

# VYSOKÉ UČENÍ TECHNICKÉ V BRNĚ

BRNO UNIVERSITY OF TECHNOLOGY

FAKULTA CHEMICKÁ  
ÚSTAV CHEMIE MATERIÁLŮ

FACULTY OF CHEMISTRY  
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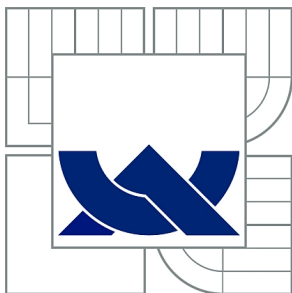
EFFECT OF ADDING MULTIDIRECTIONAL ORIENTED FIBERS ON  
MECHANICAL PROPERTIES OF DENTURE BASE RESIN

DIPLOMOVÁ PRÁCE  
MASTER'S THESIS

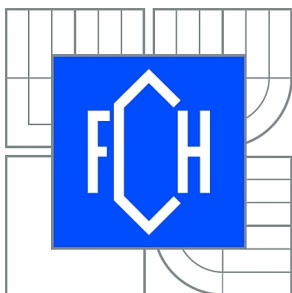
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## EFFECT OF ADDING MULTIDIRECTIONAL ORIENTED FIBERS ON MECHANICAL PROPERTIES OF DENTURE BASE RESIN

VLIV PŘÍDAVKU VÍCESMĚRNĚ ORIENTOVANÉ VÝZTUŽE NA MECHANICKÉ VLASTNOSTI  
MATERIÁLU PRO PŘÍPRAVU CELKOVĚ SNÍMATELNÝCH NÁHRAD

DIPLOMOVÁ PRÁCE  
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### Název diplomové práce:

Vliv přídavku vícesměrně orientované výztuže na mechanické vlastnosti materiálu pro přípravu celkově snímatelných náhrad

### Zadání diplomové práce:

Cílem práce je charakterizace mechanických vlastností kompozitního materiálu skládajícího se z křehké termoplastické matrice vyztužené vícesměrně orientovanými splétanými vlákny. Hlavní pozornost je věnována vlivu tohoto vyztužení na houževnatost takto připraveného materiálu a jeho srovnání s běžně komerčně dostupnými řešeními.

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## **Abstract**

This Diploma thesis deals with effect of adding multidirectional oriented fibres on mechanical properties of denture base resin. Three sets of samples: PMMA resin, PMMA resin reinforced with Dentapreg Mesh and PMMA resin reinforced with stainless steel Mesh Strengthener were used for testing. Static three-point bending test and Charpy instrumented impact test were used to measure the behaviour of the samples under the static and dynamic loading conditions. Viscoelastic properties were determined by dynamic-mechanical analysis (DMA). Scanning electron microscopy (SEM) was used to characterize fracture surfaces. During the static loading of the samples the reinforcement does not play an important role in comparison with unreinforced resin. However, the opposite trend was observed under the dynamic loading where the effect to the reinforcement was increasing in the row PMMA resin – stainless steel Mesh Strengthener – Dentapreg Mesh.

## **Key words**

PMMA, Dentapreg Mesh, stainless steel Mesh, three-point bending test, Charpy un-notched impact strength, SEM, DMA

## **Abstrakt**

Tato Diplomová práce se zabývá vlivem vyztužení na mechanické vlastnosti materiálu pro přípravu snímatelných náhrad. Pro testování byly použity tři sady vzorků: PMMA pryskyřice, PMMA pryskyřice s Dentapreg Mesh výztuží a PMMA pryskyřice vyztužená zesilující sítkou z nerezové oceli. Pro měření chování vzorků při statickém a dynamickém namáhání byl použit třibodový ohybový test a Charpy rázové kladivo. Viskoelastické vlastnosti byly měřeny dynamicko-mechanickou analýzou (DMA). Skenovací elektronová mikroskopie (SEM) byla použita k charakterizaci lomových ploch. Během statického namáhání vzorků výztuž nehraje důležitou roli v porovnání s nevyztuženou pryskyřicí. Nicméně byl pozorován opačný trend při dynamickém namáhání, kde efekt výztuže rostl v řadě PMMA pryskyřice – sítka z nerezové oceli – Dentapreg Mesh.

## **Klíčová slova**

PMMA, Dentapreg Mesh, sítka z nerezové oceli, tří-bodový ohybový test, Charpy rázová houževnatost bez vrubu, SEM, DMA

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#### **DECLARATION**

*I declare that the diploma thesis has been worked out by myself and that all the quotations from the used literary sources are accurate and complete. The content of the diploma thesis is the property of the Faculty of Chemistry of Brno University of Technology and all commercial uses are allowed only if approved by both the supervisor and the dean of the Faculty of Chemistry, VUT.*

.....  
Student's signature

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# **1 INTRODUCTION**

Failure of complete denture prosthesis made from polymethylmethacrylate (PMMA) is most likely from of fracture either due to fatigue or impact forces of mastication. Flexural fatigue of dentures as evidenced by midline fracture is due to the stress concentration around the micro cracks formed in the material due to continuous applications of small forces. Fracture of dentures by impact forces, on the other hand, results from the sudden application of force to the dentures. Such types of fractures are more likely due to the accidental dropping of dentures on surfaces.<sup>1</sup>

Research in this area aimed at modifying the composition or reinforcing the PMMA with stronger materials and developing new materials with better properties.<sup>1</sup> Currently starting to use reinforcement from glass fibres. They have excellent aesthetic appearance, superior mechanical properties and biological compatibility.

This Diploma thesis deals with characterization of the mechanical properties of the composite material consisting of brittle matrix reinforced with glass multidirectional oriented fibres. The main attention is focused on the impact behaviour of commercially available denture base material reinforced with commonly used stainless steel multidirectional reinforcement or with the new brand of glass fibre multidirectional reinforcement, respectively.

## **2 THEORETICAL PART**

### **2.1 The history and development of posterior denture teeth**

The earliest examples of complete dentures used in the Far East come from early sixteenth century Japan. These unique restorations were carved from a solid piece of a sweet-smelling wood, such as apricot, cherry, or boxwood.

Early eighteenth-century Europe experienced a high point in Western civilization and culture. In 1756 Phillip Pfaff first described a wax sectional impression technique to generate plaster casts of a patient's mouth. In French Fauchard applied a jeweller's enamel to teeth made from thin gold plate. He is also credited by some for originating the idea of using porcelain teeth.<sup>2</sup>

Before the early twentieth century, the occlusal anatomy of posterior denture teeth was an arbitrary creation (whether "anatomic" or "nonanatomic"). It was not designed to follow any occlusal philosophy or theory of mandibular movement. These posterior denture teeth could not be set in harmony (or to balance) with any particular determinants of occlusion.

In 1913 were introduced first anatomic teeth by Dr. Alfred Gysi. Maxillary and mandibular teeth had distinctive occlusal anatomy. Molars and premolars were properly proportioned, and teeth meant for one side could be differentiated from teeth meant for the other side.<sup>3</sup>

Dentures today are made out of all different sorts of rubbers and plastics. Of the main materials in dental prosthetics made all the known types of refunds, which are made up of either one material, or their mutual combinations. Many of the materials we use today known evolutionary three basic: metal alloys (Cu, Fe, Au, Ag), the ceramic material ("porcelain") and plastics (the most important and practically universally used are acrylic polymers, in particular still polymeric methylmethacrylates).

The manufactory of denture starts with impression. The impression must faithfully reproduce the oral situation and serve as a basis to obtain its exact model. Requirements for impression materials are so strict. According to the nature of the impression material the material is divided into chemoplastic (composite materials, the test mass) or thermoplastic (plaster, silicone mass).<sup>4</sup>

Between the main interests for dentures include the reinforcement. Use the metal mesh, fibreglass composites. The main purpose of the reinforcement is to improve the mechanical properties of the product. Furthermore, the focus is on good handling in the application and also the aesthetics of the finished product. The disadvantage of composites reinforcement is the price, and perhaps distrust of the older generation of new material (any changes).

## **2.2 Polymethyl methacrylate**

### **2.2.1 General characteristics**

Acrylics are plastics whose base polymers are polymers, monomers structurally derived from acrylic acid or copolymers of acrylic acid. An example of a popular plastic in this group is polymethyl methacrylate, also known simply as acrylic or Plexiglas.

Acrylic plastics are based generally on polymethyl methacrylate. PMMA is produced by free-radical polymerization of methylmethacrylate in mass (when it is in sheet form) or suspension polymerization.

The large forms of this material (e.g., sheet, rods, and tubes) are polymerized in situ by casting a monomer that has been partly pre-polymerized by removing any inhibitor, heating, and adding an agent to initiate free radical polymerization. This agent is typically organic peroxide (e.g. dibenzoyl-peroxid). As such, this technique is not suitable for producing injection moulding resin, but it does aid in producing material that has a large rubber plateau and has a high enough elevated-temperature strength to allow for band sawing, drilling, and other common finishing processes, as long as the localized heating does not reach the polymer's decomposition temperature. These bulk polymerized materials generally have such a high molecular weight that they cannot be subjected to additional thermal processing or fabrication without degradation. Cast acrylic is used extensively as bathtub material, such as in showers and in whirlpools; display parts; and household decorative items.

Acrylic beads are made via suspension polymerization. This produces a material with low enough molecular weight to allow for typical melt processing via injection moulding, casting, extrusion, or vacuum and pressure forming.<sup>5</sup>

### 2.2.2 Properties of PMMA

PMMA has high mechanical strength, high Young's modulus and low elongation at break. It does not shatter on rupture. It is one of the hardest thermoplastics and is also highly scratch resistant. It exhibits low moisture and water absorbing capacity, due to which products made have good dimensional stability. Both of these characteristics increase as the temperature rises. Disadvantages include brittleness and relatively large shrinkage during polymerization.

Its strength properties during injection moulding differ significantly in longitudinal and transverse direction as a result of the orientation effect. As in the case with other thermoplastics, the mechanical properties of PMMA vary as the temperature changes. This material tends to creep. It is not suitable for operation under multiple dynamic loads.

PMMA is one of the polymers that is the most resistant to direct sunshine exposure. Its strength characteristics exhibit fairly small variations under the effect of UV-radiation, as well as in the presence of ozone.

The low water absorption capacity of PMMA makes it very suitable for electrical engineering purposes.

The thermal stability of standard PMMA is only 65°C. Heat stabilised types can withstand temperatures of up to 100°C. PMMA can withstand temperatures as low as -70°C. Its resistance to temperature changes is very good.

PMMA exhibits very good optical properties – it transmits more light (up to 93% of visible light) than glass. Combined with its good degree of compatibility with human tissue, it can be used for replacement intraocular lenses or for contact lenses.

Acrylics are unaffected by aqueous solutions of most laboratory chemicals, by detergents, cleaners, dilute inorganic acids, alkalis, and aliphatic hydrocarbons – however, acrylics are not recommended for use with chlorinated or aromatic hydrocarbons, esters, or ketones. It dissolves completely in chloroform, di- and trichlorethane, which is used for production of glues. The chemical resistance will vary with stress level, temperature, reagents and duration of exposure. <sup>6</sup> Table 1 shows some of mechanical properties of PMMA.

**Table 1:** Mechanical properties of PMMA<sup>7</sup>

Properties	Value
Density, 25 °C	1,19 g/cm <sup>3</sup>
Flexural modulus	1,68 – 3,30 GPa
Flexural strength	49,0 – 131,0 MPa
Charpy Unnotched Impact Strength	13,3 – 25,3 kJ/m <sup>2</sup>
Tensile modulus	3,13 – 3,34 GPa
Tensile strength (break)	48,9 – 77,2 MPa

## 2.3 Polymerization of PMMA

### 2.3.1 Bulk polymerization

A typical charge might consist of monomer, a monomer-soluble initiator, and perhaps a charge-transfer agent. Simple rate and heat transfer problems due to reaction kinetics generally create problems with bulk polymerization. The addition of inert solvent to a bulk polymerization mass (solution polymerization) minimizes many of the difficulties encountered in bulk systems. This reduces the tendency toward auto acceleration, and the inert diluent adds its heat capacity without contribution to the evolution of heat. It also cuts the viscosity of the reaction mass at any given conversion. The heat of polymerization may also be conveniently and efficiently removed by refluxing the solvent.<sup>5</sup>

### 2.3.2 Suspension polymerization

The heat removal is facilitated by keeping the dimension of the reaction mass small. This is carried out by suspending the monomer in the form of droplets in an inert, non-solvent liquid (almost always water). In this way each droplet becomes a single bulk reactor, but with dimensions small enough so that heat removal is not a problem, and the heat can easily be soaked up by and removed from the low-viscosity, inert suspension medium.<sup>5</sup>

### 2.3.3 Free radical polymerization

Free radical polymerizations are classic chain reactions, wherein initiator thermolysis introduces free radicals at a moderate rate. Polymerization is initiated by

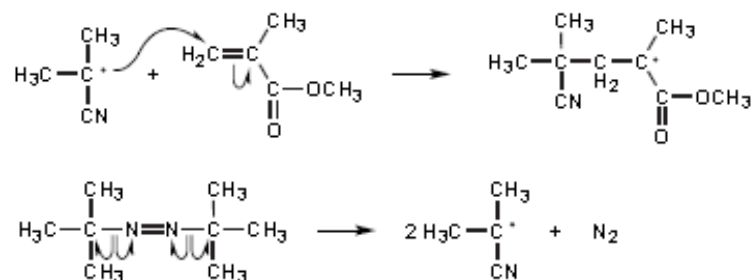
the action of free radicals, electrically neutral species with an unshared electron. Free radicals are usually generated by the photo or the thermal decomposition of organic peroxides or azo compounds.<sup>8</sup> The chain reaction involves three steps: initiation, propagation and termination.

