

Improvement of cavitation resistance of composite films using functionalized alumina particles

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Abstract

Composite films having the UV cured Bis-GMA (Bisphenol A glycidylmethacrylate)/TEGDMA (triethylene glycol dimethacrylate) as a matrix and the ferrous oxide doped alumina (Al₂O₃ Fe) based particles were prepared and subjected to cavitation. In order to improve the mechanical and adhesion properties of composites, four different surface modifications of filler particles were performed: 3-methacryloxypropyltrimethoxysilane (MEMO), vinyltris(2-methoxyethoxy)silane (VTMOEO), (3-aminopropyl)trimethoxysilane (APTMS) and biodiesel (BD). Composite films were made with 0.5, 1.5, and 3 wt.% of ferrous oxide doped alumina particles with each of the mentioned surface modifications. Composite films were prepared on brass substrates and exposed to cavitation erosion. The erosion was monitored using the mass loss while image analysis was used to observe surface defects. The composite film reinforced with Al₂O₃ Fe having VTMOEO as a surface modifier was the most resistant one in terms of mass loss, as well as the level of surface destruction. Results were compared to the same polymer matrix film and composite films prepared with fillers without surface modifications revealing that all composites with surface modified fillers exhibited some improvement in resistance to cavitation.

Keywords: polymer-matrix composites (PMCs), adhesion, surface modification, image analysis, cavitation erosion.

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1. INTRODUCTION

Composite materials have been used in a variety of fields such as surface protection layers, in parts requiring demanding properties, and also as dental restoration materials. Possibilities to use composite materials in dentistry are based on their ability to maintain mechanical properties for a long time, to achieve good adhesion to dentine and to be colored to achieve aesthetical demands [1]. Dental resin composites based on BisGMA (2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl] propane) are commonly used in dental restorations due the possibility of different forms in order to fill the cavity while the polymerization can be achieved by light in a rapid and controlled manner [2]. Light curing of dental resins and resin composites in small volumes using halogen lamps or more modern LED (light emitting diodes) units is a routine operation [3].

Dental composites are typically composed of four major components: organic polymer matrix (BisGMA, TEGDMA, urethane dimethacrylate (UDMA), etc.), inorganic filler particles (up to about 70 % mass fraction), coupling agents, and the initiator–accelerator system [4]. The BIS-GMA/TEGDMA system, was chosen in this work as it enables control of the polymer shrinkage, and the two polymers enable the viscosity control required for the resin formation. In specific, Bis-GMA serves to limit polymer shrinkage and enhance resin reactivity, while TEGDMA provides methacrylate double bond for increased conversions [5]. While the types of fillers vary, mostly used are short glass fibers, silica and ceramic particles or whiskers [6,7]. The role of fillers is to enhance the mechanical properties, but also to limit the shrinkage of the composite during light curing.

Cavitation represents formation, growth and collapse of bubbles due to the pressure fluctuations in a liquid. The bubble collapsing produces shock waves and micro jets on the exposed surface of the material, in a very short time [8].

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Repetition of the impact induces the surface damage with subsequent material loss, which depends on the material properties [9]. Cavitation is the way to test the behavior of a material in rough environments, but it can be also used to evaluate surface resistance of a coating as well as adhesion of a film to the substrate [10,11].

The aim of this study was to investigate cavitation resistance of composite films based on BisGMA/TEGDMA dental resins and ferrous oxide doped alumina particles with four different surface modifications. Surface modifications of the fillers are supposed to increase the interface cohesion between the filler particles and the matrix, but also to influence the adhesion of the composite film to the substrate. The film mechanical properties and adhesion strength influence its resistance to cavitation so that this method can be used to obtain valuable information about the film behavior in severe conditions. The obtained results could be extrapolated to show behavior of the film in prolonged times of service in dental applications. It is well known that dental materials should bear very high loads in difficult environments and the obtained information could be useful to evaluate potentials of investigated materials.

2. EXPERIMENTAL PROCEDURE

2. 1. Materials

BisGMA (bisphenol A glycidyl methacrylate, $C_{29}H_{36}O_8$), TEGDMA (triethylene glycol dimethacrylate, $C_{12}H_{18}O_6$), CQ (camphorquinone, $C_{10}H_{14}O_2$), and 4EDMAB (ethyl-4-dimethylamino benzoate, $C_{11}H_{15}NO_2$) were all supplied by Sigma – Aldrich, Darmstadt, Germany. 3-aminopropyltri-methoxysilane (APTMS, $C_9H_{23}NO_3Si$), iron chloride ($FeCl_3 \cdot 6H_2O$) potassium hydroxide (KOH), tetrahydrofuran (THF, C_4H_8O), and methanol (CH_3OH) were purchased from Fluka (Sigma-Aldrich, Darmstadt, Germany). The tris(2-methoxyethoxy)(vinyl)silane (VTMOEO, $C_{11}H_{24}O_6Si$); (3-methacryloxy propyl trimethoxy silane) (MEMO, $C_{10}H_{20}O_5Si$) used as silane coupling agents were supplied from Dynasylane, Evonik Industries, Essen, Germany. Aluminum chlorohydrate $Al_2Cl(OH)_5 \cdot 2.5H_2O$ was supplied from Clariant company under the brand name Locron L (Paris, France).

