRCs), exhibiting heterogeneous structure at multiple length scales, the interphase phenomena at various length scales were shown to be of pivotal

importance for the control of the performance and reliability of such structures. Various models based on continuum mechanics were used to describe effects of the macro- and meso-scale interphase on the mechanical response of laminates and large FRC parts, satisfactorily. At the micro-scale, the interphase is considered a 3D continuum with ascribed average properties. Number of continuum mechanics models was derived over the last 50 years to describe the stress transfer between matrix and individual fiber with relatively good success. In these models, the interphase was characterized by some average shear strength, and elastic modulus. On the other hand, models for transforming the properties of the micro-scale interphase around individual fiber into the mechanical response of macroscopic multi-fiber composite have not been generally successful.

The anisotropy of these composite structures is the main reason causing the failure of these models. The strong thickness dependence of the elastic modulus of the micro-scale interphase suggested the presence of its underlying sub-structure. On the nano-scale, the discrete molecular structure of the polymer has to be considered. The term interphase, originally introduced for continuum matter, has to be re-defined to include the discrete nature of the matter at this length scale [36].

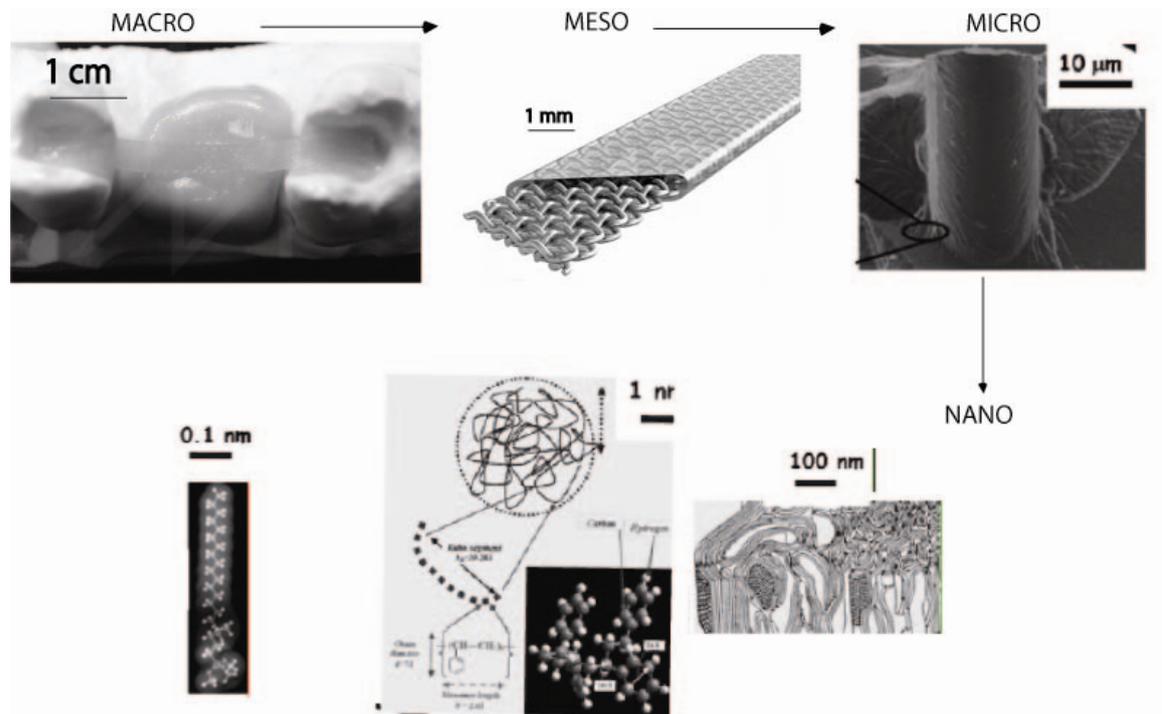


Fig. 11 Micro, meso and nano scales of FRC

3.3.3. Influence of the Interface on the Mechanical Properties of Composites

The interface between the filler or reinforcing material and the polymeric matrix is essential in polymeric composites [7]. A strong interfacial bonding increases the strength of composite material at the expense of the material toughness. A functional interlayer deposited between two phases can eliminate this conflict of material parameters. A proper thin film with adhesive bonding to both the phases results in an improvement of the material strength and toughness simultaneously [6][7].

The interface adjusts the composite's mechanical resistance: here, concentration of mechanical stress occurs. The mechanical stresses acting upon a polymer composite are

transmitted from the matrix towards the filling or reinforcing material by means of the interface. The assertion may be made that the interface regulates the mechanical properties of polymeric composites.

Thorough studies have been developed aimed at correlating the mechanical properties with the resistance of the bonds formed at the filling or reinforcing material/ polymeric matrix interface. The mechanical properties were found as maximum when, at the interface, the strength of the links among phases had been optimized. However, in the absence of a quantitative approach, the nature of interfacial links cannot be wholly understood; with a view to quantifying bonds' resistance at the interface, it was assumed that the mechanical properties of some polymer composites as filling material can be correlated with the adhesion force, which represents the stresses necessary for separation of components and breaking of such links [7].

3.4. Fracture of Adhesive Bond

The mechanical strength of the system is determined not only by the interfacial forces, but also by the mechanical properties of the interfacial zone and the two bulk phases. When an adhesively bonded structure breaks under low applied stress, the structure is often said to have „poor adhesion“. Such usage can be misleading, since the fracture may have occurred cohesively near the interface rather than along the interface [19].

At present a gap exists between the strength of adhesion which is theoretically possible and that which is obtainable in practice. The maximum possible adhesion ideally derived from all physical and chemical attractive forces requires the condition of molecular nearness. This state, however, can never be attained in practice. The inherent strength of the bond is determined by the ability of the adhesive and adherend to attain molecular nearness. Loss of adhesion due to incomplete wetting is shown as the difference between the maximum adhesion and the inherent bond strength. If the adhesive is unable to deform to accommodate stresses induced during its solidification, further loss of adhesion occurs. The residual bond strength is the difference between the inherent bond strength and the internal bond stresses. Finally, contamination of the surfaces further reduces the measured bond strength, with experimental errors representing the loss in adhesion [33].

3.4.1. Griffith Fracture Criterion

In any solid material body there are present flaws and defects. In smaller bodies, however, the probability of a presence of a large defect is much smaller than in a large solid. From analysis of experimental measurements, Griffith developed a relationship between flaw size a and the stress at failure σ , by noting that when a flaw grew in a stress body, there was a decrease in the potential energy of the body, so energy was released to be able form the new surface of the growing flaw. The energy necessary to do this was taken as the true surface energy of the solid:

$$\sigma_c = \sqrt{\frac{2\gamma E}{\pi a_c}} \quad (4)$$

for a stress plane condition, or in the case of plane stress,

$$\sigma_c = \sqrt{\frac{2\gamma E}{\pi a_c(1-\nu)}} \quad (5)$$

where E is the elastic modulus, γ surface energy and ν Poisson's ratio.

For most materials stress concentration induced at the crack tip causes large localized plastic deformation and flow, which results in more energy being dissipated prior to fracture.

$$G_c = 2\left(\gamma + \frac{G_p}{2}\right) \quad (6)$$

where G_c is the energy released rate and G_p is the energy used in plastic deformation.

Experimentally observed critical strain energy release rate, was for these materials two to three orders of magnitude bigger than the theoretical value of proposed by Griffith. It was concluded that the surface energy proposed by Griffith is to be the controlling factor in fracture is too small even for macroscopically very brittle materials. Therefore a modification of the Griffith criterion (7) for fracture, where G_c is replacing 2γ [37],

$$\sigma_c = \sqrt{\frac{2\left(\gamma + \frac{G_p}{2}\right)E}{\pi a_c}} \quad (7)$$

3.4.2. Stress at a Crack Tip

The analysis of local stress fields around crack is a reasonable approach to investigate localized phenomenon such as fracture. A crack in the solid body can be loaded principally in three basic modes (Fig. 12). Loading in the direction perpendicular to crack plane (normal stress) gives rise to an *opening mode* denoted as **Mode I**, which is the most important for the analysis of impact tests. **Mode II** or *sliding mode* denotes pure shear loading in the plane of crack. **Mode III** also called tearing mode involves both modes in a combination.

The stress conditions in front of a flaw tip are inhomogeneous and they are unlimited values. An element A of the plate at a distance r from the flaw tip and an angle φ with respect to the flaw plane, experiences normal stress σ_x and σ_y in X and Y directions respectively and shear stress τ_{12} [37].

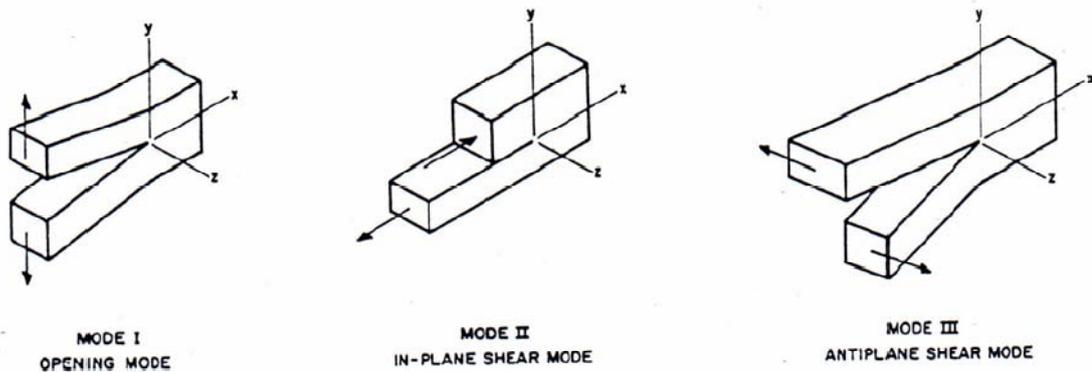


Fig. 12 Basics loading modes

$$\sigma_x = \frac{K}{\sqrt{2\pi r}} \cos \frac{\varphi}{2} \left(1 - \sin \frac{\varphi}{2} \sin \frac{3\varphi}{2}\right), \quad (8)$$

$$\sigma_y = \frac{K}{\sqrt{2\pi r}} \cos \frac{\varphi}{2} \left(1 + \sin \frac{\varphi}{2} \sin \frac{3\varphi}{2} \right), \quad (9)$$

$$\tau_x = \frac{K}{\sqrt{2\pi r}} \sin \frac{\varphi}{2} \cos \frac{\varphi}{2} \cos \frac{3\varphi}{2} \quad (10)$$

where K is the **stress intensity factor** and it determines the stress field around a crack tip and is defined as:

$$K = \sigma_0 \sqrt{\pi a} \pi \quad (11)$$

The stress intensity factor, was defined for an ideally elastic body, however its use can be extended into problems involving small scale yielding and to some extent to the contained yielding type of fracture. For calculation of K a factor of proportionality (geometrical factor) has to be introduced depending on the shape of the cracks [38].

