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The 1st
International Conference on Materials Engineering (ICME)
and
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FACULTY OF ENGINEERING UNIVERSITAS GADJAH MADA

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Opening Remarks by the Rector of Universitas Gadjah Mada

Bismillahirrahmaanirraahiim.
Assalaamu'alaikum warahmatullahi wa barakatuh.

Distinguished guests, Colleagues, Participants, Ladies and Gentlemen,

I would like to welcome all delegates of the First International Conference on Material Engineering (ICME) 2010 and the Third AUN/SEED-Net Regional Conference on Materials (RCM) 2010 to Universitas Gadjah Mada here in Yogyakarta. It is a great pleasure for me to see a large attendance to this meeting.

Please allow me to convey some details about this Conference. It is jointly organized by the Department of Mechanical and Industrial Engineering, Faculty of Engineering of UGM, ASEAN Foundation, ASEAN University Network/Southeast Asia Engineering Education Development Network (AUN/SEED-Net), JICA, and Universiti Sains Malaysia which are the host institution of the Material Engineering field. The International Conference on Materials Engineering (ICME) 2010 is the first event of subsequent series of conferences that we plan to run annually. The Conference is organized in conjunction with the Third AUN/SEED-Net Regional Conference on Materials (RCM) 2010.

The ICME 2010 and the RCM 2010 aim to encourage the sharing of experience and research results about aspects in Materials Engineering among academicians, scientists, researchers, industry practitioners and students. Those will greatly enhance each other's expertise, enabling them to take further steps in the development of this special science. The role of materials science is increasing in recent years. Indeed, this science is an area that is very promising to improve the welfare of human beings. It has the potential to contribute much for a better future of the world. It should serve, however, as a guide that ensures the concept of sustainable development.

To reach that goal, I believe that UGM will continue to work with scientists and industrial companies around the world whilst taking the initiative in leading research and innovative works in the field of materials. I believe UGM researchers, senior and junior, as well as students, will strive hard to help achieve welfare for the better civilization and the happiness of humankind.

To conclude, I would like to express my sincere and profound gratitude to all members of the Conference Committee, the Dean of Faculty of Engineering UGM, and everyone who have made this event a success. I wish you all have a productive gathering as well as a pleasant stay in Yogyakarta. Thank you.

Wassalaammu'alaikum wr. wb.

Prof. Ir. Sudjarwadi, M.Eng., Ph.D.
Rector of Universitas Gadjah Mada
Foreword from
the Chief Advisor JICA Project
for AUN/SEED-Net

It is a great honor for me once again to have an opportunity to welcome all of you to the 3rd AUN/SEED-Net Regional Conference on Materials Engineering, which is even more special this year as it is jointly organized with the 1st International Conference on Materials Engineering. On behalf of ASEAN University Network / Southeast Asia Engineering Education Development Network (AUN/SEED-Net), I would like to express my sincere gratitude to delegates from prestige Member and other Institutions, and all the people who have worked hard to organize this event, with my special thanks to the ASEAN Foundation, the main sponsor, and to Faculty of Engineering, Gadjah Mada University, the gracious and magnificent host, for successfully organizing this joint event, which will not only provide all participants with a greater chance to share research findings and trends, but also a greater chance to expand collaboration and networking among researchers in this area.

Since the start of the Project Phase I in 2003, more than 120 seminars/conferences have been organized and year by year we have been witnessing significant expansion of participants to non-member institutions inside and outside the region as well as industrial practitioners and government bodies, which indicates the very promising sustainability of this engineering network. And I believe that this network would empower researchers to tackle with ASEAN common socio-economic issues.

Today we have arrived at the middle of the Project's Phase II. So far we have been making efforts in engineering human resources development in ASEAN – which over 700 scholarships for higher degrees have been awarded, and more than half of them have already returned home to serve their roles to build the next generation of engineers. We have significantly enhanced the education and research capacity of member institutions through several types of research grant, and we have been facilitating research collaboration within ASEAN as well as between ASEAN and Japan so as to establish and strengthen this engineering network. And we promise to continue these important missions in a way that will respond to the common needs of the ASEAN region.

