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Nondestructive Hardness Assessment of Chemically Strengthened Glass

Geovana Lira Santana [1], Raphael Barbosa [2], Vinicius Tribuzi [2], Filippo Ghiglieno [2], Edgar Dutra Zanotto [1], Lino Misoguti [3] and Paulo Henrique Dias Ferreira [2,*]

[1] Graduate Program in Material Science and Engineering, Department of Materials Engineering (DEMa), Federal University of São Carlos, Rodovia Washington Luís, km 235, São Carlos 13565-905, SP, Brazil; geovanalira@estudante.ufscar.br (G.L.S.); dedz@ufscar.br (E.D.Z.)

[2] Physics Department, Federal University of São Carlos, Rodovia Washington Luís, km 235, São Carlos 13565-905, SP, Brazil; raphaelbarbosa@estudante.ufscar.br (R.B.); tribuzi@ufscar.br (V.T.); filippo.ghiglieno@df.ufscar.br (F.G.)

[3] São Carlos Institute of Physics, University of São Paulo, Av. Trabalhador São-Carlense, 400, São Carlos 13566-590, SP, Brazil; misoguti@ifsc.usp.br

* Correspondence: paulohdf@df.ufscar.br

Abstract

Chemically strengthened glass is widely used for its remarkable fracture strength, mechanical performance, and scratch resistance. Assessing its hardness is crucial to evaluating improvements from chemical tempering. However, conventional methods like Vickers hardness tests are destructive, altering the sample surface. This study presents a novel, rapid, and nondestructive testing (NDT) approach by correlating the nonlinear refractive index (n_2) with surface hardness. Using ultrafast laser pulses, we measured the n_2 cross-section via the nonlinear ellipse rotation (NER) signal in Gorilla®-type glass subjected to ion exchange (Na^+ by K^+). A microscope objective lens provided a penetration resolution of ≈5.5 μm, enabling a localized NER signal analysis. We demonstrate a correlation between the NER signal and hardness, offering a promising pathway for advanced, noninvasive characterization. This approach provides a reliable alternative to traditional destructive techniques, with potential applications in industrial quality control and material science research.

Keywords: chemically strengthened glass; nonlinear refractive index; nondestructive testing (NDT); nonlinear ellipse rotation (NER); ultrafast laser pulses

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1. Introduction

The development of novel glass is a central focus in materials science and engineering, driven by applications ranging from domestic and artistic uses to advanced optical, electronic, and medical technologies. Over the past two decades, the demand for thin, lightweight, and mechanically strengthened glass has grown significantly, particularly for specialized applications such as portable electronic device displays, solar panels, and car windshields [1]. To achieve these enhanced mechanical properties, the ion exchange process (IOX) has been employed as a method of chemical tempering. This process involves replacing smaller alkali ions in the glass surface with larger ones from a molten salt bath. The difference in ion sizes induces compressive surface stress, significantly improving the glass's fracture resistance while maintaining excellent optical transparency. Consequently, there has been notable growth in the investigation of ion exchange strengthened

(ION-XS) glass, particularly because of its relevance in mechanical performance and optical applications [2].

ION-XS glass also presents promising prospects in nonlinear optics, serving as a substrate for advanced photonic devices, as evidenced by successful waveguide production [3,4]. The most common approach for measuring the nonlinear refractive index (n_2) is the Z-scan technique. While effective for distinguishing nonlinear absorption (NLA) and nonlinear refraction (NLR), this method lacks the spatial resolution necessary to resolve these effects within the material.

A novel method, nonlinear ellipse rotation (NER), offers particular promise because it can determine local changes in the nonlinear refractive index with high spatial resolution (on the order of the Rayleigh parameter) when a tightly focused laser beam is used—an advantage not achievable with the Z-scan technique [5]. Theoretical studies indicate that different contributions can be distinguished using this technique, including pure nonresonant electronic, molecular orientational, thermal, populational, and electrostriction effects [6]. This approach is particularly significant for understanding the magnitude and depth-dependent behavior of nonlinear refraction (n_2) [7,8], especially considering that the sodium–potassium concentration variations resulting from ion exchange treatments affect n_2 values. In this context, nonlinear microscopy has emerged as a potential tool for measuring local nonlinearities [9,10]. The NER method enables the precise determination of the nonlinear susceptibility component (χ^3_{xyyx}), which is crucial for elucidating the underlying mechanisms of nonlinear optical phenomena in ION-XS glass [6,9]. A recent study [11] introduced this approach, enabling the measurement of Gorilla® Glass's local n_2. The authors observed that the substitution of sodium ions with larger potassium ions plays an important role in increasing the nonlinear refractive index of this alkali-aluminosilicate glass.

Assessing the glass hardness after IOX is essential for understanding the effect of chemical strengthening on mechanical performance and fracture resistance. Traditionally, micro- and nano-indentation are standard methods for evaluating glass hardness. However, these techniques are inherently destructive, as the indentation process induces permanent deformation and may cause surface cracking, thereby altering the material's structural integrity. Alternatively, optical methods offer a promising nondestructive testing (NDT) approach for assessing hardness. Such methods provide several advantages, including real-time observation, the ability to monitor changes without inducing damage, and broader insights into the material's behavior under mechanical stress [12,13]. These noninvasive techniques contribute to a more comprehensive understanding of material responses without compromising structural integrity.

