R-curve Behavior of $\text{Al}_2\text{O}_3/\text{ZrO}_2$ Composites Measured by Stable Fracture Test in Bending

Toshihiko Nishida^{☆1}, Naoki Maeda^{☆1}, Masahiro Kihara^{☆2} and Tomohiko Ogata^{☆2}

^{☆1} Dept. of Materials, Kyoto Institute of Technology, Matsugasaki, Sakyo-ku, Kyoto 606.

^{☆2} Toray Co., Ltd., Sonoyama, Ohtsu 520.

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SYNOPSIS

A fracture mechanics specimen shaped as a single-edge double-notched beam (SEDNB), was specially designed for evaluating the fracture properties of brittle ceramic materials. The notch-tip was sharpened to a radius $<10\ \mu\text{m}$ in order to reduce the amount of elastic energy stored at its root prior to crack extension. Furthermore, a crack stabilizer, designed for the bending geometry, was employed. The use of this special stabilizer as well as a new machining technique developed for introducing the sharp notches made it possible the detectability of near-tip R-curve behavior, and to discern it from long-crack R-curve behavior. As an application of this technique, the R-curve behavior of $\text{Al}_2\text{O}_3/\text{ZrO}_2(2.5\text{Y-TZP})$ composites has been evaluated and the micromechanism responsible for such a behavior theoretically discussed.

KEY WORDS

Ceramic composite, R-curve, Bending test, Alumina, Zirconia,

1. INTRODUCTION

Techniques adopting the double cantilever beam (DCB)^{1, 2)} or the compact tension (CT)^{3, 4)} specimen configuration have been developed to measure the resistance to crack extension (i.e., the rising R-curve behavior) of brittle ceramic materials. However, it seems more desirable to obtain R-curves from a simple bending test using small rectangular bars, especially for new engineering materials still under research and development. Nevertheless, catastrophic fracture due to the elastic energy stored upon loading both in the specimen and in the testing apparatus remains a peculiar aspect of the bend-bar configuration pre-cracked with a straight-through notch. Reasons inherent to the notch geometry and the finite notch-tip radius represent related aspects of the crack stability problem in the bending bar configuration. In this paper, we have attempted to overcome these problems and describe a fracture mechanics procedure to measure R-curves in bending bars containing a straight-through notch. It should be noted that the use of the chevron-notched configuration, which has also been proposed⁵⁾ and widely used⁶⁻⁸⁾ to obtain stable crack propagation in bending, leads often to uncertainties in the fracture mechanics computation of stress-intensity factors⁹⁾. In this investigation, the R-curve behavior of toughened $\text{Al}_2\text{O}_3/\text{Y-TZP}$ ceramic composites was monitored

to apply the proposed technique.

2. MATERIAL, NOTCHING AND TESTING APPARATUS

Two kinds of $\text{Al}_2\text{O}_3/\text{ZrO}_2(\text{Y-TZP})$ composite materials were prepared by the ordinary powder metallurgy processing. Small amounts of TiO_2 additive (3 wt%) were used as a sintering aid. Compositions and preparation conditions of the composite materials are summarized in Table 1. Sintering temperature was selected to achieve the fairly full density. Typical scanning electron (SEM) micrographs of the polished and thermally etched surface of the sintered materials are shown in Fig. 1. White and black colored grains in the photographs are Y-TZP and Al_2O_3 , respectively. The material microstructure was constituted by uniaxial grains whose average diameter was from a few μm to 10 μm . Rectangular bars 3 x 4 x 20 (B x W x L) mm in dimension were cut from the sintered bodies and a straight-through notch with a relative length, a/W , of about 0.5 was introduced in the center of the bar by a diamond cutter (the blade thickness of 0.2 - 0.5 mm). Then, to reduce the influence of the finite notch-tip radius, the bottom part of the saw-notch was processed with a sharp razor blade sprinkled with fine diamond paste (1 μm). This procedure allowed to sharpen the notch-tip to a radius $<10\ \mu\text{m}$. The experimental

Table 1 Compositions and preparation condition of Al₂O₃ / Y-TZP composites.

