# EVALUATION OF RESIDUAL STRESS INDUCED PHASE TRANSFORMATION FOR Y-TZP AND WC/Y-TZP

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## Abstract

Vickers impression was applied on the polished surface of Y-TZP and WC/Y-TZP specimens with an impression load of 50 kgf. After the surface of the specimen was again ground out and polished to remove the indentation follow, it was aged at about 150°C for various times in air. The phase transformation in the locally stressed region beneath the indentation impression was investigated by X-ray analysis, SEM observation and surface profile measurement.

## Introduction

Phase transformation toughening mechanism[1,2] of zirconia ceramics (partially stabilized zirconia and tetragonal zirconia polycrystals) leads to superior mechanical characteristics, such as high strength and high toughness. According with its superior properties, zirconia is expected to be used extensively for blades, tools and instruments, sportswear, etc. Although some commodities have already been commercially produced, one of the weak point for the zirconia materials remains the phase transformation of the metastable tetragonal phase which gradually occurs to monoclinic phase at a relatively low temperature range (about 200-300°C). It has been pointed out that the tetragonal phase remains after cooling and its transformation compromises the strengthening mechanism (so-called degradation at low temperatures)[3-6]. The degradation at low temperatures can be improved depending upon the reforming microstructure of sintered body, the amount of a selected stabilizer[5,6,7-9], and on the addition of other ceramic secondary phases[10,11]. Almost all the evaluations of the degradation phenomenon were, however, done by ignoring the influence of stressing conditions. Being the toughening mechanism of zirconia due to a stress-induced transformation, it is necessary to consider how the effect of stressing conditions may influence the degradation. According with the above point of view, one of the present authors observed the degradation of a Y-PSZ during bending test under constant loading, and it was clear that the degradation was easily progressing from the PSZ surface where the tensile stress was applied[12]. However, in the engineering applications (i.e. blades, tools, etc.), it is most likely to be subjected to local and dynamic (instantaneous) stimulation rather than to be exposed to static stressing conditions. Therefore, the gradual degradation during operation becomes a serious problem with time passing. In this work, a local residual stress was induced to generate transformation at the surface of zirconia ceramics, and the degradation behavior was investigated by micro-focused X-ray analysis, SEM observation and quantitative analysis of the specimen surface morphology.

## Experimental

The materials used in this study were a 2.7 mol% yttria-stabilized and hot-isostatically pressed tetragonal zirconia polycrystal (Y-TZP), and its composite with tungsten carbide (WC/Y-TZP). Photographs of the fracture surfaces for both specimens are shown in Fig. 1. Lapped surfaces of these sintered bodies were obtained by grinding with some kinds of SiC powder and polishing with 1.0 $\mu$m diamond paste. A Vickers diamond indentation was applied on the polished surface with a load of 50 kgf for 15 sec. It can be thought that there is a residual compressive stress around the indentation impression. The indented surface was again ground out by a grinding wheel (#400) until the depth of the impression disappeared and

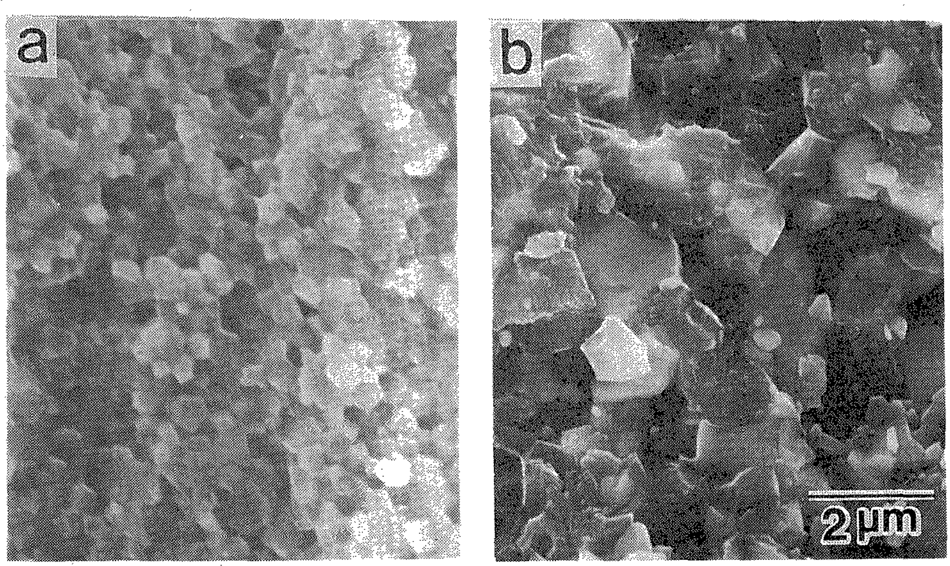



Fig. 1 SEM micrographs of (a)Y-TZP and (b)WC/Y-TZP.

