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Comparison of Mechanical Behavior of SiC Sintered Specimen to Analysis of Surface Defects

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Abstract:

Silicon carbide is the material that in sintered form is suitable for protection from impact as part of the multilayered structure. Its role is to accumulate energy from the projectile, to plastically deform the projectile and to protect other layers of the material. Standard procedures for material testing include structure testing, surface defect shape and size determination and mechanical testing. The obtained information about surface defects was used to calculate the behavior of the sample exposed to bending and the finite element method was used for this simulation. The obtained results are in accordance with the experimental ones.

Keywords: *SiC ceramics, Bending, Image analysis, Extended finite element method*

1. Introduction

Steel armours for military vehicles have been used for protection from impact for decades. The main problem using this sort of protection concerned the very heavy structure that is very energy consuming when the vehicle is moving. Very heavy structures are also very difficult to manipulate. Research was conducted to produce materials enabling the lighter structures and even better protection. The use of layered structures is one of the solutions that enable the safety and reduces problems associated with weight. The ceramic surface of those layered structures is of great importance for improvement of overall characteristics [1]

Ceramics such as silicon carbide have high hardness and are very resistant to compressive loads. Their main role in the protection is to absorb the energy from the projectile and to induce plastic deformation on the projectile. The ceramic material is very often broken during impact, but the structure under the ceramic remains intact. The study of mechanical behavior of this sort of material is crucial in understanding the process that is involved in the stress distribution in the material. The studies of flexural strength of SiC are oriented to determination of factors that influence the strength. The behavior of the material is strongly dependant on the morphology of pores in the material [2, 3]. Those parameters are strongly dependant on the process of preparation of the material [4]. All the SiC structures are having bulk pores that could be visualized using the image analysis.

Standard procedures for material testing include structural analysis using XRD, microstructural analysis, including scanning electron microscopy and optical microscopy in

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order to study surface defects and internal defects, density and strength of specimens. The correlation between the surface defects and observed behavior under three point bending test is the aim of this study. This is the way how the measured material properties could be incorporated into a mathematical model and how observed features could be studied in order to explain their importance for the quality testing of a material.

One of the easiest ways to study the SiC ceramics is to observe the surface and to test the values of the surface defects. The observed maximum diameter of the surface imperfection could be critical for the behavior of the material under load. In those results, it was proved that the material behavior under three point bending tests corresponds to the observed maximum diameter of the surface defects observed using microscopy and image analysis tools.

Crack initiation and propagation is modelled using the Extended Finite-Element Method (X-FEM). Thus, one of the aims of this study is to develop and validate a numerical model for analysis of fracture behavior of the silicon carbide plates under quasi-static loading [5]. This numerical procedure enables to model failure of the material without node separation or cohesive elements introduction and is therefore suitable, especially in the cases when the crack growth path is not known in advance.

2. Experimental

2.1 Material

The samples were plates of silicon carbide, thickness of 4 mm and the shape of a hexagonal prism with edge length of 17 mm. The samples were tested in order to obtain information on their mechanical properties as well as on the parameters of surface quality and composition. Tests have included scanning electron microscopy, optical microscopy, X - ray diffraction analysis, testing flexural strength of the sample and the density.

2.2 Mechanical characteristics

Mechanical characteristics were obtained using the standard compression test using the servo – hydraulic testing machine Instron 1332 with load cell of 100 kN and 5 kN with data acquisition system. Strain rate is 0.2 mm / min with a custom device that gives the possibility of determining the flexural strength of samples with a spacing of supports of 15 mm.

2.3 SEM microscopy

The morphology of the sintered sample's surface was examined using a scanning electron microscope (SEM) Jeol JSM 5800, operated at 20 kV. Images were captured with a magnification of 800 ×. The external surface and the fracture surface of the samples were tested.

2.4 Image analysis

Image analysis was used to determine dimensions, shape and distribution of the pores. The Image Pro Plus program was used to process the images [6]. The mean diameter of the pore is the average length of diameters measured of two degree intervals and passing through the object's centroid. Maximum diameter is the length of longest line joining two points of the object's outline and passing through the centroid. The area is measured by counting the

number of pixels in the picture and correlated with the calibration value to the rapper length of the corresponding image.

