Effect of Ta$_2$O$_5$, Nb$_2$O$_5$, and HfO$_2$ Alloying on the Transformability of Y$_2$O$_3$-Stabilized Tetragonal ZrO$_2$

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The addition of Ta$_2$O$_5$, Nb$_2$O$_5$, and HfO$_2$ enhanced the transformability of Y$_2$O$_3$-stabilized tetragonal ZrO$_2$ poly-crystal (Y-TZP), which was indicated by an increase in phase transformation temperatures and fracture toughness of Y-TZP. Comparison of the alloying effects of these oxides on the transformability and crystal structure of Y-TZP suggested that an alloying oxide which increases the c/a axial ratio (tetragonality) of TZP also increases the transformability. Empirical equations to predict the tetragonality are proposed. Calculated tetragonality showed good agreement with measured values in the systems ZrO$_2$-Y$_2$O$_3$-Ta$_2$O$_5$, -Nb$_2$O$_5$, and -HfO$_2$. [Key words: yttria, zirconia, alloys, transformations, fracture toughness.]

I. Introduction

It is well-known that for ZrO$_2$ to be utilized for technical applications the high-temperature polymorphs, namely, cubic (c) and tetragonal (t) phases, should be stabilized at ambient temperature by the formation of solid solutions which prevent delitserous tetragonal-to-monoclinic (m) phase transformation. The alloying oxides which lead to the stabilization are alkaline-earth, rare-earth, and actinide oxides and some transition-element oxides such as In$_2$O$_3$. Although there is no theoretical basis to select the stabilizer, it has been suggested that the factors which may influence the stabilization are size, valency, and concentration of solute cations and crystal structure of the solute oxide, where the valency and concentration determine the number of oxygen vacancies created by the formation of substitutional solid solutions.

Y$_2$O$_3$ is a very effective stabilizer for t-ZrO$_2$, which possesses high values of strength and fracture toughness with an optimum amount of Y$_2$O$_3$ concentration. The t-ZrO$_2$ phase retention at room temperature in this system depends on the composition, grain size, and density of the Y$_2$O$_3$-stabilized t-ZrO$_2$ polycrystal (Y-TZP). For a given grain size and density, the addition of Y$_2$O$_3$ to ZrO$_2$ lowers the transformation temperatures of $t \rightarrow m$ ($M_s$) and $m \rightarrow t$ ($A_s$), so that the chemical driving force for the transformation is reduced with increasing Y$_2$O$_3$ concentration. Thus the transformability or instability of Y-TZP decreases as the Y$_2$O$_3$ content increases. This lowered transformability is manifested by a small amount of m-ZrO$_2$ fraction observed on the fracture surface of Y-TZP and is accompanied by a decrease in the fracture toughness.

Schubert has related the compositional dependence of the stability of TZP in this system to its dependence on the thermal expansion anisotropy ($\Delta a$), which decreases with increasing Y$_2$O$_3$ concentration. $\Delta a$ is known to govern the residual stress in t-ZrO$_2$ so that the increased residual stress enhances the transformability. Since the $\Delta a$ of Y-TZP decreases with increasing Y$_2$O$_3$ content, it was expected that the composition at which the $\Delta a$ becomes zero would coincide with that at which c-ZrO$_2$ becomes stable. However, the composition determined from the linear extrapolation of the $\Delta a$ to zero was very different from the composition for c-ZrO$_2$ obtained from the measurement of lattice parameters. Consequently, the effect of alloying on the transformability cannot be predicted by the effect on the $\Delta a$ alone. On the other hand, the alloying of HfO$_2$ into ZrO$_2$ raises the $A_s$ and $M_s$ temperatures, which is attributed to an increase in the driving force for the $t \rightarrow m$ transformation.

The increased $M_s$ due to HfO$_2$ alloying has been interpreted by Burke and Garvie using the concept of the solid phonon mode. In their argument $M_s$ increases with decreasing effective cation mass of the resulting solid solution, which is decreased by the addition of HfO$_2$. This simple interpretation is not applicable to changes in $M_s$ in the other ZrO$_2$ solid solutions since these solid solutions cannot conform to the assumption that ZrO$_2$ and a solute are identical with the exception of cation masses.

