ULTRA-HIGH TEMPERATURE CREEP BEHAVIOR FOR IN-SITU SINGLE CRYSTAL Al_2O_3/YAG OXIDE CERAMIC EUTECTIC COMPOSITES

Y. Harada, T. Suzuki and K. Hirano

Institute of Mechanical Systems Engineering, National Institute of Advanced Industrial Science and Technology, AIST Tsukuba East, Namiki 1-2, Tsukuba, Ibaraki 305-8564, Japan

ABSTRACT

Creep behavior was undertaken for in-situ single crystal Al_2O_3/YAG oxide ceramic eutectic composites. Creep tests were conducted in the ultra-high temperature ranging from 1773 to 1873 K under a constant tensile stress ranging from 90 to 160 MPa in air and moisture environments under the water vapor pressure range from 0.06 to 0.6 MPa. Al_2O_3/YAG eutectic composites exhibited a stress exponent of 8-13. The tensile creep rate in air was about an order of magnitude higher than the compressive creep rate in argon at the same temperature. The presence of moisture enhanced the creep strain and the creep rate significantly. The creep rates increased with increasing the water vapor pressure up to 0.45MPa.

KEYWORDS

Single Crystal Oxide, Eutectic Composites, Ultra-high Temperature, High Pressure, Moisture, Creep Deformation

INTRODUCTION

Advanced ceramics such as SiC/SiC, Si_3N_4 and oxides are expected to be candidates for the ultra-high temperature structural materials in the field of power generation industries and aerospace industries because of their low density, high melting point, good oxidation resistance and high temperature strength. However, at high temperatures of 1477 K, it has been recently reported the degradation of SiC or Si_3N_4 in high pressure, moisture-rich environments (0.15 MPa of water vapor carried in air environment at 1.0 MPa of total pressure) [1-3]. Also, poly-crystal Al_2O_3 tested in water vapor revealed significant changes in weight, volume, microstructure and strength by grain boundary etching and grain growth [4]. These studies agree that the presence of water vapor accelerates the corrosion process.

In-situ single crystal oxide ceramic eutectic composites such as Al_2O_3/$Y_3Al_5O_{12}$(YAG), Al_2O_3/GdAlO$_3$ (GAP) and Al_2O_3/$Er_3Al_3O_{12}$(EAG), have newly been investigated and developed [5-9]. Al_2O_3/YAG eutectic composite has new microstructure in which single crystal Al_2O_3 and YAG are three-dimentionally and continuously connected. This composite has thermally stable at 1973K in air, excellent flexural strength and compressive creep resistance [5,6]. However, limited investigations have been conducted on the mechanical properties for Al_2O_3/YAG eutectic composite at ultra-high temperature. Furthermore, in a search of the

literature no reference was found to work investigating only the effect of water vapor on long-term mechanical properties for Al_2O_3/YAG eutectic composite.

Recently, our laboratory has been the first to construct to conduct a material test in simulated severe environments such as high temperature, high pressure or moisture environment [9-11]. This equipment permits mechanical tests at ultra-high temperature up to 1973 K, high pressure as high as 0.98 MPa and air, moisture, O_2 or N_2 environment. In this study, in order to ensure the long-term durability for Al_2O_3/YAG eutectic composite, tensile creep behavior at ultra-high temperature, high pressure, moisture environments was conducted by using the developed materials testing system in simulated severe environments.

EXPERIMENTAL PROCEDURES

Materials

The material investigated in this paper was the Al_2O_3/$Y_3Al_5O_{12}$ (YAG) in-situ single crystal oxide ceramic eutectic composite. Commercial α-Al_2O_3 powder (99.99 %) and Y_2O_3 (99.999 %) powder were pre-melted to obtain an ingot, and then the ingot was placed in a molybdenum crucible. It was fabricated by the unidirectional solidification at a melting temperature of 2173 K by lowering the molybdenum crucible at a speed of 5 mm/h [5,6]. The composition of the binary eutectic composite was Al_2O_3/YAG=67/33 wt.%. Figure 1 shows the microstructure for Al_2O_3/YAG eutectic composite. The microstructure consists of a fine lamellar structure and a three-dimensional network structure containing single crystal YAG phase (bright phase) distributed in a matrix of single crystal Al_2O_3 (dark phase). The homogeneous microstructure exists no pores or colonies in the eutectic composite.

