Damage accumulation caused by cyclic indentation in zirconia ceramics

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Indentation fatigue testing is carried out for four kinds of zirconia ceramics; 6Y-FSZ, 3Y-TZP, Mg-PSZ and Ce-TZP; which have different strengthening-toughening mechanisms one another. Each material shows different indentation fatigue behavior. Large sized surface chipping is produced easily in early cycles for non-toughened 6Y-FSZ, while 3Y-TZP toughened by anelasticity has high resistance against chipping. There is a possibility that anelastic characteristic works as resistance against indentation fatigue damage. Stress induced phase transformation also improves fatigue damage, but this effect depends on the extent of transformation and/or occurrence of microcracking. © *1999 Kluwer Academic Publishers*

1. Introduction

Indentation fatigue is the gradual damage accumulation in the surface layer caused by local cyclic compressive loading [1–3]. This phenomenon and the way of investigation in ceramics are firstly reported by Guiu and Vaughan [1]. Although ceramics are, as generally known, resistant to compressive load, cyclic indentation has significant effect on ceramics, which is a serious problem from an industrial and engineering point view as well. For one series of non-transformable ceramics; silicon nitride (Si_3N_4), silicon carbide (SiC), alumina (Al_2O_3) and so on, indentation fatigue was precisely investigated in previous papers [4, 5]. Each material showed different fatigue behavior. Especially important results were as follows. Silicon carbide with non-additives, which ruptures transgranularly and does not indicate Mode I fatigue, showed indentation fatigue and surface chipping. Besides, a trace of striations was seen in this region. It has been clarified that damage accumulation caused by cyclic indentation is different from general fatigue (Mode I fatigue) and depends on structures of materials (difference of grain size or extent of ductility-brittleness) intensively. On the basis of the above results, a model of indentation fatigue mechanism has been proposed [4, 5], but lateral crack propagation during cyclic indentation has not been fully elucidated yet.

Chipping, which is caused by lateral crack advancement, occurs more easily for transgranular fracture type materials rather than intergranular fracture type materials [4, 5]. This is because for the latter type materials crack branching or microcracking is liable to occur, which may work as the factor of stress relieving at the crack tip.

As above-mentioned, indentation fatigue has been fairly clarified for non-transformable ceramic materials, while few papers as for transformable zirconia ceramic materials have been reported. Therefore, it is necessary to comprehend indentation fatigue behavior systematically for a wide range of ceramics with relation to strengthening-toughening mechanisms. In this work, indentation fatigue behavior for four kinds of zirconia ceramics which have different strengthening-toughening factors was investigated in detail. The influence of anelastic behavior [6–9] and stress-induced phase transformation [10, 11] etc. on indentation fatigue was examined and discussed.

2. Experiments

2.1. Materials

Four kinds of zirconia ceramics were used for the present work. Zirconia systems are 6 mol % Y_2O_3 fully stabilized zirconia (6Y-FSZ), 3 mol % Y_2O_3 stabilized tetragonal zirconia polycrystals (3Y-TZP), 9 mol % MgO partially stabilized zirconia (Mg-PSZ) and 12 mol % CeO tetragonal zirconia polycrystals (Ce-TZP). Their structure, grain size, fracture toughness and fracture mode are summarized in Table I.

6Y-FSZ is composed of only cubic phase so that it is very brittle [11]. It is expected that 3Y-TZP has anelastic behavior, which is noticed as significant strengthening-toughening mechanism in our previous works [6, 7]. Mg-PSZ and Ce-TZP are much tougher materials than the formers. It is presumed that such high toughness values are caused by stress-induced phase transformation [10–13]. All samples were provided by commercial ceramics makers in Japan. They were made in rectangular shapes, their section size was 4×8 mm and the length was approximately not less than 12 mm. Before testing, their main surface (to be indented) was polished with 1/4 μm diamond paste so that crack growth is never

TABLE I Structure, fracture toughness, grain size and fracture mode for the materials tested

Material	Structure[a]	Grain size (μm)	Fracture toughness (MPa · $m^{1/2}$)	Main fracture type
6Y-FSZ	c	1.0–1.2	1.7	transgranular
3Y-TZP	t	0.5–0.7	3.2	intergranular
Mg-PSZ	c + t + m	0.2–0.4	7.4	intergranular
Ce-TZP	t	1.0	9.0	intergranular

