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# Effect of 24 GHz microwave heating on creep deformation of yttria partially stabilised zirconia ceramics with titania and tin oxide additives

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## Abstract

The effect of quasi-millimetre-wave (24 GHz microwave) heating on creep deformation was investigated on partially-stabilised zirconia through tensile testing at temperatures from 1100 to 1350 °C over stresses from 3 to 10 MPa. The specimens were fabricated by sintering the 3 mol% of yttria stabilised $ZrO_2$ (3YSZ) powder dispersed with $SiO_2$, $SiO_2/SnO_2$ or $SiO_2/TiO_2$ at 1600 °C. In tensile test using the quasi-millimetre wave heating, strain rates of $SiO_2$ dispersed 3YSZ were similar to that with a conventional furnace heating. The 3YSZ sample containing the $TiO_2$ addition exhibited a different strain rate when heated by millimeter-wave irradiation as against conventional furnace heating, it is proposed that this difference can be explained due to $TiO_2$ having a high-dielectric constant resulting in a non-thermal effect when subjected to quasi-millimeter wavelength radiation.


*Keywords:* Zirconia; Creep deformation; Millimetre-wave heating; Tin dioxide; Titanium dioxide

## 1. Introduction

Zirconia ceramics are promising high-temperature structural materials because of their favourable mechanical properties such as good strength, heat resistance and resistance to corrosion at elevated temperatures. They are usually stabilised zirconia compositions in which the zirconium ions have been replaced by yttrium or calcium ions. Stabilised zirconia ceramics show superplasticity or high-temperature plastic deformation at high strain rates [1–5]. This superplasticity can be leveraged to enable zirconia ceramics to be processed similarly to sintered metals, thus providing a wide range of applications for these ceramics.

Various studies concerning the superplasticity of zirconia ceramics have been conducted, with a focus mainly on the strain limit and strain rate. Their superplastic properties can be improved by adding silica, which can increase the strain limit to as high as 1038% and render them easily processable [3].

However, these materials readily deform under load at relatively low temperatures. A preferred material can be easily deformed at lower temperatures but has good creep resistance at its higher operating temperature. In the present study, 24-GHz quasi-millimetre-wave (MMW) irradiation heating was used as a specific processing condition to achieve this desired performance.

MMW irradiation heating is a type of self-heating that uses the loss of energy occurring during penetration of the radiation into the ceramic body. Diffusion enhancement by MMW irradiation can enable rapid and low-temperature sintering for many materials [6–15]. Janney et al. reported an enhancement in oxide ion self-diffusion with the 28-GHz MMW irradiation of an alumina single crystal [16]. We also reported cation interdiffusion enhancement for a perovskite ceramics diffusion couple [17].

Diffusion of the atoms located at the grain boundaries governs the superplasticity because the main mechanism of ceramic

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superplasticity is grain boundary sliding. If the diffusion of some grain boundary-segregated atoms is faster under MMW heating than with conventional heating, then MMW heating could enhance the superplasticity of the material.

Tin oxide has a large dielectric loss at ∼24 GHz, resulting in a large absorption of electromagnetic energy [18]. Such dispersoids are expected to improve the superplastic (creep) deformation properties through local heating if segregated at the grain boundaries. In the present study, tin oxide was dispersed into silica-dispersed yttria-stabilised zirconia ceramics to investigate the effects of MMW irradiation on plastic deformation. Non-thermal effects are frequently invoked to explain some unusual phenomena observed under microwave (including MMW) irradiation [19,20]. Such non-thermal effects are proportional to the dielectric constant. In this work, dispersed titanium oxide was used to examine the effect of MMW irradiation on plastic deformation because it has a larger dielectric constant than tin oxide, yet has the same crystal structure.

## 2. Experimental procedure

### 2.1. Sample preparation

Yttria-stabilised zirconia (3 mol%; 3YSZ, TZ-3Y, Toso Co. Ltd., Tokyo, Japan) was used as the starting material for the main matrix powder. Colloidal silica (Snowtex-O, Nissan Chemical Co. Ltd., Tokyo, Japan) was used to facilitate plasticity [2]. Commercial tin oxide (guaranteed grade, Kojyundo Chemical Laboratory Co., Ltd., Tokyo, Japan) and titanium oxide (guaranteed grade, Fuji Titanium Industry Co., Ltd., Tokyo, Japan) were used as dispersoids. In the present study, 3YSZ without dispersoid, 20 mol % of $SiO_2$ added to 3YSZ (S-3YSZ), 14 mol% $SiO_2$–$SnO_2$ (10 mol% + 4 mol%, SS-3YSZ) co-dispersed and 14 mol% $SiO_2$–$TiO_2$ (10 mol% + 4 mol%, TS-3YSZ) co-dispersed ceramics were fabricated by a conventional sintering process.

