BURNING OF THE PVB BINDER DURING WINDOW GLASS SINTERING ASSISTED BY AN ELECTRIC FIELD

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Sintering is a significant route for glass and glass-ceramic manufacturing; however, flash sintering of vitreous materials has only been reported for a few compositions. Some of us successfully achieved the flash sintering of commercial window glass particle compacts in the soda-lime-silica (SLS) system a few years ago. Among the challenges, the samples showed blackening upon flash sintering. The blackening appeared to be resulting from the use of a binder. Still, the mechanism of blackening and whether binder remotion is enhanced under the application of an electrical field are open questions. To answer these questions, we characterized by SEM-EDS and XPS some flash-sintered samples that showed blackening. The glass was powdered, sieved under 7 µm granulometry, and finely mixed with 2 wt% PVB (polyvinyl butyral) as a binder. The mixture was uniaxially pressed in a die with a dog-bone shape into compacts approximately 2.2-mm thick, 3.3-mm wide, 28 mm in length, and 0.6 relative density, calculated from their weights and geometries. Non-isothermal heat treatments were performed in a horizontal tubular electric furnace manufactured in-house, with a temperature stability of ± 1 °C measured by a thermocouple close to the sample. The samples were suspended in the furnace treatment zone by two platinum wires connected in central holes through their circular ends. They were also used as electrodes for the electric field application by a DC power source. The initial length between the electrodes was 20 mm. The samples were heated at 10 °C/min, first to 500 °C for debinding for 60 min, and subsequently with different applied voltages (0, 100, 200, 300, 400, and 500 V) up to a temperature at which flash was detected. The furnace heating and applied voltage were turned off at the beginning of the flash at a given cutoff current, and the samples were cooled in the furnace to room temperature. The voltage and current in the samples were measured during sintering. Digital images were simultaneously captured, recording a stop-motion movie of the fast-shrinking samples during sintering. The linear shrinkage was characterized by measuring the length between the holes through the sample ends in the recorded images relative to the initial length. The compacts underwent flash sintering at 300 V and above. The traditionally sintered (0 V) sample presented maximum shrinkage at 720 °C, whereas the samples treated at 300, 400, and 500 V achieved maximum shrinkage abruptly at ~684, 665, and 640 °C, respectively. SEM-EDS of fracture and polished surfaces did not show any pattern due to carbon distribution. As shown in Fig. 1, C-1s XPS on cross-sectional surfaces suggests that they fractured along the binder-burning residue. Since XPS only measures to a depth of ~5 nm, the glass underneath was not detected, except for a small Si peak. The blackening is consistent with the decomposition of PVB in an oxygen-deficient environment, mostly forming C-C (possibly carbon black). Our results show that PVB binder burning is impaired, and its residue is trapped in the glass bulk during electric field-assisted sintering at lower temperatures and oxygen-deficient atmosphere.

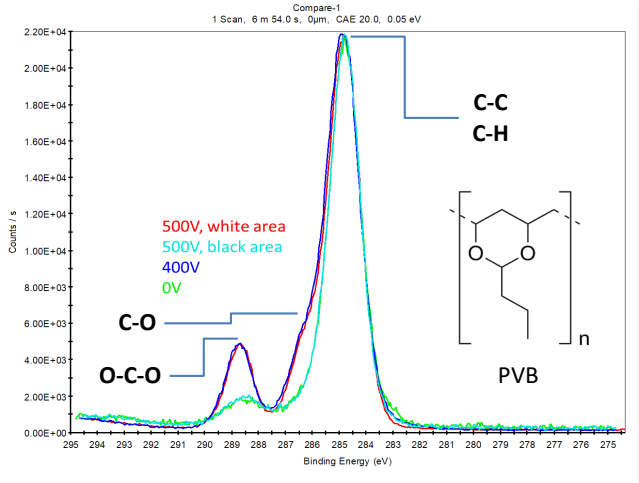

Figure 1 – C-1s XPS spectra of fractured surfaces of SLS glass powder compacts sintered conventionaly (0 V), and flash sintered at 400 and 500 V. The 500-V sample was analyzed at one of its extremities not affected by the electrical current (white), and in its central region transversed by the electrical current (black). The PVB structure is shown in the insert.