MME SEE CONGRESS 2023

5th Metallurgical & Materials Engineering Congress of South-East Europe
Trebinje, Bosnia and Herzegovina
7-10th June 2023

CONGRESS PROCEEDINGS

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Dr. Branislav Marković
Institute for Technology of Nuclear and Other Mineral Raw Materials

prof. Dr. Vaso Manojlović
Faculty of Technology and Metallurgy, University of Belgrade

Technical editor:

M. Sc. Gvozden Jovanović
Institute for Technology of Nuclear and Other Mineral Raw Materials

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PRINCIPAL COMPONENT ANALYSIS OF MORPHOLOGICAL DESCRIPTORS FOR ASSESSMENT OF SURFACE DEFECTS INDUCED BY EXTREME CONDITIONS

Sanja Martinović[1], Ana Alil[2], Aleksandar Savić[3], Dragomir Glišić[4], Dragana Živojinović[4], Tatjana Volkov-Husović[3]

e-mail: s.martinovic@ihtm.bg.ac.rs

1-University of Belgrade, Institute for Chemistry, Technology and Metallurgy, Belgrade, Serbia,
2-University of Belgrade, Innovation Center of the Faculty of Technology and Metallurgy, Belgrade, Serbia,
3-University of Belgrade, Faculty of Civil Engineering, Belgrade, Serbia,

Implementation of statistical analysis for monitoring the formation and propagation of defects that occurred due to exposure of the material to thermal shock and cavitation is presented in this study. Results of two groups of materials are shown: refractory concrete sintered at three temperatures and ceramic materials based on cordierite and zircon. Alumina-based refractory concretes were subjected to thermal stability test, while the surface morphological changes regarding the defects were monitored after a certain number of thermal shock cycles. Cordierite and zircon based materials were initially subjected to cavitation testing while after specified time periods, morphological changes in the surface defects were analyzed. Besides the standardized methods, non-destructive testing of materials was applied for monitoring the surface changes. Cameras, microscopes, and numerous software enable recording images of sample surface with appropriate magnifications as well as monitoring and quantifying the surface damage degree with various image analysis tools. Thus, this approach offers the quantification of selected morphological descriptors regarding the defects. Obtained values were then subjected to pattern recognition method, the principal component analysis that can provide information on extracted morphological descriptors by describing the main difference among observed defects and identifying variations among them. As a result, a better insight into surface degradation that occurs during exposure to extreme conditions was obtained. The multivariate analysis provided grouping and thus reduction of the descriptors while maintaining the highest percentage of variance unchangeable. More precisely, the analysis extracted the most informative descriptors that should be observed for monitoring changes in surface defects and determining a degradation mechanism.

Keywords: Thermal shock, Cavitation erosion, Surface defects, Morphological descriptors, Principal component analysis (PCA)

Introduction

In real operating conditions, many equipment parts are exposed to harsh and extreme environments, thus causing the occurrence of defects and cracks, their growth, and finally leading to failure and breakage. Therefore, it is of great significance to assess their endurance the lifetime of such material used in equipment parts production. This study presents the results regarding behavior of the two different types materials under two different extreme conditions to which each one is usually exposed. The paper will present an approach by using two contexts.

The first context is engaged with the refractory concretes that are usually used in wall linings of industrial furnaces and other thermal reactors. The extreme conditions it is exposed to are cyclic and sudden temperature changes of different intensities, often accompanied by the effect of additional loads. Such uneven cooling and heating to which the material is exposed is called thermal shock. The different parts of the walls expand and shrink, causing internal stresses that induce crack nucleation and growth, loss of strength, and fracturing of the wall. Therefore, it is very important to have information about the behavior of refractory concretes during thermal stresses caused by thermal shock [1,2].

The second context belongs to the study of two ceramic materials, based on cordierite and zirconium silicate, exposed to the cavitation. Both materials are chosen for their wide use as coatings or fillers for lining the walls within the metallurgical aggregates.