2.5 Measurement of sample density

A very important feature of ceramic samples is their density. Density is an indication of the porosity of the sample and provides an opportunity to assess the internal porosity of the sample. The theoretical density of SiC is 3.21 g cm^{-3} . The density of the samples was measured using Archimedes scales and the values were obtained in the range of $3.18 - 3.22 \text{ g cm}^{-3}$.

2.6 Extended finite element method

X-FEM is a numerical method that enables analysis of crack propagation without remeshing a cracked specimen in accordance to newly created crack faces. It employs local enrichment of the approximation functions. The method can be useful for evolving process with non-smooth characteristics in small parts of a computational domain, e.g. near discontinuity or singularity regions, as in the case of cracks for which the standard finite element method is not accurate. The X-FEM was first introduced by Belytschko and Black [7]. The enrichment is realized based on the partition-of-unity concept developed by Melenk and Babuska [8]; it allows incorporation of local enrichment functions into a finite-element approximation domain. Spatial enrichment functions with additional degrees of freedom are used for modelling the discontinuities. The general framework of this method is incorporated in the finite- element software Abaqus 6.10 [9 - 11].

In this study, two finite-element models were developed: the model with defect size $8 \mu\text{m}$ and the one with defect size $12 \mu\text{m}$ (the choice of these two values is based on experimental observations, as explained in the next chapter). They are simplified two dimensional plane strain X-FEM models used to simulate the fracture of SiC plate exposed to quasi-static loading in three point bending test. The test was simulated with the finite-element software Abaqus 6.10. Quadrilateral finite elements with linear interpolation are used. Loading, as well as both supports, is modelled through the contact with rigid bodies (Fig. 1). The FE models are used to verify the applicability of the X-FEM to analysis of the failure behavior of silicon carbide plates under loading conditions. Fig. 1 shows meshed part of the silicon carbide plate, 4 mm thickness and 30 mm length. The material properties of these models are: flexural strength of 350 MPa [12], Young modulus 200 GPa and Poisson ratio 0.2, and fracture energy of 0.05 [13].

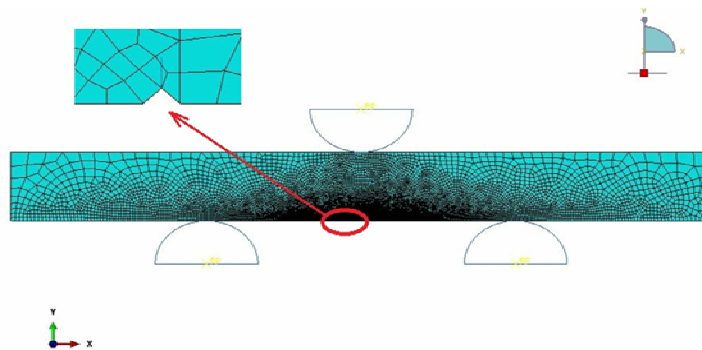


Fig. 1. Meshed silicon carbide plate subjected to three point bending test

3. Results and discussion

The analyzed sample had the shape of a hexagonal plate with thickness of about 0.4 cm. The diffractogram of the sample is shown in Fig. 2 with the identified tag and phase of the respective Miller's Index [14].

