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Environmentally assisted debonding of copper/barrier interfaces

Ryan P. Birringer [a], Roey Shaviv [b], Paul R. Besser [c], Reinhold H. Dauskardt [a,*]

[a] Department of Materials Science and Engineering, Stanford University, Stanford, CA 94305, USA
[b] Novellus Systems Inc., 4000 North First Street, San Jose, CA 95134, USA
[c] GLOBALFOUNDRIES, 1050 East Arques Avenue, Sunnyvale, CA 94085, USA

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Abstract

Environmentally assisted debonding at Cu/barrier interfaces is reported for oxidizing and reducing environments. Both moist and dry oxidizing environments are considered, and the effects of different oxidizing species and their chemical activity on the rate of debonding of a Cu/SiN interface is quantified. The type of oxidizing species is shown to play a critical role in the kinetics of environmentally assisted debonding. Additionally, the effect of varying the activity and temperature of reducing hydrogen environments is investigated. The mechanisms responsible for environmentally assisted debonding of Cu/SiN and Cu/SiCN interfaces are elucidated using an atomistic bond rupture model. An activation energy for debonding of Cu/SiCN interfaces in a hydrogen environment is calculated. Finally, a connection between environmentally assisted debonding and the time-dependent dielectric breakdown properties of Cu interconnects is proposed.
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1. Introduction

Barrier films such as silicon nitride and silicon carbide are a critical component of Cu interconnect technologies due to their diffusion barrier properties, as well as their desirable chemical and mechanical characteristics. These barrier materials can be engineered to exhibit good adhesion to both Cu and adjacent dielectric materials and are highly resistant to moisture, alkali, and Cu diffusion [1]. Adhesion of barrier materials to Cu has long been recognized as critical for the long-term reliability of Cu interconnects, significantly influencing electromigration and stress migration performance [2–4], but nothing is known about the process of time-dependent environmentally assisted debonding of Cu/barrier interfaces. Additionally, the time-dependent dielectric breakdown (TDDB) properties of Cu interconnects are significantly degraded in the presence of moisture [5–7], and environmentally assisted debonding in the interconnect structure may contribute to this accelerated TDDB failure. Understanding these environmentally assisted processes provides valuable insight regarding how chemical environment and temperature influence the mechanical integrity of the Cu/barrier interface and the kinetics of defect growth.

Not all materials or interfaces are prone to environmentally assisted cracking or debonding. For example, both bulk SiN and SiC are essentially immune to moisture-assisted cracking at room temperature [8,9]. Surprisingly, however, in this study we demonstrate significant environmentally assisted debonding of Cu/barrier interfaces in both oxidizing and reducing environments. The existence of environmentally assisted debonding in moist and oxidizing environments illustrates the necessity of a hermetic barrier which prevents diffusion of these species to the interface so that such debonding does not occur. Additionally, the detrimental effects of reducing environments, specifically molecular hydrogen, are explored. Reducing environments are often used to remove Cu oxide prior to deposition of barrier layers [3,4], and molecular hydrogen

* Corresponding author. Tel.: +1 650 725 0679; fax: +1 650 725 4034.
E-mail address: dauskardt@stanford.edu (R.H. Dauskardt).

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may be present during annealing of these nitride films due to dissociation reactions [10,11].

1.1. Environmentally assisted debonding

The critical fracture energy of an interface (G_c) is largely insensitive to the environment and debonding rate, and this value is generally reported in adhesion studies of barrier films used in Cu interconnects [2–4,12]. Unlike critical debonding, environmentally assisted debonding arises from synergistic effects involving both mechanical loads and an environmental species. This is sometimes known as subcritical cracking or debonding and is generally due to a stress-enabled chemical reaction between a chemical species in the environment and strained bonds at the crack front [13–19]. Environmentally assisted debonding generally occurs at driving forces well below the critical fracture energy G_c of an interface and is a time-dependent process controlled by rate limiting steps at the crack front.

