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CO₂-based test for the detection of defects in oxygen barrier layersMariagrazia Troia¹, Andreas Schulz¹, Matthias Walker¹, Thomas Hirth²¹IGVP, University of Stuttgart, Stuttgart, Germany ²KiT, Karlsruhe, Germany

mariagrazia.troia@igvp.uni-stuttgart.de

Barrier layers' performances for food packaging and encapsulation of electronic devices are severely limited by the presence of punctual micro- and sub-micro-metrical defects. In order to determine their density in layers obtained by means of Plasma Enhanced Chemical Vapor Deposition, their origin and overall effect on oxygen transmission rate, a new non-destructive test has been developed and optimized.

Thin films acting as oxygen barrier layers have been deposited on thin PET foils by means of an Electron Cyclotron Resonance (ECR) low-pressure, microwave-sustained plasma, with HMDSN/O₂ gaseous feeds.

The coated sample in the sealed testing cell is interposed between a pure CO₂ atmosphere and a saturated limewater solution: CO₂ permeates preferentially through the pinholes in the barrier layers, causing the precipitation of calcium carbonate crystals on top of the defects. Real-time analysis is performed by means of an optical microscope on top of the cell's transparent lid. The position of the crystals and the underlying defects can be later retrieved for further investigations at a scanning electron microscope by means of markers placed on the sample itself.

Average defect densities for different substrates and for a wide range of thicknesses and chemical compositions of silica-like barrier layers have been calculated and compared to their respective oxygen transmission rate and barrier improvement factor. The time required for crystals to appear on top of defects (i.e. the time required for CO₂ to permeate through the whole length of the defect) has also been calculated and correlated to the rise time of the oxygen and CO₂ permeation curves.

Repeated oxygen permeation analysis, after the formation of crystals on the samples surface, show remarkably reduced transmission rates, thus confirming the growth of crystals directly on top of pinholes and hinting at their formation mechanisms, as well as delimiting the applicability of a purely defect-based permeation.

Keywords

Barrier layers

Pinholes

Microwave plasma

ECR