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전기저항측정에 의한 SiC섬유강화 Si_3N_4기 복합재료의 파괴예측

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Fracture Prediction in SiC Fiber Reinforced Si_3N_4 Matrix Composites from Electrical Resistivity Measurements

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초 록 섬유강화 세라믹스 복합재료의 파괴예측 가능성을 알아보기 위해서 탄소섬유와 WC분말입자를 전기 전도상으로 이용하여 재료 스스로가 파괴예측 기능을 가지도록 한 SiC섬유강화 Si_3N_4세라믹스기 복합재료를 1773K에서 1시간 동안 hot-press하여 제작하였다. 4점 굽힘 시험하는 동안 전기저항 변화를 측정하여 파괴예측 기능을 평가하였다. 그 결과 전기저항은 재료의 파괴거동과 밀접한 관계를 가지면서 변화함을 알았다. 특히 분말형태의 전기전도상의 첨가는 본 복합재료의 파괴과정을 낮은 응력단계로부터 예측하는데 유용하였다. 결과적으로 이러한 재료설계의 신개념(파괴예측기능)의 도입은 Si_3N_4기 세라믹스를 구조재료로 이용함에 있어서 큰 문제가 되고 있는 신뢰성 확보에 새로운 가능성을 준다고 생각되었다.

Abstract SiC fiber reinforced Si_3N_4 matrix composites combined with electrical conductive phases of carbon fiber and WC powder were fabricated by hot pressing at 1773K. The ability to predict fracture in the ceramic matrix composites was evaluated by measuring simultaneous load-deflection and electrical resistance difference-deflection curves in four point bending tests. The changes in electrical resistance differences closely corresponded to the fracture behavior of the composites. Different electrical conductive phases are suited to predicting different stages and rates of fracture. These observations show that it is possible to perform "in situ" fracture detection in ceramic composites.

Key words : fracture detection, Si_3N_4 composite, electrical resistivity, bending test, conductive phase

1. Introduction

Ceramic materials are not reliable enough for use as structural parts, mainly because of their brittleness. One means of overcoming the brittleness (i.e. improving toughness) is by developing ceramic matrix composites, for example, the controlled arrangement of interlocking needle-shaped grains in Si_3N_4[1], the fabrication of materials with whiskers[2~4] or with nanometer scale particles.[5] Another method to attain reliable ceramics is to design materials with the ability to detect when they have suffered damage or are about to fail.

This self-diagnosis function is one of the key characteristics of the new class of materials known as "intelligent materials".[6]

Ceramics have various properties such as electrical conductivity, piezoelectricity and transparency, which may be exploited for fracture prediction. Muto et al.[7,8] have reported that carbon-fiber/glass-fiber reinforced-plastic (CFGFRP) composites, in which electrically conducting carbon fiber and insulating fiber reinforced-plastic (FRP) is incorporated, can perform this function. An increase of electrical resistance is observed during loading of the composite, and the increased electrical resistance remains even after the load is removed. When the load is applied, carbon fibers in the composite are broken and disconnected, causing the increase in electrical resistance, while glass fibers maintain the strength of the composite. In composites containing electrical conduction paths, therefore, fracture can be predicted by measuring the change of electrical resistance generated by micro-deformation or the change of connectivity of the conduction path under an external load.[7,9~12]

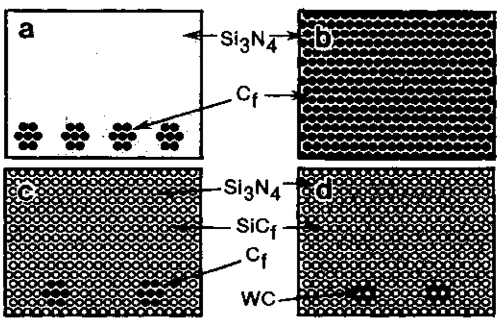
The main aim of this paper is to demonstrate the possibility and the effectiveness of adding fracture prediction functionality to ceramic matrix composites. In the present study, measurement of electrical resistance under loading was conducted on SiC fiber reinforced Si_3N_4 matrix ceramics containing electrically conductive phases of carbon fiber and WC powder.

2. Experimental Procedure

Si_3N_4 powder (Ube Industries Co., SN-COA) was used as the matrix material. Carbon fibers (C_f, Toray Industries Inc., T-400HB) and SiC fibers (SiC_f, Nippon Carbon. Ltd., Nicalon NL-401) were used for the reinforcement materials of the composites. Carbon fiber also serves as the electrically conductive material. WC powder (Japan New Metals Ltd., WC-055) was also added to provide electrical conductivity.

