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Alumina Ceramics Reinforced by Ni-coated Chopped Alumina Fiber

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Alumina composite reinforced by chopped alumina fiber was fabricated by filter-pressing the fiber slurry followed by the infiltration of alumina slurry. The chopped fiber was coated with nickel by electroless plating method. The green samples were densified by hot-pressing. Microstructures were studied by SEM and the mechanical properties such as bending strength and fracture toughness were measured. The resulting mechanical properties were analyzed in relation with processing parameters such as preform density and resulting microstructures. The load-displacement curve of the specimen with Ni interlayer but without Ni inclusion showed brittle fracture mode due to the direct contact between matrix and fiber. The load-displacement curve of the specimen with Ni interlayer and Ni inclusion in the matrix which is introduced by high applied pressure during specimen preparation showed non-brittle fracture mode due to the fiber pull-out and dutile phases in the matrix.

Key words: Alumina composite, Chopped alumina fiber, Ni coating, Non-brittle fracture

I. Introduction

Use of second phases such as whisker or particulate is a common technique to increase the strength and fracture toughness of structural ceramics. Use of continuous fiber coated with BN or C has also been intensively studied.[1,2] Continuous fiber reinforced CMC has disadvantages in that there are some limitations in selecting fiber/matrix combination and it is, in some cases, problematic to coat fiber surface homogeneously. Also there are some limitations in selecting the interface layer between the fiber and the matrix to satisfy the criteria for interface debonding.[3, 4]

The chopped fiber did not draw much attention. However, it seems that it can be possible to fabricate CMC with chopped fiber using conventional processing route. Use of chopped fiber can also provide reinforcing mechanisms such as debonding, fiber pull-out and/or plastic deformation when surface is coated with metallic phase such as Ni. Electroless deposition of Ni has been widely used for corrosion protection of steel.[5] In this study it was aimed to fabricate alumina composite reinforced by chopped alumina fiber coated with Ni interlayer in a cost effective way. The special emphasis has been put to the effect of preform density on the final properties.

II. Experimental procedure

1. Coating of Ni on chopped alumina fiber

The surface of chopped alumina fiber was coated by Ni using the wet method such as electroless deposition technique.[6,7] Catalyzing treatment of the surface of chopped alumina fiber is necessary due to its non-conducting property.

The chopped alumina fiber (ALMAX, 1 mm cut, 10 mm diameter, 99.5% α-Al_2O_3, Mitsui Mining Co., Japan) was first rinsed in distilled water under ultrasonic treatment. The process for catalyzing treatment is to sensitize the chopped alumina fiber in 1% $SnCl_2$ aqueous solution at 40°C for 5 minutes while stirring, followed by filtering and rinsing in the distilled water. The rinsed fiber was activated in $PdCl_2$ aqueous solution of which concentration is 2 g/liter at 40°C for 10 minutes in order for Sn^{2+} on the fiber surface to be replaced by Pd^{2+} which is conductive and acts as the nucleation site for Ni plating. Table 1 shows the composition of plating solution used. $NiCl_26H_2O$ is used as Ni source and $N_2H_4H_2O$(hydrazine) as reducing agent. Operation conditions were 80°C and pH of 11.7.

Table 1. The Composition of Plating Solutions Used and the Operating Conditions

Constituent	Composition [gr/liter]
$NiCl_26H_2O$	24.1
$KHCO_2$	45.1
K_2CO_2	69.1
K_2HOP_4	87.1
KOH	16.3
$N_2H_4H_2O$	32.6
pH	11.7
Temperature	80°C

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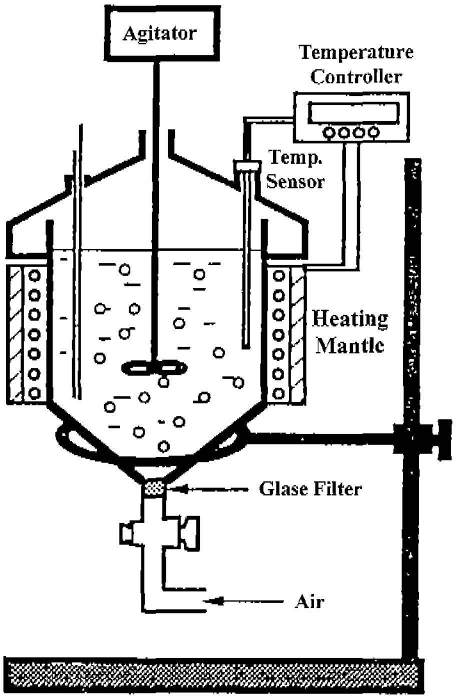

Fig. 1. Schematic drawing of plating bath.

