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Quantitative characterization of the interfacial adhesion of Ni thin film on steel substrate: A compression-induced buckling delamination test

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ABSTRACT

A compression-induced buckling delamination test is employed to quantitatively characterize the interfacial adhesion of Ni thin film on steel substrate. It is shown that buckles initiate from edge flaws and surface morphologies exhibit symmetric, half-penny shapes. Taking the elastoplasticity of film and substrate into account, a three-dimensional finite element model for an edge flaw with the finite size is established to simulate the evolution of energy release rates and phase angles in the process of interfacial buckling-driven delamination. The results show that delamination propagates along both the straight side and curved front. The mode II delamination plays a dominant role in the process with a straight side whilst the curved front experiences almost the pure mode I. Based on the results of finite element analysis, a numerical model is developed to evaluate the interfacial energy release rate, which is in the range of 250–315 J/m^2 with the corresponding phase angle from $-41°$ to $-66°$. These results are in agreement with the available values determined by other testing methods, which confirms the effectiveness of the numerical model.

1. Introduction

Over the past few decades, the development of thin film devices has spurred interest on buckling instabilities. As it is well known, thin films deposited on substrates play an important role in many applications such as semiconductor devices, magnetic storage media, and surface coatings (Mei et al., 2007). A common failure form in their applications is buckling of thin films, resulting in interfacial delamination and fracture (Hutchinson et al., 1992). The quality of interfacial adhesion of a film/substrate system is essential to the lifetime of a device. Therefore, how to quantitatively characterize the interfacial adhesion of a thin film is the key in its practical application.

A number of methods have been available for determining interfacial adhesion. Of these methods, the most commonly used ones are peeling (Wei, 2004), indentation (Drory and Hutchinson, 1996; Zheng and Zhou, 2005), bulge (Gent and

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Lewandowski, 1987; Jensen, 1991; Zhou et al., 2003; Jiang et al., 2008, 2010) and bending tests (Lane et al., 2000). However, there are still a lot of challenges associated with each of these techniques. For example, in the peeling test, when the thickness of a film is at micro or nano-scale, it is difficult to peel it off from substrate. Although indentation can overcome this limitation that in principal can be applied to specimens with any geometry, it frequently results in fracture within the coating that introduces uncertainty. Moreover, to make a film delaminate, the indenter needs to penetrate through the film, which leads to plastic deformation in substrate (Drory and Hutchinson, 1996). The bulge test is a useful method for quantitatively characterizing the interfacial adhesion of a film–substrate system that is available for both brittle and ductile films, however, the preparation of samples is difficult and the relevant theoretical models are complicated. Furthermore, the bending test is only suitable to brittle films (Lane et al., 2000). In consideration of these disadvantages, it is necessary to seek a new method that can be applied to evaluate the interfacial adhesion of a film–substrate system.

Thin films on thick substrates may buckle due to residual or external compressive stress. Such a phenomenon is prevalent, in which several configurations exhibit from circular, linear to telephone cord, and even to a network of folds (Wang and Evans, 1998; Moon et al., 2002; Kim et al., 2011). The associated mechanics of energy release rate (ERR) and mixed modes have been established (Hutchinson and Suo, 1991; Hutchinson, 2001; Jensen and Sheinman, 2001; Sorensen and Jensen, 2008). If the residual compressive stress in a film is low, there are no spontaneous buckles. Thus it is necessary to apply the external compressive stress to force thin films to buckle away from substrates. A strain-to-fail method was studied by Zhao et al. (2011), which was employed to examine the interfacial adhesion of electron beam–physical vapor deposited thermal barrier coatings (TBCs) with a Pt-diffused γ/γ' bond coat. The adhesion energies of Cr and indium tin oxide films on polyimide substrates, which were determined by measuring the buckle geometry under tension, were investigated by Cordill et al. (2010) and Jia et al. (2012), respectively. They found that channel cracks initiate and propagate in polyimide-supported Cr and indium tin oxide films along the direction perpendicular to tension. Upon further tension, some film fragments delaminate and buckle away from substrate, driven by compressive stress perpendicular to the tensile loading direction in films due to Poisson's effect. Although these phenomena are undesirable, one may take benefit of it for characterization of interfacial adhesion properties of a film–substrate system.

The purpose of this study is to characterize the interfacial adhesion of an elastic–plastic material such as Ni film on steel substrate by compression. The experimental method is presented in Section 2, including the synthesis of samples and the compression-induced buckling delamination test. Based on the measured buckling profiles, the ERR can be determined by a numerical model. In Section 3, the ERR and phase angle along the crack front are calculated by a three-dimensional (3D) finite element analysis. Based on the results of finite element simulations, a numerical model is developed to evaluate the interfacial ERR in Section 4. Finally, the values obtained by the numerical model are compared with the available ones determined by other testing methods.

2. Experimental

2.1. Synthesis of samples

The samples are Ni thin film deposited on steel substrate by the electrodepositing technology. Before deposition, steel substrate was cut into the size of $40\times5\times5\ \text{mm}^3$ by an electrical discharge wire-cutting machine and polished with diamond paste, then cleaned up by acetone and absolute alcohol. The steel substrate with an area of $200\ \text{mm}^2$ was put into the electrodepositing solution for 1 h with an electric current density of $50\ \text{mA/cm}^2$. The planting temperature was adjusted in the range of 50–60 °C to an accuracy of ±2 °C. The thickness of Ni thin film was in the range of 50–60 μm. Finally, the samples were cut into the size of $10\times5\times5\ \text{mm}^3$ for compression-induced buckling delamination tests.

2.2. Compression-induced buckling delamination tests

The test procedure is illustrated in Fig. 1. By using a REGER/2000 mechanical testing machine, the pancake indenter was impressed through one end of the sample and compression was applied along the direction parallel to the film/substrate interface. The load systematically increased until a typical buckling happened. With this method, the buckling-driven delamination was performed in a quasi-static manner. A charge-coupled device camera was put in front of the cross-section of the sample to monitor and record the buckling process.

Micro-flaws exist along the free edges of film/substrate, which are introduced during processing or machining. Under the action of residual stresses within film or an externally applied load, these micro-flaws are initiated and propagated along the interface. In consideration of the evolution of buckling morphologies under different stress states, their characteristics can be used to evaluate the interfacial adhesion.

2.3. Buckling profiles

As shown in Fig. 2a and b, the straight-sided buckle starts from an edge flaw, and then propagates along the straight side and curved front. The buckling surface topography looks like a half-penny (see Fig. 2b), which is similar to buckling above an