3.4.3. Locus of Failure

The interface may be sharp or diffuse. When interfacial diffusion is sufficient, the interface is diffuse, such as between thermoplastic polymers of similar polarities. In this case, clean interfacial separation is obviously impossible (except perhaps at zero rate), as molecular segments are entwined. On the other hand, when little or no interfacial diffusion occurs, the interface will be sharp, such as between polar and non-polar polymers, between a thermoplastic polymer and a cross-linked polymer, or between a polymer and an inorganic material. In this case, true interfacial separation may occur.

Interfacial separation will occur when the interfacial strength is weaker than the bulk strength. In terms of the Griffith criterion, interfacial failure will occur when

$$\sqrt{\frac{E_{12} G_A}{a_{12}}} < \sqrt{\frac{E_2 G_A}{a_{12}}} \quad (12)$$

where phase 1 is stronger than phase 2.

In the absence of interfacial chemical bonding,

$$E_{12} \cong \sqrt{E_1 E_2} \quad (13)$$

and

$$G_A \cong \sqrt{G_1 G_2} \quad (14)$$

therefore,

$$\sqrt{\frac{a_2}{a_{12}}} < \sqrt{\frac{E_2 G_A}{E_1 G_1}} \quad (15)$$

is the condition for interfacial separation. There are several possibilities for crack propagation during failure an adhesive joint:

- cohesive failure in the bulk of phase 1,
- cohesive failure in the bulk of phase 2,
- cohesive failure in a thin layer in phase 1 very near the interface,
- cohesive failure in a thin layer in phase 2 very near the interface, and
- true interfacial failure [19].

4. EXPERIMENTAL PART

In this investigation a total of 84 samples, were created and measured. The samples were divided into fourteen different groups (*Fig. 13*). The four groups were made of samples without any interlayer. The other ten groups were divided according the composition of the interlayer (thickness and resins used). PFC of the specimens differed in the filler content (0, 10, 40 wt. %). The FRC rod was identical for all samples.

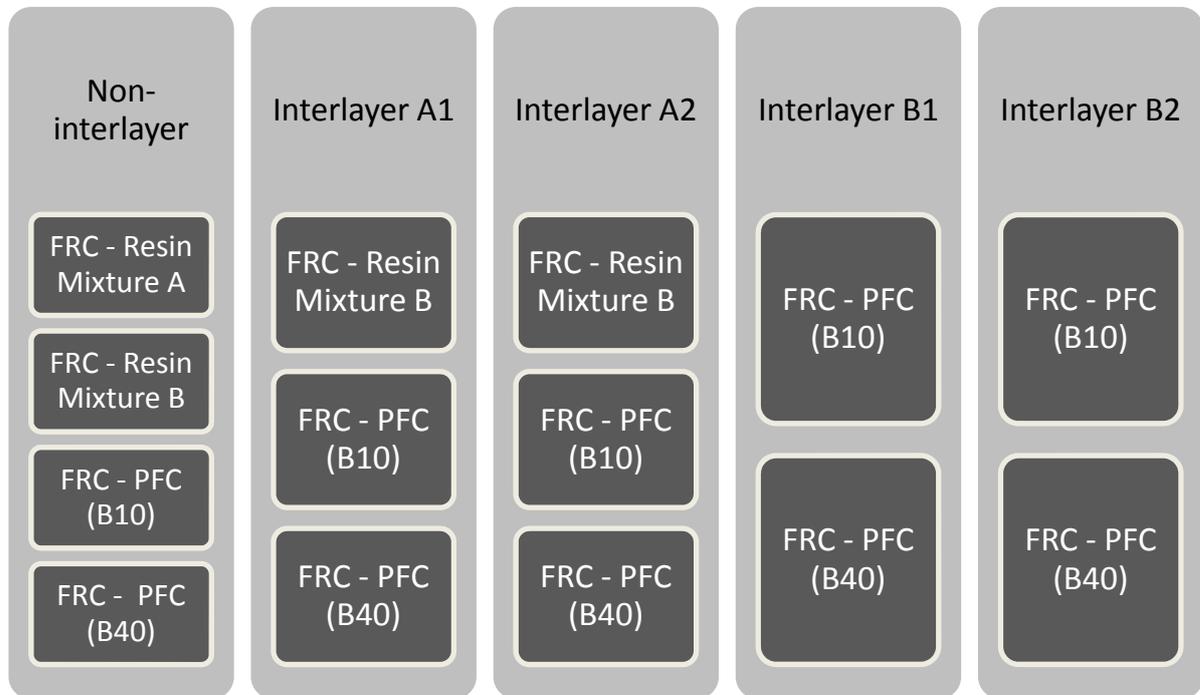


Fig. 13 Structure of the investigation.

4.1. Materials and Devices

For this investigation diverse materials were used. Those are described below.

4.1.1. Resins and Photo-initiation Complex

Commercial dimethacrylate resin provided by Advance Dental Materials - ADM a.s., was used as the matrix phase of FRC rods.

For the resins mixtures Bis-GMA (Sigma-Aldrich, Germany) was used as monomer. PEGDMA (Sigma-Aldrich, Germany) and TEGDMA (Sigma-Aldrich, Germany) were used as viscosity modifiers for the resin mixtures used for different interlayers and PFC preparation.

The photo-initiation complex for the resin's polymerization, was based on Camphorquinone (Sigma Aldrich, Germany) and N,Ndimethylaminoethylmethacrylate (Sigma-Aldrich, Germany).

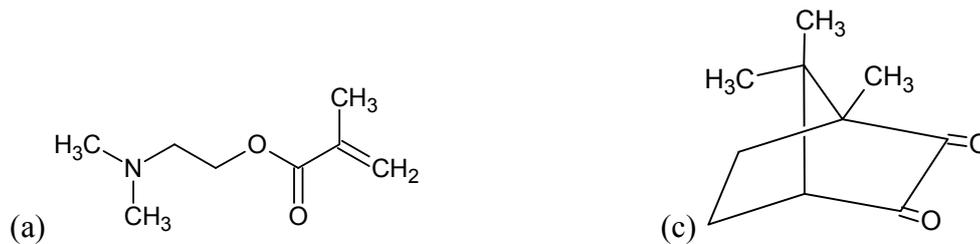


Fig. 14 (a) *N,N*-dimethylaminoethylmethacrylate and (b) camphorquinone.

4.1.2. Reinforcements

To create FRC rods, three roving of *S-2 Glass® Yarns* with continuous filaments yarns from the company AGY (Belgium), were used. As filler for PFC rings, untreated *barium glass powder* (Schott GM 27884, Germany) was used.

4.1.3. Devices

The following devices were used during the investigation

Dentapreg™ UV lamp (ADM s.r.o, Czech Republic).

Targis Power light curing chamber (Ivoclar, Liechtenstein)

MICROMOT 50/E Cutting Tool (Proxxon, Germany)

Universal Testing Machine Zwick Z010 (Zwick, Germany)

Scanning electron microscope (SEM) Tesla BS 340 (Tesla, Czech Republic)

4.2. Resin Mixture Preparation

Two resin mixtures were used for the preparation of composite veneers and interlayers. The mixtures were based on bis-GMA and the viscosity modifier TEGDMA and PEGDMA.

The procedure used for the preparation of resin mixture was the follow:

1. Bis-GMA and viscosity modifiers, were heated up to 75°C, to reduce the viscosity and make easier the manipulation.
2. Viscosity modifiers, TEGDMA for *resin mixture A* and PEGDMA for the *resin mixture B* were weighed and added into a beaker wrapped in aluminum foil.
3. The monomer bis-GMA was added to create a mixture in the rate of 50 wt. % and the mixture was mixed during 15 minutes at 65°C.
4. The photo-initiation complex was added. Camphorquinone (0.3 wt. %) and *N,N*-dimethylaminoethylmethacrylate (0.2 wt. %) and mixed for 15 minutes more.
5. The resins mixtures were stored in a beaker wrapped by aluminum foil in a dark place to prevent light interaction and premature polymerization of the resins.

4.3. PFC Preparation

For the preparation of the particle filled composites, the mixture resins based on the mixture of Bis-GMA and PEGDMA mentioned above was used.

Barium glass powder was added to the resin mixture. The powder was progressively added to create PFC with different filler content, 10 wt. % and 40 wt % respectively. The resin mixture and the barium glass powder were mixed in the dark, to prevent premature polymerization of the resin mixture, for approximately 40 minutes.

4.4. FRC Preparation

FRC specimens were prepared using a pultrusion mechanism, in the form of round cross section rod containing continuous S-2 glass fibers with unidirectional orientation. Glass content in the matrix was calculated as 35 wt. % (*Fig. 15*).

The samples were prepared as follow:

1. Three continuous roving of S2-Glass fibers on the top of the device, were pulled with a loading weight of 1.1 Kg.
2. Fibers went through two commercial dimethacrylate resins baths for a better impregnation, where the fibers were shaped, impregnated and light cured by UV lamp.
3. The rods of 90 cm were cut into 30 cm and polymerized by the UV light of 460 nm, in the light curing chamber for 10 minutes.
4. The sample was cleaned by dropping acetone and then cut to the final length of 50 mm, using the cutting tool.

