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Journal of the European Ceramic Society 25 (2005) 1191–1199

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# Application of eutectic composites to gas turbine system and fundamental fracture properties up to 1700 °C

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Available online 8 March 2005

**Abstract**

Single crystal eutectic composites have recently been researched and developed as the bulk materials. They are generally expected to be the most interesting and attractive as the high temperature, environmental resistant structural material in the field of aeronautics, aerospace and power generator technologies. They have high temperature strength characteristics, high creep and oxidation resistance as well as the fairly good machinability and manufacturability. An overview provides explain the National project of application of eutectic composites for 1700 °C-class gas turbine system that does not require thermal and environmental barrier coatings. Pure mode-I and -II, mixed-mode fracture toughness are investigated up to 1700 °C in air to determine materials reliability and also discussed fatigue crack growth characteristics in relation to materials durability. Key technical issues are summarized with prospects for a wide practical application.
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*Keywords:* Composites; Engine components; Fracture; Toughness and toughening; Technical issues for application

## 1. Introduction

Recently, single crystal eutectic composites have been researched and developed as the bulk materials[1,2] as well as the reinforcement fibers.[3] They are generally expected to be the most interesting and attractive as the high temperature, environmental resistant structural material in the field of aeronautics, aerospace and power generator technologies. They have many potentialities that the flexural and tensile strength are maintained right below the melting point temperature mainly due to fine phases interfaces, increases with decreasing of a characteristic dimension of the network microstructure without the reduction of creep resistance and plastically deformed over approximately 1550 °C. They also show a fairly good machinability and manufacturability of complex shape structural components in comparison with conventional sintered engineering ceramics. However, low fracture toughness and thermal shock resistance originally resulted from the oxide/oxide composite are critical technical issues for a practical application. It is still necessary for the further research to search new eutectic composites and to improve the materials reliability and durability. Binary to ternary eutectic composite is considered to be one of the possibilities to improve materials performances.

This paper briefly explains the National project of application of eutectic composites for 1700 °C-class gas turbine system[4] that does not require thermal and environmental barrier coatings (TBC and EBC). Pure mode-I and -II, mixed-mode (I + II) fracture toughness are determined to assess the mixed-mode fracture criterion up to 1700 °C in air. Fatigue crack growth characteristics are also presented in relation to materials durability. Some technical key issues are summarized with prospects for a wide practical application.

## 2. Application to gas turbine system

From FY1998 to FY2000, feasibility studies[5] had already finished on (1) search for new MGC (Melt Growth Composite was firstly named by Prof. K. Suzuki, former director general of Institute of Materials Science at Tohoku University

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doi:10.1016/j.jeurceramsoc.2005.01.003

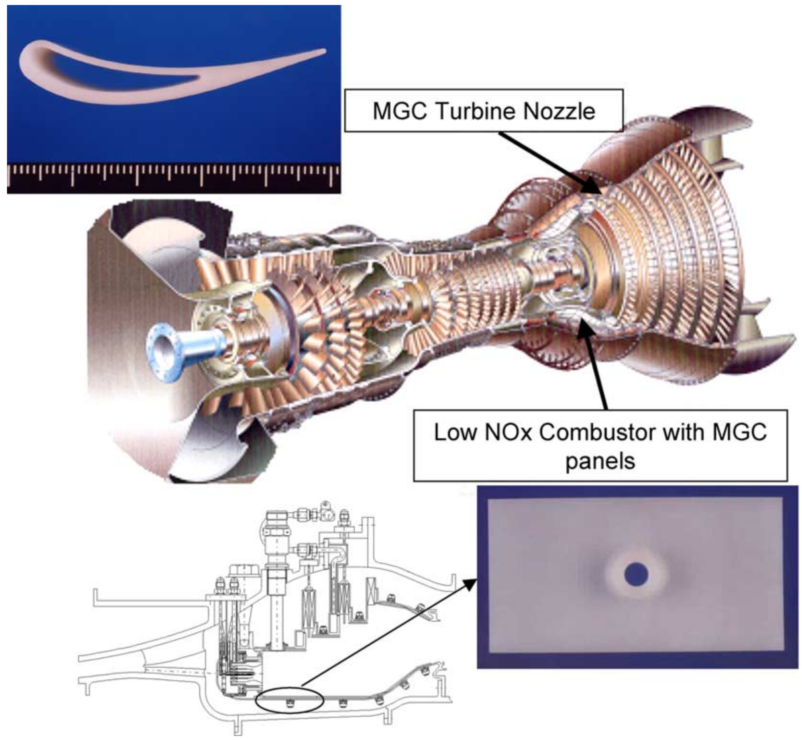


Fig. 1. Research targets components for gas turbine.[4]

