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Fracture behaviour of Al_2O_3/SiC nanocomposite ceramics after crack healing treatment

Zdenek Chlup[a,*], Petr Flasar[a], Ando Kotoji[b], Ivo Dlouhy[a]

[a] *Institute of Physics of Materials Academy of Sciences of the Czech Republic, Zizkova 22, 616 62 Brno, Czech Republic*

[b] *Department of Energy and Safety Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, Yokohama 240-8501, Japan*

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Abstract

The self-crack-healing ability was systematically studied using flexural strength measurement of beams containing a semi-elliptical surface crack made by Vickers indenter. Beams of Al_2O_3/SiC ceramic nanocomposite with a good self-crack-healing ability predetermined for four-point and three-point bend test were prepared by ceramographic techniques. Flexural strength and fracture toughness were determined using three- and four-point bend method. Thermal aging in range from 1000 °C up to 1300 °C for 1 h was applied to find optimal conditions to heal the samples pre-cracked by the indentation technique. Fractographical analyses using an optical and/or scanning electron microscopy has been carried out to investigate the surface crack behaviour. Very good self-crack healing ability has been proved for surface micro-cracks having length up to 250 μm and depth no higher than 100 μm.

Keywords: Composites; Strength; Al_2O_3/SiC; Crack healing

1. Introduction

Thanks to new ceramic based materials development and development of methods for their exact assessment at extreme loading conditions the materials are used in structural applications that were unacceptable due to their inherent brittleness until now. Structural ceramics and ceramic matrix composites (CMCs) are step by step exploited in selected specific applications, where high resistance against high temperatures, corrosion environment, wear, etc. is expected at the same time as advantageous ratio of material density, hardness and/or flexural strength. Except for microstructure optimisation, toughening effects are introduced, i.e. these applications are conditioned by exact material properties definitions, by methods of their accurate determination, and calculation (modelling) methods proving either microstructural (toughening) effects synergy or contributing directly to the component design. The brittleness of these materials is a very limiting factor, which constraints its use in many otherwise suited applications.

In general, there are two ways of how to solve the problem. The first way (1) is to enhance fracture toughness of the ceramic matrix (reinforcing it by fibres and whiskers, ductile or brittle particles, by means of phase transformation, crack tip shielding and crack deflection, etc.) and the second one (2), which is the aim of this study, is to produce a perfect state of the material (material without flaws and other mainly surface defects originated by forming, sintering, finishing processes such as grinding, polishing, etc.). To reach this goal either careful inspection of the component (specimen) surface and/or crack healing process has to be applied.[1]

A vast progress has been made in the first area and a number of works is still increasing. Only few studies have been done in the second area although, if a crack healing process is applied to structural ceramics components, many advantages are considerable: increase of reliability of component, decrease of machining cost and prolongation of component lifetime.

From this point of view, the development and production of ceramic materials with crack healing ability is desired. Current CMCs systems with high crack healing ability are mostly based on oxide ceramic matrix with well distributed SiC nanoparticles: Al_2O_3/SiC,[2–4] mullite/SiC,[5] etc. Nevertheless the non-oxide ceramic systems are also in focus of interests, e.g. Si_3N_4/SiC.[2,4,6–10]

In this work, we have focused on the investigation of mechanical behaviour and crack healing ability of alumina/SiC nanocomposite. At first, standard mechanical properties have

* Corresponding author. Tel.: +420 532 290 335; fax: +420 541 218 657.
E-mail address: chlup@ipm.cz (Z. Chlup).

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been investigated. The influence of surface quality on flexural strength was systematically studied. Secondly, the basic crack healing behaviour and critical size of surface defect of studied material was examined using an indentation technique applied to bend specimens.