20μ m
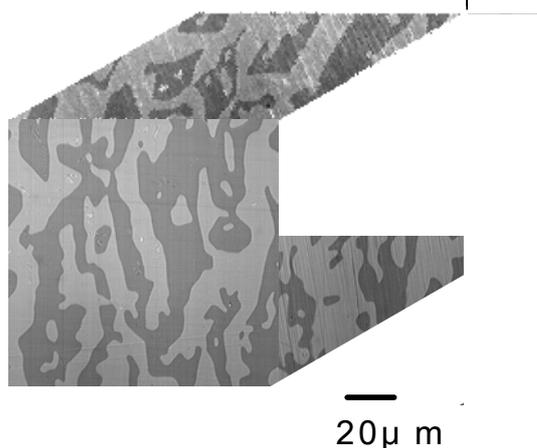
Figure 1: Microstructure for Al_2O_3/YAG eutectic composite

Creep test

Tensile creep specimens having a 2.5 mm diameter and 10 mm gage-length were machined with their axis parallel to the solidified direction. In the case of the tensile creep test at 1873K, the flat, dog-bone shaped specimen having a 3×2 mm cross-section and 10 mm gage-length was machined. The overall length of all specimens was 65 mm. After machined, all specimens were homogenized at 1873K for 1 hour in air.

Tensile creep tests were undertaken by using a developed materials testing system in simulated severe environments [9-11]. By using this developed material testing system, tensile creep tests were conducted at 1773 K or 1873 K in air and 1773 K in moisture environments. The test temperature was measured by thermocouples located in the near vicinity of the specimen gage length, which was maintained within 1 K of the set point throughout each test. The constant load was controlled by a closed loop servomotor system. The displacement during creep test was measured by the extensometer using a linear variable differential transducer. Creep strain was measured by assuming that deformation occurred only in the gage section. The strain increment was calculated from the displacement after the deformation of the initial loading. Stress dependence on the creep rate was examined by stepwise loading after a creep curve showed the secondary creep behavior at a lower stress, because of the limited supply of specimens.

In addition, crept specimens were characterized by scanning laser microscopy, scanning electron microscopy

(SEM) and Energy dispersive X-ray spectroscopy (EDS).