and fundamentally almost the same to single crystal eutectic composite) and improvements for materials performance, (2) possibility of low-cost processing and manufacturing technologies for large complex, near-net shaped components, (3) aero-mechanical design methodology for gas turbine components based on computer fluid dynamic (CFD) and (4) preliminary turbine cycle analysis and system integration technology. Then, five-years national project, that is, NEDO Project on MGC Applied Gas Turbine System had been started under the sponsorships of Ministry of Economic, Trade and Industries (METI) from FY2001.[4]

Final targets of this research project are summarized as follows: (1) gas turbine output power of 5000 kW-class, (2) overall pressure ratio of 30, (3) turbine inlet temperature (TIT) of 1700 °C, (4) non-cooled MGC turbine nozzle, (5) lower $NO_x$ emission than 25 ppm (15% $O_2$) and (6) MGC combustor liner panel (see Fig. 1). In order to realize these research targets, it focuses on two major R&D themes, first is, gas turbine system integration technology.

- Total MGC gas turbine system and cycle analysis
- MGC turbine nozzle and vane
- Low $NO_x$ emission combustor with MGC liner panels

Second is innovative, breakthrough processing and manufacturing technology.

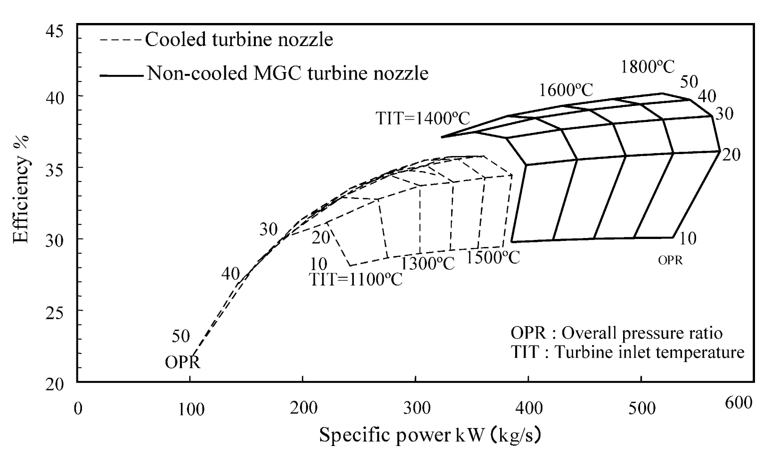


Fig. 2. Gas turbine performance curve as a function of specific power.[6]

- Near-net shape casting of complex shape components
- Improvements of materials reliability and durability under simulated severe environments (highly water vapor pressurized at high temperatures)

MGC applied turbine nozzle and combustor have preliminary been designed and determined the overall structure of the total gas turbine system. On the assumption that thermal barrier coating/environmental barrier coating (TBC/EBC) coated metal-based turbine blade can be available, it has been confirmed the possibility of the realization of the 1700 °C-class MGC gas turbine system. Turbine cycle analysis has also been done. The gas turbine performance curve is shown in Fig. 2 as a function of specific power. It can be seen from this figure that compared to current gas turbine for electric power, the possibility of approximately maximum 9% efficiency improvement can be prospected by applying MGC materials to non-cooled turbine nozzle at TIT of 1700 °C and overall pressure ratio of 30. [6]

### 2.1. Developments of MGC turbine nozzle and vane

The three-pieces separated, non-uniform-thickness hollow-type turbine nozzle was designed and manufactured by ultrasonic machining from a round-bar $Al_2O_3$/GAP eutectic composite as shown in Fig. 3.[7] The turbine nozzle firstly was examined in hot gas flow test at a maximum temperature level of 1400 °C. Under the steady-state, maximum thermal stress generated at pressure side was estimated from temperature distribution, and successfully reduced at approximately 0.431 to ultimate materials strength. In this hot gas flow test, the structural integrity of the separated turbine nozzle was also ensured during heating and cooling conditions. The 1700 °C-level nozzle rig has been improved to continuously measure the temperature distributions on the nozzle surface by using infrared cameras. It has just planned to conduct the test at the inlet gas temperature of 1700 °C level in order to ensure the structural integrity under the steady state and thermal cycle conditions.

