



# Effects of mixed-mode ratio and step-shaped micro pattern surface on crack-propagation resistance of carbon-fiber-reinforced plastic/adhesive interface



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## ABSTRACT

This study investigated the effects of the mixed-mode ratio of applied loads ( $G_{II}/G$ ) and aspect ratio  $A$  of step-shaped micro patterns on the crack-propagation resistance of a carbon-fiber-reinforced plastic (CFRP)/adhesive interface fabricated by in-mold surface modification. Experiments showed that the fracture behaviors change and that the apparent mixed mode fracture toughness  $G_C$  increases with  $G_{II}/G$  and  $A$ . We used the Benzeggagh–Kenane (B–K) failure criterion for the mixed-mode fracture toughness considering the transition of the failure mode of the step-shaped micro patterns. The B–K criterion agreed well with the improvement of  $G_C$  due to an increase in  $G_{II}/G$  for various fixed values of  $A$ . We clarified the relationship between the aspect ratio  $A$  and the parameter  $\eta$ , which is required to describe the B–K criterion, and therefore,  $\eta$  can be estimated from  $A$ . Consequently, it was verified that  $G_C$  of the CFRP/adhesive interface with step-shaped micro patterns can be predicted for arbitrary  $G_{II}/G$  and  $A$  values by substituting the  $\eta$ – $A$  relationship in the B–K criterion.

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## 1. Introduction

In recent years, the automotive industry has witnessed a strong push toward lightweight vehicles with a view to reducing fuel consumption and therefore improving efficiency. Toward this end, composite materials such as carbon-fiber-reinforced plastics (CFRPs) are expected to find widespread use [1]. Composite structures are generally formed using adhesively bonded joints; at the same time, additional surface modifications such as grid blasting [2,3], plasma treatment [4], and peel ply [5] are often required to achieve high adhesion strengths. However, these conventional surface modification techniques have some drawbacks. First, they involve secondary fabrication processes that make them excessively time consuming for application to mass production in the automobile industry. Second, workers without appropriate protective gear may be exposed to the harmful air used in these processes [6]. Third, although recycling is important, especially for mass-produced automobile structures [7], disassembly of joint parts usually has not been considered.

To address the issue of applying composites to the mass production of automobiles or in the automotive industry, we have investigated an in-mold surface modification process that uses imprint lithography to produce adhesive joints on composite surfaces. Then, we confirmed that the interfacial fracture toughness of a composite/adhesive interface with microstructures modified by this process can be affected by the aspect ratio  $A$  [8–10]. Interfacial failure occurred at the composite/adhesive interface under mode I [8,9], and interfacial failure and cohesive failures that break microstructures on the composite surface occurred under mode II [10]. For controlling the crack resistance, some studies investigated the effects of microstructures on the adherend on adhesive strength or interfacial fracture toughness. Byskov-Nielsen et al. [11] confirmed that the adhesive strength of a polymer/metal interface with microholes fabricated by laser machining was found to increase as the distance between the holes decreased or the depth of the holes increased. Shahsavan and Zhao [12] confirmed that the interfacial energy release rate by peeling off polymer bilayers containing micropillars fabricated by photolithography increased as the contact area increased. Janarthanan et al. [13] confirmed that the increase in the mode I fracture toughness of a polymer bilayer with grooves depended on the groove orientation. Kim et al. [14] confirmed that the mode II interfacial fracture toughness of CFRP/metal interfaces with micropatterns fabricated by photolithography improved as

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the micropattern width increased. They showed that the crack resistance is largely affected by the fracture toughness of the adhesives in addition to the configuration of the microstructure of the adherend surface.

Many fracture criterion models based on the mixed-mode ratio have been proposed according to specific adherend types and fracture behaviors [15,16]. Whitcomb's power law [17] is generally applied [15] for constant fracture behaviors whose fracture criterion can be described by a linear or ellipsoidal form. However, once cohesive failure occurs when the mixed-mode ratio increases, the fracture criterion based on the power law cannot fit the experimental value of  $G_c$  because the mode I component of  $G_c$  may become greater than the pure mode I fracture toughness  $G_{IC}$  [18,19]. Recently, some new fracture criteria have been proposed to consider the change in fracture behavior from interfacial to cohesive failure [20–22]. In particular, the Benzeggagh–Kenane (B–K) criterion [23] is often applied to a composite/adhesive interface in which cohesive failure occurs [24–27].

However, the studies have not yet clarified the effects of the mixed-mode ratio and microstructure shape fabricated on an adherend surface on the crack resistance and fracture behaviors because they have been limited to simple conditions such as mode I or II for the evaluation of the adhesive properties of microstructures on an adherend surface. In addition, few studies have evaluated the failure criteria of a composite/adhesive interface with micro pattern structures when including fracture transition under the mixed mode. The fracture behaviors of a CFRP/adhesive interface with microstructures may change depending on the dimensions of the microstructures in addition to the mixed-mode ratio. Thus, the failure criteria for a CFRP/adhesive interface with microstructures have to be investigated by simultaneously considering the effects of the mixed-mode ratio and the dimensions of the microstructures.

