

# A New Technique for Precracking Ceramic Specimens in Fatigue and Fracture

Ashok Kumar Ray\*

National Metallurgical Laboratory, Jamshedpur-831007, Bihar, India

(Received 23 June 1997; accepted 19 December 1997)

## Abstract

*This paper highlights the design and performance of an articulated bridge fixture to precrack ceramic specimens with 100% success. The precracked specimens were subsequently used to determine and study the fatigue crack growth rate and fracture toughness of a 25 wt% SiC whisker reinforced  $Al_2O_3$  composite. Precracking was initially done by using Vickers indentation as a cracker starter. However one can argue that the residual stress introduced due to the indentation might influence the fracture toughness and fatigue crack growth rate values determined subsequently. The success achieved in precracking by using this fixture led us to substitute the Vickers indentation with a 0.1 mm deep groove as a crack starter. The fracture toughness values obtained from V notched and precracked specimens were about 7.2% lower than those obtained from indentation as a crack starter in indented and precracked specimens. However, fatigue crack growth rates using both these crack starters were not widely different. © 1998 Elsevier Science Limited. All rights reserved*

## 1 Introduction

Precracking of ceramic samples is extremely difficult as these materials have very low toughness and high E (modulus of elasticity) values. Besides, due to the difficulties in their machining and surface preparation, the size of ceramic specimens are usually very small. The crack initiation in such materials often requires a load which is higher than that of crack extension. Therefore, a crack propagates as soon as it initiates, making it practically

impossible to precrack the specimen. To facilitate precracking by bridge indentation technique, and for growth of stable cracks a special bridge fixture is therefore required.

In general, measurements of fatigue crack growth rate (FCGR) and  $K_{IC}$  (fracture toughness) of such materials are very difficult, as the load, displacement and in most cases crack length which are required for the determination of  $K_{IC}$  and FCGR are very small and their precise measurements are extremely difficult in practical situations.

The conventional bridge is normally being used elsewhere. By using this technique most of our specimens failed before even the crack had grown in the specimen in a controlled manner. This is because the precision and dimensional tolerance of the grips and fixtures used to precrack the specimen, are not adequate to avoid spurious mode III type of loading in the specimen.

The bulk of the fracture toughness data were generated in ceramic/ceramic composites<sup>1–6</sup> generally by indentation technique, indentation-strength method<sup>2</sup> or by using compact tension specimens.<sup>7,8</sup> The bridge indentation technique was first proposed by Sadahiro and Takatsu<sup>9</sup> and further developed by Warren and Johannesson.<sup>10</sup> In this technique, an initial crack is created on the top surface of the specimen by means of a row of Vickers indentations aligned so that cracks emanating from the diagonal of each indentation link up. A compressive load (F) is then applied to the bend bar through the bridge indentation device as illustrated in Fig. 1. At a certain load the surface crack extends unstably (denoted 'pop-in') through the thickness down to the depth where the stresses are low enough to arrest the crack tip. The critical load and the depth of arrest are dependent on the material properties, the span (d in Fig. 1), the number of hardness indentations, and the applied hardness indentation load. The load is increased until the crack front became even and a depth of at least 2 mm was reached. A span of 12 mm with

\*Present address: The Institut für Werkstoffe der Energietechnik (IWE-1), KFA, Forschungszentrum, D-52425, Jülich, Germany.

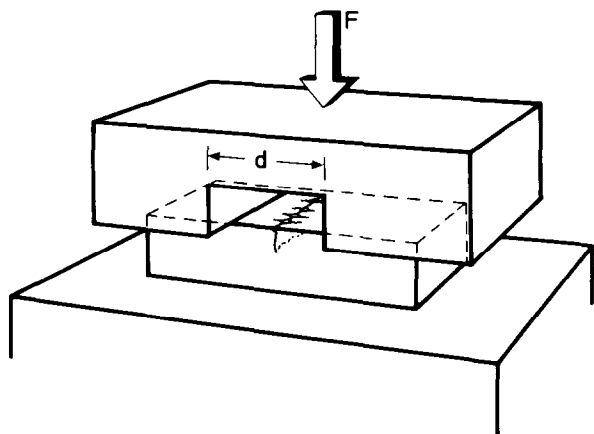


Fig. 1. Device for precracking brittle materials by the bridge indentation technique.

bridge punch contact area of  $3 \times 16$  mm on each side is used by Hansson *et al.*<sup>11</sup> in the same bridge indentation technique for evaluating the  $K_{IC}$  of a hot pressed alumina reinforced with 33 vol% silicon carbide whiskers.

Using bridge indentation technique, Warren and Johanneson had investigated the effect of indentation load and span on the fracture toughness of WC-6Co alloy and were able to create stable cracks in hard metals.<sup>10</sup> Later Nose and Fuji<sup>12</sup> had reported  $K_{IC}$  tests performed on precracked single-edge beam specimens of alumina, sialon and silicon carbide and studied the effect of the precracking procedure, loading rate and precrack length on the  $K_{IC}$  values. The work of Baron *et al.*<sup>13</sup> reported the  $K_{IC}$  values of two silicon carbides alumina and an yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) also by using bridge indentation method meant for precracking of beam specimens. The effects of precracking parameters including the effect of indentation load and bridge span on the precrack length or  $K_{IC}$  of the materials were also studied.<sup>13</sup>

This work is innovative in the sense that we were able to grow a stable crack using an articulated fixture and hence study the crack growth rate behaviour under normal four-point bend loading and later on evaluate the  $K_{IC}$  values from indented as well as V notched four-point bend specimens of the ceramic composite in the present investigation.

