Indentation Fatigue of Silicon Carbide and Silicon Nitride*

Eiju Takakura** and Susumu Horibe**

The fatigue failure mechanism of ceramic material has been hardly made clear under cyclic loading. In this study, we examine the damage of the surface area in ceramics under local cyclic compressive loading by using the cyclic indentation method (indentation fatigue), and discuss its mechanism. Four kinds of materials (transgranular and intergranular fracture type) were used for the experiment. The indentation fatigue test was carried out by indenting repeatedly onto the same point of the specimen surface by using a Vickers hardness tester. The median/radial crack length, the number of cycles to chipping and the size of the chipping area were measured, and the lateral crack growth rate was computed. The fracture surface in the chipping area was also observed by a scanning electron microscope. The median/radial crack growth was slightly accelerated by cyclic loading in any material. During cyclic indentation, the lateral crack propagated, which resulted in the chipping phenomenon. Such damage accumulation remarkably occurred in the transgranular fracture type material rather than in the intergranular fracture type one. Striations were observed in the chipping area and the striation spacing approximately corresponded to the size of the chipping area divided by the cycles to chipping, i.e., the lateral crack propagation rate. The observed zigzag propagation of the lateral crack is considered to be attributable to the difference between the stress fields under loading and unloading. The reason why the lateral crack growth and the resultant chipping behavior are suppressed in intergranular fracture type materials is most likely that crack branching and microcracking easily occur in those materials so that the stress concentration at the crack tip is relieved.

(Received February 19, 1991)

Keywords: silicon carbide, silicon nitride, indentation fatigue, radial crack, lateral crack, chipping, striation, transgranular fracture, intergranular fracture

I. Introduction

The fatigue in ceramic materials under cyclic loading has been actively studied since several years ago. As for significant engineering materials such as silicon nitride⁽¹⁾⁻⁽⁴⁾, silicon carbide⁽⁵⁾⁽⁶⁾, aluminum oxide⁽⁵⁾⁽⁷⁾⁻⁽¹⁰⁾ and zirconium oxide⁽¹¹⁾⁽¹²⁾, their fatigue properties at room temperature have so far been reported phenomenologically. However, the fatigue failure mechanism of these materials is hardly clarified, yet⁽¹³⁾. Therefore, the elucidation of such mechanism is an important subject which we should set about from now on.

In this study, we carried out cyclic indentation tests by using a Vickers indenter. We examined the damages caused by cyclic application of a local compressive load onto surfaces of silicon carbide and silicon nitride ceramics (we called it indentation fatigue), and discussed their mechanism. Guio *et al.*⁽¹⁴⁾⁽¹⁵⁾ have first used this method for indentation fatigue of aluminum oxide, and reported that a lateral crack propagates with indentation cycles which results in chipping behavior. This is supposed to be very important from the view point of engineering practice. Moreover, it is also significant for fundamentally understanding fatigue mechanism in ceramic materials such as the damage accumulation in crack-asperity contact area (the region in which both crack faces are misfitting because of residual stress)⁽¹⁶⁾.

Fig. 1 Schematic illustration of the crack systems produced in a brittle material by a Vickers indenter: (a) Side view, (b) Top view.

When brittle material is indented by a Vickers indenter, semicircular median/radial cracks approximately perpendicular to the material surface and sectorial lateral cracks parallel to it are produced (Fig. 1(a), (b)).

In this work, we have examined the propagation of these two types of cracks during cyclic indentation and especially studied the lateral crack propagation mechanism.

* This paper was originally published in Japanese in J. Japan Inst. Metals, **54** (1990), 611.

** Tsukuba Laboratories, National Research Institute for Metals, Tsukuba 305, Japan.

II. Experimental Procedure

1. Materials

The tested materials are two kinds of silicon carbide and silicon nitride ceramics; i.e., four types of materials in total. Their fabrication process, Vickers hardness and fracture toughness are shown in Table 1. SiC-T, SiC-I and Si₃N₄-I were produced by pressureless sintering with additives shown in Table 1. Si₃N₄-T[†] was prepared by HIP process without additives⁽¹⁷⁾. Fracture surfaces of these materials produced by flexural strength testing in the air are shown in Fig. 2. SiC-T and Si₃N₄-T are transgranular fracture type materials, whereas SiC-I and Si₃N₄-I are intergranular fracture type ones. The Mode I cyclic fatigue behavior in these materials has been reported elsewhere⁽³⁾⁽⁶⁾⁽¹⁸⁾ by one of the authors, and it has

[†] Si₃N₄-T was produced by Kobe Steel, Ltd. in "Research and Development Project on Basic Technologies for Future Industries".

been revealed that an effect of cyclic loading on crack propagation appears in only intergranular fracture type materials⁽¹⁸⁾. The dimensions of specimens are 20 mm × 4 mm × 3 mm and indentation was applied onto a surface of 20 mm × 4 mm.

