Strain Rate Dependence of Tensile Behavior and Environmental Effect in Zirconia Ceramics

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(Received on August 31, 2002; accepted in final form on October 22, 2002)

The tensile strength of four kinds of zirconia ceramics (two kinds of Y-TZP with different grain sizes, Mg-PSZ and Ce-TZP) has been investigated for a wide range of strain rates \(3 \times 10^{-9} - 1.0 \times 10^{-1} \text{ s}^{-1}\) in both air and vacuum. The obviously unique strain rate dependence was observed in Y-TZPs and Mg-PSZ, which have anelastic properties. On the other hand, such strain rate dependence was not observed over the whole range of strain rates for Ce-TZP, which does not exhibit anelasticity. For all of the samples investigated, there was no significant difference between both values of tensile fracture strength in air and vacuum. It was found that a decrease in strength with decreasing strain rate should not be associated with the static fatigue growth of microcracks nucleated during loading. It was concluded that the extent of the occurrence and the exhaustion of anelasticity predominantly controls the tensile strength of zirconia ceramics.

KEY WORDS: anelasticity; ADOV; ceramics; fatigue; fracture and fracture toughness.

1. Introduction

Plastic deformation in metallic materials is mainly dominated by the movement of dislocations. Because the dislocations are thermally activated, the ease of deformation of the matrix increases under conditions of lower strain rate or higher temperature. However, it has been found that even brittle ceramic materials, with little included plastic strain, show a similar strain rate dependence for fracture stress.\(^2\)\(^-\)\(^7\)\) In the case of ceramics, only a few dislocations exist in the original matrix, and even this small number of faults find it difficult to move because of the high Peierls potential (except for semi-brittle ceramic materials, such as MgO\(^2\)). Under conditions where there is a lower strain rate, not only the ease of dislocation slip, but also environmental effects (static fatigue; stress corrosion attack) increase during loading. Accordingly, such multiple effects might cause a significant undesirable decrease of fracture stress in various engineering materials.

The effect of strain rate on the strength of ceramics has been extensively studied by Lankford et al.\(^2\)\(^-\)\(^5\)\) and Subhash and Nasser.\(^6\)\(^7\)\) So far, Lankford et al.\(^2\)\(^-\)\(^5\)\) have investigated the compressive strength of various ceramic engineering ceramics over a wide range of strain rates, \(10^{-5} - 10^{-3} \text{ s}^{-1}\), and found that some materials exhibited a strain rate dependence for compressive fracture stress. This behavior could be interpreted in terms of the growth of axial microcracks nucleated during loading by tensile stress component. In regions where the strain rate is \(<10^{-2} \text{ s}^{-1}\), the relationship between \(\sigma_c\) (compressive strength) and \(\dot{\varepsilon}\) (strain rate) can be represented as\(^2\)\(^-\)\(^4\)

\[
\sigma_c \propto \dot{\varepsilon}^{1+\eta_c}
\]

where \(\eta_c\) is a constant value. \(n_c=m\); \(m\) is the exponent in the crack growth power law (Paris’ law), as given by

\[
V=AK^n
\]