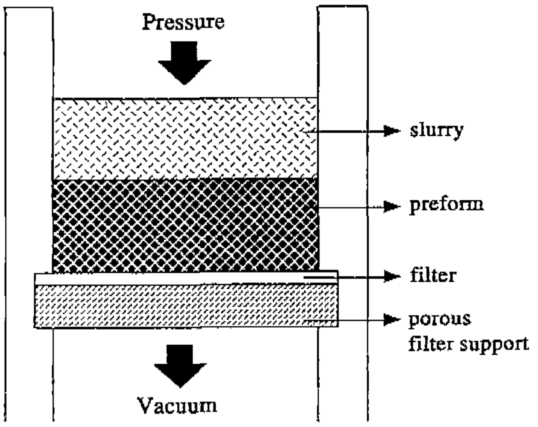

Fig. 2. Schematic drawing of filter pressing equipment.

Plating bath is conical shape as shown in Fig. 1. Mechanical stirring as well as air blowing from the bottom were employed for homogeneous distribution of chopped alumina fiber in the plating bath. The amount of Ni coated on chopped alumina fiber was calculated by weighing after plating, rinsing and drying.

2. Preform preparation

Ni-coated chopped alumina fiber was dispersed in distilled water (solid : liquid=10 : 90) using ultrasonic homogenizer (4710 series, Cole-Parmer Instrument Co., U.S.A.). The slurry was poured into filter pressing equipment which is shown in Fig. 2. Different pressures were applied to same amount of slurries in order to change the height of the preform, i.e., the preform density.

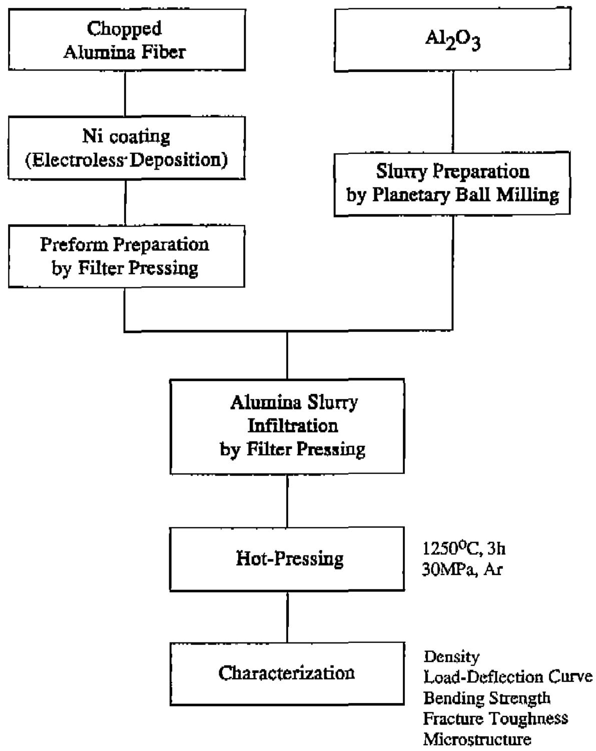

Fig. 3. Schematic flow diagram of the experiment.

3. Alumina slurry preparation

Alumina slurry has been prepared using alumina powder (AKP-30, Sumitomo, Japan). 70% of alumina powder was mixed with 30% of distilled water in a planetary ball mill using alumina jar and balls. 0.3 wt% of polyacrylic ammonium (Aron, A-6114, Toagosei Chemical Industry Co., Japan) was used as deflocculant.

4. Specimen preparation

Preforms with different densities have been infiltrated by the alumina slurry. Special caution has been taken in order to have alumina slurry to be infiltrated homogeneously throughout the preform by controlling the time for vacuum suction. In some cases excess amount of alumina slurry was poured to preform so that excess alumina slurry can form alumina layer on the top of infiltrated preform as shown in Fig. 2. The specimens were dried in vacuum drying oven at 80°C for overnight and hot-pressed in graphite mold at 1250°C for 3 hours under 30MPa in Ar atmosphere. The schematic flow diagram of the experiment is shown in Fig. 3.

