The Evaluation of the Actual Fracture Energy of the Carbon Fiber Reinforced Silicon Nitride Composite by the Kr-BET Adsorption Technique

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Kr-BET 吸着法による炭素繊維強化窒化ケイ素複合材料の実破壊エネルギーの評価

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The fracture surface area of the chevron-notched carbon fiber reinforced silicon nitride composite was measured by the Kr-BET adsorption technique. The energy-absorbing mechanism of the composite was discussed on the basis of the actual fracture energy. The average actual fracture energy of the composite is nearly the same as the fracture energies of polycrystalline ceramics. This fact suggests that the large energy-absorbing capacity of the composite depends on how the fracture surface area can increase. Furthermore, application of the loading/unloading technique reveals that the actual fracture energy of the composite increases to a maximum at an initial stage of the failure, and then decreases to a constant value at a final stage. This indicates that the energy-absorbing mechanism changes from the formation of the fracture surface to the pull-out of fibers.

Key-words : Fracture energy, Composite, BET adsorption

1. Introduction

Many studies¹⁾⁻¹⁶⁾ have been carried out on the work of fracture to evaluate the fracture energy of ceramic materials. Most of polycrystalline ceramics have the fracture energies from 10 to 100 J/m², which are ten times larger than the thermodynamic surface energies. Recently, the work of fracture technique has been applied to fiber reinforced ceramic matrix composites,¹⁷⁾⁻¹⁹⁾ because the main crack of the composites can not be clearly observed, and so ordinary fracture mechanical techniques have not been strictly used to composites. The observed fracture energies of composites are more than 1000 J/m².

In previous studies, however, a projection area of a notch-ligament in the specimen was used as the nominal fracture surface area. Therefore, the discussions about the fracture energies reported previously, have less physical meaning. That is to say, it is necessary to know the actual fracture surface area after (and also during) the fracture test. The purpose of this study is to measure the actual fracture surface area of the fiber reinforced ceramic matrix composite by the Kr-BET adsorption technique, and to evaluate the actual fracture energy of the composite.

2. Experimental

2.1 Specimen

The carbon fiber reinforced silicon nitride composite^{20),21)} was used as a specimen, which was supplied by Noritake Co., Ltd. for the activities of the committee (The Ceramic Society of Japan) for the standardization of testing methods for fracture energy. The composite was unidirectionally reinforced by carbon fibers (60% in volume), and main crystalline phases of matrix were α -Si₃N₄ and Si₂N₂O.²²⁾ The bulk density was 2.55 g/cm³, and the open porosity was about 3%.²²⁾ The specimens were cut down in a rectangular shape (4 mm width * 3 mm thickness * 40 mm length), and a chevron-notch (vertical angle=120 degree) as shown in Fig. 1, was introduced into the specimen.

2.2 Measurement of a surface area by the Kr-BET adsorption technique

Brunauer, Emmett, and Teller²³⁾ made up the adsorption theory of a multilayer of molecules, by expanding Langmuir's adsorption theory²⁴⁾ of a monolayer. According to their theory, a total volume of adsorbing gas at an equilibrium pressure P is expressed as follows,

$$\frac{P}{V(P_0 - P)} = \frac{1}{V_m C} + \frac{C-1}{V_m C} \left(\frac{P}{P_0} \right) \quad (1)$$

where P_0 is a saturation pressure of adsorbing gas, V_m is a volume of a monolayer, and C is a constant depending on the heat of adsorption. Therefore, a plot of data for $P/V(P_0 - P)$ versus P/P_0 gives a straight line (BET plot), and a volume of a monolayer V_m can be calculated from the intercept $1/V_m C$ and the

Fig. 1. The configuration of the chevron-notch of the specimen.

slope $(C-1)/V_m C$ of the line. The value of V_m converts to a total surface area of the specimen using the area occupied by one molecule.

In this study, the total surface area of a chevron-notched specimen was measured before and after the three-point-bending test. The measurement was conducted with the ORR surface-area pore-volume analyzer (Micromeritics: 2100A) at a temperature of 77.23 K. Kr gas was used as an adsorbing gas, and the reduction of a pressure resulting from adsorption was measured repeatedly for one specimen, by changing the value of P/P_0 from 0.05 to 0.3 where the Eq. (1) can be used. The calculation was conducted using a saturation pressure, 2.42 Torr and the area occupied by one molecule, 0.208 nm². The increase of a total surface area after the three-point bending test is defined as the actual fracture surface area in this study.

2.3 Three-point-bending test

The chevron-notched specimen was loaded into a three-point-bending fixture with a span length of 30 mm at a crosshead speed 0.1 mm/min. The average actual fracture energy $\langle \gamma \rangle$ is expressed as follows,

$$\langle \gamma \rangle = \frac{\Delta W}{\Delta A} \quad (2)$$

where ΔW is the work done during the whole fracture process, and ΔA is the change in the total surface area of the specimen after the bending test. The number of the specimens is two.

