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Fatigue damage in ceramic materials caused by repeated indentation

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Indentation fatigue is the damage accumulation in the surface layer of ceramics caused by local cyclic loading. It has been found that there are two types of indentation fatigue behaviour, Types I and II. Type I is characterized by a slight increase in indentation-induced damaged zone with increased number of indentation cycles, followed by the abrupt chipping of the surface layer. On the other hand, Type II behaviour is characterized by pronounced enlargement of the damaged zone in early cycles. Lateral cracking behaviour during cyclic indentation in various ceramics was also studied by scanning electron microscopy and scanning acoustic microscopy. It was confirmed that a lateral crack zig-zag propagates with repeated indentation in silicon nitride of transgranular fracture type. The relations between lateral cracking, microcracks and the resultant damaged zone in alumina and magnesium oxide were investigated in detail and their indentation fatigue mechanism is discussed.

1. Introduction

It is widely known that static fatigue in ceramic materials is caused by stress corrosion cracking due to moisture in air [1–3]. On the other hand, it has been believed for a long time that cyclic fatigue never occurs in ceramic materials because of the absence of plasticity. However, the latest active works made it clear that crack growth occurs under cyclic loading conditions in silicon nitride [4–7], alumina [8–12] and zirconia [13, 14], and is inexplicable by the subcritical cracking mechanism due to static fatigue (environmental-assisted cracking process) alone [15]. Recently, a technique using the repeated indentation on to the surface of ceramics was proposed as a method for investigating cyclic fatigue behaviour in ceramic materials by Guiu and co-workers [16, 17]. They found that a lateral crack propagates with indentation cycles, which results in chipping behaviour [16, 17], and that a fatigue limit exists in terms of cyclic compressive forces across the lateral crack faces [18]. Elucidation of the mechanism of this behaviour is supposed to be very important not only from the view point of engineering practice, such as contact or fretting fatigue, but also for fundamentally understanding the fatigue mechanism of the damage caused by crack-asperity contact (misfitting of crack faces because of residual stress release, or trapping of debonded particles).

In previous papers we have shown that in transgranular fracture-type materials, striations are seen in the chipping area (Fig. 1) which is attributable to lateral crack propagation, and we proposed a model of indentation fatigue mechanism on the basis of the difference between the stress fields under loading and unloading (Fig. 2) [19, 20]. However, lateral crack propagation behaviour during indentation fatigue has not been fully elucidated because of difficulty in observing the inside of materials.

In this work, fatigue damage in various kinds of ceramics caused by repeated indentation was investigated in terms of brittleness/ductility (i.e. covalent bonding/ionic bonding characteristics) and grain size, and the indentation fatigue mechanism was systematically discussed.

2. Experimental procedure

2.1. Materials

The tested materials were silicon carbide, silicon nitride, sialon, alumina and magnesium oxide, which were produced by sintering and/or hot isostatic pressing (HIP) processes. Silicon carbide, silicon nitride and sialon are highly covalently bonded materials, which are characterized by a brittle manner in fracture. Magnesium oxide is a highly ionic bonded material and more ductile than the silicon compounds. Alumina has a medium nature between the former and the latter. In alumina, magnesium oxide and sialon, two kinds of different grain-sized materials are used. Their final fabrication processes, additives, Vickers hardness, grain size, and fracture appearance in the conventional flexural strength testing in air at room temperature, are shown in Table I. These specimens were ground and lapping-polished with diamond paste and alumina aqueous slurries. The specimen dimensions were 20 mm × 4 mm × 3 mm and the indentation was applied on to a 20 mm × 4 mm surface.

2.2. Testing procedure

Indentation fatigue tests were carried out by indenting repeatedly on to the same point of the specimen

