Contact strength test of Si_3N_4 and SiC with opposite roller loading

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Abstract

The opposite-roller contact test has been used for the strength measurement of Si_3N_4 and SiC based ceramics and the results have been compared to the results of the four-point bending strength test. Ceramographic and fractographic methods have been used for the characterisation of the strength degrading defects. The characteristic strength values for the silicon nitride ceramics are very similar in contact and bending strength tests but for the silicon carbide the characteristic strength in bending mode is lower compared to the value of contact strength. This is caused by the presence of the relatively large processing flaws in the SiC ceramics. The Weibull moduli obtained for the investigated materials using contact strength method are approximately a half of the moduli of bending methods. Contact end cracks, median and lateral cracks have been found as the main damage mechanisms which lead to the fracture of the specimens in contact strength test.

K e y w o r d s: contact strength, bending strength, cylinder loading, Weibull parameters

1. Introduction

Silicon nitride and silicon carbide based ceramics are promising materials for a variety of structural applications due to their unique combination of mechanical properties (e.g. hardness, wear resistance, high-temperature strength, corrosion resistance, etc.), [1–4]. Over the last few decades, these materials were intensively developed with the aim to optimise their final mechanical properties such as strength, fracture toughness, creep behaviour, etc. [3]. The relationship between processing, microstructure and fracture/mechanical properties, reliability and lifetime were investigated in detail at different mechanical loading configurations and in different environments using different test methods [2–4]. Apart from the standard mechanical tests (hardness, strength, fracture toughness etc.), non-standard indentation tests, for example Hertzian contact tests, have also been applied to the characterisation of advanced ceramics [5]. Under the Hertzian load, the nature of contact damage is very different compared to homogeneous brittle materials (e.g. glasses) or different composites. Examination of Hertzian indentation in a heterogeneous SiC with coarse and elongated grains revealed significant transition from classical Hertzian cone fracture to quasi-plastic damage [6].

Usually, the strength of ceramic materials is measured by doing bending tests resulting in a stress state represented, mostly, by a uniaxial stress with a relatively small gradient. Considering practical applications of different tools/parts, the mechanical loading is often represented by a multi-axial stress state to be significantly non-homogeneous. In regard to the determination of the strength of ceramic materials, the multi-axial and non-homogeneous stress state can be obtained through contact loading using line or point loads. Over the last few years contact strength tests have been introduced and applied for the study of contact strength and cone crack formation of different ceramics [7, 8]. Fett et al. [8] successfully applied this approach as applicable at least in cases where the stress variations are small and sufficient constant stresses can be assumed over the size of natural flaws. This was a reason for a study, in which the effective volumes and surfaces for the two tests were compared and the influence of the strong stress gradients was considered.

Fett et al. [8] performed contact and bending strength tests on several structural ceramics and found

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that:

– linear relation exists between the characteristic strength σ_0 for bending mode and contact mode:

$$\sigma_{0,\text{bend}} \approx \sigma_{0,\text{cont}},$$

– the Weibull exponents in the contact strength (m_{cont}) tests are smaller than those of the four-point bending tests (m_{bend}) and approximately equate to:

$$m_{\text{bend}} \approx 2m_{\text{cont}}.$$

The aim of this contribution is to compare strength, strength degrading defects and fracture characteristics of silicon nitride and silicon carbide ceramics tested in the opposite roller and bending mode as well as to verify Fett's theory concerning the relationship between their Weibull parameters obtained in these testing modes.

2. Experimental procedure

Two silicon nitride and one silicon carbide ceramics have been used for this investigation. The first was a gas pressure sintered silicon nitride (SN I) with ~ 3 % Al_2O_3 and ~ 3 % Y_2O_3 additives, produced by CeramTec (Plochingen, Germany). It was provided by the manufacturer in the form of plates with dimensions of 47 × 11 × 102 mm [10]. The second material (SN II) used in this investigation was also a gas pressure sintered monolithic Si_3N_4, prepared using SNE-10 powder with addition of ~ 3 % Y_2O_3 and ~ 1 % Al_2O_3 [11]. The third material (SC1), a SiC ceramic was prepared as a mixture of the commercially available β-SiC powder (HSC-0.59, Superior Graphite) with Si_3N_4, Al_2O_3 and Y_2O_3, provided by Institute of Inorganic Chemistry SAS Bratislava in the form of plates with dimensions of 60 × 60 × 6 mm. The SiC ceramic was hot pressed at 1870 ℃/1 h in a nitrogen atmosphere and subsequently annealed at 1650 ℃/5 h [12].