The ceramic composite was prepared by mixing  $\alpha$ -alumina powder of particle size ( $< 1 \mu\text{m}$ ) with 25 wt%  $\beta$ -silicon carbide whisker.<sup>2</sup> The length of the whisker varied between 10 and  $80 \mu\text{m}$  and measured about 0.45 to  $0.65 \mu\text{m}$  in diameter,<sup>14</sup> (Fig. 2). The composite was hot pressed and sintered (25 MPa/1000–1850 °C/30 min) in the form of a billet of ( $12.5 \times 50 \times 150$  mm) from which four point bend specimens ( $3 \times 4 \times 50$  mm) were made. The grain size of the matrix varied between

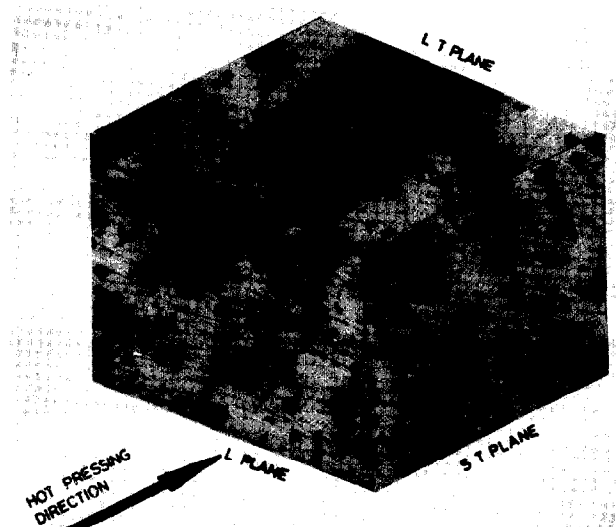


Fig. 2. Montage of the microstructures showing the distribution of the SiC whiskers along the three planes.<sup>14,16</sup>

1 and  $4 \mu\text{m}$ <sup>14</sup> and the porosity content in this composite was 4.89%.<sup>2</sup> Opposite side of the four point bend specimens were diamond-ground flat and parallel with a  $30 \mu\text{m}$  diamond wheel, and the prospective tensile surface was polished with  $9 \mu\text{m}$  diamond paste. The modulus of elasticity, fracture strength and the hardness of this composite are 340 GPa, 559 MPa and 20 GPa, respectively.<sup>2,14</sup> The details of fabrication, processing and material characterization has been described elsewhere.<sup>2,14</sup>

With a Vickers indentation as a crack starter, the loads are high and the crack lengths obtained using an indenter are generally very small. Also, as a result of indentation, residual stresses would have been induced which would effect small specimen behaviour towards fatigue and fracture. Therefore, to find out the effect of residual stresses induced on the  $K_{IC}$  and FCGR behaviour of the material, it is also essential to fabricate similar specimens with a grooved notch as crack starter for examining the nature and variation of residual stresses present ahead of the crack front emanating from such origins of crack. The magnitude and nature of the residual stresses present in the parent sample due to grinding while fabrication and ahead of both the crack starters along the crack propagation direction were determined by X-ray diffraction (XRD), using an X-ray stress analyser, AST-X2001 and have been reported.<sup>15</sup>

Since we had lost 24 out of 36 specimens by resorting to the conventional bridge technique while precracking, an articulated bridge fixture was therefore designed to precrack successfully all the remaining specimens by removing the spurious loading of the specimen in modes other than the mode I. The indentation fracture toughness and FCGR data of 25 wt% silicon carbide whisker

reinforced alumina ceramic, and their fractographic features are reported elsewhere.<sup>14-16</sup> The articulated bridge fixture and its efficiency in precracking these specimens are discussed in this paper.

Our objective, therefore was to briefly outline the articulated bridge fixture and also to find out the effect of indentation and notch as crack starters on the  $K_{IC}$  and FCGR in a 25 wt% SiC whisker reinforced  $Al_2O_3$  composite.

## 2 Experimental Procedure

### 2.1 Precracking of the ceramic specimens

To facilitate precracking, V-notches were grooved on 50% of the specimens with the help of a carborundum wheel to a depth of 0.1 mm. The width of the notch was 0.3 mm. These notch was grooved in the ST (short transverse) plane of the billet (Fig. 2) and exactly at the centre of the  $3 \times 50$  mm surface, i.e. the tensile surface of the four-point bend specimen. (Fig. 3). On the remaining samples a Vickers indentation was produced at 0.8 kN, also at the midpoint of the tensile surface of the specimen (Fig. 3), so that after precracking, the crack propagation was parallel to the LT (long transverse) plane<sup>14</sup> of the billet (Fig. 2). Since the material flow along the L (longitudinal) plane was high during hot pressing, the whiskers were aligned normal to the LT plane (Fig. 2) and the crack propagation direction was parallel to the 4 mm dimension of the specimen, which is perpendicular to the hot pressing direction.<sup>14</sup> Thus the whiskers are embedded perpendicular to the crack propagation direction and the crack plane was parallel to