2. 2. Ferrous oxide doped alumina particle preparation via a sol-gel route

The alumina particles were synthesized via the sol gel technique: $Al_2Cl(OH)_5 \cdot 2.5H_2O$, $FeCl_3 \cdot 6H_2O$ and demineralized water as a solvent. Demineralized water and aluminum chlorohydrate were mixed using a magnetic stirrer until complete dissolution and then 1.5 wt.% of $FeCl_3 \cdot 6H_2O$ was added under continued stirring. The mixture solution was then poured into a Petri dish and allowed to gel. The resulting mixture was ground in a mortar and the powder was calcined at 900 °C for two hours. The amount of Fe_2O_3 precursor was adjusted so as to obtain 10 wt.% Fe_2O_3 in the final particle composition. The gel was heat-treated at 900 °C in order to obtain the appropriate crystal structure of the reinforcement [12]. The particle size of alumina doped with iron oxide was previously determined by particle size analyzer. The most commonly used metrics when describing particle size distributions are d-values (d(0.1), d(0.5) and d(0.9)) which are the diameters from the intercepts for 10%, 50% and 90% of the cumulative number of particles. The diameters of such obtained particles are: d(0.1) = 0.412 μm , d(0.5) = 0.608 μm , d(0.9) = 1.208 μm (in other words, the values in brackets represents the percentage of analyzed particles in the range of 0.0-1.0 having diameter up to the specified value) [13]. According to the obtained results, calcined Al_2O_3 Fe powder was of submicron sizes.

2. 3. Synthesis of methyl ester from linseed oil fatty acid (biodiesel – BD)

In a four-necked glass reactor of 2 L, equipped with a reflux condenser, mechanical stirrer, thermometer and a dropping funnel, 929 g (3.3 mol) of linseed oil, dissolved in 85 ml of methanol, was added. Next, a potassium hydroxide solution in methanol (0.12 mol of KOH in 102 ml of methanol) was added drop wise. The reaction mixture was then heated to 58–62 °C for 3 hours, and left to cool down. The bottom layer, *i.e.* mainly raw glycerin, was separated, and the upper layer was treated with active charcoal and filtered through diatomaceous earth. After drying with sodium sulfate, the obtained linseed oil methyl ester (biodiesel, BD) was purified by vacuum distillation under nitrogen. Characteristics of the obtained mixture, termed BD were: the acid value (AV) 5 mg KOH/g; ester content 97 %; iodine value 152 [14].

2. 4. Surface modification of alumina based particles

Alumina particles (1 g) were dispersed in toluene (75 ml) using a mechanical stirrer in under reflux under the flow of nitrogen. When the boiling point of toluene was reached (110.6 °C), 1 g of silane coupling agent (VTMOEO/MEMO/APTMS) was added and the reaction was kept for 22 h. After the completion of the reaction time, the particles were filtrated and washed with hexane in order to remove unreacted silane. The particles were dried at 40 °C in an oven for 12 h and then used for preparation of composites [14]. The Al_2O_3 Fe particles modified with VTMOEO, MEMO and APTMS were denoted as Al_2O_3 Fe – VT, Al_2O_3 Fe – ME, and Al_2O_3 Fe – AM, respectively.

2. 5. Second step of alumina modification with BD

The modified alumina particles with terminal amino groups (Al_2O_3 Fe – AM), from the first step of alumina modification, were dispersed in 50 ml THF and 1.56 g of methyl ester of linseed oil fatty acids in a three-necked glass reactor, equipped with a magnetic stirrer, thermometer, reflux condenser and a calcium chloride protection tube. The reaction was carried out for 12 h at 25 °C, whereupon the mixture was kept at 60 °C for 2 hours. The obtained particles were filtered under vacuum, followed by dispersion in THF and filtration (two times), washed with absolute ethanol and dried at 40 °C for 12 h. The particles obtained in this modification step were denoted as Al_2O_3 Fe – BD.