polished again with 1.0 μm diamond paste to obtain a flat surfa because the surface containing the indentation follow is not suita for a quantitative evaluation of the degradation.

Because it has been reported that the degradation of Y-TZP much accelerated under a condition keeping at temperatures rangi from 200 to 250°C[7,13], the indented and, then, polished specime were aged in air bath at a temperature about 150°C to examine 1 degradation behavior under a moderate condition. The degradat behavior which can be observed beneath the indentation was inves gated by micro-focused X-ray analysis, SEM observation and surfa profile measurement by using a roughness tester (Sakata Ind. C DSF-300).

## Results and Discussion

Shape of the indentation-induced crack

When a Vickers diamond indents on the surface of brittle ma rials, cracks are usually starting from the four-edges of the inden tion. Two types of crack formation mechanism have been suggest for the Vickers indentation induced cracks. One is a harf-pen shaped crack formed beneath the indentation. This crack is cal median crack and it spreads radially to the surface of the specin while residual compressive stresses remaining beneath the indentat after unloading (median/radial crack)[14]. Another one is a shall horizontal crack formed at the lateral part of the indentation. It called Palmqvist crack and is obtained during a step way of med crack being formed[15]. The latter crack can be observed in the ca of indentation of toughened materials under relatively low indentat load. An detailed consideration of the mechanism of responsible the indentation induced crack is an important step in driving forwa the fracture mechanics analysis of ceramics subjected to t elastic/plastic field formed around the indentation[16]. As a matter fact, it has also been known as a concern for the choice of equatic for fracture toughness calculation (IF method)[17,18].

It can be decided that the indentation-induced crack is median/radial or a Palmqvist-type upon the fracture surface observ tion[19,20] or by analytical techniques in which the dependence crack length on the indentation load is established[21,22]. The obse vation of fracture surfaces by using SEM or optical microscope w however, very difficult in the case of sintered zirconia specimens.

A SEM photograph of a specimen surface obtained by fracturi from a Vickers indentation induced crack in bending is shown in F

2. It is very difficult to distinguish the configuration of the indentation crack as mentioned above, and also, the original configuration of the indentation crack may be changed during the fracture process. Figure 3 shows the traces of the internal cracks in the specimen whose surface was removed for about 30 $\mu$m from the polished surface prior the indentation. It is observed from this SEM microphoto-

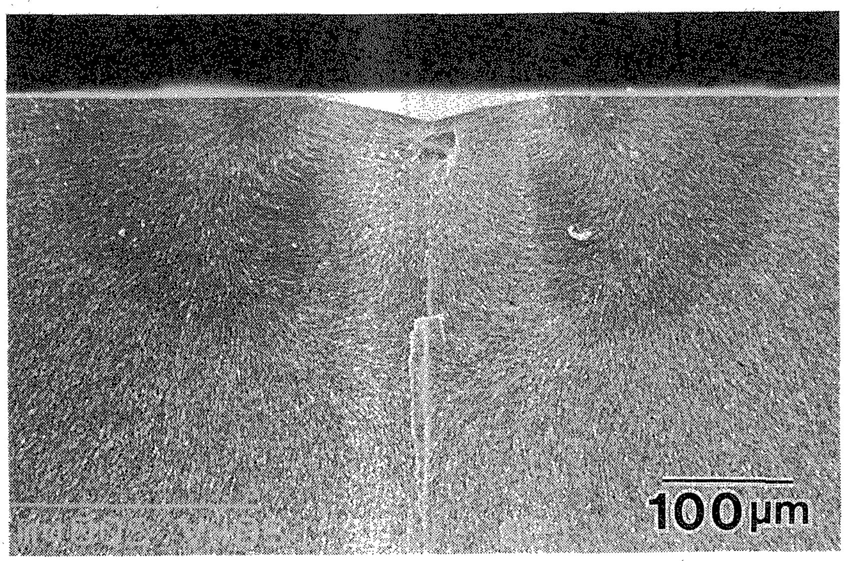



Fig. 2 Fracture surface of Y-TZP showing the crack induced by Vickers indentation.