[a]c: cubic, t: tetragonal, m: monoclinic

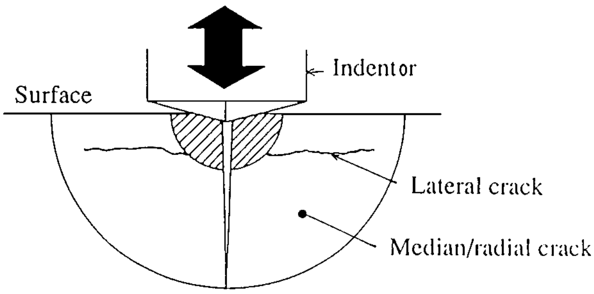

Figure 1 Schematically illustration of the testing procedure for indentation fatigue.

interrupted by surface scratches and we could measure crack length precisely.

2.2. Test method

The indentation fatigue testing was carried out by pressing the diamond indentor onto the same point of the sample main surface repeatedly in air, as shown schematically in Fig. 1. The testing machine was a digital type vickers hardness tester (DVK-2S, Matsuzawa seiki co., LTD., Tokyo, Japan). The applied indentation load was in the range from 9.8 to 196 N, the loading speed was 70 μm/s and the load holding time was 10 s. The median/radial crack growth behavior during cyclic loading was measured by optical microscope fixed in the testing machine. This enabled us to measure crack length without taking the sample out of the machine or shifting indentation point accidentally. Besides, lateral crack length, number of cycles to chipping and the chipping or fatigue damage zone size were observed and measured. (The region different from surrounding area in morphology was defined as fatigue damage zone, which was caused by phase transformation and/or microcracks and so on.) The indentation was applied repeatedly not more than 30 times during testing. If chipping of the surface layer occurred, just then, testing was finished and the number of cycles was recorded. The form and condition of the indent was observed in detail by not only monitoring optical microscope but also scanning electron microscope (SEM). As for 3Y-TZP, in order to confirm whether the accumulative damage (crack advancement during cyclic indentation) was caused by cyclic fatigue (repetition of loading) or static fatigue, indentation fatigue testing was carried out by changing the load holding time from 10 to 150 s corresponding to 15 times repetition (i.e., $10 \times 15 = 150$ s). The median/radial crack growth behavior under this condition was compared with the results of above-mentioned original testing.

3. Results and discussion

3.1. The morphology of damaged zone

Fig. 2 is a scanning electron micrographs, which show one example of damaged zone of each material tested. They had different features; 6Y-FSZ and 3Y-TZP produced straight median/radial cracks from each corner of the indent, while Mg-PSZ did not produce such straight median/radial cracks from the corner but many radial cracks (microcracks) around the indent. Ce-TZP brought about so short cracks under the present condition of indentation loading.

6Y-FSZ has frequently caused extremely large sized chipping as shown Fig. 2a, which also represents brittleness of the material. On the other hand, 3Y-TZP hardly caused chipping under all loading conditions. Fig. 2b shows an exceptional result. This will be also detailed in the next section. Slight uplifting was found around the indent on both Mg-PSZ and Ce-TZP. This uplifting was thought to be caused by phase transformation, which prevents the median/radial cracks from extending [12].

3.2. The advancement of median/radial cracks and lateral cracks

Fig. 3 shows the extending behavior of the median/radial cracks produced by cyclic indentation, on four kinds of zirconia ceramics tested; 6Y-FSZ, 3Y-TZP, Mg-PSZ and Ce-TZP. Although it is supposed that such extending behavior is essentially of Mode I fatigue, these materials showed considerably different behavior one another.