the hot pressing direction.<sup>14,16</sup> Since the majority of the whiskers were oriented normal to the crack plane, they would tend to bridge the crack plane more effectively and enhance the toughness of the composite. Therefore, the  $K_{IC}$  for the crack planes parallel to the hot pressing direction (the L and ST planes of the billet) should be substantially higher than that for crack planes which are perpendicular to hot-pressing direction.<sup>14</sup> Recently, Hansson *et al.*<sup>11</sup> studied the fracture toughness anisotropy of a hot pressed alumina reinforced with 33 vol% SiC whiskers and clearly showed that the  $K_{IC}$  for crack planes parallel, and crack growth direction at right angles to the hot-pressing direction is substantially higher than that for crack plane normal, crack growth at right angles to the hot pressing direction and crack plane parallel, crack growth parallel to the hot pressing direction.<sup>11</sup> This justifies the orientation of the crack plane and the direction of crack propagation in our composite also, for reporting a high  $K_{IC}$  value. Before indentation, and after inserting slots, all the specimens were coated with aluminium in a vacuum evaporator by physical vapour deposition technique to about  $0.03 \mu\text{m}$  thickness, to facilitate location of the crack tip in the scanning electron microscope.

Precracking was accomplished using an articulated bridge fixture at a force of 4 to 5 kN, load ratio,  $R = 0.1$  and at a frequency,  $f = 20$  Hz, under fatigue. The crack growth was carefully monitored on the specimen surfaces and after the crack had advanced significantly with regular increments of number of cycles, the specimens were unloaded and the crack length was measured with the help of the micron marker in the scanning electron microscope (JEOL 840A).

#### 2.1.1 Design of the articulated bridge fixture

The articulated bridge fixture designed, fabricated and used is shown in Fig. 4. The articulated bridge fixture differed from the conventional one in that here the specimen was allowed to rest on a free to rotate rod made of tool steel, of diameter 16 mm and length 120 mm which in turn was housed in a block fixed to the actuator. On the free to rotate

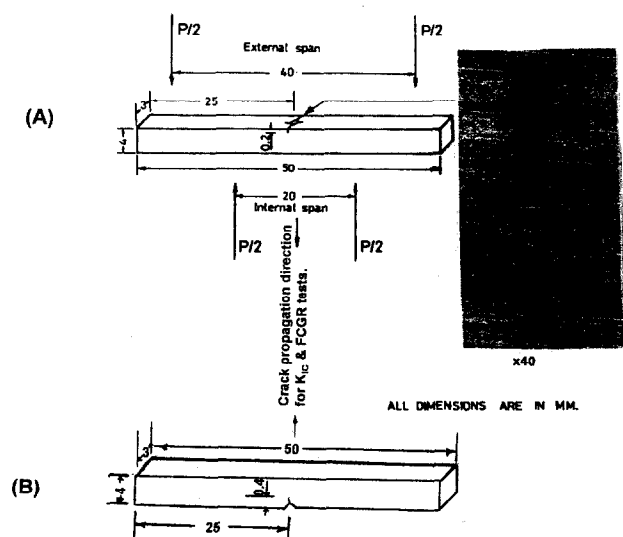


Fig. 3. (A) Indented and precracked specimen used in four point bend loading. Cracks are located at the four corners of indentation. (B) Notched and precracked specimen also used in four-point bend loading.

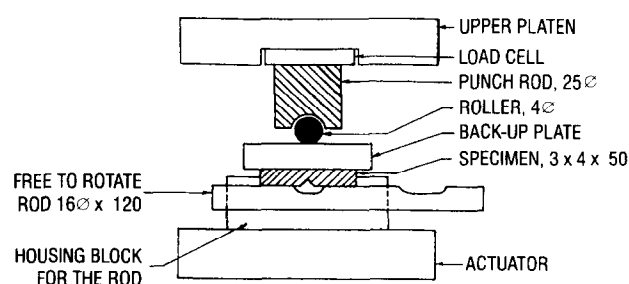


Fig. 4. An articulated bridge fixture designed and used for precracking ceramic specimens with 100% success.

rod, two bridges of varying span (of one's choice) were accommodated. This was made because the span of the bridge has also an effect on the length of the precrack and  $K_{IC}$  of the specimen.<sup>13</sup> The bridge span used in our case was 12.5 mm. The advantage of the articulated fixture is that the specimens no longer fail before controlled crack growth by avoiding spurious mode III loading. In the conventional bridge fixture, the four point specimen rested on a fixed bridge block attached to the actuator and was backed up by a plate on its top, just below the punch rod (of diameter 25 mm). The premature failure of the sample in this case, therefore, is probably related to the fixture stiffness more than anything else. The punch rod rested exactly at the centre of the back up plate on the opposite side of the notched or indented surface, as in the case of the articulated bridge fixture. An examination of the changing crack path trajectory on the fractured surfaces of the failed samples<sup>14,16</sup> in the conventional bridge fixture revealed that instead of mode I loading, the specimens experienced a combined mode loading with mode III loading playing a significant role in the crack extension. Therefore the conventional bridge technique was rejected and a new articulated bridge fixture for precracking ceramic specimens was designed and fabricated.

