

Fracture toughness of Al_2O_3 fibers with an artificial notch introduced by a focused-ion-beam

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Abstract

The present work was carried out to estimate the fracture toughness of two types of Al_2O_3 fibers (85 Al_2O_3 –15 SiO_2 , Altex[®] (Sumitomo Chemical Co., Ltd) and α - Al_2O_3 , Almax[®] (Mitsui Mining Co., Ltd)) and to elucidate the transition from the intrinsic defects-induced fracture to introduced notch-induced one. With an application of the focused-ion (Ga^+)-beam micromachining method, a mode I type straight-fronted edge notch with a notch-tip radius around 25 nm was introduced in fiber specimen. The fracture toughness K_{Ic} was estimated for each fiber specimen based on the fracture mechanical approach in which the measured values of notch depth, fiber diameter, fracture strength and calculated correction factor were substituted. The fracture toughness values of the 85 Al_2O_3 –15 SiO_2 and α - Al_2O_3 fibers were estimated to be 1.86 ± 0.24 and $2.05 \pm 0.13 \text{ MPa m}^{1/2}$, respectively. The fracture toughness value was almost independent of the fiber diameter and notch depth in both fibers tested. From the obtained fracture toughness value and the measured fracture strength of the original fiber, the notch depth at the transition from intrinsic defects-induced fracture to notch-induced one, corresponding to the equivalent size of the intrinsic defects that determines the strength of the original fiber, were estimated to be 0.3 and 0.8 μm for 85 Al_2O_3 –15 SiO_2 and α - Al_2O_3 fibers, respectively.

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1. Introduction

The small diameter fibers such as Al_2O_3 , SiC and C (carbon) fibers are used as reinforcements for composite materials. For estimation of fracture toughness value of these fibers, the difficulty arises from the small physical dimensions, due to which proper method to introduce small notches is limited. In addition, the fiber diameter is not unique, being different among the test fiber specimens (it has been reported that the diameters of SiC, C and Al_2O_3 fibers are distributed in the range of around 7.5–14, ^{1–5} 5.1–11^{6,7} and 8–17 μm ,^{1,2,8–10} respectively.) Thus, the influence of the fiber diameter on fracture strength shall be incorporated in estimation of fracture toughness for each fiber test specimen.

Due to the difficulties mentioned above, the fracture toughness has been estimated with the indentation fracture method or the empirical method (hereafter noted simply as mirror zone size method), which uses the relation between the fracture toughness and size of the mirror zone in fracture surface. However, concerning the indentation fracture method, it has been shown that indentation-induced subthreshold flaws on fused silica fibers in an inert environment behave differently from the post-threshold ones, due to which consistent result cannot be obtained for the specimens with different flaw size.^{11,12} The mirror zone size method can be applied only to amorphous or amorphous-like fibers that exhibit mirror, mist and hackle zones in fracture surface, but not to crystalline fibers that do not show such zones.

Thus there is a need to develop a method to introduce a sharp artificial notch directly in the small diameter crystalline fibers for estimation of fracture toughness. In our preceding work,¹³ it was attempted to introduce a sharp artificial notch in small diameter fiber with a focused-ion-beam (FIB). With this method, a