In the present paper, a systematic investigation of the effect of ternary element alloying on the transformability of Y-TZP was conducted in an effort to elucidate the origin of the compositional dependence of the transformability of t-ZrO$_2$. Primarily, the effect of Ta$_2$O$_5$ alloying on the transformation temperatures, fracture toughness, and lattice parameters of Y-TZP was investigated. In addition, Nb$_2$O$_5$ and HfO$_2$ were employed separately as ternary allowing oxides in Y-TZP to add to a generalization of the role of alloying oxides on the transformability. An effort was made to predict the alloying effect on the transformability which leads to a selection criterion for the stabilizer of t-ZrO$_2$. 

II. Experimental Procedure

Starting powders were prepared by coprecipitation from aqueous solutions of Zr(SO$_4$)$_2$·4H$_2$O, Y(NO$_3$)$_3$·6H$_2$O, and TaCl$_5$, NbCl$_5$, or HfOCl$_2$·8H$_2$O with ammonia. For some compositions in the system ZrO$_2$-Y$_2$O$_3$-Nb$_2$O$_5$, ZrOCl$_2$·8H$_2$O was used for ZrO$_2$ because of the decomposition of Zr(SO$_4$)$_2$·4H$_2$O with high concentration of NbCl$_5$ solution in an aqueous mixture. The precipitates were dried for 20 h at 120°C after washing with distilled water and decomposed to the oxides by calcination for 3 h at 920°C. The oxide agglomerates which contained Ta$_2$O$_5$ or HfO$_2$ were dry-milled in a planetary mill using an alumina jar and balls. The powders in the system ZrO$_2$-Y$_2$O$_3$-Nb$_2$O$_5$ were obtained by attrition milling in isopropyl alcohol using ZrO$_2$ balls. Bend test specimens were prepared by pressing the powders isostatically at 170 MPa, and sintering for 1 h at 1500°C. The density of sintered specimens was determined by the Archimedes method.

The fracture toughness was determined by two methods. One method involved the fracture of polished four-point bend specimens of dimensions 2.5 mm $\times$ 3.0 mm $\times$ 30 mm containing three 147-N Vickers indentations on the tensile surface within the inner span after Cook and Lawn. The dimensions of the inner and outer spans were 10 and 20 mm, respectively. The samples were tested at a crosshead speed of 0.5 mm/min. 

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The fracture toughness of each composition was obtained from an average value of three measurements. The other method employed the indentation technique applying the equation proposed by Anstis et al., where the value of 205 GPa was used for the modulus of TZP. For the indentation method the indent load was varied from 196 to 490 N and the value of the fracture toughness was determined again by the average of three measurements. Four-point bend strength was obtained from the fracture of the bend specimens also at a crosshead speed of 0.5 mm/min.

The monoclinic fraction on the specimens was determined from X-ray diffractometer (XRD) peak intensities after Garvie and Nicholson. A dilatometer was used to measure the \( A_\text{t} \) and \( M_\text{s} \), transformation temperatures, which correspond to the starting points of the discontinuous volume shrinkage and expansion of the samples during heating and cooling, respectively. The dilatometry samples were heated from room temperature to 900°C and cooled at the same rate of 7.5°C/min. The average grain size was measured by the linear intercept method with the use of a correction factor of 1.56 after Mendelson. The lattice parameters were determined by least-squares refinement of XRD data, obtained using CuK\( \alpha \) with Si as a standard and utilizing the program LCLSQ.

### III. Results and Discussion

The effect of \( \text{Ta}_2\text{O}_5 \) alloying on the transformability of Y-TZP was examined by determining the effect on the transformation temperatures, \( A_\text{t} \) and \( M_\text{s} \), of 2 and 3 mol\% Y-TZP. As shown in Fig. 1, both \( A_\text{t} \) and \( M_\text{s} \) of each Y-TZP increase with the addition of \( \text{Ta}_2\text{O}_5 \), so that the chemical driving force for the \( t \rightarrow m \) transformation of Y-TZP is enhanced because of the \( \text{Ta}_2\text{O}_5 \) alloying. This \( \text{Ta}_2\text{O}_5 \) alloying effect is contrasted to the effect of \( \text{Y}_2\text{O}_3 \), a stabilizer for TZP, whose addition decreases the transformation temperatures.