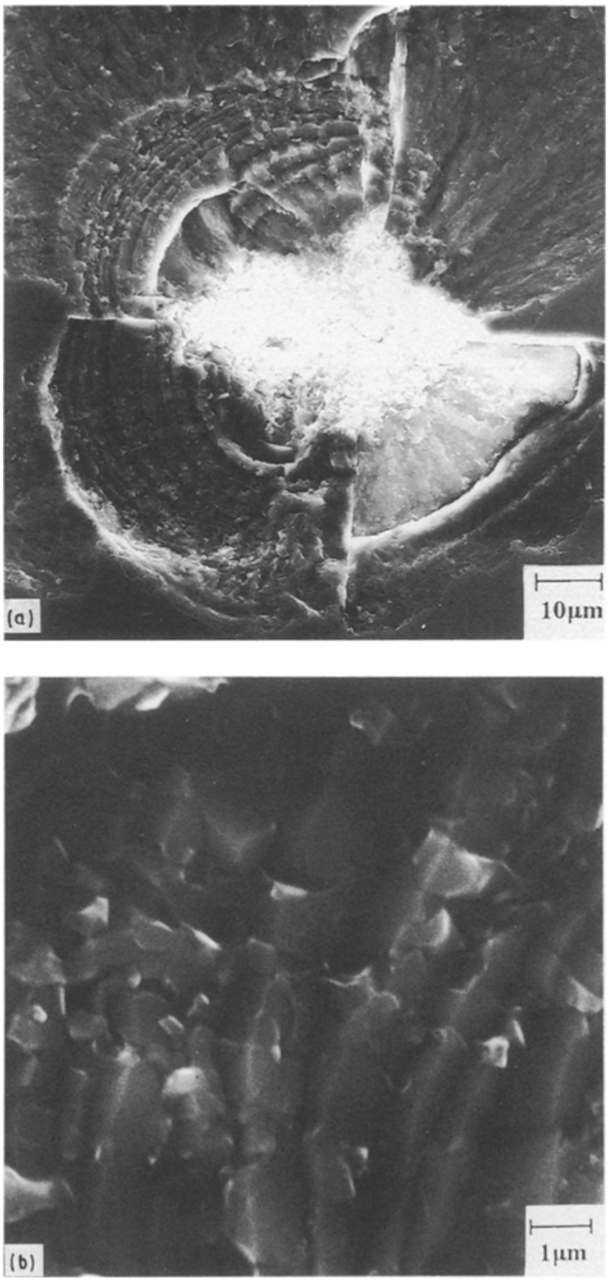

Figure 1 Scanning electron micrographs of the chipped area produced in silicon nitride of transgranular fracture type. Note that striations like concentric circles are observed: (a) low magnification, (b) high magnification.

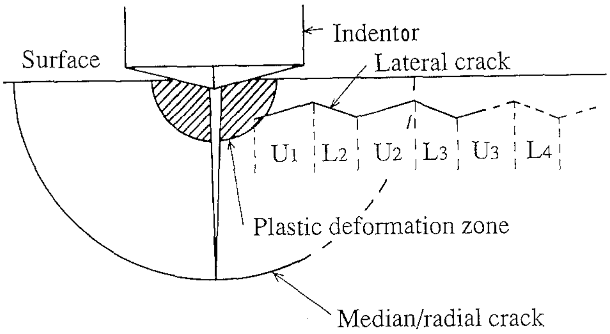

Figure 2 The lateral crack propagation mechanism. U_i, unloading process; L_i, loading process.

surface using a Vickers hardness tester or a Microvickers hardness tester, as shown schematically in Fig. 3. The applied load was in the range 9.8–490 N. The holding time for indentation was 5 s and the intervals were approximately 60 s. The surface cracking behaviour was observed by optical microscopy. Subsurface cracking behaviour was examined by scanning acoustic microscopy (SAM) at frequencies of 200 and 400 MHz, with a water medium. The fracture surface in the damaged area was also inspected by scanning electron microscopy (SEM).

3. Results and discussion

3.1. Damage accumulation caused by cyclic indentation

Fig. 4 shows the extending behaviour of the damaged area produced by cyclic indentation [21]. In silicon carbide, silicon nitride and sialon ceramics with strong covalent bonding, very slight increase of the damaged zone size was observed during cyclic indentation for some tens of cycles, followed by the abrupt extension of this area at a certain number of cycles (Fig. 4a). On the other hand, in magnesium oxide, which has small but significant plasticity attributable to ionic bonding, it has been found that the damaged zone is pronouncedly enlarged in the early stages, with the number of indentation cycles (Fig. 4b). Fine-grained alumina behaved in a similar fashion to silicon carbide and silicon nitride, while this damage accumulation in coarse-grained alumina resembled the behaviour of magnesium oxide. Consequently, with respect to the appearance of such damage accumulation, materials can be classified into two groups, Types I and II, as shown in Fig. 5. Type I indentation fatigue behaviour implies that the lateral crack propagation is caused by cyclic loading, which results in chipping of part of the surface layer. It is of interest that alumina behaved as

TABLE I Fabrication process, additives, Vickers hardness, grain size, and fracture appearance in the conventional flexural strength testing for the materials tested

Material	Final fabrication	Additives	Grain size (μm)	Hardness (GPa)	Fracture appearance
MgO	Pressureless	None	25	5	Transgranular
MgO	Pressureless	None	10	5	Transgranular
Al_2O_3	HIP	None	10	13	Intergranular
Al_2O_3	HIP	None	2	18	Intergranular
SiC	Pressureless	B, C	5	24	Transgranular
SiC	Pressureless	Al_2O_3	1	19	Intergranular
Si_3N_4	HIP	None	2	18	Transgranular
Si_3N_4	Pressureless	Y_2O_3, $MgAl_2O_4$	0.5	13	Intergranular
Sialon	HIP	Y	1	19	Intergranular
Sialon	HIP	Y	0.3	19	Intergranular

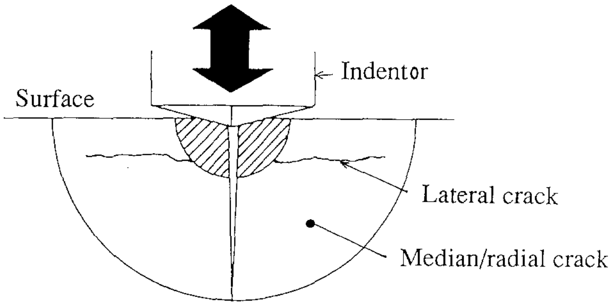

Figure 3 Schematic illustration of the testing procedure for indentation fatigue.