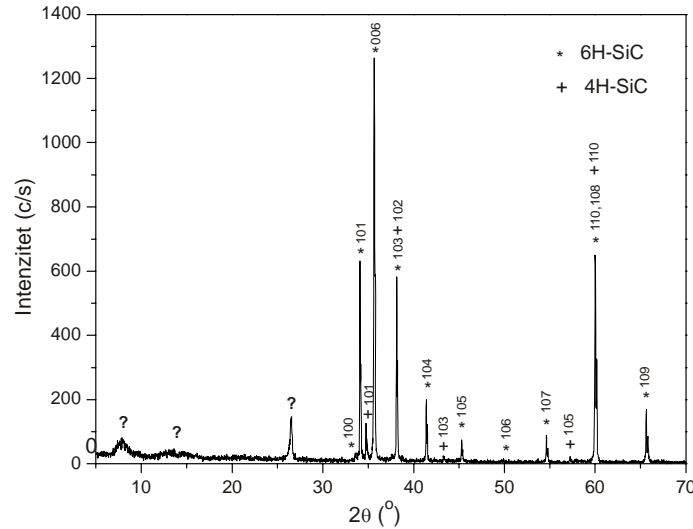


Fig. 2. The diffractogram of the test sample silicon carbide

Two phases of silicon carbide were identified in the sample. Both phases belong to the hexagonal crystal system, space group P63mc. The first and most common stage of the α -SiC, the most common modifications of SiC, with crystallographic labeled 6H-SiC, while the second phase is present indicates a 4H-SiC.

These two phases present had narrow characteristic diffraction peaks, indicating that they have a high degree of crystalline. The approximate weight ratio of the two phases in the sample is 90:10. The unit cell parameters of these phases are in good agreement with literature values.

In addition to the two mentioned phases in the sample there is at least one unidentified phase with only one clearly defined peak at 26.53° 2θ ($d = 3.36 \text{ \AA}$) (6.31 \AA) indicates the presence of a vitreous (amorphous) phase. 7.85° ($d = 11.3 \text{ \AA}$) indicates the presence of some substance poorly crystallized. At such low values, the corners of the peaks are typically silicates, but this can occur and the peaks of other phases. It is quite possible that the disclosed hill and a peak derived from the additive promote the sintering. The parameters of the unit cell were shown in Tab. 1.

Tab. I The unit cell parameters of the identified phases

Phase	The unit cell parameters (\AA)		Literature
	Experiment	Literature	
6H-SiC	a = 3,081 c = 15,119	a = 3,081 c = 15,125	156190-ICSD *
4H-SiC	a = 3,080 c = 10,119	a = 3,073 c = 10,053	24170-ICSD *

- ICSD is Inorganic Crystal Structures Database.

According to the manufacturer, additives used in the sintering of SiC were the following substances: C, B, Al₂O₃ and Y₂O₃. However, in the sample was identified none of the above compounds or any of their possible combinations.

Two samples were tested which showed different hardness values so as to determine whether there is a relationship between the difference in the strength and the morphology of the surface and cross-section. On the other hand, this was also a way to determine the quality of the surface. The results of the analysis area can then be used for comparison with the results obtained by optical microscopy. Fig. 3 shows the microstructure of samples ((a) sample 1 and b) sample 2) of the obtained cross-sectional imaging of the scanning electron microscope.

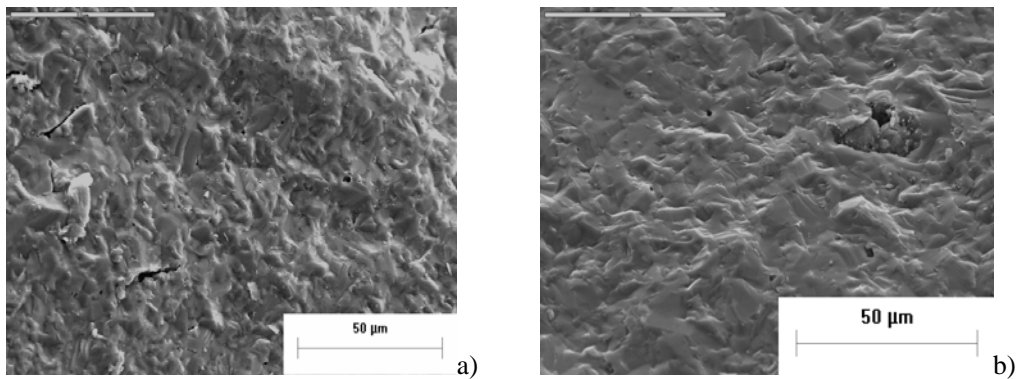


Fig. 3. SEM micrograph cross - section area of sample 1 a) and sample 2 b)

The microstructure section showing the dense sintered structure with the appearance of small pores. The dimensions of these pores corresponding to the dimensions of pores on the surface of the sample.

Fig. 4 shows the details of the microstructure of the surface of the samples ((a) sample 1 and b) sample 2).