5. Measurements

Bulk density was measured by electronic densimeter (ED-120T, Taiyo Trading Co., Japan). The three-point bending strength was measured by the universal testing machine (Instron, Model No. 4206, U.S.A.) with a span of 18 mm and a crosshead speed of 0.5 mn/min. Fracture toughness was calculated using the equation of Lawn and Fuller[8] after Vickers indentation using 10 kg load. Crack propagation mode after indentation as well as fracture surface after bending test were investigated using SEM (JSM 35-CF, JEOL, Japan).

III. Results and Discussion

Figure 4 shows the SEM micrographs of Ni-coated chopped alumina fiber. Coating layer is very irregular and the average thickness of the coating layer is approximately 1-2 microns.

Two specimens with different preform densities have been chosen for comparison and their properties are shown in Table 2. Specimen No. 1 which had lower preform density (11.5% TD) showed slightly lower green density after infiltration (56% TD) while specimen No. 2 which had relatively higher preform density (20% TD) showed slightly higher green density after infiltration (61% TD). The differences in preform density, green density after infiltration and fiber volume percent originate from the difference in heights of the specimen No. 1 and 2. Higher bending strength of specimen No. 2 seems to be due to higher sintered density after hot-pressing at 1250°C for 3 hours under 30 MPa in Ar atmosphere, as shown in Table 2. Fracture toughness measured by Vickers indentation method is also higher for specimen No. 2.

SEM micrographs of specimen No. 1 and 2 are shown in Fig. 5. SEM micrograph of specimen No. 1 consists of alumina matrix with Ni-coated alumina fiber although the cross section of Ni coating layer has been deformed to elliptical shape perpendicular to its hot pressing direction. The schematic drawing showing the deformation of Ni layer on

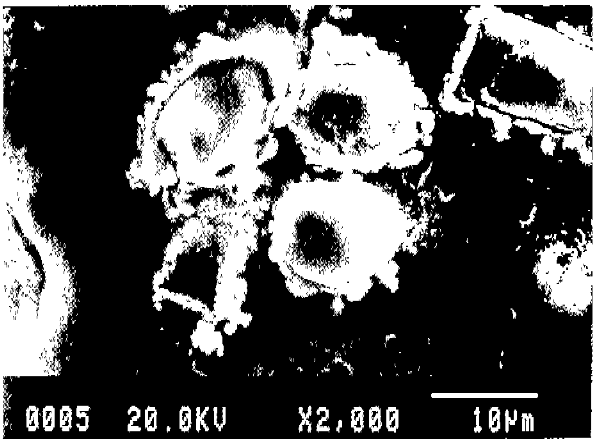

Fig. 4. SEM micrograph of Ni-coated chopped alumina fiber.

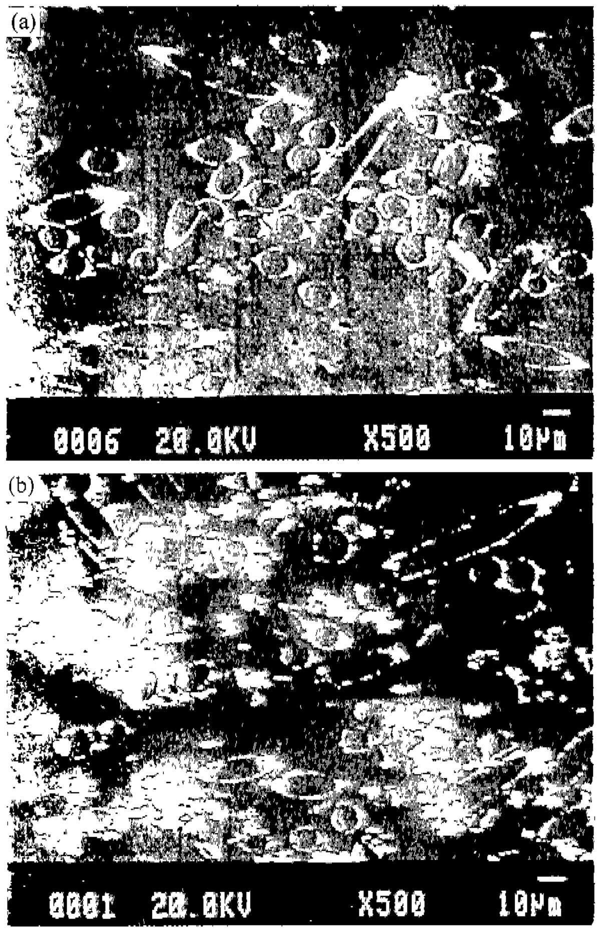

Fig. 5. SEM micrographs of the polished section of specimen No. 1 (a) and 2 (b).