**Initiation** is the first step in the free radical polymerization process. During initiation, free radicals are generated, usually by the decomposition of an initiator. The free radicals that are formed then react with the monomer to form other free radicals that are capable of chain growth.

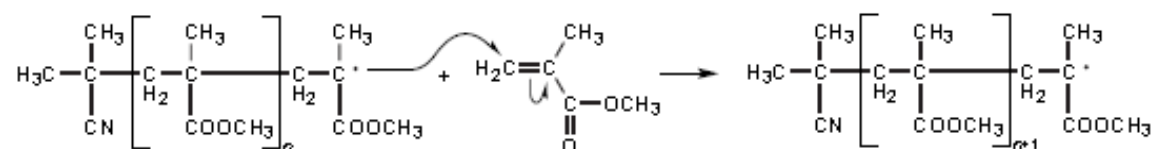
**Propagation** is the second step in a free radical polymerization process. The chain radicals that are formed in the initiation step are capable of adding successive monomers to propagate the chain.

The **termination** step stops the chain growth. Generally, this occurs because of the strong tendency of radicals to react in pairs to form a paired-electron covalent bond with loss of radical activity.<sup>5</sup> Scheme of free radical polymerization of PMMA is shown in Figure 1.

## Initiation

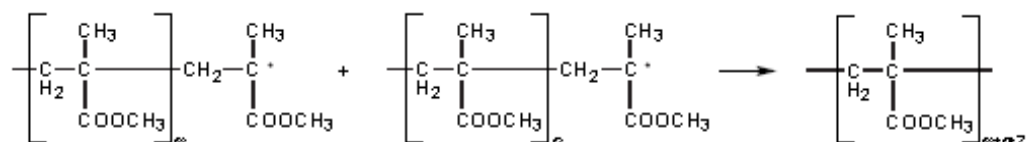


## Propagation

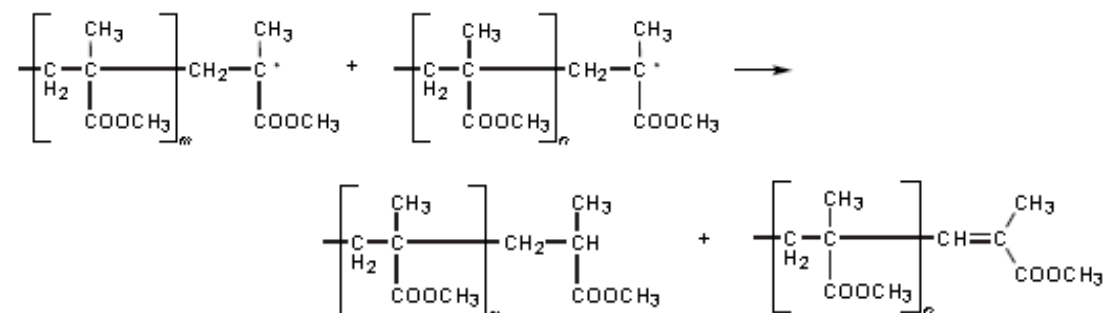


## Termination

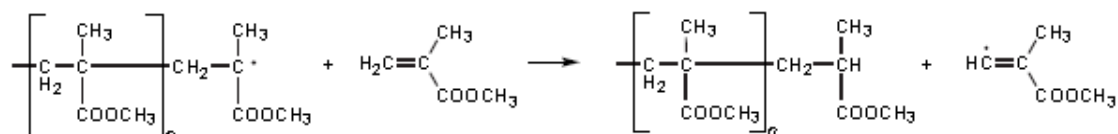
### Combination



### Disproportionation



## Transfer



**Figure 1** Free radical polymerization of MMA with AIBN as the initiator.<sup>9</sup>

## 2.4 Composites

It is called the composite material in which are combined in a specific way two or more components or phases of significantly different physical and chemical properties. Composite materials can be divided according to the type of filler on the particle and fibre.<sup>10</sup>

### 2.4.1 Particulate composites

For the plastics is one of the components of the polymeric matrix and the other called the continuum are discrete particles mostly inorganic fillers. Particle size in most practically usable filled plastic ranges is between 1-20  $\mu\text{m}$ . An important structural parameter for particle composites is the content of fillers, which is expressed as a volume fraction of filler:

$$V_f = \frac{V_f}{V_{total}},$$

where  $V_f$  is the amount of fillers contained in the composite, and  $V_{total}$  is the total volume of the composite.<sup>10</sup>

#### 2.4.1.1 The function of filler in the particular composites

Particle composites are produced industrially by mixing powdered and liquid matrix. Matrix particle composites are the bearer of all essential mechanical-physical and chemical properties. The bearer of the stiffness of the particle composites is a filler and polymeric matrix. The role of the matrix increases with decreasing content of fillers.

The primary mechanism to increase the rigidity of conventional particle composites is the replacement of the volume of the low-module matrix high-module material. With conventional fillers such as limestone ( $\text{CaCO}_3$ ), aluminium hydroxide ( $\text{Al}(\text{OH})_3$ ), talc is the rate of growth of the flexibility with the content given by the shape and size of the filler particles. For particles with non-spherical shape ( $\text{Mg}(\text{OH})_2$ , a short glass fibres) appears a significant effect of particle fillers orientation, which is more pronounced than the effect of the orientation of the structure of the polymer matrix. In these cases, the modulus of elasticity is growing with the content of fillers for composites faster than spherical particles.<sup>10</sup>



### 2.4.2 Fibre composites

Unreinforced resins has a low density is easily workable and has a relatively good stability against exposure to a wide range of environment and chemicals. The disadvantage is the low modulus of elasticity, low strength, fragility and relatively little creep resistance. On the other hand fibres have the required strength and rigidity, but they are very fragile and susceptible to damage in aggressive environments. Another disadvantage is difficult to maintain them in the desired space layout. By combining resins and fibre reinforcement in required ratio we are able to prepare suitable composite material for different type of applications.<sup>10</sup>

Fibre composites are often classified according to the orientation and length of the reinforcing fibres:

- **unidirectional** (the fibres are oriented predominantly in one direction):
  - short-fibres (length/diameter ratio  $L/D < 100$ )
  - long-fibres ( $L/D > 100$  or continuous fibres with a length equal to the size of the entire panel)
- **multidirectional** (the fibres are oriented randomly or regularly two or more directions):
  - short-fibres ( $L/D < 100$ )
  - long-fibres ( $L/D > 100$ )

#### 2.4.2.1 Types of fibres

The main technological structural parameter that controls the properties of the resulting composite is given the type of the fibre and resin fibre content. For technological process is given the weight fraction of fibres, which can be used directly for the dosage in production. Among the most commonly used fibres are glass and carbon fibres.<sup>10</sup> Table 2 shows some mechanical properties of chosen fibres.

#### Glass fibres

Glass fibre refers to individual filaments made by attenuating molten glass in special fibre-forming furnaces. A continuous filament is a glass fibre of great or indefinite length; a staple fibre is a glass fibre of relatively short length.<sup>5</sup>

The most common glass fibre in the world produced mainly from the enamel known as glass E. It is based on a system of oxides of  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{MgO}$ - $\text{CaO}$ - $\text{B}_2\text{O}_3$ . Today, these fibres are produced by continuous technology.<sup>10</sup>

C-glass is designed for use where optimum chemical resistance is required. D-glass (a highboron-content glass) has very good electrical properties, particularly the dielectric constant, and is used in electronic applications. S-glass is used for high strength and stiffness, whereas R-glass is a lower-cost fibre than S-glass.<sup>5</sup>

### **Carbon fibres**

Carbon fibres have demonstrated a wide variety of strengths and module. These fibres are made from a pitch or polyacrylonitrile (PAN) precursor by an oxidation, carbonization, and graphitization process.

They are now used in such areas as plastic composites for automotive, aerospace, and sporting goods. Carbon fibres can be used to reinforce both thermosets and thermoplastics.<sup>5</sup>

Carbon fibre has certain properties that make it potentially useful in dentistry. It is biocompatible, corrosion-resistant, and strong. Most reports of the potential uses of carbon fibre in dentistry are limited to the reinforcement of existing restorative materials and as a possible post material. The carbon fibre post is reported to have a modulus of elasticity that is nearly identical to that of dentine, so that it causes less tooth stress and hence, fewer root fractures.<sup>11</sup>

They also lower the coefficient of friction and provide excellent resistance to most environmental exposure conditions and chemicals. The properties of the final composite are very much dependent on the degree of orientation of the fibres within the resin matrix. When the fibres are in complete alignment (e.g., unidirectional or all fibres are at an orientation of  $0^\circ$ ), the properties of the composite are more affected by the fibre than if the fibres are randomly aligned.

Compared with glass fibre-reinforced plastics, the carbon fibre composites generally feature greatly improved tensile and flexural strengths and module. Thermal expansion of the carbon formulations is lower, and mould shrinkage ranges from about one-half to one-fifth that exhibited by the non-reinforced resin. Thermal conductivity of carbon composites is about twice that of equivalent glass-reinforced formulations.<sup>5</sup>

### Aramid fibres

Kevlar is a trade name for an aramid fibre (or aromatic polyamide fibre) manufactured by Du Pont. When introduced in 1972, Kevlar essentially revolutionized pressure vessel technology because of its great strength in consistency coupled with low density, resulting in much more weight-effective designs for rocket motors. The specific tensile strength of Kevlar was, at its introduction, the highest for any fibre. Carbon/graphite fibres, because of advances in processing, have the highest values now.

The tensile strength of Kevlar fibre for reinforcing plastics is better than twice that of nylon or polyester and 15 percent better than that of E-type fibreglass. Elongation is low, and the density of the fibre is 40 percent lower than glass. Aramids are as strong as steel at 1/5 the weight.

Aramid fibres also have good chemical resistance except for strong acids and bases. They have high stress rupture life and fracture toughness, good cyclic tensile fatigue resistance, and fair damping characteristics. Environmental stability of composites is generally good except in extremely hostile environments.<sup>5</sup>

### Polyethylene (PE) fibres

The polyethylene fibres have the same shear and compression property drawbacks as the aramids, but they also suffer from a low melting temperature that limits their use to composites that cure or operate below 150°C and a susceptibility to degradation by ultraviolet (UV) light exposure. Both the aramids and the polyethylenes have wide use in personal protective armour, and the polyethylene fibres have found wide use as ropes and lines for mountain climbing as well as boating and sailing because of their high strength and low density. They float on water and have a pleasant feel or hand as a rope or line.<sup>5</sup>

**Table 2** Mechanical properties of selected fibres.

<b>Fibre</b>	<b>E-glass <sup>10</sup></b>	<b>S-glass <sup>10</sup></b>	<b>Carbon <sup>10</sup></b>	<b>Aramide Kevlar 49 <sup>12</sup></b>	<b>PE <sup>12</sup></b>
Density [g.cm <sup>-3</sup> ]	2,5	2,5	1,6	1,5	0,9
Tensile strength [GPa]	3,5	4,7	2-2,5	2,8	3,0
E-Modulus [GPa]	74	87	400-500	130	172

## 2.5 Determination of flexural properties

Three-point bending is used to investigate the flexural behaviour of the test specimens and for determining the flexural strength ( $\sigma_{fM}$ ), flexural modulus ( $E_f$ ) and other aspects of the flexural stress/strain relationship under the conditions defined. It applies to a freely supported beam, loaded at mid-span (three-point loading test).

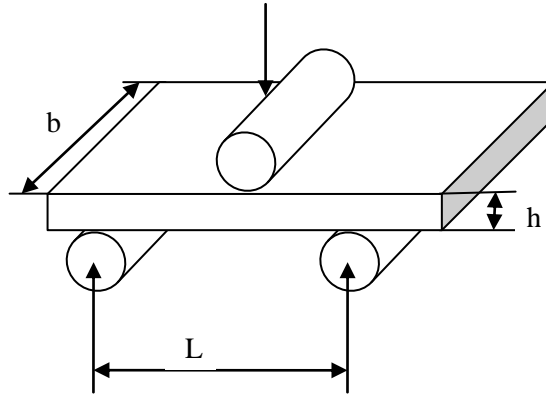
The method is suitable for use with the following range of materials:

- thermoplastics moulding and extrusion materials, including filled and reinforced compounds in addition to unfilled types; rigid thermoplastics sheets;
- thermosetting moulding materials, including filled and reinforced compounds; thermosetting sheets.
- fibre-reinforced thermosetting and thermoplastic composites incorporating unidirectional or non-unidirectional reinforcements such as mat, woven fabrics, woven roving, chopped strands, combination and hybrid reinforcements, roving and milled fibres, sheet made from pre-impregnate materials (prepregs), including filled and reinforced compounds,
- thermotropic liquid-crystal polymers.

Flexural properties can only be used for engineering design purposes for material with linear stress/strain behaviour. For non-linear behaviour, the flexural properties are only nominal. The bending tests should preferentially be used with brittle material, for which tensile tests are difficult.<sup>13</sup>

### 2.5.1 Three-point bending test

The test specimen, supported as a beam, is deflected at a constant rate at the mid-span until the specimen fractures or until the deformation reaches some predetermined value. During this procedure, the force applied to the test specimen is measured.<sup>13</sup> Scheme of a three-point bending is shown in Figure 2.



**Figure 2** Scheme of three-point bending.

### 2.5.2 Calculation

**Flexural strength** – maximum flexural stress sustained by the test specimen during a bending test, it is expressed in megapascals (MPa).