2. Experimental

The investigated nanocomposite material was prepared from alumina (Al_2O_3 powder AKP-20; mean grain size, 0.4–0.6 μm; purity, 99.99%, Sumitomo Chemical Ltd., Tokyo, Japan) with 15 wt% of SiC particles (ultrafine, mean grain size 0.2 μm, Ibiden Ltd., Gifu, Japan) well distributed in the alumina matrix. The powder was mixed together for 48 h, hot pressed at 1600 °C and 35 MPa for 4 h.[4]

The material was cut into standard specimens of dimensions 3 mm × 4 mm × 45 mm.[11] The beam surface was polished by ceramographic techniques to mirror quality and edges on the tensile side were chamfered. The flexural strength σ_0 and elastic Young's modulus E was determined by means of three-point and four-point bending tests carried out according to standards.[12] Additionally a resonance method was used for Young's modulus determination.[13] Fracture toughness was determined on specimens with a chevron notch; the three-point bending test method was applied.

Crack healing ability of the composite is given by presence of well-distributed ultra-fine SiC particles. These particles react with oxygen from the atmosphere under formation of SiO_2 which is involved in the crack healing. This reaction is conditioned by addition of energy in this case carried into effect by increase of temperature (Fig. 1). The healing mechanism was proved on commercially available monolithic sintered silicon carbide Rocar S1 produced by CeramTec, Czech Republic.

To investigate the crack healing behaviour of the composite specimens with a semi-elliptic surface crack produced by the Vickers indentation in the middle of the beam at a load of 20N were prepared (Fig. 2). Annealing of the specimens in temperature range of 1000–1300 °C for 1 h in air was applied. The level of flexural strength σ_0 together with the fractographically identified fracture origin was determined.

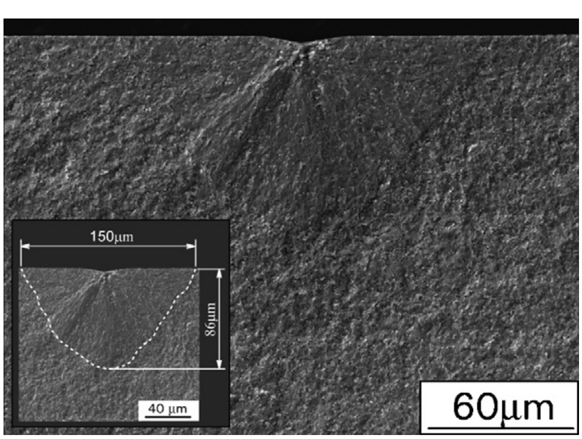

Fig. 1. Micrograph of surface crack (perpendicularly to beam surface) prepared by Vickers indenter at a load of 20N.

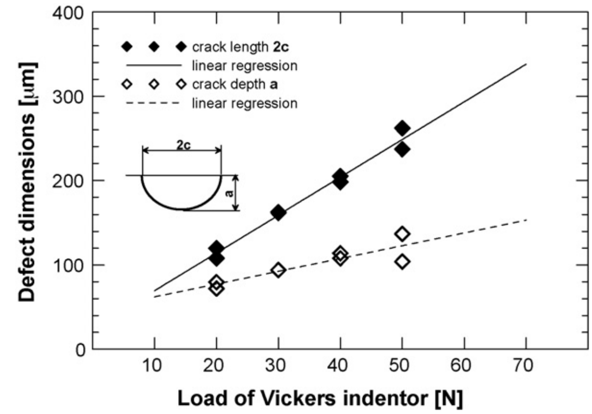

Fig. 2. Indicative dimensions of the crack caused by application of different load during Vickers indentation.

A critical flaw-size, i.e. the largest surface defect that is possible completely heal, was determined. One surface crack in the middle of the beam using Vickers indenter at different load (10N, 20N, 30N, 40N, 50N and 70N) was produced on each of specimens. Shapes of the typical cracks are shown in Fig. 3. Both the pre-cracked and the smooth specimens were annealed under the same conditions at 1300 °C for 1 h in air. The three-point bend test was applied for flexural strength σ_0 determination to prove the fracture behaviour only in close vicinity of the pre-cracked and healed zone.

