Mechanical Characteristics of Nanocrystalline (ZrO$_2$·20 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ Synthesized via Pulse Electric Current Consolidation of the Amorphous Powder

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The isothermal compressive forming is carried out in the cylindrical nanocrystalline tetragonal (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample, as prepared by consolidating the attrition milled amorphous powder in the absence of any additive, using the thermo-mechanical processing equipped with electric current heating, in order to provide a quantitative analysis for the superplastic flow inherent to the nanocrystalline ceramics. The nanocrystalline tetragonal (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample shows a large decrease in true plastic strain rate (r) up to a high compressibility of 0.75 with a decrease in true stress (σ) without void formation and grain growth at the temperature from 1440 to 1590 K. The strain rate sensitivity exponent (m) as defined by the relationship of the form, $m = \beta \ln \sigma / \ln r$ shows the constancy of 0.7 at a higher level of the strain rate compensated by temperature and reciprocal true stress. $\dot{e} = \sigma^{-1} \exp(Q/RT)$, following an increase from approximately 0.3 at a lower level of the parameter, for the superplastic flow in nanocrystalline (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$. The flexural strength of consolidated tetragonal (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ with the longitudinal crystallite size of approximately 40 nm is greatly enhanced by compressive forging relative to that of consolidated sample, and then characterized by the size effect with aspect ratio of sample width (W) to height (H) having nearly 2 GPa at the maximum in the case of $W/H = 1$. [doi:10.2320/matertrans.MRA2008152]

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Keywords: amorphous ceramic powder, pulse current nanocrystalline synthesis, high-speed superplastic flow, strain rate sensitivity exponent, high strength, size effect

1. Introduction

The bulk nanocrystalline synthesis is now becoming one of the most promising structure control methods for developing advanced ceramics with innovated mechanical properties necessary for the realization of next-generation technologies. The high pressure condensation of the nanoparticle obtained by gas deposition has been a well-known buildup processing of bulk nanocrystalline ceramics, but used to synthesize a porous and small mass product when the single nanometer sized grain is maintained.\(^{1,2}\) On the other hand, some novel powder metallurgy methods have been proposed to prepare large scale nanocrystalline ceramics. These processing include the pressure sintering of the amorphous ceramic powder prepared by alkoyx method,\(^{3}\) spray pyrolysis\(^{4}\) and mechanochemical synthesis.\(^{5,6}\) Especially, the electric field assisted consolidation of the mechanically synthesized amorphous powder\(^{7}\) is successfully employed to obtain a full-density nanocrystalline ceramic with a nearly threshold of approximately 10 nm for the average crystallite size in the absence of any additive.\(^{8}\) Thus-obtained nanocrystalline ceramics have exhibited excellent mechanical properties\(^{9}\) such as high-speed superplastic flow at a relatively low temperature and extremely high fracture toughness by compressive forging; these are expected to be a trigger to make a breakthrough in structural ceramics.

Until now, the superplastic compressive flow on heating has been conveniently used to manifest a relatively high strain rate of more than $1 \times 10^{-2}$ s$^{-1}$ at a low temperature of approximately 1400 K for consolidated nanocrystalline (ZrO$_2$·$\beta_60$(Al$_2$O$_3$)$_{20}$.\(^{10}\) Then, the superplastic extrusion has been performed to achieve a high-speed net-shape forming without void formation in monolithic nanocrystalline tetragonal (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$.\(^{11}\) While, in sub-micron meter grain sized and/or nanoparticle dispersed ceramics, one found it difficult to overcome a limited condition for the occurrence of the superplastic flow including considerably low strain rate and relatively high temperature, and accompanying grain coarsening and void formation.

We here are going to conduct the isothermal compressive forming and three-point bend testing for the nanocrystalline (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$, in order to characterize the superplastic flow and the high strength inherent to nanocrystalline ceramics through variables and parameters with clear physical justification.

