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DC-electro softening in soda lime silicate glass: An electro-thermal analysis

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ABSTRACT

DC-electric field-induced softening was investigated in soda lime glass. The application of a DC current causes a migration of Na ions towards the cathode resulting in the formation of a sodium depleted layer close to the anode where a localized voltage drop ignites electrical arcs through the glass. This effect is strongly asymmetric with respect to the applied DC polarity and, at the anode, it induces strong photoemission (optical transition of alkali elements) sharp rise in temperature and increased electrical resistance. It appears that electrolytic effects and sodium migration play a fundamental role as triggering mechanisms of DC-electric field-induced softening.

Recent studies have shown that the application of an external DC-electric current through a green body results in an extraordinary reduction in both firing time and sintering temperature [1–9]. This promising consolidation technology is known as flash sintering and is characterized by three unusual features: a sudden electrical resistivity drop for the ceramic [10,11], rapid densification [1] and photoemission [12,13]. More recently, McLaren and co-workers have shown that a similar process can be applied to dense bulk alkali-containing silicate glasses at temperatures even lower than 400 °C; the DC-field application results in a sharp resistivity drop and discrete photoemission lines with a large decrease in flow stress [14]. For this reason, the phenomenon has been referred as “Electric Field-Induced Softening” (EFIS). Even if the mechanism underpinning EFIS has not been fully understood, it could allow new forming glasses [15,16] and glass- ceramics [17–19].

In the present work, we propose a more critical investigation of EFIS where thermal imaging analysis, electrical conductivity measurements and optical emission spectroscopy were applied to commercial soda lime glass. In particular, we considered localized effects occurring at the electrodes.

Well characterized microscope slides (Menzel glasses, Agar scientific, nominal composition: 72.2 SiO_2 - 14.3 Na_2O - 1.2 K_2O - 6.4 CaO - 4.3 MgO - 1.2 Al_2O_3 - 0.3 SO_3 wt%) were cut into rectangular specimens ($\approx 12 \times 2.5 \times 1.5$ mm^3) by a diamond saw. Two Pt wires were wrapped around the sample narrow edges and employed as electrodes. Experiments were carried out both with and without the addition of a Pt-based conductive paste (Gwent, C60903P5) to enhance and reduce the electrode-glass contact surface, respectively. Electrodes were connected to a DC power supply (TTi, PLH250-P) and to two multimeters (Mercury, MTTR01) to record voltage and current concurrently. The samples were then preheated using a custom built electric furnace ($T = 700 \pm 15$ °C). A thermocouple was placed close to the sample (≈5–8 mm from the sample center) to monitor the local temperature before applying an electric field. An optical fiber connected to a spectrometer (Avantes Starline, AvaSpec-2048) was positioned ≈5 mm from the anodic region; the obtained spectra were analyzed using the Plasus Specline software (the procedures are analogous to those reported in [20]). The experiments were visually recorded using a digital camera (Canon, Legria HF-R48) and an IR thermo-camera (FLIR, 655sc) both at ≈30 cm from the sample. EFIS treatments were carried out with an applied potential of 200 V and a current limit of 50 mA.

To investigate possible variations and differences in electrical conductivity around the electrodes and within the bulk, 4 point conductivity measurements were carried out. In this case, the sample was symmetrically drilled at 4 points along its gauge length: the distance

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between the two external holes was ≈8 mm, while the internal ones were at ≈4 mm. Four Pt wires were inserted into the cavities and the gap was filled with powder of the same glass. The test sample was then heated to 700 °C for 1 h allowing the powder to consolidate. Thus, the metal electrodes were truly embedded within the glass specimen during the EFIS experiments, achieving the best possible contact of the Pt-glass. The external wires were connected to a DC power supply (Sorensen DLM 300-2) with an applied voltage of 200 V and current limit of 50 mA. Multimeters (Keithley 2100) were used to collect the potential drops in the different portions of the sample gauge length and to register the current (a sketch of the circuit is shown below in Fig. 4(a)). A Nabertherm P330 furnace was used to perform the EFIS experiments under isothermal conditions at 700 ± 10 °C. Finally, EDS analyses were carried out using a JEOL IXRF SYSTEMS 500 with Iridium Ultra software. The analysis were carried out within a SEM Jeol JSM5500 using 20 kV electrons.