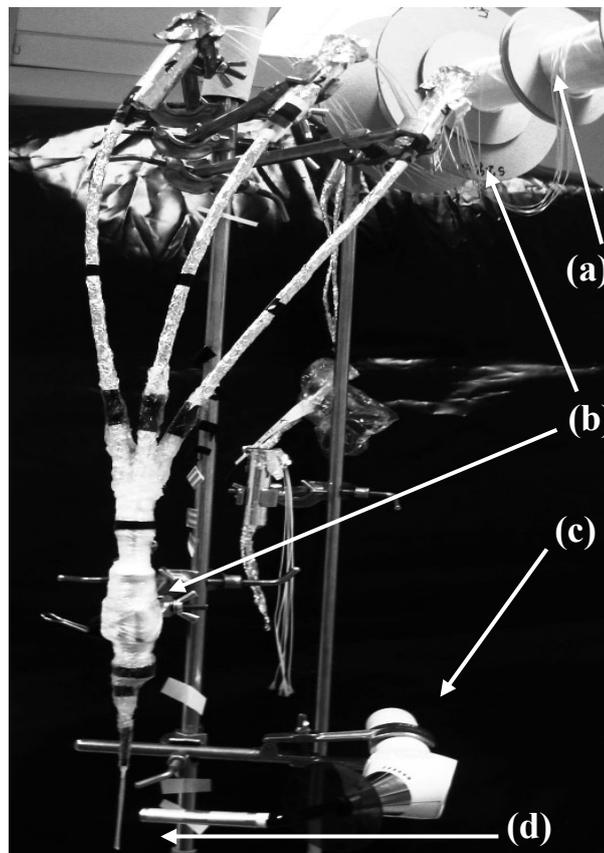


Fig. 15 Pultrusion device for FRC rod preparation. (a) continuous roving of fibers, (b) resin bath – upper pre impregnation guidance, lower post impregnation guidance, (c) curing mechanism (d) pulling mechanism.

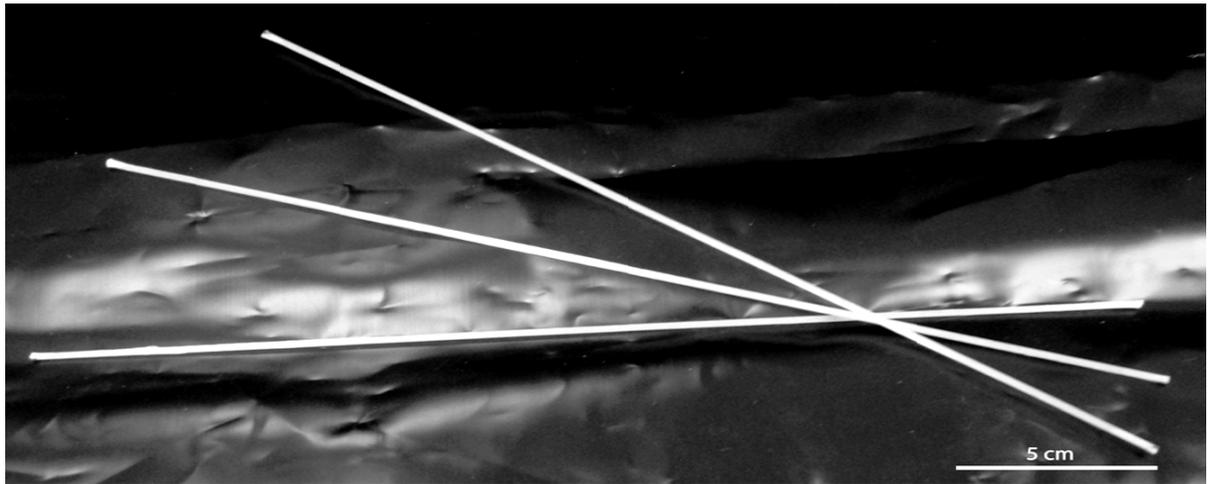


Fig. 16 FRC rods (25cm)

4.5. Interlayer Preparation

For the preparation of samples with interlayer, FRCs posts were introduced into a recipient containing resin mixture (A or B). The sample was held with the help of a click and the FRC posts were left to flow for one minute. After this the curing started for 30 seconds with the help of UV lamp. In this way a thin layer of about 0.03 mm, covered the FRC post.

For making the thickness of the layer bigger, the FRC posts were introduced twice into the resin recipient in that way we achieved a layer of a thickness about 0.05 mm, and then after polymerized in the same way as mentioned above (*Fig. 17*). After this FRC with interlayers were introduced in the curing chamber for 2 minutes.

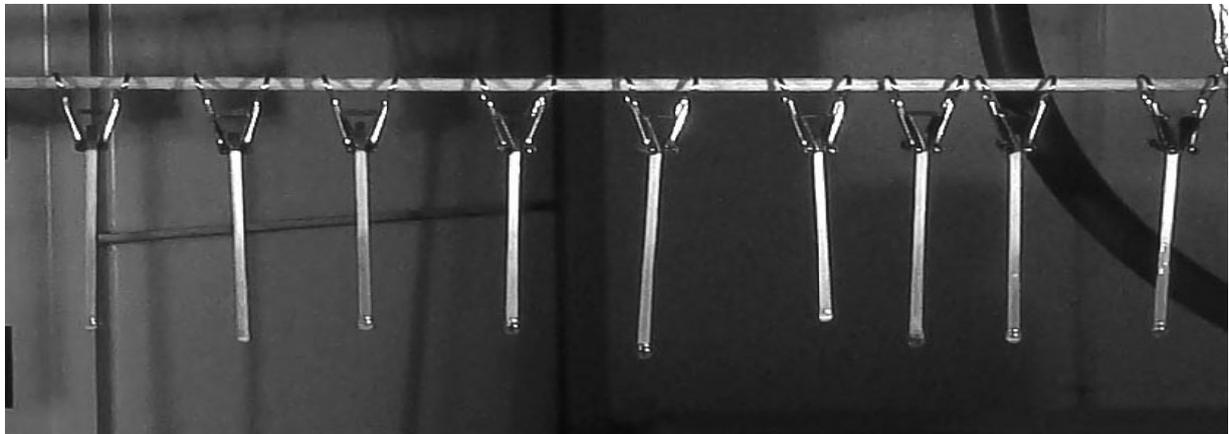


Fig. 17 FRC rods during the polymerization by UV light lamp after submersion in resin mixture.

4.6. Sample Preparation

The test specimen was fabricated by placing the FRC rod (with any or different interlayers) into a transparent *Lukopren* rubber mold. The mold was closed together and unpolymerized PFC was added in order to create a ring that surrounds the FRC rod. A small piece of *lukopren* was putted in the surface of the specimen and light cured for 10 seconds in order to achieve a uniform structure of the PFC ring and prevent the formation of bubbles

within the PFC. Then prepare samples (Fig. 19) were introduced into a light curing chamber for 10 minutes. The samples were removed from the mold and cleaned with acetone (Fig. 18).

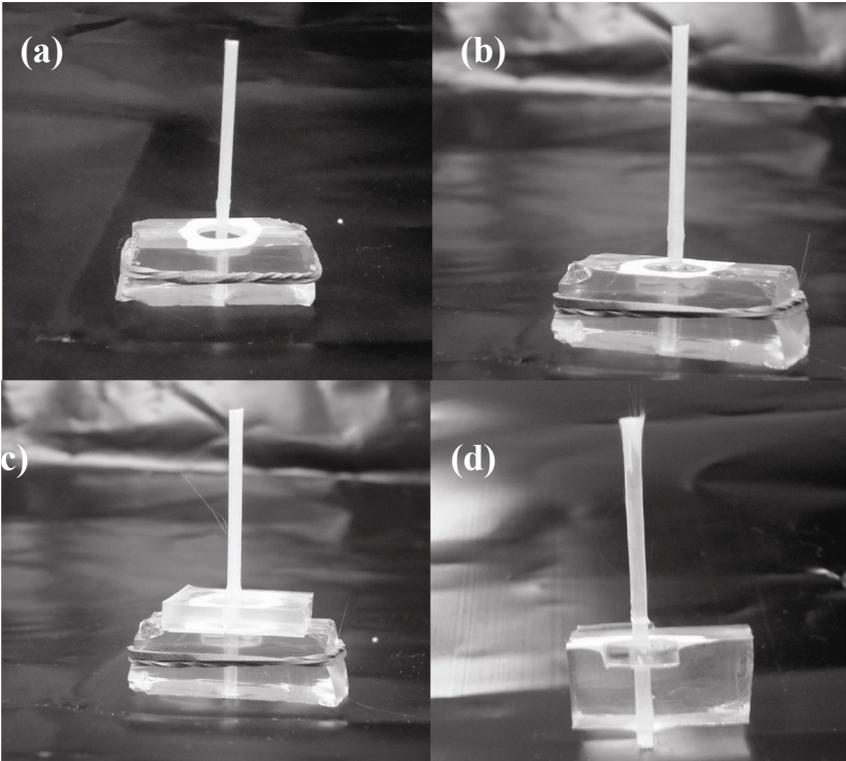


Fig. 18 sample preparation. (a) FRC in Lukopren mold, (b) application of PFC, (c) bubbles prevention (d) final sample.

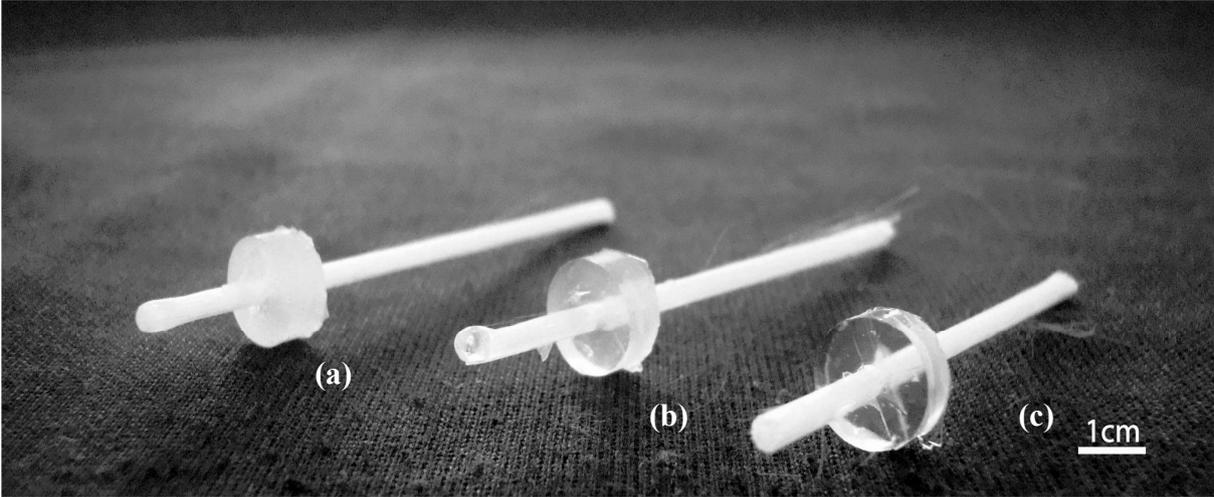


Fig. 19 Final test samples with different PFC rings; (a) 40 wt. %, (b) 10 wt. %, and (c) 0 wt. %.