The arrows in Fig. 3 represent that chipping occurred just after the cycle, which clarifies that 6Y-FSZ tends to cause chipping more easily in early cycles than other materials observed in the present work. The easiness and extent of chipping should be related with brittleness, in other words, the absence of toughening factors enhances lateral crack propagation. There was one case that, under the minimum loading of 9.8 N, chipping did not occur in spite of 30 times cyclic indentation. Besides, by a supplementary experiment, it was confirmed that chipping never occurred under the condition of lower indentation load 4.9N-30 times. Therefore, an indentation fatigue limit of 6Y-FSZ may exist near the load 9.8 N (See Section 3.3). From the observation of large sized chipping in Fig. 2a it is assumed that chipping can occur more easily in 6Y-FSZ, of transgranular fracture type, as well as SiC without additives [4, 5]. In effect, crack extension of 6Y-FSZ was terminated due to this chipping in early cycles. To the contrary, 3Y-TZP showed crack extension over the whole indentation cycles.

In 3Y-TZP there are two interesting features; (i) chipping seldom occurred through whole cyclic indentation and (ii) crack hardly nucleated under the condition of the minimum load 9.8 N tested. The latter is, especially, an astonishing result, in respect that in Mg-PSZ crack nucleated even under the loading condition of 9.8 N though the produced crack length is shorter than that of 3Y-TZP as a whole. We think that one of the causes may be the anelastic behavior of Y-TZP [6–9], as discussed in the next section.