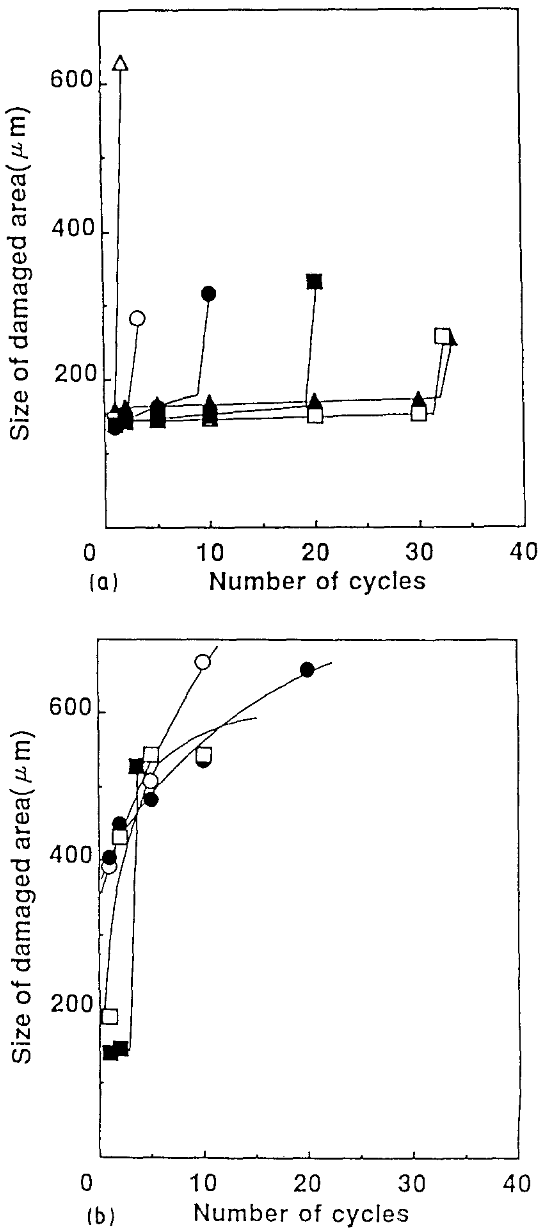

Figure 4 Development of the damaged area under cyclic indentation; peak applied load = 196 N. (a) Covalently bonded materials: SiC (○) transgranular, (●) intergranular; Si_3N_4 (△) transgranular, (▲) intergranular; sialon (□) 0.3 μm, (■) 1.0 μm. (b) Ionically bonded materials. MgO (○) 10 μm, (●) 25 μm; Al_2O_3 (□) 10 μm, (■) 2 μm.

either Type 1 or Type II, depending upon the grain size.

Fig. 5 illustrates the dependence of indentation fatigue type upon the grain size and ductility/brittleness.

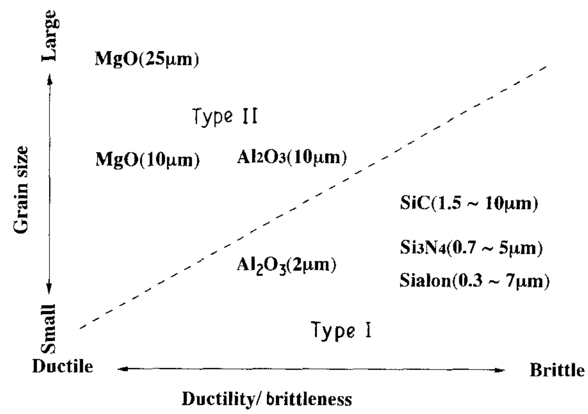

Figure 5 The dependence of the type of indentation fatigue behaviour on grain size and plasticity.

From this figure, it is significantly clear that Type I materials are characterized by brittleness and/or small grain size, whereas Type II materials are plastic and/or have a large grain size.

3.2. Type I indentation fatigue

In order to identify the sectional view of a lateral crack, we broke the indented specimens of transgranular fracture type silicon nitride by deliberately making a median/radial crack grow, and observed the fractured surfaces by SEM (Fig. 6). When a single indentation of 9.8 N was applied, it was seen that the microcracking area was produced beneath the indent and that the lateral crack was initiated from the boundary between this area and the surrounding elastic zone (Fig. 6a). On the fractured surface of the specimen indented for five cycles, zigzag propagation of a lateral crack was observed (Fig. 6b). From this figure, we presume that the lateral crack initiates and propagates slightly upwards (towards the surface) during the unloading processes, and propagates downwards during the loading processes. When a 49 N load was applied, a semicircular crack, not seen in the 9.8 N load sample, was observed apart from the microcracking area (Fig. 6c). The lateral crack was initiated not from the boundary between the microcracking area and the surrounding elastic zone, but from the crack region. It is usually considered that lateral cracking is initiated from the border between the plastic-deformation zone and the surrounding elastic zone. Hence, the semicircular crack is supposed to be produced along the border between the plastic zone and the surrounding elastic zone, and the geometry of this crack is considered to be hemispherical. Presumably such a hemispherical crack is produced at the same time and also in a similar manner to the lateral crack formed during the unloading process of indentation stress. The fact that no such crack is observed for the 9.8 N indentation implies that there exists a critical stress for that crack to initiate.

It is possible to observe the lateral crack behaviour prior to chipping during cyclic indentation by SAM. Fig. 7 shows the SAM micrographs of silicon nitride of transgranular fracture type after the indentation of 1 cycle and 10 cycles. The lateral crack was seen in the