The test specimen dimensions were the follows (Fig. 20):

L length of the FRC rod specimen approx. 50 mm

d diameter of FRC rod specimen approx. 1.65 mm

l adhesive joint length approx. 3 mm

b diameter of PFC ring approx. 8 mm

δ Interlayer's thickness approx 0.03 and 0.05 mm

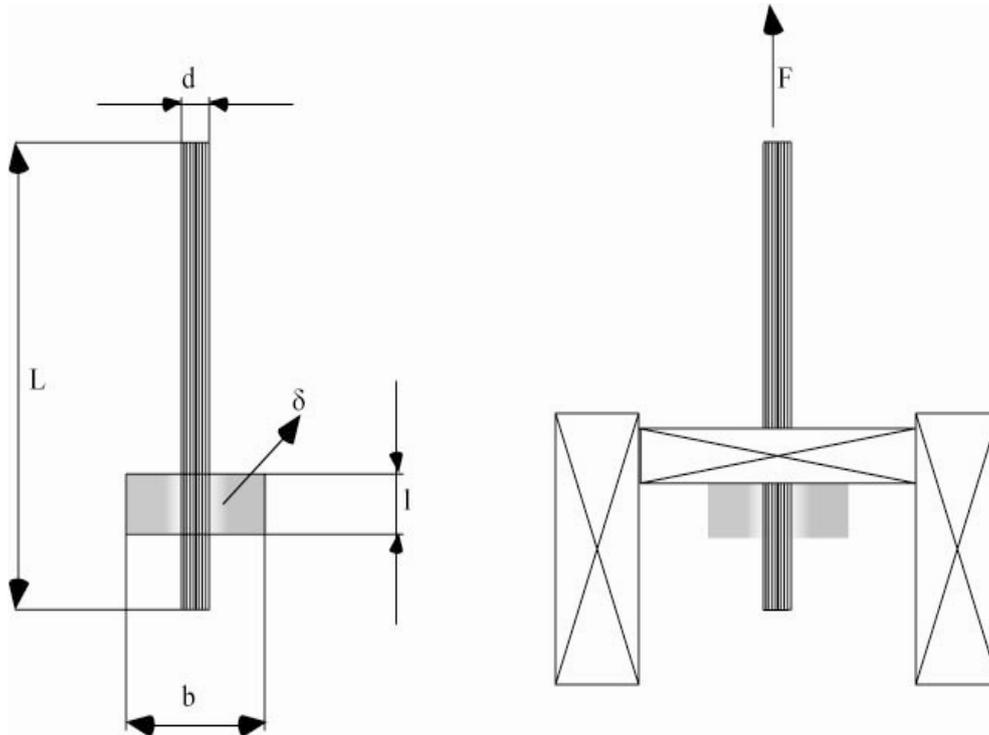


Fig. 20 Diagram of specimens dimensions and tethe applied load F .

4.7. Measurement Techniques

Universal Testing Machine Zwick Z010 (Zwick, Germany) testing instrument was used to perform the shear bond strength at room temperature (Fig. 21). Shear bond strength of adhesion was measured using a newly developed technique based on modification of the fiber pull-out test used for single filament adhesion testing. Special steel clamp was used to mount the specimens. Cross head speed of 2mm/min was used in all measurements and the test was run until fracture occurred. Maximum stress, stress at failure, deformation at failure were recorded using the Zwick data processing software.

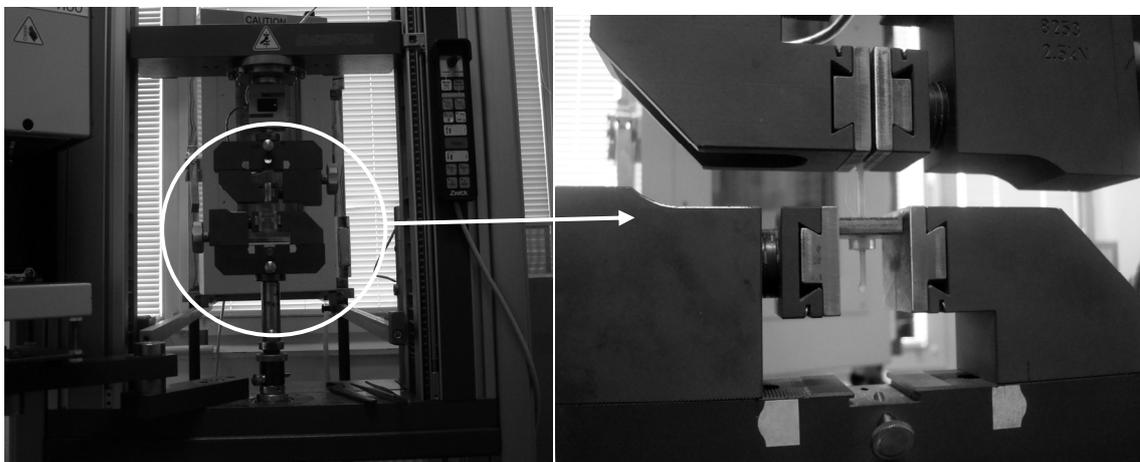


Fig. 21 Universal Testing Machine Zwick Z010 (Zwick, Germany)

Scanning electron microscope (SEM) was used to inspect the loci of failure in order to determine the type of fracture. Surfaces were mounted on a sample holder fixed with a carbon-acrylate adhesive; since the samples were not ductile a process of sputtering was necessary to coat the samples with a thin ductile layer created by plasma prior to placing in the SEM chamber.

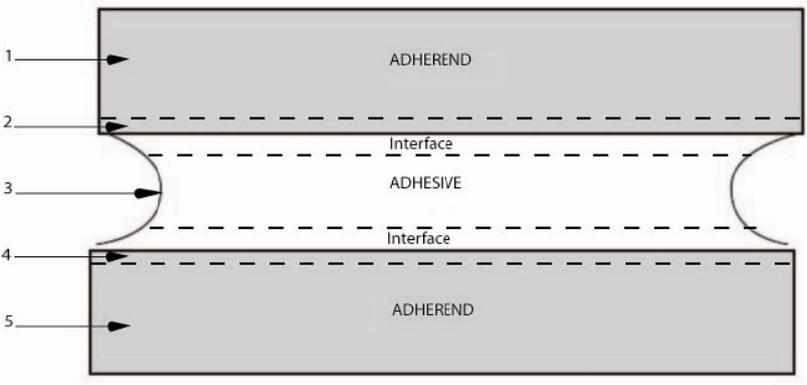


Fig. 22 five possible failure locus in an adhesive joint

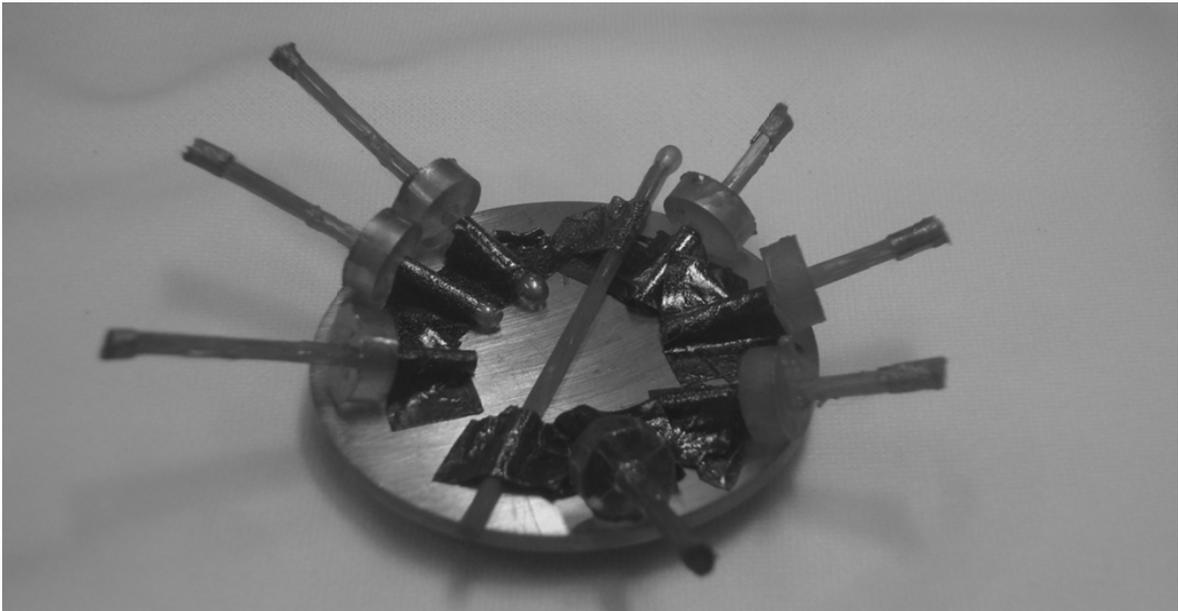


Fig. 23 Samples after the sputtering for SEM measurements

4.8. Equations and Calculations

For calculation of the shear bond strength was used following equation

$$\tau_a = \frac{F_{max}}{\pi \cdot d \cdot l} \tag{16}$$

where τ_a is the interfacial shear bond strength, F_{max} is the load at failure, d is the diameter of the FRC rod specimen and l is the adhesive joint length.

5. RESULTS AND DISCUSSIONS

The shear bond strength (τ) was determined for all the 14 groups of sample, each group consisted int. The difference between those groups was based on the interlayer composition (thickness and resin used) and the PFC filler content, whereas FRC rods remained identical for all the specimens. The measurement was run under laboratory conditions using a pre-load of 2 N and using a cross head speed of 2mm/min.