The starting powders were weighed in predetermined ratios and then mixed in a planetary ball mill using zirconia balls and ethanol. After drying under an infrared lamp, the powder mixtures were crushed and ground in an alumina mortar. The powder mixtures were uniaxially pressed in a 34-mm-diameter steel die under 25 MPa of pressure, then sintered at 1600 °C for 2 h in ambient air. Dumbbell-shaped tensile specimens with an initial length of 12 mm were fabricated from the pressed pellets using an ultrasonic cutting machine, as shown in Fig. 1.

### 2.2. Characterisation

The apparent density was measured for each sintered specimen using the Archimedes method. Crystalline phases were identified by X-ray diffraction (XRD; Multiflex, Rigaku, Tokyo, Japan). Microscopic observations were carried out using scanning electron microscopy (SEM; model S-4300, Hitachi Co. Ltd., Tokyo, Japan) on cross-sectional surfaces following flash-annealing at 1600 °C for 10 min. Grain sizes were calculated from the SEM images using ImageJ freeware (National Institutes of Health, Bethesda, MD, USA). Energy-dispersive X-ray spectroscopy (EDX) was used to determine the atomic distribution relative to the microstructure.

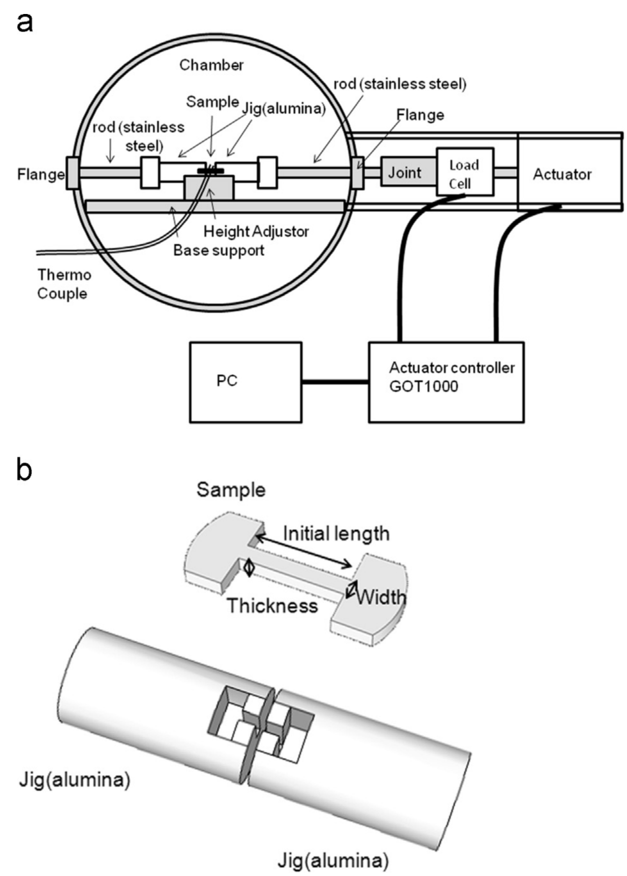


Fig. 1. Schematic illustration of sample set-up for tensile test under the MMW irradiation. Detail figures of sample and jigs are shown in Fig. 1(b).

Both the real ($\varepsilon'$ ) and the imaginary ($\varepsilon''$ ) parts of the complex dielectric constant (relative permittivity) were measured over a frequency range of 18–26.5 GHz (K-band) using a network analyser (model 8510C, Agilent Technologies, Santa Clara, CA, USA). The measurement was conducted at approximately room temperature using the WR42 waveguide method.

### 2.3. Tensile test

A conventional tensile creep test was conducted under 3–10 MPa of pressure over a temperature range of 1100–1350 °C using a universal testing machine (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) equipped with an image furnace. Tensile testing under MMW irradiation was conducted under 3–10 MPa of pressure at temperatures below 1300 °C using a tensile machine (EDA-1kN100-S, Daiichi Denki Co., Fujisawa, Kanagawa, Japan) attached to the MMW irradiation chamber (24-3GS, MSP Ltd., Osaka, Japan; Fig. 1).

The tensile strain rate data $\varepsilon^{\bullet}()$ from both tensile tests, which used different heating methods, was used to estimate the activation energy $Q$ for creep deformation according to the following equtions.:

$$\text{Activation energy } \dot{\varepsilon} = A\frac{\sigma^n}{d^p}\exp\left(-\frac{Q}{RT}\right) \quad (1)$$