**Flexural stress** – nominal stress of the outer surface of the test specimen at midspan, it is calculated according to equation and is expressed in magapascals (MPa).

$$\sigma_f = \frac{3FL}{2bh^2}$$

where  $F$  is the applied force, in newtons;  $L$  is the span, in millimetres;  $b$  is the width, in millimetres, of specimen and  $h$  is the thickness, in millimetres, of the specimen.

**Flexural modulus** – to determine flexural modulus, calculate the deflection  $s_1$  and  $s_2$  corresponding to the given values of the flexural strain  $\varepsilon_{f1} = 0,0005$  and  $\varepsilon_{f2} = 0,0025$  using the following equation:

$$s_i = \frac{\varepsilon_{fi} L^2}{6h} \quad (i = 1; 2)$$

where  $s_i$  is one of the deflection, in millimetres;  $\varepsilon_{fi}$  is the corresponding flexural strain, whose values  $\varepsilon_{f1}$  and  $\varepsilon_{f2}$  are given above;  $L$  is the span, in millimetres and  $h$  is the thickness, in millimetres, of the specimen.

Calculate the flexural modulus, expressed in megapascals, using the following equation:

$$E_f = \frac{(\sigma_{f2} - \sigma_{f1})}{(\varepsilon_{f2} - \varepsilon_{f1})}$$

where  $\sigma_{f1}$  is the flexural stress, in megapascals, measured at deflection  $s_1$  and  $\sigma_{f2}$  is the flexural stress, in megapascals, measured at deflection  $s_2$ .<sup>13</sup>

## 2.6 Determination of Charpy impact properties

The method is used to investigate the behaviour of specified types of specimen under the impact conditions defined and for estimating the brittleness or toughness of specimens within the limitations inherent in the test conditions. It may also be used for the determination of comparative data from similar of material.

The method is suitable for use with the following range of materials:

- rigid thermoplastic moulding and extrusion materials, including filled and reinforced compounds in addition to unfilled types, rigid thermoplastics sheets,
- rigid thermosetting moulding materials, including filled and reinforced compounds, rigid thermosetting sheets, including laminates,
- fibre-reinforced thermosetting and thermoplastic composites incorporating unidirectional or non-unidirectional reinforcements such as mat, woven fabrics, woven roving, chopped strands, combination and hybrid reinforcements, roving and milled fibres, sheet made from pre-impregnate materials (prepregs), including filled and reinforced compounds,
- thermotropic liquid-crystal polymers.<sup>14</sup>

### 2.6.1 Un-notched Charpy test

The test specimen, supported near its ends as a horizontal beam, is impacted by a single blow of a striker, with the line of impact midway between the supports, and bent at a high, nominally constant velocity.<sup>14</sup>

### 2.6.2 Calculation

**Charpy un-notched impact strength ( $a_{cU}$ )** – impact energy absorbed in breaking an un-notched specimen, referred to the original cross-sectional area of the specimen. It is calculated according to equation and is expressed in kilojoules per square metre.

$$a_{cU} = \frac{E_c}{h \cdot b} \times 10^3$$

where  $E_c$  is corrected energy, in joules, absorbed by breaking the test specimen,  $h$  is the thickness, in millimetres, of the test specimen and  $b$  is the width, in millimetres, of the test specimen.<sup>14</sup>

## 2.7 Dynamic Mechanical Analysis (DMA)

Dynamic mechanical methods characterise the viscoelastic properties of materials as function of both frequency of applied oscillatory stress (or strain) and temperature.<sup>15</sup>

DMA is a technique where a small deformation is applied to a sample in a cyclic manner. This allows the materials response to stress, temperature, frequency and other values to be studied. The term is also used to refer to the analyzer that performs the test.

Dynamic Mechanical Analyzer deforms a sample mechanically and after that it measures the sample response. When a force is applied on a material it suffers a change in shape, that is, it deforms. The deformation can be applied sinusoidally, in a constant (or step) fashion, or under a fixed rate. The response to the deformation can be monitored as a function of temperature or time. A force to resist the deformation is also set up simultaneously within the material and it increases as the deformation continues. If the material is unable to put up full resistance to external action, the process of deformation continues until failure takes place. The deformation of a body under external action and resistance to deform are referred to by strain and stress respectively.

DMA measurements explain how a material behaves at the moment and future.

Dynamic Mechanical Analyzer is useful for these tests: mechanical properties, morphology of polymers, loss factor ( $\tan \delta$ ), loss angle ( $\delta$ ), impact resistance, dynamic viscosity, curing kinetics, correlation with materials formulation, ageing, damping, glass transition temperature ( $T_g$ ), industrial products stiffness, rheological properties, secondary transitions, specimen stiffness, young Modulus, thermal stability, creep behaviour, tension test, stress-strain.<sup>16</sup>

### 2.7.1 DMA Modulus parameters

**The Modulus (G)** - Modulus is an intrinsic material property (does not change with material size or shape), defined as the ratio of stress ( $\sigma$ )/strain ( $\varepsilon$ ) in a body under a particular mode of deformation (such as shear, bending, torsion, etc.). Thus modulus is a measure of materials overall resistance to deformation. The modulus is the ratio of a component of stress to a component of strain, in rheology.

$$G = \frac{\sigma}{\varepsilon}$$

**The Elastic (Storage) Modulus ( $G'$ )** - The storage modulus is measure of elasticity of material. It is also called “the ability of the material to store energy”. It is equivalent to the ability of a sample to store energy, i.e. its elasticity. Energy storage occurs as molecules are distorted from their equilibrium position by application of a stress. Removal of the stress results in a return to equilibrium position of the molecular segments.

$$G' = G \cdot \cos \delta$$

**The Viscous (Loss) Modulus ( $G''$ )** - Loss modulus represents the capability of a material to dissipate energy (mechanical, acoustic) as heat, owing to viscous motions inside the material itself. It is limited to the molecular motion within the sample that dissipates energy as heat. In rheology, loss modulus is the imaginary part of the complex modulus.

$$G'' = G \cdot \sin \delta$$

**Tan Delta** - It is measure of material damping - such as vibration or sound damping. Damping refers to damping the loss of mechanical energy as the amplitude of motion gradually decreases. It means also the ability of a material to dissipate mechanical energy by converting it into heat. Tan Delta is a useful index of material viscoelasticity since it is a ratio of viscous and elastic moduli.  $\tan \delta$  is an important indication of viscoelasticity of materials, it is independent from the shape and dimension of samples and it is dimensional.<sup>16</sup>

$$\tan \delta = \frac{G'}{G''}$$

## 2.8 Scanning electron microscopy (SEM)

SEM can provide information on surface topography, crystalline structure, chemical composition and electrical behaviour of the top 1  $\mu\text{m}$  or so of specimen. Various specialised stages can be attached to enable behaviour under various conditions to be examined.

SEM has many advantages. SEM benefits from a large depth of field so most of the specimen surface is simultaneously in focus whatever the surface roughness. There is much higher magnifications can be achieved (up to 1,000,000x), with an ultimate resolution of 1 nm. SEM has the possibility of getting more information than just the



surface topography, e.g. crystal structure, chemical composition and electrical properties.

In an SEM the incident electrons (from an electron gun) typically have energies of 2-40 keV. There are three types of electron gun in general use:

- The most common is the tungsten hairpin filament which is heated (by passing a current through it) to over 2500°C to produce thermal emission of electrons from its tip.
- Lanthanum hexaboride (LaB<sub>6</sub>) filaments also work by thermionic emission, but advantages include a larger maximum beam current, i.e. a “brighter” beam (because LaB<sub>6</sub> has a lower work function than tungsten) and a longer working lifetime.
- Field emission guns (not heated, so also known as “cold cathode” emitters) provide the brightest beam with very small deviations in electron energy by applying a very high electric field to a finely pointed tip until quantum mechanical tunnelling of electrons occurs.<sup>17</sup>

### **2.8.1 Principle of measurement**

The electron gun at the top of the column produces an electron beam that is focused into a fine spot as small as 1 nm in diameter on the specimen surface. This beam is scanned in a rectangular raster over the specimen and the intensities of various signals created by interactions between the beam electrons and the specimen are measured and stored in computer memory. The range of accelerating voltages is from 50 to 30 000 volts. There is no necessary to penetrate the specimen.<sup>18</sup>

### **The interactions between the beam electrons and sample atoms**

The specimen itself emits secondary electrons. Some of the primary electrons are reflected backscattered electrons (BSE). These backscattered electrons can also cause the emission of secondary electrons as they travel through the sample and exit the sample surface. The specimen emits X-rays. Electrons are absorbed by the specimen. The specimen sometimes emits photons of visible light (cathodoluminescence).<sup>18</sup>

**Electron detection**

Detectors for backscattered electrons and secondary electrons are usually either a scintillation detector or a solid-state detector. In the scintillator case, electrons strike a fluorescent screen, which emits light that is amplified and converted into an electrical signal by a photomultiplier tube. The solid-state detector works by amplifying the minute signal produced by the incoming electrons in a semiconductor device. A third type of detector monitors the net current absorbed by the specimen (beam current less secondary and backscattered electron emission) or the current induced in a semiconductor junction by the incoming beam electron. These absorbed current and EBIC (electron beam induced current) measurements permit the study of dynamic electrical phenomena in electronic devices.<sup>18</sup>

### **3 EXPERIMENTAL PART**

#### **3.1 Materials and samples preparation**

##### **3.1.1 Materials**

- Lukopren, type N1 522 – two-component silicon rubber for preparation of the form for the samples (Lučební závody Kolín, The Czech Republic)
- Duracryl® Plus (PMMA resin) – self-curing denture base resin (SpofaDental a.s., The Czech Republic)
  - powder: polymethylmethacrylate (PMMA) (>95%), dibenzoyl- peroxid (DBP) (<5%)
  - liquid: methylmethacrylate (80-90%), dimethyl-paratoluidine (<5%), ethylene glycoldimethacrylate (3-10%)
- Dentapreg MESH (Glass reinforcing mesh) – glass fibre reinforced heat curing composite for reinforcing and repair of removable dentures and acrylic devices (ADM, a.s., The Czech Republic)
- MESH Strengtheners (Metal reinforcing mesh)– stainless steel (MEDIN, a.s., The Czech Republic)

##### **3.1.2 Resin preparation**

The resin was prepared from Duracryl® Plus powder and liquid. It was mixed in the volumetric ratio 1,5 : 1 (1,5 parts of powder, 1 part of liquid). The powder was poured into the liquid. The mixture was mixed up for 10 seconds at laboratory temperature.

##### **3.1.3 Samples preparation**

For mechanical testing three sets of samples were prepared: Duracryl Plus (cross-linked PMMA), PMMA resin reinforced with Dentapreg Mesh (multidirectional glass fibre reinforcement) and PMMA resin reinforced with stainless steel Mesh Strengtheners (multidirectional metal reinforcement). Each set consists of 12 samples. Samples from clear resin were used as a reference. Test samples were prepared in the

form of rectangular with dimensions of 15 mm (b) × 3 mm (h) × 60 mm (l). Dimensions of the samples were in accordance with ČSN EN ISO 178. Stainless steel or fibre composite reinforcement was placed in the bottom of the sample. Reinforcement size was similar to the sample 60 mm (l) × 15 mm (b). For DMA analysis the test specimens for each type of material with dimensions of 10 mm (b) × 3 mm (h) × 50 mm (l) were prepared. For DMA analysis 2 samples of each type of material were measured.

Preparation of the test samples were carried out in several steps. At first, the reinforcement, with exception of the samples from pure resin, was place on the bottom of Lukopren form. Then the resin from Duracryl Plus was prepared. Subsequently, the reinforcement was poured with the resin in the form and let for 30 minutes at laboratory temperature. After this period, the specimens were cured and removed from the form. Finally, the specimens were grinded off to required dimensions using disc grinder equipment.

### **3.2 Measurement of flexural properties**

Flexural properties of the prepared samples were measured by static three-point bending test using an universal testing machine Instron 3366 under laboratory conditions. The sample was supported by two supports with a span (L) 48 mm and loaded by loading edge in the middle of sample at a constant speed (v) 1 mm/min. Radius of the supports was 2 mm, force transducer 5 kN. Test was carried out until the break of the sample or until the loading force decrease about 50% from its maximum.

Obtained values of maximum loading force and samples deflection were used for calculation of  $\sigma_{fM}$ ,  $E_f$  and  $a_c$  utilizing Bluehill software.

### **3.3 Determination of Charpy un-notched impact strength**

Impact hammer (Resil Impact Junior, DAS 4000) was used for measuring mechanical properties of the samples at dynamic testing conditions. The hammer impact velocity was 1,2 m/s at initial angle 40°. The nominal pendulum energy was 7,5 J and the support span was 48 mm. Measured values of acting force and sample deflection were collected with DAS 4000 external unit and impact energy ( $a_{cU}$ ),  $\sigma_{fM}$  and  $E_f$  were calculated utilizing CEAST DAS4WIN Extender software according to equations:

$$a_{cU} = \frac{E_c}{h \cdot b} \times 10^3 \text{ [kJ/m}^2\text{]},$$

$$E_f = \frac{F \cdot l^3}{4 \cdot Y \cdot b \cdot h^3} \text{ [MPa]},$$

where F is the applied force in newtons, l is the span in millimetres, Y is deflection of the sample in millimetres, b is the width and h is the thickness of the specimen in millimetres, respectively. CEAST DASFILTERING software was used for smoothing of the force-time curves obtained during the dynamic tests.

### 3.4 DMA

The specimens at frequency sweep mode were loaded with the force 0,5 N at strain  $5 \times 10^{-3}$  % and in the frequency range 0,1-100 Hz. During the thermal sweep the temperature was increasing from 35 to 140°C at speed 3°C/min at frequency 1 Hz. From obtained data loss modulus, storage modulus and tan delta were calculated utilizing Trios software.