Tables 2, 3 and 4 show the value of shear bond strength, which were determine as an average from 6 measurements, together with their standard deviation. Appendixes 1 and 2 show typical stress-strain curves obtained during the measurements. The shear bond strengths of the different samples are represented as a function of the interlayer thickness (*Fig. 23*) and the filler content in PFC (*Fig. 24*).

Table 2 Values of shear bond strength and their respective standard deviation for samples without interlayer.

Without interlayer	τ [MPa]	Standard deviation [%]
FRC – A¹	15 ± 4	25%
FRC – B²	8 ± 1	19%
FRC - B10³	15 ± 2	15%
FRC - B40⁴	17 ± 1	6%

Table 3 Values of shear bond strength and their respective standard deviation for samples with interlayer A (resin mixture A – TEGDMA/bisGMA)

Thickness A	0.03 [mm]		0.05 [mm]	
	τ [MPa]	Stand. deviation [%]	τ [MPa]	Stand. deviation [%]
FRC- B	16 ± 2	16%	15 ± 2	17%
FRC - B10	18 ± 3	19%	15 ± 3	19%
FRC - B40	19 ± 1	8%	14 ± 3	18%

Table 4 Values of shear bond strength and their respective standard deviation for samples with interlayer B (resin mixture B – PEGDMA/bisGMA)

Thickness B	0.03 [mm]		0.05 [mm]	
	τ [MPa]	Stand. deviation [%]	τ [MPa]	Stand. deviation [%]
FRC - B10	19 ± 2	7%	21 ± 2	9%
FRC - B40	23 ± 3	11%	26 ± 3	11%

¹ Sample made of FRC rod – Resins mixture A (TEGDMA/bis-GMA)

² Sample made of FRC rod – Resins mixture B (PEGDMA/bis-GMA)

³ Sample made of FRC rod – PFC ring based on mixture B with 10 wt. % of barium glass powder

⁴ Sample made of FRC rod – PFC ring based on mixture B with 40 wt. % of barium glass powder

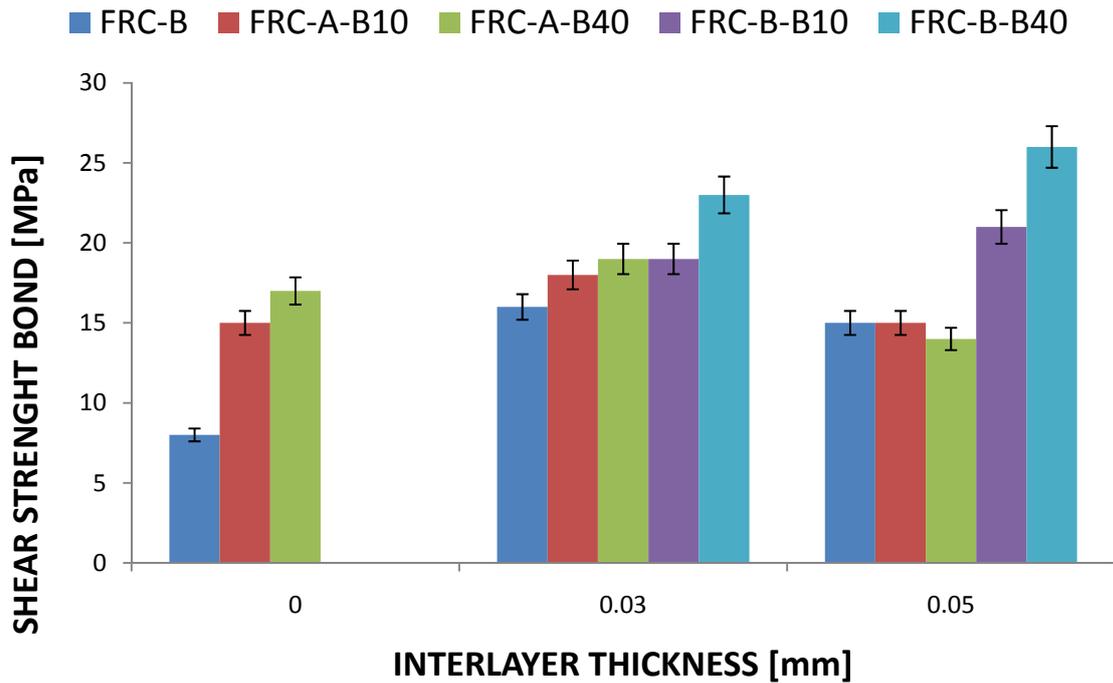


Fig. 24 The dependence of shear bond strength on interlayer's thickness (0 – without interlayer; 0.03 and 0.05 mm).

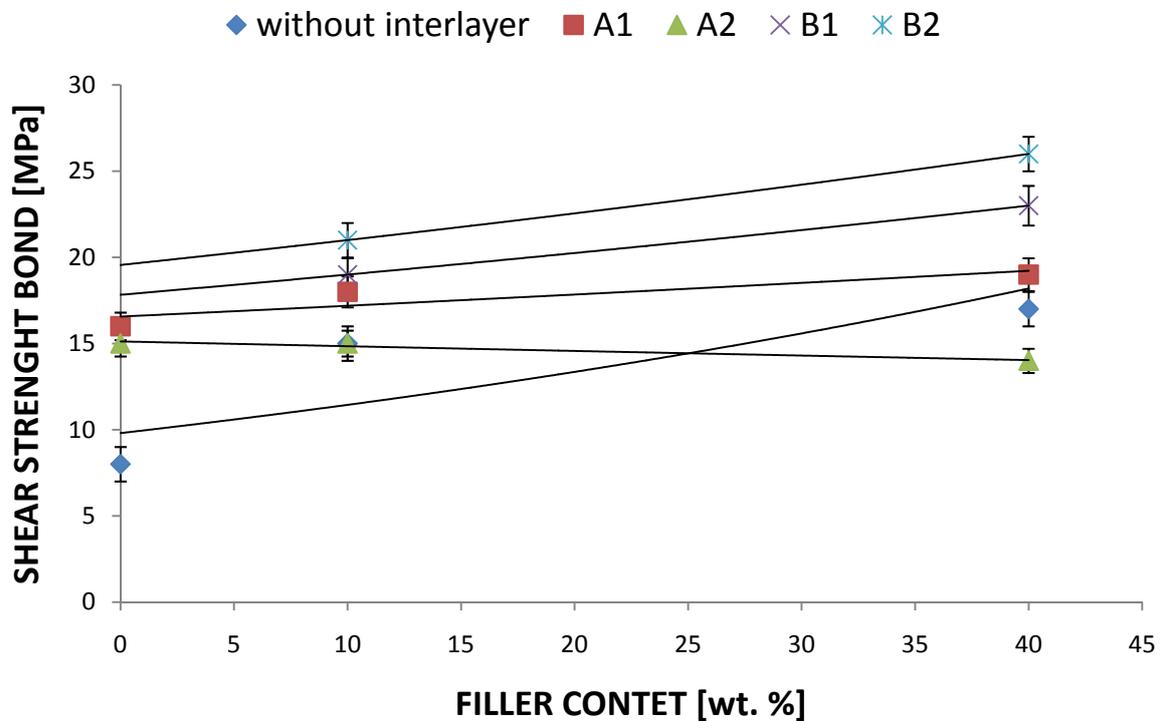


Fig. 25 The dependence of shear bond strength on filler content in PFC (0, 10 and 40 wt %). A1 Interlayer of resins mixture A (thickness of 0.03 mm), A2 Interlayer of resins mixture A (thickness of 0.05 mm), B1 Interlayer of resins mixture B (thickness of 0.03 mm), B2 Interlayer of resins mixture B (thickness of 0.05 mm)

The measured group without any interlayer was measured to define the standard shear strength bond between FRC and PFC. The failure in specimens FRC-A, FRC rod and ring made of resins mixture TEGDMA/bis-GMA, without an interlayer, was due the failure of the matrix (crack of the ring – Fig. 26).

Results show, that the composition of the interlayer between FRC and PFC materials seems to have a remarkable effects on the ultimate properties of the joint. This can be seen since, all the groups having interlayers compounded from the same resins mixture as the PFC matrix (groups with interlayer B), showed better response in terms of shear strength. At the same time a clear increase of these values was obtained from the specimens having an interlayer's thickness of 0.05 mm over the specimens with the interlayer's thickness of 0.03 mm.

On the other hand groups with interlayer A, also showed improvement of the strength in comparison with the same samples without an interlayer; but not more than samples with the same chemical composition of the interlayer and the PFC matrix. However for this group the influence of the interlayer's thickness was not clear because the results showed lower values for specimens with 0.05 mm thin interlayers than the ones having interlayers with a thickness of 0.03. Moreover results obtained from specimens with a bigger interlayer thickness were almost constantly no matter the content of filler particles in the PC ring.

Scanning Electron Microscope was used to determine the loci of failure of the specimens (Fig. 26 -32). Any of the groups with interlayers showed fibers interruption, whereas the cohesive failure of the matrix was the most common type of failure, meaning that the interfacial strength was stronger that the strength of the particle filled composite. This demonstrates that with the addition of interphase layer, a better wetting of the FRC rod is achieved and the adhesion bond is improved.

Is assumed that interphase regulates the materials differences between the FRC and the PFC, creating in that way an exponential dependence of the strength of the bond on gradient of the thickness of the interlayers, which adjust the composite's mechanical resistance of the adhesive bond. It is also important to mention that, it is highly probable that interphases are not homogeneous in the sense that their composition, structure and, therefore, properties could vary across their depth, for that reason the measurement of the adhesion and the influence of the interlayers could be a problem and a good preparation of samples is required, to minimize the standard deviation.

It has been proved that the interlayer's composition has a very big influence on the shear bond strength between FRC and PFCs. Since this interphase region is very sensitive to various environments, the study of these materials under conditions simulating the *aggressive* mouth environment, could have practical outcomes, about how parameters such a moisture and temperature can affect the adhesive bond of these materials used for clinical applications. This could be a subject matter of future investigations.

