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Quantitative adhesion characterization of antireflective coatings in multijunction photovoltaics

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A B S T R A C T

We discuss the development of a new composite dual cantilever beam (cDCB) thin-film adhesion testing method, which enables the quantitative measurement of adhesion on the thin and fragile substrates used in multijunction photovoltaics. In particular, we address the adhesion of several 2- and 3-layer antireflective coating systems on multijunction cells. By varying interface chemistry and morphology through processing, we demonstrate the marked effects on adhesion and help to develop an understanding of how high adhesion can be achieved, as adhesion values ranging from 0.5 J/m^2 to 10 J/m^2 were measured. Damp heat (85 °C/85% RH) was used to invoke degradation of interfacial adhesion. We demonstrate that even with germanium substrates that fracture relatively easily, quantitative measurements of adhesion can be made at high test yield. The cDCB test is discussed as an important new methodology, which can be broadly applied to any system that makes use of thin, brittle, or otherwise fragile substrates.

1. Introduction

Solar power generation with high-efficiency multijunction photovoltaic (PV) cells has been an area of significant interest in recent years, as improved concentrator systems have advanced towards cost effective terrestrial deployment [1–3]. While CPV systems have held a clear advantage in conversion efficiency over traditional silicon PV, with efficiencies that have long exceeded 40% and even recently reaching as high as 46% [1,4,5], questions regarding long term reliability remain as availability of field use data is limited [1,6]. Furthermore, environmental degradation is of greater concern as cells are subjected to higher incident flux of ultraviolet light and larger temperature cycles.

The application of multijunction PV cells, with their complex layered structures, in terrestrial applications requires an improved understanding of thermomechanical reliability and testing metrologies as the basis for improved lifetime predictions [7]. The ability to establish bankability via accelerated life testing stands as a key hurdle to any new solar technology if it hopes to overcome the relative safety provided by silicon panels [3,8–11].

While there have been studies of performance degradation [12–15], little has been done to understand and quantify the underlying materials properties that lead to degradation. Of particular concern is the adhesion of the many internal interfaces including those involving backside metal contacts, substrates, active layers, antireflective (AR) coatings, and frontside metal gridlines, as cracking and delamination of these materials has been cited commonly as a primary failure mode [14,16–18]. The specific effects of stressing parameters that include mechanical stress, temperature, and humidity from terrestrial environments are also of significant interest [12,14,16].

Thus far the lack of reliable quantitative testing methods has prevented any meaningful study of adhesion within multijunction PV structures. In particular, studies of AR adhesion in the past have commonly been limited to qualitative or indirectly-quantitative methods such as the cross-hatch test, wiping test, or tape-peel test [19–21]. In the course of exploring techniques for measuring adhesion on such fragile, thin substrates in this study, several well-known thin film adhesion testing methods were applied. These included the dual cantilever beam (DCB), single cantilever beam (SCB), and four-point bend (4PB) techniques, all of which are commonly used to quantify the fracture of thin films [22–26]. However, the existing methods were prone to fracture within the substrate, resulting in very low sample yield and necessitating the creation of a new testing method.

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In this study, we develop a composite dual cantilever beam (cDCB) adhesion test, and apply it to the measurement of adhesion of AR coatings deposited on top of state of the art multijunction PV cells. As is fairly typical in multijunction PV systems [18,27,28], Al_2O_3/TiO_2 AR layers are used, and are deposited via both high and low energy deposition methods. Processing conditions were varied including the use of adhesion promoting layers, to demonstrate the sensitivity of the cDCB method for quantifying improvements in adhesion. The effect of Damp Heat aging conditions on interfacial adhesion was also examined [29]. While this study focuses specifically on adhesion of AR layers deposited on multijunction photovoltaic cells, the methodologies can be broadly applied to any system that makes use of thin, brittle, or otherwise fragile substrates in order to make high-yield quantitative measurements of adhesion.

2. Experimental

2.1. Antireflective coating deposition and aging

Antireflective (AR) layers (Al_2O_3/TiO_2) were deposited atop epitaxially grown multijunction photovoltaic cells on 180 μm germanium substrates (Spectrolab). A schematic of these test structures is shown in Fig. 1. Each type of AR layer is identified with number of layers (2 or 3), deposition method (L-low energy, or H-high energy), and type of adhesion layer (A, B, or C), and is hereafter referred to by a 3-digit signifier such as '3LA'. In one set of specimens, deposition of the AR layers was carried out as normal, while in the other a lower temperature heat treatment was applied. Higher temperature heat treatments have been qualitatively observed to significantly increase adhesion of the AR layers, and thus provides an opportunity for methodology validation. Following deposition, a series of wafers were subsequently exposed to accelerated aging conditions at 85 °C/85% relative humidity in a Thermal Products Solutions Inc. Blue M FRS-361F chamber for specified durations, up to 2000 h. Damp Heat is applied in excess of the 1000 h requirement in the IEC 62108 module design qualification and type approval test. The Damp Heat test, however, well exceeds the expected moisture concentration typical to the interior of a CPV module.

