Microstructural evolutions and thermomechanical properties of enameled glass for automotive applications

Antoine Lejeune*†[1,2], Jérôme Chevalier[1], Sylvain Meille[1], Philippe Steyer[1], Laurent Gremillard[1], and Juliette Jamart[2]

[1]Matériaux, ingénierie et science [Villeurbanne] – Université Claude Bernard Lyon 1, Institut National des Sciences Appliquées de Lyon, Centre National de la Recherche Scientifique – France
[2]Saint-Gobain Research Compiègne – Saint-Gobain Research Compiègne – France

Abstract

In automotive applications, black enamels are mostly used for their opacifying property, especially on soda-lime-silica glass substrates for laminated products such as windshields. Enamels are composed of low-temperature sinterable Bi2O3 − B2O3 − ZnO − SiO2 − N2O glass frit systems (N being an alkali element), black pigments, and mineral additives. Final properties and microstructure of enamel coatings are achieved through pre-firing and subsequent bending of automotive glazing. Through these process steps, enamel undergoes several microstructural transformations: sintering, glass transition, crystallization, porosity evolution... potentially leading to a weakening of the glass substrate.

These transformations are highly influenced by the chemical composition of enamel, the respective component proportions, and their particle size distribution. The thermomechanical properties of the enamel coating such as thermal expansion coefficient and Young's modulus may vary to a large extent. The resulting thermomechanical properties mismatch between enamel coating and SLS glass substrate is of great importance for the resulting weakening phenomena, but also for the optical quality in areas where high optical quality in transmission is required.

In the present work, two chemically different enamels are characterized using XRD, SEM, EDS, X-ray tomography, DSC, bending tests, color and curvature measurements to provide correlations between composition, microstructure, and mechanical properties. *In-situ* characterization by XRD, environmental-SEM and curvature evolutions enable to monitor the evolution of the enamel microstructure during thermal treatments, allowing correlations with its mechanical behavior according to Stoney's assumptions. Combination with ex-*situ* characterization in X-ray tomography and XRD led to a global understanding of the impact of thermal processing on porosity and crystallization ratio. These transformations were also characterized through the evolution of thermomechanical properties by curvature measurement.
It was found that initial particle size of enamel glass frit had a significant impact on porosity after sintering. Final microstructure after sintering, originating from initial particle size and

*Speaker
†Corresponding author: antoine.lejeune@saint-gobain.com

thermal treatment, has a low but still significant impact on failure mechanism of enameled glass. Moreover, it was shown that chemical composition of enamel powder (glass frit systems, mineral fillers, pigments) deeply influenced the sintered enamel microstructure and therefore their strength and mechanical properties as well.

Keywords: Enamel, thermomechanical properties, in, situ, crystallization, microstructure.