3. Results and discussion

The flexural strength of the composite under investigation was determined on the level of about 800 MPa (three-point bending, smooth specimen). The fracture toughness measured using chevron notch technique was on the level of 3 MPa $m^{1/2}$. The elastic Young's modulus was determined by three different methods and reached the value of 402 GPa at four-point bending, 392 GPa at three-point bend loading and 406 GPa using a resonance method, respectively.

The crack healing ability of the sintered silicon carbide Rocar S1 was determined by means of flexural strength values obtained on pre-cracked and healed specimens. Flexural strength

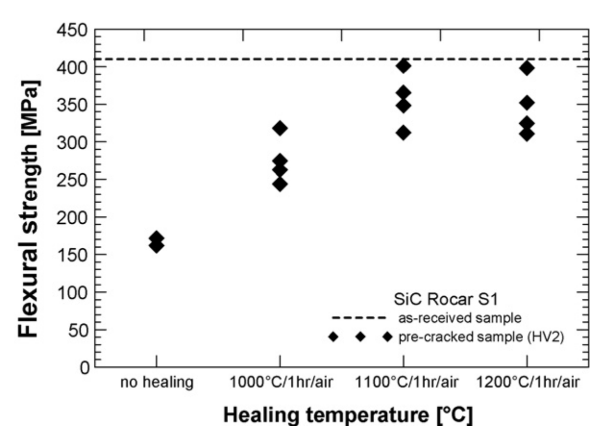

Fig. 3. Dependence of flexural strength of the pre-cracked specimens by Vickers indenter (load 20N) on healing temperature for the Rocar S1.

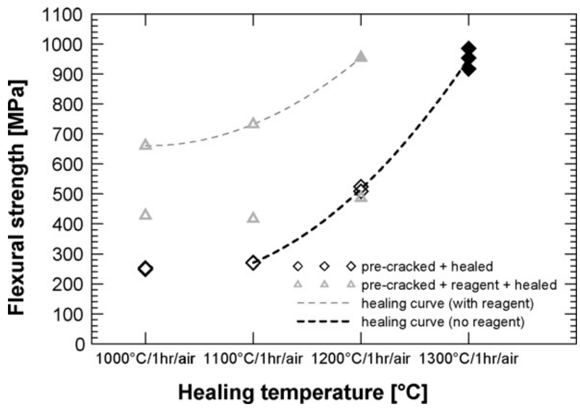

Fig. 4. Dependence of flexural strength of the pre-cracked specimens by Vickers indenter (load 20N) on healing temperature for the composite.

of smooth samples was on the level of 410 MPa and of the pre-cracked without application of healing procedure on the level of 170 MPa. The comparison of flexural strength values of specimens under various healing conditions is displayed in Fig. 1. The healing conditions providing sufficient fracture resistance were practically identical with those determined further on the composite material.

In addition, a crack healing behaviour was investigated on the alumina-based composite. Fig. 4 shows flexural strength values for pre-cracked and healed specimens. For the healed specimens at temperature of 1300 °C is evident that reference values of σ_0 smooth (unflawed) specimens have been successfully reached and fracture origin was out of pre-cracked zone (full symbols). Therefore these healing conditions seem to be the optimal. The maximal value of flexural strength for pre-cracked specimens (Vickers 20N) was about 250 MPa, at the same time, maximal flexural strength of pre-cracked and healed specimen reached more than 1000 MPa. There is an indication that by application of appropriate reactant before healing on the surface can be lowered either healing temperature or healing time. Results of application of oxidizing agent are shown in Fig. 4 by grey symbols and grey dashed line illustrates the maximal reached effect up to now.