This environmentally assisted behavior is well known in a number of ceramics and glasses [12–16,18,19], including low-k dielectric materials [17,20], however, it has never been investigated in Cu/barrier materials. A quantitative understanding of this behavior in bulk materials or at bimaterial interfaces is essential in making accurate lifetime predictions and understanding the kinetics and extent of defect growth as a function of environment, temperature, and driving force.

A schematic of a typical environmentally assisted debonding curve containing a number of different regions is shown in Fig. 1. The curve quantifies the rate at which an interface debond or crack will grow as a function of applied crack driving force, and each region results from a different rate limiting step in the debonding process.

Region 1 represents a reaction-controlled region, where the kinetics are controlled by the rate of the chemical reaction at the crack front. The slope of this region is controlled by the dependence of the reaction on the applied driving force. A lower bound or threshold (G_{Th}) below which crack growth is assumed to be arrested may be present if the stress-enabled chemical reaction is not energetically favorable at driving forces below this value or if steric hindrance effects prevent the reactive environmental species from reaching strained bonds at the crack tip [16,17]. The threshold is particularly relevant when assessing defect evolution, as it represents the driving force above which an interface defect will continue to grow.

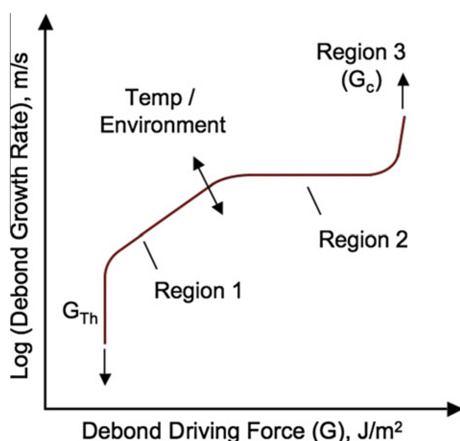

Fig. 1. A schematic of an environmentally assisted debonding curve illustrating different rate-controlled regions.

Region 2 represents a transport-controlled region, where the kinetics are controlled by diffusion of the reactive species to the crack tip. In this region the rate of chemical reaction at the crack front is higher than the rate at which the reactive species can be supplied to the strained bonds at the crack tip. For this reason the kinetics of debond growth are generally insensitive to the applied driving force and are mainly influenced by factors which affect the rate of diffusion [14,16–19]. The transport-controlled region may or may not be present, depending on the reaction rate and the mobility and concentration of the reactive species. The position of both the reaction- and transport-controlled regions, as well as the threshold, may be dependent on the activity of the reactive species, as well as the temperature.

As the driving energy for debonding is increased the critical fracture energy of the interface is eventually reached (Region 3). At this driving force the energy necessary to separate the interface becomes independent of the chemical environment. The critical value, however, may still be influenced by the temperature, as the yield properties and extent of plasticity in adjacent layers may be affected.

2. Experimental

2.1. Sample preparation

Two different sets of thin films were investigated in this study. The first sample consisted of an electroplated Cu film capped with a SiN barrier, while the second sample consisted of a Cu film deposited using physical vapor deposition (PVD) and a SiCN barrier cap. The first sample was fabricated by depositing a 50 nm PVD Cu seed layer onto 300 mm silicon substrates already containing a dense oxide and a Ta barrier film. Five-hundred nm of Cu was electroplated onto the seed layer and then annealed at 225 °C for 90 s. The Cu film was reduced to 200 nm total thickness using chemical mechanical polishing. A NH_3 reducing pretreatment was performed in order to reduce the surface of the Cu film immediately prior to the deposition of a 50 nm SiN dielectric diffusion barrier film by chemical vapor deposition (CVD). The film stack was then capped with a 1000 nm SiO_2 overlayer, as shown in Fig. 2a.

The second sample was fabricated by PVD deposition of a 37 nm Cu film onto 300 mm silicon substrates containing a 400 nm dense oxide and a Ta barrier film. This PVD Cu film was capped with a 35 nm SiCN film by CVD, as shown in Fig. 2b.

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