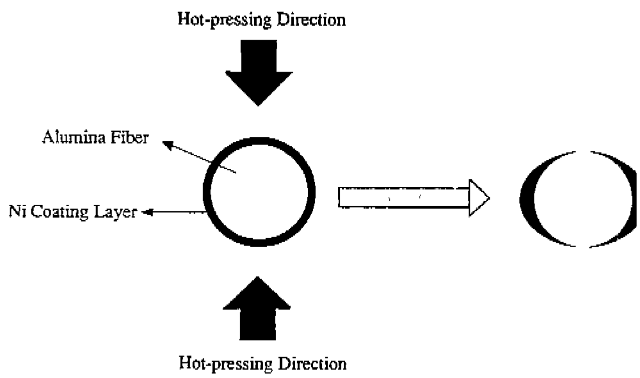

Fig. 6. Schematic drawing showing the deformation of Ni layer on chopped alumina fiber during hot pressing.

Table 2. Characteristics of Specimen No. 1 and 2

Specimen No.	Density of Preform [%TD]	Density of Specimen After Infiltration [%TD]	Fiber Vol %	Sintered Density [%TD]	Bending Strength [MPa]	Fracture Toughness [$MPa \cdot m^{1/2}$]
1	11.5	56	15	94	280	3.3
2	20	61	25	97.5	460	4.2

chopped alumina fiber during hot pressing is shown in Fig. 6. It is noticeable that Ni layer on chopped alumina fiber has been deformed to be pushed away to result in the direct contact between matrix alumina and chopped alumina fiber during hot pressing. SEM micrograph of specimen No. 2 in Fig. 5 shows more complex pattern compared with that of specimen No. 1. It consists of matrix alumina, chopped alumina fiber with deformed Ni layer as well as lots of Ni inclusions. The occurrence of Ni inclusions seems to be resulted from the Ni-coated chopped alumina fiber. Its reasonable when we remind that specimen No. 2 has been prepared using higher pressure during preform preparation. Ni layer on chopped alumina fiber has been deposited irregularly as shown in Fig. 4 and, when high pressure is applied during the preparation of preform, some debris will be formed which will be located in void spaces in the preform. When the preforn is infiltrated with alumina slurry during the fol-

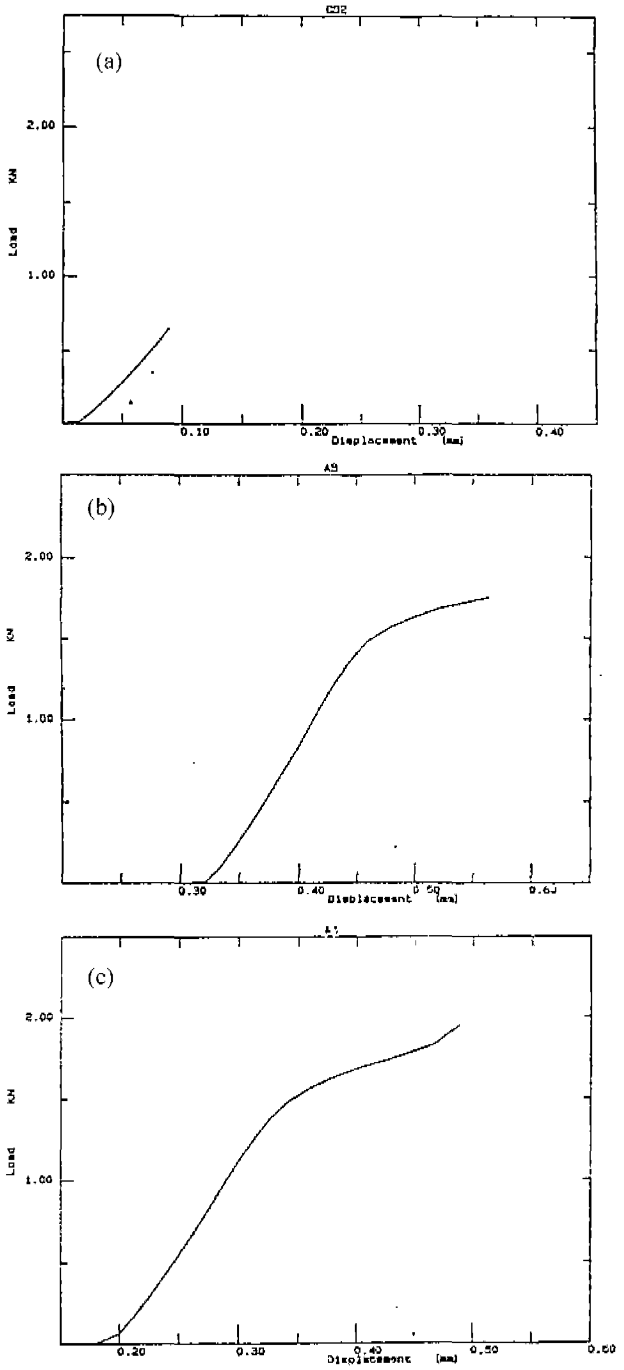

Fig. 7. Load-displacement curves during bending test for (a) specimen No. 1, (b) specimen No. 2 and (c) specimen with layered structure.

