Dislocation shielding and flaw tolerance in titanium nitride

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Abstract

Titanium nitride is a very brittle and flaw sensitive ceramic material at temperatures below 750°C. In this study, we present experimental evidence of room temperature dislocation-based plasticity in the material as well as insensitivity to flaws in form of single edge notches. We performed in-situ fracture experiments inside the transmission electron microscope on 150–300 nm thick, 5 µm wide freestanding films fabricated from titanium nitride/titanium multi-layers with titanium nitride as the notched and titanium as un-notched layers. The calculated stress concentration factor for the 800 nm to 1.5 µm long notches were greater than 8, however, the terminal cracks always nucleated at the un-notched edge of the specimens and not at the notch tip. To explain such remarkable flaw tolerance, we observe motion of dislocations (pre-existing and nucleated away from the notch) towards the notch tip. We suggest that the room temperature dislocation activities are facilitated by the residual stresses in the multi-layer specimens and the thickness dependence of image forces, which reduces the effective shear modulus to promote dislocation motion. The migration of dislocations towards the notch tip shields it from stress concentration to manifest the flaw tolerance in 150 nm specimens, which is observed real time in the microscope.

1. Introduction

Titanium nitride (TiN) is a very hard and chemically resistant ceramic material that has long been used as corrosion, erosion and wear protective coatings or as diffusion barrier layers in micro-electronics technology (Wu et al., 1990). Because of the ionic and covalent atomic bonding in ceramic materials, dislocations have very high activation energy (Carter and Norton, 2007) and plastic deformation is observed at temperatures above 0.4Tm, where Tm is melting temperature (Meyers and Chawla, 2009). For example, the brittle to ductile transition for TiN takes place at around 750–1000 °C (Yamada et al., 1980). However, there is ample evidence of strong specimen size effect on the brittle to ductile transition temperature (Bobylev and Ovid’ko, 2008; Jang and Greer, 2010; Kim et al., 2004; Michot et al., 1998; Zhu et al., 2008). Increased interest in this research area is due to the more recent experiments exhibiting low temperature ductility on thin films (Shigeki et al., 2008), nanowires (Han et al., 2007; Smith et al., 2010) and nanopillars (Minor et al., 2005; Östlund et al., 2009) of macroscopically brittle materials. It is proposed that dislocations can nucleate and move even in ceramic materials because of the lower activation energy at the nanoscale (Gerberich et al., 2009; Han et al., 2007). Altogether, the size effects on strength, ductility, fracture toughness, and fatigue – all point strongly towards dislocation plasticity as the underlying mechanism (Gerberich et al., 2009; Hartmaier and Gumbsch, 1999; Hirsch and Roberts, 1997; Michot and de Oliveira, 2001).