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Observation of nanometer waves along fracture surface

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Abstract

The fracture in solid materials is ideally referred as a two-dimensional surface formed by a crack moving through a planar, straight-line path. In reality, the fracture has a complicated morphology. Recent studies have developed a dynamic model in which, a moving crack results in three-dimensional, elastic waves that generate morphology along the fracture surface. The waves are defined by their wavelengths of millimeters or higher scales. We present the observation of nanometer waves along the fracture surface of the silicon dioxide layers (thickness ~0.5–2 μm). These waves with the wavelengths of ~200 nm form a well-defined surface structure.

1. Introduction

Many recent studies have focused on the elastic waves formed by a moving crack. These waves with intrinsically three-dimensional property do not exist in the classical theories of crack propagation [1]. Their formation provides an explanation of the complex fracture morphology in nature (e.g. rock patterns) [2,3].

The mechanism of formation of waves in an ideal, homogeneous material is related to the instability of the propagation of a straight crack. Sharon et al. have shown that when a crack propagates at a speed of approximately 0.4 the speed of sound across a free surface (i.e. Rayleigh speed, $\sim$3.3 km s^{-1}), it becomes unstable and will proceed via formation of micro-branches [2]. Branching leads to the increase of cracking energy and triggers the formation of waves. Similarly in heterogeneous materials, interaction of the crack with material inhomogenity leads to an energy fluctuation that also generates the waves via formation of micro-branches [2]. The thermal stress in the material also influences the formation of waves. In particular, a transition from straight to wavy fracture occurs as the thermal stress increases, due to the concomitant increase of cracking energy. This is commonly referred to as a Hopf bifurcation [4–6].

The average wavelengths of these waves are usually of millimeters or higher scales, although the wavelengths of several micrometers have recently been observed [2]. In this Letter, nanometer scale waves are reported. These nano-waves are uniformly distributed along the fracture surface of the amorphous SiO_2 thin film layers. They generate a well-defined surface structure not observed previously at nano-scale. Previous studies have suggested that the fracture surface remains rough at this scale [7]. The roughness is varied with varying the cracking energy and crystallographic properties. In our experiments, the formation of nano-waves is favoured as the thermal stress in the films increases.

2. Results and discussion

The nano-waves are created spontaneously following cleaving the micrometer thick, amorphous films of silicon dioxide (Fig. 1). These waves form the mirror images on the opposing fracture plane. For the film with thickness of ~2 μm, the wavelengths are estimated as 200 nm. These SiO_2 films are prepared by thermal oxidation of Si wafers at 1000 °C (see Section 4). By comparison, the films previously prepared via

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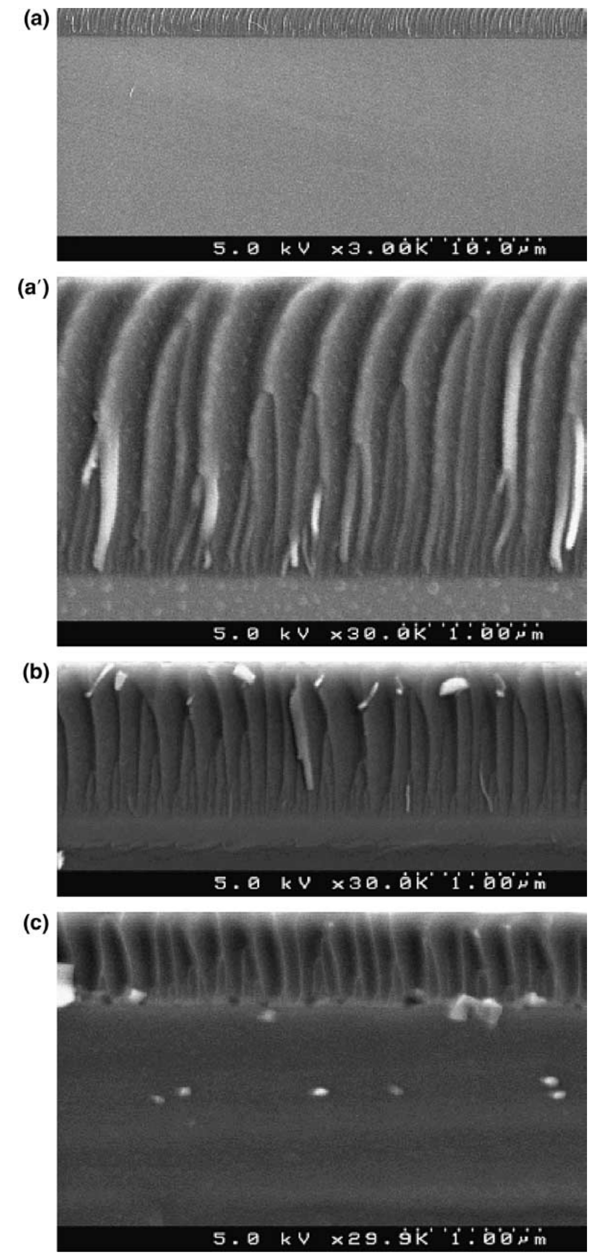