2. Experimental Procedure

The amorphous (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ powder was prepared via solid state reaction of the elemental powder mixture using the instrumented rotating-arm ball mill equipped with the tank and arms of ZrO$_2$.\(^{12}\) This amorphous powder was packed into the graphite die, and then consolidated at 1390 K without any additive by employing the pulse electric current system under an applied stress of 150 MPa.\(^{11}\) The consolidated cylindrical (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample with 10 mm in diameter and 10 mm in height consists of the elliptical tetragonal grain in a range of the diameter from 20 to 40 nm, as estimated from X-ray line broadening method, up to the consolidation temperature of approximately 1560 K.\(^{13}\) The size and shape of the nanocrystallite in consolidated (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ has been confirmed by high resolution transmission electron microscopy as has reported in the previous papers.\(^{9,14}\) Such monolithic tetragonal (ZrO$_2$·3 mol% Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ synthesized via crystallization is an appropriate material by which to set up the phenomenology of the superplastic flow in nanocrystalline ceramics, even though remaining finely divided particles\(^{6}\) and suggested precipitates of Al$_2$O$_3$ may serve as a pinning effect by suppressing a grain growth that ought to occur at

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high temperatures. The mid-section and fracture surface of the forged sample were characterized by X-ray diffraction using Cu Kα radiation, and the crystallite size ($D$) was deduced using the Scherrer formula of $D = 0.94\lambda/(B\cos\theta)$, where $B$ is the X-ray line broadening at half-maximum, $\lambda$ is the X-ray wavelength (0.1542 nm for Cu Kα) and $\theta$ is the scattering angle.

The cylindrical sample of nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ was compressed under an initial applied stress ($\sigma_o$) of 115 MPa at a holding temperature from 1490 to 1590 K using the thermo-mechanical processing equipped with electric current heating and servo-controlled hydraulic pressing. The displacement ($Z$) is measured in real time, and is used to determine the compact height and hence its true strain ($\varepsilon$) according to the equation of $\varepsilon = \ln(1 + \varepsilon_o)$ where $\varepsilon_o$ is the nominal strain. Then, the true stress ($\sigma$) is obtained from the equation of $\sigma = \sigma_o(1 + \varepsilon_o)$. The true plastic strain rate ($\dot{\varepsilon}$) at a constant temperature is defined by a relation of the form:

$$\dot{\varepsilon} = \frac{de}{dt}$$  

where $t$ is the time. The die temperature during compression is measured via the thermocouple and is corrected through a temperature profile between the sample and graphite die to estimate the temperature ($T$) at the surface of the sample.

The fracture stress ($\sigma_f$) in the three-point bending test is given by

$$\sigma_f = \frac{3PL}{2WH^2}$$  

where $P$ is the applied load at fracture, $L$ is the distance between both fulcrums, $W$ and $H$ is the sample width and height, respectively. On the other hand, Young’s modulus ($E$) is deduced by

$$E = \frac{3PL}{2\varepsilon_bWH^2}$$  

where $\varepsilon_b$ is the elastic strain of the sample. The bend sample of forged nanocrystalline (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ was prepared with a height of 15 mm by mechanical cutting and polishing. In order to examine an effect of the sample geometry on the flexural strength of nanocrystalline ceramics, the width ($W$) of the bend sample varied in a range from approximately 1 to 3 mm. The rectangular sample with a sharp edge and shiny surface was tested in three-point bending at a room temperature under a cross head speed of 0.01 mm min$^{-1}$ using an Instron testing machine.

3. Results

3.1 Isothermal compressive forming

Figure 1 shows the displacement, the temperature at the surface of the sample and direct current versus time at an initial applied stress of 115 MPa. This measurement was taken during compression of the cylindrical nanocrystalline (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample. Nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ shows the smooth and monotonic plastic flow up to the displacement of 7.5 mm, avoiding the formation of any macroscopic cavity and void, under the dc of 2750 A at 1590 K, following a rapid displacement from approximately 1400 K on high-rate heating with approximately 8 K s$^{-1}$. Figure 2 shows the nanocrystalline (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample subjected to compressive forging at 1590 K and the cylindrical consolidated sample. This forged nanocrystalline ceramics has a relatively high value of 0.75 for the level of compressibility ($\phi$); its shape looks like a disk rather than a barrel, indicating a continuous free forming with a less frictional sliding as occurs in a ductile material. Besides, some small cracks at the surface of the disk occasionally occur along a loading axis due to lateral tension in a final forged stage of nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ without a monoclinic phase.