where \(V\) is the crack growth velocity under tensile Mode I stress in a fracture mechanics-type specimen, and \(A\) is a constant depending on the material and the environmental conditions. Therefore, because \(n_c=m\), it has been assumed that the strain rate dependence of compressive strength is brought about by thermally activated microcrack growth by tensile stress component (quasi-static fatigue) in ceramics, with no dislocation slip. In this case, microcracks are produced axially, parallel to the stress axis.\(^2\)\(^-\)\(^5\)\) In some cases, however, this coincidence between \(n_c\) and \(m\) cannot be attained.\(^2\)\(^-\)\(^5\)\) For example, the discrepancy trend, \(n_c\neq m\), has been observed in transformable Mg-PSZ (MgO doped partially-stabilised zirconia),\(^9\)\(^10\) where \(n_c\) is nearly twice \(m\), suggesting that something other than, or in addition to, sub-critical crack growth is responsible for this discrepancy. Lankford\(^4\) has asserted that this effect is caused by a strain-induced martensitic transformation,\(^8\) which is thermally activated and which occurs under high compression in Mg-PSZ. Recently we have found unique time-dependent anelastic behavior in \(Y_2O_3\)-doped partially-stabilised zirconia,\(^9\)\(^10\) (see Fig. 1), which might be caused by the existence of oxygen vacancies and should be also of a thermally activated process, as described in the following section. Thus, the existence of a thermally activated mechanism makes it more difficult to systematise the strain rate dependence of fracture strength. Recently, it has been found that some kinds of ceramic
materials are degraded in hydrogen environment.\textsuperscript{11,12} However, the extent of the strength degradation or static fatigue in hydrogen is lower than that in air, as reported in alumina and zirconia ceramics.\textsuperscript{11,12} Therefore, it is much more important to examine the mechanical behavior in air from the view point of clarifying the environmental effect in the ceramic materials loaded. There are quite a few reports that discuss the strain rate effect in zirconia ceramics.\textsuperscript{4,6,7} Moreover, these studies have been carried out under compression testing, with the discussion focusing on tensile crack growth.\textsuperscript{2–7} In this study, we conducted dynamic tensile testing over a wide range of strain rates for four kinds of zirconia ceramics, which have different mechanical properties, transformability, anelastic properties\textsuperscript{9,10,13–18} and so on. Dynamic fracture testing was also carried out in vacuum in order to verify the true effect of subcritical microcrack growth on the strain rate dependence of tensile fracture strength.

2. Possible Mechanism of Anelasticity in Zirconia Ceramics

In our recent work,\textsuperscript{17,18} we have investigated anelastic behavior in some kinds of zirconia ceramics. Figure 2 shows the anelastic behavior in four kinds of materials (Y-TZP, Y-FSZ, Mg-PSZ and Ce-TZP) at 100 MPa and load-holding time of 10 h.\textsuperscript{18} This figure indicates that anelastic strain is produced in the Y\(_2\)O\(_3\)–ZrO\(_2\) and MgO–ZrO\(_2\) systems, but almost not in the CeO\(_2\)–ZrO\(_2\) system, irrespectively of the kind of crystallographic phase. This anelastic phenomenon appears even at extremely low stress level (<100 MPa) and produces time-dependent and recoverable strain.\textsuperscript{9,10} Therefore, such a unique behavior cannot be explained by conventional mechanism, for example, stress-induced phase transformation,\textsuperscript{10–21} ferroelastic domain switching,\textsuperscript{22–25} microcracking\textsuperscript{26–28} and so on. It is well known that the large amount of oxygen vacancy is introduced in matrix of the Y\(_2\)O\(_3\)–ZrO\(_2\) and MgO–ZrO\(_2\) systems in order to keep electrical balance and, on the other hand, the CeO\(_2\)–ZrO\(_2\) system seems to have few vacancies.\textsuperscript{29–31} From these concepts, it is assumed that the mechanism of anelasticity is concerned with the existence of oxygen vacancy, \textit{i.e.} slight shift of ions. Therefore, this anelasticity due to oxygen vacancy (ADOV) should be thermally activated\textsuperscript{14} and can work at relatively low stress level.\textsuperscript{9,10} As a result, ADOV is regarded as one of the significantly important strengthening-toughening mechanisms.

In a previous study\textsuperscript{13} we have investigated the relationship between fracture strength and stress rate in various pre-cracked zirconia ceramics and found a unique stress rate dependence in Y-TZP, indicating extremely high strength in the lower stress rate region at elevated temperature. It is assumed that activated ADOV\textsuperscript{9,10,13–18} releases stress concentrations in the region of the crack tip and thus causes an increase in fracture strength. Since ADOV has a thermally-activating factor,\textsuperscript{14} the mechanical properties of the materials should depend on the strain (stress) rate and the temperature.\textsuperscript{13,14} ADOV might therefore have some effect on the strain rate dependence of fracture strength, even in a smooth specimen.