For the pre-cracked specimens the fracture was initiated in surface indentation cracks. For the pre-cracked and healed specimens three typical fracture origins can be identified: the indentation crack, surface defects and defects under surface. Within the flexural strength determination a big defect which has occurred in the material during sintering was several times identified as the fracture origin. An example of such surface defect being located out of the initial pre-crack plane is shown in Fig. 5.

In Fig. 6 the dependence of flexural strength σ_0 on different initial surface crack size after healing at 1300 °C for 1 h in air is illustrated. The indicative defect size is represented by the surface length 2c and depth a and is corresponding to the level of indentation load for simplicity. The reference values obtained on smooth specimens (specimen without any surface crack annealed under same conditions as pre-cracked specimens with the aim to eliminate possible differences in strength values caused by microstructural changes) are represented by points

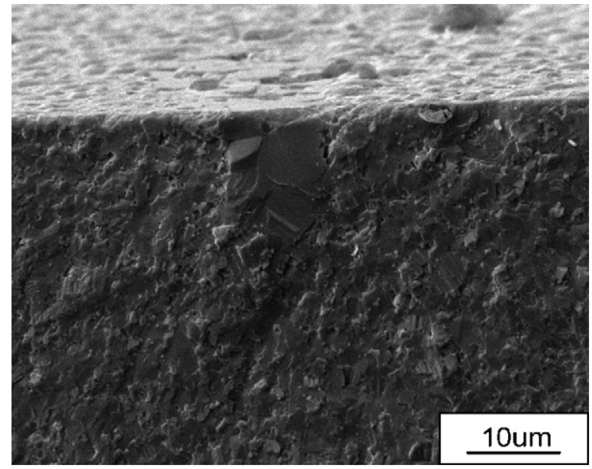

Fig. 5. Processing defect as a fracture origin, located at the tensile surface of the specimen (SEM, sample tilted by 10°).

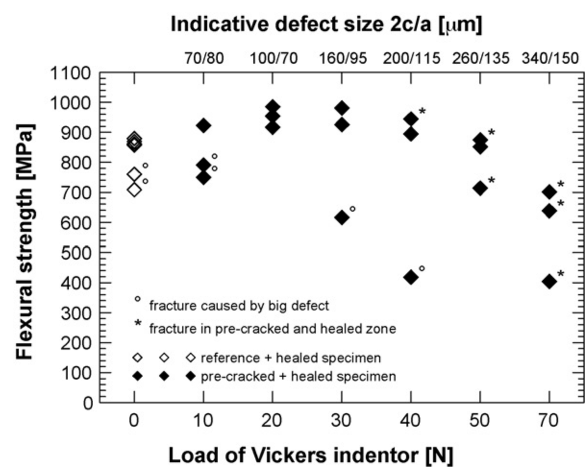

Fig. 6. Flexural strength of cracked and healed specimens in dependence on the primary surface defect.

at zero load. The average σ_0 value of reference specimens was about 800 MPa. The symbol "°″ indicates the beams broken on big material defect, via symbol "*" the failure initiation within crack-healed zone is marked.

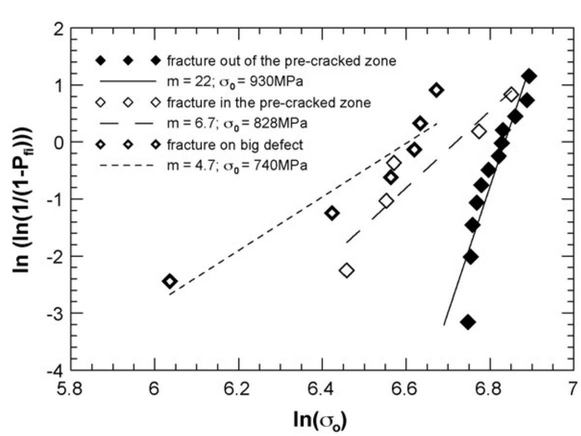

Fig. 7. Weibull diagram for pre-cracked and healed samples with respect to the fracture origin.