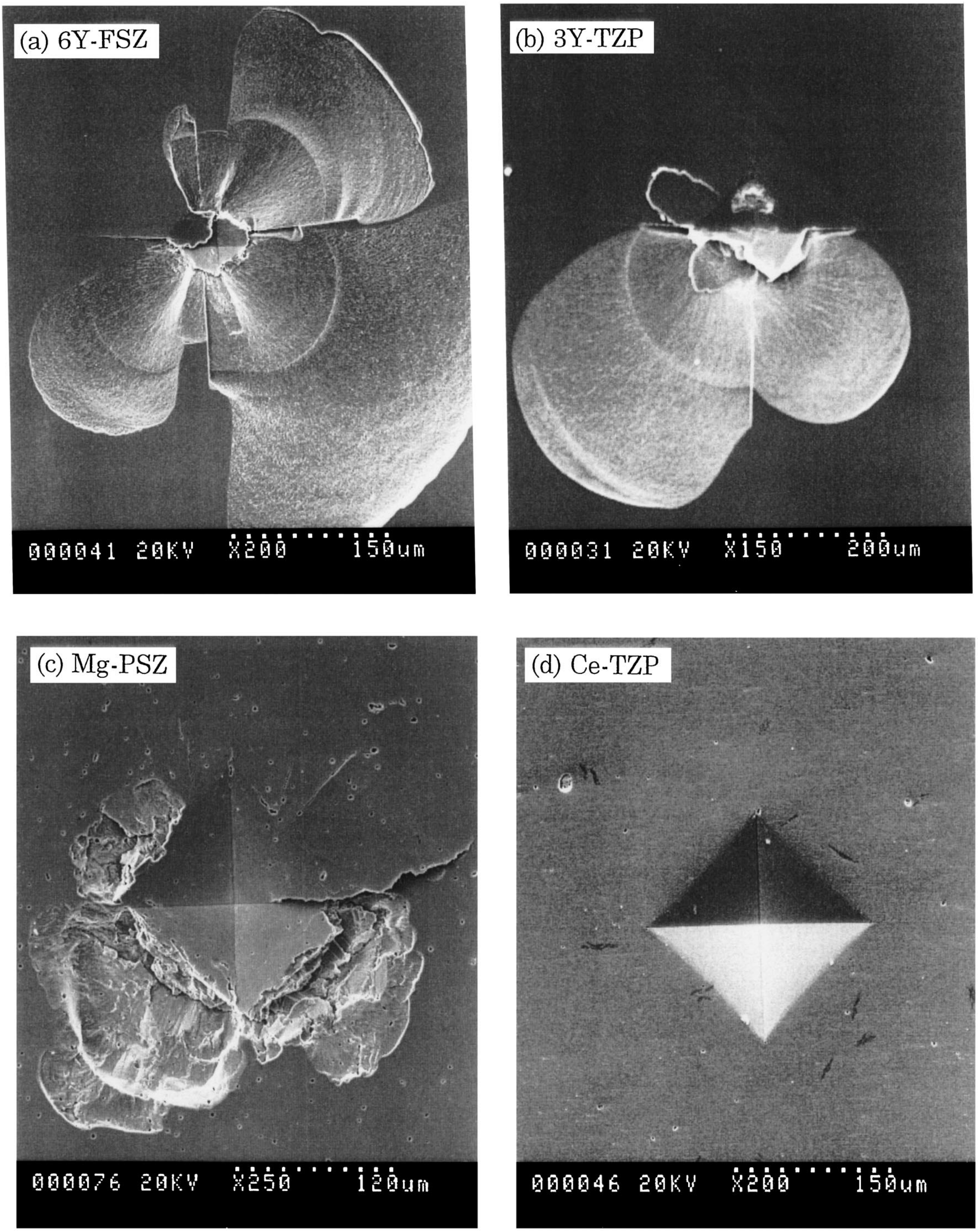

Figure 2 Scanning electron micrographs of indent of each material tested: (a) 6Y-FSZ in 49N-2times; (b) 3Y-TZP in 196N-6times; (c) Mg-PSZ in 196N-2times; and (d) Ce-TZP in 196N-30times.

Mg-PSZ did not produce large crack extension as much as 3Y-TZP. Mg-PSZ tends to cause chipping more easily than 3Y-TZP in spite of possessing a strong transformation toughening mechanism. According to Table I, toughness of Mg-PSZ is much higher than that of 3Y-TZP. Furthermore, it is considered that Ce-TZP having strong stress-induced transformability as well as Mg-PSZ should show strong resistance against crack nucleation-propagation and occurrence of chipping. Under the loading condition of 9.8 N cracks did not occur, and under the next smallest loading of 49 N short cracks was found once in a while, which indicates that crack occurrence limit exists between both load levels. Chipping did not occur at all under the