I strongly believe that the results of this 3rd AUN/SEED-Net Regional Conference on Materials Engineering will certainly have huge impacts and make path to future innovations and network expansion. Please let me wish all of you the valuable knowledge dissemination, the fruitful prolific discussion, as well as the wonderful stay in Yogyakarta. Let us together look forward to the smooth and successful conference.

Thank you very much.

Prof. Dr. TSUTSUMI Kazuo
Chief Advisor JICA Project for AUN/SEED-Net
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The Effect of Thermal Shock on Bending Strength of Zirconia Ceramics

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Abstract. This research aims to investigate the effect of thermal shock on bending strength of zirconia stabilized with 3 % Yttria called 3Y-TZP (Tetragonal Zirconia Polycrystal stabilized with 3 % mol Yttria). The zirconia powder with mean particle size of 0.2 - 2 μm was obtained from Goodfellow UK. Zirconia powder was uniaxially pressed with a pressure of 100 MPa to produce cylindrical green body with a diameter of 15 mm and thickness of 8 mm and green body specimens of rectangular cross-section bar (8x5x50 mm3). The cylindrical green body was pressureless sintered at various temperatures of 1250, 1300, 1350, 1400 and 1450 °C for 1 hour. The density of each sintered specimen was measured using Archimedes method. It was found that 1450 °C was the best sintering temperature. The rectangular green bodies were then pressureless sintered in a furnace at temperatures of 1450 °C for 1 hour with a heating rate of 5 °C/min. The sintered samples were polished. Group of the sintered samples were then heated up with a heating rate of 5 °C/minute to various temperatures of 25, 225, 275, 325 and 425 °C with a holding time of 1 hour. They were then thermal shocked by rapid cooling (quenching) to a water media with a temperature of 25 °C. The strength of samples was evaluated using four point bending test. The results show that the thermal shock resistance of the specimens is between 200 °C and 250 °C where the bending strength decreased drastically.

Keywords: thermal shock, zirconia, bending strength, crack.

1. INTRODUCTION

Ceramic materials usually have low brittleness and low thermal conductivity which leads to low resistance of thermal shock. When a ceramic material is quenched from high temperature, the rapidly cooled surface will want to contract, but it will be restrained from doing so by the bulk of the body, so stresses will develop called thermal stresses. If this thermal stresses (caused by such thermal gradient) large enough and greater than its strength, the ceramic will crack [1]. Other factors that can generate thermal stresses in materials are an-isotropy of atomic structure, volume change due to phase transformation and difference in thermal expansion coefficient of each constituent in composite materials [2].

Becher and Warwick [3] carried out research about thermal shock resistance on Al2O3-14%vol ZrO2. The specimens were thermal shocked with difference temperatures of 200, 300, 400, 500, 600, 700, 800, 900, 1000, 1100 and 1200 °C. The specimens were then quenched into a water media with temperature of 100 °C. The bending strength of specimens at ΔT = 0 °C was 700 MPa. This strength decreased severely to 400 MPa after thermal shocked at ΔT = 1000 °C. Wildan and Rusiyanto [4] studied about the fabrication of kaolin and thermal shock resistance (TSR) of kaolin. The thermal shock resistance was performed by heating the specimens and followed by quenching in water media. They found that found that the TSR of kaolin is in between 200 – 300 °C.

Kobayashi, et.al. [5] carried out a research on thermal shock of porcelain reinforced with alumina. They found that after thermal shock on specimen at ΔT = 30 °C and 140 °C, crack did not appear. However, at higher temperature difference of thermal shock at ΔT of 150 and 160 °C cracks developed.