Furthermore, The chevron-notched specimen was loaded, and then unloaded (loading/unloading technique¹²⁾). This cycle was repeated until the specimen fractured completely. The actual fracture energy γ is expressed as follows,

$$\gamma = \frac{dW}{dA} \quad (3)$$

where dW is the work done during one cycle of loading and unloading, and dA is the change in the total surface area of the specimen after one cycle. The number of the specimen is two.

3. Results and discussions

3.1 Reproducibility and accuracy of surface area

The surface area was measured twice with one specimen to investigate reproducibility and accuracy of the measurement. Figure 2 shows the BET plots of these two data. Closed circles are the data of the first measurement, and open circles are those of the second measurement. The linear relationships in BET plots indicate that the Eq. (1) exactly consists with the data. Reproducibility of the measurement is also held in the experiments. Analyzing two data, the total surface areas in the first and second measurements are 0.214 m² and 0.213 m², respectively. Therefore, it is suggested that accuracy of the measurement is appropriate to this study.

Fig. 2. The BET plots of data measuring one specimen twice. Closed circles are the data of the first measurement, and open circles are those of the second measurement.

3.2 The average actual fracture energy of the composite

The typical load/deflection curve of the composite is shown in Fig. 3. It is shown that the specimen has fractured after the manner of typical fiber reinforced composites. In the middle of the failure, the pullout of fibers was observed at a notch-ligament. The final pullout-length of fibers is 1–2 mm, as shown in Fig. 4.

The work calculated from an area under the load/deflection curve was about 0.25 J, and the change in the total surface area after the three-point-bending test was about 0.016 m². Using an area of the notch-ligament (5.9×10^{-6} m²) as the nominal fracture surface area, the apparent fracture energy of the composite is about 42 kJ/m². This value is nearly the same as the fracture energies of this specimens reported

Fig. 3. The load/deflection curve of the carbon fiber reinforced silicon nitride composite.

Fig. 4. The pullout of fibers after the bending test.

previously.^{20),21)}

On the other hand, applying the Eq. (2), the average actual fracture energy $\langle \gamma \rangle$ of the composite, is about 16 J/m^2 . This value is nearly the same as the fracture energies of polycrystalline ceramics reported previously.¹⁾⁻¹⁶⁾ Therefore, it is considered that fiber reinforced ceramic matrix composites almost have microscopically the same fracture processes as polycrystalline ceramics. These results also suggest that the large energy-absorbing capacity of composites, depends on how the fracture surface area can increase until the specimen fractures completely.

3.3 The change in the actual fracture energy during the fracture process

The typical load/deflection curve of the composite is shown in Fig. 5. The change in the total surface area (normalized by an initial surface area) is plotted in Fig. 6. It is shown that the total surface area rapidly increases at an initial stage of the failure, and saturates to the constant value at a final stage of the failure.

Next, the actual fracture energy against the deflection at the maximum load was plotted as shown in Fig. 7. From this figure, it is shown that the actual fracture energy of the composite rapidly increases at an initial stage of the failure, and reaches at the same level of the fracture energies of polycrystalline ceramics (several 10 J/m^2). This result suggests that the cracks extend actually in the notch-ligament at an initial stage, and the actual fracture energy mainly depends on the formation of the fracture surface. Whereas, the actual fracture energy slightly reduces at a final stage of the failure, and gives the value of

Fig. 5. The load/deflection curve of the specimen using a loading/unloading technique.

Fig. 6. The change in the normalized surface area during a bending test.

Fig. 7. The change in the actual fracture energy of the composite during the bending test.

several J/m^2 . This means that the main energy-absorbing mechanism changes from the formation of the fracture surface to the pullout of fibers.

As mentioned above, applying the Kr-BET adsorption technique to the fracture of composites, we can get the quantitative information about the energy-absorbing mechanism of fiber reinforced ceramic matrix composites.

4. Conclusions

The fracture surface area of the chevron-notched carbon fiber reinforced silicon nitride composite, was measured by the Kr-BET adsorption technique. The energy-absorbing mechanism of the composite was discussed on the basis of the actual fracture energy. The average actual fracture energy of the composite is nearly the same as the fracture energies of polycrystalline ceramics. It suggests that the large energy-absorbing capacity of the composite depends on how the fracture surface area can increase. Furthermore, applying the loading/unloading technique to the composite, the actual fracture energy of the composite increases to a maximum at an initial stage of the failure, and then decreases to a constant value at a final stage. This indicates that the energy-absorbing mechanism changes from the formation of the fracture surface to the pullout of fibers.

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