3. Experimental Procedure

In this study, 4 kinds of zirconia ceramics were used, comprising of 2 different kinds of Y-TZP (3 mol% Y\(_2\)O\(_3\)-doped tetragonal zirconia polycrystals) with different sized grains and anelastic properties, Mg-PSZ (9 mol% MgO-doped partially stabilised zirconia) and Ce-TZP (12 mol% CeO\(_2\)-doped tetragonal zirconia polycrystals). Y-TZPs having different grain sizes were fabricated. The specimens of smaller grain size (Y-TZP(S)) were obtained by sintering at about 1 500°C, whereas the specimens of larger grain size (Y-TZP(L)) were produced by heat-treating of Y-TZP(S) at 1 650°C. As the grain size is increased (Y-TZP(S)→(L)), anelastic strain production is also increased (Sec. 4.3). These two kinds of Y-TZP were prepared to clarify the effect of ADOV on tensile deformation and fracture behavior. The details of the mechanical properties and microstructure of each of these materials are summarised in Table 1. These materials were prepared as test pieces with two notches for tensile rupture testing, as shown in Fig. 3. The two notches and two pinholes were carefully polished using 1/4 \(\mu\)m diamond paste before testing. Two strain gauges were attached to the central parts of the main surfaces (between the two notches). This two-gauge method effectively eliminates any strain error caused by slight bending of the
specimens.

Monotonic tensile rupture testing was carried out by stroke control at various cross-head speeds, over a wide range between $3 \times 10^{-3}$ mm/min and $1.0 \times 10^{1}$ mm/min, corresponding to strain rates of about $3 \times 10^{-2}$–$1.0 \times 10^{-1}$ s$^{-1}$. Testing was conducted both in air and in vacuum in order to clarify the environmental effect on fracture behavior (strain rate dependence of fracture stress). The testing machine was an Autograph AG-10kNE, made by Shimadzu, Kyoto, Japan. In the cases where we tested in vacuum and at loading speeds below $1.0 \times 10^{-2}$ mm/min ($1.0 \times 10^{-6}$ s$^{-1}$, the super-low strain rate region), an electro-hydraulic type servo-pulser, known as the EHF-EB 10kN-10L (Shimadzu), was used. The vacuum testing was carried out at below $8.0 \times 10^{-3}$ Pa pressure. The tensile fracture stress, $\sigma_f$, was evaluated as a function of the strain rate, $\dot{\varepsilon}$. The values of load and strain produced during loading were simultaneously captured in an oscillographic recorder as a voltage signal and were later analysed on a personal computer so that the stress vs. strain curves and the stress vs. non-elastic strain curves could be obtained. The non-elastic strain, $\varepsilon_{\text{non-elastic}}$, was calculated as follows.

$$\varepsilon_{\text{non-elastic}} = \varepsilon_{\text{total}} - \sigma/E$$ .................................. (3)

where $\varepsilon_{\text{total}}$ is the total strain, $\sigma$ the stress (If the stress concentration caused by notch radius is taken into consideration, the stress values should be multiplied by 1.77.) and $E$ is Young’s modulus. $E$ was measured from the slope of the linear part of the stress–strain curve at the highest strain rate in order to avoid the interfusion of anelastic strain.

4. Results and Discussion

4.1. Strain Rate Dependence of Tensile Fracture Strength and Effect of Environment

The results of tensile fracture testing in air for the four materials are shown in Fig. 4(a). The fracture strength depends on the strain rate in Y-TZP and Mg-PSZ. The fracture strength decreases with decreasing strain rate from the high strain rate to the low strain rate region (region A; $10^{-6}$–$10^{-1}$ s$^{-1}$), and then either saturation or a slight increase in strength (inverse strain rate dependence) is found in the super-low strain rate region below $10^{-6}$ (region B). On the other hand, the strength of Ce-TZP seems to be approximately independent of the strain rate over the whole region investigated. The tensile fracture stress $\sigma_T$ can be described as a function of the strain rate $\dot{\varepsilon}$, analogous to Eq. (1) under the compression loading condition$^{2,4}$;

$$\sigma_T \propto \dot{\varepsilon}^{1/n_T}$$ .................................. (4)

where $n_T$ is a constant indicating the extent of the strain rate dependence on the fracture strength. This relationship has been shown by Evans$^{1}$ to correlate tensile strength $\sigma_T$ with $\dot{\varepsilon}$ through dynamic bending testing, and the equivalence between $n_T$ and $m$ (the exponent of Paris’ power law, Eq. (2)) has been accepted as proof that the strain rate dependence of strength is based upon thermally activated tensile microcrack growth.$^{1,4}$ Analysis of the data from region A in Fig. 4(a) according to Eq. (4) yields the $n_T$ values given in Table 2, with $m$ values from our experimental data and references.$^{32,36}$ Good agreement is seen between $n_T$ and $m$ in Y-TZP and Mg-PSZ, suggesting the possibility that the

![Table 1](image)

Fig. 3. Test piece used for tensile fracture testing.