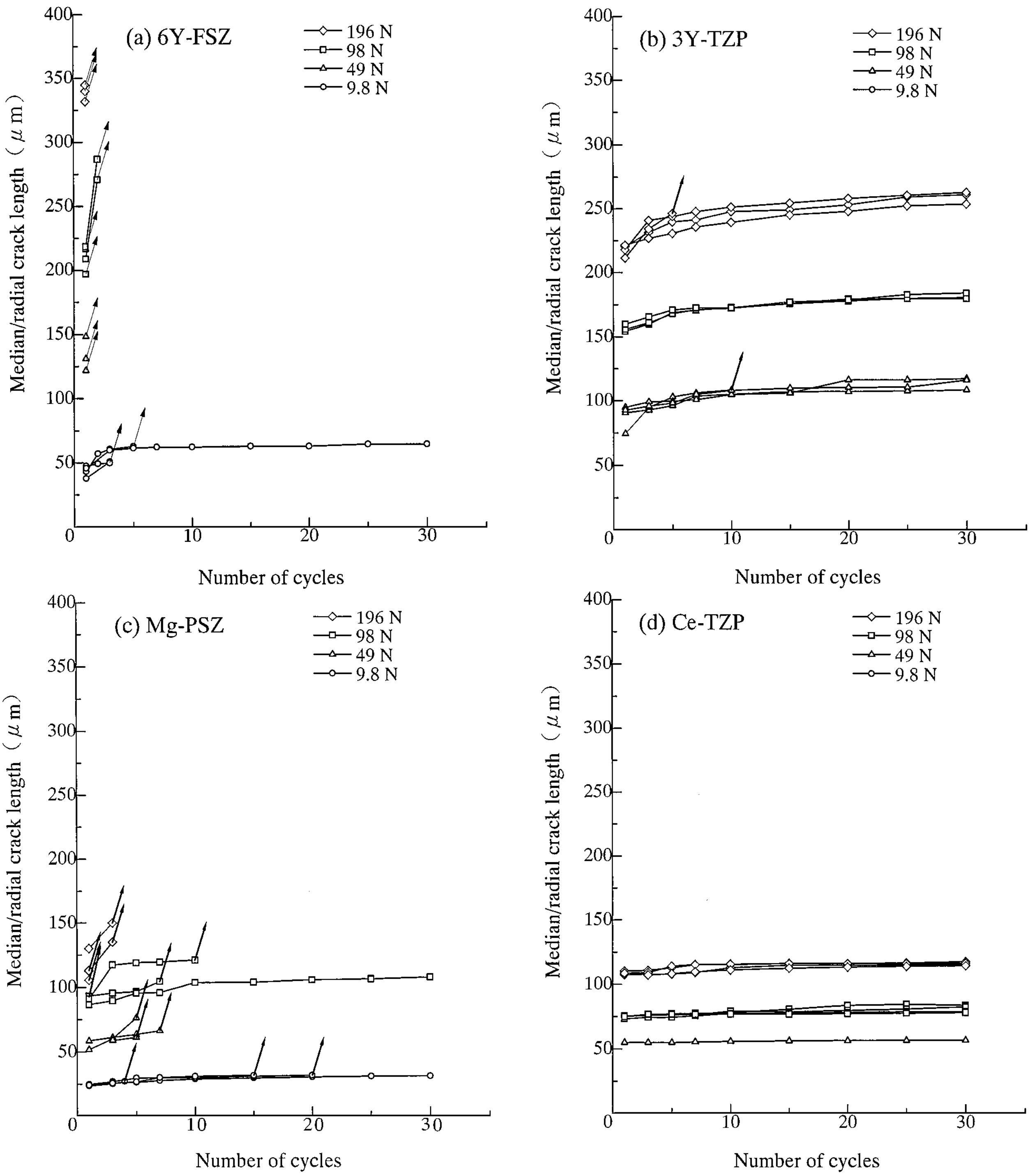

Figure 3 The advancement of median/radial crack under cyclic indentation. (a) 6Y-FSZ, (b) 3Y-TZP, (c) Mg-PSZ and (d) Ce-TZP.

present condition of indentation loading. Such different behavior between two materials having similar strong transformation-toughening characteristic might come from the difference of critical stress [14] for transformation. (or something else.?)

3Y-TZP produced larger extent of median/radial crack advancement. Fig. 4 shows the effect of load holding time on indentation fatigue in 3Y-TZP, which is the comparison between the result of Fig. 3b and that obtained by the test of long load holding time corresponding to 15 time-indentations. It looks that both curves are very different, but crack advancement caused by the 2nd and the 3rd indentation cycles under the condition of 150 s holding time is roughly equal to that caused by the 2nd and the 3rd indentation cycles under the condition of 10 s holding time. This clarifies that median/radial crack advancement in 3Y-TZP by cyclic indentation is determined almost by load repetition and not by load holding time.

Since 6Y-FSZ is translucent, it is possible to observe the process of lateral crack advancement, and the resultant chipping only in this material by optical microscope. Fig. 5 shows lateral crack advancement in this material under cyclic indentation of 9.8 N. After relatively large advancement in early cycles, lateral cracks propagate with repeating growth and arrest, followed by chipping finally. The striations observed in SiC [4, 5] were, unfortunately, not seen in the chipping region.

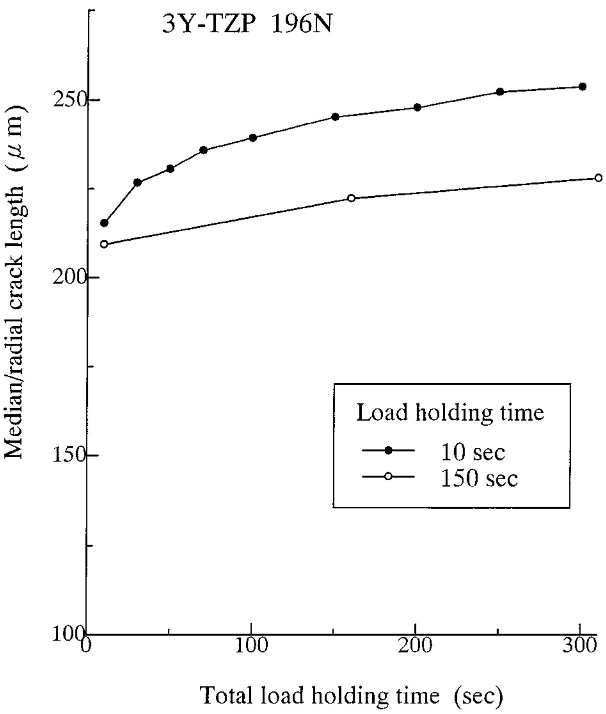

Figure 4 The effect of load holding time on the advancement of median/radial crack in 3Y-TZP. (Indentation load is 196 N).