### 2.2. Developments of low $NO_x$ emission combustor with MGC panels

MGC panel attachments to combustor liner were designed. The structural integrity was first verified on the basis of transient thermal stresses analysis. It has also been determined the staging combustor configuration for low $NO_x$ emission on the basis of numerical fuel flow analysis. These computer simulations suggest that the axial swirling method was prospected to be lower pressure-loss and better mixing in air and fuel flows rather than the radial swirling. Combustion tests at a level of 1400 °C had already carried out by using the model combustor (see Fig. 4).[7] It is confirmed that the fuel nozzle achieved to low $NO_x$ emission level of 8 ppm (15% $O_2$). Now, it has planned to continue the combustion tests up to 1700 °C level.

### 2.3. Innovative, breakthrough processing and manufacturing technology

The most important to control the microstructures for eutectic composites is temperature gradient in the heating and the melting zones, in particular at the solid–liquid interface during the process. A large Bridgman-type furnace was newly designed and developed to accurately control many materials process parameters. Using this new large equipment, the temperature of both the melting zone and the solidified zone are independently controlled, and successfully kept at a constant the temperature gradient at the solid–liquid interface by heat insulator.

The near-net shape casting methodology has been researched and developed. An example is shown in Fig. 5 for the casting mold and core for turbine nozzle.[7] Molybdenum was chosen as the most suitable material for casting mold

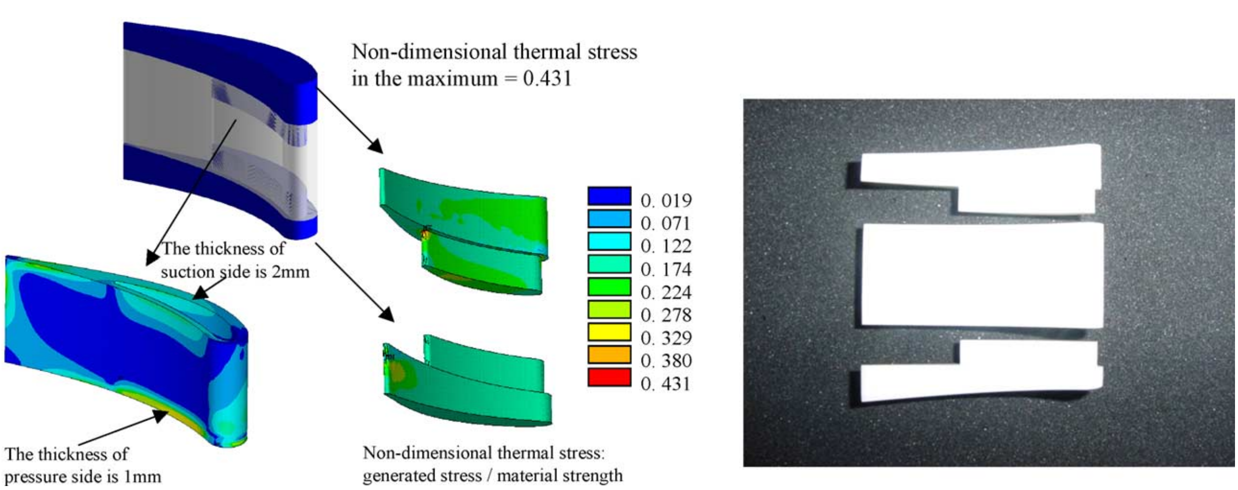


Fig. 3. Reduction of thermal stress by the separated hollow-type turbine nozzle.[7]