Recommendation on water quench test procedures of thermal shock resistance of materials may refer to the method proposed by Lewis III [6]. Some of the recommendations are as follows:
- the water media should be cleaned and the container should be large enough
- the specimens should be in the form of circular rods, square bar or rectangular cross-section bars
- the quenching should be done as rapidly as possible
- bending test can be done using three-point or four-point bending test
- the specimens size should be as large as practical

If a bar of length is heated or cooled, it expands or contracts in proportion to the coefficient of thermal expansion, α, the length change Δ is given by [1] [7]:

ΔL = α L ΔT

(1)

If the bar is fixed at both ends, stresses are developed:

σ = E ε = E α ΔT

(2)

where E is Young's modulus and ε is the strain.
Thermal stresses can be induced by differential thermal expansion in multiphase materials, anisotropy in the thermal expansion coefficients of a single-phase solid or thermal gradient in a material [1].

Rapid heating or cooling of a ceramic will often result in failure, which is known as thermal shock. This occurs when thermal gradient and corresponding thermal stresses exceed the strength of ceramic. Thermal shock resistance (TSR) is the ability of a material to withstand from rapid heating or cooling. Thermal shock resistance can be evaluated by heating specimens to various temperatures $T_{\text{max}}$. The specimens are then rapidly cooled from $T_{\text{max}}$ to a medium (usually water with a temperature $T_{\text{amb}}$). The retained strength of the specimens is then measured. The typical relationship between the retained strength and $\Delta T = T_{\text{max}} - T_{\text{amb}}$, can be seen in Figure 1.

\[ U_{\text{st}} = U_o - U_{\text{strain}} + U_{\text{surf}} \]  (4)

Where $U_{\text{st}}$ is the energy of the stress- and crack-free crystal of volume $V_o$, $U_{\text{strain}}$ and $U_{\text{surf}}$ are strain energy and surface energy, respectively.

As it is assumed that the stress fields were non-interacting, in the presence of $N$ cracks, $U_{\text{st}}$ is equal to

\[ U_{\text{st}} = U_o + \frac{V_o}{2E} \left( \sigma_{\text{thermal}}^2 - N \sigma_{\text{normal}}^2 \right) + A \left( -\pi N \sigma_{\text{normal}}^3 - \frac{4}{3} \pi N \sigma_{\text{normal}}^3 \right) \]  (5)

Where $\sigma_{\text{thermal}}$ is thermal stress, $E$ is Young's Modulus and $v$ is Poisson’s ratio

Where the third term of Equation (5) represents the strain energy released by the existence of the cracks and the last term is the energy needed to extend them, $G_e$ is the toughness of material.

By differentiating the Equation (5) with respect to $\sigma_{\text{thermal}}$, then equating the resulting expression to zero, and rearranging terms, that for $\Delta T > \Delta T_s$, where $\Delta T_s$ is given by

\[ \Delta T_s \geq \sqrt{\frac{G_e (1-2v)^2}{\alpha^2 E c_i}} \]  (6)

The cracks will grow and release the strain energy. Conversely, for $\Delta T \leq \Delta T_s$, the strain energy that develops is sufficient to extend the crack, which in turn implies that the strength should remain unchanged.

The crack propagation in thermal shock is finite, where the crack will grow up to a certain length of $c_f$ that is commensurate with the strain energy available to them and then stop. To estimate the $c_f$, one simply equates the strain energy available to the system to the increase in surface energy, or

\[ \pi N c_i \left( \frac{c_f^2}{2} - \frac{c_i^2}{2} \right) = \frac{\alpha \Delta T_s^2}{2(1-2v)} \]  (7)

For short initial cracks, that is, $c_i > c_f$, substituting for the value of $\Delta T$ from Equation (6), one obtain ;

\[ c_f \leq \sqrt{\frac{1}{\alpha N c_i}} \]  (8)

which interestingly enough does not depend on any material parameters.

