SURFACE ANALYSIS OF COMPOSITE MATERIAL EXPOSED TO EXTREME CONDITIONS

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The purpose of this study was quantitative analysis of material surface degradation by program for image analysis. Composite material, alumina based low cement concrete, was synthesized, cured, and then sintered at 1600 °C with the dwell time of 3 h. Prepared samples were subjected to the influence of following extreme conditions: thermal shock, cavitation, and low level laser (LLL) beam. Surface degradation was monitored during the exposure to the above tests. The results show that surface of the samples exhibited minimal level of destruction during all the tests.

Keywords: composite material, cavitation, thermal shock, LLL

1. Introduction

Refractory concretes are considered as composite materials since they consist of coarse phase (aggregate) and matrix (filler, additive, binder). They are often applied under critically high temperatures in complex constructions, thin sections and hard-to-reach areas. Since low cement refractory concretes have excellent mechanical properties, great wear, corrosion, and thermal shock resistance, low open porosity and permeability, it can be expected that they will be very suitable for application in extreme conditions, such are thermal shock, cavitation, and LLL beam exposure. Recently, there have been some studies focused on ceramic materials that are high temperature refractory ceramics, and their characterization for nuclear, electric, and electronics applications. Due to their exceptional thermal stability, superior mechanical strength, chemical stability, and dielectric strength, it can be assumed that these materials can find application as a material for electrical insulation for electric / hybrid electric vehicles. Besides, good wear resistance of material exposed to the cavitation test indicates that the material can be used for producing the turbine blades, as well. Also, monitoring the behavior when they are exposed to the excimer laser radiation increasingly attract the attention of the researchers worldwide. New approach to the analysis of material exposed to the extreme conditions was applied. This new approach has involved an important method for assessing damage of materials, the non-destructive image analysis of the specimen surface changes [1—4]. The Image Pro Plus program was used for image analysis to monitor the development of surface damage in alumina based low cement concrete. The photographs of samples were taken before and during thermal shock, cavitation and LLL beam tests in order to observe the differences between undamaged and damaged surface of the material. All results were calculated and compared to the ideal surface and finally they were presented in percentages ($(P/P_0)100\%$). Since certain level of degradation occurred at the surface and inside the bulk even before tests, P_0 was determined according to the ideal facets of 4×4 cm in case of surface degradation monitoring.

2. Experimental

Composite alumina based material was prepared by using tabular alumina as an aggregate, fine and ultrafine alumina as a filler, dispersed alumina as an additive and calcium-aluminate cement as a binder. All components were mixed with water (< 5 %), cast, cured, dried and finally sintered at 1600°C [3]. Prepared samples were subjected to the thermal shock, cavitation, and laser beam effect.

Test of thermal shock resistance which implies behavior of the material in conditions of cyclic temperature changes was realized by water quench test (ISC 81.080 SRPS BD8.308). Each cycle consisted of several consequent steps: slow heating up to the quench temperature set at 950°C, keeping samples at this temperature for 30 minutes and finally the quenching into the water bath at temperature of 23°C. Conventional testing is based on the cyclical heating and cooling till the samples' surface degrades up to 50% of the original surface. This method is rather subjective since it does not provide quantitative measure of material degradation, but only visual control. Therefore, by using the non-destructive method such as image analysis, it was possible to quantify the changes in the surface [1—3].

The laboratory testing of the material cavitation resistance was performed by modified vibratory cavitation test method. The setup consisted of a high frequency generator of 360 W, an electro-strictive transducer, a mechanical vibrations transformer and a water bath containing the test specimen. The same conditions developed for the metallic materials testing were applied to the tested refractory concrete. Total duration of the cavitation test was 3 h with analysis periods after each 30 minutes [1—3].

Continuous non-polarized semiconductor laser with MGL-S-532 integrated collimator from “CNI” Company was used in experiments for laser beam exposure. Power in a stationary temperature regime was measured using Laser Check from "Coherent" Company and was 366 mW. The wavelength was 532 nm. Beam profile was oval with the axis ratio of 3:2, with a Gaussian distribution of power. Focus was placed on the sample surface using built-in optics and achieved beam major axis length of 0.46 mm. Laser beam was pointed to different places marked on the samples. After the plugging in, the laser was working for 10 minutes before starting experiment related to the behavior of the samples. Different times, from 5 to 25 minutes, with interval of 5 minutes were selected to investigate the resistance of samples to LLL beam [4].

