



## Effects of substrate condition on calcium corrosion and its role in the calcium test for water vapour transmission rate



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### ABSTRACT

The electrical Ca test is a standard method for the estimation of the water vapour transmission rate through the encapsulation barriers. Here, we discuss the corrosion process of thin Ca films under different preparation conditions, and the impact of the substrate temperature. It is demonstrated that without vacuum drying of substrate, the Ca films corrode from the substrate side, even though they are encapsulated. The thickness dependence revealed its influence on lifetime, but not on its electrical properties. These observations reveal the strong impact of the Ca fabrication technology and measurement procedure on the corrosion process of Ca thin films.

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### 1. Introduction

Organic electronics devices such as organic field-effect transistors (OFETs), organic light-emitting diodes (OLEDs) or organic solar cells (OSCs) are promising candidates for the next generation of electronic devices, where low-cost and printable methods are available for device fabrication [1,2]. Recent progress showed that energy conversion efficiencies, achieved using organic materials, are comparable to inorganic materials such as amorphous silicon [3]. However, the long-term operation stability of these organic devices is far below what would be considered acceptable for commercial products, with the replacement costs negating the cost savings from inexpensive manufacturing. This is because organic materials are more susceptible to degradation from oxygen and water than inorganic materials. A number of studies have been carried out showing that the stability/degradation issue is quite complex and not yet fully understood, though gradual progress has been made.

As a result, device encapsulation technologies have been developed to provide barriers to water vapour and oxygen [4,5]. Many research efforts exist to develop barrier materials that are both flexible and transparent, but which are still inexpensive enough to make the final product cost competitive. To characterize the

quality of the barrier, the water vapour transmission rate (WVTR) is evaluated. There are suggested various test methods, such as the cup test [6], the isostatic method [7], the mass spectroscopy method [8], or the Ca test [9,10]. The first two mentioned are standards of the American Society for Testing and Materials (ASTM). However, the sensitivity is lower than requirements for organic electronics devices [4]. On the other hand, mass spectroscopy is sensitive enough, but the drawback is high initial investment. Since Ca is extremely sensitive to oxygen and water [11], an alternative method, based on the evaluation of Ca thin film degradation, has been proposed. This method is widely used and few modifications have been introduced [12–15]. In other words, the Ca corrosion is not only interesting for Ca applications, but also as a reference corrosion process, which can be used for comparison with other degradation processes.

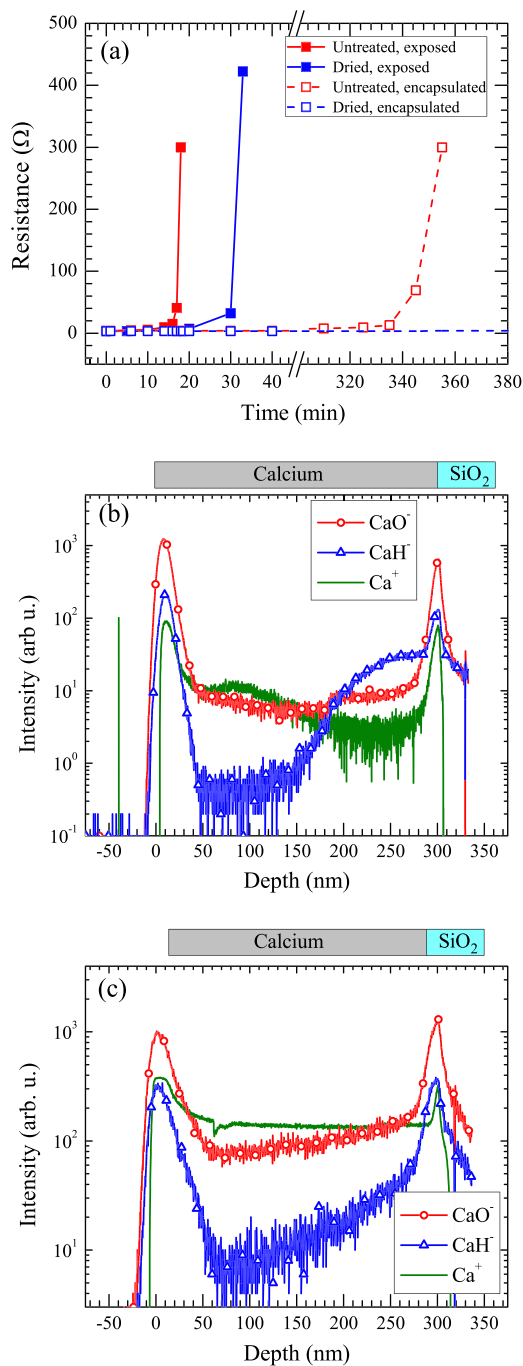
In the Ca test, the resistance change of Ca thin films is used to evaluate the degree of Ca corrosion, because oxidized Ca salts have very low conductivity. Assuming homogeneous corrosion of Ca thin film from the surface, and a step change in the conductivity between corroded and virgin Ca, the total film resistance  $R$  is inversely proportional to the thickness  $h$  of remaining conductive Ca,

$$R = \frac{\rho \ell}{wh}, \quad (1)$$

where  $\rho$  stands for the Ca resistivity, and  $w$  and  $\ell$  are the thin film width and length, respectively. However, the sheet resistance  $R_{sq} = \rho/h$  is commonly used to obtain a film-area independent