Figure 2 compares the alloying effect of \( \text{Ta}_2\text{O}_5 \) with that of \( \text{HfO}_2 \) on \( M_\text{s} \) of 2 mol\% Y-TZP. The \( \text{HfO}_2 \) has been known as the only solute which increases the \( M_\text{s} \) temperature. The increasing trend of \( M_\text{s} \) due to \( \text{Ta}_2\text{O}_5 \) alloying is steeper than that due to \( \text{HfO}_2 \) alloying, which implies that \( \text{Ta}_2\text{O}_5 \) is a more effective destabilizer for TZP than \( \text{HfO}_2 \). The density of the specimens was 297% of the theoretical density. No \( m \)-\( \text{ZrO}_2 \) phase was detected in the as-sintered specimens, except in the sample with 1.5 mol\% \( \text{Ta}_2\text{O}_5 \) which contained about 8% \( m \)-\( \text{ZrO}_2 \). The fracture toughness increases from 5 to about 3.0 MPa m\(^{1/2}\) for 2 and 3 mol\% Y-TZP prepared by sintering at 1500°C for different times.

The alloying effect on the fracture toughness of Y-TZP was investigated and the result is shown in Fig. 3, where \( \text{Ta}_2\text{O}_5 \) is added to both 2 and 3 mol\% Y-TZP, \( \text{HfO}_2 \) is added to 2 mol\% Y-TZP, and \( \text{Nb}_2\text{O}_5 \) is added to 3 mol\% Y-TZP. The toughness value of the specimen of 2 mol\% Y-TZP with 0.5 mol% \( \text{Ta}_2\text{O}_5 \) could not be obtained since most of the \( t \)-\( \text{ZrO}_2 \) phase transformed to \( m \)-\( \text{ZrO}_2 \) phase during sample preparation. The alloying showed little effect on the microstructure of the specimens sintered at 1500°C for 1 h. Typically, the grain size of TZPs in the system \( \text{ZrO}_2 \)-Y\(_2\text{O}_3\)-\( \text{Ta}_2\text{O}_5 \) and \( \text{HfO}_2 \) was determined to be about 0.5 \( \mu \)m for all alloy additions. The density of the specimens was 97% of the theoretical density. No \( m \)-\( \text{ZrO}_2 \) phase was detected in the as-sintered specimens, except in the sample with 1.5 mol% \( \text{Ta}_2\text{O}_5 \) which contained about 8% \( m \)-\( \text{ZrO}_2 \). The fracture toughness increases from 5 to about

![Fig. 1. Influence of \( \text{Ta}_2\text{O}_5 \) alloying on phase transformation temperatures of 2 and 3 mol\% Y-TZP prepared by sintering at 1500°C for 15 h.](image1)

![Fig. 2. Comparison of \( \text{Ta}_2\text{O}_5 \) and \( \text{HfO}_2 \) alloying effects on \( t \rightarrow m \) transformation temperature of 2 mol\% Y-TZP. Specimens were prepared to have grain sizes of about 1.0 \( \mu \)m by sintering at 1500°C for different times.](image2)