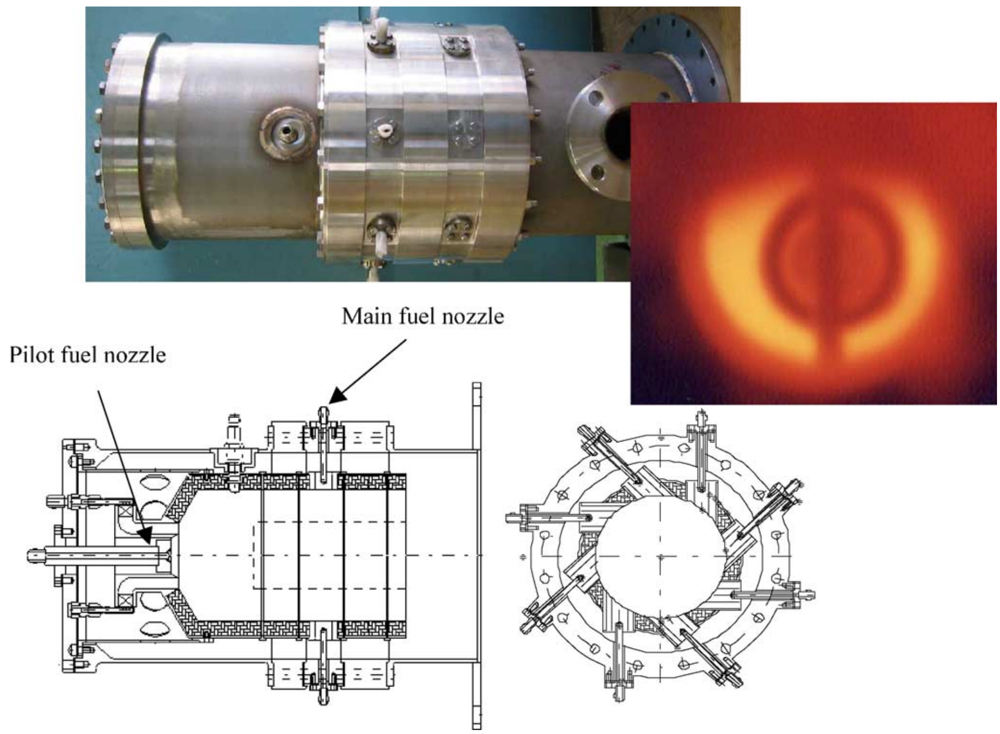


Fig. 4. Schematics of MGC panel attachments to combustor liner.[7]

and core on the basis of thermal expansion coefficient and thermal stability. Turbine nozzle core made of copper was treated by plasma spaying coating method.

It has also been modeled and simulated the casting process by computer in order to optimize process parameters for near-net shape casting. The computer simulations were expected to be useful to accurately control the overall temperature of the large Bridgman-type furnace. These are also contributed to establish the near-net shape casting methodology.

Fig. 5. Examples of casting mold and core for turbine nozzle.[7]

### *2.4. Evaluation of materials reliability and mid-term durability*

Thermal stability of microstructure and residual strength characteristics are also examined in relation to the materials reliability and mid-term durability (approximately few thousands hours). SEM examinations of microstructures are shown in Fig. 6 after exposure at 1700 °C in air for 250 h.[7] These are typical results for $Al_2O_3$/YAG and $Al_2O_3$/GAP eutectic composites. After the exposure for 500 h, there are no microstructure changes and no reduction in residual flexural strength. These eutectic composites show good thermal stability and residual strength characteristics. Also, MGC components show excellent mid-term oxidation resistance and there are no changes in weight, shape and dimensions of components after exposure at 1700 °C in air for 500 h.

Hot corrosion resistance has also been investigated at 1700 °C in addition to 30 wt.% moisture environments. No weight-loss and no strength-reduction were observed even after exposure for 10 h. The MGC materials fundamentally displayed very superior hot corrosion resistance. The details of materials reliability and mid-term durability as well as innovative, breakthrough processing and manufacturing technology also includes computer simulation of the casting process were presented at Directionally Solidified Eutectic Ceramics Workshop (held on May 5–7, 2003 in Paris).[8–11]

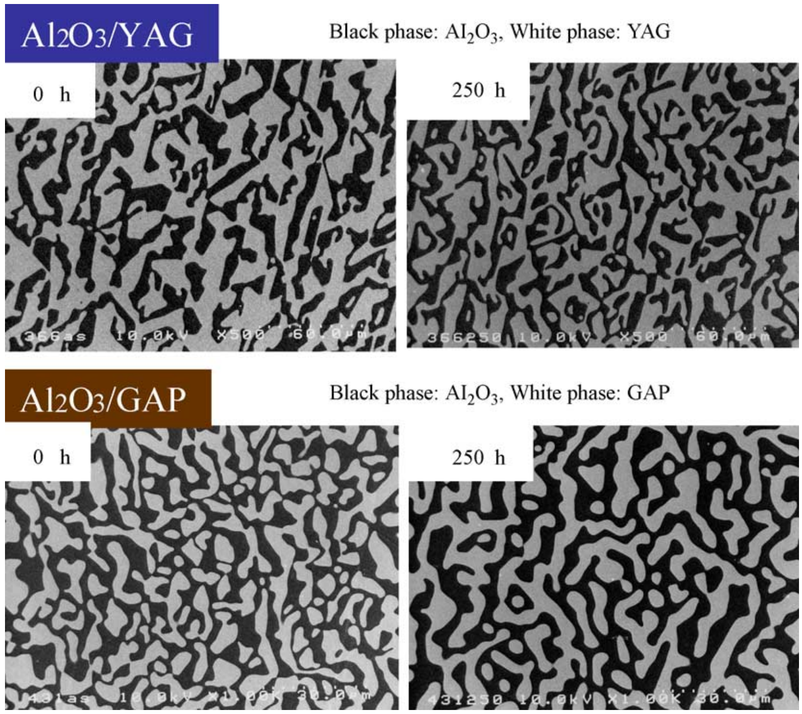


Fig. 6. SEM examinations of microstructures after exposure at 1700 °C in air for 250 h.[7]