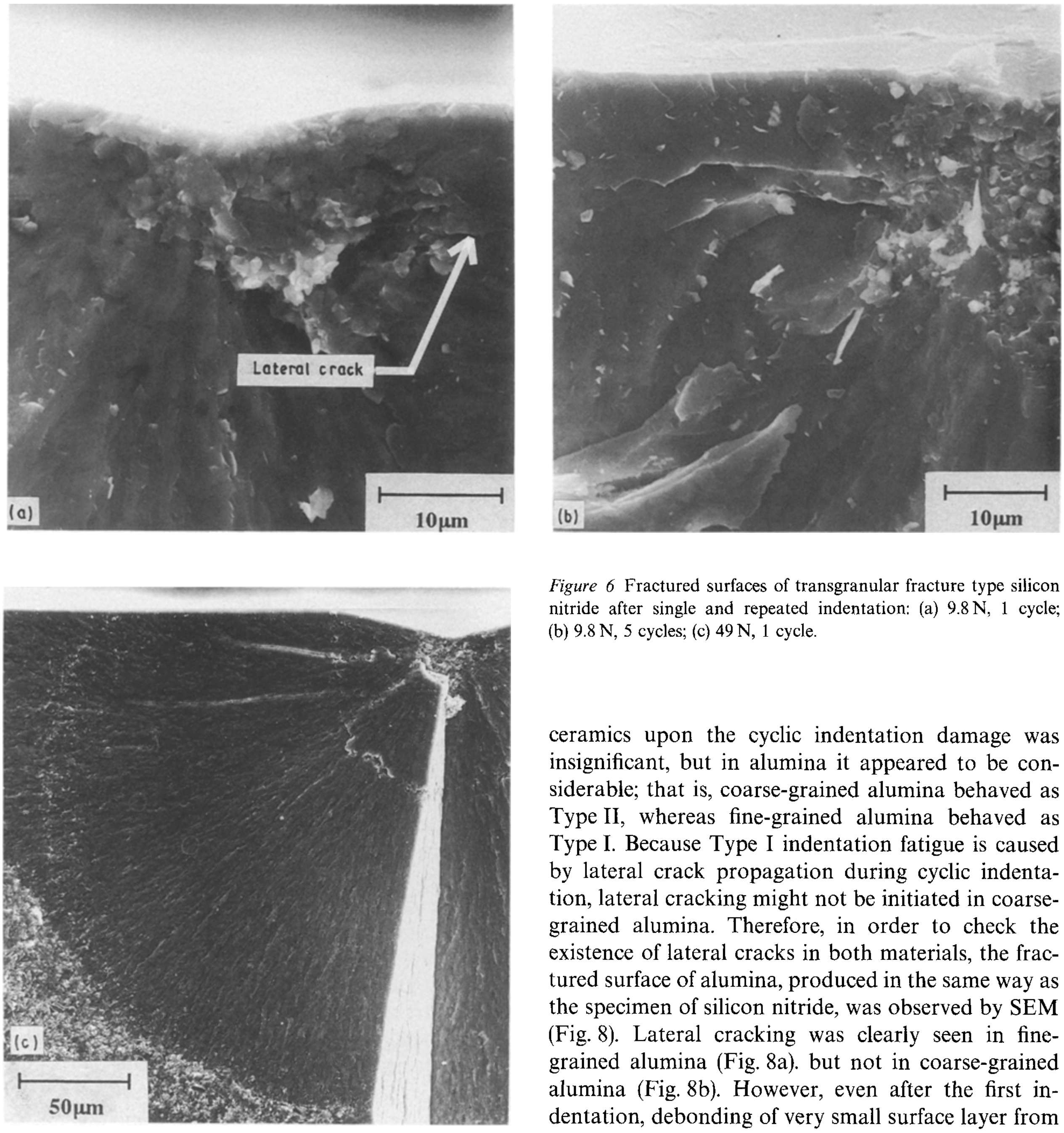

Figure 6 Fractured surfaces of transgranular fracture type silicon nitride after single and repeated indentation: (a) 9.8 N, 1 cycle; (b) 9.8 N, 5 cycles; (c) 49 N, 1 cycle.

bright region to be very short after indentation of 1 cycle (Fig. 7a), but its considerable growth was observed after that of 10 cycles (Fig. 7b). Because the striped pattern seen in the chipping area (Fig. 1) is formed in the area of lateral cracking during cyclic indentation, this pattern is confirmed as the striation.

3.3. Type II indentation fatigue

Type II indentation fatigue is characterized by a pronounced enlargement of the indentation-induced damaged zone in early stages of the indentation cycles. This behaviour was observed in materials having a large grain size or plastic deformation characteristic.

3.3.1. Influence of grain size on indentation fatigue

From the results shown in Fig. 4, it was found that the effect of grain size in magnesium oxide and sialon ceramics upon the cyclic indentation damage was insignificant, but in alumina it appeared to be considerable; that is, coarse-grained alumina behaved as Type II, whereas fine-grained alumina behaved as Type I. Because Type I indentation fatigue is caused by lateral crack propagation during cyclic indentation, lateral cracking might not be initiated in coarse-grained alumina. Therefore, in order to check the existence of lateral cracks in both materials, the fractured surface of alumina, produced in the same way as the specimen of silicon nitride, was observed by SEM (Fig. 8). Lateral cracking was clearly seen in fine-grained alumina (Fig. 8a). but not in coarse-grained alumina (Fig. 8b). However, even after the first indentation, debonding of very small surface layer from the matrix was observed in coarse-grained alumina, so that the surface after debonding which was flat might be associated with lateral cracks.

The specimens were also observed by scanning acoustic microscopy (Fig. 9). A lateral crack was clearly seen, irrespective of the grain size, and it was found that a lateral crack was produced by a single indentation, even in coarse-grained alumina (Fig. 9c). After indenting for 10 cycles, lateral cracking was hardly seen outside the damaged zone in coarse-grained alumina (Fig. 9d). Furthermore, it was found that the lateral crack length after the first indentation nearly corresponds to the damaged zone size after the tenth indentation, implying that the lateral crack did not propagate with repeated indentation. Fig. 4b, clarifies the size of the damaged zone to be a maximum at about 10 cycles. Consequently, it was assumed that the extension of the damaged zone was determined by the length of lateral cracks produced by the first indentation; i.e. the volume above the lateral crack face is exclusively damaged.