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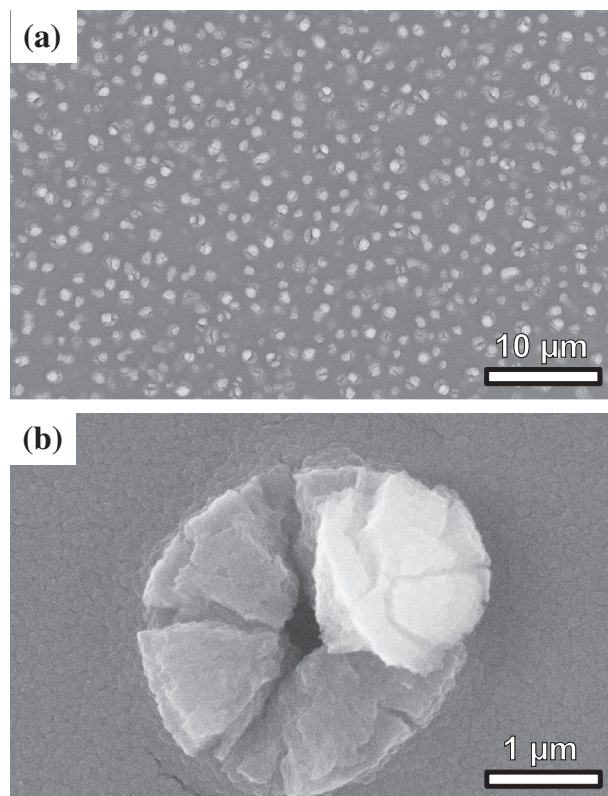
E-mail address: [martin.weis@stuba.sk](mailto:martin.weis@stuba.sk) (M. Weis).



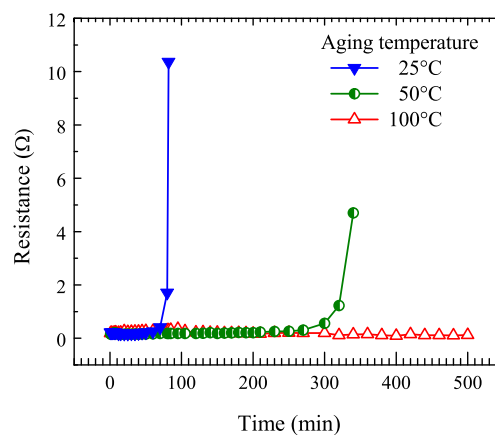
**Fig. 1.** (a) Temporal evolution of the resistance of 300 nm thick Ca film deposited on untreated or vacuum dried SiO<sub>2</sub> substrate. For comparison, the data for encapsulated films are also shown. SIMS depth profiles of Ca films encapsulated by Au layer. SiO<sub>2</sub>/Si substrates were (b) untreated and (c) dried in vacuum prior to the Ca deposition. Sputter depth has been shifted to zero for the Ca film surface position.

variable with unit Ω/sq, where sq stands for ‘square’ abbreviation. Strictly speaking such a unit (Ω/sq) does not exist, but it is commonly used since it can be measured directly. That is, the resistance and the sheet resistance is a primary result, whereas the resistivity is a secondary result, which requires the determination of the film thickness.

The chemical reactions of Ca in ambient atmosphere are assumed as follows,



**Fig. 2.** SEM SE surface morphology of 300 nm thick Ca film deposited on untreated surfaces in initial state of degradation: (a) large-area image, and (b) detail of one of the defects.



**Fig. 3.** Temporal evolution of the Ca film resistance for various aging temperatures.



It should be noted here that the reaction with water vapour at room temperature is far more significant than the reaction with oxygen [13,11,17]. Differences in obtained results by different research groups [10,13,15] are usually discussed in the meaning of the edge-seal limitation, since the quality of Ca films is assumed to be identical. However, it has been reported that for the same encapsulation various laboratories estimated WVTR using a Ca test in the range of  $0.48\text{--}1.56 \times 10^{-4} \text{ g/m}^2/\text{day}$  [18]; *i.e.* the values differ by a

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