## 3. Fracture toughness

### 3.1. Improvement of fracture toughness by ternary eutectic composite

Fig. 7 shows two-parameter Weibull's plots of fracture toughness determined according to the indentation fracture (IF) method for $Al_2O_3$/YAG/$ZrO_2$ ternary eutectic composite. Similar results for $Al_2O_3$/YAG binary eutectic composite[12] show that there is a remarkable orientation dependency and large scatter in fracture toughness at every plane. The fracture toughness is lower for the samples parallel to the solidification direction than perpendicular direction. Fundamentally, fracture toughness for YAG phase is lower than $Al_2O_3$ phase. Fine interfaces have little influence on fracture toughness. Low fracture toughness is predominantly controlled by YAG phase in the microstructures.

Mean value of fracture toughness was approximately 1.3 times larger in comparison with $Al_2O_3$/YAG binary eutectic composite. Shape parameter *m* value of the Weibull's distribution is also large, and the scatter in fracture toughness is less than that for binary eutectic composite. It is concluded here that the materials reliability was improved by ternary eutectic composite. It is also very interesting that there is a quite difference in orientation dependency between binary and ternary eutectic composites, and fracture toughness is not always to be lowest in parallel to the solidification direction for ternary eutectic composites.

### 3.2. Temperature dependence of mode-I fracture toughness

Pure mode-I and -II, mixed-mode (I + II) fracture toughness testing were performed for $Al_2O_3$/YAG binary eutectic composite according to chevron-notched (CN) diametrical compression test method[13–15] up to 1700 °C in air. The test method has an advantage that is simple and applicable to high temperature ranges. Mode-I fracture toughness $K_{IC}$ are plotted as a function of test temperature in Fig. 8 .[16] It is found that fracture toughness also depends on test method and there is a little bit of a variation. Room temperature fracture toughness is ranging from 2.5 to 4.0 MPa $m^{1/2}$. By considering these scatter band, fracture toughness are nearly constant and there is no temperature dependence up to 1700 °C. It is due to no drastic transition in fracture mechanism. It is almost the same to the temperature dependency of flexural and tensile strength characteristics. It is also found that fracture toughness in air has a tendency to be slightly lower than in vacuum.

Why is the mode-I fracture toughness for $Al_2O_3$/YAG eutectic composite independent of temperature? Fracture toughness for single crystal $Al_2O_3$, that is, sapphire rapidly decreases from 4.5 (RT) to 1.0 MPa $m^{1/2}$ (1000 °C) and then slightly increases to around 1.5 MPa $m^{1/2}$ with increasing of test temperature as shown in Fig. 9. These are also identical to the experimental results determined according to the controlled surface flaw method.[17] On the other hand, it had already reported that single crystal YAG increases with test temperature from 2.2 MPa $m^{1/2}$ (RT) to 4.5 MPa $m^{1/2}$ (1600 °C) in air.[18] That is to say, temperature