![Fig. 3. \( \text{Ta}_2\text{O}_5 \), \( \text{Nb}_2\text{O}_5 \), and \( \text{HfO}_2 \) alloying effects on fracture toughness of 2 and 3 mol\% Y-TZP prepared by sintering at 1500°C for 1 h.](image3)
with that of Ta$_2$O$_5$ alloying. The trends of Ta$_2$O$_5$ and HfO$_2$,
January 1990 Effect of formability of t-ZrO$_2$, due to the alloying shown in Fig. 1. The influence of Nb$_2$O$_5$ alloying is similar to that due to Ta$_2$O$_5$, but the effect of HfO$_2$ alloying is not noticeable as compared with that of Ta$_2$O$_5$ alloying. The trends of Ta$_2$O$_5$ and HfO$_2$ alloying effects on the fracture toughness of 2 mol% Y-TZP are analogous to those on the $M_r$ temperature shown in Fig. 2. Thus, Ta$_2$O$_5$ and Nb$_2$O$_5$ are strong destabilizers which enhance the transformability of Y-TZP, as compared with HfO$_2$. Figure 4 shows the Ta$_2$O$_5$ alloying effect on the bending strength and $m$-ZrO$_2$ fraction on the polished surface of 3 mol% Y-TZP. The bending strength slightly increases with the addition of Ta$_2$O$_5$ up to 1.0 mol% and decreases with further addition of Ta$_2$O$_5$. This decrease in the bending strength of the samples with 1.5 mol% Ta$_2$O$_5$ is due to the presence of a large amount of $m$-ZrO$_2$ phase on the polished sample surface. Increasing the sintering time, which results in grain growth, causes a further drop in the strength of the samples with 1.5 mol% Ta$_2$O$_5$ due to the increased amount of $m$-ZrO$_2$ phase.

The alloying effect on the transformability has been demonstrated by measuring the $m$-ZrO$_2$ phase fraction on the fracture surface of the bend test specimens, where the increasing trend of $m$-ZrO$_2$ fraction after the fracture as a function of Ta$_2$O$_5$ content shows excellent correlation with the fracture toughness. The fracture toughness values measured by the multiple-crack method were identical to those obtained by indentation as long as the minimum requirement of $c \geq 2a$ suggested by Anstis et al. is met, where $c$ and $a$ are the half crack length from a Vickers diamond pyramid indent and the half diagonal of the indent, respectively. Otherwise, the values by the indentation technique are overestimated by about 10% to 15% as compared with the values by the multiple-crack method. This is the case for the specimens with 1.0 and 1.5 mol% Ta$_2$O$_5$ or Nb$_2$O$_5$ after indentation with the maximum load of 490 N employed in the present study.

The effects of Ta$_2$O$_5$, Nb$_2$O$_5$, and HfO$_2$ alloying on the changes in the $c/a$ axial ratio (tetraxiality) of Y-TZP are illustrated in Fig. 5, and the lattice parameters of the resulting $t$-ZrO$_2$ solid solutions at room temperature are listed in Table 1. As shown, the alloying of these oxides into Y-TZP increases the tetraxiality. On the other hand, the addition of oxides such as Y$_2$O$_3$, Yb$_2$O$_3$, Gd$_2$O$_3$, and CeO$_2$ to stabilize $t$-ZrO$_2$ is found to decrease the tetragonal distortion as summarized in Fig. 6. This is the exactly opposite effect of the alloying of Ta$_2$O$_5$, Nb$_2$O$_5$, and HfO$_2$, which enhances the transformability of Y-TZP. Thus the destabilizer and the stabilizer for the $t$-ZrO$_2$ might be differentiated depending on its role in the changes of the tetraxiality of the resulting solid solution. Again, the effects of Ta$_2$O$_5$ and Nb$_2$O$_5$ alloying on the tetraxiality are distinguished from the effect of HfO$_2$. In particular, the influences of Ta$_2$O$_5$ and HfO$_2$ alloying on the tetraxiality of 2 mol% Y-TZP as shown in Fig. 5 are consistent with those on the $M_r$ temperature and fracture toughness, as illustrated in Figs. 2 and 3, respectively. To see how the transformability of these ternary TZPs changes with the tetraxiality, the $M_r$ temperatures of the system ZrO$_2$–Y$_2$O$_3$–Ta$_2$O$_5$ shown in Fig. 1 are plotted as a function of the tetraxiality in Fig. 7, where it can be seen that the $M_r$ temperature of 2 and 3 mol% Y-TZP increases linearly with increasing tetraxiality. The effect of the tetraxiality on the transformability is apparent in Fig. 8, where the fracture toughness values are from Fig. 3. As a result, at least phenomenologically, the alloying effect on the transformability of TZP is closely related to the effect on the tetraxiality of $t$-ZrO$_2$ solid solution in such a way that the transformability increases with the tetraxiality.