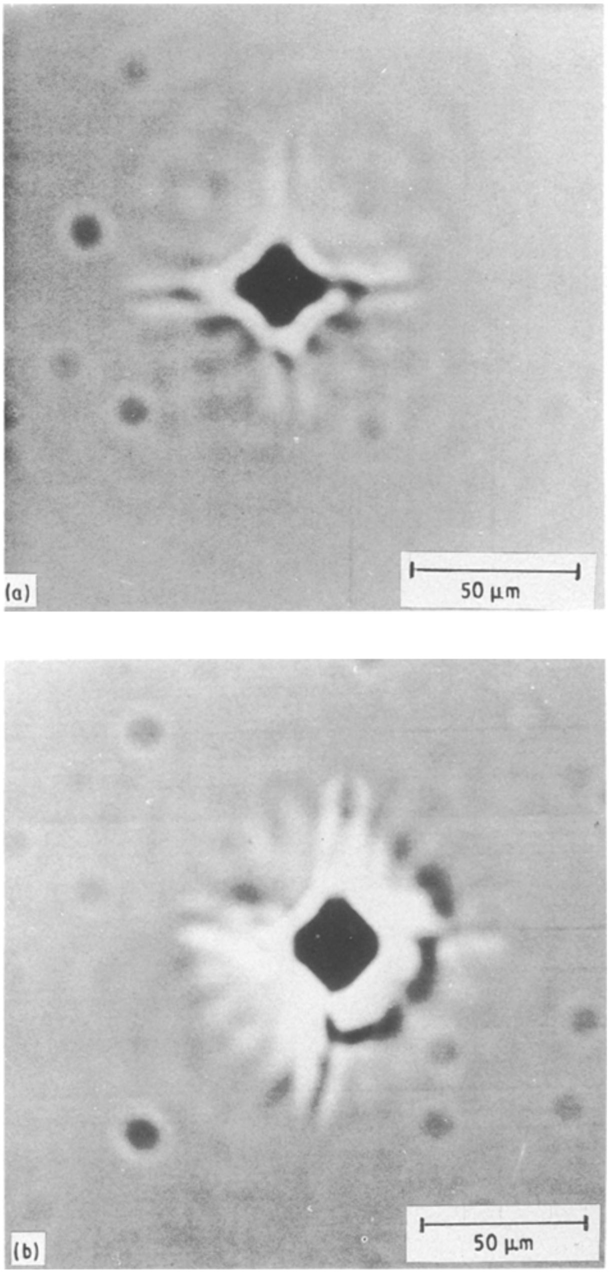

Figure 7 SAM photographs of transgranular fracture type silicon nitride after indentation with 9.8 N load: (a) 1 cycle; (b) 10 cycles.

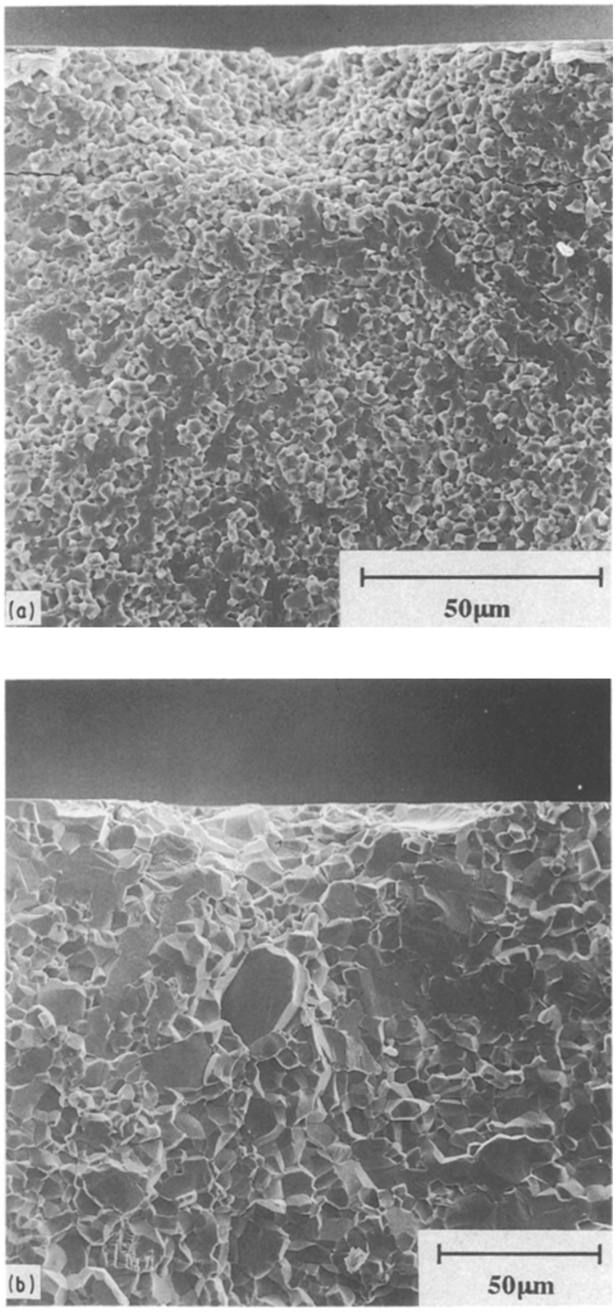

Figure 8 Fractured surfaces produced by flexural strength testing in alumina: (a) fine-grained, (b) coarse-grained.