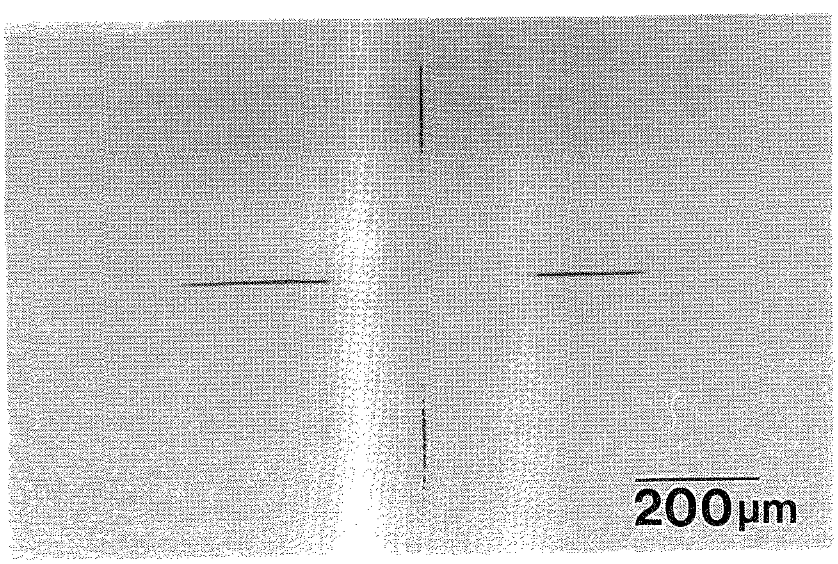



Fig. 3 Optical micrograph of a ground and polished surface after Vickers indentation with an impression load of 50 kgf for Y-TZP.

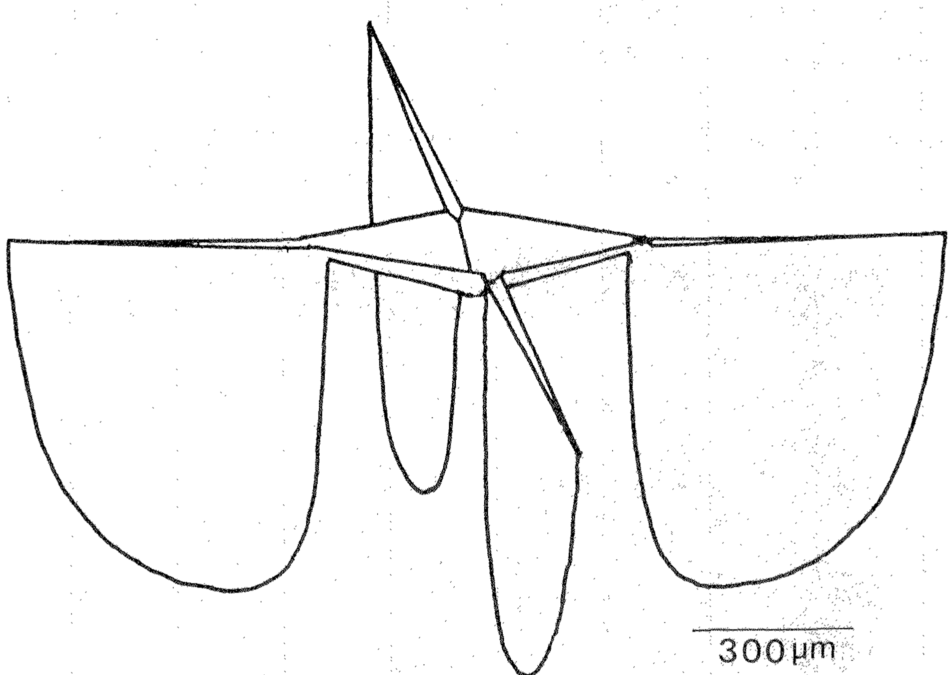



Fig. 4 Schematic drawing of Vickers indentation induced crack in Y-TZP.