In order to obtain a direct measure of failure under contact loading, two cylinders made of hardened steel are pressed onto the rectangular specimen with a force P, as illustrated in Fig. 1.

The maximum tensile stress in the specimen is reached at the upper and lower surfaces, directly at the contact of the specimen and rollers [8]. At these locations:

$$\sigma_{\max} = 0.490\sigma^*, \quad \sigma^* = \frac{p}{Ht}. \tag{1}$$

In the contact strength tests, the maximum stress value was identified as the strength. The experiments have been carried out at crosshead speed of 0.5 mm min^{-1}. With the aim to investigate the evolution of the contact damage in the specimens, they have been loaded to the approximately 70 % of the failure load and after unloading cut at the long axis. The ceramography together with SEM investigation have been used for the study of damage mechanisms.

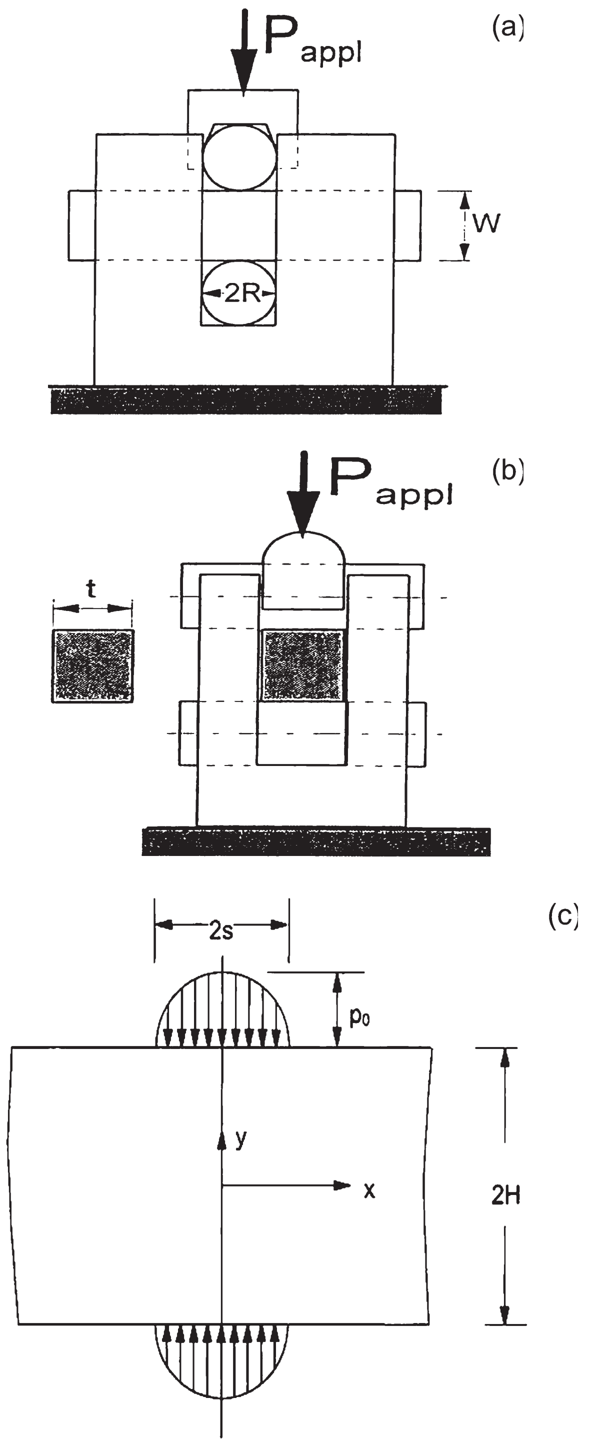

Fig. 1. A contact strength test fixture with two opposite rollers [2].