It is noteworthy that precracks were produced from a single Vickers indentation at the centre of the tensile surface of the specimen, in the articulated bridge technique. The articulated bridge technique is only a modified bridge indentation technique. Normally, in the 'bridge indentation technique' for precracking four point bend specimens using vickers indentation as crack starter, an initial crack is created on the top surface of the specimen by means of a row of Vickers indentations aligned so that cracks emanating from the diagonal of each indentation link up with the application of a compressive load to the bend bar, through the bridge indentation device.<sup>9,10</sup>

### 2.1.2 Crack length measurement during precracking by the articulated bridge technique

In the case of four point bend specimens with the indentation as crack starter at the mid point of the tensile surface of the specimen, cracks were developed at the four corners of indentation (Fig. 3). Before precracking, crack length of the two cracks emanating from the diagonal, i.e. from the two opposite corners of indentation across the thickness of the specimen, was measured in the SEM-JEOL 840A at a magnification of 40. These two cracks are of prime importance for the present investigation. The tip of the crack was located through observation at higher magnification which

often showed evidence of crack branching.<sup>14,16</sup> The branch extending farthest was considered to be the crack tip and the crack length was measured accordingly. During precracking, the specimen was fixed in the articulated bridge fixture and was subjected to fatigue loading. The length of the advancing crack across the thickness of the specimen was measured with regular increments of the number of fatigue cycles, and the crack front often showed deflections<sup>14</sup> (Fig. 5). The bridge load was then increased for stable extension of the crack. While measuring the crack length, the specimen was first unloaded from the servohydraulic machine and the current crack length was measured in the SEM. Later, after these two advancing cracks had spanned right across the specimen thickness and until the crack front became even, the crack length was measured on both the adjacent planes ( $4 \times 50$  mm surfaces), till it achieves a length of 0.2 mm to 0.4 mm ( $a/W = 0.05$  to 0.1). The average of the crack length measured on both the  $4 \times 50$  mm surfaces gave the crack length  $a$ . The articulated fixture was then unloaded from the MTS-880 servohydraulic test machine and the specimen was loaded under normal four point bend loading as shown in Fig. 3. As mentioned earlier, the hot pressing direction of the specimens was normal to the direction of crack propagation under four point bend loading. The crack length was measured in a similar manner during FCGR tests under four point bending, which preceded the  $K_{IC}$  testing. Since the determination of FCGR required a precise measurement of the crack length,  $a$  during FCGR was measured with a special care in locating the crack tip.

In case of notched four point bend specimen, the notch was not exactly V, but in the form of a U, with a depth of 0.1 mm. Therefore, in order to get a sharp crack, the specimen was also precracked

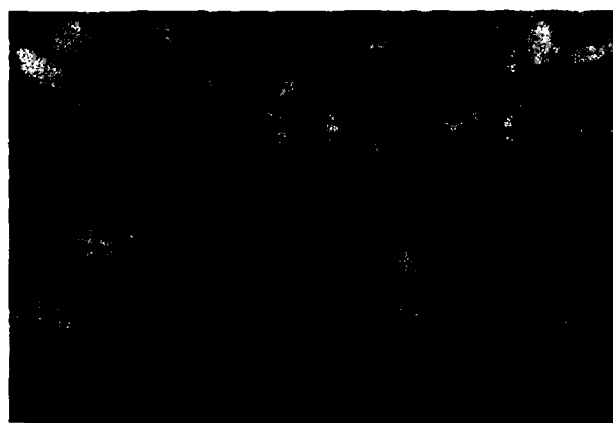


Fig. 5. Crack deflection observed in the low  $\Delta K$  region of FCGR test<sup>14</sup> and during precracking, of 25 wt% SiC whisker reinforced alumina. The crack shown in the fracture surface was at an angle to the final fracture

using the articulated bridge fixture till a crack length equivalent to 0.3 to 0.4 mm was achieved, inclusive of the depth of the groove or notch. While precracking, the crack length was also measured in a similar manner, at a magnification of 40 in the SEM.

## 2.2 Determination of FCGR and $K_{IC}$

FCGR was determined with both types of crack starter, i.e. V notch with precrack and indentation with precrack, in laboratory atmosphere and at ambient temperature, after the crack had grown significantly on the  $4 \times 50$  mm surface and achieved length  $a$  equivalent to  $a/W \approx$  to 0.05 to 0.1. The four point bend specimen is not recommended in ASTM standard E-647 and therefore, the  $K_I$  values of the specimen was calculated using the standard formula reported in ASTM STP-410.<sup>17,18</sup> Except for this aspect,<sup>14</sup> the procedure used here to determine FCGR conformed to the recommendations given in ASTM standard E-647. The tests were conducted in a MTS-880 servohydraulic test machine using a 1 kN load cell under four point bend loading. The test frequency was 1 Hz and the value of load ratio in fatigue,  $R = 0.1$ . The loading rate was  $0.25 \text{ N s}^{-1}$ . Typically the specimens were cycled within the load range between 11 and 111 N, when  $a/W \approx 0.1$ . The crack lengths were measured at regular intervals of increments of number of cycles giving  $\approx 0.05$  to 0.1 mm of crack growth. Thus, the fatigue cracking was interrupted after a predetermined number of cycles. This was continued until the crack length increased to a value giving  $a/W \approx 0.45$  to 0.5.