Composition Code \ (wt%)	Al ₂ O ₃	Y-TZP*	TiO ₂
A Z 65 T	62.0	35.0	3.0
A Z 80 T	77.0	20.0	3.0

CIP : 19.6MPa, Pressurless Sintering : 1500°C, 3h *Y₂O₃ : 2.5mol%

Fig. 1 Microstructure of Al₂O₃/Y-TZP composites. Polishing and successively thermal etching at 1450°C for 2 h were performed prior to observation.

details and a critical evaluation of this double-notch technique have been previously published 10). SEM pictures of a double-notched Al₂O₃/Y-TZP specimen (side view) are shown in Fig. 2.

Three-point bending test (with span S=16 mm) was carried out at room temperature in air by using an oil-pressure type testing machine (load capacity of 10 ton) under the relatively fast pressing-ram speed of 0.1 mm/min. The fast speed was selected to avoid the effect of subcritical crack growth during the measurements. To achieve fully stable fracture in bending geometry, it is well known for improving the rigidity of testing machine¹¹⁻¹³⁾. In the present experiment, the notched specimen was set in the stabilizer designed by

Fig. 2 Photographs of a sharpened (double) notch introduced in the Al₂O₃/Y-TZP material (A). In (B), a magnified micrograph of the sharpened notch-tip region is shown.

Nojima¹⁴⁾. This crack stabilizer for bending geometry allows to detect directly both the applied load and the load-point deflection. The stabilizer system can be expressed by using the equivalent spring model shown in Fig. 3(b). In this model, the compliance of the testing system, Λ_{eq} , excluding the effect of the specimen compliance, $\lambda(a)$, can be expressed as;

$$\Lambda_{eq} = \Lambda_1 + \Lambda^* \Lambda_2 / (\Lambda^* + \Lambda_2) \tag{1}$$

Although the compliance of usual testing machine, Λ^* , is about 1 mm / 10 kN, Λ_{eq} can be improved to 1 mm / 73 kN, for example, by applying a stabilizer with $\Lambda_2 = 1 \text{ mm} / 360 \text{ kN}$ and $\Lambda_1 = 1 \text{ mm} / 91 \text{ N}$. In this case, the stiffness of the testing system can be rises to about seven times that of the test machine without stabilizer. This stabilizer also has the advantage of detecting the stress-strain relation with high accuracy owing to their direct measurements by semiconductor strain gauges. The strain gauges are attached on both the loading bar (number 3 in Fig. 3(a)) and the detection plate placed under the specimen (number 5 in Fig. 3(a)).

A photograph of the stabilizer, specimen and loading machine set up is also shown (Fig. 3(c)).

3. TESTING RESULTS AND DISCUSSION

A typical load – (load–point) displacement curve, as detected in the present $\text{Al}_2\text{O}_3/\text{Y-TZP}$ material during stable fracture, is shown in Fig. 4. As seen, during the stable crack propagation, load could be arrested and quickly released at selected values of the displacement, P_x . The specimen, containing the stably propagated crack, was then immersed in black ink and the length of the stable crack extension, Δa_x , measured by optical microscopy after completely fracturing the specimen at fast rate (cf. low magnification photograph of

Fig. 3 Set-up for stable crack extension in bending test. Draft (A), equivalent spring system (B), and photograph (C) of the stabilizer. The numeration shown in (A) refers to the following parts of the apparatus: 1, Upper anvil; 2, Strain gauges for detection of load; 3, Stainless-steel stabilizer bars; 4, (Double) notched beam specimen; 5, Strain gauges for detection of specimen deflection; 6, Lower anvil, and in (B) refers to the following compliance: Λ^* , Testing machine; Λ_1 , Load cell; $\lambda(a)$, Specimen; Λ_2 , Crack stabilizer.

fracture surface shown in Fig.5). The crack extension resistance, K_R , was calculated from the total crack length value, a_x , and the fracture load, P_x , at

Fig. 4 Typical load – load–point displacement curve as measured in the present $\text{Al}_2\text{O}_3/\text{Y-TZP}$. The curve loses its linearity at the load value P_i , (i.e., just below the maximum load, $P_{\max}$). Then, during stable propagation, the load is quickly released at the selected value P_x to check the crack extension.

the onset value before unloading by the following equation 12).

$$K_R = (P_x S / BW^{3/2}) \{ (3/2) (a_x / W)^{1/2} Y(a_x / W) \} \quad (2)$$

where S is the span width, Y is function related to the loading geometry and relative crack length 12), and a_x is the total crack length equal to $(a_0 + \Delta a_x)$, with a_0 being the notch length. a_0 and Δa_x are clearly recognized in the black and gray areas in Fig.5, respectively.