### 3.5 SEM

Fracture surfaces of samples were examined on a scanning electron microscope Zeiss EVO LS10. From each set was measured one sample. Samples were coated with a conducting layer of Au-Pd on Polaron Range – Sputter Coater. Thickness of the surface layer was in the range 7-10 nm, current 12 mA, voltage 1,5 kV and time of deposition 60 s.

For a sample reinforced with Dentapreg Mesh the number of fibres in the bundle was calculated.

### 3.6 Weight and volume fraction of the reinforcement

Samples were burned in furnace for 30 minutes at 700 °C. Reinforcement weight and volume fraction of reinforced samples were calculated according to equations:

$$w_f = \frac{m_f}{m_{total}} \times 100 [\%],$$

$$\varphi_f = \frac{V_f}{V_{total}} \times 100 [\%]$$

where  $m_f$  is the weight of reinforcement (g),  $m_{total}$  is the weight of the sample (g),  $V_f$  is volume of reinforcement ( $cm^3$ ) and  $V_{total}$  is volume of the sample ( $cm^3$ ). Three samples of each type were measured and the results were averaged. Density of stainless steel is 7,70 g/ $cm^3$ .

## 4 RESULTS AND DISCUSSION

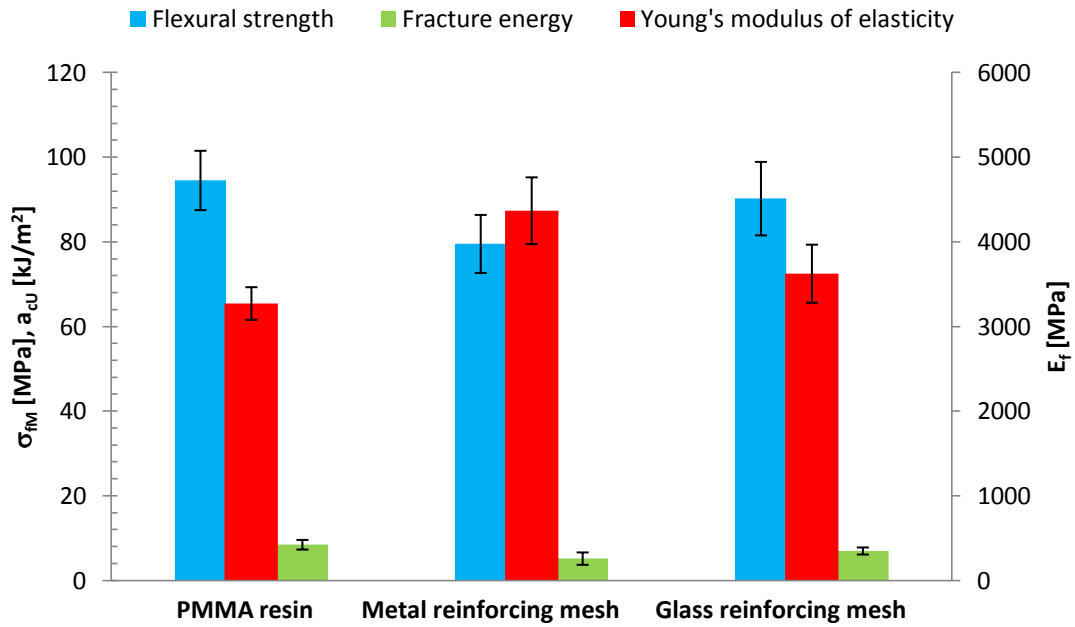
Three sets of samples were tested for flexural strength. The resulting values for each set are summarized in tables and graphs. Location of reinforcement in the bottom part of the sample played an important role, because during the three-point bending test this part is exposed to the maximal tensile force instead of the upper part of the sample where the compression occurs. Reinforcement should be uniformly located on the bottom side of the sample. Due to the liquid resin was difficult to ensure the precise placement of the reinforcement in the sample.

### 4.1 Static three-point bending test

The average values of calculated maximal flexural strength and Young's modulus of elasticity and fracture energy including standard deviations are shown in Table 3 and Figure 3.

**Table 3** Average values of maximal flexural strength, Young modulus of elasticity and fracture energy including standard deviations of measured samples, static three-point bending test

	$\sigma_{fM}$ [MPa]	SD [MPa]	$E_f$ [MPa]	SD [MPa]	$a_{cU}$ [kJ/m <sup>2</sup> ]	SD [kJ/m <sup>2</sup> ]
<b>PMMA resin</b>	95	7	3273	192	8,4	1,1
<b>Metal reinforcing mesh</b>	80	7	4369	395	5,2	1,5
<b>Glass reinforcing mesh</b>	90	9	3625	343	7,0	0,8



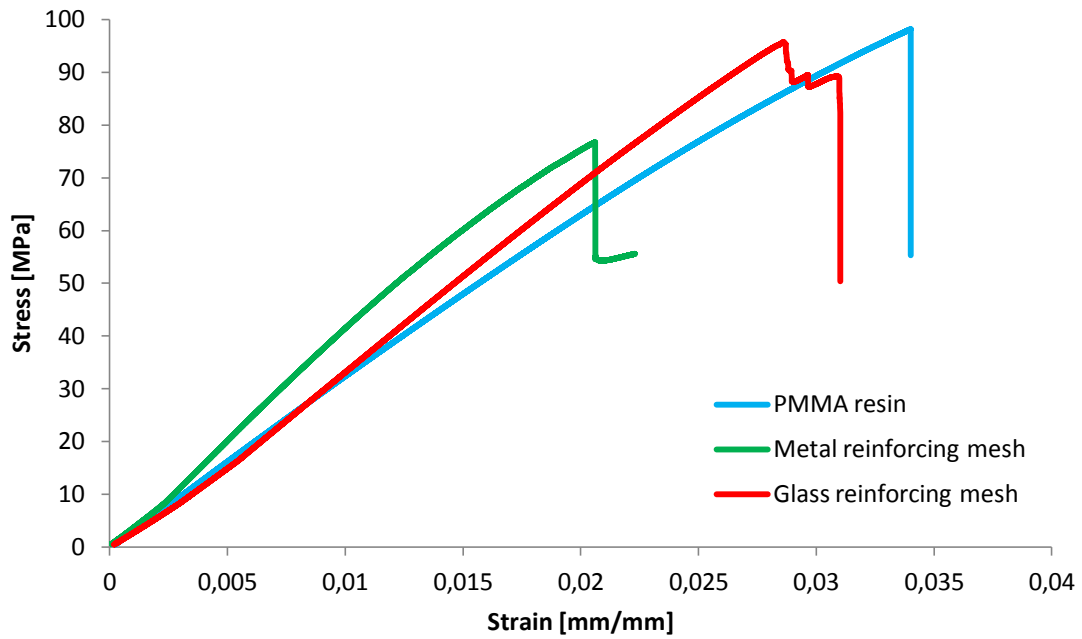
**Figure 3** Average values of maximal flexural strength, Young modulus of elasticity and fracture energy including standard deviations of measured samples, static three-point test

Flexural strength of PMMA resin was 95 MPa, Metal reinforcing mesh had flexural strength 80 MPa and Glass reinforcing mesh 90 MPa. Young's modulus of elasticity reached 3272 MPa for PMMA resin, for Metal reinforcing mesh it was 4369 MPa and for Glass reinforcing mesh it was calculated to 3625 MPa. Fracture energy of PMMA resin was calculated to 8,4 kJ/m<sup>2</sup>, Metal reinforcing mesh reached 5,2 kJ/m<sup>2</sup> and Glass reinforcing had fracture energy 7,0 kJ/m<sup>2</sup>.

The calculated values of  $\sigma_{fm}$  are practically the same for unreinforced and reinforced materials. These results signalized that during the static loading of the samples the reinforcement does not play an important role in comparison with unreinforced resin. The same trend is apparent from  $E_f$ , where the modulus is increasing in the row PMMA resin – Glass reinforcing mesh – Metal reinforcing mesh, however this increasing is not so significant as was expected. It should be consequence of the relatively small amount of the reinforcement in the test samples and the weak or poor adhesion between the stainless steel or glass fibre reinforcement and PMMA resin. The fracture energy is increasing in the row Metal reinforcing mesh – Glass reinforcing mesh – PMMA resin. This trend signalizes that the metal reinforcement exhibits very poor adhesion with PMMA matrix and that the fracture is brittle in all material cases.



The fracture energy effect of glass fibre reinforcement is not apparent in comparison with pure PMMA matrix at low speed loading. The characteristic stress – strain curves are shown in Fig 4.



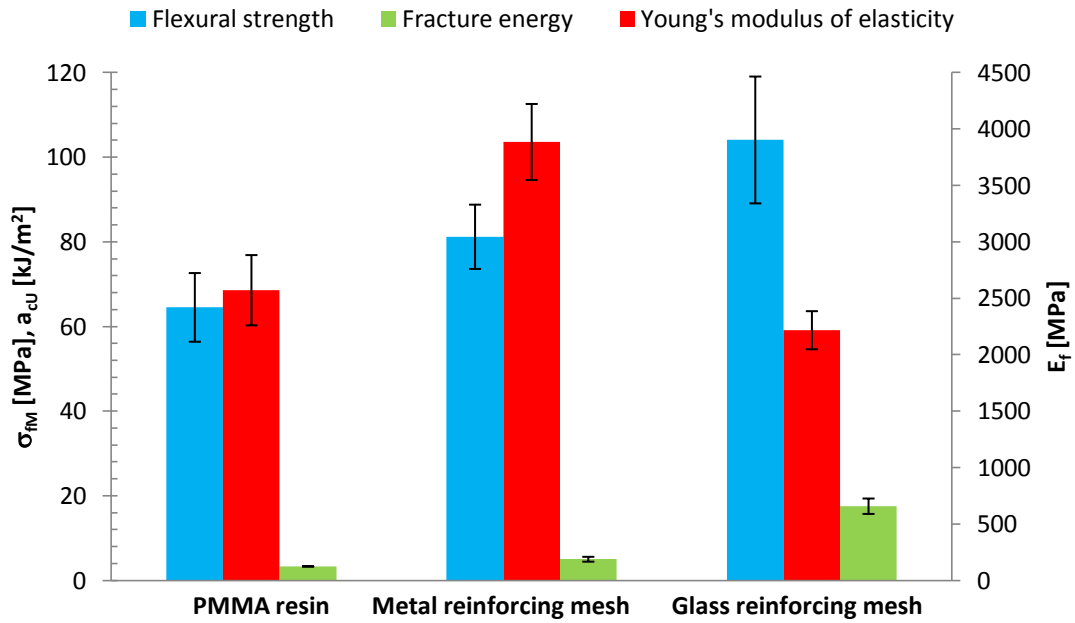
**Figure 4** Characteristic stress – strain curves for tested samples

## 4.2 Charpy instrumented impact test

Charpy instrumented impact un-notched test was used to measure the materials properties under dynamic test conditions. According to equations (2.5.2, 2.6.2, 3.3) values of  $a_{cU}$ ,  $E_f$  and  $\sigma_{fM}$  were calculated. Calculated values of  $a_{cU}$ ,  $E_f$  and  $\sigma_{fM}$  including standard deviations are summarized in Table 4 and Figure 5.

**Table 4** Average values of maximal flexural strength, Young's modulus of elasticity and fracture energy including standard deviations of measured samples, dynamic Charpy un-notched impact test

	$\sigma_{fM}$ [MPa]	SD [MPa]	$E_f$ [MPa]	SD [MPa]	$a_{cU}$ [kJ/m <sup>2</sup> ]	SD [kJ/m <sup>2</sup> ]
<b>PMMA resin</b>	65	8	2572	311	3,3	0,0
<b>Metal reinforcing mesh</b>	81	8	3886	337	5,0	0,6
<b>Glass reinforcing mesh</b>	104	15	2218	169	17,6	1,8

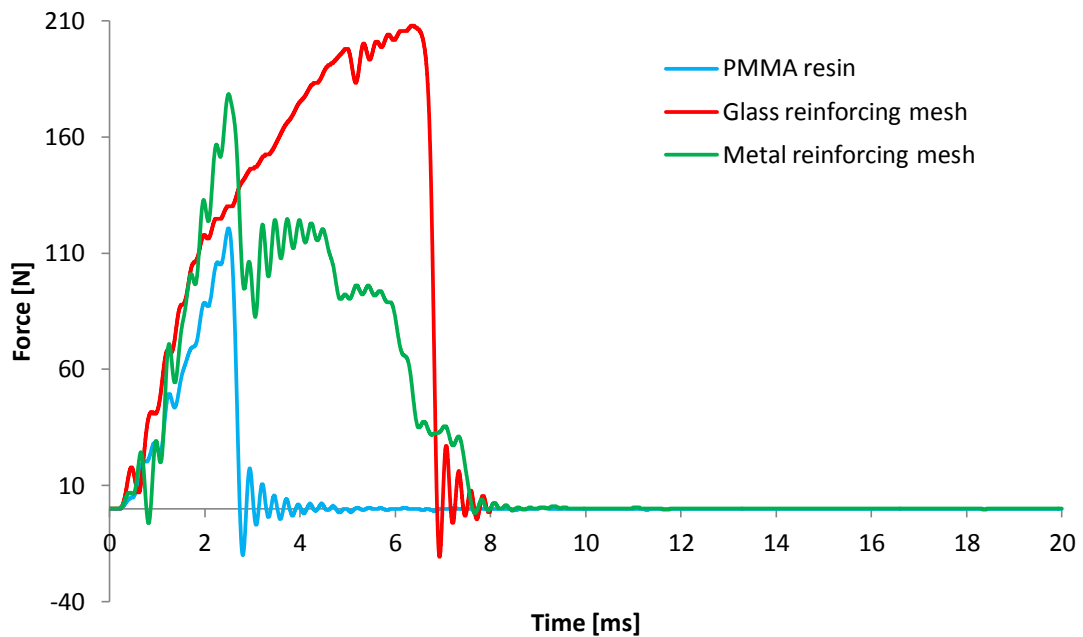


**Figure 5** Average values of maximal flexural strength, Young's modulus of elasticity and fracture energy including standard deviations of measured samples, static dynamic Charpy impact test