The extreme conditions they are subjected to are the abrasion caused by dust, loading charge materials, molten slag, and molten metal.

For these reasons, it is very important to examine the installed material on cavitation resistance. During cavitation, the material surface erodes, resulting in the creation of numerous cavities or pits that are randomly distributed. Formed defects have various dimensions, shapes, and depths that increase, propagate, and merge into larger ones over time. The presence of such defects in the structure is considered undesirable because they greatly deteriorate the material properties [1-3].

This study presents a method to quantitatively monitor the changes in morphological descriptors of defects using image analysis, while their processing by the statistical method offers the extraction of the most informative parameters that should be observed for lifetime prediction purposes. The chosen procedure is a multivariate analysis of a large dataset to recognize and extract the most informative and representative parameters using pattern recognition techniques, such as principal component analysis (PCA). Namely, PCA can provide information on extracted morphological descriptors, it describes the main difference among the observed defects and identify variations among them. This technique is not only useful for reducing the dimensionality of such datasets but also for providing simple visualization of relationships among variables. Basically, PCA is an unsupervised pattern recognition technique used to reduce the n-dimensional space of morphological descriptors to a low-dimensional space (two-dimensional in this case) of maximal variance. Principal component scores make it possible to recognize differences among the objects (i.e. internal structure of the dataset) while loading values enable the extraction of morphological descriptors that are the most informative for the dataset structure. Consequently, the most informative descriptors can be used to characterize not only the thermal or cavitation resistance of the material but also the structure of the material [4-6].

Materials and methods

Low cement castable, as well as cordierite and zircon based ceramics, were synthesized, cured, and sintered according to the procedures given in previously published study [1-3,6]. Refractory concrete was prepared using tabular alumina as an aggregate, whereas the matrix was composed of fine fractions of tabular alumina, reactive alumina, dispersing alumina, and calcium aluminate cement. The fresh castable mixture was cast in the shape of cubes for sample preparation (4×4×4 cm). After demolding, the samples were cured and then sintered at 1100, 1300, and 1600 °C for three hours. Cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) as the refractory coating was synthesized using kaolin, alumina, and sepiolite in powdered form. The components were firstly homogenized, then pressed under the pressure of 1 MPa, and finally sintered at the temperature of 1200 °C for 2 h. Zircon was obtained by mechanical processing, purifying, and milling of zircon sand. The samples were obtained by pressing the prepared powder with 106 Pa and afterward sintered at a temperature of 1200 ºC.

The thermal shock tests were realized by water quenching according to the standardized procedure (ICS 81.080 SRPS B.D8.308). The standard procedure for determining the material resistance to thermal shock includes only a visual inspection based on a subjective assessment of the sample's surface, with an estimate of the testing end when 50 % of the original sample surface was degraded. Such a method requires a large number of samples for obtaining statistically representative results, while many samples become useless for further testing, as they are already destroyed. Besides, the method does not provide quantitative measures of degradation. Therefore, the authors have made an effort for years to improve the standard procedure through the implementation of various non-destructive methods. This study proposes using of image analysis to provide a quantitative measure of the damage level during the thermal shock.

The cavitation erosion test was performed by using an ultrasonic vibratory method (with a stationary specimen) according to the standard procedure ASTM G32 with operational parameters that are taken from the recommended standard values. The total duration of the tests was 80 min.

The standard procedure for determining the material resistance to cavitation implies measuring the mass loss during the testing.

Defects that occurred during both tests were characterized by morphological descriptors, through their determining and quantifying using software for image analysis. The basic elements of methodology regarding the morphological analysis of defects are partially shown in earlier published papers [1-3,6]. High-resolution images (1200 dpi) of the sample surfaces were scanned and processed by the Image-Pro Plus (IPP) 6.0 software package (Media Cybernetics, 2006, Rockville, MD) after each certain time period of testing. The IPP allows extract damaged areas from the surface and quantify the selected morphological descriptors of defects.

