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## The Use of Image Analysis for Determination of Surface Deterioration Level of Improved Alumina Based Materials Subjected to Cavitation

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### Abstract:

*Alumina based specimens having different content of alumina based fibers were investigated for possible application as cavitation resistant material. Cavitation damages of the alumina based specimens were tested by the modified vibratory cavitation set up. Erosion rates were measured based on the method developed for metallic samples, mass loss was measured during the experiment. Surface erosion was determined during the experiment simultaneously to mass loss measurements. Image Pro Plus Program was applied for surface analysis during testing. Results indicate that investigated material exhibit excellent mechanical properties and very good resistance to cavitation erosion.*

**Keywords:** *Composites, Fibers and filaments, Fracture, X-ray methods, Image analysis*

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### 1. Introduction

Brittleness of structural ceramics in engineering is the main factor limiting their application. Various methods have been used to improve the toughness of ceramics, such as transformation toughening, second phase particle reinforcing, whisker or fiber toughening. The attractive ceramic properties of ceramic fiber composites include operating temperatures of up to about 1000°C, high temperature structural integrity and high temperature corrosion resistance. Sintering process is widely used for synthesis and consolidation of advanced materials.

Ceramics composites reinforced with ceramic fibers have been known for many years for their improved strength and toughness. The high toughness of these composites has been associated with limited bonding between the fibers and the matrix in order to give substantial fiber pull-out as a major source of toughening. Ceramic fiber composites have been developed to take advantage of the attractive properties of ceramics minimizing their brittleness. In fiber reinforced composites, strong and stiff fibers are usually embedded into a ductile matrix with the aim of enhancing mechanical properties, mainly strength, strength-to-weight ratio, etc. Under load, the matrix transmits the force to the fibers, which carry the most of

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applied load. The geometry and arrangement of fibers are also important in controlling the mechanical properties of a fiber reinforced composite.

Ceramic matrix composites (CMC<sub>s</sub>) can display quasiductile deformation behavior when mechanisms such as crack deflection, fiber pull-out, crack bridging and debonding can be made to operate by optimizing strength of the interface between fiber and matrix [1-4]. In this investigation alumina based refractory samples were improved using the addition of different amount of alumina based fibers were used.

Cavitation, one of the mechanisms of liquid erosion, characterized by the generation and the collapse of vapor structure in liquid, occurs frequently in hydraulic machinery such as pumps, turbine and propellers. The pressure waves emitted during the collapses of vapor structures interact with neighboring solid surfaces, leading to material damage [5]. Cavitation damage is often tested for metallic materials. As many composites and ceramic materials are expected to replace some of the metallic components, the goal of this investigation was to apply cavitation testing on the ceramic composite samples.

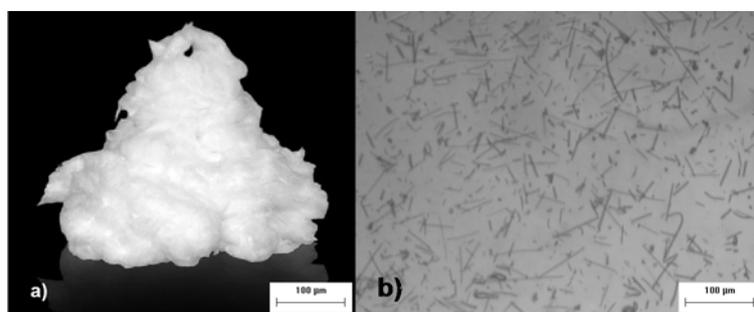
In this study image analysis was used for the determination of damage surface level before and during testing. Samples were scanned using the scanner having high resolution that enables the observation of the surface and the determination of damaged and non damaged areas. Results were as ratio P (damaged surface) and P<sub>0</sub> (ideal surface before quenching).

## 2. Materials

The raw materials selected for specimen preparation in this study were bauxite, chamotte and clay [6-7]. Raw materials were mixed in mortar. One series consists of raw materials without the addition of alumina fibers and other two series of samples were prepared using the addition of 1 wt.% and 2 wt.% (139.2 μm mean lengths) of alumina fibers (it had aspect ratio  $l/d \approx 17$ ). The samples were sintered at 1200 °C for 2h. Samples were cylinders having 30.0 mm diameter and 8.0 mm high.