The values of  $da/dN$  were generated from the crack length,  $a$  versus number of cycles,  $N$  plot at a given  $a$  and plotted against  $\Delta K$  (stress intensity range). The  $\Delta K$  value is given by  $K_{\max} - K_{\min}$ . With load and  $a$  known,  $K_I$  values were calculated from<sup>17,18</sup>

$$K_I = Y3P\sqrt{a}(L_1 - L_2)/2bW^2 \quad (1)$$

where

$$Y = 1.99 - 2.47(a/W) + 12.97(a/W)^2 - 23.17(a/W)^3 + 24.80(a/W)^4$$

$P$  = load;  $L_1$  = external span;  $L_2$  = internal span;  $b$  = thickness of specimen;  $W$  = depth or width of the specimen;  $a$  = crack length. The values of  $K_{\max}$  and  $K_{\min}$  were calculated using eqn (1).

With the completion of the FCGR tests and after the crack had advanced to a level of  $a/W \approx 0.45$  to 0.5,  $K_{IC}$  was determined by subjecting the precracked four-point bend specimens to monotonic

loading.  $K_{IC}$  testing followed here conformed in all respects to the ASTM STP 410 method,<sup>17,18</sup> since four-point bend specimens were used. The test record of load versus mid-point displacement of the specimen was obtained during monotonic loading; the ramp rate was  $0.25 \text{ N s}^{-1}$ . The load value corresponding to the onset of fast fracture was used in eqn (1) to obtain the  $K_{IC}$  value.

## 3 Results and Discussion

### 3.1 Performance of the articulated bridge fixture

The articulated bridge fixture (Fig. 4) designed and fabricated to precrack the ceramic specimens for the present investigation, was able to precrack all the specimens with 100% success. Therefore this technique proves to be an efficient method for precracking ceramic specimens which are very brittle and expensive.

### 3.2 Fracture toughness, $K_{IC}$ testing

Fracture toughness values determined by using both the types of crack starter and as reported in Table 1, show that the average  $K_{IC}$  values obtained from V notched and precracked specimens were about 7.2% lower than those obtained from indentation as a crack starter in indented and precracked specimens. This is attributed to the presence of more tensile residual stresses in the notched sample compared to the residual stress present in the indented sample<sup>15</sup> and has been confirmed from the measurement of the residual stresses ahead of notch and indent by XRD technique. The effect of near-surface residual stresses would be to influence surface cracks. Thus their effect on fracture toughness here is merely through their influence on the observed, apparent crack length since in this case, the crack length was measured only on the surface. In the case of the machined V-notch the observed residual stress exist

**Table 1.** Fracture toughness determined by the ASTM STP 410 method for 25 wt% SiC whisker reinforced  $\text{Al}_2\text{O}_3$  composite

Fracture toughness, ( $\text{MPa}\sqrt{\text{m}}$ )		
ASTM STP 410 method		Indentation-strength method <sup>2</sup>
Notched and precracked	Indented and precracked	$5.35 \pm 0.17$
5.5	5.98	
5.44	6.1	
5.63	5.87	
5.52	6.0	
5.6	6.0	
5.47	5.93	
$5.53 \pm 0.01$	$5.96 \pm 0.05$	
Average	Average	

along the whole notch tip and not only at the side surfaces. Similarly, indentation as a crack starter has also been supposed in earlier work<sup>2,11,14</sup> to create tractions on the entire precrack (and not only on the side surfaces). Data collected for X-ray stress work and XRD pattern on the composite samples over a range of  $2\theta$  range 132 to 135° using  $\text{CrK}_\alpha$  (6 mA, 30 kV) radiation showed that the order of tensile residual stresses in the as-received parent material was  $325 \pm 0.4$  MPa.<sup>15</sup> These were the grinding stresses introduced into the parent material. Due to indentation a certain amount of compressive residual stresses (of magnitude  $-40.20 \pm 0.4$  MPa) had been induced ahead<sup>15</sup> of the indent and also along the entire precrack in the specimen. Nevertheless, the process of inserting a V-notch in the specimen resulted in a considerable amount of tensile residual stresses (of the order  $396.7 \pm 0.9$  MPa) ahead and along the whole notch tip.<sup>15</sup> Each measurement was taken using four tilt angles ( $\Psi = 0$  to 45°). The tilt angle  $\Psi$  is the angle between normal to the surface and normal to the plane of reference. The (119) reflection from  $\text{Al}_2\text{O}_3$  corresponding to a  $2\theta$  value of 135°<sup>19</sup> and the (222) reflection from  $\beta$ -SiC (cubic 3c) corresponding to a  $2\theta$  value of about 132° are suitable for X-ray stress work.<sup>20</sup> To partially compensate for anisotropic behaviour along the (hkl) planes, the elastic constants  $E$  (modulus of elasticity) and  $\gamma$  (Poisson's ratio) specific to that plane was used rather than the bulk value of the aggregate.<sup>20</sup> These constants are reported in the work of Abuhasan *et al.*<sup>20</sup> The residual stress  $\sigma_\phi$  is directly proportional to the peak shift  $\Delta d$  of the X-ray diffraction curve due to the presence of the residual stresses in the material, and was calculated with the help of a computer software using the following expression<sup>19,20</sup>

$$\sigma_\phi = E\Delta d(1 + \gamma) \sin^2 \Psi \quad (2)$$

It should be noted that the term compressive in the present case is used only in a relative sense in order to delineate from the tensile residual stresses already present in the parent specimen as well as in the specimen with a notch.