2. 6. Composite film preparation

The substrate was brass 260 ½ hard, (ASTM B36 250 µm-thick (ASTM B36, K&S Engineering, USA). The substrate chemical composition is: copper (68.5-71.5 %), zinc (remainder), lead (0.07 % max) and iron (0.05 % max). Prior to film deposition, the brass substrate required activation in 20 % sulfuric acid solution [15]. After drying of the substrate, the film was deposited. The composite matrix was prepared from: BisGMA 49.5 %, TEGDMA 49.5 %, CQ 0.2 % and 4EDMAB, 0.8 % and cured under UV light. Composite films were made with 0.5, 1.5, and 3 wt.% content of modified fillers as presented in the previous work [12]. A drop coating method was used for polymer film deposition on the brass substrate. In specific, a thin cover on the brass substrate was formed by depositing a single drop of the monomer solution on its surface. The film was then polymerized under UV light for 3 minutes. The ultimate thickness of the deposited film was controlled by the weight of a glass cover, which was placed over the drop, so that the obtained film had a thickness value of 70 ± 5 µm.

2. 7. Characterization methods

Fourier-transform infrared spectroscopy (FTIR) was used for characterization of chemical compositions of four different modifications of alumina based particles and the obtained composite films. Tests were performed using a Nicolet 6700 spectrometer (Thermo Scientific, USA) in the attenuated total reflectance (ATR) mode with a single bounce 45 °F Golden Gate ATR accessory with a diamond crystal, and an electronically cooled DTGS detector. FTIR spectra were obtained at 4 cm^{-1} resolutions with ATR correction. The Nicolet 6700 FTIR spectrometer was equipped with OMNIC software and recorded the spectra in the wavelength range from 2.5 µm to 20 µm (*i.e.*, $4000 - 500 \text{ cm}^{-1}$) [16].

Alumina based particles modified with four different surface modifiers were examined using a field emission scanning electron microscope (FESEM), MIRA3 from TESCAN ORSAY HOLDING, *a.s.*, Czech Republic, operated at 20 kV. The images were used to extract morphology information of each of the used fillers. Morphological parameters were determined using the automatic object detection tool and the particles were analyzed on 2 different images and for every sample of particles. Number of objects is given in Table 1.

Cavitation tests were performed by using an ultrasonic vibratory cavitation device in accordance with the ASTM G32-92 Standard (the stationary specimen method). The device consisted of a 360 W high frequency generator, electrostrictive transducer, transformer for mechanical vibrations and a water bath containing the test specimen. Cavitation testing was accomplished using recommended standard values [17]:

- vibration frequency: 20 ± 0.5 kHz
- vibration amplitude at the top of the transformer: 50 µm
- gap between the test specimen and the transformer: 0.5 mm
- water temperature in the bath: 25 ± 1 °C
- water flow: from 5 to 10 ml/s.

Cavitation was monitored using mass loss measurements, similarly as in studies of metallic materials and by measuring the corresponding surface degradation during the experiment. Specimens were dried in a dryer at 110 °C for 1 hour and the mass losses were measured on an analytical balance with an accuracy of ± 0.1 mg. Surfaces of the specimens were photographed using a scanner in order to minimize the influence of light conditions. Surface damage was then determined from the photographs according to differences in gray levels. Image analysis of the photographs of sample surfaces was performed using the image analysis software (Image-Pro Plus 4.0, Media Cybernetics Inc., Cambridge, UK) that provided measurements of the surface damage levels [18-21]. The results were presented as a surface erosion ratio.

Optical microscopy technique was applied to analyze the effect of the erosion and to interpret the results of cavitation tests. In this paper, the images after 0, 1, 2 and 3 min of cavitation were used for detailed analysis. Image analysis was also used for image data processing and quantification of the damage characteristics. Image analysis was also used for image data processing and quantification of the damage characteristics. Results of image analysis are given in Figure 5. The image analysis software was necessary to recognize the tiny differences in the grey level on images in order to discover the surface destruction level.

3. RESULTS AND DISCUSSION

3. 1. Microstructure of the particles

Morphologies of Al_2O_3 Fe with four different surface modifications (MEMO, VTMOEO, APTMS and BD) are shown in Figure 1 together with the measured particle diameter distribution.