The bending strengths were determined by doing a four-point bending test in laboratory atmosphere (inner/outer span of 20/40 mm). It was applied at crosshead speed of 0.5 mm min^{-1} and evaluated by means of two-parameter Weibull distributions, yielding the characteristic bending strengths σ_0 at a failure probability of 63 % and the Weibull moduli m, which decrease with increasing statistical scattering of the bending strengths σ_0:

$$P_{\mathrm{f}} = 1 - \exp\left[-V\frac{\sigma_{\mathrm{f}} - \sigma_{\mathrm{u}}}{\sigma_0}\right]^m. \qquad (2)$$

The fracture surface of the specimens failure in bending was investigated by means of scanning electron microscopy and the types, sizes and locations of the failure-initiating defects have been identified and characterised.

3. Results and discussion

The microstructure of the Si_3N_4 material shows moderately elongated Si_3N_4 grains with the aspect ratio of approximately 3 (Fig. 2a). Some microporosity and some iron containing inclusion (approximately 0.1 wt.%) have been found in the microstructure, which is likely to have remained from the original powder. The second material investigated – Si_3N_4 – has a bimodal grain size distribution with large grains, up to 20 μm in diameter and small ones, < 1 μm in diameter (Fig. 2b). The microstructure of the hot pressed SiC material consists of the fine submicron-sized equiaxed SiC grains with a low aspect ratio (Fig. 2c). Additionally, all materials contain an intergranular phase in the form of a very thin grain boundary films and in the form of a triple point.

The characteristic strength and Weibull modulus, computed by using two-parameter Weibull distribution for all three testing ceramic materials and differences in contact results comparing to bending results are illustrated in Table 1 and Fig. 3. The ratios of the Weibull parameters are presented in Table 2.

According to the results, the characteristic strength values for the silicon nitride ceramics are very similar in contact and bending strength tests but for the silicon carbide the characteristic strength in bending mode is lower compared to the value of contact strength. The Weibull moduli obtained for the investigated materials using contact strength method are approximately half of the moduli of the bending methods. The very low strength characteristic in bending mode in the case of SiC is caused by the presence of relatively large processing flaws in this material. Such flaws with different size, shape and location (distance from the tensile surface) not only decrease the characteristic bending strength but also lead to the low