Figure 6 shows the rising R-curve behavior as found in the present $\text{Al}_2\text{O}_3/\text{Y-TZP}$ materials. The lowest value of the stress intensity factor, denominated K_{I0} hereafter, was calculated from the initial notch configuration, a_0 , and the onset load value for non-linearity in load – deflection curve, P_i shown in Fig. 4. In this paper, we have considered the calculated K_{I0} value as the critical stress intensity for crack initiation. The K_{I0} value of AZ65T and AZ80T shown by open marks in Fig. 6, were $4.0 \text{ MPam}^{1/2}$. This value may correspond to the actual stress–intensity necessary for crack initiation in the present composite materials, if the effect of subcritical crack growth is negligible under the present testing condition. On the other hand, the lowest K_R value which could be determined from examining the fracture surface after dye–penetration treatment was slightly higher (i.e., about $4.5 \text{ MPam}^{1/2}$) and corresponded to a crack extension of about ten microns. Another interest–

ing feature was found in comparing the present K_R values with the stress intensity factor measured by the standardized single-edge pre-cracked beam (SEPB) testing procedure^{13,14}. This latter value, although is referred to in the standard as the " K_{IC} " of the material, was markedly higher (i.e., 5.5 (AZ65T) and 5.2 (AZ80T) MPa $m^{1/2}$) than the K_{I0} value measured by the present procedure. This was likely due to shielding forces operated by grain interlocking across the faces of the pop-in pre-crack¹⁵). This circumstance suggests that, for

Fig. 5 Optical micrograph showing the fracture surface of the SEDNB specimen after the dye-penetration treatment: (a) notched area; (b) area of stable crack propagation; (c) area rapidly fractured after test interruption for observing the area (a), (b).

Fig. 6 R-curve of the Al_2O_3/Y -TZP composites as obtained from the stable fracture test in bending. The open marks were the values calculated from the initial notch configuration, a_0 , and the onset load value for non-linearity in load - deflection curve, P_1 shown in Fig. 4.

Fig.7 SEM photographs of the fracture surface of the tested Al_2O_3/Y -TZP material (AZ65T). (A) stably fractured area; (B) area rapidly fractured after the test interruption at load P_x for measuring the extent of the stable fracture.

Fig.8 SEM photographs showing some grain bridging along the crack extension (AZ80T).

materials showing sharply rising R-curve behavior as the present $\text{Al}_2\text{O}_3/\text{Y-TZP}$ composites, the standardized SEPB procedure may be unsuitable and lead to dangerous overestimations in K_{IC} .

SEM micrographs of typical fracture surface in the regions of stable and brittle fracture are shown in Fig. 7(a) and (b), respectively. Both the micrographs were taken on regions in the neighborhood of the crack front at the time of the crack arrest (i.e., at the time of load release). The area of brittle fracture refers to the unstable propagation produced for measuring the crack length after partial crack extension. In both the regions, higher percentage of transgranular fracture is observed in the composite grains. On the other hand, extensive interlocking grain is observed in the stable region of the fracture surface as compared to the monolithic alumina polycrystal, as a consequence of intergranular crack propagation. Also the crack extension from the side surface clearly showed occurrence of the grain bridging caused by the Y-TZP phase as shown in Fig. 8. These observation result may suggest that surface tractions by grain interlocking, namely the shielding forces operating in a process zone behind the crack front, produce the main toughening mechanism in the present $\text{Al}_2\text{O}_3/\text{Y-TZP}$ ceramic composites.

4. SUMMARY

A procedure for measuring the R-curve properties of ceramics in bending was proposed and applied to toughened $\text{Al}_2\text{O}_3/\text{Y-TZP}$ composite materials. This testing procedure is based on the introduction of a straight-through notch with sharp tip radius and the use of a crack stabilizer specially designated for bending geometry. The procedure is simple and requires only minimum amount of material. It can be generally applied to determine the fracture mechanics characteristics of ceramics and related composites. The detectability of the K_{I0} value, namely the onset stress intensity for crack initiation, seems also a very suitable characteristic of this testing method on the design side. The total length of crack extension which can be monitored by testing in the present geometry is about 1 mm. However, this limitation seems not to be a serious problem for the fracture mechanics evaluation of engineering ceramics with sharply

rising R-curve behavior.

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