From the results mentioned above, damage accumulation in coarse-grained alumina is considered to occur as follows (shown schematically in Fig. 10). The first indentation initiates a lateral crack around an indent (Fig. 10a). From SEM observation of the indentation damaged zone in coarse-grained alumina (Fig. 11), it was found that the fracture was characterized mainly by intergranular cracking. Grain-bridging peculiar to coarse-grained alumina [22, 23] presumably occurs even in the lateral crack system. Subsequent indentation allows bridging grains to rotate or slide (Fig. 10b), which produces secondary microcracks at grain boundaries and/or inside the grains. Such grain-bridging prevents the lateral cracks from propagating during subsequent cycles because the residual stress, playing the role of driving force for lateral crack propagation, is relieved by microcracking. Meanwhile, the cracks reach the surface of the material (Fig. 10c) and the volume surrounded by cracks finally chips (Fig. 10d). The nearer the grains are to the indent, the larger is the force which makes the grains rotate or slide. Consequently, the surface layer gradually chips from the edge of the indent. However, these microcracks are induced exclusively within the volume above the lateral crack face, so that the damaged zone was propagated to the extent of the lateral crack length at most, and the extension of the damaged area was saturated at a certain number of cycles (Fig. 4b).

3.3.2. Influence of plasticity on indentation fatigue

As mentioned in Section 3.1, the effect of plasticity is considered to be one of the most significant factors in

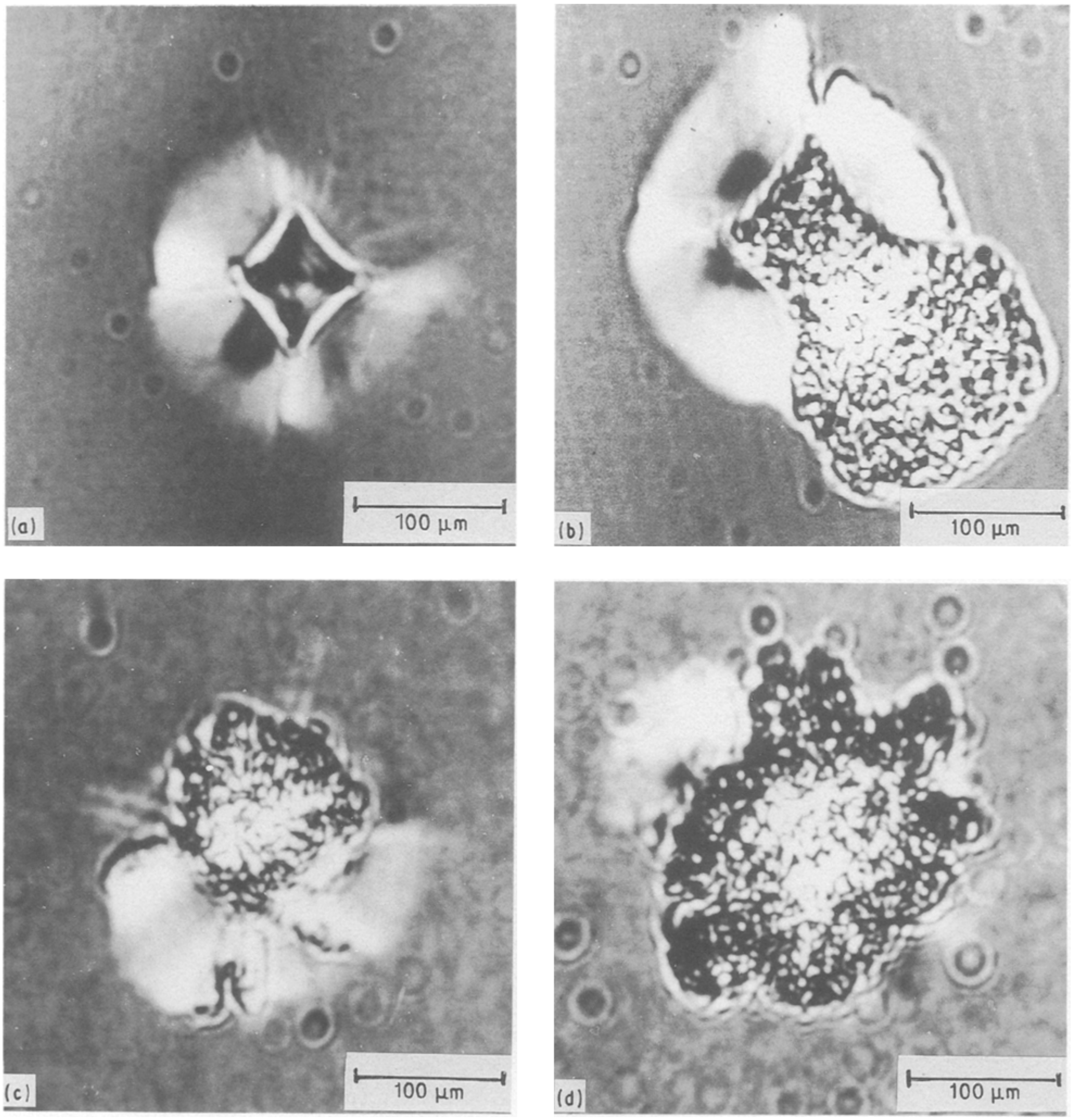

Figure 9 SAM photographs of alumina after single and repeated indentations at 98 N load. The dark region represents the damaged zone, whereas the bright region is the lateral cracks: (a) fine-grained, 1 cycle; (b) fine-grained, 10 cycles; (c) coarse-grained, 1 cycle; (d) coarse-grained, 10 cycles.