Fig. 1. Cross-section scanning electron microscopy of differently thick SiO_2 films. The films are prepared by thermal oxidation of Si(1 0 0) wafers at 1000 °C between 1 and 14 days. (a) Formation of the nano-waves along the fracture surface of the 2 μm thick film; (a′) enlargement of (a). The wavelengths are of ~200 nm; (b)–(c) formation of the nano-waves along the fracture surface of the films with thickness of ~1 and 0.5 μm, respectively. The fracture sections of the underlying Si substrates remain virtually planar.

radio-frequency sputtering, chemical vapour deposition and sol–gel resulted in a smooth or porous fracture [8]. Rapid thermal oxidation has been extensively used for preparation of the ultra-thin SiO_2 films (thickness of few nanometers) that may be used as a dielectric layer in Si integrated devices [9]. By contrast, a relatively extreme condition is used in our experiments with the oxidation time being substantially extended (see Section 4).

In fact, the extreme condition for preparation of SiO_2 film is related to the formation of nano-waves. It is well known that these films are accompanied with a large thermal stress, as a result of the significant volume expansion during oxidation of Si (oxidation at 1000 °C should create a thermal stress in SiO_2 of $\sim 3 \times 10^8$ N m^{-2}) [9]. Our results are in agreement with those from Yuse and Sano [5] who showed that formation of millimeter waves on the fracture surfaces were favoured as the crack propagated through a large thermal stress field. In their experiments, cracks were formed when a glass plate was heated and transferred to a cold-water bath. As the heating temperature and therefore thermal stress was increased, the redistribution of stress gave rise to a systematic transition from planar to sinusoidal and other wavy fracture.

To test the influence of stress to the formation of nano-waves, we examine the fracture morphology of SiO_2 films with lower thermal stress. The films are prepared from oxidation of Si at temperature of 600 °C. This should result in the thermal stress of $\sim 1.4 \times 10^8$ N m^{-2} [9], i.e. decreased by a factor of two compared to the above films. For this film, the fracture surface remains virtually planar (Fig. 2). Although, the onset of

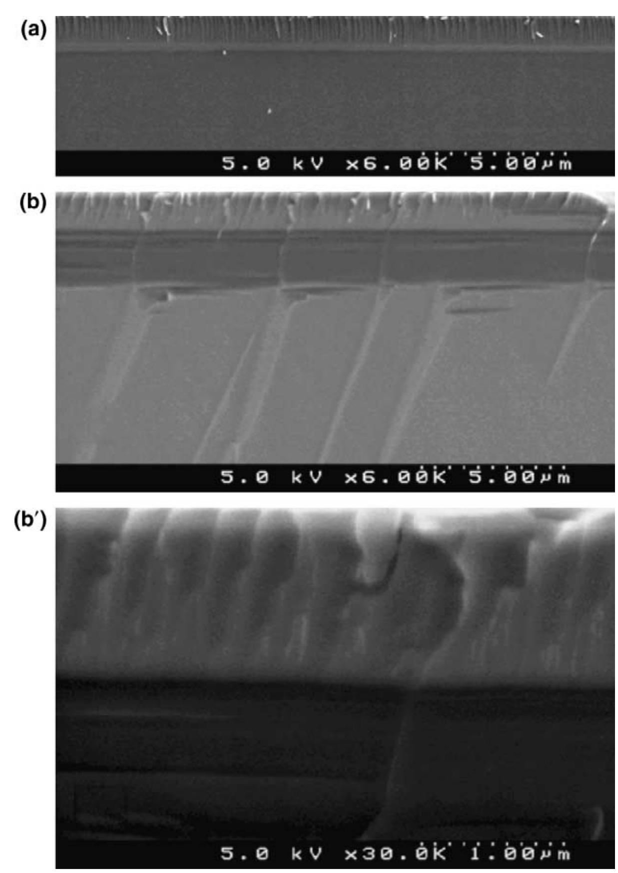

Fig. 2. Scanning electron microscopy showing the fracture morphology of the 1 μm thick SiO_2films prepared by thermal oxidation of Si(1 0 0) at (a) 1000 °C and (b) 600 °C. (b)′ the enlargement of (b).