RESULTS AND DISCUSSION

Creep curve
Tensile creep tests were conducted at 1773 K and 1873 K in air environment with the applied stresses ranging from 100 to 160 MPa. Figure 2 shows the relationships between the strain increment, $\Delta\varepsilon$, and time for tensile creep of Al_2O_3/YAG eutectic composite at 1773 K and 1873 K in air environment. All creep curves under constant stresses have essentially the same shape and can be divided into primary and secondary creep region. In the initial part of the curve, the creep rate decreases with increasing strain and reached a minimum forming a brief quasi-steady state condition. At the temperature of 1773 K under the stress of 160 MPa, failure occurred in the transient creep region.
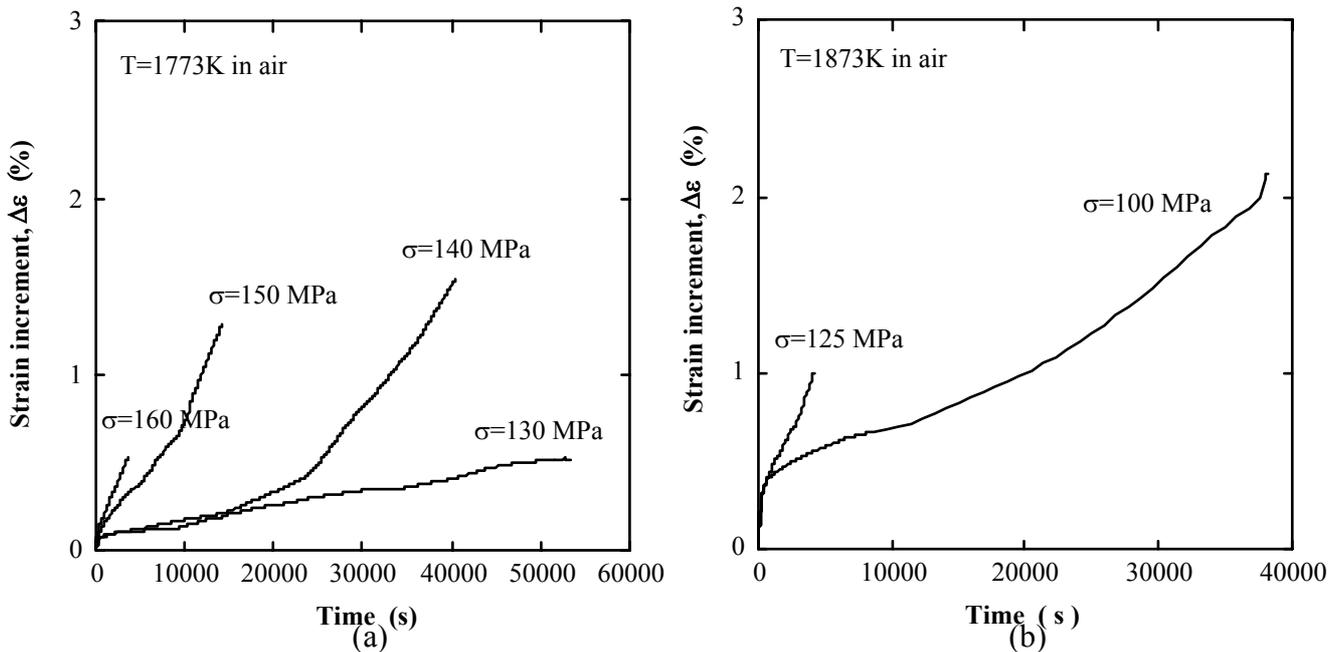
Figure 2: Strain increment as a function of time for Al_2O_3/YAG eutectic composite, (a) 1773 K and (b) 1873 K in air environment
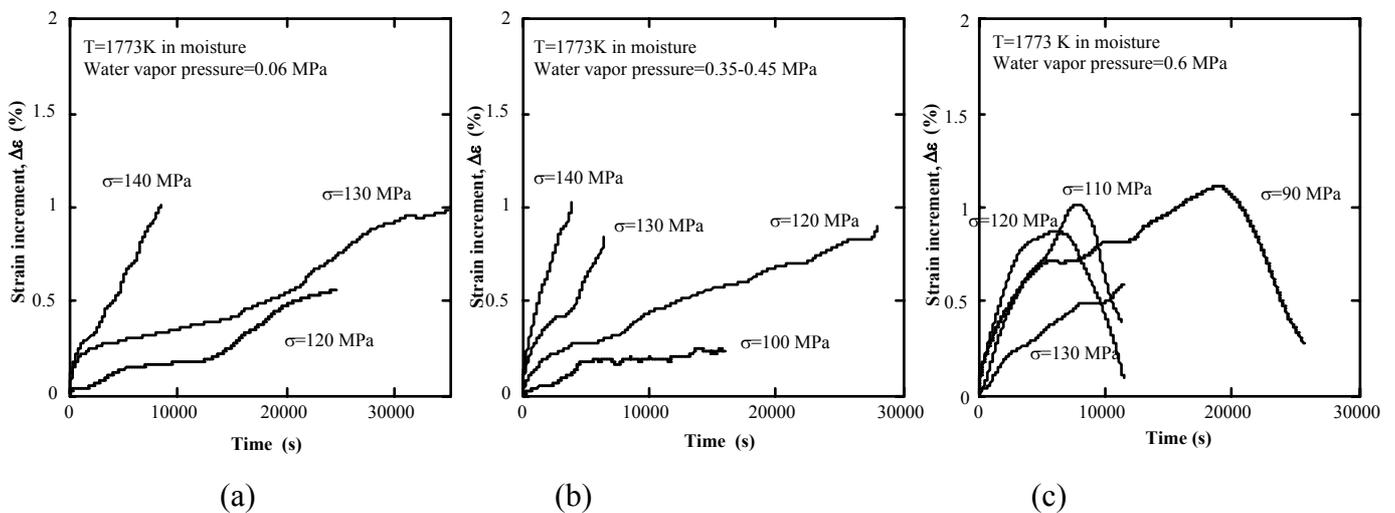
Figure 3: Strain increment as a function of time for Al_2O_3/YAG eutectic composites at 1773 K in moisture environments, under the water vapor pressure of (a) 0.06 MPa, (b) 0.35-0.45 MPa and (c) 0.6 MPa