The relationship between transformability and tetraxiality is supported by consideration of $t$'–ZrO$_2$, which is known as nontransformable $t$-ZrO$_2$. The $c/a$ ratio of $t$'–ZrO$_2$ in the system Y$_2$O$_3$–ZrO$_2$ is closer to unity than that of transformable $t$-ZrO$_2$. The smaller tetragonality of $t$'–ZrO$_2$ is likely responsible for its nontransformability. Another indication is in the system Y$_2$O$_3$–CeO$_2$–ZrO$_2$, where the substitution of Y$_2$O$_3$ for CeO$_2$ causes a decrease in the fracture toughness and an increase of the phase stability of Ce-TZP. This Y$_2$O$_3$ alloying effect is predictable since the decrease in the tetragonality due to Y$_2$O$_3$ is more pronounced than that due to CeO$_2$, as shown in Fig. 6. The alloying effect of Y$_2$O$_3$ and CeO$_2$ on tetragonality is consistent with that on the $m$–$t$ transformation temperature; that is, the decrease in $A_t$ temperature per mole percent solute is reported to be 170–7° C for YO$_{1.5}$ and 71.2°C for CeO$_2$. Evidently, $t$'–ZrO$_2$ solid solutions become unstable as their tetraxiality increases toward 1.020 (Fig. 6), which corresponds to the $b/a$ axial ratio of $m$-ZrO$_2$ at room temperature. On the other hand, they become stable as the tetraxiality decreases toward unity, which corresponds to $c$-ZrO$_2$. This relationship allows the classification of oxides into either a stabilizer (decreasing tetraxiality) or a destabilizer (increasing tetraxiality) for the $t$-ZrO$_2$ phase at room temperature.
Table 1. Comparison of Measured and Calculated Lattice Parameters of t-ZrO₂ Solid Solutions

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<tr>
<th>Composition (mol%)</th>
<th>Measd</th>
<th>Calcd</th>
<th>Δa (%)*</th>
<th>Measd</th>
<th>Calcd</th>
<th>Δc (%)*</th>
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<tr>
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<td>0.51883</td>
<td>0.05</td>
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*[(measured value - calculated value)/(measured value)] x 100.

Fig. 6. Compositional dependence of c/a axial ratio of t-ZrO₂ solid solutions stabilized by various oxides (Refs. 21 to 23).

In order to control the fracture toughness of TZP through alloying, besides the changes in the microstructure, it is desirable to formulate equations which predict changes in the lattice parameters of t-ZrO₂ as a result of compositional variations since the tetragonality is related to the transformability. The empirical equations for the changes in the lattice parameters, a and c, of t-ZrO₂ solid solutions at room temperature are expressed as follows:

\[
a = 0.5086 + \sum_k (0.0255\Delta r_k + 0.00015\Delta z_k) m_k
\]

\[
c = 0.5189 + \sum_k (0.0213\Delta r_k + 0.00064\Delta z_k) m_k
\]

where a and c are in nanometers, \(\Delta r_k\) (in nanometers) is the difference in ionic radius (\(r_k - 0.084\)) of the kth solute cation (\(r_k\)) and Zr⁴⁺ (0.084 nm) in eightfold coordination from Shannon's compilation, \(\Delta z_k\) is the cation valency difference (\(z_k - 4\)), and \(m_k\) is the mole percent of the kth solute, in the form of \(MO_x\), \(x = 4\). The term \(m_k\), which corresponds to \(M_k\) for \(n_k = 1\), is calculated by