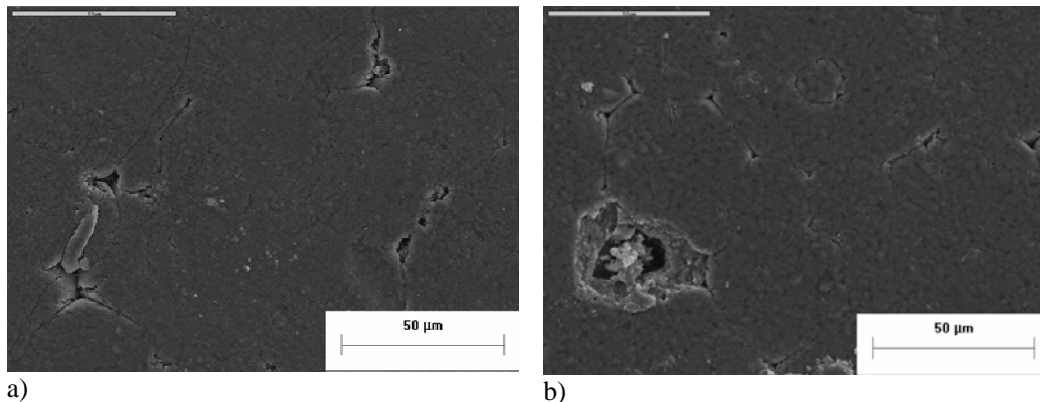


Fig. 4. The microstructure of the surface of the sample 1 a) and sample 2 b)

The pores are generally in the corners of the particle where the sintering process was slower in terms of filling the interstitial spaces. Just a sample of 2 was observed pores whose dimensions are much larger than others as evidenced by statistical data measurement, consists of two parts whose individual diameters of about 12 μm.

Tab. II shows the statistical data obtained by determining the morphological properties of the pores observed by scanning electron microscope on the surface of samples.

Tab. II Comparison of statistical indicators for the pore morphology of sample 1 and 2

Sign of value	Area of pore Sample 1	Area of pore Sample 2	Max diameter of pore Sample 1	Max diameter of pore Sample 2	Mean diameter of pore Sample 1	Mean diameter of pore Sample 2
Average value, μm	1,061	1,145	1,568	1,474	1,103	1,048
Standard deviation	0,100	0,260	0,070	0,084	0,042	0,051
Median, μm	0,465	0,470	1,183	1,163	0,851	0,860
Minimum, μm	0,229	0,232	0,621	0,653	0,546	0,585
Maximum, μm	16,22	46,91	8,408	12,08	4,745	7,797

Fig. 5 shows Histograms of characteristic defects on the surface of samples 1 and 2.