Tensile creep tests were conducted at 1773 K in moisture environments with applied water vapor pressure ranging from 0.06 to 0.6 MPa. The applied stress was ranging from 90 to 140 MPa. Figure 3 shows the

relationships between the strain increment $\Delta\varepsilon$ and time for tensile creep of Al_2O_3/YAG eutectic composite at 1773 K in moisture environments. Creep curves under the water vapor pressure of 0.06 MPa and 0.35-0.45 MPa have the deformation in primary creep and a stable secondary creep as well as Figure 2. On the other hand, in the case of the water vapor pressure of 0.6 MPa, it can be seen that after the deformation in primary creep have reached at a maximum strain, the specimen begins to contract. This phenomenon defined as short-term negative creep [12,13]. However, under the stress of 130 MPa, as applied load is increased the negative creep is reduced, even disappeared, and failure occurs in the transient creep region. The more details are under investigation.

Stress dependency of the creep rate

The steady-state creep region stated here is the region where the creep curve is a straight line after the leveling treatment. Figure 4 shows the stress dependence of the tensile creep rates for Al_2O_3/YAG eutectic composite at 1773 K and 1873 K in air environment. These creep rates increase linearly with increasing applied stresses on the logarithmic scales. Figure 4 illustrates that the stress exponent is around 8-13 and decreases with increasing temperatures (from n=13 at 1773 K to n=8 at 1873 K). It is assumed to consider a dislocation backstress argument to explain the high stress exponents observed. One possible source for this backstress is an Orowan pile-up and bowing mechanism occurring in the single crystal Al_2O_3 matrix due to the presence of the more creep resistant single crystal YAG phase or the presence of the interphase boundary etching. The more details are under investigation.
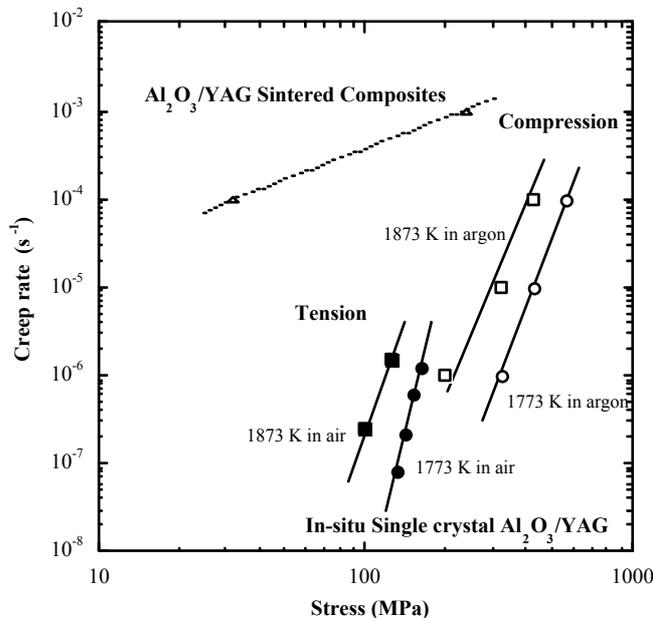
Figure 4: Stress dependence of the tensile creep rate for Al_2O_3/YAG eutectic composite at 1773 K and 1873 K in air environment, compared with the compressive creep rates for Al_2O_3/YAG eutectic composite and sintered composite at 1773 K and 1873 K in argon environment