![Fig. 4](image)

Fig. 4. The relationship between tensile fracture strength and strain rate; (a) in air and (b) in vacuum.
monotonic decrease in tensile strength with decreasing strain rate observed in region A might be brought about by subcritical (static) growth of the microcrack nucleated during loading, as well as the other results previously reported in compression testing. On the other hand, the $n_t$ value considerably differs from $m$ in the case of Ce-TZP. It has been reported that fatigue crack growth progresses via repeated propagation and arrest, due to extensive stress-induced phase transformations produced in the region of the crack tip in Ce-TZP. Therefore, measurement of the $m$ value is in fact difficult, and seems to involve considerable data scattering, which might introduce significant discrepancies between $n_t$ and $m$ in Ce-TZP.

In order to investigate how much static microcrack growth contributes to the strain rate dependence of tensile fracture stress, the same dynamic fracture testing was carried out in vacuum. The relationship between tensile strength and strain rate in vacuum conditions is shown in Fig. 4(b). Since the number of specimens is limited, we could not conduct the enough tests. However, the figure shows quite unexpectedly astonishing results, which indicate that the strength in a vacuum is approximately the same as that in air for each sample examined. This result leads us to the significant conclusion that the decrease in strength with decreasing strain rate and the agreement between the $n_t$ and $m$ values (at region A) are not principally caused by static microcrack growth, but by other, thermally activated, mechanisms. Although it is generally known that the stress-induced phase transformation, regarded as significant strengthening-toughening mechanisms in zirconia ceramics occurs athermally, it has been reported that such phase transformation occurs thermally or isothermally in some kinds of zirconia ceramics under certain conditions. Therefore, serious attention is required in order to comprehend the effect of phase transformation on strain rate dependence of strength. For example, Lankford has investigated the relationship between compressive fracture strength and strain rate in Mg-PSZ, and has found a discrepancy in the exponent values, $n_t \neq m$, and concluded that a strain-induced martensitic transformation contributes to the strain rate dependence in particularly high stress situations. The strain-induced transformation would act thermally and form deformation bands in the matrix. In the tension situation, however, it is assumed that the ceramic material would break down at a relatively lower stress level before the strain-induced transformation could occur, since the accompanying high stress concentration should simultaneously initiate microcracking and cause catastrophic fracture. Therefore, we insist that this concept should not be applied to the case of the tensile fracture behavior of zirconia ceramics. Besides which, taking into account that at most, only a few dislocations originally exist in the matrix and that even if there are any, slip is so much harder to achieve in brittle ceramics than metal, ADOV presents itself as the most probable thermally-activated mechanism that causes the strain rate dependence in zirconia ceramics. It should be noted that the strain rate dependence of tensile fracture strength is also observed only in Y-TZP and Mg-PSZ, which have the anelastic properties shown in Fig. 2.

### 4.2. Analysis of Stress–Strain Behavior in Air and Vacuum

Stress–strain curves for each material investigated, both in air and in vacuum, are shown in Fig. 5. The data for the stress rates $1.0 \times 10^{-1}$, $1.0 \times 10^{-6}$ and $3 \times 10^{-9}$ s$^{-1}$, which represent a high strain rate, a low strain rate and a super-low strain rate respectively, are exhibited for convenience of presentation and comprehension. (The curves at the super-low strain rate in vacuum are not shown in Fig. 5 because the electric noises caused by the vacuum system disturbed the precise measurement of stress–strain relations at the lower stress region. However, there was no problem for measurement of fracture strength (Fig. 4).) A relatively high linear relationship is observed up to fracture in all samples, which indicates that the non-linear strain portion is much smaller than the elastic portion. However, the former significantly influences the fracture behavior for brittle ceramics. The strain rate dependence of the slope can be confirmed in the low stress region (full elastic region expected) in Y-TZP. The slope decreases with decreasing strain rate. While a slight strain rate dependence on the slope is also observed in Mg-PSZ, all of the curves nearly match one another, regardless of strain rate, in the case of Ce-TZP, which has no anelastic properties. These results suggest that time-dependent ADOV should produce non-elastic strain, even in the low stress region, and could cause such a strain rate dependence, resulting in the interesting stress–strain behavior that we observed.