Figure 3 shows the true plastic strain rate ($\dot{\varepsilon}$) under an initial applied stress of 115 MPa for nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ at various temperatures of 1440, 1510 and 1590 K. The strain rate increases up to $3 \times 10^{-3}$ s$^{-1}$ at 1590 K on heating, and then greatly decreases at the holding temperatures for nanocrystalline (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ in compression in the case of the holding temperature of 1590 K. We see a concurrent decrease in strain rate with a decrease in true stress that is caused by an increasing cross-sectional area on the single nanocrystalline (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ specimen. Figure 5 shows the X-ray diffraction patterns of the nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample as forged at 1440, 1510 and 1590 K and as consolidated one. All forged tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ does not show any significant difference in X-ray peak intensity and position from as consolidated sample within the accuracy of this experiment; its crystallite size that is perpendicular to 111 plane of a tetragonal phase, as deduced by the line broad-
enlarging method, is approximately 40 nm; this value is equal to that of as consolidated sample. Therefore, an apparent grain elongation and growth could be suppressed during superplastic forming up to a relatively high compressibility of 0.75 at 1590 K for supersaturated nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ as synthesized via crystallization of the amorphous phase.

### 3.2 Three-point bending

Figure 6 shows the stress-strain curve in three point bending for the nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ sample forged with $\phi = 0.75$ at 1590 K having the width in range from 1 to 3 mm. The forged monolithic sample with the longitudinal crystallite size ($D_o$) of 40 nm shows a fracture in a brittle manner, and a flexural stress dependent on sample width having a high value of approximately 2 GPa at the maximum in the case of $W = 1.5$ mm. Table 1 summarizes the fracture stress, apparent Young’s modulus, dimension, volume and aspect ratio of bend sample for forged nanocrystalline tetragonal (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$, Young’s modulus is evaluated at 211 GPa for forged nanocrystalline (ZrO$_2$-3 mol%Y$_2$O$_3$)$_{80}$(Al$_2$O$_3$)$_{20}$ with the smallest width of 1.02 mm; this value is comparable to a commercial tetragonal zirconium oxide and lower than that of the (ZrO$_2$-2 mol%Y$_2$O$_3$)-20 vol%Al$_2$O$_3$
Mechanical Characteristics of Nanocrystalline (ZrO₂·20 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ Synthesized

Displacement arisen from a point contact of the spherical composite in a literature. Besides, we see a decrease in apparent Young’s modulus as increasing width, but this decrease may be caused by superimposing a transverse displacement arisen from a point contact of the spherical plunger. Table 2 summarizes the fracture stress in three-point bending for consolidated tetragonal (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ sample. As consolidated monolithic tetragonal nanocrystalline (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀, shows an increasing flexural strength with increasing sample volume within the experimental range of this study; this dependence is not accounted for by a fracture event as is governed by a weakest link model. It is noteworthy that the fracture strength can be greatly enhanced by compressive forming with φ = 0.75 as well as the fracture toughness as evaluated by the indentation microfracture method.

Figure 7 shows the nanocrystalline tetragonal (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ specimen fractured in three-point bending. This fractured sample is mainly composed of two fragments with the plane inclined to tensile axis, whereas conventionally processed ceramics commonly exhibits scattering into pieces. The fracture morphology of nanocrystalline tetragonal (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ may be coupled to a shearing crack extension accompanied by plastic flow. Figure 8 shows the X-ray diffraction pattern at the fractured surface of forged nanocrystalline (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀. X-ray diffraction confirms that forged nanocrystalline (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ at the fracture surface of bend sample remains a monolithic and tetragonal phase, showing the disappearance of a mechanically driven tetragonal-monoclinic phase transformation as occurred in a commercial partially stabilized zirconium oxide.

Table 2 The flexural stress, the dimension, volume and aspect ratio of the bend sample for consolidated tetragonal (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ with a longitudinal crystallite size of approximately 40 nm.