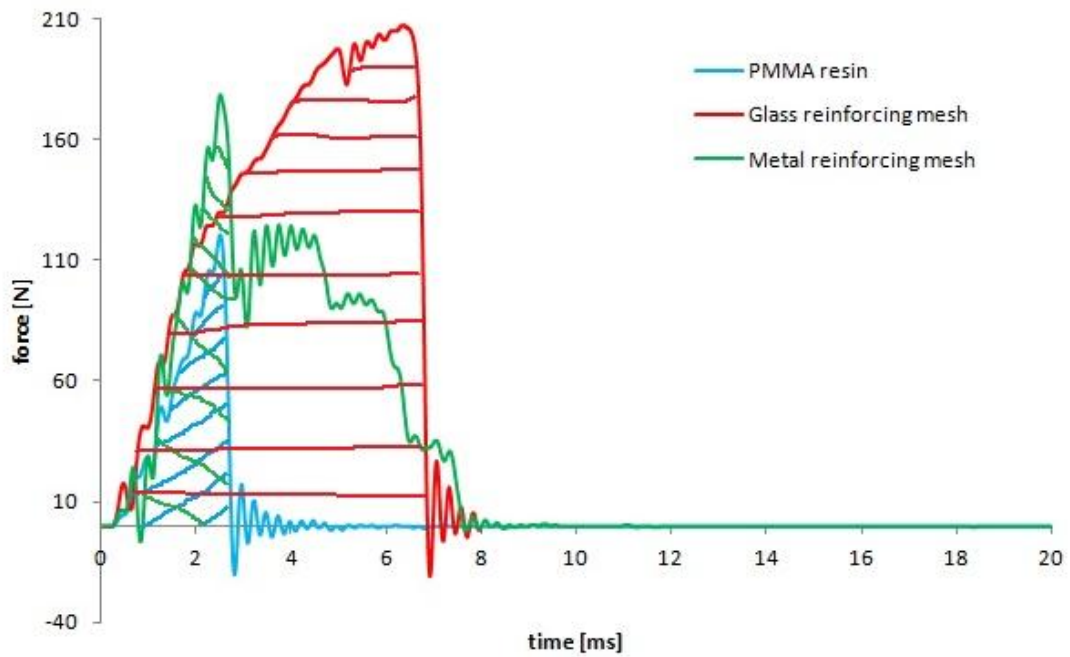
Flexural strength of PMMA resin was 65 MPa, Metal reinforcing mesh had flexural strength 81 MPa and Glass reinforcing mesh 104 MPa. Young's modulus of elasticity reached 2572 MPa for PMMA resin, for Metal reinforcing mesh it was 3886 MPa and for Glass reinforcing mesh it was calculated to 2218 MPa. Fracture energy of PMMA resin was calculated to 3,3 kJ/m<sup>2</sup>, Metal reinforcing mesh reached 5,0 kJ/m<sup>2</sup> and Glass reinforcing had fracture energy 17,6 kJ/m<sup>2</sup>.

The calculated values of  $\sigma_{FM}$  shows 20 % increase for PMMA resin compared with Metal reinforcing mesh. The same trend shows Glass reinforcing mesh compared with Metal reinforcing mesh. The flexural strength is increasing in the row PMMA resin – Metal reinforcing mesh – Glass reinforcing mesh. The row shows effectiveness of reinforcement. These results signalized that during the dynamic loading of the samples with Glass reinforcing mesh is transmitted stress the best of the used reinforcements. The Young's modulus of elasticity is increasing in the row Glass reinforcing mesh – PMMA resin – Metal reinforcing mesh. The Metal reinforcing mesh contributes to increase the Young's modulus of elasticity compared with Glass reinforcing mesh. Glass reinforcing mesh shows a decrease of Young's modulus of elasticity compared with PMMA resin. The fracture energy is increasing in the row PMMA resin – Metal

reinforcing mesh – Glass reinforcing mesh. The fracture energy of Glass reinforcing mesh is almost 6 times greater than for PMMA resin and 3 times greater than for Metal reinforcing mesh. These results are the consequence of the very poor adhesion between the Metal reinforcing mesh and PMMA matrix as is apparent from the SEM pictures (see Fig. 11). On the other hand a certain adhesion is apparent between the Glass reinforcing mesh and PMMA matrix (see Fig. 15) which contributes to higher impact resistance of this material in comparison with Metal reinforcing mesh. The characteristic force – time curves with trend lines are shown in Figure 6. Figure 6a shows shaded areas used for calculations of fracture energy.

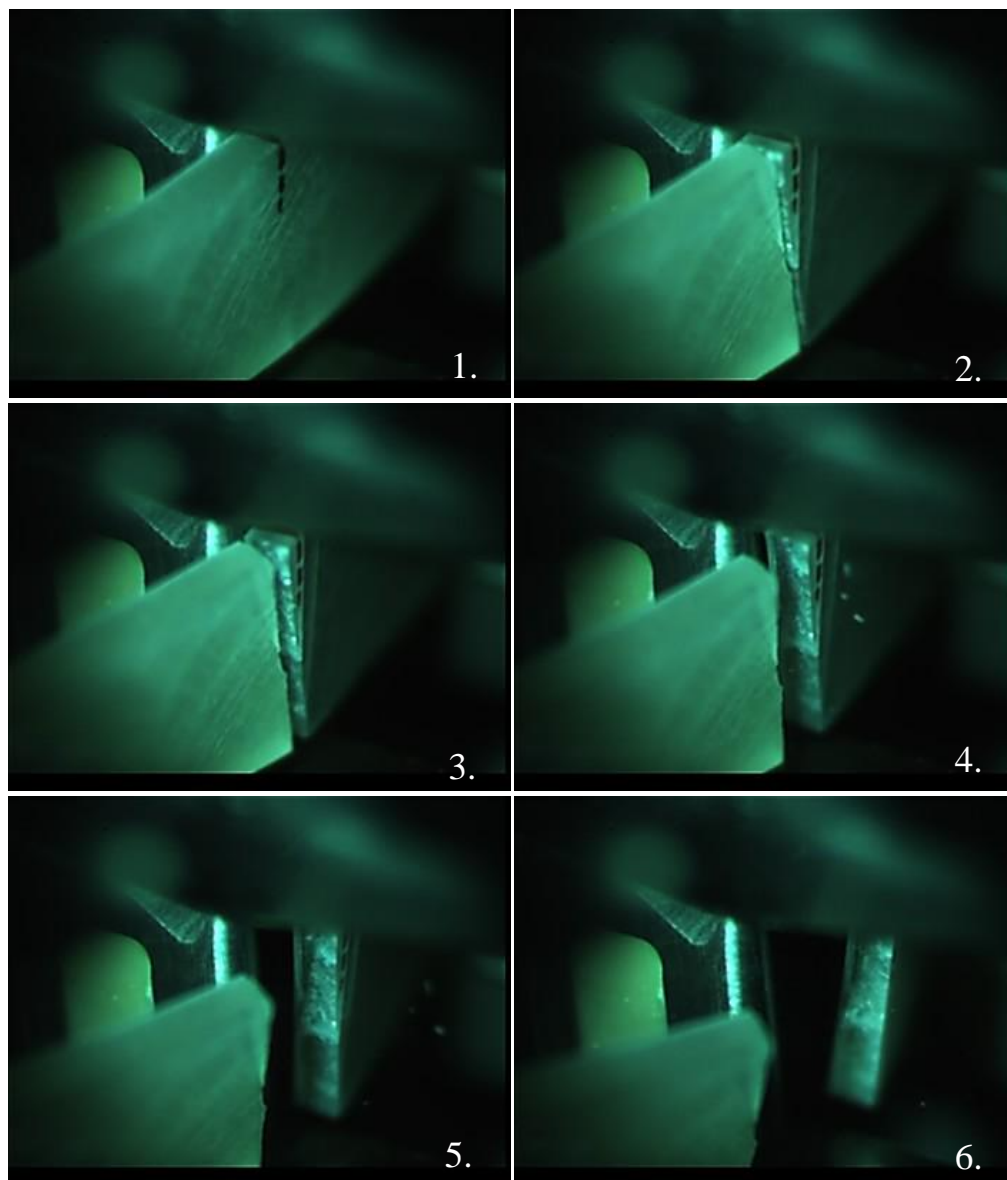


**Figure 6** Characteristic force – time curves with trend lines for tested samples

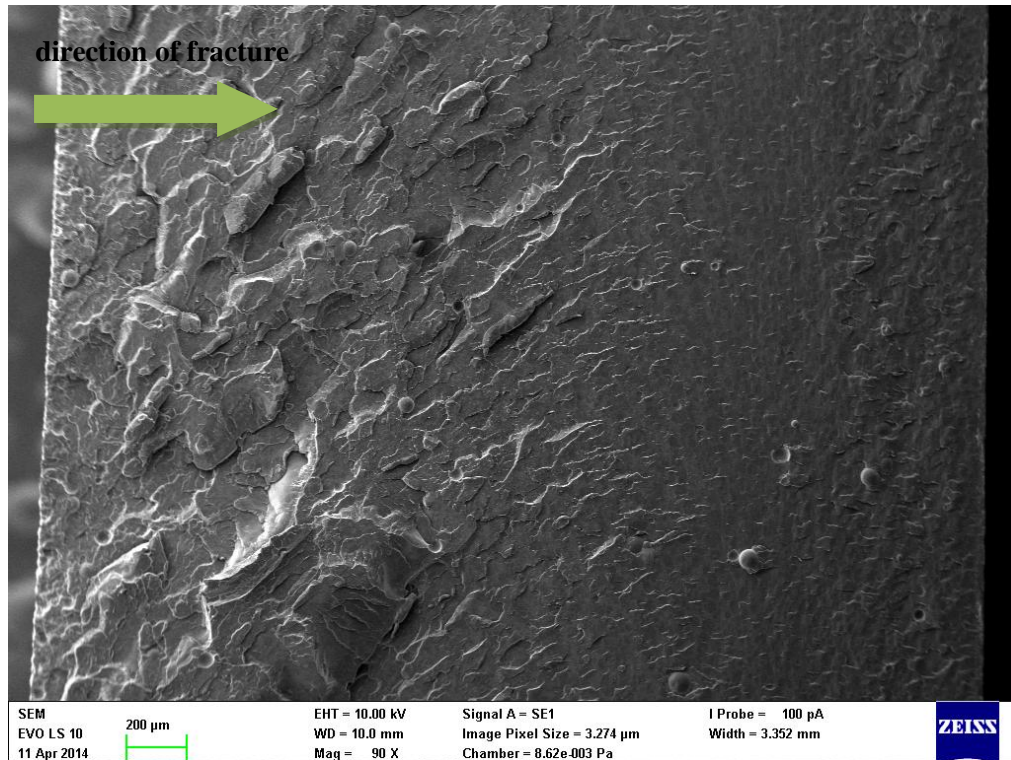


**Figure 6a** Characteristic force – time curves for tested samples, shaded areas for calculation

Fracture of the material is shown in the direction of the arrow in SEM images. The fracture is brittle in all material cases. An images sequence from high-speed camera shows fracture of matrix (Figure 7). Brittle fracture and direction of fracture for PMMA resin sample is shown in Figure 8.