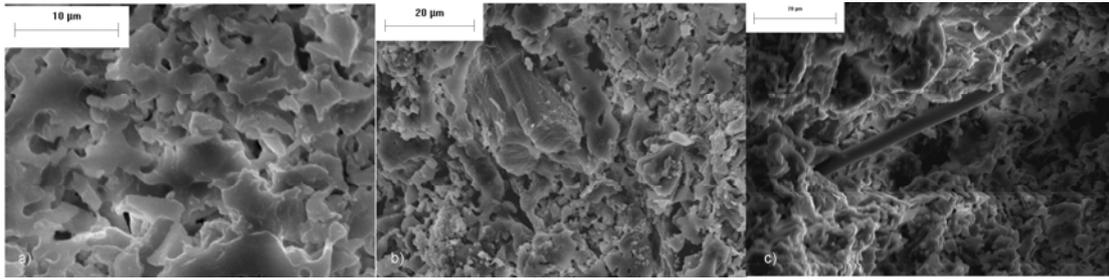
In this experiment Thermal Ceramics bulk fibers are used. Maximum temperature range for usage of those fibers is 1260° - 1549° C. They provide good chemical stability and resistance to chemical attack.

The fibers were cracked in mortar in order to obtain fibers having defined lengths to diameter ratios. Image of bulk fibers is presented in the fig.1a and the micrograph of cracked fibers is given in fig. 1b.



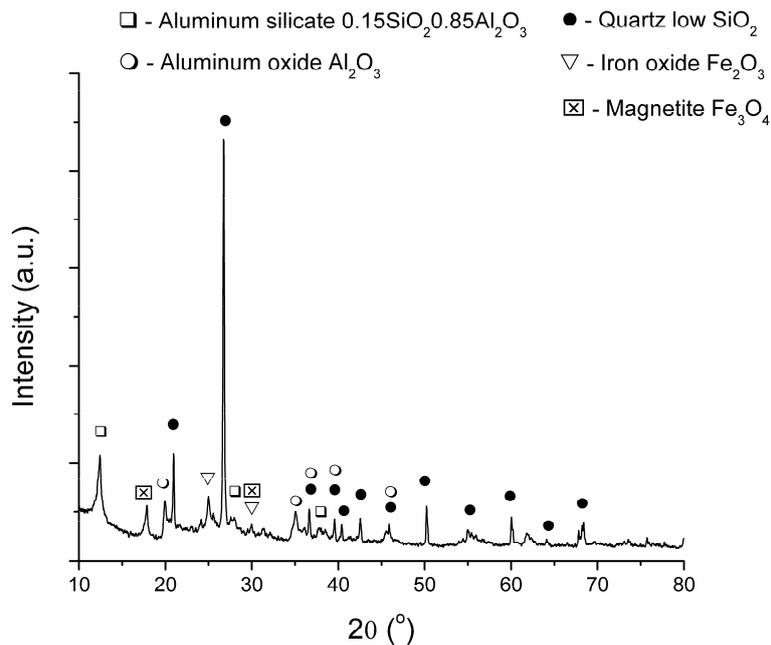
**Fig. 1.** Ceramic fibers used in sample preparation: (a) bulk ceramic fibers; (b) fibers after preparation for sample preparation.

The SEM micrograph of the samples without fibers and with fibers is given in Fig. 2. It could be seen that the fibers were well chosen as they establish good contact with the matrix and they could be well bonded so as to enable better mechanical properties of the material.



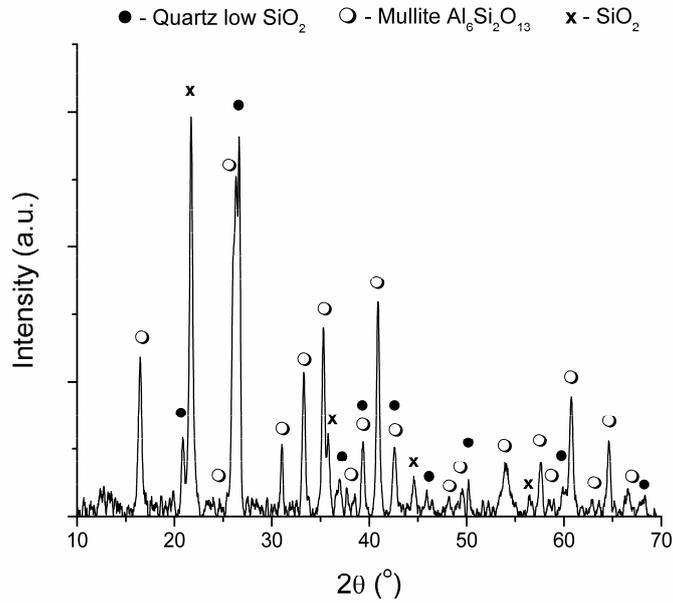
**Fig. 2.** The microstructure of the samples: a) without fibers, b) with 1% of fibers, c) with 2% of fibers showing imbedded into the structure of the matrix.