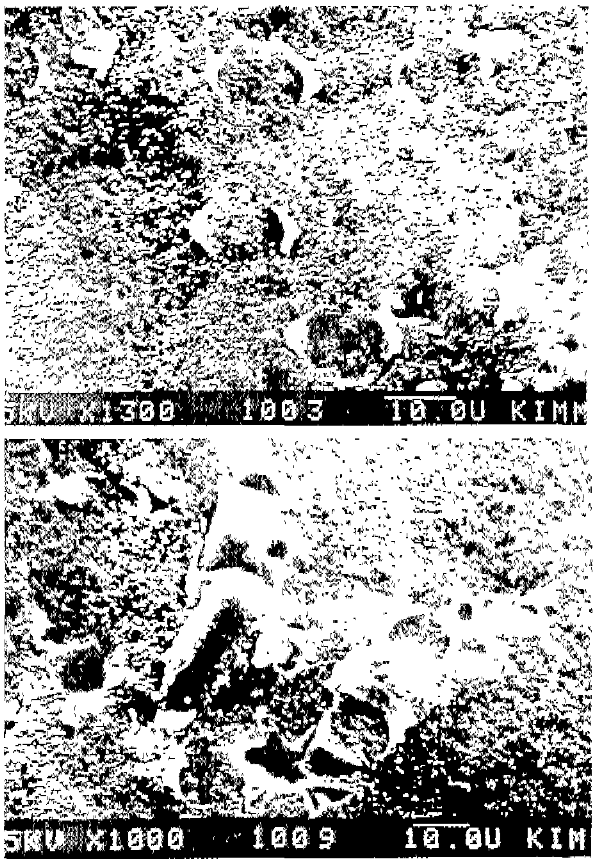

Fig. 8. SEM micrographs of the fracture surface of the speci men No. 1.

Fig. 9. SEM micrograph of the fracture surface of the speci men No. 2 showing fiber pull-out.

lowing step, some Ni-debris will be located among alumina particles, which will give rise to a distribution of Ni-inclusions inside alumina matrix after hot pressing.

The load-displacement curves during bending test are shown in Fig. 7. Specimen No. 1 shows the typical brittle fracture mode. Ni-coated chopped alumina fiber didnt contribute much to modify the fracture mode because some of Ni layer has been pushed away during hot pressing to cause direct contact between alumina matrix and chopped alumina fiber (Fig. 8). Specimen No. 2 shows non-brittle fracture mode in which it shows two-stage fracture mode. The slope of the load-displacement curve becomes steeper and steeper to a certain point beyond which it become slower. When the crack tip starts to propagate and meet the alumina matrix it propagates through possibly grain boundaries. When it meets Ni-coated chopped alumina fiber to a favorable direction the fiber pull-out may occur (Fig. 9). The steep increase in the slope of the load-displacement curve seems to be due to the fiber pull-out. The sudden drop which is usual case in continuous fiber reinforced composites has not been observed. Instead, it showed the turning stage in which the slope of the curve suddenly changed. This stage may be explained by the Ni-coating layer. Contrary to other coating layers like BN or C, Ni coating layer itself on the chopped alumina fiber can deform plastically resulting in the slower increase in the slope of the curve (Fig. 10). The slope of the curve becomes slower just after the turning stage. It possibly suggests that the plastic deformation of the Ni inclusion may play a dominant role in this stage.

When we add excess amount of alumina slurry than the amount enough to fill the void space in preform during infiltration process, alumina layer will be formed on the top of the specimen. This layer-structured specimen is also hot-pressed at 1250°C for 3 hours under 30 MPa in Ar atmosphere. The resulting load-displacement curve is shown in Fig. 7(c). The general tendency is similar to that of the specimen No. 2 except that it has the tail which corresponds to the fracture behavior of alumina layer. When we consider that work of fracture is proportional to the area of the curve, the layered structure will show higher fracture resistance.