In-situ observations of DC-EFIS involve an asymmetric photoemission both with and without the employment of the Pt paste, the anodic region being much brighter than the cathodic one in both the testing conditions. As an example, Fig. 1 shows images and temperature

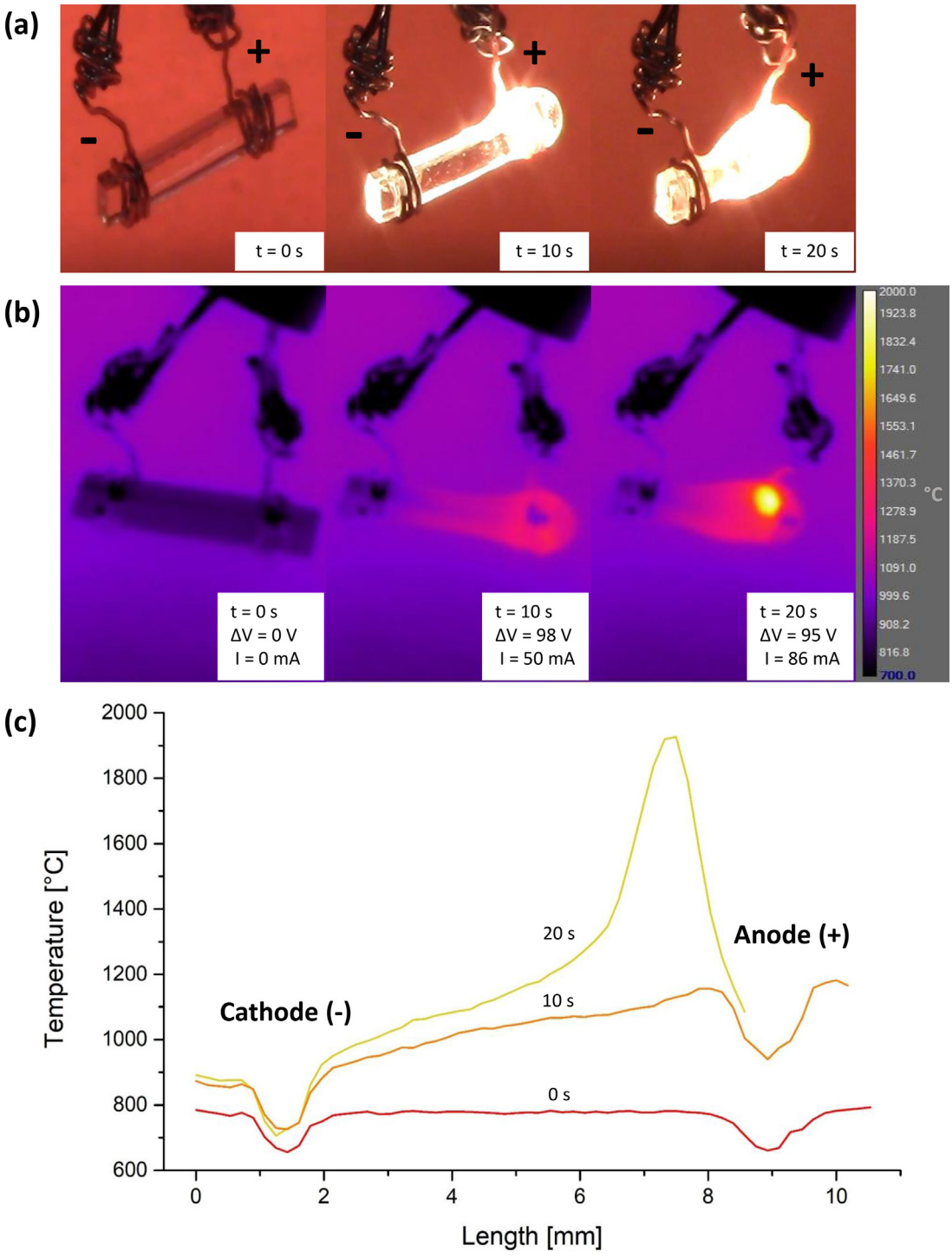

Fig. 1. Video stills taken every 10 s using a digital camera (a) and the IR-thermo-camera (b) upon an EFIS experiment. Temperature profiles relative to the images above collected along the gage length (c). Time = 0 s corresponds to the instant when the power was applied.