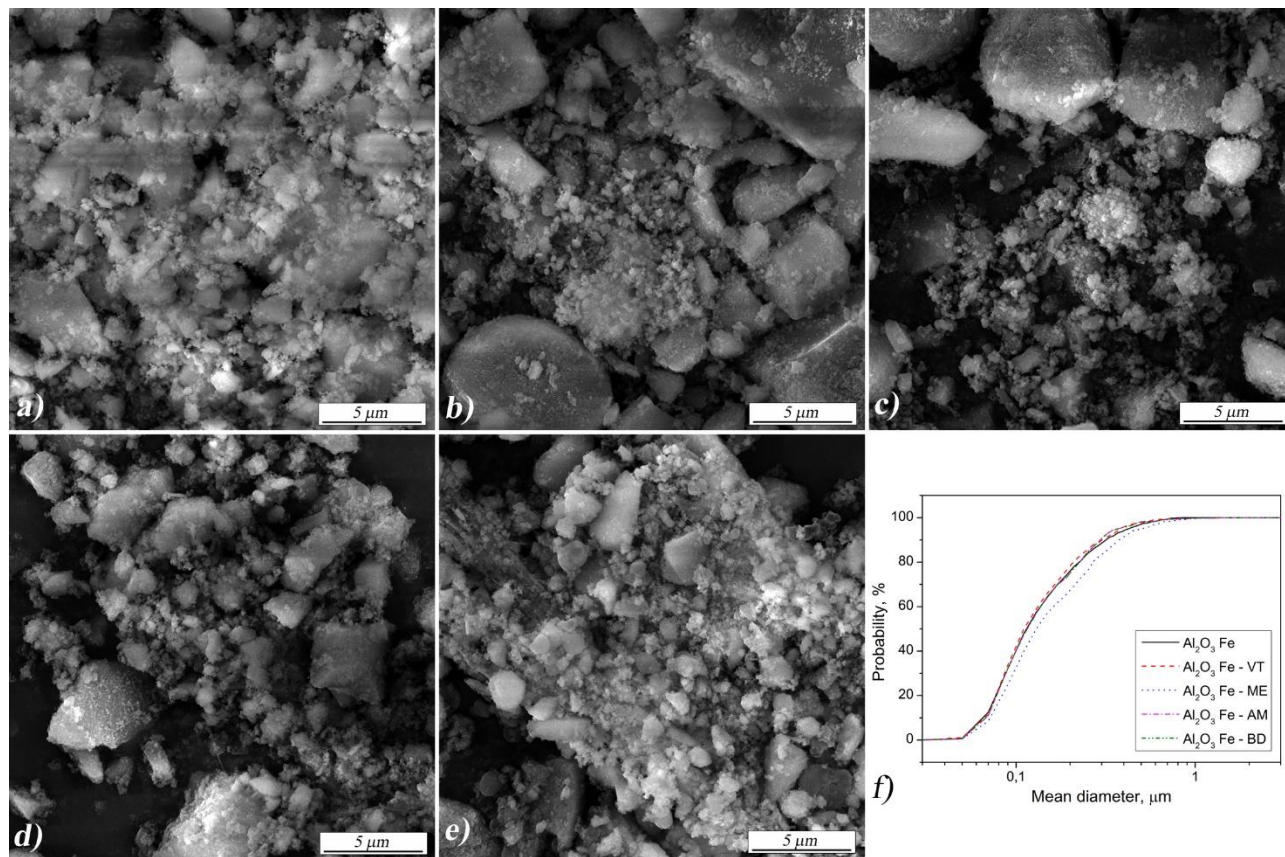


Figure 1. The FE-SEM micrographs of: a) Al_2O_3 Fe, b) Al_2O_3 Fe – VT, c) Al_2O_3 Fe – ME, d) Al_2O_3 Fe – AM, e) Al_2O_3 Fe – BD and f) cumulative particle diameter of analyzed particles.

Statistical data of morphological properties of Al_2O_3 Fe with and without surface modification are given in Table 1.

Table 1. Statistical data of morphological particle properties (values in parentheses represent standard deviation)

Statistical data	Area, μm^2	D_{\max} / μm	D_{\min} / μm	D_{mean} / μm	Roundness	Fractal dimension	No. of objects analyzed
Al_2O_3 Fe	0.026 (0.055)	0.294 (0.270)	0.075 (0.054)	0.171 (0.123)	5.502 (5.220)	1.153 (0.074)	1197
Al_2O_3 Fe – VT	0.023 (0.050)	0.280 (0.267)	0.074 (0.055)	0.165 (0.124)	5.266 (4.339)	1.146 (0.075)	756
Al_2O_3 Fe – ME	0.037 (0.077)	0.343 (0.337)	0.094 (0.074)	0.200 (0.155)	5.716 (6.475)	1.142 (0.074)	315
Al_2O_3 Fe – AM	0.027 (0.084)	0.296 (0.314)	0.076 (0.055)	0.170 (0.128)	5.337 (5.107)	1.149 (0.071)	757
Al_2O_3 Fe – BD	0.048 (0.167)	0.367 (0.387)	0.095 (0.080)	0.207 (0.175)	5.723 (5.910)	1.142 (0.075)	818