Morphological parameters that determine and describe defects (shape, area, perimeter, roundness, etc.) present carriers of information that can be used for further analysis of material resistance to thermal shock or cavitation, based on comparing the differences in defects morphology in defined time intervals. The parameters that were measured and were found useful for describing the extracted defects since providing the most information, and therefore are suitable for further analysis are as follows: Area, Area (Polygon), Aspect ratio, $\text{Axis}_{(\text{minor and major})}$, $\text{Diameter}_{(\text{max, min, mean})}$, $\text{Density}_{(\text{max})}$, Hole Area, Hole Ratio, $\text{Radius}_{(\text{ratio, min, max})}$, Perimeter, Perimeter 2, Perimeter 3, Roundness, Fractal dimension, Box width, Box height, $\text{Size}_{(\text{length and width})}$. The IPP allows export of all measured data directly into Microsoft Excel, while as a result, large datasets of measured values are obtained and can be subjected further to the statistical and graphical procedures. Data collected in such a manner presents numerous datasets of selected morphological parameters that are then processed using multivariate analysis, for which PCA is the most suitable. The aim was that measurable characteristics of selected morphological descriptors related to the induced surface defects were subjected to a PCA in order to recognize patterns in high dimensional data and extract the main difference or similarities within observed datasets as well to identify variations among them and reduce the number of measurable parameters (selected morphological descriptors). The PCA, as a nonsupervised technique, reduces dimensionality of original data matrix retaining maximum amount of variability allowing relationship between variables and recognizing data structure. This is a suitable tool to reduce the dimensionality of large experimental data sets providing easy visualization of relationships among variables. PCA was performed using the IBM SPSS Statistics 19 software package (IBM SPSS Statistics, Chicago, IL, 2019).

Results and discussion

Before and during testing, sample surfaces were scanned, Figure 1. Material surface that is exposed to cavitation erodes, randomly distributing numerous cavities or pits. In the case of thermal shock, cracks appear not only at the material surface but also in the internal structure. However, this study only focuses on changes at the surface. Such formed defects in both cases will grow until they reach a critical level, causing the material to degrade and eventually break. Formed defects have various dimensions, shapes, and depth that increase, propagate, and merge into larger ones over time. Therefore, it is important to determine which particular parameters of defect are essential. Images of the sample surfaces are subjected to image analysis using software tools to distinguish damaged and undamaged areas, Figure 2. This procedure is performed automatically based on the detection of differently colored damaged and undamaged parts appearing on the samples surface using groups of characteristic color pixels. All surfaces areas of cubes subjected to thermal shock were analyzed, while examined eroded area of samples exposed to cavitation was outlined in red. Morphological parameters that describe surface defects have been subjected to quantification and analysis in order to determine which ones can best characterize certain damages.

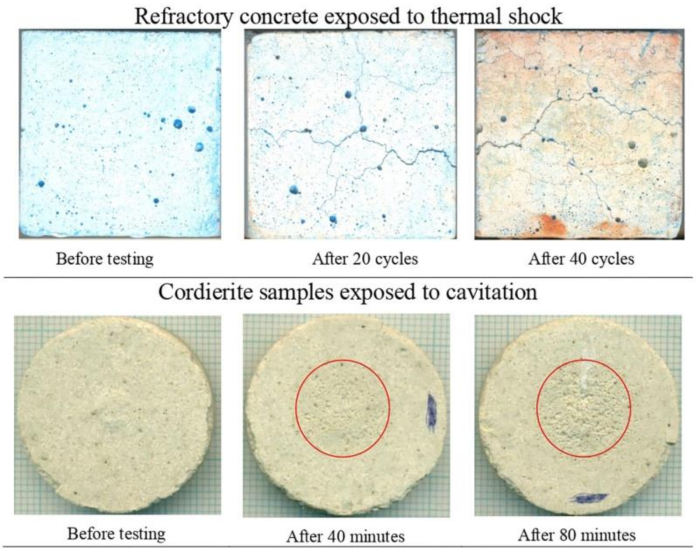

Figure 1. Samples image during thermal shock and cavitation testing.