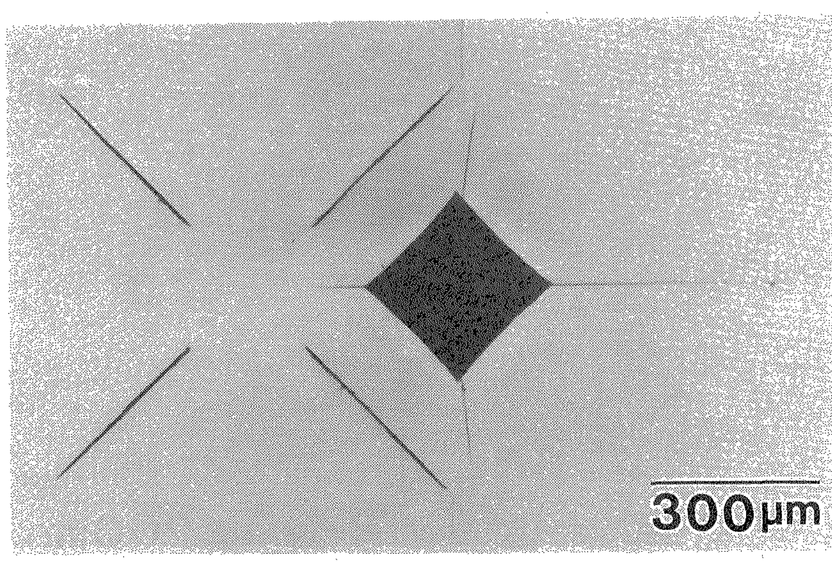



Fig. 5 Crack propagation behavior at around the ground/polished region beneath the indentation for Y-TZP.

graph, that no crack formation is found beneath the indentation. Repeating this operation from the surface to the interior to get a successive complete mapping, the shape of the indentation-induced crack pattern of TZP was reconstructed as schematically shown in Fig. 4. A considerable finding was that the crack apparently seen as a Palmqvist-type crack, is extending rather deeply as ordinary lateral cracks are not. The reason can be found in the stress-induced phase