<table>
<thead>
<tr>
<th>No.</th>
<th>W, L, H (mm)</th>
<th>W·L·H (mm³)</th>
<th>Aspect ratio (W/H)</th>
<th>Fracture stress, σf (GPa)</th>
<th>Young’s modulus, E (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.28, 10.22, 1.68</td>
<td>90.6</td>
<td>3.14</td>
<td>0.847</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>5.27, 10.22, 1.80</td>
<td>97.0</td>
<td>2.92</td>
<td>0.727</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>5.24, 15.00, 4.00</td>
<td>314.4</td>
<td>1.31</td>
<td>1.010</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>5.25, 10.00, 4.70</td>
<td>246.8</td>
<td>1.11</td>
<td>1.216</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 5 X-ray diffraction patterns of the monolithic nanocrystalline (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ samples subjected to the superplastic forging at 1440, 1510 and 1590 K and as consolidated one at 1390 K in thermo-mechanical testing equipped with pulse current heating.

Fig. 6 The stress-strain curve in three-point bending for the forged tetragonal nanocrystalline (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ sample with the different width.

Table 1 The dimension, volume and aspect ratio of the bend sample, and the fracture stress and Young’s modulus in three point bending for monolithic forged tetragonal (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀.

<table>
<thead>
<tr>
<th>No.</th>
<th>W, L, H (mm)</th>
<th>W·L·H (mm³)</th>
<th>Aspect ratio (W/H)</th>
<th>Fracture stress, σf (GPa)</th>
<th>Young’s modulus, E (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>1.02, 10.0, 1.51</td>
<td>15.4</td>
<td>0.66</td>
<td>1.80</td>
<td>211</td>
</tr>
<tr>
<td>II</td>
<td>1.51, 10.0, 1.51</td>
<td>22.8</td>
<td>1.00</td>
<td>1.96</td>
<td>190</td>
</tr>
<tr>
<td>III</td>
<td>3.03, 10.0, 1.51</td>
<td>45.8</td>
<td>2.00</td>
<td>1.60</td>
<td>170</td>
</tr>
</tbody>
</table>

Fig. 7 The nanocrystalline tetragonal (ZrO₂·3 mol%Y₂O₃)₈₀(Al₂O₃)₂₀ sample fractured in three-point bending.
4. Discussion

4.1 An analysis of time dependent compressive displacement

Let us provide an analysis for the superplastic forming using the single compressive specimen. The superplastic flow is semi-empirically expressed by following equation:

\[
\dot{\varepsilon}^m = A \sigma^{(1/d)p} \exp(-Q/kT)
\]  
(4)

where \( A \) is the pre-exponential term, \( m \) is the strain rate sensitivity exponent, \( d \) is the grain size, \( p \) is the apparent inverse grain size exponent and \( Q \) is the apparent activation energy. The \( Q \) is related to \( mQ_m \) where \( Q_m \) is the activation energy at a certain level of the \( m \), and the \( p \) is given by \( p = \rho p_m \) where \( p_m \) is the reciprocal grain size exponent at each \( m \). Under the constancy of \( d \) and \( T \), the \( m \) is defined by

\[
m = \partial \ln \sigma / \partial \ln \dot{\varepsilon}
\]  
(5)

A logarithm of eq. (4) gives the relationship of the form,

\[
\ln(\dot{\varepsilon}^m/\sigma) = \ln A - p \ln d - Q/kT
\]  
(6)