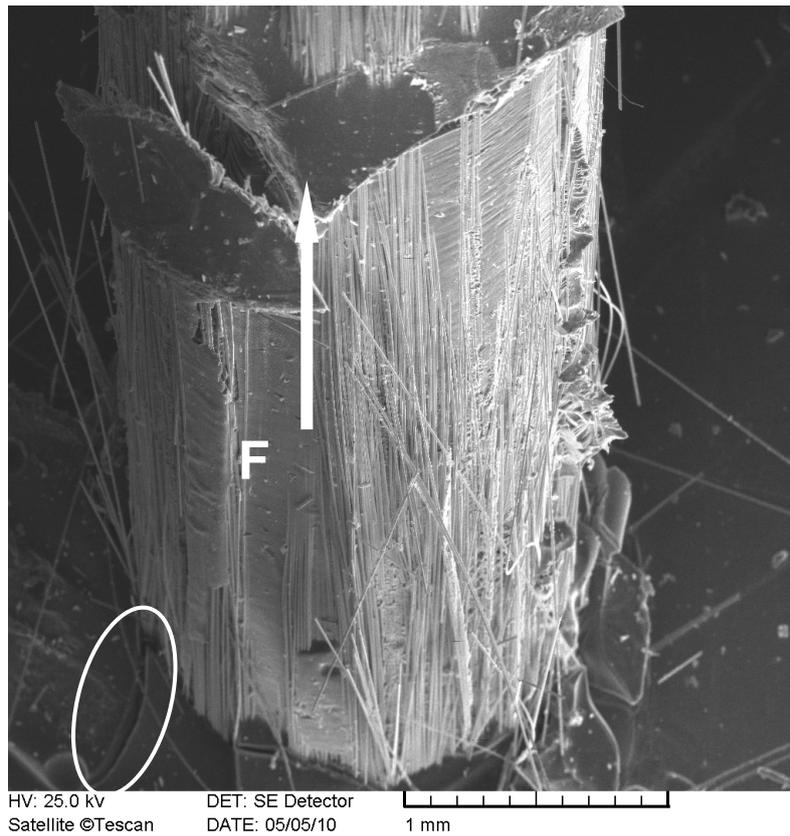


Fig. 26 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/mixture resin A (TEGDMA/bis-GMA) without interlayer, magnitude 100x.

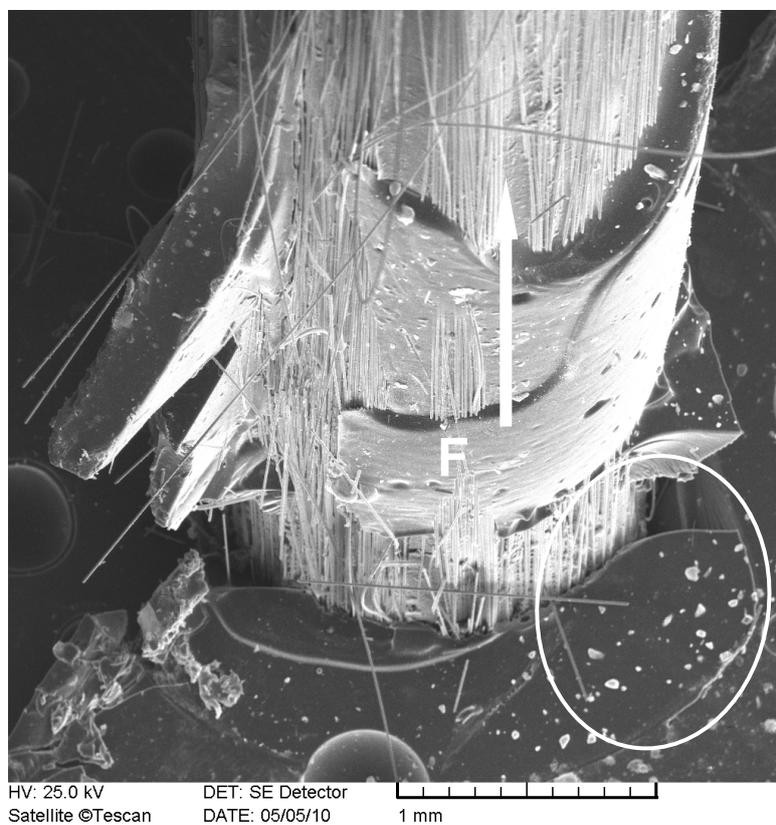


Fig. 27 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/PFC-B10 without interlayer, magnitude 100x.

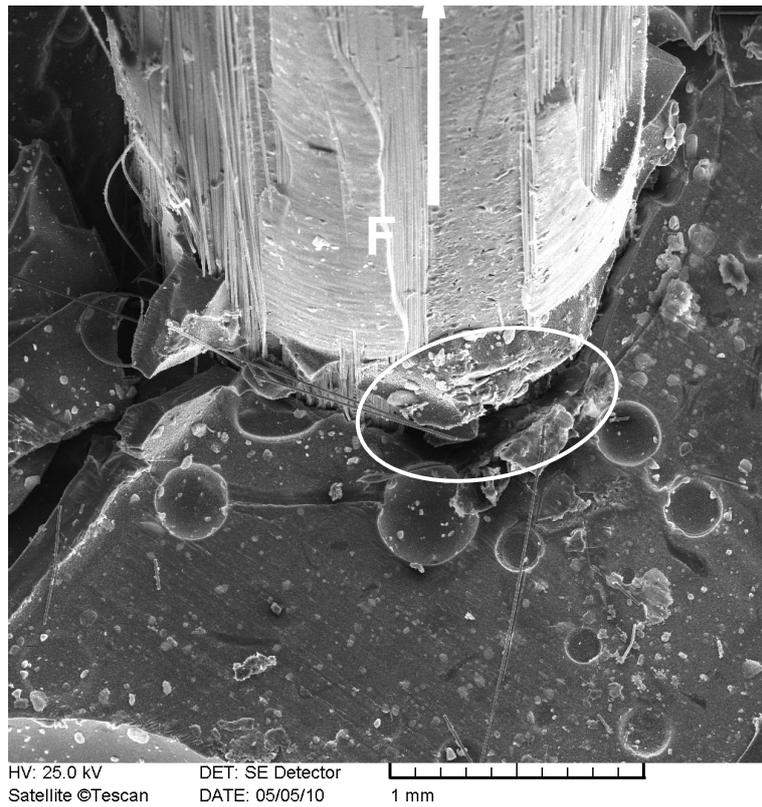


Fig. 28 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/PFC-B40 without interlayer, magnitude 100x.

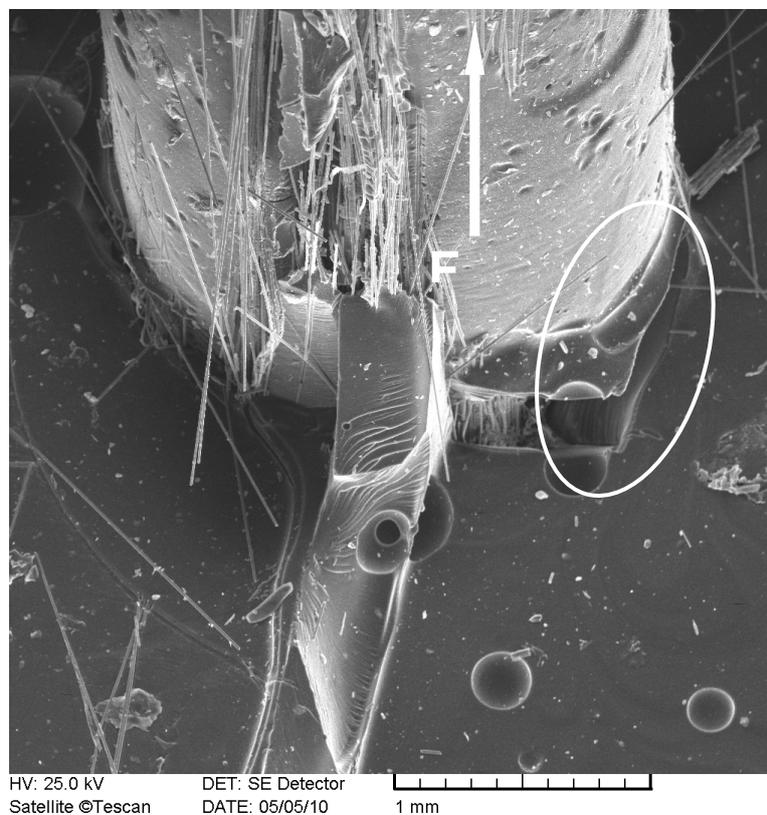


Fig. 29 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/A1/PFC-B10. Interlayer type - A1 (resin mixture TEGDMA/bis-GMA; thickness approx. 0.03 mm), magnitude 100x.

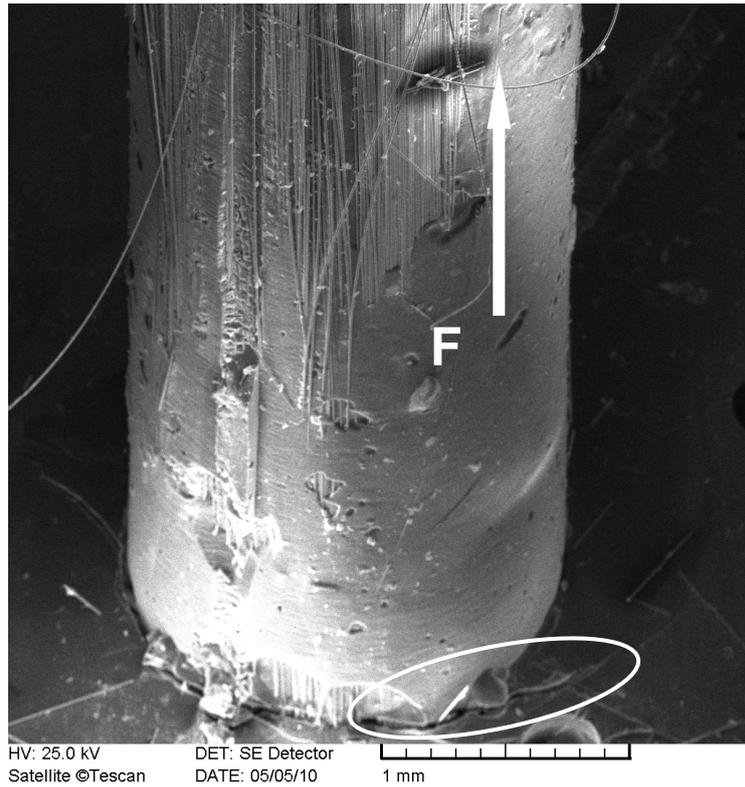


Fig. 30 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/A1/PFC-B40. Interlayer type - A1(resin mixture TEGDMA/bis-GMA; thickness approx. 0.03 mm), magnitude 100x.