Four types of prepregnated specimens (prepregs) - Si_3N_4 containing 6 vol% C_f, Si_3N_4 containing 40 vol% C_f, Si_3N_4 with 40 vol% SiC_f and 3 vol% C_f, and Si_3N_4 with 40 vol% SiC_f and 4 vol% WC - were fabricated by the filament winding method: C_f and SiC_f bundles were soaked into either a surry of Si_3N_4-5wt% ($Y_2O_3+Al_2O_3$) or WC in methanol and then dried and cut into rod-rike prepregs. One or two types of prepregs were uni-directionally stacked into carbon molds to form continuous electrically conductive components. The stacked prepregs were hot-pressed at 1773K, 40 MPa for 1hr in N_2 atmosphere and then ground by diamond wheel into $4\times3\times40mm^3$ specimens.

Figure 1 shows schematic drawings of the cross-sections and components of the four types of materials. In composite (a) C_f was positioned at about 500㎛ above the lower face (the tension side in bending) of the specimen. Composite (b) contained carbon fibers arranged uniformly throughout the specimen. In composite (c) SiC_f was used to reinforce the material and C_f was substituted for SiC_f around 500㎛ from the lower surface.



composite	matrix	reinforcing fiber	conductive phase
a	Si_3N_4	——	6 vol% C_f
b	Si_3N_4	40 vol% C_f	40 vol% C_f
c	Si_3N_4	40 vol% SiC_f	3 vol% C_f
d	Si_3N_4	40 vol% SiC_f	4 vol% WC powder

Fig. 1. Schematic drawings of the structure and components of electrically conductive Si_3N_4 ceramic matrix composites.

Composite (d) was reinforced only by SiC_f and the electrically conducting WC powder was arranged in continuous lines from end to end of the specimen.

The fracture response of the composites was evaluated by the four point bending test (inner span 20mm, outer span 30mm, crosshead speed 0.1mm/min), with simultaneous measuring of the load-deflection curve and electrical resistance difference-deflection curve at room temperature. Scanning electron microscopy was performed to examine the fracture surface after the bending test.

3. Results and Discussion

Figure 2 shows the relation between the load-deflection and electrical resistance difference-deflection curves of the Si_3N_4-6vol%C_f composite. The composite exhibits a linear load-deflection relationship for small loads, and brittle fracture behavior. The strength of the composite is almost the same as the Si_3N_4 matrix on its own, but the hardness decreases slightly.

Electrical resistance changes only slightly in the elastic region but increases suddenly when the specimen fails catastrophically. In such a brittle material, it seems to be difficult to predict local fracture by measurement of electrical resistance, since no characteristic changes in resistance can be distinguished before failure.

Figure 3 shows the relationship between the load-deflection and electrical resistance difference-deflection curves of the Si_3N_4-40vol%C_f composite. The composite has a non-linear load-deflection curve and its fracture behavior is not brittle. It is noted that the electrical resistance difference increases together with deflection and discontinuous changes in the electrical resistance

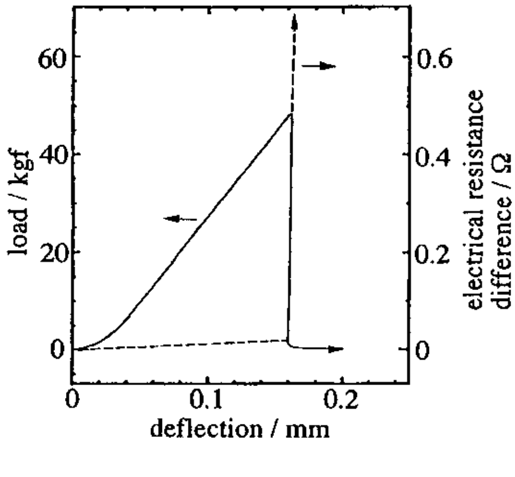


Fig. 2. Load-deflection curve and electrical resistance difference under bending for the Si_3N_4-6vol%C_f composite.

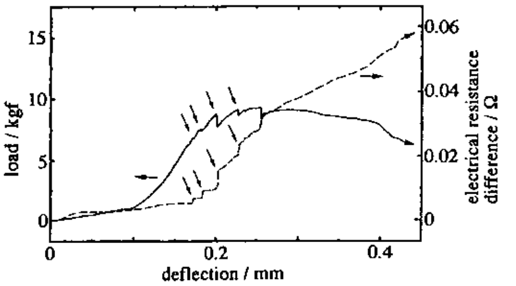


Fig. 3. Load-deflection curve and electrical resistance difference under bending for the Si_3N_4-40vol%C_f composite.