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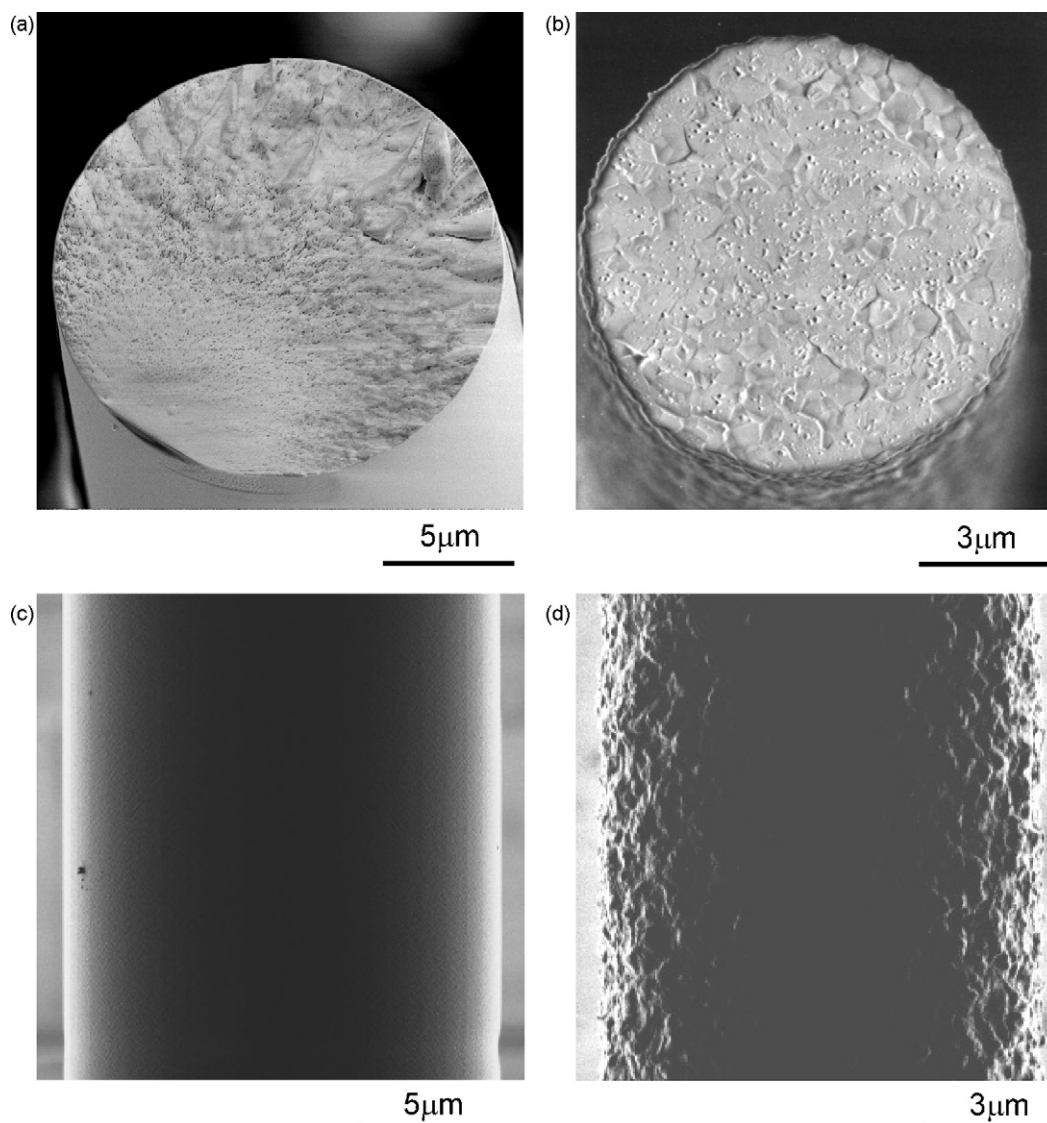


Fig. 1. Fracture surface of the unnotched (a) $85\text{Al}_2\text{O}_3$ – 15SiO_2 and (b) α - Al_2O_3 fiber specimens, together with the appearance of the side surface of (a') $85\text{Al}_2\text{O}_3$ – 15SiO_2 and (b') α - Al_2O_3 fibers.

sharp straight-fronted edge notch with a notch-tip radius around 25 nm could be introduced in the polycrystalline (Tyranno-SA[®], grade 3, Ube Industries) and amorphous (Tyranno-ZMI[®], Ube Industries) SiC fibers. In the present work, as the method to introduce a notch, the FIB method was applied to two types of the alumina fibers ($85\text{Al}_2\text{O}_3$ – 15SiO_2 fiber, Altex[®], Sumitomo Chemical Co., Ltd., where the figure of 85 for Al_2O_3 and that of 15 for SiO_2 refer to the chemical composition in wt %, and α - Al_2O_3 fiber, Almax[®], Mitsui Mining Co., Ltd.).

Fig. 1 shows the fracture surface and side surface of the $85\text{Al}_2\text{O}_3$ – 15SiO_2 and α - Al_2O_3 fibers. The $85\text{Al}_2\text{O}_3$ – 15SiO_2 fiber showed mirror, mist and hackle zones in fracture surface but not the α - Al_2O_3 fiber. This means that the mirror zone size method could be applied to the estimation of fracture toughness value of the $85\text{Al}_2\text{O}_3$ – 15SiO_2 fiber but not to that of

the crystalline fiber (α - Al_2O_3). To reveal the fracture toughness values of both fibers, it is needed to apply the common method to both fibers other than the mirror zone size method. The present approach using the FIB-introduced notch makes it possible to estimate the fracture toughness values comprehensively.

The main aims of the present work were (i) to reveal the fracture toughness values of these fibers, (ii) to examine whether the fracture toughness value is dependent on the fiber diameter and notch depth or not, and (iii), based on the obtained fracture toughness values and the strengths of original fibers without notch, to reveal the critical notch depth at the transition from intrinsic defects-induced fracture to notch-induced one, which corresponds to the equivalent size of the intrinsic defects that determine the strength of the original fiber without notch.

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