Figure 4 also shows the stress dependence of the compressive creep rates for Al_2O_3/YAG eutectic composite and the sintered composite shared the same composition and constitutional phase at 1773 K and 1873 K in argon gas reported by Waku [5]. The compressive creep rates in argon environment decreases by approximately an order of magnitude compared with the tensile creep rates in air environment at each temperature. The stress exponent varies between 5 and 6. It was reported the compressive deformation mechanism followed the dislocation creep model as observed dislocations in crept specimens [5]. While, for the sintered composite, the compressive creep rate in argon environment is approximately 2.5 orders of magnitude higher than the tensile creep rates or 3 orders of magnitude higher than the compressive creep rates in argon environment for Al_2O_3/YAG eutectic composite. The exponent is close to 1. It is assumed that the creep deformation mechanism followed the diffusional creep such as Nabarro-Herring or Coble creep models.

Effects of moisture environments on creep rate
Figure 5 shows the stress dependence of the tensile creep rate for Al_2O_3/YAG eutectic composite at 1773K in moisture environments without the water vapor pressure of 0.6 MPa. This figure also shows the results for Al_2O_3/YAG eutectic composite at 1773 K and 1873 K in air environment. These creep rates increase linearly with increasing the applied stress on logarithmic scales. The creep rates in moisture environment are larger than that at 1773 K and smaller than that at 1873 K in air environment. Also they increase with increasing the water vapor pressure up to 0.45 MPa. The stress exponent is around 9.4-10 and slightly decreases with increasing the water vapor pressure.
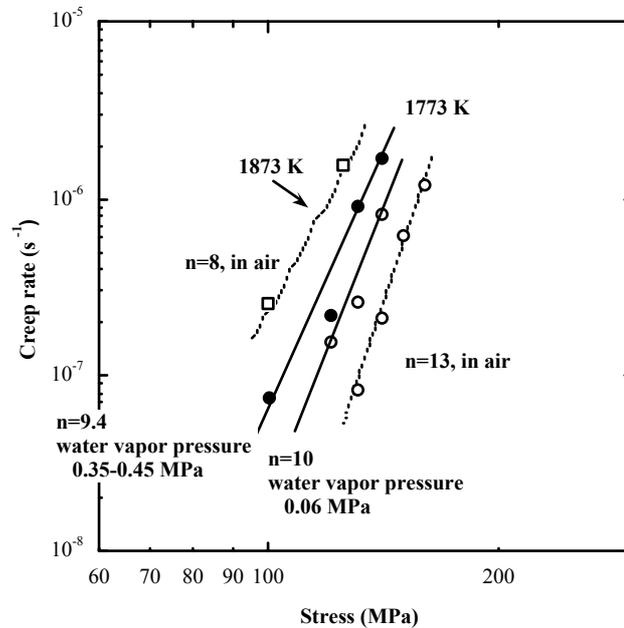
Figure 5: Stress dependence of the tensile creep rate for Al_2O_3/YAG eutectic composite
in air and moisture environments

It was widely recognized that metals, alloys and intermetallics can be severely embrittled by moisture. Impurities in moisture can dissolve in most materials and affect their properties. For instance, the $Ni_3(Si, Ti)$ alloys exhibited substantially reduced tensile ductility and intergranular fracture when tested in air or hydrogen containing environments [14]. In contrast, hydrogen introduced in palladium enhances its deformation by promoting diffusional creep [15]. In the case of single crystal ceramics, it is reported that the presence of water enhanced weakening of sapphire with introduction of water defect as a result of enhanced dislocation mobility [16].