The difference between both of these curves in air and in vacuum is only very small in the majority of cases, as would be expected from the results of the stress–strain rate dependence in Fig. 4. While a high linear relationship continues up to the final fracture in every Y-TZP sample, a relatively large deviation from the linear relationship is observed in Mg-PSZ, especially for lower strain rates. On the other hand, a remarkably large non-linear strain is produced in the case of Ce-TZP just before the specimen is ruptured. This non-linear strain is not caused by static microcrack growth, because the same phenomena can even be observed in vacuum. This pronounced increase in strain seems to be caused in large part by a stress-induced transformation, as indicated by the high transformability of this material.

In order to comprehend the deformation behavior under conditions of varying strain rate, the stress–non-elastic strain curves were obtained using Eq. (3). The results are shown in Fig. 6. The difference in non-elastic strain behavior between both the materials and the strain rates now becomes clearer. It is assumed that the time dependent anelastic strain should be limited under conditions of high strain rate, since the samples rupture instantly (some few microseconds). Therefore, the non-elastic strain observed for higher stress region under high strain rate conditions is not caused by ADOV, but by other mechanisms, which operates.

<table>
<thead>
<tr>
<th>Materials</th>
<th>$n_t$</th>
<th>$m$</th>
</tr>
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<tbody>
<tr>
<td>Y-TZP(Si)</td>
<td>21</td>
<td>20</td>
</tr>
<tr>
<td>Y-TZP(LJ)</td>
<td>52</td>
<td>51</td>
</tr>
<tr>
<td>Mg-PSZ</td>
<td>27</td>
<td>23</td>
</tr>
<tr>
<td>Ce-TZP</td>
<td>332</td>
<td>15</td>
</tr>
</tbody>
</table>
Fig. 5. Stress–strain curves for four kinds of zirconia ceramics in air (1) and in vacuum (2); (a) Y-TZP(S), (b) Y-TZP(L), (c) Mg-PSZ and (d) Ce-TZP.
Fig. 6. Stress–non-elastic strain curves for four kinds of zirconia ceramics in air (1) and in vacuum (2); (a) Y-TZP(S), (b) Y-TZP(L), (c) Mg-PSZ and (d) Ce-TZP.
at stresses higher than the certain level, such as stress-induced transformation\(^{19-21}\) or microcracking.\(^{26-28}\) In short, the large amount of non-elastic strain in the lower strain rate regime is predominantly composed of anelastic strain. The contribution of ADOV is recognised as the difference in the strain from that observed at high strain rate. A slight negative, non-elastic strain is found at one part of the curve at high strain rate, especially in the Y-TZP. This is just a problem in the calculation using Eq. (3). There is a possibility that this “curvature” portion is caused by the “stress dependence of Young’s modulus”.\(^{41}\) If the modulus did not depend on stress, such a curvature might not be observed. The existence of the stress dependence in Young’s modulus for ceramic materials is of great interest, but this point is ignored in this study. We consider that there is no problem in comparing the stress–non-elastic strain curves with the change of strain rate.

In both examples of Y-TZP, a relatively large amount of non-elastic strain is produced by the ADOV with decreasing strain rate (See Figs. 6(a-1) and 6(b-1)). Especially in the case of Y-TZP(L), a larger amount of non-elastic strain is produced at low strain rate (1.0×10\(^{-6}\) s\(^{-1}\)) than for Y-TZP(S) sample. Moreover, while the non-elastic strain production increases with decreasing strain rate from the low strain rate to the super-low strain rate regime in Y-TZP(S), the stress–non-elastic strain curves at both strain rates are not significantly different in the case of Y-TZP(L). In other words, the anelastic strain has been already saturated at 1.0×10\(^{-6}\) s\(^{-1}\) in the Y-TZP(L), indicating the highest anelastic strain productivity under these conditions. Although there is some degree of difference between the curves in air and in vacuum, no outstanding difference is found in terms of the curve shape (see Figs. 6(a-2) and 6(b-2)).