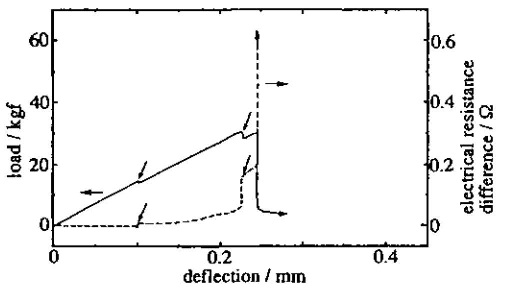


Fig. 4. Load-deflection curve and electrical resistance difference under bending for the Si_3N_4-40vol%SiC_f-3vol%C_f composite.

difference-deflection curve correspond to saw tooth changes in the load-deflection curve. Over the maximum load, the electrical resistance difference increases gradually with deflection.

Figure 4 shows the curves for the Si_3N_4-40vol%SiC_f-3vol%C_f composite. The composite displays non-linear load-deflection behavior and discontinuous electrical resistance difference changes similar to the Si_3N_4-40vol%C_f composite (see Fig. 3). However, the electrical resistance difference is about 10 times larger than that of Si_3N_4-40vol%C_f. This seems to be related to the number of C_f bundles set near the tension side of the material.

The changes in electrical resistance difference can be classified into three stages. In the first stage, up to a deflection of 0.1mm, a relatively small increase in electrical resistance was observed. In the second stage, the electrical resistance difference increased gradually after a small increment, and then jumped to about 0.2Ω at the same time as the load relaxed at around 30kgf. During the final stage, the electrical resistance difference increased more rapidly until catastrophic failure occurred. The large step-like increase in electrical resistance difference at the end of the second stage shows the failure of the composite from the onset of fracture in the reinforcing fiber.

Figure 5 shows the relation between load-deflection

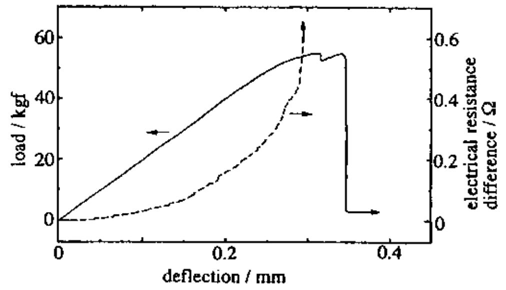


Fig. 5. Load-deflection curve and electrical resistance difference under bending for the Si_3N_4-40vol%SiC_f-4vol%WC composite.

and electrical resistance difference-deflection curves of the Si_3N_4-40vol%SiC_f-4vol%WC composite. The electrical resistance difference of the composite increases gradually with deflection and then rises rapidly to infinity at a load before the final failure. This result indicates that rupturing of the conduction paths has occurred before catastrophic failure. This change in electrical resistance can be used to predict the fracture of the ceramic composite matrix.

Generally, fiber reinforced ceramic composites are considered to fail by the following process: the first stage is local fracture of the matrix phase (matrix cracking) because of its low strength, the second is debonding and bridging of fibers, and the final is pull-out of fibers.[3] Figure 6 shows a schematic drawing of fracture behavior in fiber reinforced Si_3N_4 matrix composites containing an electrical conductive phase of C_f or WC powder. In the composites reinforced by a large amount of fibers, non-linear behavior is exhibited by the load-deflection curves (Figs. 2, 3 and 4) and a lot of pull-out of fibers was observed on the fracture surface of specimens. Figure 7 shows an SEM micrograph of the fracture surface of the Si_3N_4-40vol%C_f and Si_3N_4-40vol%SiC_f-4vol%WC composites. Pull-out of many C_f and SiC_f can be seen at the fracture surface, indicating breaking of fibers during deformation of the composites. This means that the standard fracture process mentioned above is dominant in these ceramic composites.

Addition of C_f as a continuous conductive phase is useful for fracture prediction of fiber reinforced composites, especially when the C_f content is low. In Si_3N_4-40vol%C_f and Si_3N_4-40vol%SiC_f-3vol%C_f composites, electrical conductivity through the C_f phase change, slightly up to the point where debonding and bridging of fibers occurs. The remarkable increase in electrical resistance is caused by pull-out of carbon fibers. The step-like changes in electrical resistance correspond to

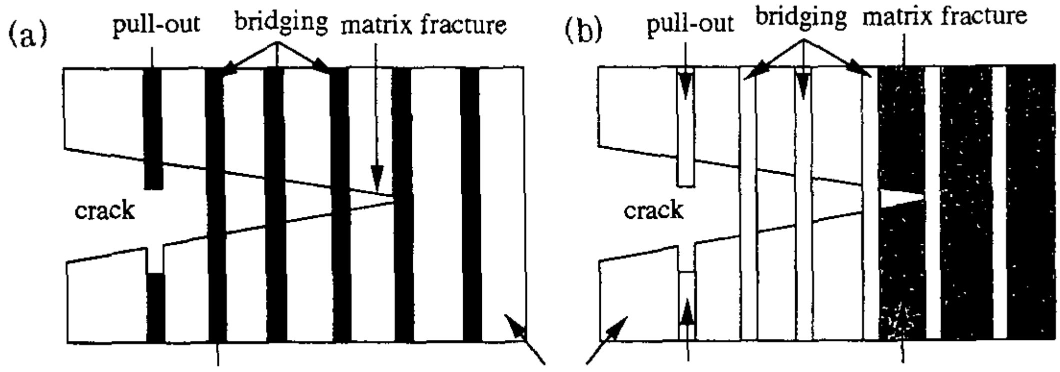


Fig. 6. Schematic drawings of the fracture behavior of fiber reinforced Si_3N_4 matrix composites with conductive phases of (a) C_f and (b) WC powder.