2. Experiment

Indentation fatigue test was carried out by indenting repeatedly onto the same point of the specimen surface by using a Vickers hardness tester or a Microvickers hardness tester, as schematically shown in Fig. 3. Applied load was in the range of 4.9 N to 294 N. Holding time for indentation was 5 s and the interval was approximately 60 s.

Median/radial crack growth was examined with an optical microscope by measuring the crack length at periodic intervals. As for the propagation of subsurface lateral crack, the number of the cycles to chipping, which is the final result of the lateral crack growth, was measured. The size of chipping area defined as the distance from a center of an indent to the end of the chip-

Table 1 Fabrication process, mechanical test data and microstructural observation for selected silicon carbide and silicon nitride.

Material	Fabrication	Additives	Hardness (GPa)	K_{IC} (MPa · m ^{1/2})	Fracture appearance in flexural testing
SiC-T	Pressureless sintering	B, C	24	2.6	transgranular
SiC-I	Pressureless sintering	Al ₂ O ₃	19	4.8	intergranular
Si ₃ N ₄ -T	HIP	non	18	2.9	transgranular
Si ₃ N ₄ -I	Pressureless sintering	Y ₂ O ₃ + MgAl ₂ O ₄	13	6.5	intergranular

Fig. 2 Fracture surface produced by flexural strength testing: (a) SiC-T, (b) SiC-I, (c) Si₃N₄-T and (d) Si₃N₄-I.

Fig. 3 Schematic illustration for the indentation fatigue.

ping region was also measured with an optical microscope. The fracture surface of the chipping area was observed by a scanning electron microscope.

III. Results

1. Median/radial crack

The growth behavior of median/radial crack during cyclic indentation loading is shown in Fig. 4. Applied load was 98 N in Si_3N_4 -I and 9.8 N in other three kinds of specimens. In any material, the crack tended to slightly propagate in the early stage of cyclic indentation, followed by arresting. It seems important to clarify which causes such growth of median/radial crack, static fatigue or cyclic fatigue. Therefore, the indentation of 45 s holding time, corresponding to the sum of holding times from second indentation to tenth one, was applied to the specimen which had experienced the first indentation of 5 s holding. The increment in this indentation Δa_{static} was compared to that in the cyclic indentation (the difference between the crack length after the first indentation and that after the tenth one) Δa_{cyclic} (Table 2). From these results, it has been revealed that in any material not only

Fig. 4 The development of radial crack under cyclic indentation.

Table 2 Comparison of radial crack growth between static and cyclic loading.

Sample	Indentation load (N)	Δa_{cyclic} (μm)	Δa_{static} (μm)
SiC-T	9.8	9.4	3.4
SiC-I	9.8	1.2	0.1
Si_3N_4 -T	9.8	8.2	3.4
Si_3N_4 -I	98.0	21.9	6.1

static fatigue but also cyclic fatigue is involved in such median/radial crack growth.

2. Lateral crack

The relation between the indentation load and the number of cycles to chipping N_f is shown in Fig. 5. It is found that N_f s of intergranular fracture type materials such as SiC-I and Si_3N_4 -I are much larger than those of transgranular fracture type ones. Plots with arrows show that no chipping occurred in the corresponding numbers of cycles.

The relation between the size of chipping area S_c and the indentation load (peak applied load L_p) in three kinds

Fig. 5 Number of cycles required to produce chipping in silicon carbide and silicon nitride under cyclic indentation.

Fig. 6 The dependence of chipping size on peak applied load.

Fig. 7 SEM photographs of morphologies of chipping: (a) SiC-T (9.8 N), (b) Si₃N₄-T (9.8 N) and (c) Si₃N₄-I (49 N). Note that striations are observed in SiC-T and Si₃N₄-T, but not in Si₃N₄-I.

of materials in which chipping was observed, is shown in Fig. 6. The chipping size of intergranular fracture type material is smaller than that of transgranular fracture type one.

In order to examine the crack growth behavior morphologically, we observed the surface of chipping area by SEM (Fig. 7). In SiC-T and Si₃N₄-T of transgranular fracture type, the stripes like concentric circles were clearly found around an indent (Fig. 7(a), (b)). These are supposed to be striations which are developed in every indentation cycle. On the other hand, in Si₃N₄-I of intergranular fracture type, such clear stripes were not seen and the fracture surface was rather uniform (Fig. 7(c)).