IPP uses tools for automatic measuring, counting, and classifying data about analyzed objects, and also communicate directly with the Excel, thus creating large datasets of measured values that can be subjected further to statistical and graphical procedures, Figure 2.

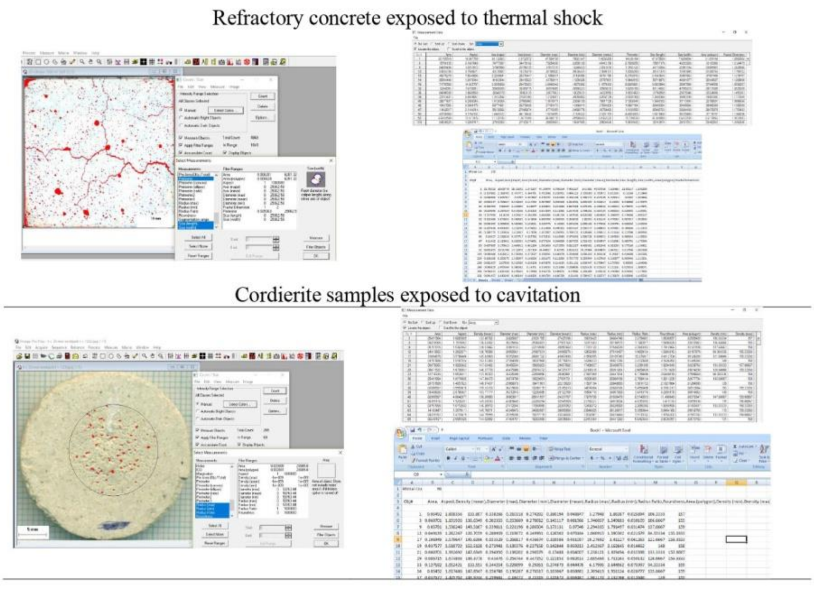

Figure 2. Screen shot of window during image analysis procedure.

The first context studied the refractory concretes exposed to thermal shock. Selected defects were analyzed by 15 morphological descriptors and the resulting data were grouped into three datasets according to sintering temperature. There was an additional grouping as a function of exposure to thermal shock cycles in order to correlate morphology with the properties. The PCA of morphological descriptors after 35 thermal shock cycles showed that samples sintered at 1100°C and 1300°C have similar damage morphology compared to samples sintered at 1600°C, Figure 3. The PCA extracted several morphological descriptors, and results showed that the most informative ones for separability among three series were $Density_{max}$, $Density_{mean}$, and Aspect, due to their highest loading values along the PC2-axis.

Aspect was related to the size and shape of defects, while Density was related to the depth of damage.

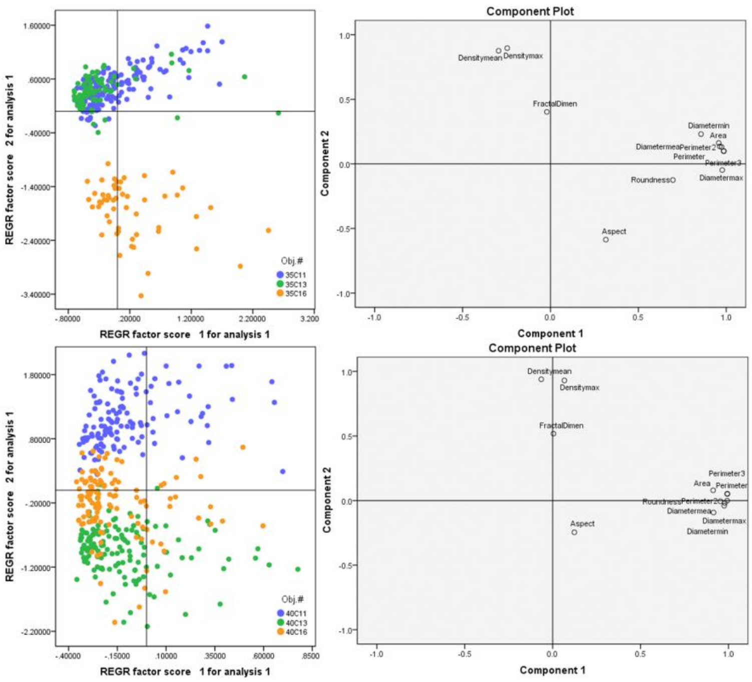

Figure 3. Percentage of preserved variance along the axis and loading plots in PC1-PC2 space[2].