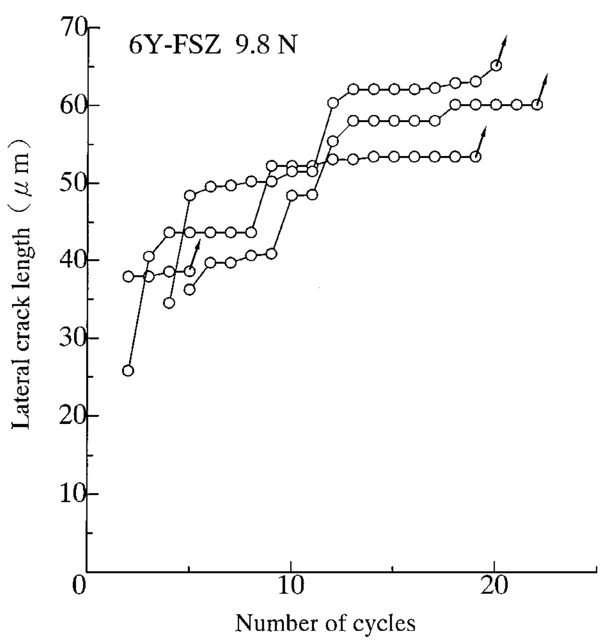

Figure 5 The advancement of lateral crack under cyclic indentation in 6Y- FSZ. (Indentation load is 9.8 N).

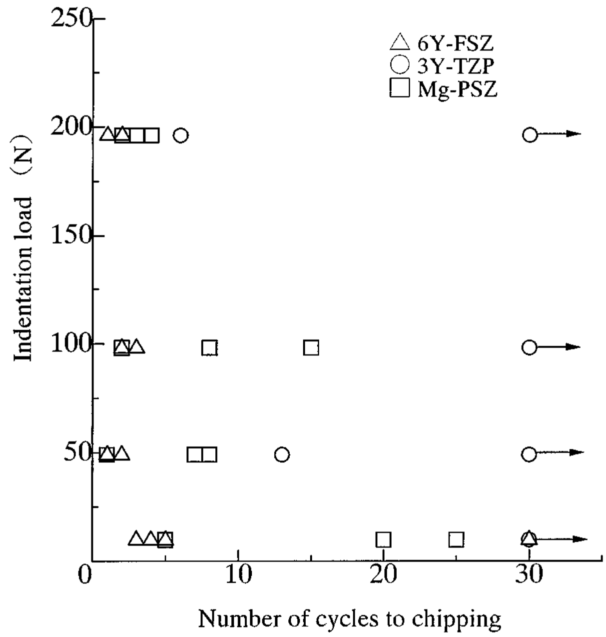

Figure 6 Relation between indentation load and number of cycles to chipping.

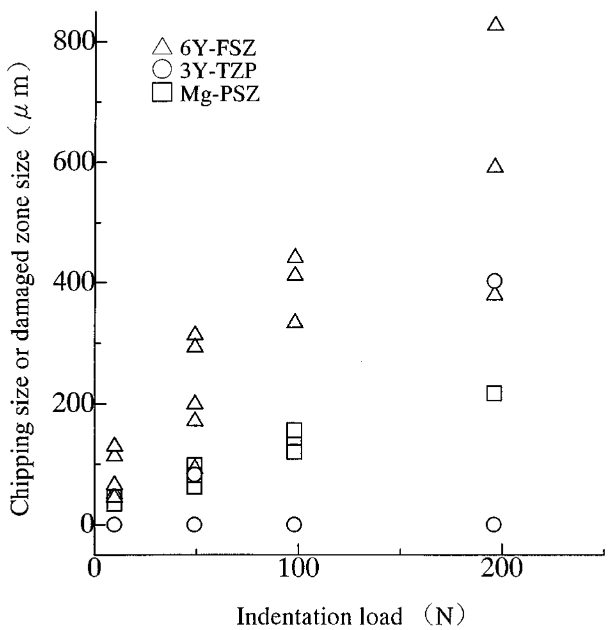

Figure 7 Relation between chipping size or damaged zone size and indentation load.