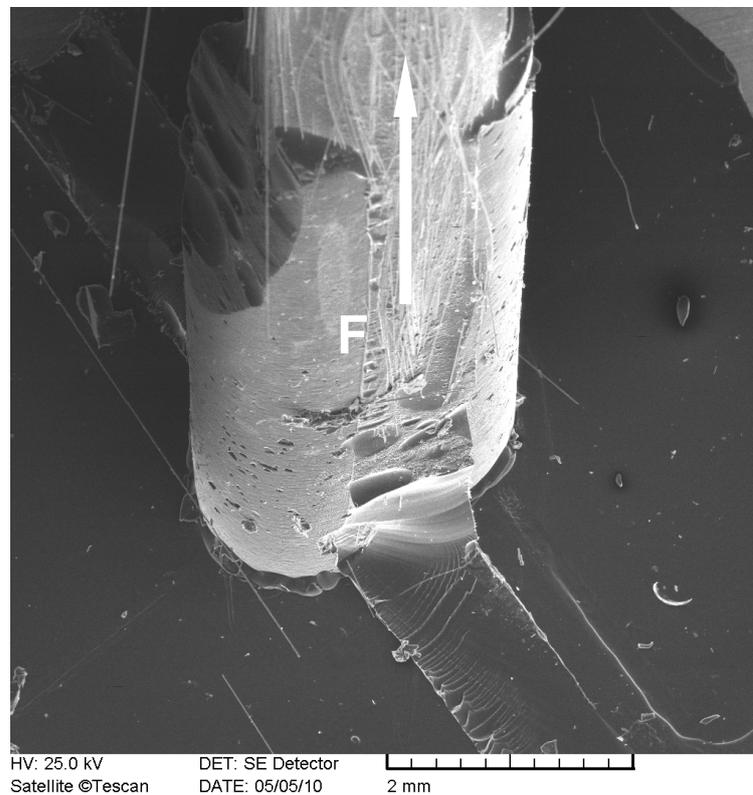


Fig. 31 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/A2/PFC-B10. Interlayer type – A2(resin mixture TEGDMA/bis-GMA; thickness approx. 0.05 mm), magnitude 49x.

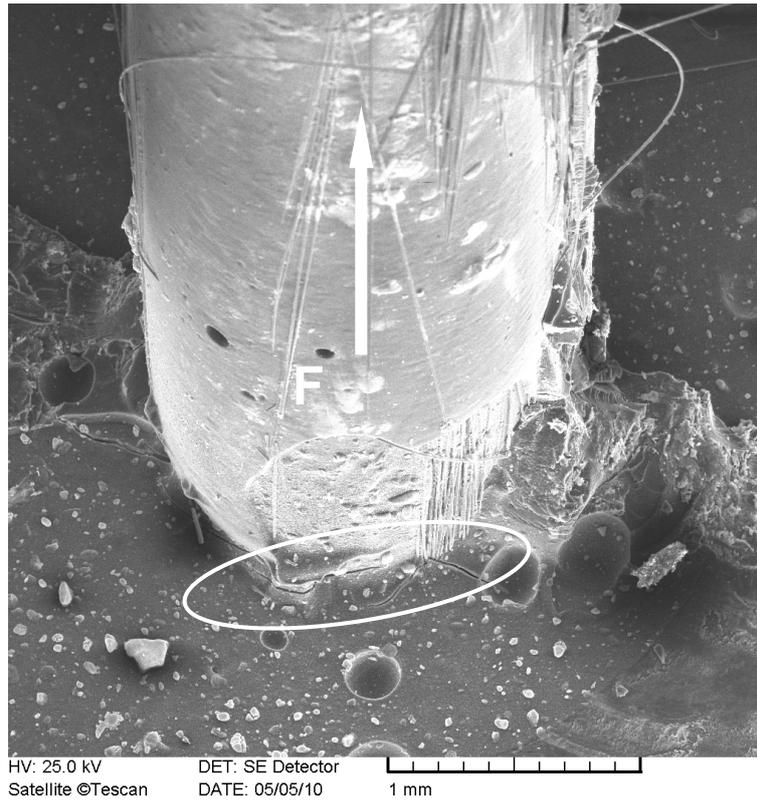


Fig. 32 Scanning electron micrograph of fracture surface from shear bond test of sample FRC/A2/PFC-B40. Interlayer type – A2(resin mixture TEGDMA/bis-GMA; thickness approx. 0.05 mm), magnitude 49x.

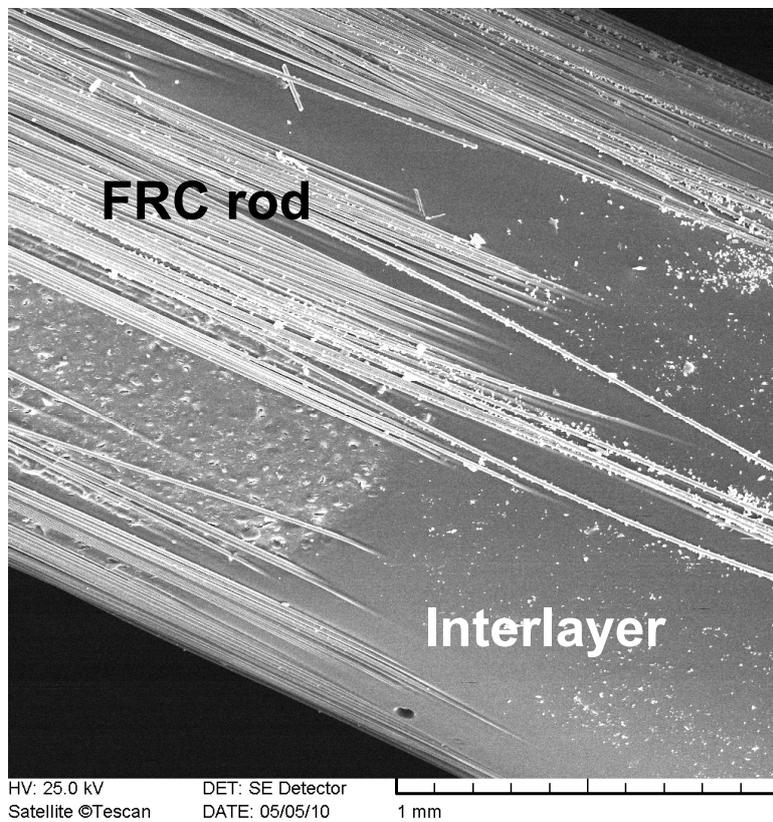


Fig. 33 Scanning electron micrograph of FRC rod coated with interlayer type – B1 (resin mixture PEGDMA/bis-GMA; thickness approx. 0.03 mm), magnitude 148x.

6. CONCLUSION

In this investigation, the influence of the interlayer's composition on the shear strength of adhesive bond between fiber-reinforced composites (FRC) and particle filled composites (PFC) was studied. The shear bond strength was determined by a pull-out test run under laboratory conditions by the testing machine ZWICK until failure of the sample occurred. The failure of the adhesive joint was localized using SEM observations.

The results obtained show that the biggest strength was in samples that had an interlayer of 0.05 mm based on the same chemical composition as the matrix of the PFC ring, which was 26 ± 3 MPa for PFC with a 40 wt. % of filler content of PFC rings and 21 ± 2 for PFCs with a 10 wt. % of filler content. In the same time samples with just a 0.03 interlayer also registered an improvement of the strength values 23 ± 3 and 19 ± 3 MPa for 40 wt. % and 10 wt. % PFCs respectively.

On the other hand depositing an interlayer between FRC and PFC materials of a different resin from the FRC/PFC system, registered as well an improvement of the strength bond of the joint, but not as big as the one using the same resin as interlayer. The values registered were 19 ± 1 and 18 ± 3 MPa for PFCs with 40 and 10 wt. % of barium glass powder.

From the literal research and from the obtained results, it can be concluded that thin interlayers, have remarkable effects in the adhesive joint between different composite materials. The addition of an interlayer between the fiber-reinforced and particle filled composite, results into an improvement on the shear strength bond. This is of vital importance, since the use of FRC/PFC systems have been progressively increasing in dentistry due the combination of the characteristics such as strength, wear resistance and esthetics. Although during the past years there has been a significant increase of the uses of composite materials in clinical application, there is small information about the effect of interlayers on the bonding of FRC and PFC.