Microstructure of sintered samples was studied using X-ray analysis. The diffraction patterns were obtained for raw materials such as clay, bauxite and chamotte. Clay was composed of quartz ( $\text{SiO}_2$ ), aluminum oxide ( $\text{Al}_2\text{O}_3$ ), iron oxide ( $\text{Fe}_2\text{O}_3$ ) and magnetite ( $\text{Fe}_3\text{O}_4$ ), as seen in Fig. 3., while for chamotte powder quartz ( $\text{SiO}_2$ , JCPDS number 05-0+50), silicon oxide ( $\text{SiO}_2$ , JCPDS number 27-0605) and mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ , JCPDS number 15-0776) were identified, Fig. 4.

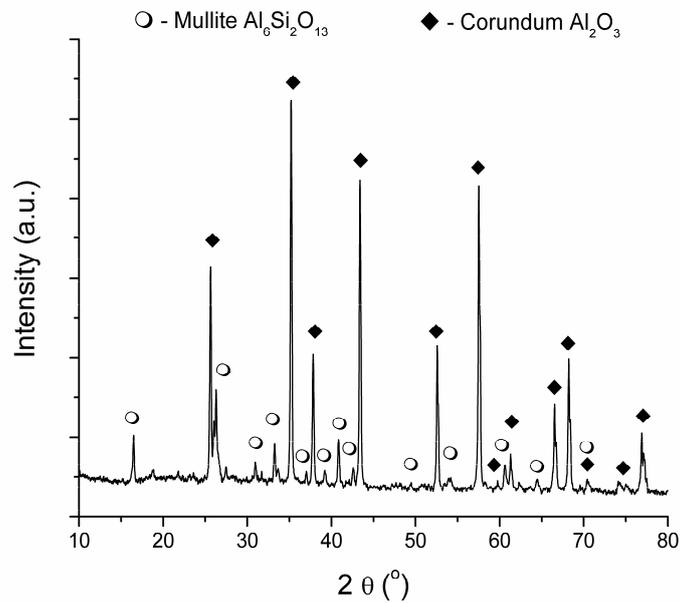


**Fig. 3.** Mineralogical composition of clay powder used for sample preparation. Phases identified in the specimen are: quartz, aluminum oxide, iron oxide and magnetite.

Bauxite powder consisted of minerals mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ , JCPDS number 15-0776) and corundum ( $\text{Al}_2\text{O}_3$ , JCPDS number 10-0173), Fig. 5. The sintered sample consisted of corundum ( $\text{Al}_2\text{O}_3$ , JCPDS number 10-0173), quartz ( $\text{SiO}_2$ , JCPDS number 33-1161), silicon oxide ( $\text{SiO}_2$ , JCPDS number 27-0605) and mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ , JCPDS number 15-0776), Fig. 6, and therefore the fibers inserted into this composition were chosen to be of aluminium-silicate (JCPDS number 37-1+60) nature so as to enable the good bonding between the fibers and the matrix.



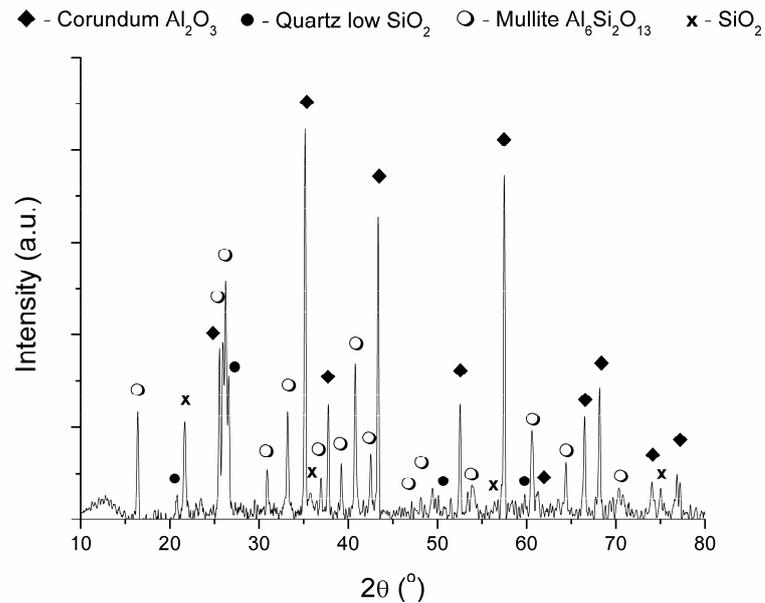
**Fig. 4.** Mineralogical composition of chamote powder used for sample preparation. Phases identified in the specimen are: quartz, silicon oxide and mullite.



