

Multilayer Coatings for Polycarbonate Glazing: Influence of Mechanical Properties on the Abrasion Performance of the Coating System

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ABSTRACT

Coated polycarbonate (PC) is a leading engineering thermoplastic used in automotive glazing for replacing laminated glass. Mechanical properties of multi-layer coating systems were investigated using a nano-indenter and the fracture behavior of coating during nano-scratch was studied employing scanning electron and atomic force microscopy. A set of coated samples was prepared, with two layers, namely Layer-1 and Layer-2. Layer-1 was applied directly to the PC substrate and used as adhesion promoter. Layer-2 was prepared with different mechanical properties. Abrasion performance of the coated system was characterized using an ASTM abrasion test methodology. Regression analysis was performed to establish correlation between the mechanical properties of the coating system and its abrasion performance. Fracture behavior of the coating systems and their plausible relationship with abrasion performance was also discussed. From the above study, it was observed that the abrasion performance of a 2-layer coating system mostly depends on mechanical properties of the sufficiently thick Layer-2. For multilayer coating it was observed that the hardness, hardness over modulus and critical load to fracture are the dominant factors for optimum coating performance.

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INTRODUCTION

Polycarbonate (PC) automotive glazing technology has matured enough to replace glass used at selected locations to exploit the benefits of high ductility, lower weight and easy manufacturability of plastics [1]. However, there is scope for further improvement in the area of abrasion and weatherability of PC glazing. Generally, in order to improve both abrasion and weatherability, multilayer hard coatings are being used on PC. Acrylic, siloxane and vapor deposited glass-like coatings are usually used to protect the PC surface. While developing a coating system with different functional layers it is important to know the effect of the properties for each layer on the performance of the coating system.

Various analytical tools are being used to assess the coating properties and adhesion of coatings to their substrates [2, 3]. Nano-indentation and nano-scratch techniques are used

extensively to understand the coating properties in nano-scale [4, 5]. Scratch testing is a simple test that gives quantitative information on the coating performance without complicated sample preparations.

In this paper, we have studied the surface mechanical properties of multilayer coating systems using nano-indentation and nano-scratch techniques. Surface mechanical properties (hardness, modulus and critical load to fracture) were calculated and correlated with critical abrasion testing methodology. Microscopic techniques were employed to further investigate the coating fracture behavior. Influence of the properties of Layer-2 on the abrasion performance of coating system is discussed.

II. EXPERIMENTAL

A. Samples

Schematic of a typical 2-layer coating system is shown in Figure 1. In this system, the PC substrate is initially coated with a thin ($\sim 1 \mu\text{m}$) layer of acrylic based coating (Layer-1) which helps adhering between the PC substrate and hardcoat layer. Siloxane based hardcoats (Layer-2) were coated on top of the primer layer using a flow coating method. Thicknesses of Layer-2 were in the range of $3\text{--}12 \mu\text{m}$. Coated samples were then annealed at different times ($t_1\text{--}t_5$) and temperatures ($T_1\text{--}T_5$) in order to cure them at different levels, at the following temperatures $T_5 > T_4 > T_3 > T_2 > T_1$ and times $t_5 > t_4 > t_3 > t_2 > t_1$ respectively. It is expected that the as-treated coatings on PC would have different mechanical properties.



Figure 1. Schematic of a typical two-layer coating system.

B. Nano-Indentation and Nano-Scratch

Nano-indentation and nano-scratch experiments were conducted using the Nano-Indenter[®] XP (Agilent System Corporation, Oak Ridge, TN). A three-sided Berkovich diamond tip with end diameter $\sim 100\text{nm}$ was used for both indentation and scratch experiments. Using this nano-indenter, the maximum distance allowed for the indenter to travel normal to the sample surface is about 1.5 mm . Over this entire range, displacement resolution is less than 0.1 nm . The maximum load capacity for this system is 500 mN with a precision of less than $1 \mu\text{N}$. The load and displacement data obtained in the nano-indentation tests were analyzed according to the model developed by Oliver and Pharr [6]. Continuous stiffness measurement (CSM) technique was used for depth dependent hardness and modulus analysis [7]. CSM technique is ideal for investigating a multilayer coating system in order to measure coating properties independent of substrate properties. The CSM option works by imposing a small oscillation on the indenter during loading. The amplitude of the force oscillation relative to the amplitude of the displacement oscillation allows one to calculate stiffness as a continuous function of penetration depth. Indentation was done with a constant strain rate of $0.05/\text{s}$. The properties of the film alone are calculated by averaging over an appropriate displacement range and the range depends on the thickness of the film. In this study, measurement of modulus of a coating is done at approximately 10% of the coating thickness and hardness at 18–20% of the coating thickness in

order to measure coating properties with minimal substrate effect.

In a typical scratch test, the normal force on the sample is controlled and can be held constant, increased, or decreased at a linear rate. In this experiment, progressive load ($0\text{--}120\text{mN}$) was used as it helps in increasing the level of stress on the coating by the indenter as load increases. If tensile stress increases beyond the cohesive energy of coating, it starts cracking. Scratch velocity was held constant throughout the scratch experiment at $10 \mu\text{m/s}$. It is prudent to mention here that the coating fracture behavior is also heavily dependent on the tip size and geometry. A typical scratch experiment was performed in four stages; an original profile, a scratch segment, a residual profile and a cross profile [8]. Typical measured parameters are scratch width, scratch depth, penetration curves, scratch fracture and delamination behavior of coatings.

C. Abrasion Testing

Abrasion testing was performed following ASTM D1044d. This abrasion test is a regulatory requirement for safety glazing. The instrument used is the Taber[®] Rotary Platform Abrasion Tester (Model 5155, Taber Industries, USA). The arms that hold the abrasive wheels have free rotation with the load of 500g on each wheel arm. The abrasive wheels are manufactured by Taber Industries and are specified in regulations ECE R43 and ANSI Z26.1 as CS-10F wheels. Samples were cleaned before and after abrasion with Isopropyl Alcohol followed by deionized water. Samples were then abraded for 1000 revolutions at $23^\circ\text{C}/50\%\text{RH}$. Percent Haze was measured before and after abrasion. Percent delta haze is then measured and compared with various mechanical properties.

D. Optical and Scanning Electron Microscopy (SEM)

Optical microscopy was performed using the Leica DMI3000 M (Leica Microsystems GmbH, Germany). Scanning electron microscopy (SEM) is a commonly used technique to study the surface topography. In this study, SEM is used to investigate the fracture morphology of nano-scratched surface. The sample was coated with gold to avoid charging. Imaging was done on environmental SEM Quanta 400 (FEI, NL). Images at appropriate magnifications were captured on fractured surfaces created during the nano-scratch experiment.

E. Atomic Force Microscopy (AFM)

Atomic force microscopy (AFM) was employed to investigate the surface morphology of the scratch marks on coating system. AFM experiment was performed using Bruker AFM (NanoScope V). The AFM scanner was calibrated by imaging a grating of defined spacing's before imaging the samples. The height images were recorded in the tapping mode of AFM using silicon tip