Analysis of the processed FE-SEM images, has demonstrated submicron Al_2O_3 Fe particle sizes achieved by the sol-gel technique. The mean diameter (D_{mean}) of neat Al_2O_3 Fe particles was $0.171 \pm 0.123 \mu\text{m}$ (Table 1). Obtained high standard deviations (with values close to the mean) indicate broad distribution of particle size in a wide diameter range. The obtained diameter ranges by image analysis were $0.007 - 2.119 \mu\text{m}$ for neat Al_2O_3 Fe particles; $0.008 - 2.623 \mu\text{m}$ for Al_2O_3 Fe – VT; $0.010 - 3.355 \mu\text{m}$ for Al_2O_3 Fe – ME; $0.009 - 3.255 \mu\text{m}$ for Al_2O_3 Fe – AM; and $0.013 - 4.351 \mu\text{m}$ for Al_2O_3 Fe – BD. Still, the predominance of smaller particles led to small mean diameters (Table 1).

Surface modification by VT induced a decrease in the surface area and particle diameter, while that by BD increased the particle size, which was shown later to lead to a weaker reinforcement. Thus, the surface modification type influences particle agglomeration and consequently the apparent particle size, where lower agglomeration leads to better composite reinforcement.

3. 2. FTIR characterization of fillers and composite films

FTIR spectra of neat Al_2O_3 Fe particles as well as particles with surface modifications (MEMO, VTMOEO, APTMS and BD) are shown in Figure 2.

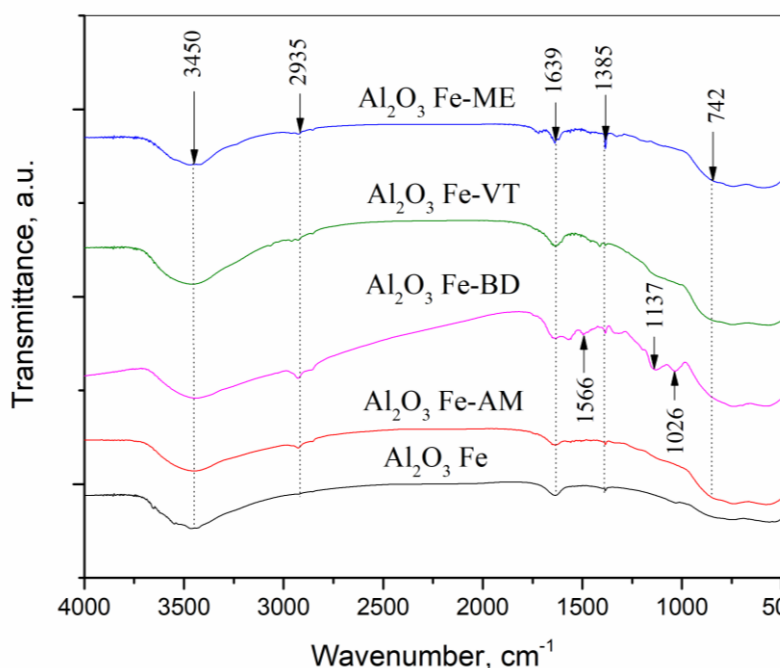


Figure 2. Comparison of FTIR spectra of Al_2O_3 Fe based particles used in composites preparation.

The characteristic peaks of investigated particles observed at 3460 cm^{-1} can be assigned to O-H groups [22]. The following peak at $\sim 1639\text{ cm}^{-1}$ relates to the H_2O vibrational bending mode [23, 24]. Absorption at 740 cm^{-1} could be attributed to vibrational modes associated with $[\text{M}^{\text{II,III}}(\text{OH})_6]^{4-3-}$ complexes distributed along the double hydroxide (LDH) layers mostly attributed to the γ crystalline structure of alumina [25-27].

Introduced surface functionality on the particles has led to the emergence of new peaks. The peaks at 2935 cm^{-1} are attributed to the ethylene C-H stretching bands of attached silane. The peaks at $1636/1639\text{ cm}^{-1}$ showed skeletal C=C double bond vibrations from silane functional groups at MEMO modified particles that overlapped with H_2O vibrational bending mode.

3. 3. Investigation of cavitation erosion

During the cavitation experiment, mass losses of dried samples were found (Figure 3). The obtained data enable comparison of mass losses in composite films with modified particles to those, previously reported [12], where the content of particles was much more elevated in order to obtain similar result.

Pure polymer films are not very resistant to cavitation as they get destructed after only 1 min of testing. The addition of ferrous doped alumina fillers increases this time to 2 min and lowers the destruction rate represented as the lower rate of mass loss of the specimen. When the filler particles were modified, reinforced composite is obtained with improved adhesion to the substrate. Every modified film behaved better than the pure polymer in terms of longevity. The AM and BD modified fillers exhibited longer exposure times, but a significant mass loss in the last minute of the process, comparable to that of the pure polymer. The VT and ME films exhibit longer film exposure times as well as a significantly lower mass loss during the whole testing period. Behavior of these films is better in terms of mass loss due to cavitation as well as of the film durability. The VT and ME modifications can be considered as a successful mean for enhancing both cavitation resistance and adhesion to the metal substrate.