Next, as for Mg-PSZ, it is of interest that a remarkable increase in the non-elastic fracture strain is observed at lower strain rates (Fig. 6(c-1)). According to our previous work (Fig. 2),\(^{18}\) anelastic strain productivity in the Mg-PSZ is less than for the Y-TZP. Therefore, we do not consider that such large amount of non-elastic strain is produced only by the ADOV. Not only ADOV, but also a time-dependent transformation due to the quasi-static stress condition might be involved.\(^{50}\) Nakanishi and Shigematsu\(^{37}\) have observed isothermal (time-dependent) phase transformation in Y-TZP and asserted that this transformation is due to a fairly amount of oxygen vacancies, that is, a high diffusibility of oxygen ions through their vacancies. Mg-PSZ includes larger amount of oxygen vacancies than Y-TZP and Ce-TZP.\(^{29}\) Our recent work has clarified that stress-induced transformation occurs only in Mg-PSZ isothermally even at room temperature.\(^{18}\) Therefore, a remarkable increase in the non-elastic strain in Mg-PSZ (Fig. 6(c-1)) is also relevant to this isothermal transformation, in addition to ADOV. From the concept that anelastic strain is produced by slight shift of ions as described in Sec. 2, Mg-PSZ is supposed to have higher anelastic strain productivity than Y-TZP because of the extent of the amount of oxygen vacancies. However, actual experimental result indicated an inverse tendency (anelastic productivity; Mg-PSZ<Y-TZP).\(^{17,18}\) Therefore, it seems that the productivity of ADOV might be controlled also by the size of dopant ion.

Strangely, non-elastic strain seems to be produced more effectively in vacuum than in air. The ambient temperature in vacuum was different from that in air due to the cooling system of vacuum chamber; the former was several degrees lower than the latter, which might result in promotion of stress-induced transformation, although the detail is unclear.

Finally, as in the case of Ce-TZP, the curves coincide with one another and do not have a different dependence on strain rate than any of the other materials investigated (Fig. 6(d-1)). This shows the essential fact that Ce-TZP has no anelastic properties,\(^{17,18}\) resulting in the strain rate being independent of the strength. Some intervals can be found between the dotted points in the curves just before fracture occurs, which indicates that the significant increase in non-elastic strain just before fracture is generated due to transformation or microcracking. The evidence for microcracking is seen in the neighborhood of the fracture surface, as shown in the SEM photograph (Fig. 7). Figure 7 includes

(a) Ce-TZP

(b) Mg-PSZ

Fig. 7. SEM image of neighborhood of fracture surface; (a) Ce-TZP and (b) Mg-PSZ. (Loading condition: at high strain rate in vacuum.)
the images of Mg-PSZ as comparison, indicating that microcracks are produced more effectively just before catastrophic fracture in Ce-TZP. Furthermore, a more linear relationship was observed in vacuum than in air (Fig. 6(d-2)). This result might be also related to the occurrence of microcracking influenced by the environment. Although Mg-PSZ has a high transformability as well as Ce-TZP, the significant increase in non-elastic strain just before fracture cannot be observed in Mg-PSZ. This is because the crack initiates more easily in Mg-PSZ than in Ce-TZP, which limits the extent of the subsequent transformation, as verified in our previous indentation testing. In short, the first occurrence of microcracking easily tends to lead to the fatal failure in the case of Mg-PSZ. Even if microcracking occurs in Ce-TZP, it is instantly closed by a remarkable transformation and then the stress concentration is released, resulting in a large disparity between $n_T$ and $m$ (Sec. 4.1). After the occurrence of microcracking, the stress concentration area is modified by the crack-closure effect in the case of smooth specimens.

4.3. The Effect of ADOV on Strain Rate Dependence of Strength

Obviously, ADOV has some effect on the strain rate dependence of strength in zirconia ceramics. Strain rate dependence is found in Y-TZP and Mg-PSZ, which have anelastic properties, but not in Ce-TZP which has no anelastic properties, as indicated in Sec. 4.1 and Fig. 4. The relationship between the non-elastic strain and the grain size (Y-TZPs(S) and (L)) gives us an important estimation. It is assumed that a major part of the non-elastic strain should be anelastic strain under the conditions investigated. From Fig. 6, therefore, it is confirmed that anelastic strain production increases as the grain size increases.