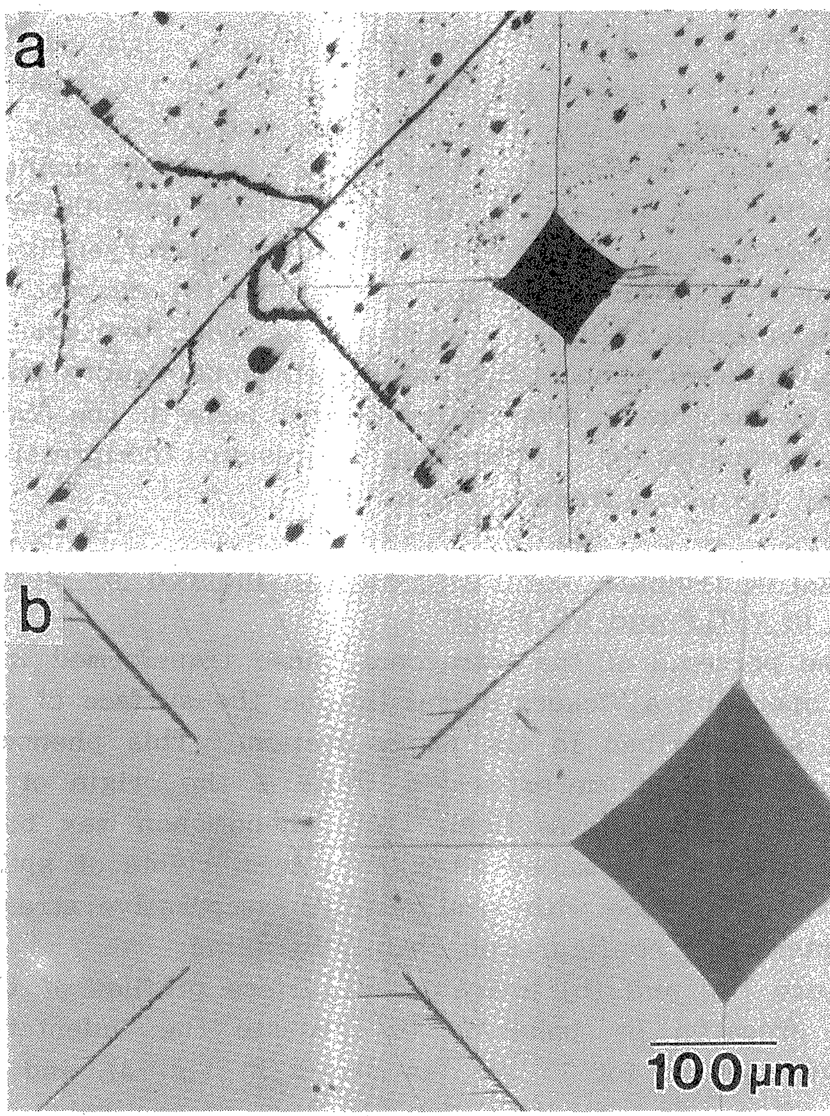


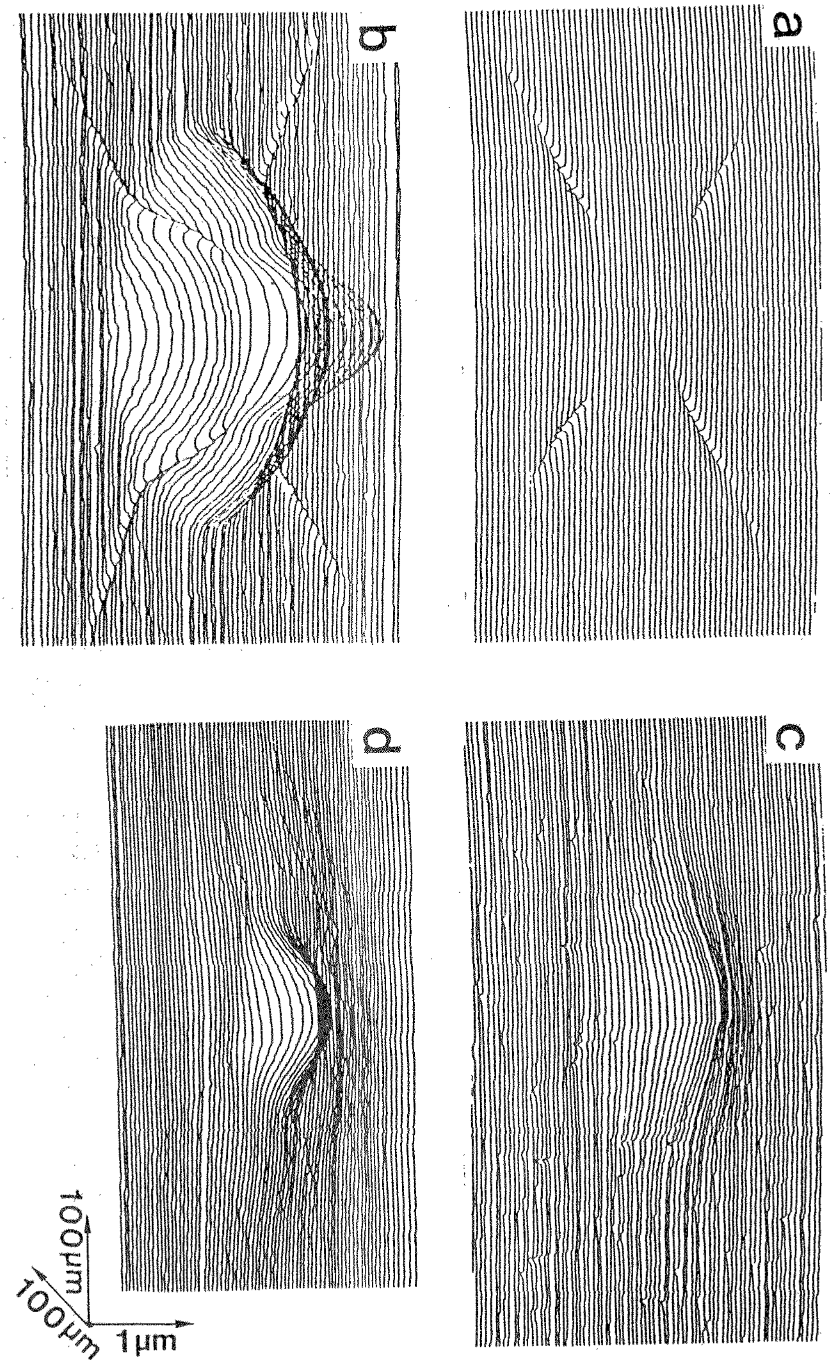

Fig. 6 The same testing results shown in Fig. 5 for alumina (a) and annealed Y-TZP (b). The indented and then polished Y-TZP specimen was heat treated at 1400°C for 30 min before testing.
(a) alumina polycrystal, (b) annealed Y-TZP