Figure 9 shows the relationship between true strain rate and true stress in logarithmic at the testing temperatures of 1440, 1510 and 1590 K for nanocrystalline tetragonal (ZrO\(_2\)-3 mol\%Y\(_2\)O\(_3\))\(_{20}\)(Al\(_2\)O\(_3\))\(_{80}\). The \( m \) shows an increase from 0.2 to 0.7 as increasing strain rate and then has a shift to a higher strain rate by rising temperature. The obtained \( m \) value clearly shows that non-Newtonian flow occurs in nanocrystalline tetragonal (ZrO\(_2\)-3 mol\%Y\(_2\)O\(_3\))\(_{20}\)(Al\(_2\)O\(_3\))\(_{80}\) with the crystallite size of 40 nm, whereas Newtonian viscous flow underlies a powder consolidation of the nanocrystalline cubic zirconium oxide with the mean average crystallite size of 12 nm. Figure 10 shows \( \dot{\varepsilon}^m/\sigma \) at various \( m \) values against the reciprocal temperature for nanocrystalline tetragonal (ZrO\(_2\)-3 mol\%Y\(_2\)O\(_3\))\(_{20}\)(Al\(_2\)O\(_3\))\(_{80}\). In this plotting, the activation energy is deduced from the slope of the straight line in the case of each \( m \). Here, we see a straight line in this Arrhenius plot; its slope permits derivation of the value of 312 ± 40 kJ mol\(^{-1}\) for the level of the apparent activation energy. In other words, the constancy of the \( Q \) means that the \( Q_m \) is roughly in inverse proportion to the \( m \), and then estimated at 445 kJ mol\(^{-1}\) in the case of \( m = 0.7 \); this value is lower than approximately 600 kJ mol\(^{-1}\) for diffusion creep\(^{16}\) in the ZrO\(_2\)-Al\(_2\)O\(_3\) system.

Consider the process window and control for superplastic forming. From the constancy of the \( d \) and the \( Q \), the constant \( A \), related to a mechanism underlying superplastic flow, varies according to the relationship of the form, \( A = \dot{\varepsilon}^m \sigma^{-1} d^p \exp(Q/kT) \). In order to set up a relationship between the \( m \) and the \( \dot{\varepsilon} \), we take the true strain rate necessary for the occurrence of the superplastic flow at each level of the \( m \) ranging from 0.2 to 0.7 for the compressive superplastic flow in nanocrystalline (ZrO\(_2\)-3 mol\%Y\(_2\)O\(_3\))\(_{20}\)(Al\(_2\)O\(_3\))\(_{80}\) at different temperatures of 1440, 1510 and 1590 K as shown in
considerably large amorphous volume. In order to gain a comprehensive understanding on a mechanism underlying superplastic flow in nanocrystalline ceramics, there-fore is written by

\[ m = F(\dot{\varepsilon} \sigma^{-1} \exp(Q/kT)) \]  

4.2 Size effect on flexural strength

Figure 12 shows the flexural strength in three-point bending as a function of aspect ratio of the width to height for the forged nanocrystalline (ZrO$_2$·3 mol\%Y$_2$O$_3$)$_{(90\%)(Al_2O_3)_{20}}$ specimen with the compressibility of 0.75. This figure includes the result of the consolidated (ZrO$_2$·3 mol\%Y$_2$O$_3$)$_{(90\%)(Al_2O_3)_{20}}$ sample. We see a good correlation between the flexural strength and the parameter of $W/H$ for both forged and consolidated samples of tetragonal (ZrO$_2$·3 mol\%Y$_2$O$_3$)$_{(90\%)(Al_2O_3)_{20}}$ with the crystallite size of 40 nm. This relation between flexural strength and aspect ratio seems to be similar to a transition of the fracture toughness between plane stress and plane strain state for a highly strengthened alloy. The aspect ratio is now recognized as a useful dimensional parameter by which to integrate both effects of $W$ and $H$ on the strength of nanocrystalline ceramics instead of the sample volume.

5. Conclusions

The isothermal compression in thermo-mechanical processing provides a convenient method for analyzing the superplastic flow in nanocrystalline ceramics. For tetragonal (ZrO$_2$·3 mol\%Y$_2$O$_3$)$_{(90\%)(Al_2O_3)_{20}}$ with the crystallite size of...
40 nm, as synthesized without the additive, the strain rate sensitivity exponent is described by a master curve using the proposed parameter of temperature and reciprocal stress compensated strain rate, $\dot{\varepsilon} = \dot{\varepsilon}_0 \exp(Q/kT)$ with the proper value of the $Q$, with the constancy of 0.7 at its higher level. The flexural strength of monolithic nanocrystalline tetragonal $(\text{ZrO}_2\cdot3\%\text{Y}_2\text{O}_3)_{80}\cdot(\text{Al}_2\text{O}_3)_{20}$ is greatly enhanced by superplastic forging with the high compressibility of 0.75, and is characterized by the size effect with the aspect ratio of the sample, having the highest value of nearly 2 GPa at $W/H = 1$.

REFERENCES