Mass loss for the composite film having the best cavitation resistance with a ferrous oxide doped alumina based particles with VT surface modification having different filler contents is presented in Figure 4. It can be observed that the addition of 3 wt. % of the modified filler significantly improves the cavitation resistance of the film. The adhesion of this film should be significantly better as compared to the pure polymer film. On the contrary, addition of 0.5 as well as 1.5 wt. % of the filler does not seem to be sufficient to improve the cavitation resistance of the composite film.

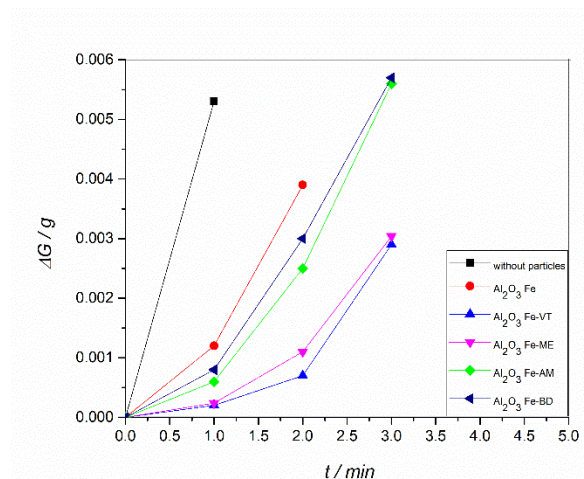


Figure 3. Mass loss during cavitation resistance testing for pure composite films and composite films with 3 wt. % of different alumina based particles.

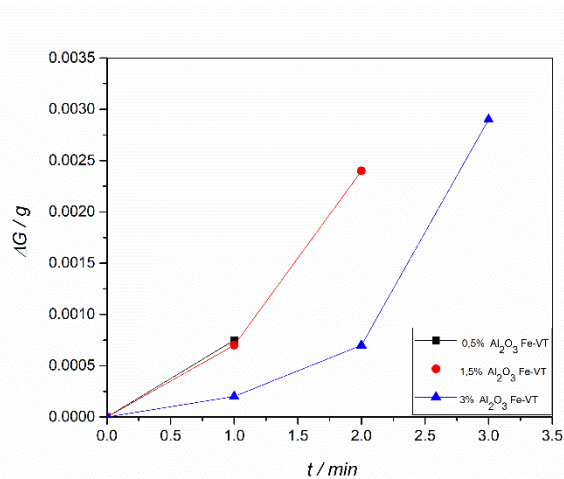


Figure 4. Mass loss during cavitation resistance testing for composite films reinforced with Al_2O_3 Fe – VT

3. 4. Image analysis of cavitation damages

Images showing surface defects after 1 min of cavitation for samples made of composite films with 0.5 wt. % of different alumina based particles are shown in Figure 5.