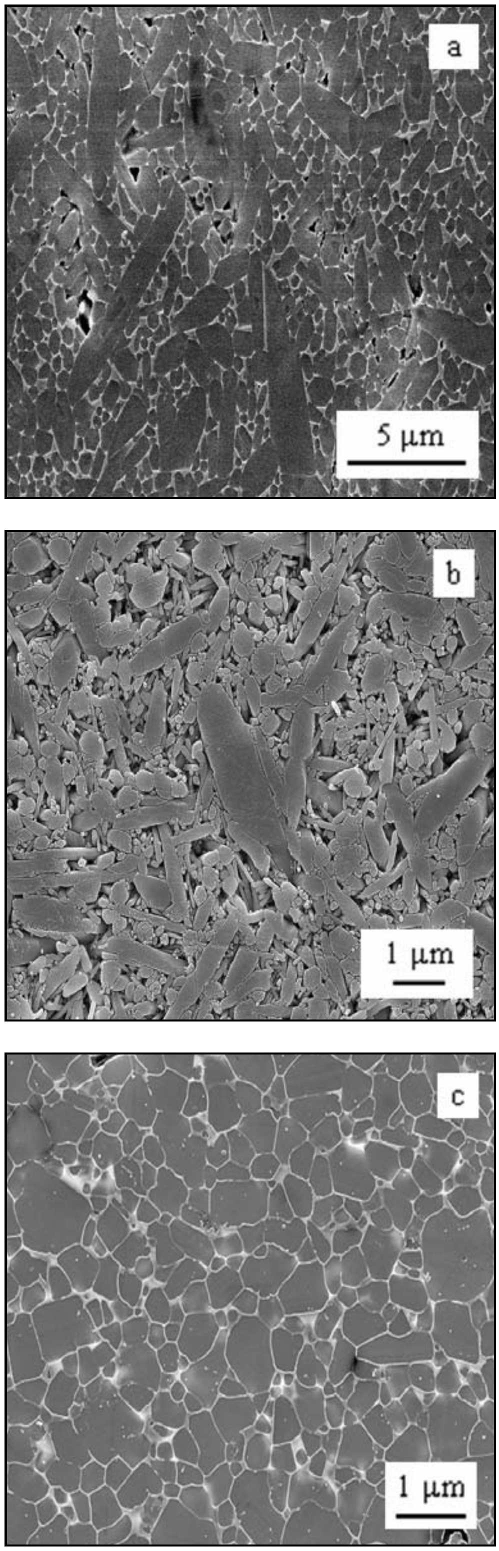

Fig. 2. Scanning electron micrographs of characteristic microstructure of Si_3N_4 (SN I) plasma etched (a), Si_3N_4 (SN II) chemically etched (b) and SiC plasma etched (c) materials.

Weibull modulus for this material. The significantly different Weibull moduli in comparison to the bend-

Table 1. Strength results for contact and bending tests with interval of results in brackets

Material	Contact results		Bending results (4P)	
	σ_0 (MPa)	m	σ_0 (MPa)	m
Si_3N_4 (SN I)	766.3 (559–826.8)	7.45 (5.6–8.2)	727.8 (596.7–767.1)	13.9 (12.9–14.57)
Si_3N_4 (SN II)	1065.1 (910–1139)	5.7 (4.1–6.52)	1012.3 (832.3–1084)	14.1 (12.22–16.14)
SiC	617.1 (511.4–707.4)	8.95 (7.25–9.89)	437.1 (347.2–485.6)	10.6 (9.4–11.12)

Table 2. The ratio of the Weibull parameters

Material	m_{cont}	m_{bend}	m_{bend}/m_{cont}	Assumption
Si_3N_4 (SN I)	7.45	13.9	1.86	2
Si_3N_4 (SN II)	5.70	14.1	2.47	2
SiC	8.95	10.6	1.18	2
	σ_{cont} (MPa)	σ_{bend} (MPa)	$\sigma_{cont}/\sigma_{bend}$	Assumption
Si_3N_4 (SN I)	766.3	727.8	1.05	1
Si_3N_4 (SN II)	1065.1	1012.3	1.052	1
SiC	617.1	437.1	1.41	1

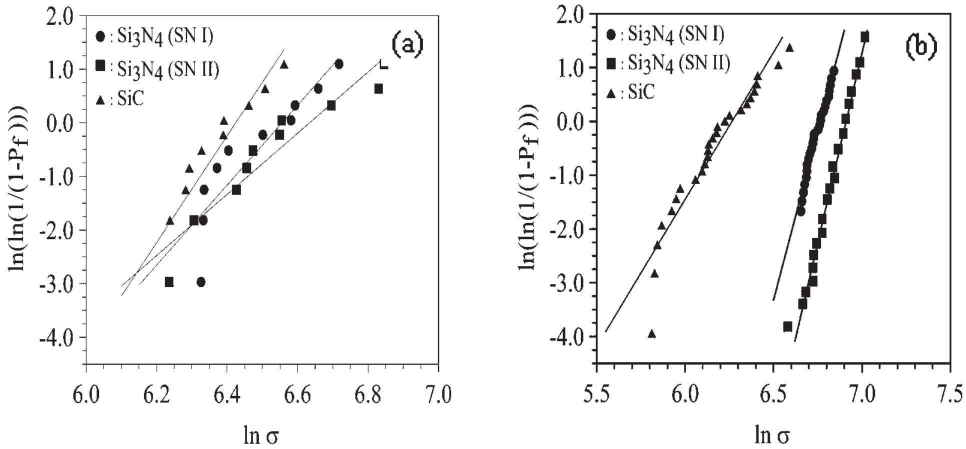

Fig. 3. Weibull distribution of strength value of Si_3N_4 (SN I), Si_3N_4 (SN II) and SiC materials in contact mode (a) and four-point bending mode (b).

ing and contact strength tests can also be explained by the existence of strong stress gradient near the contact areas in the case of contact strength test in contrast to the bending test with significantly lower stress gradient.