A residual compressive surface stress would decrease the apparent surface crack length, while a residual tensile surface stress would do the reverse. There are various ways in which surface stresses could be introduced into a material; of particular relevance to the production of samples for indentation testing is the process of surface grinding of brittle materials, e.g. using SiC or diamond abrasive wheels on glass, ceramics and cermets.<sup>19</sup> It is well documented that this surface finishing method induces residual stresses in materials such as alumina,<sup>19</sup> polyceram C906 glass-ceramic<sup>21</sup> and zirco-

nia-toughened alumina.<sup>22</sup> If the slope of  $K_{\text{IC}}$  (indentation fracture toughness) versus  $a^{1/2}$  (square root of crack length) plot is positive, it indicates that the nature of residual stress present is compressive but if the slope is negative, then tensile residual stresses are present.<sup>1,14,16,23,24</sup> Marshall and Lawn showed that such plots for tempered soda-lime glass plate produced a positive slope indicating that the nature of residual stress is compressive.<sup>23</sup> In line with Marshall and Lawn model,<sup>23</sup> Ikuma and Virkar<sup>24</sup> thus concluded that a positive linear dependence of  $K_{\text{IC}}$  on  $a^{1/2}$  for transformation-toughenable ceramics indicates the presence of residual surface compressive stresses induced by the tetragonal to monoclinic transformation of  $\text{ZrO}_2$  or  $\text{HfO}_2$  particles in the near-surface layers upon surface grinding. In this composite also, a similar behaviour was observed in evaluating  $K_{\text{IC}}$  and studying the nature of  $K_{\text{IC}}$  versus  $a^{1/2}$  plot in indentation fracture toughness tests.<sup>14,25</sup> The variation and dependence of  $K_{\text{IC}}$  on the precrack lengths of the same material in the present investigation, have been reported and discussed in details elsewhere.<sup>14,25</sup> The indentation fracture toughness,  $K_{\text{IC}}$  of this material has been compared with the  $K_{\text{IC}}$  values using precracked specimens with indentation as a crack starter and is reported elsewhere.<sup>14</sup> From Table 1, it is apparent that average  $K_{\text{IC}}$  value obtained from indented and precracked specimens were high compared to the  $K_{\text{R}}$  (fracture resistance) value reported by Krause *et al.*<sup>2</sup> in indentation-strength tests. The large difference in toughness between the ASTM STP 410 method in the present study and the indentation-strength method,<sup>2</sup> could primarily be accounted for the difference in the crack tip loading rates and the technique used for determination of toughness.<sup>14</sup> The scatter in the  $K_{\text{IC}}$  values obtained from the precracked specimens (Table 1), could be due to the non-uniform distribution of the SiC whiskers, apart from the system error inherent in the  $K_{\text{IC}}$  determination. For the same composite, Krause *et al.*<sup>2</sup> used a parametric representation of the fracture resistance as a fractional power function in crack extension of the form:

$$K_{\text{R}} = K_0(\Delta C/C_0)^m$$

where  $K_{\text{R}}$  is a parameter describing the fracture resistance for an  $R$ -curve type material,  $m$  measures the relative steepness of the  $R$ -curve,  $K_0$  is the fracture resistance that corresponds to the smallest indentation crack radius,  $C_0$  is the smallest indentation crack that causes failure in flexural testing and  $\Delta C$  is the crack extension ( $= C - C_p$ ).  $C$  is the total crack length.  $C_p$  is the traction-free portion from the outset, so  $C_p = 0$ . When  $m = 0$   $K_{\text{R}}$  is

invariant with  $\Delta C$  and  $K_R = K_0 = K_c$ . The values of  $m$ ,  $C_0$  and  $K_0$  for the same composite under the present investigation were optimised to be 0.08,  $18.1 \mu\text{m}$  and  $5.35 \text{ Mpa}\sqrt{\text{m}}$ , respectively.<sup>2</sup> They have observed that  $K_R$  of the same composite in the present investigation, increased with crack extension, the so-called rising  $R$ -curve phenomenon. This behaviour was explained in terms of the whiskers acting as elastic restraints to the point of rupture at small crack wall separations.<sup>2</sup> In  $R$ -curve type materials, the toughness varies with crack extension, so that if one attempts to measure the toughness with a procedure that ignores this as do most of the earlier indentation techniques, one would find that the 'measured toughness' depends on indentation loads.<sup>2</sup>

The microstructural toughening of this composite is essentially due to the grain bridging phenomenon of the SiC whiskers.<sup>2,14,16</sup> The crack deflection (Fig. 5) and the branching behaviour of the cracks<sup>14,16</sup> at the corners of the indentation in the present material has been explained in terms of a grain bridging phenomenon associated with microstructural toughening and could partly account for the rising trend in  $K_R$ .<sup>2,14</sup> Possibly, on the scale of grain sizes, the grains of alumina were entangled with the SiC whiskers in such a way that, because of the presence of a complex residual stress field, high resistance to crack propagation was created and hence the crack tended to find a new starting point at the weakest area away from this region.

In future, the fracture toughness anisotropy of this composite should also be studied to estimate the variation of  $K_{IC}$  with orientation of whiskers and with respect to the crack plane perpendicular, crack growth at right angles and crack plane parallel, crack growth parallel to the hot-pressing direction of the billet. As a scope for further work in this area, the effect of bridge span (which is an important precracking parameter), on the precrack length and  $K_{IC}$  of the material needs to be studied.