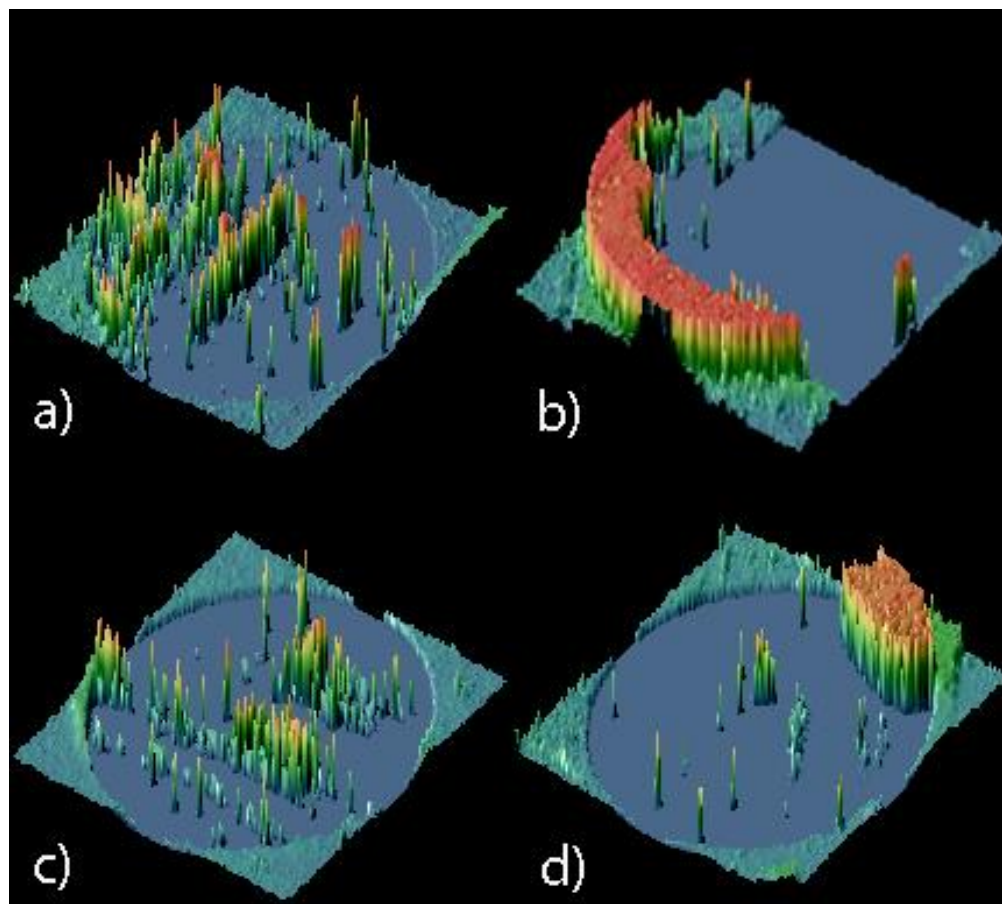


Figure 5. Surface plot images after 1min of cavitation for composite films with: a) 0.5 wt. % of Al_2O_3 Fe – AM, b) 0.5 wt. % of Al_2O_3 Fe – BD, c) 0.5 wt. % of Al_2O_3 Fe – ME and d) 0.5 wt. % of Al_2O_3 Fe – VT.

The Image Pro Plus program was used for determination of the surface deterioration level during testing and the results are presented in Figure 6.

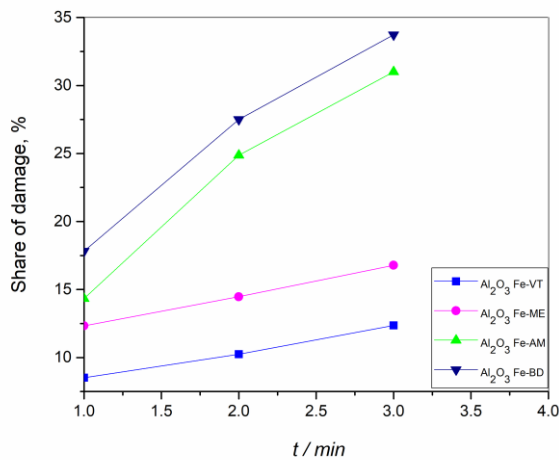


Figure 6. Share of observed damage to composite films vs. time for samples with 0.5 wt. % of different alumina based particles.

The surface degradation is considerable in the film with BD modified fillers, which is in accordance with mass loss measurements. The film with ME modified particles behaves better in terms of mass loss, but the surface degradation level is comparable to that with the BD modified filler. The mass loss of this composite is lower as compared to the composite with AM modified fillers, which exhibits better surface resistance in terms of the surface damage ratio. These findings could be related to the size of defects observed in images of composite films after exposure to cavitation. The ME film exhibit larger damaged areas, but the depth of those damages remains lower. The best performance in terms of the surface degradation level, as well as in terms of mass loss, has the composite with VT modified fillers. The overall surface destruction share after 3 min. shows that at the end of the testing procedure the BD modified films have the highest surface degradation level of about ~33.7 % while the surface degradation level is the lowest in VT modified films amounting to ~12.3 %.

Cross sections of films after cavitation, for the composite film reinforced with Al₂O₃ Fe – VT, exhibiting the best adhesion and the cross section of the film reinforced with Al₂O₃ Fe – BD exhibiting the weakest adhesion, are shown in Figure 7.