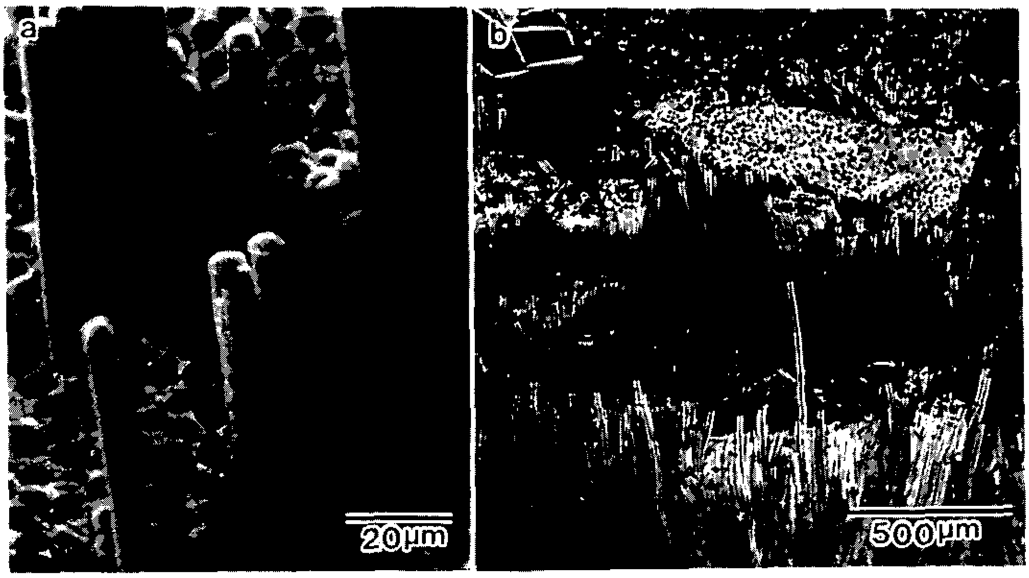


Fig. 7. SEM photographs of fractured surface of specimens. (a) Si_3N_4-40vol%C_f, (b) Si_3N_4-40vol%SiC_f-4vol%WC.

discontinuous propagation of fiber fracture. However, the electrical resistance difference in Si_3N_4-40vol%C_f is very complicated due to competing compressive and tensile stresses in the composite during bending tests. In the Si_3N_4-40vol%SiC_f-3vol%C_f composite, C_f placed near the tension side of the specimens results in electrical resistance increase due to fiber pull-out as shown in Fig. 6(a). The fracture process in Si_3N_4-40vol%SiC_f-4vol%WC is illustrated by Fig. 6(b). The continuous and conductive phase of WC powder that was substituted for part of the Si_3N_4 matrix functions as a means of predicting cracking of the matrix. This conclusion is justified by the sensitive response of the electrical resistance difference to small deflections in the composites.

Various methods exist for trying to improve the sensitivity of fracture detection in ceramic matrix composites further; e.g., using a thinner WC layer or more brittle material. In other words, it is possible that a variety of different electrically conductive phases can be used for fracture prediction. In this paper, we have given a few examples of how fracture in these composites can be predicted. By developing this concept for the design of intelligent materials, it should be possible to greatly improve the reliability of structures and machinery containing ceramic components.

4. Conclusions

The Si_3N_4-6vol%C_f composite exhibits a linear load-deflection relation and brittle fracture behavior. Electrical resistance changes little in the elastic region but increases suddenly at catastrophic failure.

The Si_3N_4-40vol%C_f composite has a non-linear load-deflection curve and its fracture behavior is not brittle. The electrical resistance difference increases with deflection and discontinuous changes correspond to saw tooth changes in the load-deflection curve. The Si_3N_4-40vol%C_f-3vol%C_f composite has a non-linear load-deflection relationship and discontinuous electrical resistance changes. In the Si_3N_4-40vol%SiC_f-4vol%

WC composite, the electrical resistance difference increases gradually with deflection and then rises rapidly to infinity at a certain deflection before the maximum load.

As these results indicate, electrical resistance changes are can be related to the fracture behavior of the ceramic matrix composites. It is therefore possible to predict fracture of ceramic composites by incorporation of electrically conductive phases.

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