### 3.3 Fatigue crack growth rate, FCGR studies

It was possible to study the fatigue crack propagation behaviour of this material using four point bend loading. The results of FCGR studies on this composite using indented and precracked samples have already been reported.<sup>14</sup> Recently, FCGR and  $K_{IC}$  data of  $\approx 12.64 \text{ wt}\%$  SiC whisker reinforced alumina composite has been reported by Dauskardt *et al.* using compact tension (CT) specimens.<sup>8</sup> The difference in toughness and FCGR of this composite containing different whisker volume and porosity content, with that of the composite under present investigation has already been discussed.<sup>14</sup> Figure 6 reveals the FCGR behaviour of

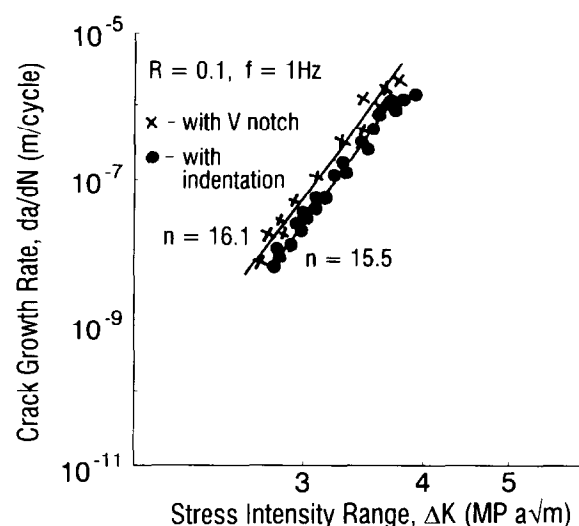


Fig. 6. Fatigue crack growth data of 25 wt% SiC reinforced  $\text{Al}_2\text{O}_3$  composite.

this material using precracked four point bend samples with notch as well as indent as crack starters. Plots of  $da/dN$  versus  $\Delta K$  for 25 wt% SiC whisker reinforced alumina composite (Fig. 6) exhibited a single linear stage without the presence of detectable threshold. This composite is therefore susceptible to fatigue crack growth phenomenon in a manner similar to the metallic materials, but with high  $n$  values. This behaviour was observed earlier too.<sup>14</sup>

Table 2 shows the Paris law constants from FCGR data. The FCGR data of this material were fitted to the usual Paris equation  $da/dN = A(K)^n$ ,<sup>8,14</sup> and the fatigue crack growth rate ( $da/dN$ ) increased linearly with the stress intensity range ( $\Delta K$ ) in a log-log plot (Fig. 6). It has already been discussed earlier that if the residual surface stress ahead of the advancing crack in fatigue<sup>14</sup> is tensile in nature, it would increase the the apparent surface crack and thus enhance the FCGR of the material. It is clearly demonstrated from Fig. 6 that FCGR at a given  $\Delta K$ , for a V-notch with precrack as a crack starter was slightly high compared to that in the case of indentation with precrack as a crack starter. This is mainly due to tensile residual stresses induced in the specimen in the process of inserting a V-notch.<sup>15</sup> This is also indicative of a higher exponent,  $n = 16.1$  of the the Paris law equation (Table 2), resulting in a minor increase in the crack growth rate at a given  $\Delta K$  (Fig. 6), compared to that of the indented and

Table 2. Paris law constants ( $A$  and  $n$ ) for 25 wt% SiC whisker reinforced  $\text{Al}_2\text{O}_3$  composite from FCGR data

Type of four-point bend specimen	$A$ , [ $\text{m/cycle} (\text{Mpa}\sqrt{\text{m}})^{-n}$ ]	$n$
Notched and precracked	$5.579 \times 10^{-16}$	16.1
Indented and precracked	$3.4 \times 10^{-15}$	15.5

precracked specimens. However, unlike the fracture toughness data, the fatigue crack growth rates using both the crack starters were not widely different.

#### 4 Conclusion

There was 100% success in precracking ceramic specimens with the help of the articulated bridge fixture. The use of indentation as a crack starter influenced mainly the  $K_{IC}$  value possibly due to creation of tractions on the entire precrack and also owing to the presence of certain amount of compressive residual stresses ahead of the advancing crack. Machining a notch produced significantly higher residual stresses along the whole notch tip and so the  $K_{IC}$  values in the notched and precracked specimens were 7.2% lower than those of the indented and precracked specimens. However, fatigue crack growth rates using both the crack starters were not widely different.

#### Acknowledgements

The author is grateful to NIST (National Institute of Standards and Technology), Gaithersburg MA, USA for supplying 30 bend specimens of the ceramic composite to NML (National Metallurgical Laboratory), Jamshedpur, India. The author is highly obliged to Dr C. K. Gupta of BARC (Bhabha Atomic Research Centre), Bombay, for supplying us the additional six specimens of the same material from NIST, USA, for repeating the tests. The author would also like to thank Professor S. Banerjee, Director, RDCIS-SAIL, Ranchi, India for his important suggestions in designing the articulated bridge fixture. Finally, the author expresses his thanks to Professor P. Ramachandra Rao, Director, NML Jamshedpur, India, regarding his permission for publishing this manuscript.