The comparison of results of the present investigation to those presented in the literature of different structural ceramics [9] show that they agree well, Fig. 4a,b.

The most significant deviation from the Fett's theory is in the case of the results of the SiC ceramic material, in regard to the ratio of the Weibull moduli but also in regard to the ratio of the characteristic strength values. The low m_{bend}/m_{cont} ratio is caused by the low m_{bend} and the high $\sigma_{cont}/\sigma_{bend}$ ratio by the low σ_{bend} and as it was found by fractographic analysis, all these results are determined by the fracture origins/technological defects with unusual size and size distribution (from 5 μm to 10 μm).

Based on the results of fractography of fracture surface (Fig. 5a) after the contact tests, it is evident that the failure starts from the surface or subsurface flaws, however by the study of the fracture surface the critical strength degrading defect could not be identified. The identification of the strength degrading defects (fracture origins) in the bending mode is significantly easier (Fig. 5b). They are technological defects or defects arising during the specimens preparation (grinding), located in the volume or surface of the specimen. In the case of silicon nitrides, the

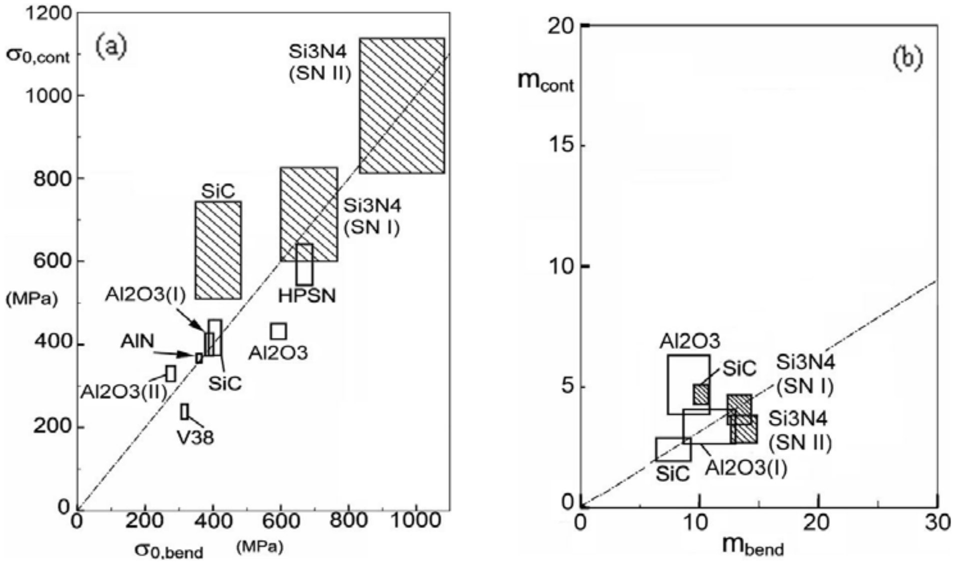

Fig. 4. The comparison of the results of the present investigation (shaded areas) with previous results published in the literature [9], for characteristic strength σ_0 (a) and Weibull exponent m (b).