It is most important to understand how ADOV contributes to strength variation. An exhaustion of ADOV leads to a decrease in strength and toughness. There is a possibility that the decrease of strength with decreasing strain rate (Region A in Fig. 4(a)) might be related to the extent of exhaustion of the ADOV. If this strength degradation were due to exhaustion of ADOV, the strength could be determined deliberately by controlling the extent of exhaustion of the ADOV. In order to confirm this possibility, an additional experiment was conducted involving changing the strain rate during loading by using Y-TZP(L). After the ADOV was saturated (exhausted) by keeping a certain stress level (450 MPa) for a sufficiently long time (15 h), the material was ruptured at a high strain rate (exhaustion-rupture test). If the strength were determined by the extent of exhaustion of ADOV, the material should be ruptured at a considerably lower strength level, lower than that at high strain rate under conditions of non-exhaustion of ADOV. The experimental result is shown in Fig. 8 with the series of curves (schema) obtained in air. Contrary to what we had expected, a curious result was obtained, indicating that not only the strength but also the non-elastic strain increased significantly. This obviously suggests that the strength cannot be determined only by the extent of exhaustion of ADOV. The large amount of non-elastic strain produced after saturation of ADOV seems to be mainly caused by stress-induced transformation, because the loading speed is so high that anelastic strain cannot be produced. X-ray diffraction (XRD) profiles were obtained from the neighborhood of fracture surface in order to confirm this probability. Figure 9 shows the volume fraction of monoclinic phase of the specimen after the exhaustion-rupture test with the data of the specimen fractured at the high strain rate (sample A). It is considered that transformation produced non-elastic strain of about 100 με and 300 με in high strain rate rupture test and exhaustion-rupture test, respectively. Since the lattice volume dilatation during tetragonal to monoclinic phase transformation is about 4%, the simple calculation assuming isotropy of material indicates the difference of strain (about 200 με) should correspond to about 1.5% increase of volume fraction of monoclinic phase. This theoretical value approximately coincides with the experimental data seen in Fig. 9. Therefore, this result implies that the progress of stress-induced transformation is promoted under conditions of saturation or exhaustion.
tion of ADOV, resulting in an increase in strength. The energy given to the material is predominantly divided between the occurrence of ADOV and stress-induced transformation. The lower the strain rate is, the more energy is expended to the occurrence of ADOV and not to stress-induced transformation. In the final fracture process in the additional experiment, the energy is all expended as the driving force for the occurrence of phase transformation.

A saturation or slight increase in strength (inverse strain rate dependence) is observed in the super-low strain rate region (Region B in Fig. 4) in Y-TZP and Mg-PSZ. It is difficult to precisely determine the cause of the inverse strain rate dependence. However, we can qualitatively explain the behavior in the super-low strain rate region, by referring to the result in Fig. 8. Since the material is always in the anelasticity-saturated state in the super-low strain rate region, the given deformation energy might easily contribute to a phase transformation. As a result, an increase in strength may be caused by a stress-induced transformation.

While ADOV is activated at elevated temperature,\(^\text{14}\) it is usual that stress-induced transformation is inversely restrained.\(^\text{21,33}\) Therefore, in order to clarify the true effect of ADOV and stress-induced transformation on the strain rate dependence of strength, it is necessary to conduct a fracture testing experiment similar to the present one at elevated temperature.

5. Conclusions

The relationship between tensile fracture strength and strain rate was investigated in air and vacuum for four different types of zirconia ceramics that possess different anelastic properties. The results of our research can be summarised as follows.

(1) Strain rate dependence is observed in Y-TZP and Mg-PSZ, which have anelastic properties, but not in Ce-TZP, which exhibits no ADOV (anelasticity due to oxygen vacancy). Furthermore, saturation or inverse strain rate dependence of strength is observed in the super-low strain rate region in Y-TZP and Mg-PSZ.

(2) There is no significant difference between both the tensile fracture strength values (strain rate dependence of strength) in air and vacuum, for all of the zirconia samples investigated. It is assumed that the strain rate dependence is not caused by static fatigue microcrack growth, but by some other effect concerned with the ADOV.

(3) The tensile strength of zirconia ceramics is controlled by the extent of the occurrence and exhaustion of ADOV. The activity of the ADOV strongly depends on the strain rate, resulting in the enhancement of a strain rate dependence of the strength.

Acknowledgements

This research was supported by (i) Special Coordination Funds of Ministry of Education, Culture, Sports, Science and Technology of the Japanese Government and (ii) Waseda University Grant for Special Research Projects (2000A-524). The authors wish to acknowledge these supports.

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