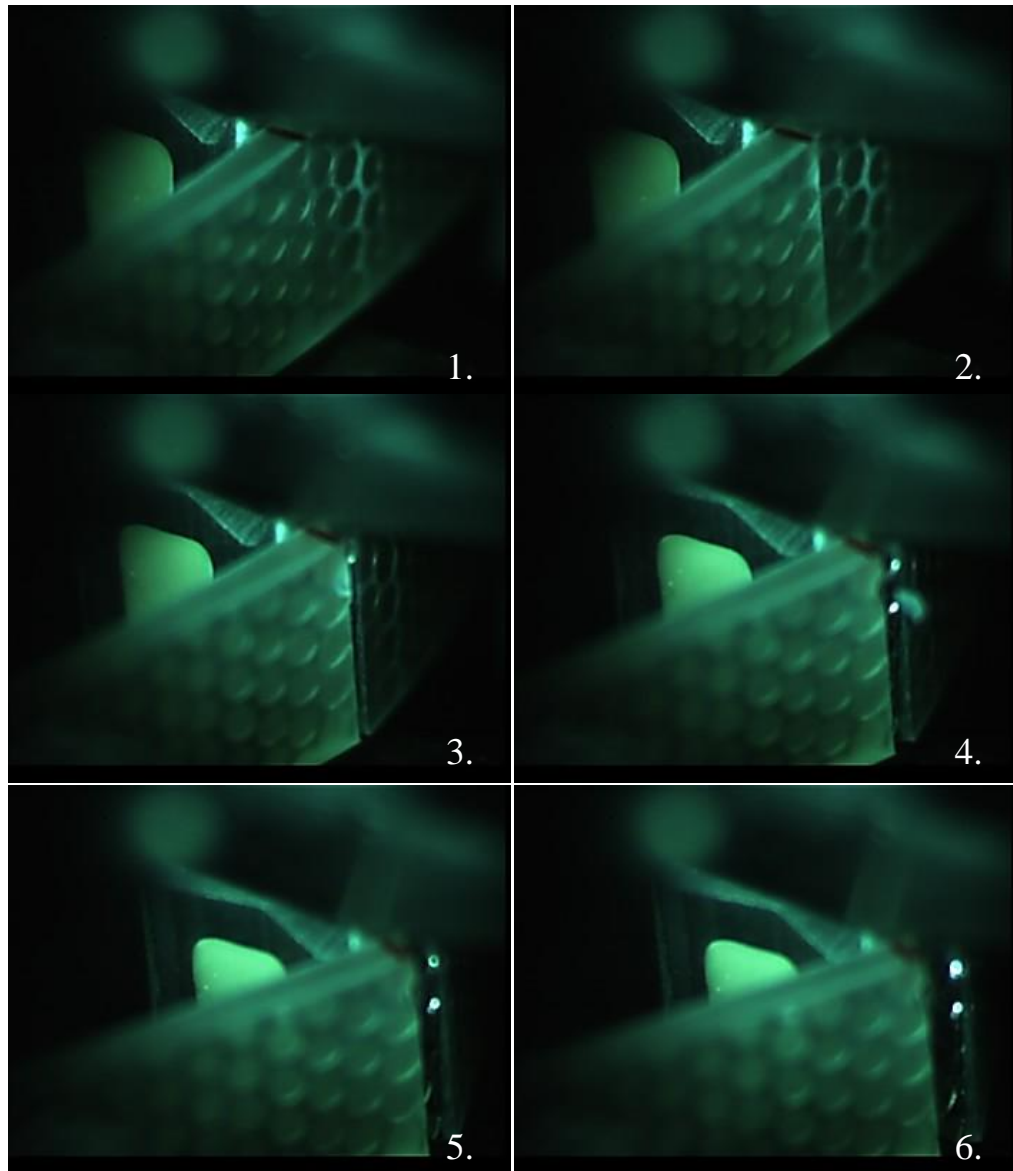


**Figure 7** Fracture images of PMMA resin sample, high-speed camera



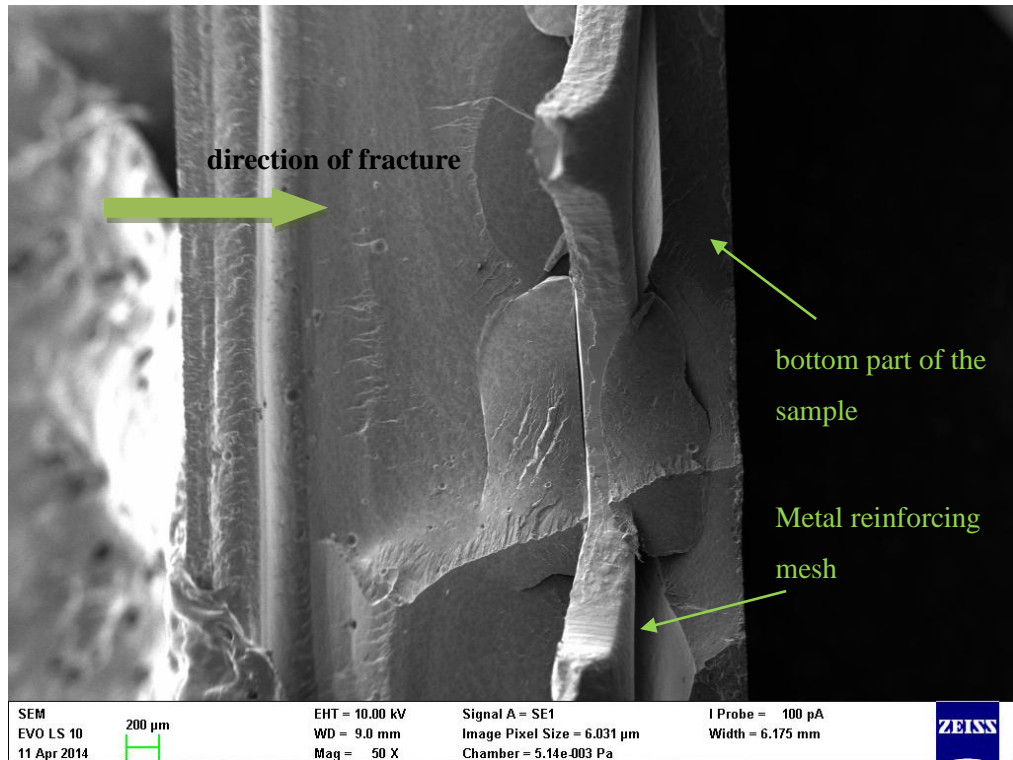
**Figure 8** SEM image of PMMA resin sample, magnification 90×

In Figure 9 are shown images of Metal reinforcing mesh sample from high-speed camera. There are shown individual steps of sample fracture. At first, brittle break of the matrix occurred. The both parts of the sample were hold together with the Metal reinforcing mesh which was consequently fractured too. Brittle fracture, direction of fracture and location of reinforcement for Metal reinforcing mesh sample is shown in Figure 10. The Figure 11 shows gap, which was created as a result of polymerization shrinkage, Figure 12 shows poor adhesion of reinforcement with PMMA matrix. Poor adhesion reduces the mechanical properties of sample. In Figure 13 is shown described SEM image width gap between PMMA matrix and the reinforcement.