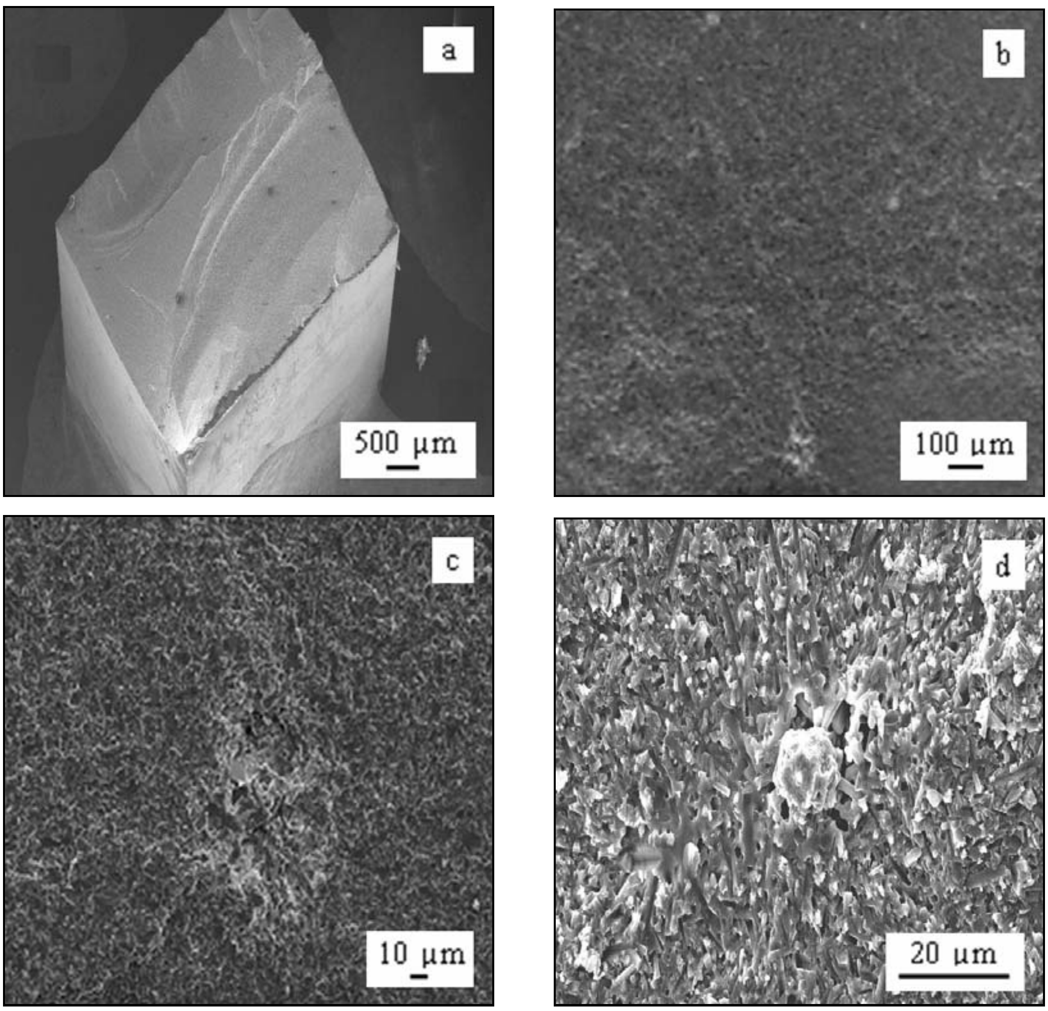

Fig. 5. Characteristic fracture surfaces of the investigated materials. Macrofractography of the specimens (Si_3N_4 (SN II)) tested in contact mode between two cylinders (a). Fracture origin in the specimen SiC tested in bending mode (b). Details of the fracture origins in the specimens SiC (c) and Si_3N_4 (SN I) (d) tested in bending mode.