IV. Discussion

1. Median/radial crack growth

The median/radial crack growth during cyclic indentation is supposed to be produced by Mode I stress cycling. Accordingly, this is consistent with the data reported so far⁽¹⁾⁻⁽⁴⁾⁽¹⁸⁾ and easily understood that an effect of cyclic loading was found in intergranular fracture type materials. On the other hand, it is not easily understandable that median/radial crack slightly grew under cyclic indentation in transgranular fracture type materials, but we consider it as follows. Median/radial crack growth is influenced by lateral crack growth behavior. As supposed by the size of chipping area shown in Fig. 6, lateral crack propagates over the corresponding median/radial crack length. In such case, the median/radial crack growth can be achieved by breaking only the volume between a surface and a lateral crack. Therefore, it is assumed that the median/radial crack growth observed on the surface is made easier as a lateral crack propagates.

2. Lateral crack growth

The lateral crack growth rate S_c/N_f is shown in Fig. 8, which was computed by using the mean values of the chipping area size S_c and the number of cycles to chipping N_f on the basis of the results presented in Fig. 5 and Fig. 6. In this figure, an interval of the stripes s clearly seen in the SEM pictures of chipping area (Fig. 7(a), (b)) is also plotted (solid symbols). Since lateral crack tends to propagate to a large extent just before chipping occurs, it seems that S_c/N_f is larger than s . However, both are approximately in the same order so that such stripes are concluded to be striations.

From this figure, it is also revealed that the lateral crack propagation rate is considerably larger in transgranular fracture type materials than in intergranular fracture type ones.

3. Lateral crack propagation mechanism

Lateral crack propagation during cyclic loading is explicable by a mechanism presented in Fig. 9. The first indentation produces median/radial cracks and subsequent unloading generates lateral cracks. This has been confirmed by the analysis of the indentation stress field

Fig. 8 Lateral crack propagation rate as a function of peak applied load: open symbols indicate the chipping size divided by the cycles to chipping, and closed symbols the striation spacing.

Fig. 9 Lateral crack propagation mechanism. (U_i : unloading, L_i : loading)

by Chiang *et al.*⁽¹⁹⁾ In the second indentation the median/radial crack slightly propagates, because the new stress field different from that in the first indentation is produced by the lateral crack formation (initiation and propagation), as mentioned in IV.1 (realization of irreversible process). Since the striations like steps are seen in the chipping area (Fig. 7(a), (b)), lateral crack seems to propagate again during the second indentation. At this moment, a shear stress acts on the lateral crack to push out the region above a crack face and as a result, Mode II crack growth is presumably induced. Such lateral crack propagation during loading process produces a new residual stress field, which makes the lateral crack propagate again during the following unloading process (mainly because of normal tensile stress). It is supposed that the repetition of the above processes makes lateral crack zigzag propagate and results in chipping behavior. This unique crack propagation behavior leaves its trace as a striation.

4. Why is chipping behavior (the lateral crack growth) restrained in intergranular fracture type materials?

From the results shown in Fig. 5 and Fig. 8, it is found that the lateral crack growth and the resultant chipping

tend to easily occur in transgranular fracture type materials but not in intergranular fracture type ones. This is mainly because crack branching and microcracking readily occurs in intergranular fracture type materials so that the stress concentration at the lateral crack tip is relieved. Furthermore, in these materials, Mode II fracture by shear stress during a loading process might be suppressed because of the ruggedness of crack surfaces. Being affected by these factors, lateral crack growth is supposed to be restrained in intergranular fracture type materials.

The chipping behavior caused by indentation is a problem of "small breakage of brittle materials". It is an unfavorable phenomenon. As shown in Table 1, transgranular fracture type materials in which chipping easily occurs generally have lower fracture toughness rather than intergranular fracture type ones. Therefore, improving the toughness in engineering ceramics is an important subject from the view point of protection against the indentation fatigue.

V. Conclusion

Indentation fatigue in silicon carbide and silicon nitride ceramics of intergranular fracture type and transgranular fracture type was investigated and the following conclusions were obtained.

- (1) In every material, median/radial crack slightly propagated during cyclic indentation. This crack growth is caused by not only static fatigue but also cyclic fatigue.
- (2) Lateral crack propagated under cyclic indentation, which resulted in chipping. Such behavior occurs remarkably in transgranular fracture type materials rather than in intergranular fracture type ones.
- (3) Striations, which correspond to the lateral crack growth per cycle, have been observed in the chipping area of transgranular fracture type materials.
- (4) On the basis of these experimental results, lateral crack propagation mechanism was proposed.
- (5) Lateral crack growth and the resultant chipping behavior were suppressed in intergranular fracture type materials. This is mainly because crack branching and microcracking easily occur in those materials so that the stress concentration at the crack tip is relieved.

Acknowledgment

The authors thank for the help from Science and Technology Agency, Agency of Industrial Science and Technology, Engineering Research Association for High Performance Ceramics and Kobe Steel, Ltd. Discussion with Dr. M. Nakamura, N.R.I.M. greatly stimulated and clarified the authors' thoughts about this work.

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