Image analysis, non-destructive method for macro and micro structural characterization, was used in order to monitor surface damages caused by the thermal shock, cavitation, and LLL beam. Image Pro Plus special software program enables work in all known formats of images, automatically measures, counts and classifies all data obtained about analyzed objects. Results were presented as surface degradation ratio during the testing time [1—3].

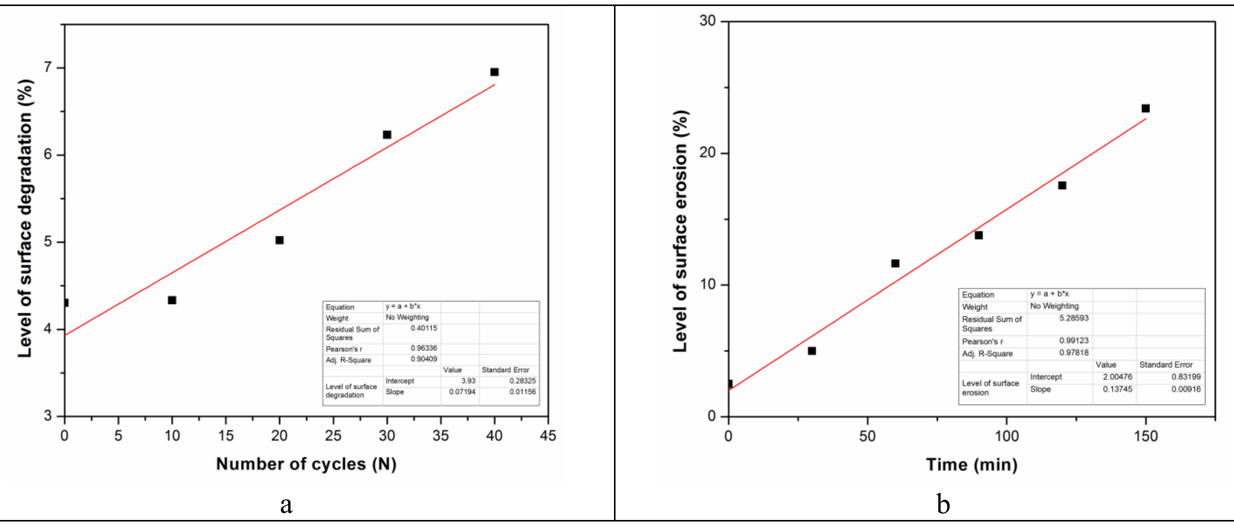
3. Results

Photographs of the tested samples' surfaces were taken and analyzed. Analysis was based on detection of differences between damaged and undamaged areas of the material. All results were calculated and compared to the ideal surface. Finally, the results were presented in percentages $(P/P0)\cdot 100\%$.

The results of image analysis showed that the certain levels of surface degradation was evident even before the tests began, which can be explained by the fact that after the preparation of concrete its structure has irregularities and defects such are pores, micropores and cracks that are considered as undesirable. It can be expected that behavior of the samples during the tests was significantly affected by the initial levels of material degradation.

Test results on the surface degradation level during thermal stability and cavitation tests depending on the number of thermal shock cycles and time of cavitation exposure are shown in Fig. 1.

The degradation level of sample surface before thermal shock testing was 4.3%, while after 40 cycles it was 6.95% (see Fig. 1, *a*). It is obvious that surface degradation during thermal shock was not dominant and the spalling of the surface typical for refractories was negligible. Linear regression analysis and slope of the curve proved that degradation was happened very slowly. Previous studies [1—3] showed the presence of $CaO\cdot Al_2O_3$ (CA_6) phase which is minority phase presented and distributed within the Al_2O_3 (A) phase, abounded phase in the matrix. Since both phases had very small values of the thermal expansion coefficient, which is an indicator of good thermal stability, such material behavior was expected.



a b

Fig. 1. Surface damage during thermal shock (*a*) and cavitation (*b*) testing

Results on the level of surface degradation during the cavitation testing are shown in Fig. 1, *b*. The obtained results show good correlation (R^2) of the surface erosion level with the cavitation exposure time, as well as linear trend. After 150 min of exposure to the cavitation, surface erosion level was below 25% presenting small damage during the testing. The results suggest that this material can be a good candidate for application where the cavitation resistance is required.