Sample 1

Sample 2

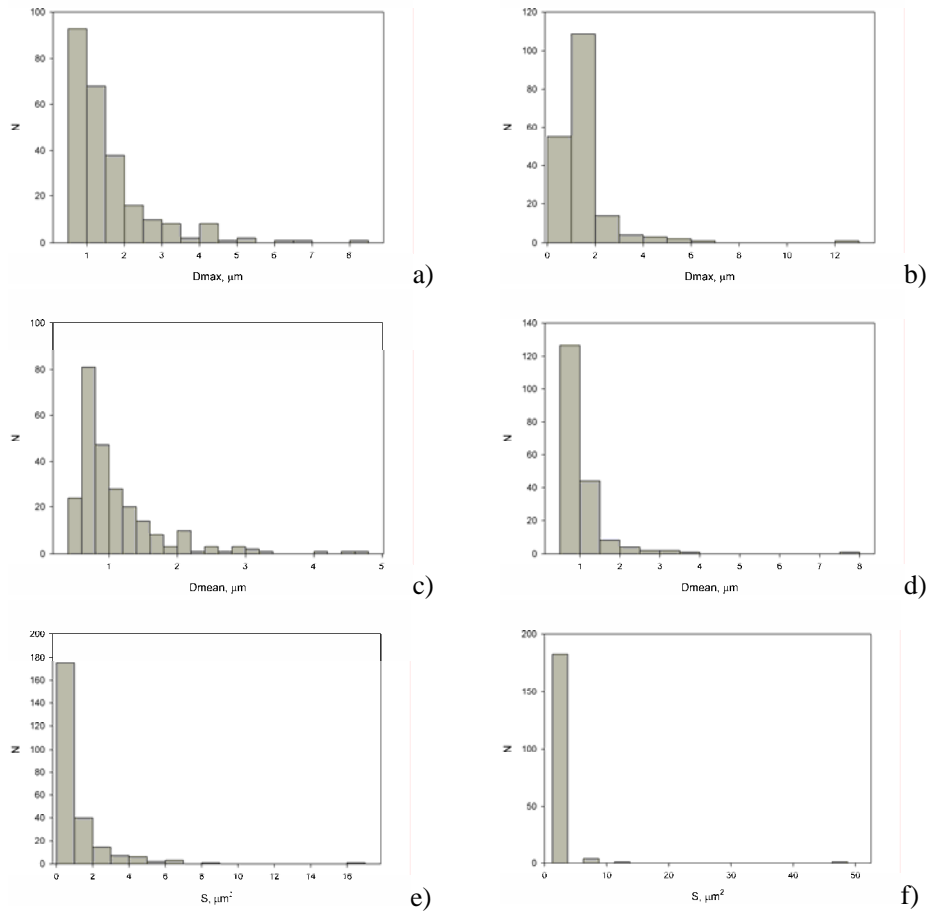


Fig. 5. Histograms of characteristic defects on the surface of samples 1 a, c, e) and 2 b, d, f)

An optical microscope was used to examine the surface of the samples. Different lenses are used for testing in order to achieve good reproducibility tests. Fig. 6 shows the microstructure of the samples captured with metallographic microscope.

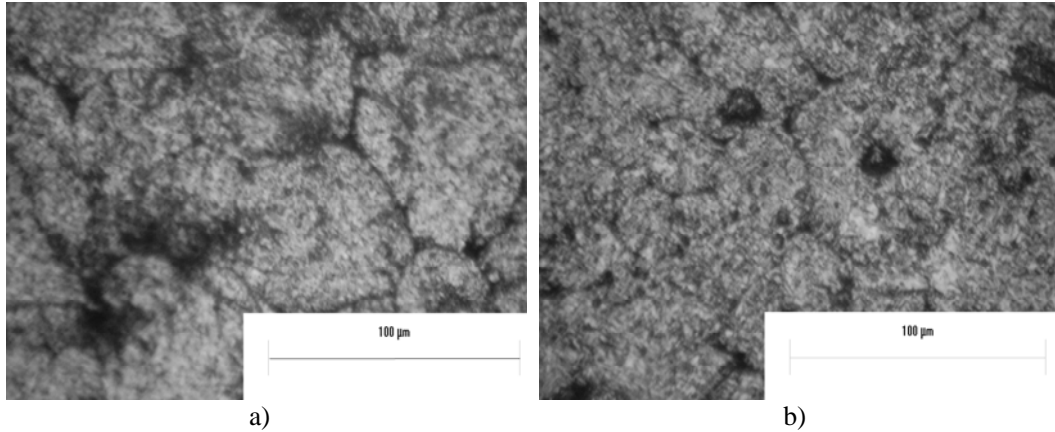


Fig. 6. The microstructure of the surface of the sample 1 a) and sample 2 b) captured with metallographic microscope

There were surface irregularities, morphologically very similar to those observed in the scanning electron microscope. The five samples were tested for determining the mechanical properties. The obtained results are given in Tab. III

Tab. III The results of the flexural strength of silicon carbide samples

	h	s	W_x	l_s	F_{max}	M_{smax}	σ_{smax}
	mm	mm	cm ³	mm	N	Nm	MPa
SiC 1	4,15	30,1	0,0864	15	9363	35,11	406
SiC 2	4,21	30,7	0,0907	15	6499	24,37	269

Diagrams obtained during tests of flexural strength are shown in Fig. 7.