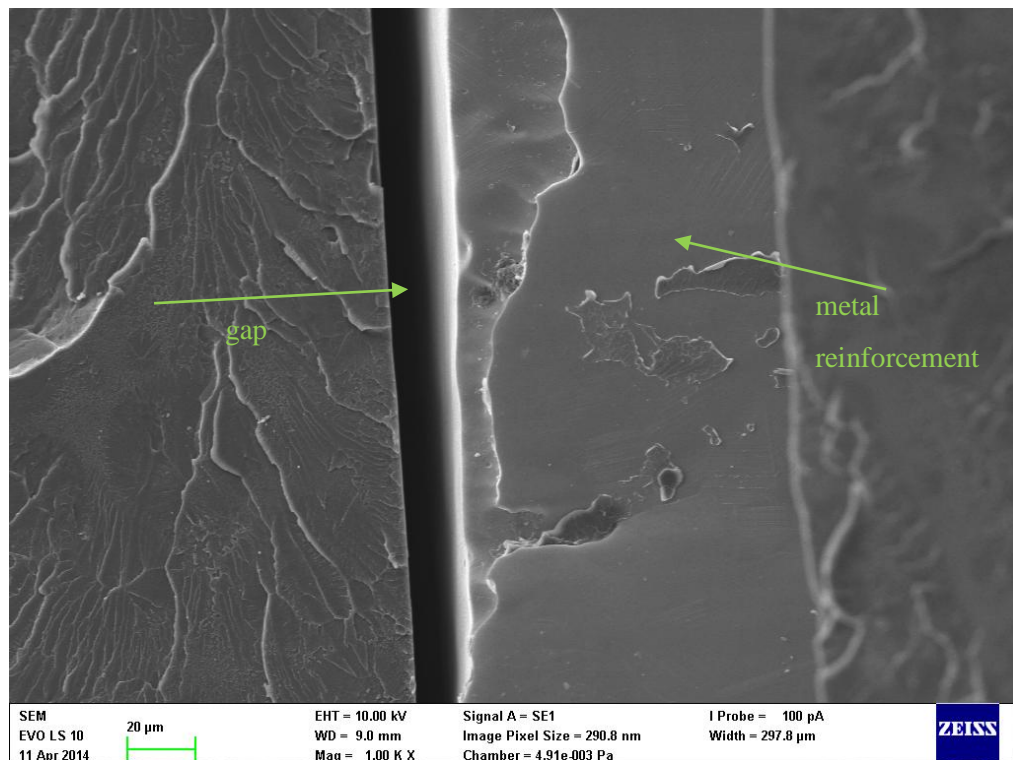


**Figure 9** Fracture images of Metal reinforcing mesh sample, high-speed camera



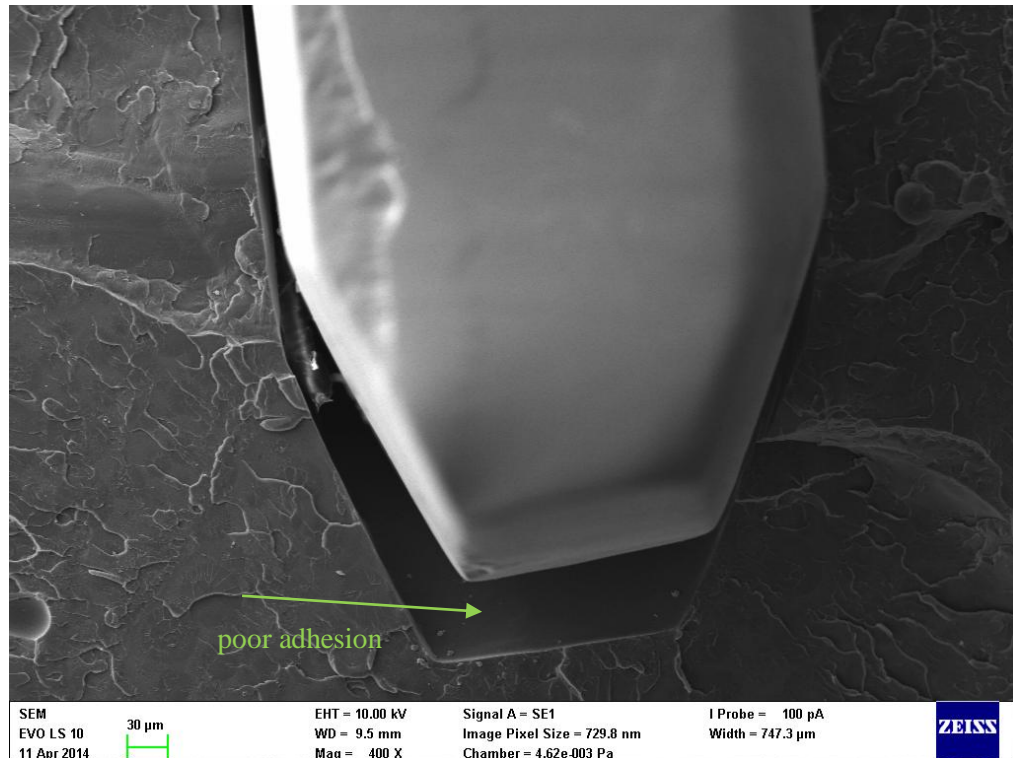


**Figure 10** SEM image of Metal reinforcing mesh sample, magnification 50×



**Figure 11** SEM image of Metal reinforcing mesh sample, gap and magnification 1000×



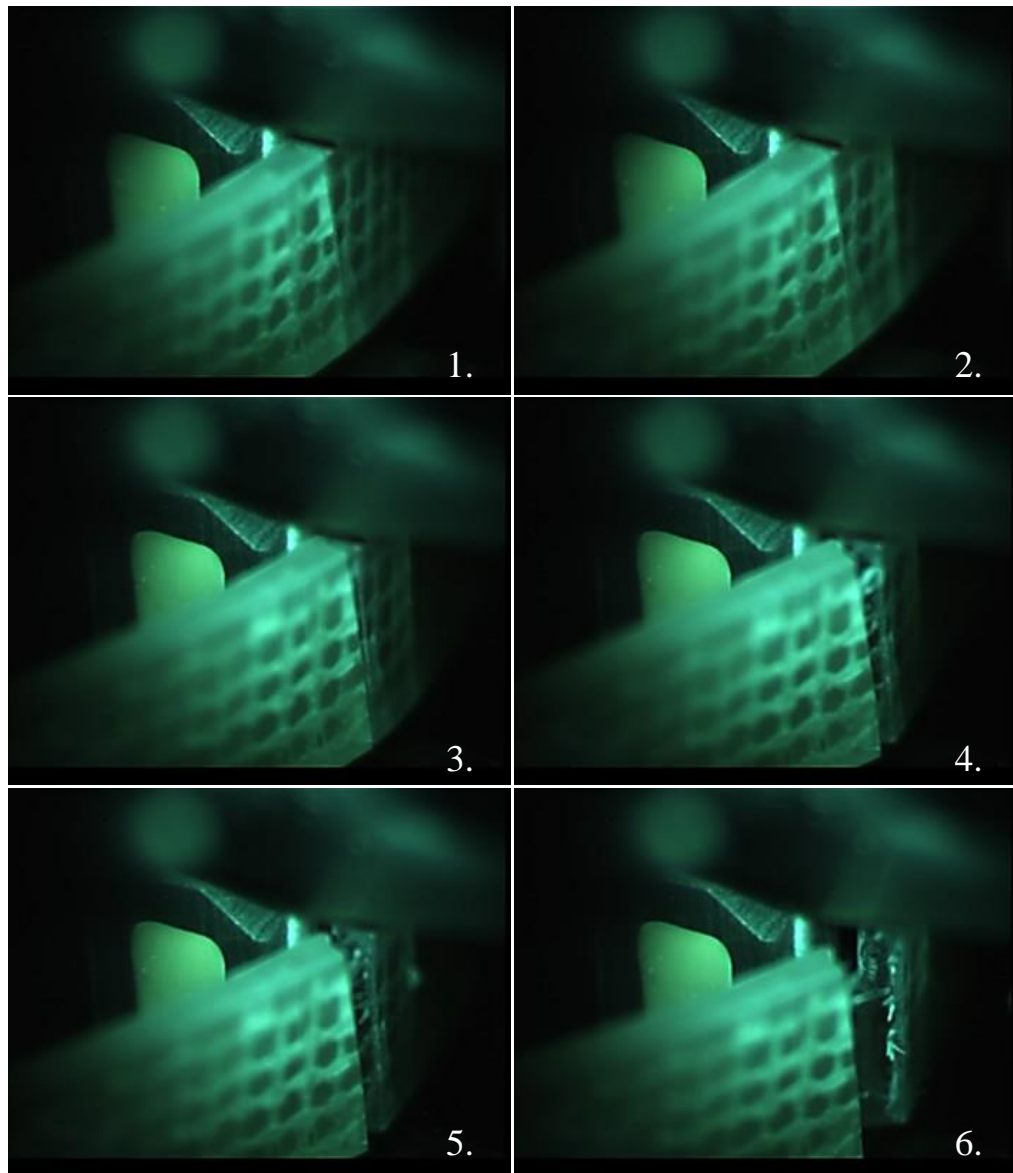


**Figure 12** SEM image of Metal reinforcing mesh sample, edge of reinforcement, poor adhesion, magnification 400×

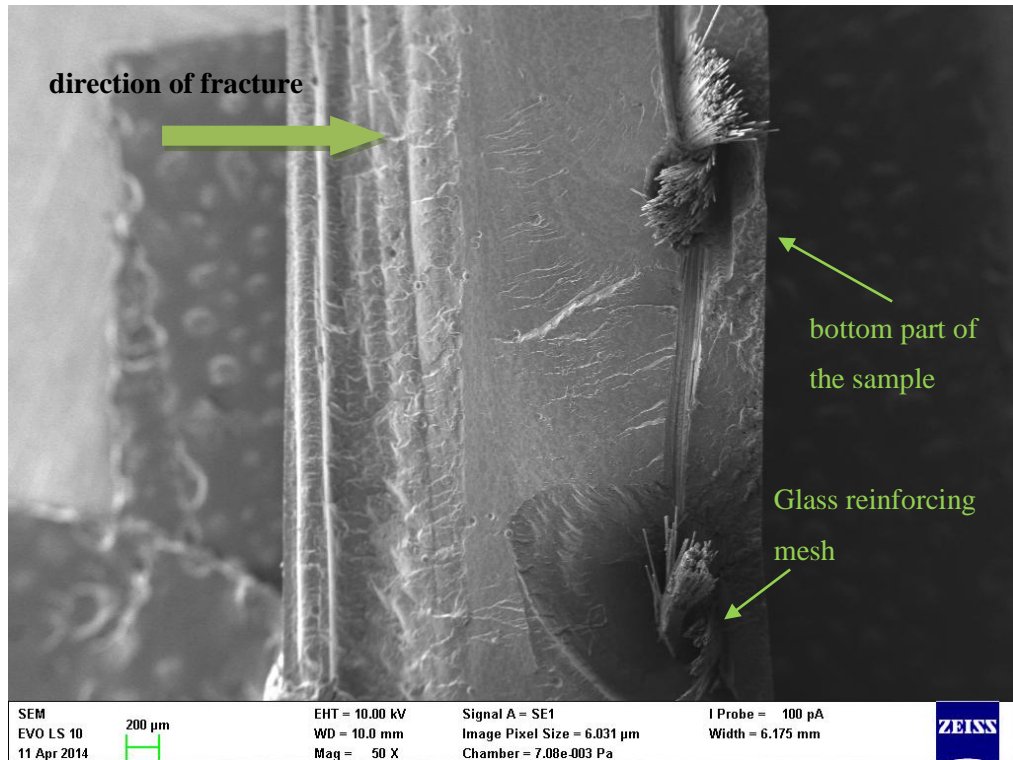
In Figure 14 are shown images of Glass reinforcing mesh sample from high-speed camera. There are shown steps of sample fracture. At first, the brittle break of matrix occurred, and then the reinforcement transverse part of stress and at last break of reinforcement together with matrix occurred. Brittle fracture, direction of fracture and location of reinforcement for Glass reinforcing mesh sample is shown in Figure 15. Figure 16 shows fibres protruding from matrix and better adhesion (compared with Metal reinforcing mesh) of fibres with PMMA matrix. For this sample was calculated average number of fibres in the bunch (Table 5). Value was calculated from the described SEM images (Appendix, Figure 17,18).

**Table 5** Calculate value of average number of fibres in bunch

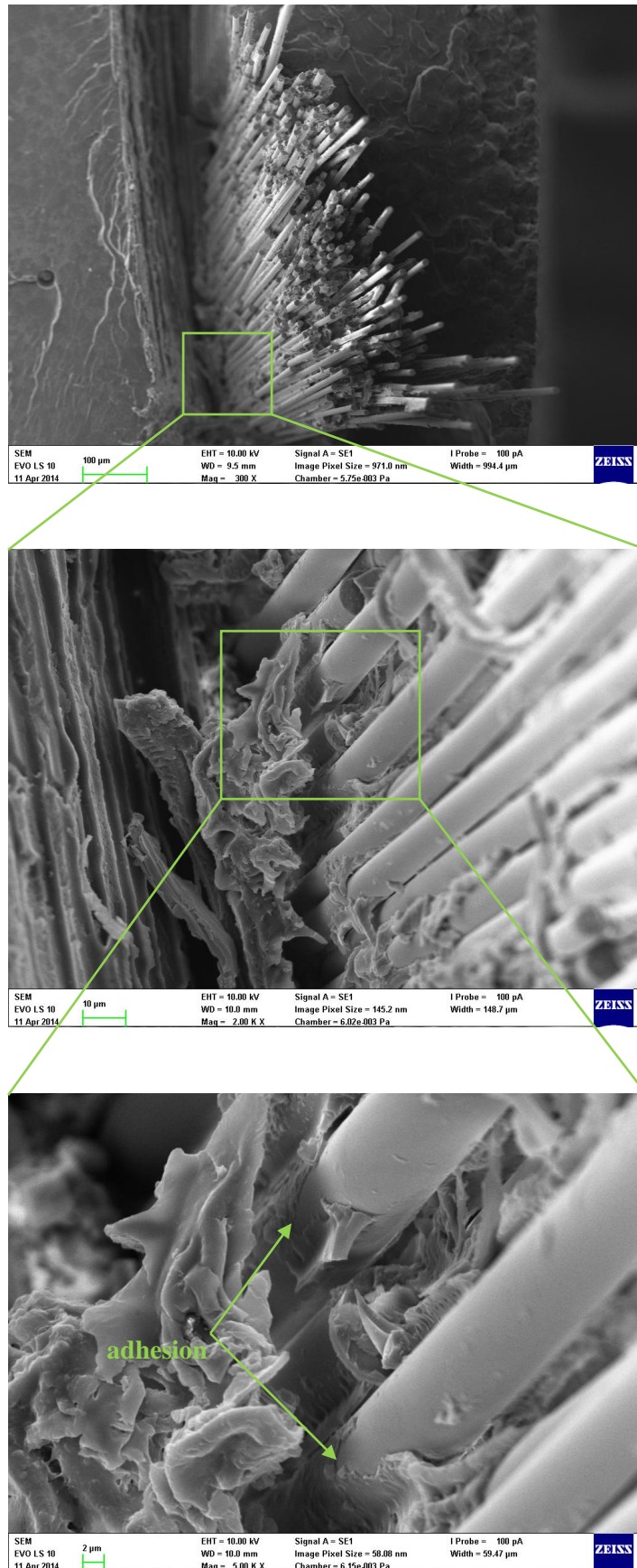
average number [fibres]	1174
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**Figure 14** Fracture images of Glass reinforcing mesh sample, high-speed camera



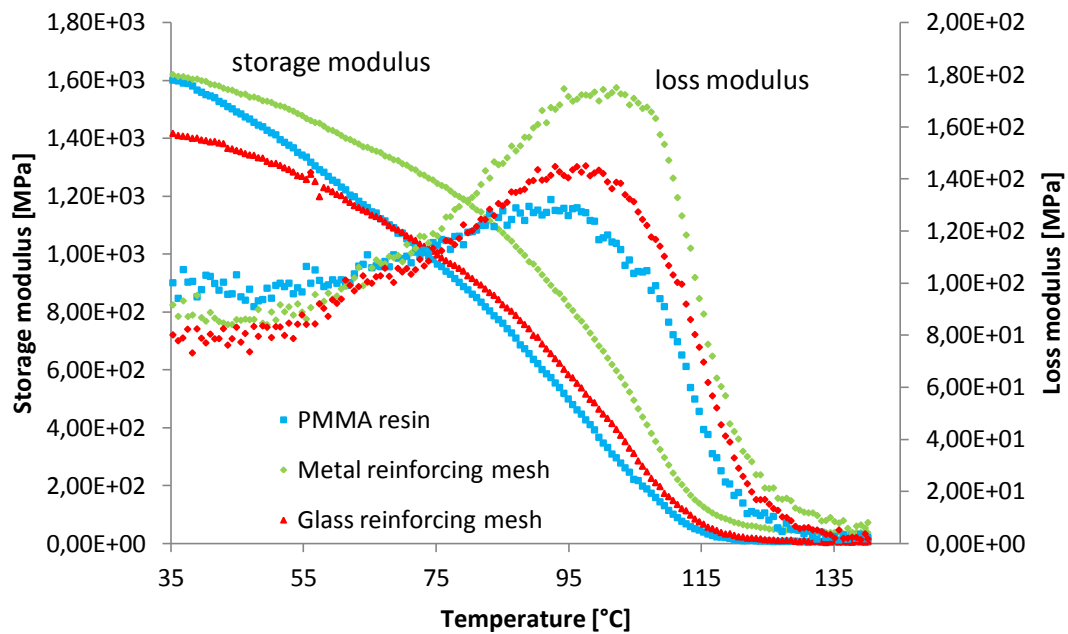
**Figure 15** SEM image of mesh sample, magnification 50×



**Figure 16** SEM images of fibres from mesh, magnification 300 $\times$ , 2000 $\times$  and 5000 $\times$

### 4.3 DMA analysis

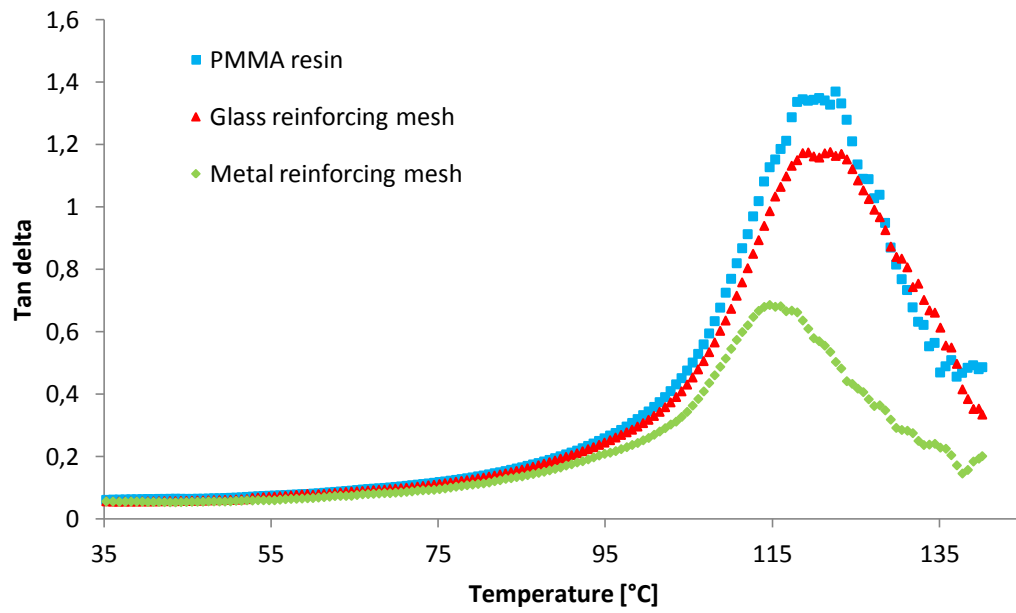
Elastic part ( $E'$ ) of a complex modulus ( $E^*$ ) at a given temperature, frequency and amplitude increases with increasing the Young's modulus of elasticity used reinforcement PMMA resin – Glass reinforcing mesh – Metal reinforcing mesh, while loss part ( $E''$ ), which is associated with dissipated energy during the mechanical vibration shows the opposite trend, Metal reinforcing mesh – Glass reinforcing mesh – PMMA resin. Figure 19 shows the dynamic properties vs. temperature for tested samples.