**Fig. 5.** Mineralogical composition of bauxite powder used for sample preparation. Phases identified in the specimen are: mullite and corundum.

Cavitation was monitored using the mass loss measurements, as it is done when metal materials are studied and by measuring corresponding surface degradation during the experiment. The surface of specimens was photographed using the scanner in order to minimize the influence of light conditions. The surface damage was determined from the photographs according to difference in gray level in the picture. Image analysis of the

photographs of the samples surfaces was done using image analysis software that allowed to measure level of the surface damage during cavitation erosion [9-10]. Results were presented as surface erosion ratio. Mass losses of the test specimens were measured on an analytical balance with an accuracy of  $\pm 0.1$  mg. Before being weighted, the test specimens were dried in a dryer at  $110^{\circ}\text{C}$  for 1 hour. The measurements were performed after subjecting each test specimen to cavitation for 30 minutes. The overall duration of the tests was 3 hours. Optical microscopy technique was applied to analyze the effect of the erosion and to interpret the results of cavitation tests.



**Fig. 6.** Mineralogical composition of sintered sample. Phases identified in the specimen are: corundum, quartz, silicon oxide and mullite cavitation.

### 3. Results and Discussion

In Table I mechanical properties of specimens were. The compressive strengths were measured using the compression test and tensile strengths were evaluated using the Brazilian test [11]. All mechanical parameters were improved with addition of fibers.

**Tab. I** Mechanical properties of ceramic materials studied.

Samples	$\sigma$ , MPa (compression)	$\sigma$ , MPa (tensile)
Without fibers	35.81124	6.34
1% of fibers	39.11237	7.92
2% of fibers	43.68792	9.16

Mass loss during cavitation experiment was measured using analytical balance with accuracy  $\pm 0.1$  mg. For the quantification of the expected mass loss, after every cycle of experiment, samples were dried at  $105^{\circ}\text{C}$  for 1 hour [12-20]. Dried samples exhibited mass loss during cavitation experiment, which is presented in the Fig. 7. Mass loss during experiment showed the excellent correlation with the time.

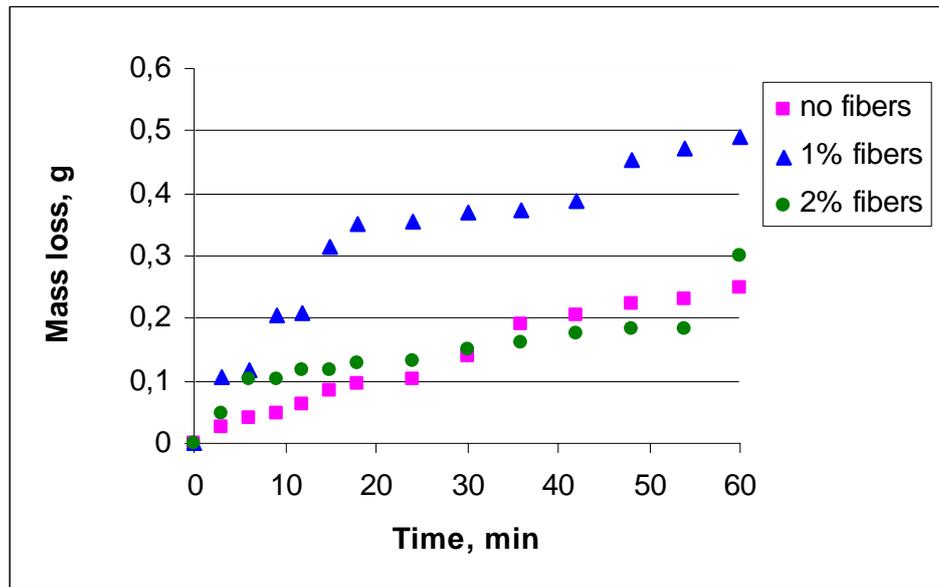


Fig. 7. Mass loss during cavitation resistance testing.