The samples sintered at 1600 °C showed the highest variability of Aspect indicating that surface defects developed after the 35th cycle of thermal shock differing much more in size and shape than depth. Based on the PCA of morphological descriptors after 40 cycles, it was obvious that although the most informative descriptors remained the same, some changes in material's response to thermal shock were evident.

The second context studied ceramic coatings exposed to cavitation. For illustrative purposes, PCA score plots for cordierite samples after 40 and 80 min of cavitation testing are shown, Figure 4.

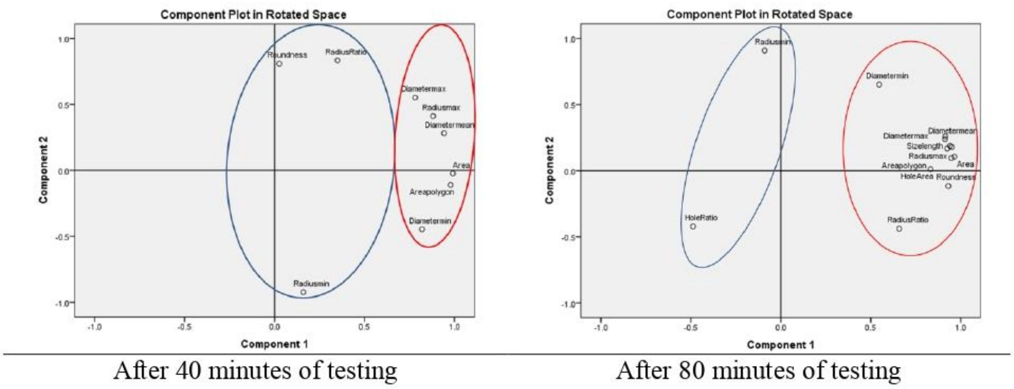

Figure 4. Loadings plots in PC1-PC2-space for cordierite based samples exposed to cavitation [3].

The original dataset of measured quantities was subjected to PCA with the aim of revealing relationships among the parameters per a specific time periods. Data reductions were performed by transforming the data into orthogonal components that were a linear combination of the original variables.

Dimension reduction was performed to PC1-PC2 two-dimensional space while retaining maximal variance to enable easy visualization of dataset's structure and to emphasize differences between different material structures. PCA analysis was realized on the dataset of values obtained for isolated damages and selected morphology parameters for each period of cavitation exposure.

As different materials are tested, different approaches were applied to select morphological descriptors. For both tested types of materials and for most of the testing period, the most informative parameters are Area and Area polygon, as these variables have values close to 1 and correlate strongly with PC1. This observation indicates that these parameters should be followed up in order to monitor changes in surface defects. The parameters that can be excluded are outlined with blue color from the further analysis since these variables have values closer to 0, indicating a weak influence on the PC1. More precisely, they did not find it sufficiently informative because they did not carry data that can accurately describe the defects.

Conclusion

The analysis that was carried out indicates the importance of the proposed approach in industrial or engineering practices since proposes applying the PCA technique to monitor the defects that occur on the material surface during exposure to extreme conditions such are thermal shock or cavitation. The PCA provides an effective tool for quickly and inexpensively monitoring, diagnosing, and predicting the level of complex changes occurring on the surface of components exposed to such conditions. A particular benefit of this method is that it offers the ability to monitor components that are difficult to access and/or require disassembly complex setups to check the level of material degradation.

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