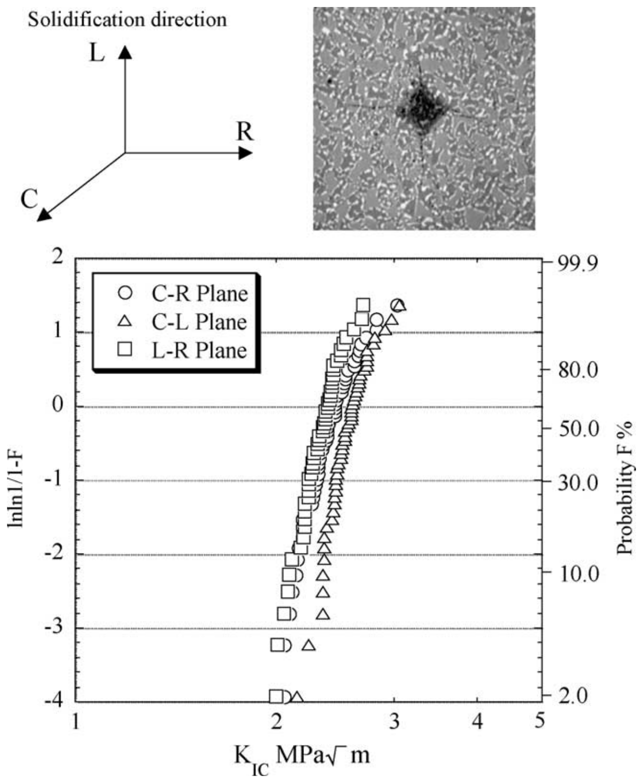


Fig. 7. Orientation dependency and variation characteristics of fracture toughness for $Al_2O_3$/YAG/$ZrO_2$ ternary eutectic composite.

dependencies offset each other and consequently $K_{IC}$ for single crystal $Al_2O_3$/YAG binary eutectic composite results in a constant regardless of test temperature up to 1700 °C, although the ductile fracture pattern is partially observed over 1550 °C. It is also very interesting that fracture toughness for $Al_2O_3$/YAG eutectic composite can be quantitatively predicted on the basis of the following rule of mixture (ROM).

$$K_{IC}\left(\frac{Al_2O_3}{YAG}\right) = K_{IC}(Al_2O_3)V + K_{IC}(YAG)(1-V) \quad (1)$$

where $V$ is volume fraction of $Al_2O_3$ phase. Also, Eq. (1) does not take into account the nature of the interfaces and

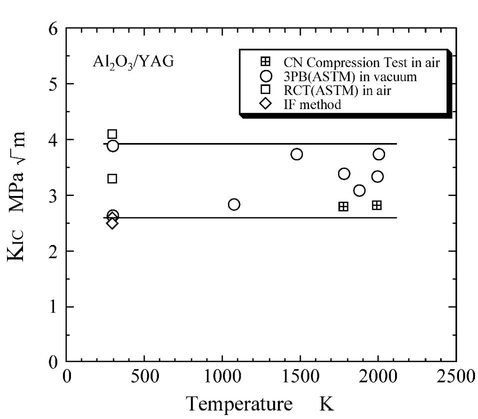


Fig. 8. Mode-I fracture toughness $K_{IC}$ as a function of test temperature.

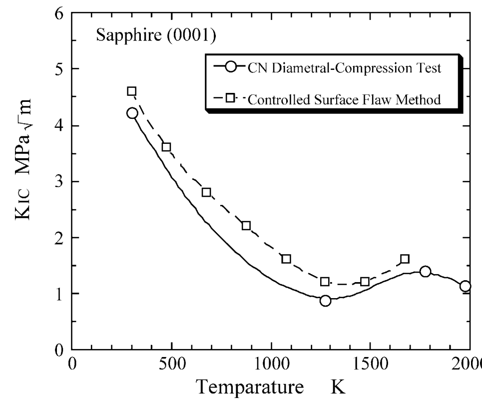


Fig. 9. Temperature dependency of fracture toughness for sapphire.

interaction of each phase. It is just a phenomenological explanation. It should be noted here that the plastic deformation capability of eutectic composites is not always contributed to improve fracture toughness even at 1700 °C.

### *3.3. Mixed-mode fracture toughness and fracture criterion*

Mixed-mode fracture toughness testing shows that pure mode-II fracture toughness $K_{IIC}$ is larger than $K_{IC}$. The $K_{IIC}/K_{IC}$ ratio for $Al_2O_3$/YAG binary eutectic composite is around 2.18 at 1500 °C. Also, there is a slight difference in macroscopic fracture manner between pure mode-I and pure mode-II loading as shown in Fig. 10a and b. Pure mode-II exhibits significantly more debris and increased roughness on fracture surface. The higher mode-II fracture toughness is related to crack-shielding effect due to roughness of fracture surface. However, fracture was predominately controlled by quasi-cleavage fracture. It should be noted here that mode-I stress component at crack tip controlled fracture even under the macroscopically mode-II loading condition.