With this background, we investigate the effects of the dimensions of the step-shaped micro patterns and the mixed-mode ratio on the crack resistance and fracture behaviors of a composite/adhesive interface modified by in-mold surface modification. In the experimental evaluation, we used single leg bending (SLB) tests under mixed-mode ratio  $G_{II}/G$  to obtain the crack resistance of the CFRP/adhesive interface with microstructures with various aspect ratios  $A$ . In addition, by observing the crack propagation of the CFRP/adhesive with the step-shaped micro patterns, we considered the effects of the mode ratio and aspect ratio of the step-shaped micro patterns on the transition of the fracture behaviors and the crack propagating resistance. Finally, the applicability of the B–K criterion for step-shaped micro patterns surface is discussed.

## 2. Materials and methods

### 2.1. In-mold surface modification

Step-shaped micro patterns were fabricated on the CFRP surface by in-mold surface modification following the nanoimprint lithography procedure [28,29] used for forming a mold with micropatterned structures. Fig. 1 shows a schematic of the in-mold surface modification process. First, after coating a releasing agent (ChemTrend, Chemlease #70) on the Al mold surface, carbon/epoxy prepregs (Mitsubishi Rayon, Pyrofil #380) were stacked on the mold. The unidirectional ply properties have been introduced in our previous study [10]. Second, the prepregs stacked on the mold were pressed by a flat Al plate. A vice was used to apply pressure to the two Al molds packed in a vacuum bag, and the in-mold surface modification for composite molding was conducted in a drying oven (Isuzu Cosmos VTN-114) under vacuum produced using a vacuum pump (ULVAC, G-10DA). The prepregs were cured

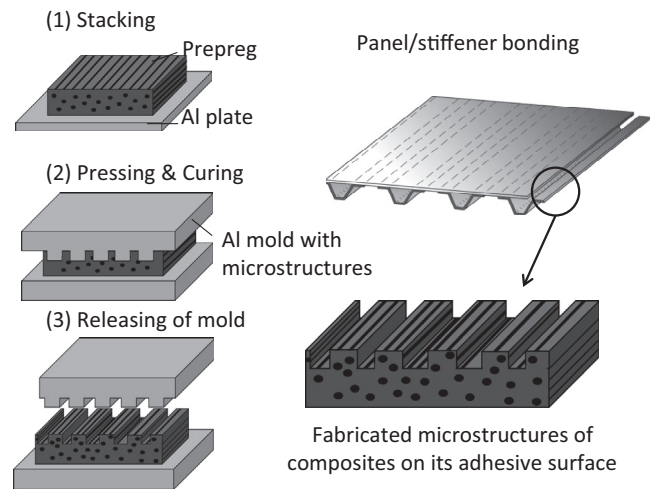


Fig. 1. Schematic illustration of in-mold surface modification by imprint lithography for composite materials.

in two steps over the glass transition temperature under a pressure of 0.6 MPa (at 85 °C for 1 h and at 135 °C for 3 h), which allowed the molten matrix resin to flow into the microstructures of the mold. Finally, the microstructures were transferred to the CFRP by demolding at room temperature.

The step-shaped micro patterns of the mold for in-mold surface modification were manufactured on an Al plate by milling, as shown in Fig. 2(a) and (b). Different sizes of the step-shaped micro patterns were prepared on a single mold. Therefore, all micro patterns on the mold can be transferred to a composite during composite molding at the same time. Here, the carbon fibers of the surface layer with microstructures must be oriented parallel to the step-shaped micro patterns so that they can be transferred to the CFRP exactly. This fiber orientation was set to avoid forming defects in the transcription of the step-shaped micro patterns on a composite, because the height of the step-shaped micro patterns is comparatively high. Forming defects such as void nucleation actually occurred, as shown in Fig. 2(c), when the fibers of the surface lamina were oriented vertically to the step-shaped micro patterns on the mold. Therefore, the transcription of the microstructures will worsen because they will not be filled with resin. As a result, the carbon fibers on the surface layer with microstructures are oriented at 90°, which is parallel to the lines of the step-shaped micro patterns to distribute resin and carbon fibers uniformly. In this study, the stacking sequence of adherends applied to the in-mold surface modification was set to  $[90/0]_{2s}$ .

To distinguish the size of the step-shaped micro patterns fabricated by in-mold surface modification, the aspect ratio ( $A$ ) is defined as the ratio of the step-shaped depth  $h$  to the sum of the widths  $w_1$  and  $w_2$  (shown in Fig. 2(d)) and is expressed by Eq. (1).

$$A = \frac{h}{(w_1 + w_2)} \quad (1)$$

The aspect ratio was adopted as the representative parameter of adherends with step-shaped micro patterns, because the fracture behaviors and fracture toughness of the adhesive interface with the step-shaped micro pattern changes largely depending on the aspect ratio compared with the sizes of the step-shaped micro patterns [8–10]. The step-shaped depth was set to a constant value of 150  $\mu\text{m}$ . In addition, the ratio of the width of a dent  $w_1$  to the width of a bump  $w_2$  was set as 1:1, i.e.,  $w_1:w_2 = 1:1$ , and the values of  $w_1$  and  $w_2$  were in the range of 300–600  $\mu\text{m}$ . Therefore, four types of microstructures were simultaneously fabricated on the CFRP with

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