2.2. Adhesion testing

2.2.1. Dual cantilever beam

DCB specimens were constructed by adhering a blank germanium wafer to the wafer of interest, and dicing the resulting stack into 5 mm × 50 mm beams. An initial crack length of 10 mm was created by deposition of a thin (~100 nm) gold release layer. The specimens were loaded in tension under displacement control, and the load, P, versus displacement, Δ, data was recorded as the crack naturally propagated from the initiated crack into the relevant interfaces. All adhesion tests were performed using a thin-film cohesion testing system (Delaminator DTS, Menlo Park, CA). The adhesion energy, G_c (J/m^2), was measured in terms of the critical value of the applied strain energy release rate, G. G_c can be expressed in terms of the critical load, P_c, at which debond growth occurs, the debond length, a, the plane strain elastic modulus, E', of the substrates and the specimen dimensions; width, B and half-thickness, h. Here, the $(E'h^3)$ term is grouped together and represents the elastic bending stiffness of the beams. The adhesion energy is then typically calculated from

$$G_c = \frac{12P_c^2a^2}{B^2\left(E'h^3\right)} \quad (1)$$

2.2.2. Composite dual cantilever beam

The dual cantilever beam test has been applied successfully to many thin film systems, but low test yield can occur if the fracture toughness of the beams is low, resulting in beam fracture rather than delamination at the interface(s) of interest. To overcome this challenge, composite dual cantilever beam specimens were constructed by adhering tough, fracture resistant beams to standard DCB specimens. These new test structures are shown in Fig. 2, with each composite beam consisting of 180 μm thick germanium bonded to 820 μm thick titanium (Grade 5 alloy, 5 × 50 mm) using a high-strength epoxy (Loctite E-20NS) under high pressure. The epoxy was cured at room temperature to avoid developing stress from mismatched thermal expansion.

Titanium is chosen as an ideal material for this test structure because it allows large elastic deflection prior to yielding or fracturing, and thus can be used to measure larger adhesion energies, particularly in the case of mechanically fragile substrates. The maximum elastic deflection (before plastic yielding) can be expressed as

$$\delta_{max} = \frac{a^2}{9G_c}\left(\frac{\sigma_y^3}{E^2}\right) \quad (2)$$

where the ratio σ_y^3/E^2 was calculated for several candidate beam materials (in MPa), including steel (0.023), aluminum (0.019), PMMA (0.039), and titanium (0.047).

It is also desirable to select a material to maximize the applied value of G for a given load, in order to reduce the maximum load required for testing. The maximum value of G_c for a given load (before plastic yielding) is given by

$$G_{max} = \frac{h}{3}\left(\frac{\sigma_y^2}{E}\right) \quad (3)$$

where the ratio σ_y^2/E is the maximum elastic energy density, which was calculated for several candidate beam materials (in MPa), including steel (2.5), aluminum (2.3), PMMA (1.6) and titanium (7.2). By both δ_{max} and G_{max}, titanium is the best commonly available material to use as the reinforcing beams in a cDCB.

The adhesion energy, G_c, can be calculated as before for a DCB test, but replacing the elastic bending stiffness with an equivalent bending stiffness for the composite bi-layer substrate, $(E'h^3)_{eq}$

$$(E'h^3)_{eq} = \frac{E_{sub}^2t^4 + E_{Ti}^2h^4 + 2E_{sub}E_{Ti}th\left(2t^2+3th+2h^2\right)}{E_{sub}t + E_{Ti}h} \quad (4)$$

where E_{sub} and E_{Ti} are Young's moduli of the substrate and

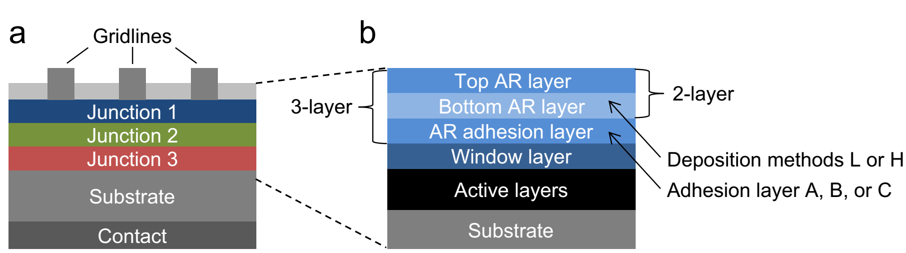

Fig. 1. Schematic representations of (a) a multijunction photovoltaic structure and (b) the test structures created for this study.