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8. LIST OF USED SYMBOLS AND ABBREVIATIONS

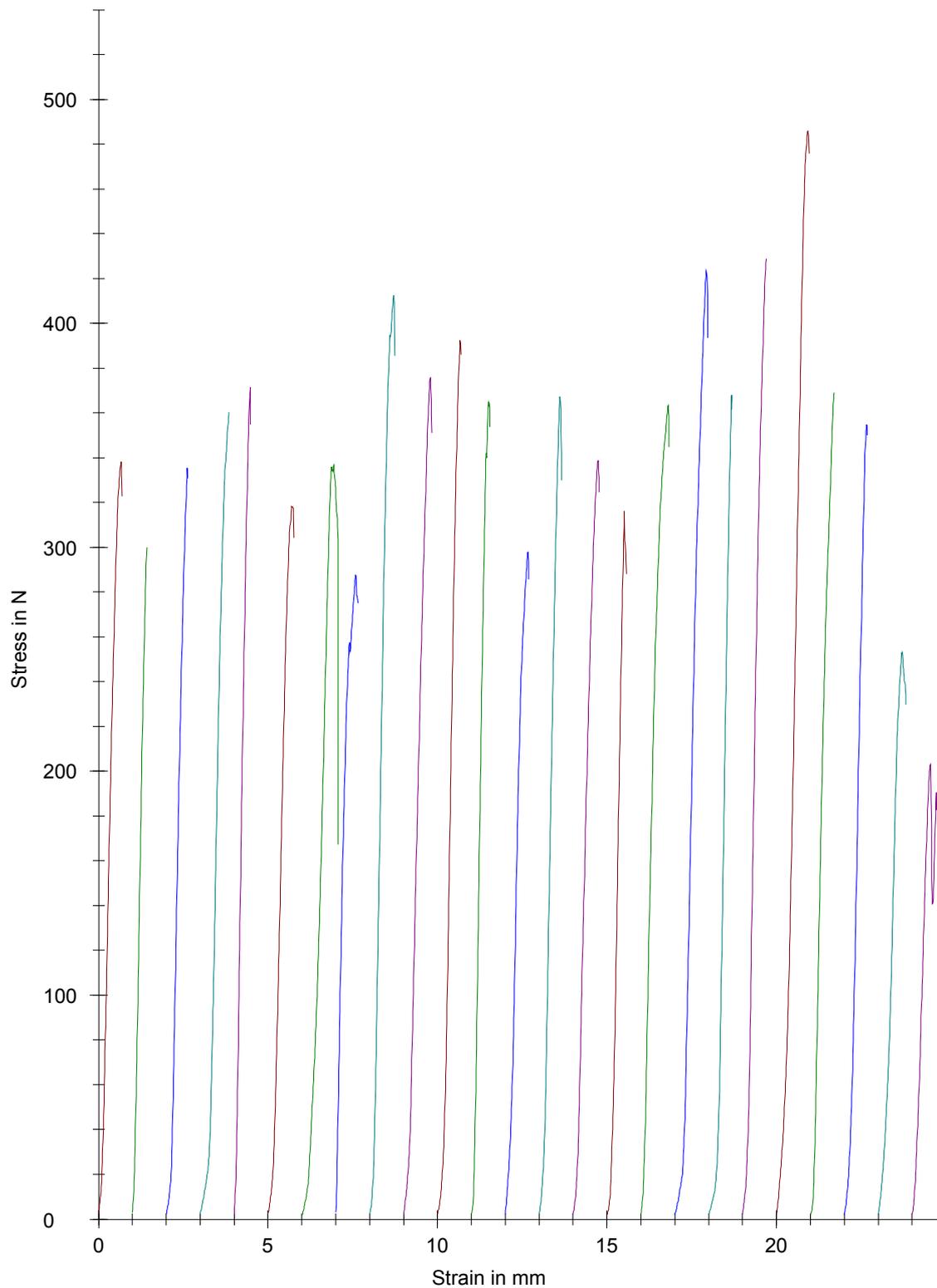
ABBREVIATIONS LIST

BPO	Benzoyl peroxide
BIS-GMA	2,2-bis[4-(2-hydroxy-3-methacryloyloxypropyl)phenyl]propane
FPD	Fixed Partial Denture
FRC	Fiber-Reinforced Composite
PEGDMA	Polyethyleneglycol dimethacrylate
PFC	Particle Filled Composite
PMMA	Poly(methyl methacrylate)
SEM	Scanning Electron Microscope
TEGDMA	Triethyleneglycol dimethacrylate
UV	Ultra Violet light
UHMWPE	Ultra-high-molecular-weight-polyethylene
VIS	Visible light

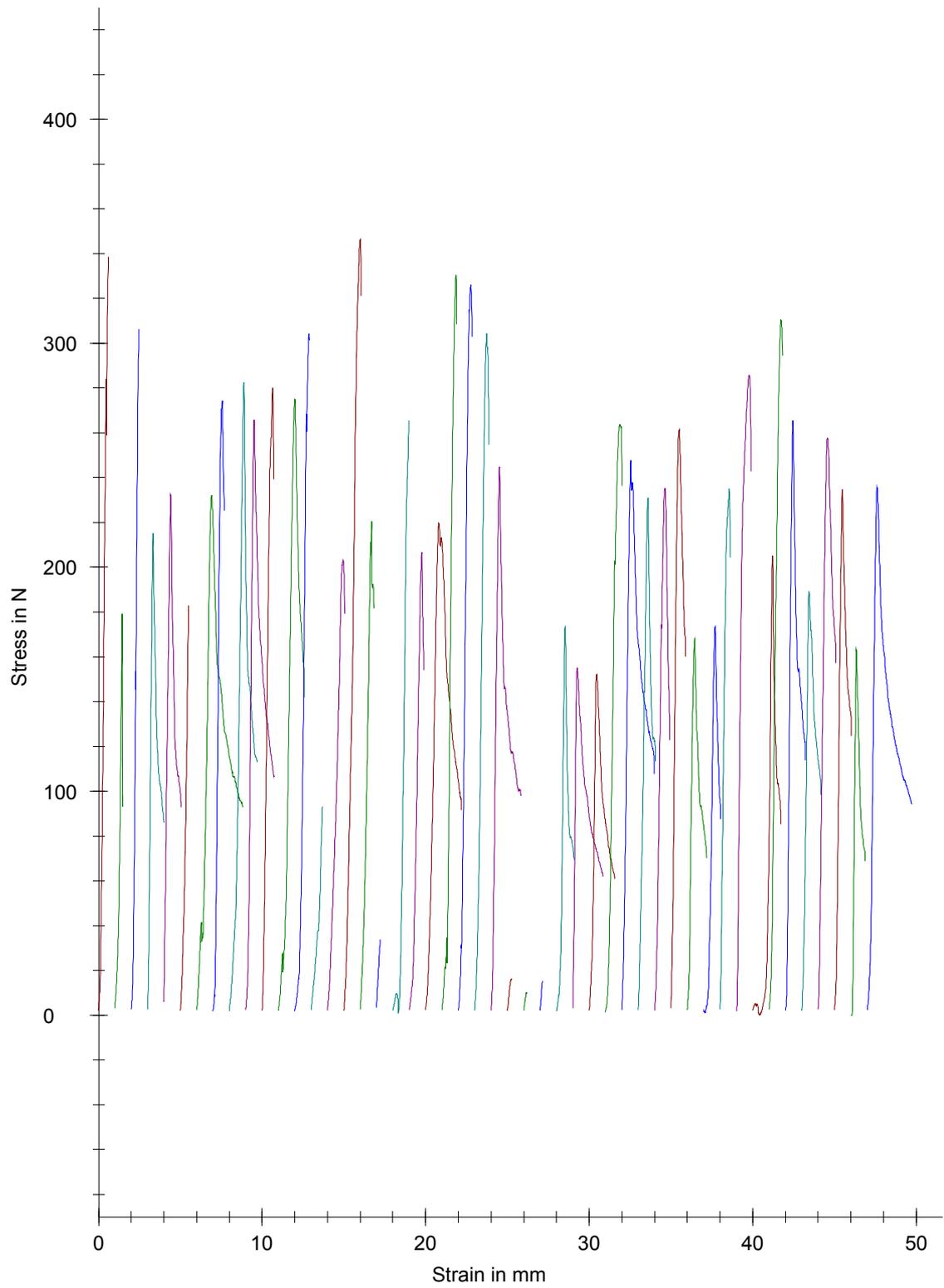
SYMBOLS LIST

l	Adhesive joint length [mm]
ρ	Density [$\text{g}\cdot\text{cm}^{-3}$]
d	Diameter [mm]
E	Elastic modulus [MPa]
G_c	Energy released rate [J]
G_p	Energy used in plastic deformation [J]
δ	Interlayer's thickness [mm]
F_{max}	Load at failure [N]
τ_a	Shear bond strength [MPa]
K	Stress intensity factor
σ	Stress at failure [MPa]
γ	Surface free energy [J]
ν	Poinsson's ratio
W_A	Work of adhesion [J],
W_c	Work of cohesion [J]

9. APENDIX



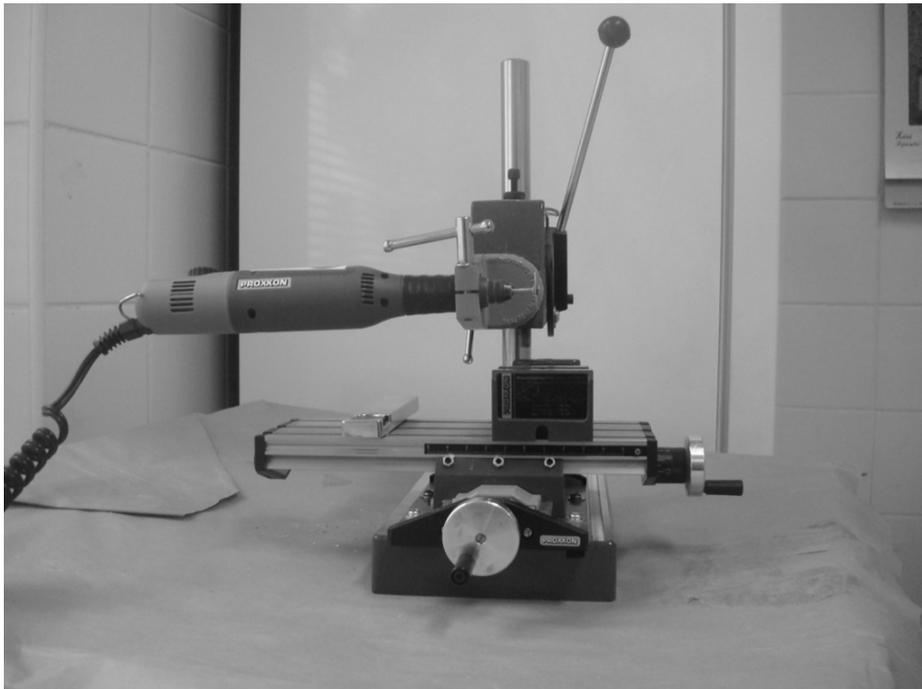
AP 1 Typical Stress-strain curves obtained during the measurement of shear strength bond, using Universal Testing Machine ZWCIK Z010. Measurement of samples with interlayer B



AP 2 Typical Stress-strain curves obtained during the measurement of shear strength bond, using Universal Testing Machine ZWCIK Z010. Measurement of samples without interlayer and with interlayer A



AP 3 Dentapreg™ UV lamp (ADM s.r.o, Czech Republic) used in the investigation.



AP 4 MICROMOT 50/E Cutting Tool (Proxxon, Germany) using to cut FRC into the length of 50 mm



AP 5 Targis Power light curing chamber (Ivoclar, Liechtenstein)