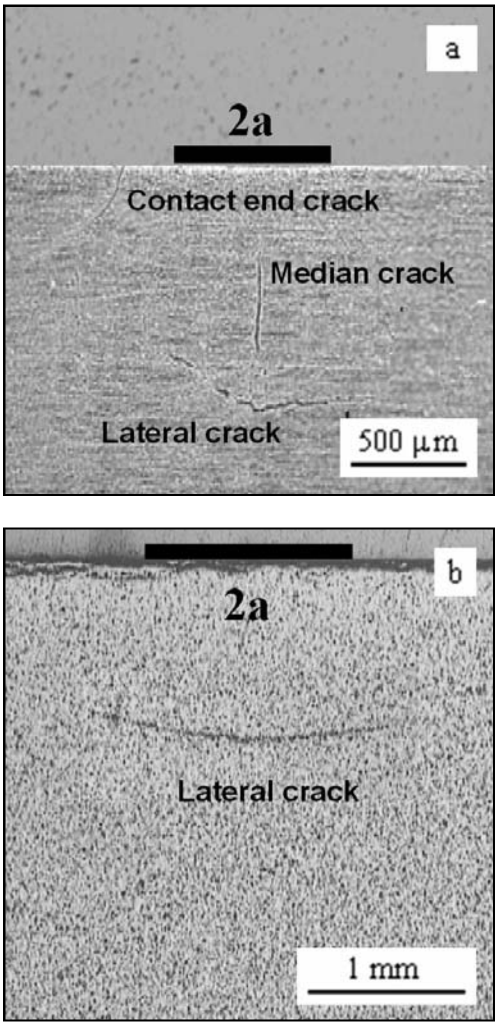

Fig. 6. Characteristic damages in contact test between two cylinders: Side view of lateral, median and contact end cracks in Si_3N_4 (SN I) material (a) and lateral crack in SiC material (b).

fracture origins are mainly surface defects and only a limited number of volume defects have been identified in the form of small pores or impurities (Fig. 5c). In the silicon carbide, the majority of specimens failed with a fracture origin in the volume in the form of large pores and clusters of pores (Fig. 5d).

According to the results of ceramographic/fractographic examination (Fig. 6), the characteristic damage mechanisms arising during the loading in contact mode were located below the surface of the specimens in the form of lateral, median and contact end cracks. The different crack types could be present at the contact surface and specimen sides of one experiment. A typical order of crack appearance was established by correlating the presence of different cracks and applied load in each experiment.

Notably, the first few cracks were always found in the specimen sides. In one or both specimen sides, a surface parallel crack was visible at a depth that was approximately one to two times the contact width $2a$. The crack typically displayed a slightly concave shape with the edges drawn towards the contact surface. In experiments that had proceeded, cracks of another type were distinguished. These cracks were vertical and located between the surface parallel crack and the contact mark. They typically started at the top side of the first crack and grew towards the contact surface. In some experiments, the cracks reached the contact surface at the contact end as shown in Fig. 6a. Following the nomenclature that is used for similar sub-surface contact cracks in ceramics, the first crack was named lateral crack (L) and second crack was named median crack (M). Last cracks were often found together in the contact surface. A few tests only contained a cracks in the corner between specimen side and surface. It was named "contact end crack" (C). Due to the contact symmetry, up to four contact end cracks may appear in one test. They meet the specimen corner at a perpendicular angle in both surface and side views (Fig. 6a,b). In the surface, they are curved towards the contact edge. These cracks originated and arose to a critical size during the loading, and caused failure and the strength degradation.

Contact damage has been widely investigated after Hertzian load and after contact strength test between balls in different glasses, ceramics and ceramic matrix composites [14]. Hertzian ring/cone cracks have been found with different modification and direction as the main damage depending on the microstructure/fracture toughness of the materials. The contact damage after the contact test between two cylinders was not investigated up to now in detail. Alfredsson and Olsson [13] found similar damages as in the present investigation at the standing contact fatigue test of hardened steel. They found that the radial surface strain outside the contact is altering the presence of cracks and that the ductility of the tested steel plays an important role in the fatigue crack initiation. In the materials with strongly limited ductility investigated in this work similar cracks can arise probably due to the complex and non-homogeneous stress states only [7]. The more detailed characterisation of the damages arising during the contact test between two cylinders will be the subject of our further investigation.

4. Conclusions

The contact strength of Si_3N_4 and SiC ceramics have been investigated using opposite rollers technique and compared to the results of bending strength test. The principle results are as follows:

In the case of Si_3N_4 ceramics, the contact strength test and bending strength test result in approximately equal values of the characteristic strength and for these materials the Weibull moduli in bending mode are approximately two times higher compared to the moduli in the contact mode. This means that for these ceramics, the Fett's theory has been proved concerning the ratio of the Weibull parameters.

In the case of SiC material, deviation has been found from this theory caused by the presence of large defects with different size/location, which are the fracture origins during the bending test and determine the Weibull parameters: low characteristic strength and low Weibull modulus.

Different damages have been found in the specimens loaded in contact mode in the form of contact end cracks, median cracks and lateral cracks. Their growth and possible interaction lead to the fast fracture of specimens at critical load.

Acknowledgements

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