Type II indentation fatigue. In magnesium oxide, many microcracks were observed around an indent (Fig. 12). Most of these microcracks are attributed to the reaction of dislocations, which is reasonably explained by a dislocation pile-up model [24]. It also seems that there are some cleavage cracks. Such cracking produces a very large damaged zone around an indent. Fig. 13 shows SAM photographs of magnesium oxide after the indentation of 1 and 10 cycles. It is assumed that in both photographs the dark region around the indents is the damaged zone and the surrounding bright region is the microcracking area. From these photographs, the cyclic indentation behaviour in magnesium oxide with a high plastic characteristic is considered to be as follows. It might be difficult for a lateral crack to propagate continuously during cyclic indentation in this material because the residual stress playing the role of the driving force for lateral cracking is easily relieved by plastic deformation or microcracking, which looks exactly like the behaviour in coarse-grained alumina. Rather, it is supposed that microcracking and the resulting local chipping occur gradually with repeated indentation, followed by saturation. In short, this behaviour is caused by successive initiation and propagation of microcracks.

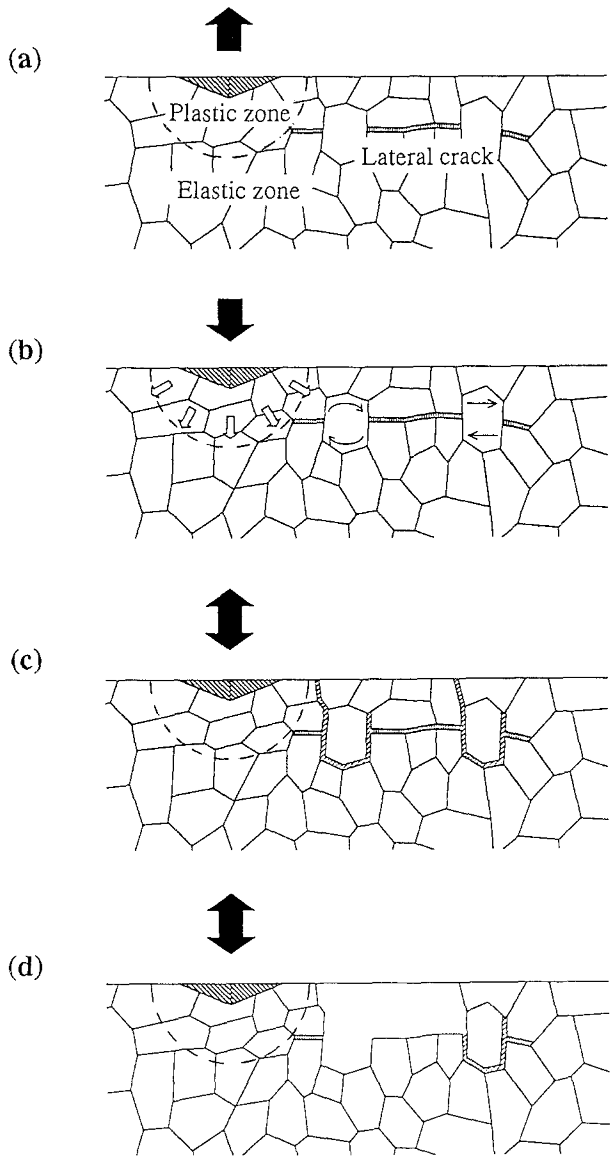

Figure 10 Schematic illustration of the indentation fatigue mechanism in coarse-grained alumina (see text).

3.4. Comparison of conventional Mode I fatigue and indentation fatigue

In materials which are characterized by transgranular fracture, no cyclic loading effect was seen in a conventional Mode I fatigue test [25]. On the other hand, as shown here, and in previous papers [19, 20], for the same materials, a cyclic loading effect was seen in indentation fatigue. Therefore, although the indentation fatigue test is very convenient as a simple method, instead of the conventional fatigue test as proposed by Vaughan *et al.* [17], it is very dangerous to predict conventional fatigue behaviour on the basis of indentation fatigue data alone; it is assumed that there are significant differences in mechanisms between conventional Mode I fatigue and indentation fatigue.

4. Conclusion

It has been found that there are two types of indentation fatigue behaviour (Types I and II). Type I indentation fatigue is characterized by a slight increase of damaged zone with indentation cycles, followed by abrupt chipping of the surface layer. This behaviour is caused by cyclic propagation of lateral cracks. On the other hand, Type II behaviour is characterized by pronounced enlargement of an indentation-induced damaged zone in early stages of the indentation cycles. Type II indentation fatigue was observed in materials