#### References

- Ponton, C. B. and Rawlings, R. D., Dependence of the vickers indentation fracture toughness on the surface crack length. *Br. Ceram. Trans. J.*, 1989, **88**, 83–90.
- Krause Jr, R. F. and Fuller Jr, E. R., Fracture resistance behaviour of a silicon carbide whisker reinforced alumina with different porosities. *J. Am. Ceram. Soc.*, 1990, **73**(3), 559–566.
- Bhattacharya, A. K. and Petrovic, J. J., Hardness and fracture toughness of SiC-particle-reinforced MoSi<sub>2</sub> composite. *J. Am. Ceram. Soc.*, 1991, **74**(10), 2700–2703.
- Swain, M. V., R-curve behaviour and thermal shock resistance of ceramics. *J. Am. Ceram. Soc.*, 1990, **73**(3), 621–628.
- Anderson, R. M. and Braun, L. M., Technique for the R-curve determination of Y-TZP using indentation-produced flaws. *J. Am. Ceram. Soc.*, 1990, **73**(10), 3059–3062.
- Ramachandran, N. and Shetty, D. K., Rising crack growth resistance R-curve behaviour of toughened alumina and silicon nitride. *J. Am. Ceram. Soc.*, 1991, **74**(10), 2634–2641.
- Dauskardt, R. H., Marshall, D. B. and Ritchie, R. O., Cyclic fatigue-crack propagation in magnesia-partially stabilized zirconia ceramics. *J. Am. Ceram. Soc.*, 1990, **73**(4), 893–903.
- Dauskardt, R. H., James, M. R., Porter, J. R. and Ritchie, R. O., Cyclic fatigue crack growth in a SiC-whisker-reinforced alumina ceramic composite: long and small crack behaviour. *J. Am. Ceram. Soc.*, 1992, **75**(4), 759–771.
- Sadahiro, T. and Takatsu, S., In *Modern Developments in Powder Metallurgy*, Vol. 14, ed. H. H. Hausner, H. W. Antes and G. D. Smith. Plenum, New York, 1981, pp. 561–572.
- Warren, R. and Johannesson, B., Creation of stable cracks in hard metals using bridge indentation. *Powder Metall.*, 1984, **27**(1), 25–29.
- Hansson, T., Warren, R. and Wasen, J., Fracture toughness anisotropy and toughening mechanisms of a hot pressed alumina reinforced with silicon carbide whiskers. *J. Am. Ceram. Soc.*, 1993, **74**(4), 841–848.
- Nose, T. and Fuji, T., Evaluation of fracture toughness for ceramic materials by a single-edged-precracked-beam method. *J. Am. Ceram. Soc.*, 1988, **71**(5), 328.
- Baron, I., Beals, J. T. and Gary, L., Fracture toughness of ceramic precracked bend bars. *J. Am. Ceram. Soc.*, 1990, **73**(8), 2519–2522.
- Ray, A. K., Fuller Jr, E. R. and Banerjee, S., Fatigue crack growth rate and fracture toughness of 25 wt% silicon carbide whisker reinforced alumina ceramic composite with residual porosity. *J. Eur. Ceramic. Soc.*, 1996, **16**, 503–513.
- Ray, A. K., Banerjee, S., Fuller Jr, E. R., Das, S. K. and Das, G., Fractography as well as fatigue and fracture of 25 wt% silicon carbide whisker reinforced alumina ceramic composite. *Bull. Mat. Sc.*, 1994, **17**(6), 893–910.
- Ray, A. K., Das, S. K., Roy, P. K. and Banerjee, S., Fractography of the fatigued and fractured regions in a silicon carbide whisker reinforced alumina composite. *J. Eur. Ceram. Soc.*, 1995, **15**, 191–199.
- Brown Jr, W. F. and Srawley, J. E., In *ASTM STP 410*. American Society for Testing and Materials, Philadelphia, PA, 1966.
- Yamade, Y. and Kishi, T., Acoustic emission study for fracture origin of sintered mullite in 4-point bending test. *The Sumitomo Search*, 1991, **45**(3), 17–24.
- Lange, F. F., James, M. R. and Green, D. J., Determination of residual stresses caused by grinding in polycrystalline Al<sub>2</sub>O<sub>3</sub>. *J. Am. Ceram. Soc.*, 1983, **66**, C16.
- Abuhasan, A., Balasingh, C. and Predecki, P., Residual stresses in alumina/silicon carbide (whisker) composites by X-ray diffraction. *J. Am. Ceram. Soc.*, 1990, **73**(8), 2474–2484.
- Cook, R. F., Lawn, B. R., Dabbs, T. P. and Chantikul, P., Effect of machining damage on the strength of a glass ceramic. *J. Am. Ceram. Soc.*, 1981, **64**, C121.
- Green, D. J., Lange, F. F. and James, M. R., Factors influencing residual surface stresses due to a stress-induced phase transformation. *J. Am. Ceram. Soc.*, 1983, **66**, 623–629.
- Marshall, D. B. and Lawn, B. R., An indentation technique for measuring stresses in tempered glass-surfaces. *J. Am. Ceram. Soc.*, 1977, **60**, 86–87.
- Ikuma, Y. and Virkar, A. V., Crack-size dependence of fracture toughness in transformation-toughened ceramics. *J. Mater. Sci.*, 1984, **19**, 2223–2228.
- Ray, A. K., Bhattacharya, D. K. and Das, G., Acoustic emission studies during indentation on ceramic and ceramic composite. In *Proceedings of 14th World Conference on Non Destructive Testing (WCNDT)*, Vol. 4, ed. C. G. K. Nain, Baldevraj, C. R. L. Murthy and T. Jaykuma, Oxford and IBH Publishing House, 1996, pp. 2445–2448.