Damage level of the samples' surface exposed to LLL beam was characterized by the image analysis of the affected pit area using Image Pro Plus program. The results for the pith area measurements are presented in Fig. 2.

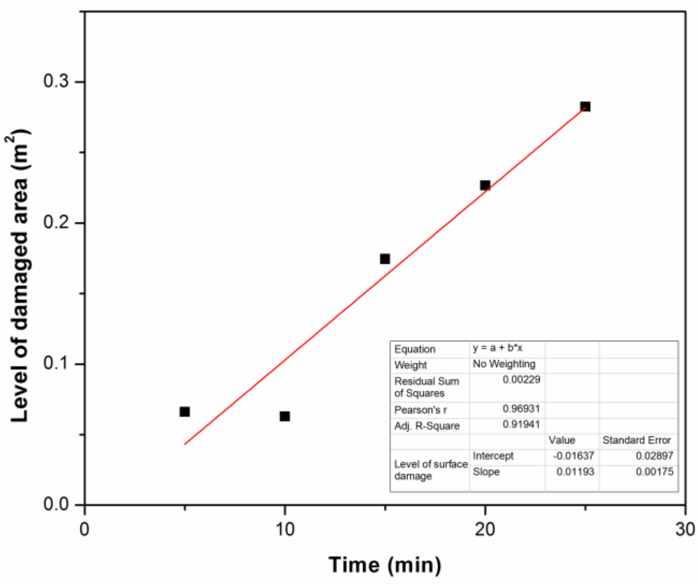


Fig. 2. Level of surface damage during exposure to LLL beam

As expected, level of damaged area was very small for a short period of time of 5 minutes. Damage level was increasing with prolongation of the LLL exposure time. Based on the obtained results, it can be noticed that the samples sintered at 1600°C exhibited excellent resistance to the influence of LLL beam. According to the previous investigations of the same material, this behavior can be related to the formation of CA_6 phase, which is expected for the sintering temperatures of over 1500°C.

Conclusions

Image analysis as a non-destructive testing method was applied in order to monitor surface level degradation of the material exposed to the extreme conditions. Tested samples have proven suitable for use in conditions that require good thermal stability as they withstood more than 40 cycles of thermal shock without cracking, while the level of material degradation was considerably below 50%, even 25%. Tested material has shown good cavitation resistance since the level of surface degradation after 150 minutes of cavitation exposure remained below 25%. Negligible surface damage was noticed at the surface of the material during the exposure to a low level laser beam. CA_6 phase is responsible for good thermal stability, cavitation resistance and excellent resistance to the impact of LLL beam. This study allows concluding that image analysis is a promising non-destructive method for damage characterization of a material caused by thermal shock, cavitation and LLL beam exposure. Beside its common applications, the results indicate the possibility of successful usage of this material for electrical insulation of electric / hybrid electric vehicles, as well as for producing turbine blades.

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Анализ поверхности композитного материала, подверженного экстремальным воздействиям.

Целью этого исследования является количественный анализ деградации поверхности материала с использованием программ для анализа изображений. Синтезированный композитный материал, бетон с низким содержанием цемента на основе глинозема, был подвергнут термоотверждению, а затем спеканию при 1600°С в течении 3 ч. Готовые образцы подвергались воздействию следующим экстремальным условиям: термический удар, кавитация, низкоуровневый лазерный пучок. При проведении вышеперечисленных испытаний контролировали деградацию поверхности. В результате исследования было обнаружено, что поверхность образцов подверглась минимальному уровню разрушения во всех экспериментах.

Ключевые слова: композитный материал, кавитация, термическая стойкость, LLL.