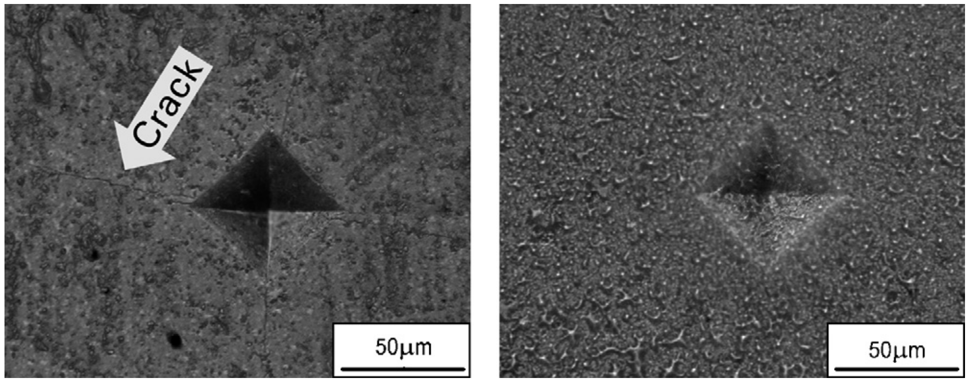

Fig. 8. Vickers indentation before (left) and after (right) annealing at 1300 °C for 1 h in air.

To establish the critical (maximal) surface crack size of the composite material two criteria were applied:

(1) complete strength recovery of pre-cracked specimens by healing, and, in addition,
(2) if any, initiation of the fracture in crack and healed zone (i.e. not on material defect and out of initial surface flaw).

Based on detailed specimen to specimen analysis taking into account the above mentioned criteria (Fig. 6) the surface flaw provided by Vickers indenter at load of 50N was established as the critical defect of studied composite material. The flexural strength σ_0 of these specimens reached the value of the smooth unflawed specimens and, at the same time, in two cases of three, the fracture was initiated in pre-crack and healed zone was initiated. Using SEM, the critical length and depth of surface crack originated by Vickers indentation at a load of 50N were measured: $2c \sim 250\ \mu m$, and $a \sim 100\ \mu m$, respectively.

The fractographical analyses of all fractured specimens result in classification on to three categories. Firstly where fracture origin was found in pre-cracked and healed zone; secondly where fracture initiation occurred out of pre-cracked and healed zone and finally those specimens where a big material defect caused fracture. Justification of this categorisation was given by statistical analyses of data. A Weibull plot was constructed and is displayed in Fig. 7. Both the Weibull module and the strength at probability of 63% were calculated for each category as can be seen in the plot legend.

An evidence of healing process is given either by flexural strength measurement or by observation on pre-cracked zone before and after application of healing procedure. The micrographs of the sample pre-cracked by Vickers indenter using load of 50N before and after application of healing procedure are displayed in Fig. 8.

4. Conclusions

For alumina matrix composite containing 15 wt% SiC particles the flexural strength and crack-healing ability was investigated. The flexural strength reached the value of about 800 MPa (three-point bending, smooth specimen). The fracture toughness was determined being on the level of $3\ MPa\,m^{1/2}$ (chevron notch technique). The elastic Young's modulus reached the value of 402 GPa at four-point bending, 392 GPa at three-point bend loading and 406 GPa using the resonance method, respectively.

The surface quality of the specimens plays a key role when the flexural strength is determined. Therefore it is essential to use the same surface quality for all specimens concerning to analyses of the healing ability.

High self-crack-healing ability has been found for the Al_2O_3/SiC composite ceramics. The healing procedure consisting of heating on the temperature of 1300 °C, holding for 1 h on the temperature in air was found to be sufficient to recover completely a semi-elliptical surface cracks. The largest flaw type surface defect (produced using Vickers indenter) that can be completely healed is a crack of length $2c \sim 250\ \mu m$ and depth $a \sim 100\ \mu m$.

Acknowledgments

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