\[
m_k = \frac{n_k M_k}{100 + \sum (n_k - 1) M_k}
\]
where \( n_k \) is the number of cations in the \( k \)th solute oxide, and \( M_k \) is the concentration of the \( k \)th solute oxide in mole percent (e.g., \( n_1 = 2 \) and \( M_1 = 3 \) for 3 mol\% \( \text{Y}_2\text{O}_3 \)). The constants in Eqs. (1) and (2) were determined from regression analyses of \( t\text{-ZrO}_2 \) lattice parameters in the systems \( \text{Y}_2\text{O}_3-\text{HfO}_2 \), \( \text{Y}_2\text{O}_3-\text{Gd}_2\text{O}_3 \), and \( \text{CeO}_2-\text{ZrO}_2 \). The details of the formalism of Eqs. (1) and (2) are described elsewhere.\(^{31}\) To illustrate, the changes in the lattice parameters of \( t\text{-ZrO}_2 \) in the system \( \text{Y}_2\text{O}_3-\text{ZrO}_2 \) as a function of the mole percent \( \text{YO}_1.5 \) can be represented as

\[
a = 0.5086 + 0.0003m_{\text{Y}}
\]

\[
c = 0.5189 - 0.00026m_{\text{Y}}
\]

by using \( \Delta c = 0.0179 \) \((r_{\text{Y}^{+}+} = 0.1019 \text{ nm})\) and \( \Delta c = -1 \). Equations (4) and (5) are in excellent agreement with the equations determined experimentally by Toraya.\(^{32}\) This suggests that the equations for the lattice parameters of \( t\text{-ZrO}_2 \) in the system \( \text{Y}_2\text{O}_3-\text{CeO}_2-\text{ZrO}_2 \), proposed by Urabe et al.,\(^{33}\) should be

\[
a = 0.5086 + 0.0003m_{\text{Y}} + 0.00033m_{\text{Ce}}
\]

\[
c = 0.5189 - 0.00026m_{\text{Y}} + 0.00028m_{\text{Ce}}
\]

from \( \Delta c = 0.013 \) \((r_{\text{Ce}^{4+}+} = 0.097 \text{ nm})\) and \( \Delta c = 0 \). The only significant discrepancy between Eqs. (6) and (7) and those determined by Urabe et al. is the coefficient of \( m_{\text{Y}} \) in Eq. (6), which was 0.0005.2

The calculated lattice parameters using Eqs. (1) and (2) are compared with the measured values in Table I. Considering the standard error (0.0003 nm) from the regression analyses in Eqs. (1) and (2) and the experimental error in the measurements, which ranges from 0.0001 to 0.00015 nm, the agreement between the calculated and measured lattice parameters is remarkable. Another good agreement can be found in the system \( \text{MgO-Y}_2\text{O}_3-\text{ZrO}_2 \), where the measured values for \( a \) and \( c \) of \( t\text{-ZrO}_2 \) were reported to be 0.5114 and 0.5138 nm, respectively, for \( M = 2.6 \) and \( M = 4.5 \) (i.e., \( m_{\text{Mg}} = 2.6 \) and \( m_{\text{M}} = 4.5 \)). These values are very close to the calculated \( a \) and \( c \) from Eqs. (1) and (2), which are 0.5108 and 0.5136 nm, respectively. These values for \( a \) and \( c \) are consistent with those of enhancing fracture toughness and \( t \rightarrow m \) transformation temperature. The trend of increasing the tetragonality due to alloying is consistent with those of enhancing fracture toughness and \( t \rightarrow m \) transformation temperature. The changes in the tetragonality due to the alloying are in remarkable agreement with the prediction by the proposed empirical equations.

IV. Conclusion

The addition of \( \text{Ta}_2\text{O}_5 \), \( \text{Nb}_2\text{O}_5 \), and \( \text{HfO}_2 \) to \( \text{Y}_2\text{O}_3 \)-stabilized tetragonal \( \text{ZrO}_2 \) (YTZP) gives rise to the high transformability of the resulting TZP. The enhanced transformability of YTZP is related to the alloying effect on the tetragonality of YTZP, so that the addition of these oxides increases the tetragonal distortion of the cubic fluorite lattice. The trend of increasing the tetragonality due to alloying is consistent with those of enhancing fracture toughness and \( t \rightarrow m \) transformation temperature. The changes in the tetragonality due to the alloying are in remarkable agreement with the prediction by the proposed empirical equations.

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