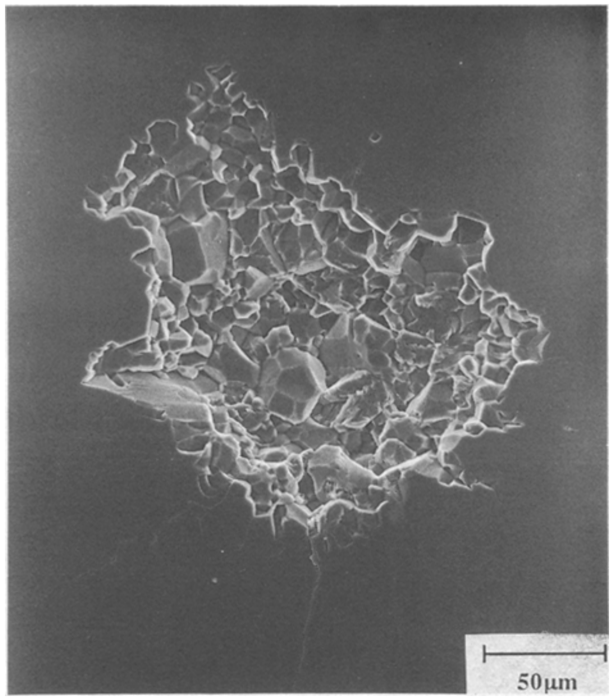

Figure 11 Scanning electron micrograph of coarse-grained alumina after repeated indentation.

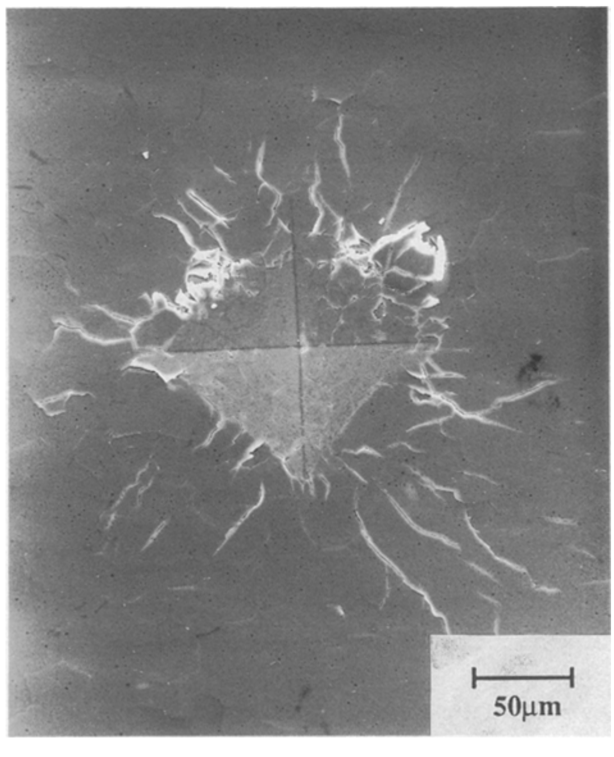

Figure 12 Scanning electron micrograph of magnesium oxide after a single indentation.

having grain-bridging or plastic-deformation characteristics. In other words, these materials can easily produce microcracks during cyclic indentation, which relieves the residual stress playing the role of driving force for lateral crack propagation, so that Type I behaviour is restrained.

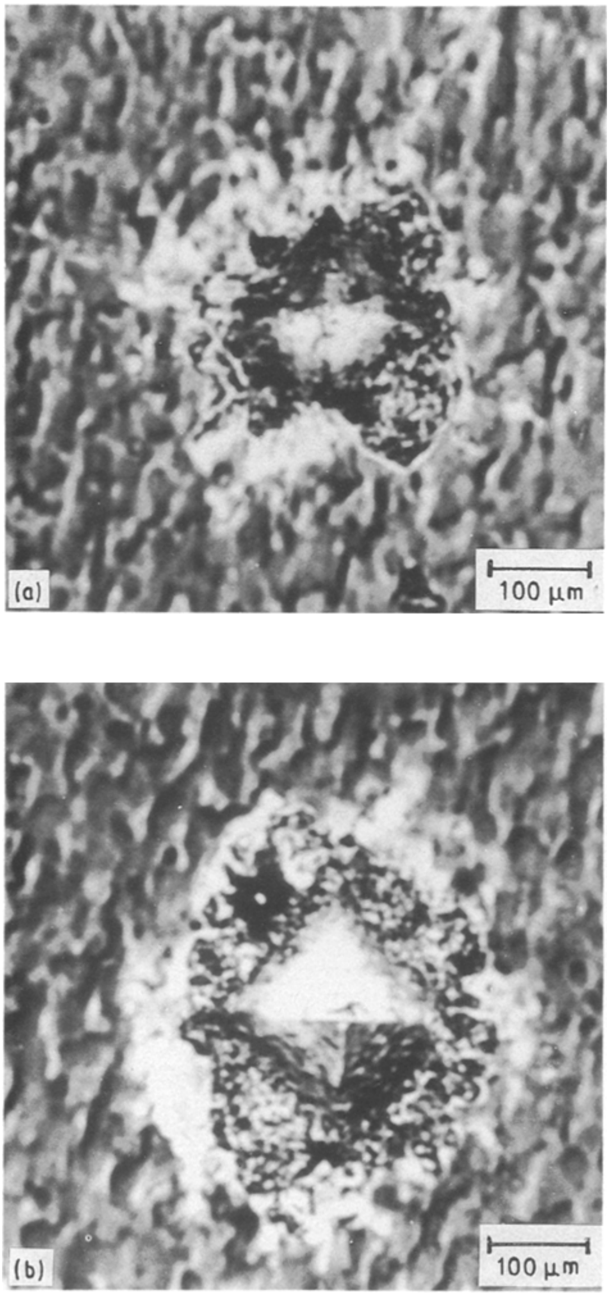

Figure 13 SAM photographs of magnesium oxide after repeated indentation: (a) 1 cycle; (b) 10 cycles.

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