transformation which is characteristic of zirconia. The metastab tetragonal phase transforms to monoclinic by complicating the stres field around the region of the impression. A phenomenon accompani by a volume expansion of about 4.6 %. The existence of a residu compressive stress can be confirmed as follows. An indented and th polished surface was prepared as previously shown in Fig. 3, and new Vickers indentation was introduced from the side portion to init ate cracks as shown in Fig. 5. The cracks can not pass through t region beneath the indentation according to the residual compressi stresses. The contents of monoclinic phase determined by X-r micro-analysis as proposed by Garvie[23] were 8.0 ± 2.0 and 0.2 ± ( vol% for locations beneath the indentation and sufficiently far aw from indentation, respectively. This shows that stress-induced pha transformation took place in Y-TZP by applying an indentation loa Results after the same testing procedure for polycrystalline alumi and annealed Y-TZP are shown in Fig. 6(a) and (b), respectively. T Y-TZP was annealed at 1400°C for 30 min in order to retransform t indentation-induced monoclinic phase to tetragonal before testing. these specimens, crack propagation occurs beneath the Vickers i pression demonstrating the absence of residual compressive stre induced by the phase transformation.

<u>Observation of residual compressive stress induced by phase tran formation in Y-TZP and WC/Y-TZP</u>

Some percents of the tetragonal phase transformed into mon clinic by operating a Vickers indentation on the surface of the Y-T specimen, as described in the above section. This phenomenons accompanied by a volume expansion which is the origin of the lo residual compressive stress field. This phenomenon may be used model the case of a damaged edge of cutters, rivets of spike sho etc. Specimen surface with local residual compressive stress show also an interesting configuration change by aging.

Figure 7(a) and (b) indicate the surface profiles of the Y-T specimen prepared by polishing to eliminate the indentation aft loading with 50 kgf for 15 sec (as shown in Fig. 3), and the sa specimen after aging at 150°C for 180 hrs, respectively. In the figures, the protrusion is emphasized by introducing a magnificati factor in the longitudinal scale. The indented/polished surface w protruding during aging in spite of the relatively moderate conditic The percentage of monoclinic content was 8 % for (a), and increas to 28 % for (b) by aging. The phase transformation effect wa




ig. 7 Surface profiles before and after aging of specimen containing residual stress by indentation.

(a) ground and polished surface after indentation for Y-TZP

(b) specimen (a) after aging at 150°C for 180 hrs

(c) specimen (a) after room temperature exposure for 240 hrs

(d) WC/Y-TZP specimen after aging at 150°C for 180 hrs however, limited only in the specimen surface because the monoclinic content decreased by removing from the specimen surface only few μm. The polished specimen (figure (a)) as found after 240 hrs at room temperature is shown in Fig. 7(c). Though a little protrusion was recognized, the monoclinic content on the protrusion was almost not changed. This result shows that in Y-TZP which contains residual compressive stress a very fast degradation can take place even under moderate temperature condition (150°C). The degradation behavior for the WC/Y-TZP, tested under conditions same as in Fig. 7(b), is shown in Fig. 7(d). It has been reported that the degradation behavior of Y-TZP can be improved by adding tungsten carbide [24]. The degradation test carried out in composites under local residual stress could also provide confirm that the combining effect are effectively operating.

Figure 8 shows the influence of aging time on the protrusion height. These plots were obtained by repeating the surface profile measurement as shown in Fig. 7 for the purpose of evaluating the stress-induced degradation behavior in Y-TZP and WC/Y-TZP specimens. Monoclinic contents on the protrusion were obtained for Y-TZP. The contents for WC/Y-TZP, on the other hand, could not be obtained because the diffraction line for (001) plane of tungsten carbide lies