The $K_{IIC}/K_{IC}$ ratio is larger than 1.0. Consequently, mixed-mode fracture toughness cannot be predicted by strain-energy release rate ($K_{IIC}/K_{IC}$ = 0.816), maximum hoop-stress (0.87) and strain–energy–density criteria (1.054). Here, the following empirical formulae were applied to predict the mixed-mode fracture toughness.

$$\left(\frac{K_I}{K_{IC}}\right) + \left(\frac{K_{II}}{CK_{IC}}\right)^2 = 1 \quad (2)$$

$$\left(\frac{K_I}{K_{IC}}\right)^m + \left(\frac{K_{II}}{K_{IIC}}\right)^n = 1 \quad (3)$$

where $C$ is $K_{IIC}/K_{IC}$ ratio. Fundamentally, Eqs. (2) and (3) also provide fairly good fits to the experimental data. There is no significant difference in these empirical formulae. One example of experimental results is shown in Fig. 11. Eq. (3) provides good fits to the data on $Al_2O_3$/YAG eutectic composite, sintered poly-crystal $Al_2O_3$ ceramic and sapphire specimens with the constants $m = n = 4$ (1500 °C), 2 (1300 °C)

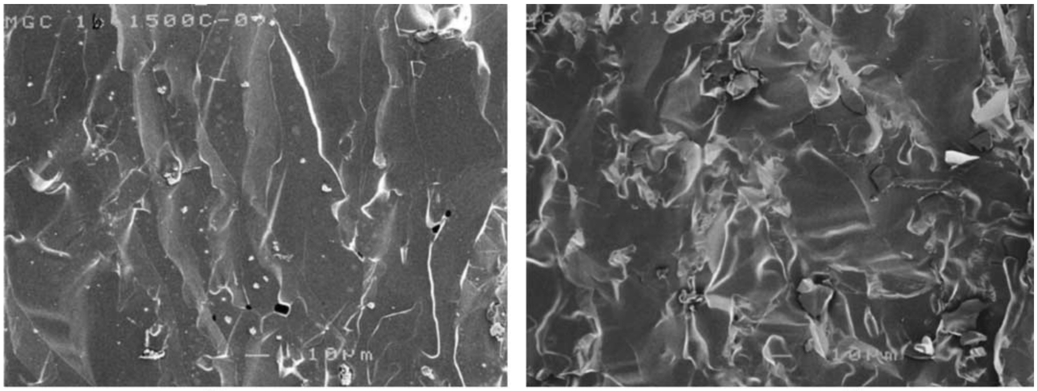
(a) (b)

Fig. 10. SEM examinations of fracture surface (a) pure mode-I loading and (b) pure mode-II loading.

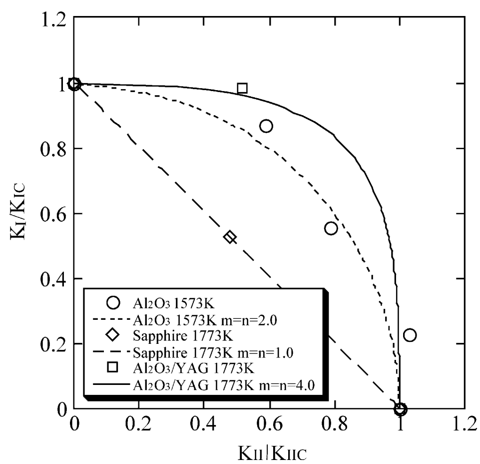


Fig. 11. Prediction of mixed-mode fracture toughness. Exponential *m* and *n* values are defined in Eq. (3).

and 1 (1500 °C), respectively. The exponential *m* and *n* values of $Al_2O_3$/YAG eutectic composite are larger than those of sintered $Al_2O_3$ ceramic and sapphire. The sintered polycrystal $Al_2O_3$ specimen fitted very closely to the predictions of the energy release rate criterion with $m = n = 2$. It is also surprised that sapphire fitted with $m = n = 1$. It is well-known that sapphire has a strong orientation dependency of fracture toughness.[17] This may be resulted from anisotropic properties.

## 4. Fatigue crack growth resistance

Fatigue crack growth resistance had already investigated in relation to materials durability.[5] The relationships between fatigue crack growth rate d*a*/d*N* (*a* is crack length and *N* is fatigue cycle) and maximum stress intensity factor $K_{max}$ are shown in Fig. 12 for $Al_2O_3$/YAG binary eutectic composite. Tests were conducted by four-point bending test at room temperature. The dotted line shows sintered poly-crystal $Al_2O_3$ ceramic.[19] The $da/dN - K_{max}$ relationship can be followed by Paris law in *R*–*L* direction (parallel to solidification direction). The *m* value as determined from slope in log–log diagram is slightly smaller than that for sintered $Al_2O_3$ ceramic. Fatigue crack growth resistance is lower at low $K_{max}$ near the threshold region and higher at high $K_{max}$ region.