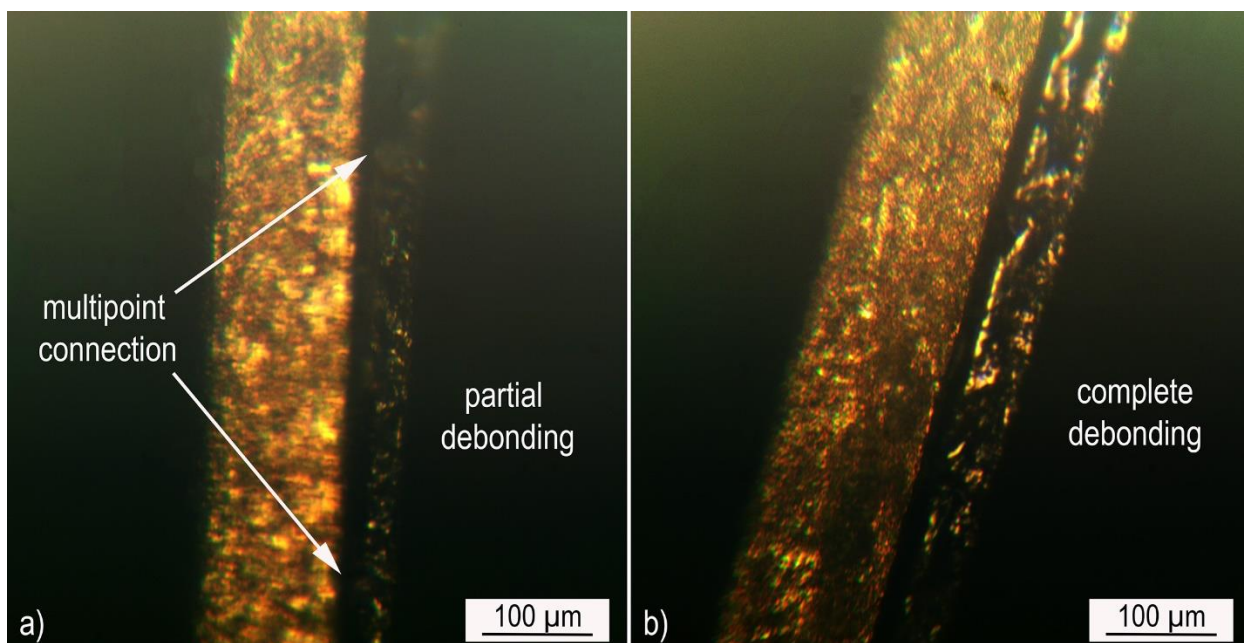


Figure 7. Cross-sections of composite films after the cavitation test, reinforced with: a) Al₂O₃ Fe – VT and b) Al₂O₃ Fe – BD particles

It can be seen that the latter composite film almost completely debonded from the substrate (Fig. 7b). On the contrary, the composite film reinforced with Al₂O₃ Fe – VT particles resisted to the cavitation erosion by stronger adhesion, which is seen by remaining multipoint connection to the substrate and the lowest share of damage (Figs 6 and 7a, respectively).

4. CONCLUSIONS

The ferrous oxide doped alumina based particles having different surface modifications (MEMO, VTMOEO, APTMS and BD) were used as reinforcement in the matrix based on BisGMA/TEGDMA composites. The FTIR analysis proved the presence of different groups on the surface of each modified particle specimen. The various surface modifications of particles induced different effects on the resistance of composite films to cavitation. The best cavitation resistance was shown for composite films with VT particle surface modification both in terms of mass loss and surface degradation level, while the worst one was for the film with BD modified particles. Better cavitation resistance also confirmed better adhesion of the composite film to the metal substrate. In our previous studies, we have shown that the best improvement in cavitation resistance was achieved in composites having 3 wt.% of unmodified particles, which was confirmed in the present study in composites with the same content of VT modified particles. Results in the present study have also demonstrated that composites with modified filler particles behaved better when subjected to cavitation as compared to those with no modified particles, while cavitation resistance was directly related to film adhesion to the metallic substrate.

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САЖЕТАК

Побољшање отпорности композитних филмова на кавитацију коришћењем функционализованих честица алуминијум оксида

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(Научни рад)

Композитни филмови на бази Bis-GMA (бисфенол А глицидилметакрилат)/TEGDMA (триетилен гликол диметакрилат) као матрице и честица на бази алуминијум оксида (Al₂O₃ Fe) припремљени су и изложени кавитацији. У циљу побољшања механичких и адхезионих својстава композита, коришћене су четири различите модификације површине честица ојачања: 3-метакрилоксипропилтриметоксисилан (ММО), винилтрис (2-метоксиетокси) силан (ВТМОЕО), (3-аминопропил) триметоксисилан (АПТМС) и биодизел (БД). Композитни филмови направљени су са 0,5, 1,5 и 3 мас.% алуминијум оксидних честица допираних гвожђе оксидом са сваком од поменутих површинских модификација. Композитни филмови су нанешени на месингану подлогу и изложени су кавитацији. Кавитациона ерозија праћена је губитком масе и коришћена је анализа слике за посматрање површинских оштећења. Композитни филм који је ојачан са честицама Al₂O₃ Fe и површинском модификацијом ВТМОЕО био је најотпорнији у смислу губитка масе и нивоа разарања површине. Резултати су упоређени са композитним филмом исте полимерне матрице и честицама без површинске модификације и сви композити који имају површински модификована ојачања показују побољшања у отпорности на кавитацију.

Кључне речи: композити са полимерном матрицом, адхезија, површинска модификација, анализа слике, кавитација