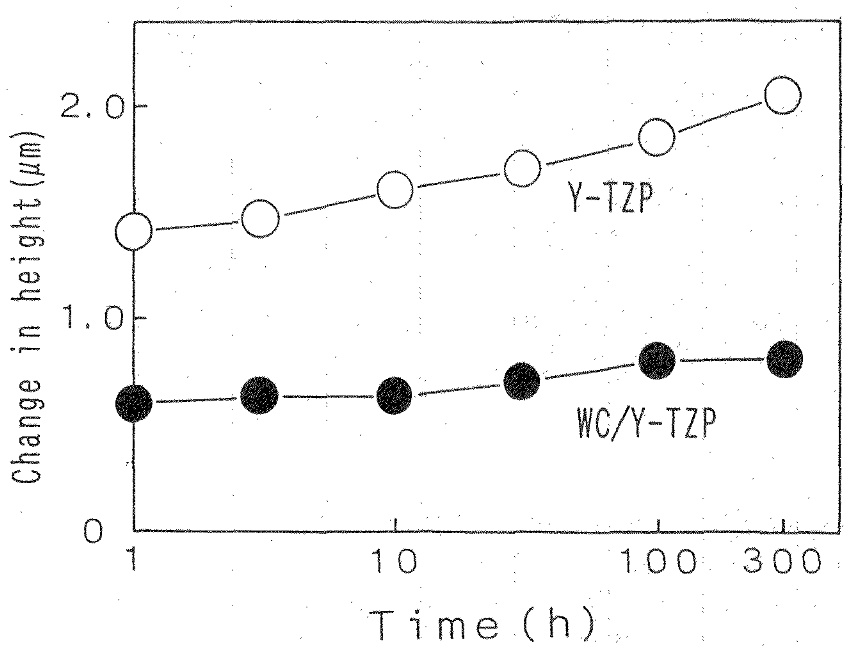



Fig. 8 Relations between protrusion height and aging time at 150°C for Y-TZP and WC/Y-TZP.

overlapping with that for (111) plane of the monoclinic zirconia. The percentage of monoclinic content in Y-TZP gradually increased with increasing aging time from 2 to 12 % at 150°C for 300 hrs even on a polished surface without residual stress. It may be due to the large grain size (0.5 $\mu$m) of the polycrystal. An accelerated protrusion at the locally stressed region beneath the indentation impression took place at the beginning of the aging (within 1 hr) also accompanied by a remarkable phase transformation occurred is also remarkable (from 8 to 40 % at 150°C for 300 hrs). Although the degradation was not found in the WC/Y-TZP aged at 200°C for 100 hrs in water[24], the growth of protrusion was observed also in this material.

The cause of degradation taking place in the locally stressed region is not easy to analyze because the stressing condition beneath the Vickers impression is not a simple hydrostatic compression but couples stresses of mode I to III loading. It is presumed that an extremely complicated micro-fracture and plastic deformation occur.

Monoclinic phase, once is produced, may act as nucleus for a continuous monoclinic formation[25,26], and also corrosive water[27,28] may be transport through the micro-cracks in the protrusion (many cracks may be generated because the volume of the protrusion is larger than that calculated from the degree of transformed phase). Further studies under different experimental conditions (atmosphere, temperature, time, etc.) should be performed.

**Summary**

The degradation characteristics at low temperatures for zirconia ceramics having local surface damages were investigated, and the preparation of a model specimen for a simple degradation tests were carried out in this paper. Specimens containing local residual stresses were prepared by firstly applying a Vickers indention to produce an impression, then by grinding and polishing correctly the specimen surface only up to the depth of the indentation follow, the residual compressive stresses remained beneath the eliminated indentation. Finally, the change in phase content and the surface morphology of the specimens after aging at lower temperatures was measured.

Conclusions are obtained as follows:

1. By applying a Vickers indentation on the surface of the Y-TZP specimen, the metastable tetragonal phase beneath the impression transformed to monoclinic together with a volume expansion. And residual compressive stress is generated by the phase transformation. This residual stress did not allow the initiation and propagation of

other Vickers indentation induced cracks.

2. Degradation occurred in the Y-TZP specimens containing local residual stresses by aging at a moderate temperature of about 150°C. Protrusion took place in the region beneath the Vickers impression especially at the first stage of aging, in which also the percentage phase transformation from tetragonal to monoclinic was remarkable.

3. For the WC/Y-TZP materials which have been considered as very resistant to the degradation, the development of the residual stress-induced phase transformation was recognized.

By describing the results obtained from this investigation, it is emphasized that the degradation at low temperature in zirconia ceramics by local residual stress is unavoidable in many applications of ceramics to engineering use.

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