Grain size of the investigated SiC is smaller than that of present 6Y-FSZ. Large grain results in low toughness and ease of chipping, which may fail to show vivid striations.

3.3. The relation between indentation fatigue damage and strengthening-toughening mechanism

Fig. 6 shows the relation between indentation load and number of cycles to chipping, which corresponds to general fatigue lifetime curve. Fig. 7 shows the relation between chipping size or damaged zone size and indentation load. It becomes clear that 6Y-FSZ produces chipping more easily than other materials tested and the size is also fairly large although data are very scattered. Next to 6Y-FSZ, Mg-PSZ produces chipping more easily. However, this chipping size was restricted, because it is thought that many cracks nucleate and propagate around the indent. Last, 3Y-TZP hardly produced chipping and had long lifetime (arrows shows non-chipping through 30 time-indentations). However, when chipping occurred rarely, these sizes were considerably large (Fig. 7), which means that lateral cracks might have always propagated beneath the sample surface. Although data of Ce-TZP are not shown, as beforementioned, chipping did not occur. It is clear that

Ce-TZP has the highest resistance of chipping of all materials tested.

Strengthening-toughening mechanism would control a pattern of indentation fatigue. In 6Y-FSZ with no strengthening-toughening factor, nothing restrains lateral crack growth and the subsequent chipping. Such chipping phenomenon of 6Y-FSZ is the same as that observed in SiC or Si_3N_4 of transgranular cleavage fracture type [4, 5]. It is reported [5, 15] that transgranular fracture type materials tend to produce much larger damaged zone or chipping size than intergranular ones, which applies to the present results. The higher the indentation load, the more scattered the chipping size data was. This means that lateral crack advancement is influenced by stress heterogeneity due to material nature such as grain size, residual stress, etc.

Note that in 3Y-TZP median/radial cracking was hard to occur under the minimum indentation load 9.8 N (Section 3.2). The damage morphology of this material is similar to that observed in intergranular fracture type SiC or Si_3N_4 [4, 5]. It is thought that some stress relaxation in the crack tip region must restrain catastrophic propagation of median/radial and lateral cracks. As to 3Y-TZP, there is a possibility that this restraining factor is its anelastic [6–9] strengthening-toughening mechanism. Ferroelastic domain switching [16–19] is also considered as alternative possible mechanism. However, considering the reports that this mechanism does not appear to contribute to the strengthening-toughening, i.e., crack initiation resistance of Y-TZP [20–22], this mechanism is not applicable to this case. We need to inquire further into the matter. The detailed analysis of the anelasticity effect is now under study.

In spite of remarkable stress-induced phase transformation, Mg-PSZ tends to produce chipping easily. It is found that chipping can occur easily even in the materials with remarkable transformation characteristic and high K_{IC} value. As to Mg-PSZ, occurrence of microcracking may lead to chipping in early cycles, while Ce-TZP having higher K_{IC} than Mg-PSZ showed the best resistance against cyclic indentation fatigue under whole indentation load investigated. Remarkable transformation must prevent crack nucleation itself strongly. Whether microcracks are nucleated or not, and the extent of microcracking must control chipping phenomenon for both materials. Therefore, it is reasonable that behavior of Mg-PSZ under the extremely low load is similar to that of Ce-TZP under the extremely high load.

4. Summary

For four kinds of zirconia ceramics (6Y-FSZ, 3Y-TZP, Mg-PSZ and Ce-TZP) that have different strengthening-toughening mechanisms, each indentation fatigue behavior was investigated. The results have clarified that damaged morphology depends on each strengthening-toughening mechanism strongly. Non-toughened 6Y-FSZ produced remarkable chipping as well as non-zirconia (non-transformable) ceramics of transgranular fracture type, while in 3Y-TZP indicating anelastic behavior, chipping was restrained remarkably. Besides, it was found that indentation damaged morphology was influenced by the extents of phase transformation and/or microcracking. In Ce-TZP crack nucleation and propagation was restrained intensively due to remarkable phase transformation. Mg-PSZ also showed similar behavior, but tended to produce chipping because of easiness of microcracking.

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