**Figure 19** DMA data for sample of each set, thermal sweep

Reinforcement affects the T<sub>g</sub> (quite slightly, pushes her to higher values). T<sub>g</sub> should roughly correspond with the intersection of the storage modulus and tan delta (for individual materials should be: 100 °C for PMMA resin, 101,5 °C for Glass reinforcing mesh and 105 °C for Metal reinforcing mesh). T<sub>g</sub> was determined from the peak of the tan delta curve (Figure 20), the values of T<sub>g</sub> are summarized in Table 6. Tan delta characterizes the viscoelastic behaviour of the material and there is a noticeable difference between the Metal reinforcing mesh and PMMA resin and Glass reinforcing mesh. Frequency sweep (Figure 21) shows that elastic and loss modulus grows with

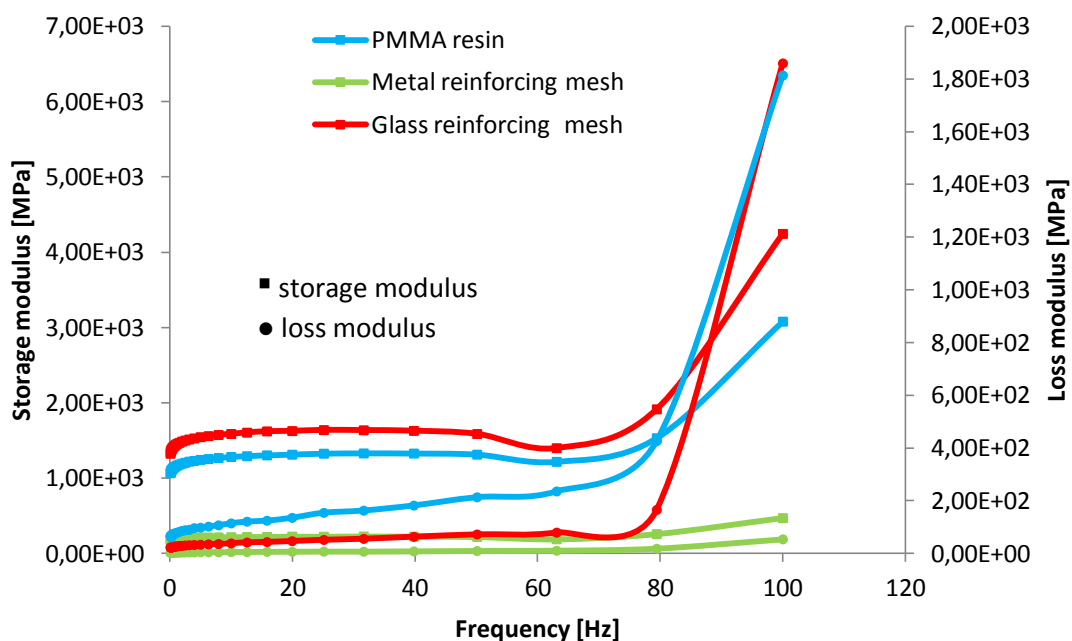
increasing frequency in the row Metal reinforcing mesh – PMMA resin – Glass reinforcing mesh.



**Figure 20** Tan delta curves for tested samples

**Table 6** Values of  $T_g$  for tested samples

	$T_g$ [°C]
PMMA resin	120,5
Metal reinforcing mesh	114,7
Glass reinforcing mesh	122,0



**Figure 21** Frequency sweep of tested samples

#### 4.4 Reinforcement weight content determination

It was also determined the mass and volume content of reinforcement in the samples. Determination was provided for Glass reinforcing mesh sample and Metal reinforcing mesh sample. Calculated values are shown in Table 7, 8.

**Table 7** Calculated values of mass content of samples

	Glass reinforcing mesh			Metal reinforcing mesh		
Sample	$m_{total}$ [g]	$m_f$ [g]	w [%]	$m_{total}$ [g]	$m_f$ [g]	w [%]
1	1,5033	0,0767	5,1	2,0765	0,4694	22,6
2	1,6287	0,0752	4,6	2,2348	0,4938	22,1
3	1,6461	0,0844	5,1	1,9959	0,4288	21,5
Ø			<b>4,9</b>			<b>22,1</b>
		SD [%]	0,2		SD [%]	0,4

**Table 8** Calculated values of volume content of samples

	<b>Glass reinforcing mesh</b>			<b>Metal reinforcing mesh</b>		
<b>Sample</b>	<b>V<sub>total</sub> [cm<sup>3</sup>]</b>	<b>V<sub>f</sub> [cm<sup>3</sup>]</b>	<b>φ [%]</b>	<b>V<sub>total</sub> [cm<sup>3</sup>]</b>	<b>V<sub>f</sub> [cm<sup>3</sup>]</b>	<b>φ [%]</b>
1	1,26	0,03	2,39	1,74	0,06	3,39
2	1,37	0,03	2,16	1,88	0,06	3,41
3	1,38	0,03	2,40	1,68	0,05	3,32
Ø			<b>2,32</b>			<b>3,41</b>
		SD [%]	0,11		SD [%]	0,07



## 6 CONCLUSION

In this Diploma thesis the influence of reinforcement from glass fibres was examined on the mechanical properties of denture base resin. For testing three sets of samples: Duracryl Plus, PMMA resin reinforced with Dentapreg Mesh and PMMA resin reinforced with stainless steel Mesh Strengthener were prepared.

Samples were tested for static loading. The material had sufficient relaxation time for chain relaxation. The values of flexural strength for each sample did not differ significantly, the Young's modulus of elasticity was increasing in the row PMMA resin – Glass reinforcing mesh – Metal reinforcing mesh and the fracture energy was increasing in the row Metal reinforcing – Glass reinforcing mesh – PMMA resin.

During dynamic loading the brittle fracture occurred for all samples. The flexural strength was increasing in the row PMMA resin – Metal reinforcing mesh – Glass reinforcing mesh, the values of Young's modulus of elasticity was increasing in the row Glass reinforcing mesh – PMMA resin – Metal reinforcing mesh. The fracture energy was increasing in the row PMMA resin – Metal reinforcing mesh – Glass reinforcing mesh, for Glass reinforcing mesh was the most significant.

DMA analysis showed that elastic part of complex modulus was increasing in the row PMMA resin – Glass reinforcing mesh – Metal reinforcing mesh. Loss part of complex modulus was increasing in the row Metal reinforcing mesh – Glass reinforcing mesh – PMMA resin. The elastic and loss modulus was increasing with increasing frequency in the row Metal reinforcing mesh – PMMA resin – Glass reinforcing mesh.

For samples reinforced with Glass reinforcing mesh and Metal reinforcing mesh was determined mass and volume content of reinforcement in the samples. Mass content corresponded to volume content.

The expected results of brittle fracture were confirmed on SEM.

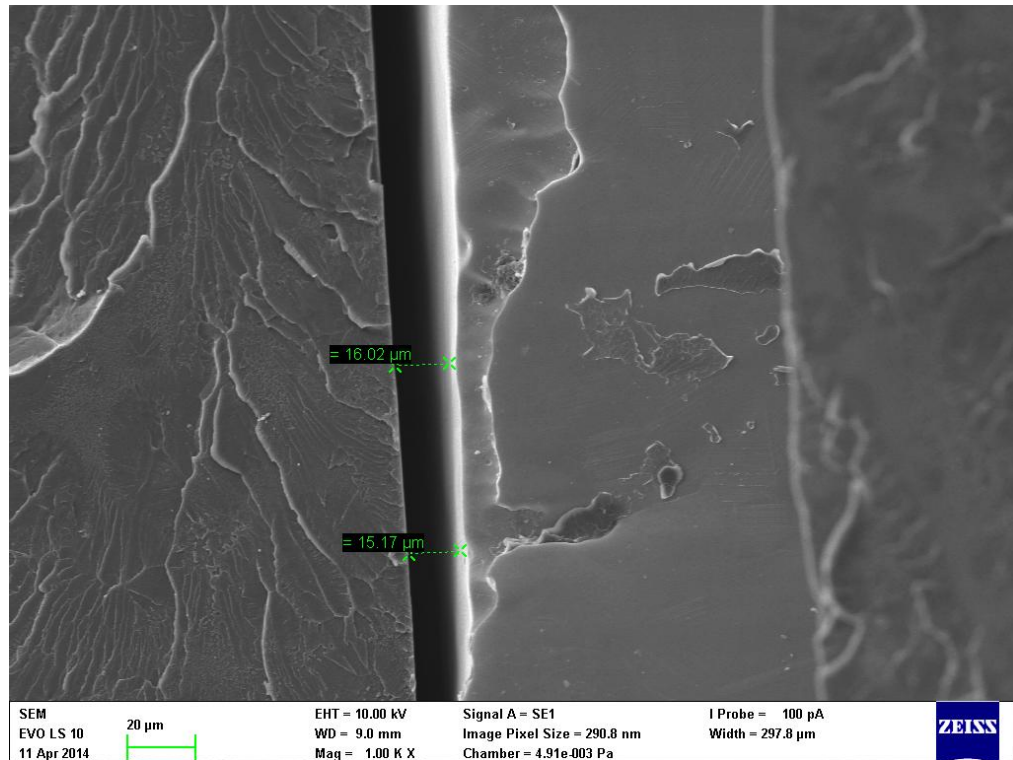
Duracryl Plus is used for restoration of removable denture. This material is reinforced by a stainless steel mesh to improve the mechanical properties. Stainless steel mesh is not perfect – effect on the mechanical properties is minimal and the stainless steel mesh is worse after the aesthetic page. Glass reinforcing mesh could be used as an alternative. With this reinforcement could be in the future work on the adhesion and colouring.

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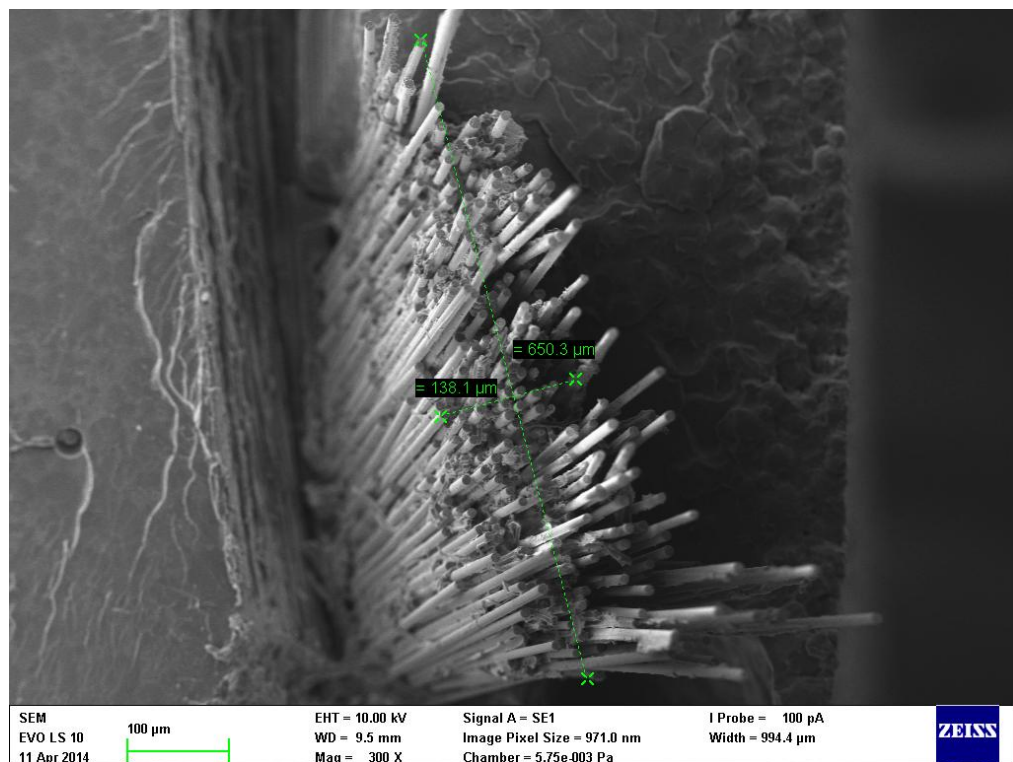
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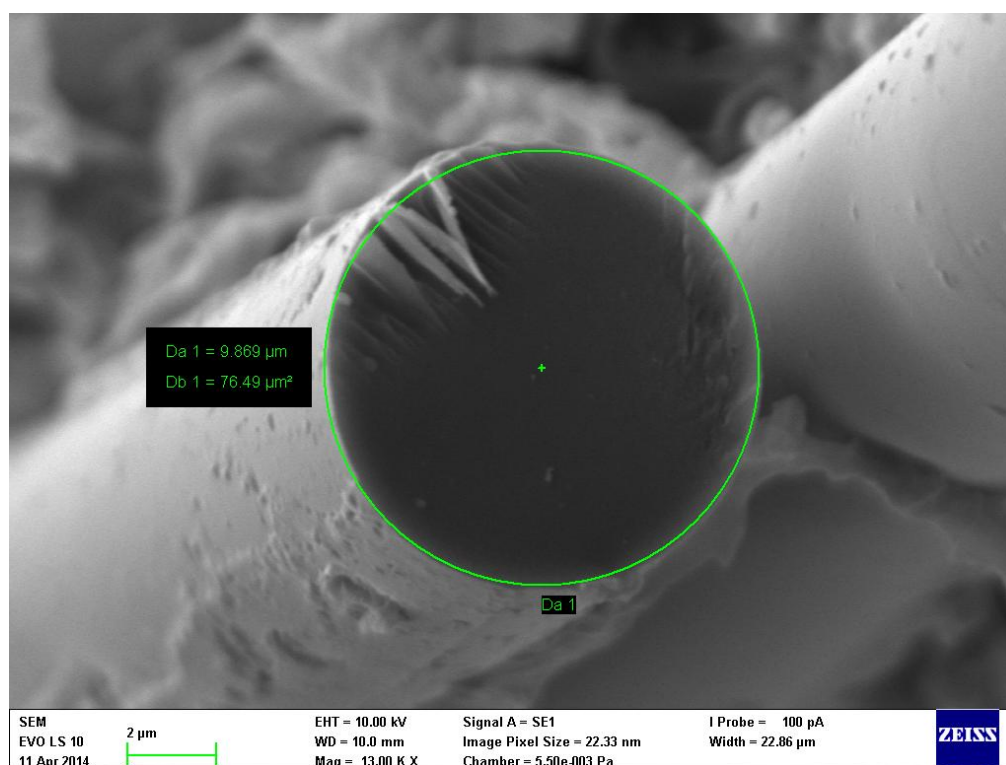
## APPENDIX



**Figure 13** SEM image of Metal reinforcing mesh sample, poor adhesion, magnification 1000×



**Figure 17** SEM image of mesh sample, dimension of bunch, magnification 300×



**Figure 18** SEM image of mesh sample, dimension of fibre, magnification 13000×