Taking into account these considerations, the accelerating of creep rate in moisture-induced environments may be due to molecular water, hydroxide or hydrogen defect in moisture in crept in Al_2O_3/YAG eutectic composites. These defects could be introduced in the bulk, by complex transport processes (pipe diffusion, solute drag, etc.) and enhance dislocation mobility. An alternative explanation for the accelerating of creep rate in moisture-induced environments may be based on the role of the Peierls potential in controlling dislocation glide and the influence of interstitial hydrogen in moisture which introduces in the specimen. The stress for the deformation is directly due to the nucleation and motion of kink pairs on dislocation. If either kink nucleation or kink motion is enhanced by the presence of interstitial hydrogen, the accelerating creep could be explained. However, the exact mechanism responsible for this accelerating creep in moisture-rich environment needs await precise observations of dislocations in crept specimens.

CONCLUSION

The tensile creep behavior for in-situ single crystal Al_2O_3/YAG eutectic composite was studied in the temperature range 1773-1873 K and the stress range 90-160 MPa in air or in moisture environments under the water vapor pressure of 0.06-0.6 MPa by using the materials testing system in simulated severe environments. The following conclusions can be drawn:

(1) Al_2O_3/YAG eutectic composite exhibited the same shape creep curve and could be divided into primary and secondary creep region. The tensile creep rates in air environment increased by about an order of magnitude compared with the compressive creep rates in argon environment. The stress exponent was 8-13, and it was assumed to consider a dislocation backstress argument.

(2) It was found that the presence of moisture enhanced the creep strain and the creep rate of Al_2O_3/YAG eutectic composites significantly. The creep rates increased with increasing the water vapor pressure up to 0.45 MPa. On the oher hand, in the case of water vapor pressure of 0.6 MPa, it was seen the short-term negative creep. As applied load was increased the negative creep was reduced, even disappeared.

REFERENCES

1. Ferber, M.K., Lin, H.T., Parthasarathy, V. and Brentnall, W. (1999) *ASME papers* 99-GT-265.
2. More, K.L., Tortorelli, P.F., Ferber, M.K. and Keiser, J.R. (2000) *J. Am. Ceram. Soc.* 83, 211.
3. More, K.L., Tortorelli, P.F., Ferber, M.K., Walker, L.R., Keiser, J.R., Miriyala, N., Brentnall, W.D. and Price, J.R. (2000) *J. Eng. Gas Turb. Power.* 122, 212.
4. Tai, W.P., Watanabe, T. and Jacobson, N.S. (1999) *J. Am. Ceram. Soc.* 82, 245.
5. Waku, Y., Nakagawa, N., Ohtsubo, H., Ohsora, Y. and Kohtoku, Y. (1995) *J. Jpn Inst. Met.* 59, 71.
6. Waku, Y. and Sakuma, T. (2000) *J. Eur. Ceram. Soc.* 20, 1453.
7. Waku, Y. (2000) *J. Jpn Inst. Met.* 64, 977.
8. Waku, Y. (2000) *J. Jpn Inst. Met.* 64, 101.
9. Hirano, K., Suzuki, T., Kamei, A. and Tamai, F. (2001) *Inter. J. Mater. Prod. Tech.* 16, 276.
10. Suzuki, T., Harada, Y., Hirano, K. and Waku, Y. (2001) *Proc. 7th International Conference on Creep and Fatigue at Elevated Temperatures,* in print.
11. Harada, Y., Suzuki, T., Hirano, K. and Waku, Y. *J. Am. Ceram. Soc.,* to be submitted.
12. Liu, X.C. and Bathias, C. (1994) *J. Mater. Sci.* 29, 4618.
13. Ren, D.G. (1998) *J. Mater. Process. Tech.* 73, 74.
14. Takasugi, T., Liu, C.T., Heatherly, L., Lee, E.H. and George, E.P. (1999) *Intermetallics* 7, 543.
15. Xu, Z.R. and Mclellan, R.B. (1998) *Acta Mater.* 46, 4543.
16. Castaing, J., Kronenberg, A.K., Kirby, S.H. and Mitchell, T.E. (2000) *Acta Mater.* 48, 1495.