Image Pro Plus Program was used for determination of surface deterioration level. Results are given in Fig. 8. Erosion rates were measured as for the metallic samples. Image analysis program was applied for surface analysis during testing. Obtained results showed that surface erosion was in strong correlation with time, and erosion of the sample surface was about 25 % of the surface area before testing. Samples without fibers and those having 2 % of fibers exhibits similar results for mass loss. Mass loss of samples with 1% fibers was 40 % larger after testing for 60 minutes.

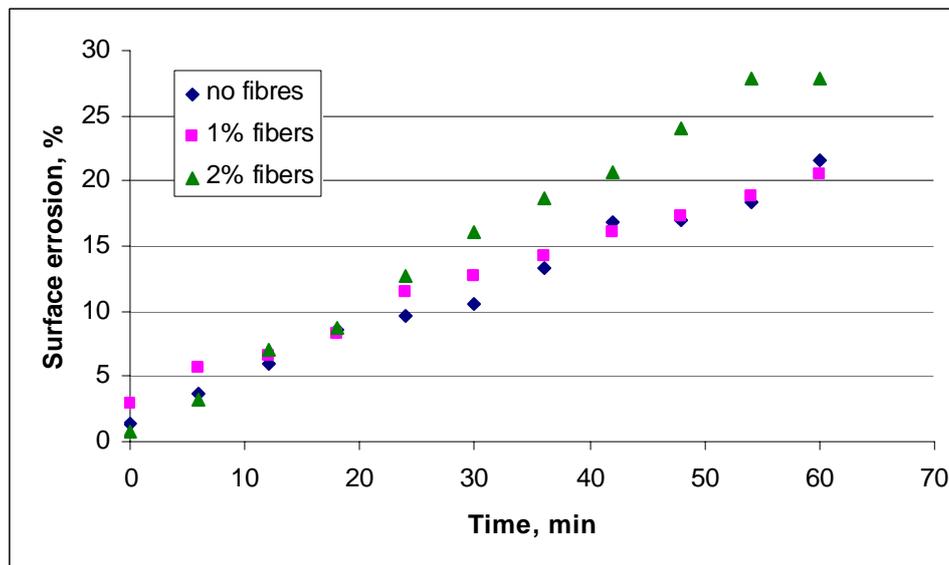
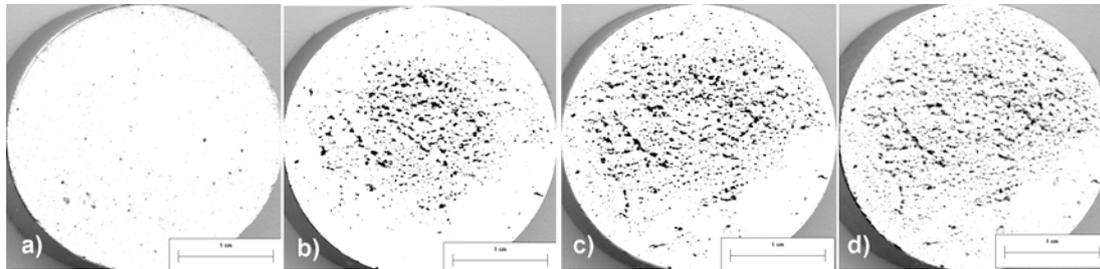


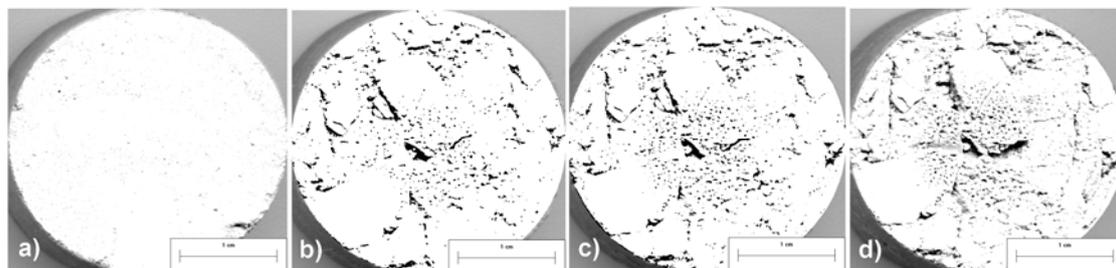
Fig. 8. Surface erosion results during cavitation determined using image analysis technique.