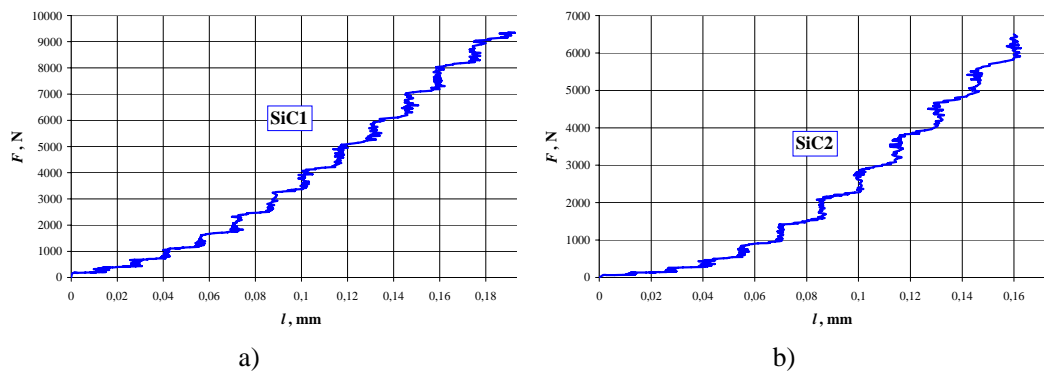


Fig. 7. Force - displacement diagram for testing the flexural strength of silicon carbide sample 1 a) and sample 2 b)

History output in Abaqus collects all the data of reaction force and displacement of the rigid body used for loading of the specimen. Diagrams force - displacement are obtained from these history output results, Fig. 8, and present the experimentally and numerically obtained results.

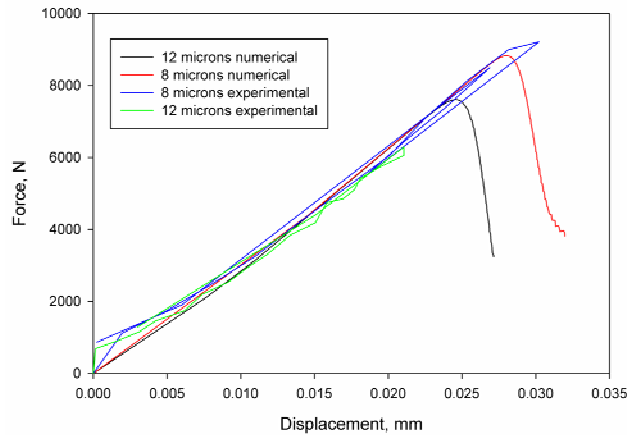


Fig. 8. Comparison of numerical and experimental diagrams

Although the differences between the numerical and experimental results exist, the model predicts the difference in deformation behavior and failure for two specimens with different pore sizes, despite the very small dimensions of the pores in comparison with plate thickness and a small difference between them (8 and 12 micrometers, respectively). Therefore, the future research will include development of more realistic models, like those with several defects or those with three dimensional geometry (i.e. without the plane strain simplification).

4. Conclusion

Non-destructive method is formed for analyzing the quality of silicon carbide, where the key parameter is measuring the greatest irregularities on the surface of the material. This is kind of a screening test that define capability of a material to bear a certain load. This ability is extremely important for the use of materials in military defense.

The silicon carbide sintered material was studied experimentally and numerically. The data about the structure and surface defects were used as the base for mathematical modeling of the behavior under bending. The results obtained for different surface defect sizes are compared to mathematical model and they enable the calculation of the behavior of the sample under bending. These data are the basis for further development of the models describing the behavior of samples under other sorts of loads. The models are also showing that the surface defects are the key factor to be determined during the quality control procedures.

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Садржај: Силицијум карбид је материјал који је у синтерованом облику погодан за заштиту од утицаја од удара пројектила као део вишеслојне структуре. Њихова улога је да акумулира енергију из пројектила, да пластично деформише пројектил и да заштити друге слојеве материјала. Стандардне процедуре за испитивање материјала укључују испитивање структуре, одређивање облика и величине површинског оштећења и механичка испитивања. Добијени подаци о површинским оштећењима коришћени су за израчунавање понашања узорка на савијање а метода коначних елемената коришћена је за ове симулације. Добијени резултати су у складу са експерименталним резултатима.

Кључне речи: керамика на бази силицијум карбида, анализа слике, метода коначних елемената