It is understood that solid ceramic materials usually contain contains pores (even at very fine pores size) which can act as micro-cracks. When there is thermal gradient due to thermal shock, these micro-cracks will develop at a certain length depend on the generated stress.
Inspecting Equation (6), it is not difficult to conclude that a good figure of merit for thermal shock resistance is

$$R_w = \text{(const)} \frac{\Delta T_c}{(\Delta T_c)^2} = (\text{const}) \frac{G_w}{\sqrt{a/E}} = \frac{K_{ic}}{\sqrt{a/E}}$$

(9)

From which it is clear that ceramics with low thermal expansion coefficients, low elastic modulus, but high fracture toughness should be resistant to thermal shock.

There is another parameter that is not included in the model, and which clearly must have an important effect on thermal shock resistance, is the thermal conductivity of the ceramic $k_{th}$. Given that thermal gradients are ultimately responsible for the buildup of stress, it stands to reason that a highly thermally conductive material would not develop large gradients and would thus be thermal shock resistant. For the same reason, the heat capacity and the heat-transfer coefficient between the solid and the environment must also play a role.

Moore [7] mentioned that thermal shock fracture parameter $R$ is constructed from Equation 2:

$$R = \frac{\Delta T}{E\alpha}$$

(10)

where $\sigma_f$ is the fracture strength

Because the rate of heat dissipation in the body is known to be important, a second parameter, $R'$ with incorporating thermal conductivity $k$, gives:

$$R' = \frac{\Delta T \cdot k}{E \alpha}$$

(11)

The stress $\sigma_f$ needs to be multiplied by $(1-\nu)$, where $\nu$ is Poisson’s ratio, to reflect the fact that the stress is biaxial, so the Equation 10 and 11 become:

$$R = \frac{\sigma_f (1-\nu)}{E \alpha}$$

(12)

$$R' = \frac{\Delta T \cdot k}{E \alpha}$$

(13)

Zirconia ($ZrO_2$) and zirconia-containing ceramics are used widely in traditional and engineering areas [8]. One of the important properties of ceramic materials is thermal shock resistance that indicates the ability of a material to withstand from failure due to rapid temperature changes. This research aims to investigate the effect of thermal shock on bending strength of 3Y-TZP (zirconia stabilized with 3 mol% Yttria).

2. MATERIALS AND RESEARCH METHOD

The material used in this research was zirconia stabilized with 3 mol% Yttria ($ZrO_2$) known as 3Y-TZP (Tetragonal Zirconia Polycrystal Stabilized with 3 %mol Yttria). The zirconia powder was obtained from Goodfellow, UK. It has mean particle size of 0.1 – 2 μm. The powder was uniaxially pressed with a pressure of 100 MPa to produce cylindrical green body with a diameter of 15 mm and thickness of 8 mm and green body specimens of rectangular cross-section bar (8x5x50 mm$^3$). The cylindrical green body was pressureless sintered at various temperatures of 1250, 1300, 1350, 1400 and 1450 °C for 1 hour. The density of each sintered specimens was measured using Archimedes method. The density of 1450 °C was the best sintering temperature. The rectangular green bodies were then pressureless sintered in a furnace at temperatures of 1450 °C for 1 hour with a heating rate of 5 °C/min. The sintered samples were polished. Group of the sintered samples were then heated up with a heating rate of 5 °C/minute to various temperatures of 25, 225, 275, 325 and 425 °C with a holding time of 1 hour. They were then thermal shocked by rapid cooling (quenching) to a water media with a temperature of 25 °C. The strength of samples was evaluated using four point bending test. Observation of the effect of thermal shock on crack propagation is also performed on pre-cracked specimen. The pre-crack is made using Vickers indentation where the cracks developed from the impression of the indentation corners.

