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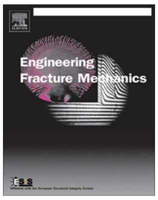

Creep fracture at interfaces of titanium nanocolumns on silicon substrate

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ABSTRACT

The dominant mechanics of creep fracture at interfaces on the nanoscale was evaluated on the basis of creep experiments conducted at room temperature on Ti oblique nanocolumns grown on a Si substrate using glancing angle deposition. To clarify whether the nanoscale stress concentration dominated the interfacial fracture caused by creep, two types of specimens were prepared: a forward specimen (loading with the column tilt direction) and a reverse specimen (loading against the column tilt direction), where the reverse specimen had a higher stress singularity at the interface edge than the forward specimen. The specimens deformed in a time-dependent manner under a constant applied force, and then the Ti nanocolumns fractured at the interface. The forward specimens required a higher applied force than the reverse specimens for a similar fracture life. The local stress distribution along the Ti/Si interface during the creep experiments was analyzed using finite element method, while giving due consideration to the creep of the Ti nanocolumn. The Mises stresses near the edge in the region of about 5 nm were very close in forward and reverse specimens with similar fracture lives, even though the stresses outside this region were very different. This suggested that the creep interfacial fracture was dominated by the local stress field in the nanoscale region.

1. Introduction

In bulk materials, fracture is a phenomenon governed by local stress fields at stress concentration sites such as crack tips and notch roots, and fracture mechanics is widely used to evaluate the crack propagation strength or fracture toughness. The concept is extended to crack initiation at bimaterial interface edges, where singular stress fields appear even without cracks or notches because of a deformability mismatch [1–5]. However, the validity of using fracture mechanics for nanoscale interfaces remains an open question. As the component size shrinks, the singular stress region near the interface edge is proportionally scaled down. In nanoscale components, the singular stress region is inherently confined to the nanoscale [6–9]. In such a case, it is questionable whether the local singular field still dominates the fracture. A few studies have been conducted to clarify the applicability of fracture mechanics to the interfacial fracture of submicron- or nano-components such as multilayered thin films [8,9], cantilevers [7,10], islands [6], and columns [11] on substrates. The results of these studies demonstrated that a singular stress zone of a size ranging from several nanometers to several tens of nanometers still governs the crack initiation.

On the other hand, fracture mechanics is also used for characterizing time-dependent fractures such as the creep crack growth in bulk materials where a singular stress or strain rate field (HRR field) dominates the crack extension [12–16]. A

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Nomenclature

α	incident angle from substrate normal (°)
H	height of Ti thin film (nm)
W	width of Ti thin film (nm)
ρ	radius of curvature of interface edge (nm)
θ_{Ti}	corner angle of Ti (°)
θ_{Si}	corner angle of Si (°)
F_{N}	normal force (μN)
F_{L}	lateral force (μN)
δ_{N}	normal displacement (nm)
δ_{L}	lateral displacement (nm)
t	time (s)
T	temperature (K)
F_{NC}	fracture normal force (μN)
τ_{C}	fracture nominal shear stress (MPa)
F_{Nap}	applied normal force (μN)
τ_{ap}	applied nominal shear stress (MPa)
δ_{NI}	initial displacement (nm)
δ_{NF}	fracture displacement (nm)
t_{F}	fracture time (s)
$(d\delta_{\mathrm{N}}/dt)_{\mathrm{a}}$	average displacement rate (nm/s)
E	Young's modulus (GPa)
ν	Poisson's ratio
σ_{eq}	Mises stress (MPa)
$\dot{\epsilon}_{\mathrm{eq}}$	Mises strain rate (s^{-1})
A	power law creep coefficient ($\mathrm{MPa}^{-n}\,\mathrm{s}^{-1}$)
n	power law creep exponent
σ_{ij}	stress tensor (MPa)
r	distance from edge (nm)
λ	stress singularity

similar singular stress field appears at interface cracks [17] or interface edges between power-law creeping and elastic materials [18]. Experiments on creep interfacial fractures in a submicron-thick tin component on a silicon substrate [19] demonstrated that the stress intensity near the interface edge correlates well with the crack initiation life, which suggests that the local stress field influences creep crack initiation on the submicron scale. However, it is still an open question whether nanoscale singular stress fields in creeping materials govern the creep fracture. To the authors' knowledge, no experimental studies have been conducted to clarify this.

A remarkable geometrical characteristic of nano-components is their high surface and interface area-to-volume ratio. Because the diffusion of atoms occurs actively at solid surfaces and interfaces, the creep deformation is enhanced in such small structures [20–23]. Thus, the issue also has practical importance because time-dependent fractures occur easily in nanoscale devices, even under a small stress.

The purpose of this study was to clarify the dominant mechanics of the creep interfacial fracture in nanoscale components. The authors previously developed an experimental method for evaluating the interface strength of nanocolumns on a substrate [11], where the nanocolumns were fabricated using glancing angle deposition (GLAD). GLAD is a method for fabricating discretely grown oblique nanocolumns by utilizing the shadowing effect caused by the highly inclined incident angle used for physical vapor deposition [24–26]. In this study, creep interfacial fracture experiments were conducted at room temperature on titanium (Ti) oblique nanocolumns grown on a silicon (Si) substrate using GLAD. On the basis of the experimental results, the time-dependent stress field at the interface is numerically analyzed, and the dominant mechanics of creep interfacial fracture is discussed.

2. Experiments

Fig. 1 shows a field emission scanning electron microscope (FESEM, Hitachi Ltd., S-4500) image of the cross-section of a sample that was tested. The sample consisted of a Si (100) substrate with a Ti oblique nanocolumn array (thickness: 439 ± 10 nm) grown using GLAD, and a Ti thin film (thickness: 1219 ± 9 nm). The interface between the Ti nanocolumn array and the Si substrate was the target of this study. Each nanocolumn was at an oblique angle of 48 ± 1° from the substrate normal. The interface between a single Ti nanocolumn and the Si substrate was 41 ± 9 nm long in the column-tilt direction and 64 ± 10 nm wide in the orthogonal direction. The Ti nanocolumns and thin films were deposited using electron beam