Results of surface erosion are presented in Fig. 8 and compare the destruction of surface in specimens having no fibers and having the addition of 1% volume and 2% volume fibers. The effects of fiber addition on surface preservation were not as beneficial as for mechanical properties. The mechanical properties are improved with fiber addition, but erosion rates and overall erosion levels were lower in specimens having no fibers than in

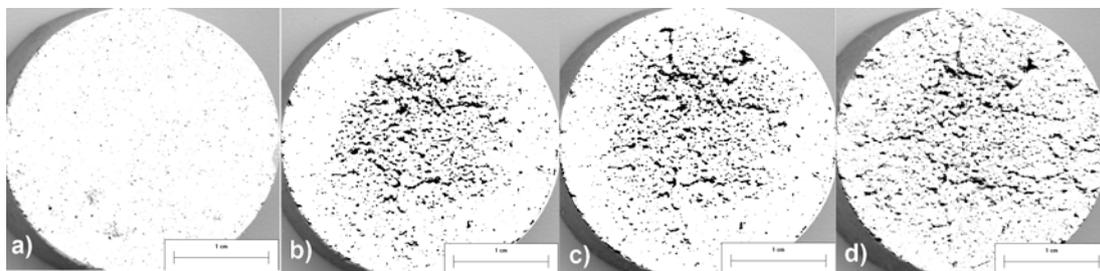
specimens having fibers in their composition. Cavitation resistance could not be significantly improved using the addition of short fibers of given length to diameter ratio.



**Fig. 9.** Samples without fibers before and during testing (before, after 24, 42 and 60 minutes).



**Fig. 10.** Samples with 1 % fibers before and during testing (before, after 24, 42 and 60 minutes).



**Fig. 11.** Samples with 2 % fibers before and during testing (before, after 24, 42 and 60 minutes).

During experiments where surface erosion was monitored, samples without fibers, and with 1 % of fibers exhibited similar behavior. These results are different if results for mass loss are considered (Fig. 7.). Samples with 1 % fibers exhibited larger values for surface erosion, but not as different as in monitoring mass loss during experiment. If the results of mass loss and surface degradation are compared, it is clear that the surface for specimens having 2% of fibers exhibit larger surface degradation and less mass loss. The depth of surface destruction which is less important when 2 % of fibers are added into the composition compared to the destruction of surface having no fibers added. So fiber addition has influence on the morphology of eroded surface defects giving shallower defects compared to specimens having no fiber addition.

#### 4. Conclusion

Synthesis of improved refractory (alumina based) materials and cavitation resistance characterization were the goal of this work. Obtained conclusions were as follows:

- Samples without fibers, as well as with content of 1% and 2 % fibers exhibited very good cavitation resistance behavior.
- Influence of fiber content was more significant for mass loss monitoring, than for surface erosion during testing.
- Results for surface erosion showed similar behavior of the samples without fibers and with 1 % fibers.
- Surface erosion for specimens having no fiber addition resulted deeper defects compared to those obtained with specimens having 2% of fibers added.

Applied nondestructive method (image analysis) showed advantages in monitoring samples behavior during cavitation resistance testing. Results obtained by non destructive measurements give much better possibility of monitoring the sample behavior during testing. Measuring the degradation level is a convenient method for prediction of material behavior, it is also a good tool for enabling optimum design where cavitation resistance is to be included and monitored in exploitation.

#### Acknowledgements

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**Садржај:** *Алуминозни материјали, којима је због побољшања својстава додат различит садржај влакана на бази алуминијум оксида, коришћени су у испитивањима отпорности на кавитацију због могуће примене у сличним условима. Кавитациона оштећења алуминозних узорака тестирана су модификованом ултразвучном методом за испитивање брзине кавитационе ерозије. Мерена брзина ерозије базирана је на методи која је развијена за металне узорке, а током експеримента мерен је губитак масе и промена оштећења површине. Анализа слике примењена је за површинску анализу током тестирања. Резултати показују да испитивани материјал има одлична механичка својства и веома добру отпорност на кавитацију.*

**Кључне речи:** *Композити, влакна, анализа слике, метода X-зрацима, прелом*

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