3. RESULTS AND DISCUSSION

Density of sintered cylindrical samples was measured using Archimedes method. The sintering at various temperatures (of 1250, 1300, 1350, 1400 1450 °C) of the cylindrical samples was carried out in order to determine the sintering temperature for producing specimens for thermal shock test. The percentage of theoretical density (relative density) of specimens increases with increasing sintering temperature from 1250 °C to 1450 °C as shown in Figure 2. Almost fully dense samples were obtained on the specimens sintered at 1450 °C. Therefore sintering temperature of 1450 °C was selected for producing specimens for thermal shock tests. The higher density at higher sintering temperature indicates that the interaction among the particles during sintering process becomes better at higher temperatures.
Rectangular bar specimens were sintered at 1450 °C for 1 hour. Thermal shock was performed on the bar specimens by heating them up to a certain temperature and then they were quenched in water media at room temperature. The thermal shock test was performed at various temperature differences (ΔT) between the heating temperature and the water temperature. Bending strength of the quenched samples was evaluated using four point bending test. Figure 3 shows the bending strength of the specimens as a function of temperature difference (ΔT) between the heating temperature and the water temperature. It can be seen in Figure 3 that the strength of samples sintered at 1450 °C slightly decrease from 304 MPa (at ΔT = 0 °C) to 280 MPa (at ΔT = 200 °C), and then it decreases drastically to 36 MPa (at ΔT = 250 °C). The bending strength of specimens after performing thermal shock with temperature difference 250 °C does not change significantly. It means that the specimens have Thermal Shock Resistance (TSR) of the specimens is between 200 – 250 °C.

Ceramic materials usually have low brittleness and low thermal conductivity which leads to low resistance of thermal shock. When a ceramic material is quenched from high temperature, the rapidly cooled surface will want to contract, but will be restrained from doing so by the bulk of the body, so stresses will develop called thermal stresses. If these thermal stresses (caused by thermal gradient) large enough and greater than its strength, the ceramic will crack [1]. As shown in Figure 3, the bending strength of samples does not change significantly after it experienced thermal shock with temperature difference lower than 200 °C. The bending strength however, decreases drastically after thermal shock with temperature difference at ΔT 250 °C and above are applied. It is understood that the original specimen may already contains pores (even at very fine pores size) which can act as micro-cracks. When there is thermal gradient due to thermal shock, these micro-cracks will develop at a certain length depend on the generated stress. If the temperature difference less than 200 °C the stress that develops is insufficient to extend the micro-cracks significantly (still less than the critical length). The temperature difference (ΔT) 250 °C, however, causes the micro-cracks propagate significantly reaching the critical length and yielding the decrease in bending strength.

Those phenomena can be explained by observation of crack propagation on the specimens. A pre-crack is created on the surface of specimen using Vickers indenter until cracks developing from the corner of the impression of the indentation as shown in Figure 4a and 5a. The specimen (containing the pre-crack) was then thermal shocked at the various temperatures. It can be seen in Figure 4b that the pre-crack does not propagate significantly on the specimen after thermal shock at ΔT 200 °C. This corresponds with the bending strength of the specimen after it was thermal shocked at ΔT 200 °C where the bending strength is still high. However, the pre-crack propagates significantly on the specimens after experiencing thermal shock with a ΔT = 250 °C as shown in Figure 5b. This crack propagation is caused by thermal the generated stress and reduced the bending strength.
4. CONCLUSION
Conclusions withdrawn from this research as follows:

- The bending strength of zirconia 3Y-TZP sintered at 1450 is 304 ± 4 MPa. This bending strength does not change significantly after thermal shock treatment at ΔT 200°C.
- The bending strength of zirconia 3Y-TZP sintered at 1450 decreases drastically after they are rapidly cooled with temperature difference of thermal shock ΔT 250°C.
- The bending strength of zirconia 3Y-TZP sintered at 1450, after rapid cooled at ΔT = 250 °C is 36 ± 8 MPa.
- The thermal shock resistance (TSR) of the zirconia 3Y-TZP is between ΔT = 200 - 250 °C.
- The existing cracks will develop during thermal shock process due to thermal stress generation.

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REFERENCE

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