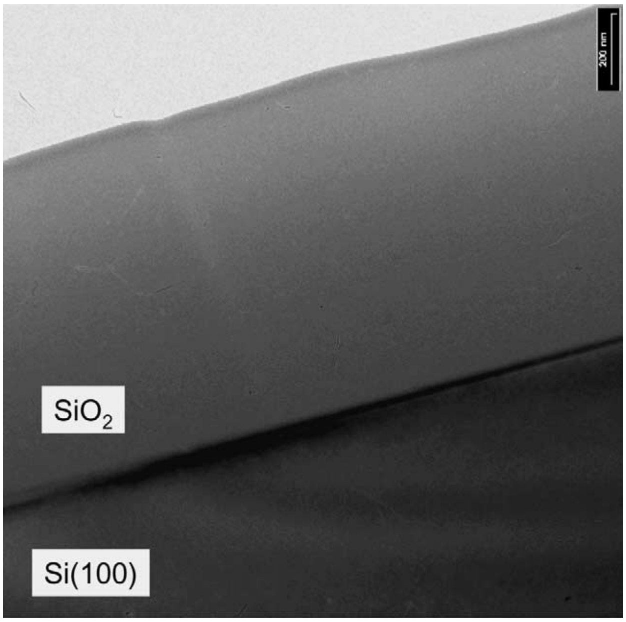

Fig. 3. Transmission electron microscopy of the 1 μm thick SiO_2film prepared by thermal oxidation of Si(1 0 0) at 1000 °C. The film is not columnar. For this experiment, due to the sample preparation procedure, the fracture surface is heavily damaged and therefore the nano-waves are not observed.

waves is evidenced via the formation of various thin lines across the surface. This observation is similar to that of the planar or sinusoidal wavy fracture in low-stress material [5]. The combined results indicate that the morphological transition in SiO_2 fracture could also be referred as a Hopf bifurcation [4–6].

In addition, there are possibilities that the nano-structures of the films influence the formation of waves. In particular, the columnar structures commonly observed in ceramic films may also lead to formation of wavy fractures [10]. For this, transmission electron microscopy shows that the films are not columnar and are amorphous (Fig. 3). The amorphous nature is confirmed from X-ray diffraction measurements. Previous studies have also shown that the crack in crystalline Si propagated along a crystallographic plane results in a planar fracture [11–13]. This can be confirmed from our results, where planar fractures of the underlying Si substrates are clearly distinguishable with wavy fractures of the films (Figs. 1 and 2). These combined results indicate that formation of waves is not related to a specific nano-structure of the films or substrates.

3. Summary

We report the observation of a fracture surface created by the well-defined nanometer waves. The mechanism of formation of waves at much larger scales also explains these nano-waves. Our observation therefore suggests there exists a universal mechanism for formation of fracture morphology over a wide range of scales. Further research into propagating speed is necessary in order to understand whether the formation of nano-waves involves dynamic crack (fast propagation) or quasi-static crack (slow propagation). It would also be of interest to carry out studies on whether the intrinsic properties of SiO_2 films such as short-range atomic structures, interfacial stress or density have any influence on the formation of waves.

4. Methods

The amorphous films of SiO_2 have been prepared from thermal oxidation of Si with (1 0 0) crystallographic orientation. Our experimental conditions were relatively extreme compared to the conventional thermal oxidation process. In particular, we carried out oxidation in air between 1and 14 days. This resulted in the film thickness of maximum of 2 μm. The films were cleaved using a diamond cleaver. Cleavage was performed along the Si(1 0 0) direction and crack propagated along the (1 0 0) plane. We did not observe the formation of nano-waves at the beginning of the crack tip. This section remained rough. The roughness was probably related to the application of a large amount of crack driving force [12]. With increasing length of crack, the roughness decreased and the waves were grown uniformly. Similar effects were also observed by Fineberg et al. [14].

Acknowledgements

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