Crack propagation modes after Vickers indentation for specimen No. 1 show little hindrance due to the direct contact between alumina matrix and chopped fiber, while those of specimen No. 2 with Ni-inclusions show hindrance of crack propagation possibly due to the plastic deformation of metallic Ni (Fig. 11). These also confirm that Ni-coated chopped alumina fiber as well as Ni inclusions play an important role in fracture resistance.

IV. Summary

Alumina ceramics reinforced by Ni-coated chopped alumina fiber showed two different fracture behaviors, brittle or non-brittle mode, depending on sample preparation methods. When the applied pressure is low enough to give low preform density during preform preparation, i.e, coating layer on chopped alumina fiber is not damaged, most of Ni layers on chopped fibers will deform during hot pressing to cause chopped alumina fiber to be exposed to the matrix, which results in the direct bond to the matrix during hot pressing. This is the major reason why they show brittle

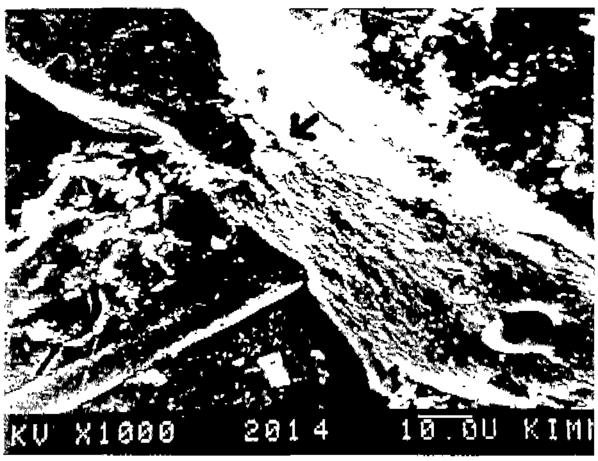

Fig. 10. SEM micrograph of the fracture surface of the specimen No. 2 showing the plastic deformation of Ni layer on the fiber.

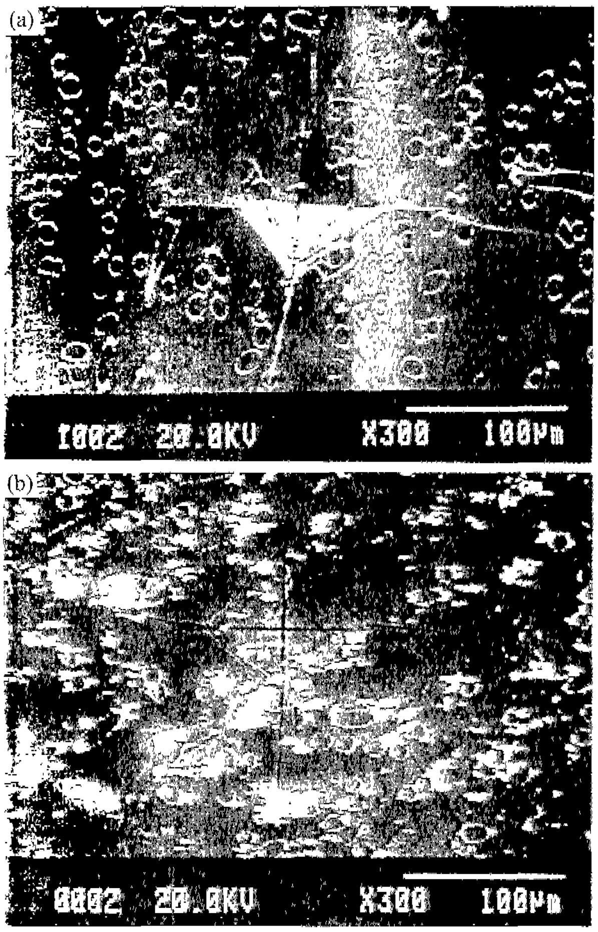

Fig. 11. Crack propagation modes of specimen No. 1 (a) and 2 (b).

fracture mode. On the other hand, when the applied pressure is high enough to damage some of the Ni layers on chopped alumina fibers during preform preparation, the debris will act as Ni inclusion in alumina matrix which is, in part, responsible for the non-brittle fracture mode. In both cases fiber pull-out seemed to play a role to a certain extent. Especially, Ni inclusions in alumina matrix exist as cluster of irregular shape which seems to be beneficial to increase fracture toughness because crack tip can not easily deflect around the irregular-shaped Ni inclusions.

Acknowledgements

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