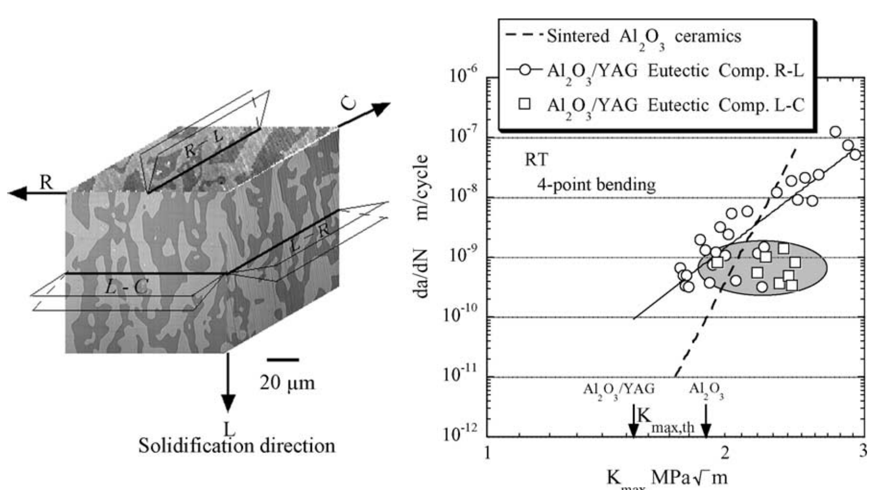


Fig. 12. Relationships between fatigue crack growth rate d*a*/d*N* and maximum stress intensity factor $K_{max}$.

Fundamentally, there is no transition in fatigue failure mechanism of $Al_2O_3$/YAG binary eutectic composite and predominantly controlled by cleavage fracture. On the other hand, in the *L–C* direction (perpendicular to solidification direction), the $da/dN - K_{max}$ relationship cannot be followed by Paris law and $da/dN$ are nearly constant regardless of $K_{max}$. The explanation for the constant $da/dN$ is that fatigue crack growth is stopped at the interface and then restarted after several ten thousands fatigue cycles. That is to say, the fatigue crack growth is discontinuous. In summary, interface has a remarkable influence on sub-critical crack growth characteristics for eutectic composite.

Creep resistance especially under highly water vapor pressurized environments and thermal shock resistance for eutectic composites can also be referenced to the literatures.[20–25]

## 5. A perspective: key technical issues for application

In summary, single crystal eutectic composites are high performance as the high temperature, environmental resistant structural material except for low fracture toughness and thermal shock resistance. They also have a fairly good machinability and manufacturability in comparison with conventional sintered engineering ceramics. However, the most important challenge is the low-cost processing and manufacturing technology for a wide practical application. It is still necessary for the future research to develop the integrated modeling and computer process simulation technique. Key technical issues to be urgently solved may be summarized as follows:

- Improvements of fracture toughness and thermal shock resistance
- Long-term durability and accelerated testing methodology
- Establishment of materials and design database

The materials database for eutectic composites has concurrently been started to construct.[26] Basically, researches are still in research and development stage. It is also focused on the processing and manufacturing technologies. Main contents of this database are the following properties.

- Thermal properties
- Chemical properties
- Mechanical properties
  - Elastic modulus/flexural strength/tensile strength
  - Measurements of residual stress
  - Fracture toughness/thermal shock resistance/fatigue strength
  - Creep resistance/environmental effects

A distinctive feature of this materials database is to cover crystallographic analysis, solidification analysis and computer simulation techniques of phase diagram.

## 6. Concluding remarks

Eutectic composites have many advantages as high temperature, environmental resistant structural materials. Japanese national project on application of these materials to gas turbine system was briefly introduced focusing on system integration and innovative, breakthrough processing and manufacturing technologies. The mechanical properties, fracture toughness and fatigue crack growth resistance were presented on the basis of improvements of materials reliability and durability. It is necessary for the further research to solve some key technical issues for a wide practical application.

## Acknowledgements

This research has been promoted as NEDO Project on MGC Applied Gas Turbine System under the sponsorship of Ministry of Economic, Trade and Industry (METI). I would like to highly acknowledge all members of Technical Committee, especially, Drs. Kenji Kobayashi and Thousaku Takamura, Ishikawajima-harima Heavy Industries, Drs. Yasuhiro Kinoshita and Masanori Yoshida, Kawasaki Heavy Industries, Drs. Yoshiharu Waku and Narihito Nakagawa, Ube Co. Ltd., and Dr. Yoshihisa Harada, National Institute of AIST for their supports and fruitful discussions.

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