In this study, we analyzed NER measurements of n_2 to evaluate the hardness of chemically strengthened glass. By correlating the measured hardness of ION-XS glass specimens treated for varying durations with their nonlinear refraction, we propose a novel, rapid, and NDT approach for hardness assessment. Our methodology represents an advancement in nonlinear optics and laser-based instrumentation, offering a potential solution to a long-standing challenge. Leveraging advanced laser systems, a long-working-distance objective with distinct spatial resolution, and ultrafast pulses, we achieved measurements of the NER signal at the inner layers of ION-XS glass.

Nonlinear Ellipse Rotation (NER) Theory

The nonlinear ellipse rotation (NER) phenomenon is observed as a distinct third-order refractive nonlinear effect, which occurs when an elliptically polarized laser beam of significant intensity propagates through a nonlinear medium [14]. This effect, crucially dependent on the medium's isotropy, results in a polarization rotation directly proportional to the nonlinear refractive index, n_2. Comprehensive methodologies for NER measurements, par-

ticularly those utilizing dual-phase lock-in techniques, have been extensively documented in the literature [5,15,16].

For reference, we present the main equation used to fit experimental data. The average angle of rotation measured by a dual-phase lock-in for a temporal Gaussian pulsed laser beam with transversal Gaussian mode, in the absence of linear absorption and for pure electronic nonlinearity, is

$$\langle \alpha(z) \rangle_{lock-in} = \frac{\omega}{c} \left(\frac{sin2\phi}{3\sqrt{2}} \right) n_2(z) n_0 z_R I \left[tan^{-1}(z_b/z_R) - tan^{-1}(z_a/z_R) \right], \quad (1)$$

where $z_b = z + L/(2n_0)$ and $z_a = z - L/(2n_0)$, L is the sample thickness, z_R is the Rayleigh range, n_0 is the linear refractive index, ϕ is the angle of the quarter-wave plate used to produce the elliptically polarized laser beam, ε_0 is the vacuum permittivity, c is the speed of light, n_2 is the nonlinear refractive index, ω is the laser frequency, and I is the laser irradiance.

For the quarter-wave plate, setting ϕ to 0° and 45° produces linear and circular polarizations, respectively. Most measurements are conducted at $\phi = 22.5°$, where a good signal-to-noise ratio is achieved.

2. Materials and Methods

Commercial 1 mm thick glass (non-IOX—before ion exchange—Gorilla® Glass) was used as-received from Corning (Corning, NY, USA) [17]. The ion exchange process was performed in an electric furnace at 450 °C in molten potassium nitrate (> 99.9% KNO_3) for durations ranging from 2 to 48 h. The treatment temperature of 450 °C (T_g ~620 °C) was selected to promote ionic diffusion within an acceptable timeframe while minimizing stress relaxation effects. To prevent thermal shock, the samples were placed in the furnace for 15 min prior to immersion in the KNO_3 salt bath. After the IOX treatment, the glass samples were cleaned with deionized water in ultrasonic baths to ensure the complete removal of salt residues.

Before quantifying the nonlinear refractive index, n_2, and cross-sections and conducting the ion-exchange process, X-ray Fluorescence (XRF) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) were employed to evaluate the chemical composition, focusing primarily on the initial amounts of lithium and sodium, the main ions involved in the exchange process. Differential Scanning Calorimetry (NETZSCH DSC 404, NETZSCH Analyzing & Testing GmbH, Selb, Germany) was also used to determine the glass transition temperature (T_g)—see Figure S1 in the Supplementary Material. The potassium concentration was assessed using Energy Dispersive Spectroscopy (EDS) as a function of depth (μm). The ion penetration depth was calculated using EDS curves and the classical tangent method.

Glass hardness was evaluated by micro-indentation using a diamond Vickers indenter (Micro Indentation Tester—MHT—Anton Paar (São Paulo, SP, Brazil)) following the ASTM C1327-15 protocol [18]. Each indentation experiment was performed with a maximum load of 5 N and a holding time of 15 s in a controlled environment (40% humidity at 22–25 °C). Ten indentations were made in each glass specimen.

Optical measurements were performed using an NER setup with a long-working-distance objective (20 × 1.5 cm working distance) and ultrafast laser pulses produced by an amplified Ti:Sapphire CPA system (Dragon, ~40 fs, 780 nm, 1 kHz, K&M Labs, Boulder, CO, USA). The NER setup closely resembles the open-aperture Z-scan technique but includes an additional quarter-wave plate to generate an elliptical polarized laser beam. A standard computer-controlled z-translation stage with 1 μm resolution was used to move the sample. With our experimental setup, illustrated in Figure 1, we performed localized

NER measurements, achieving a spatial resolution of approximately 5.5 μm (Rayleigh range) along the *z*-axis (voxel size around 700 μm^3). This approach significantly enhances the spatial precision of nonlinear optical measurements.

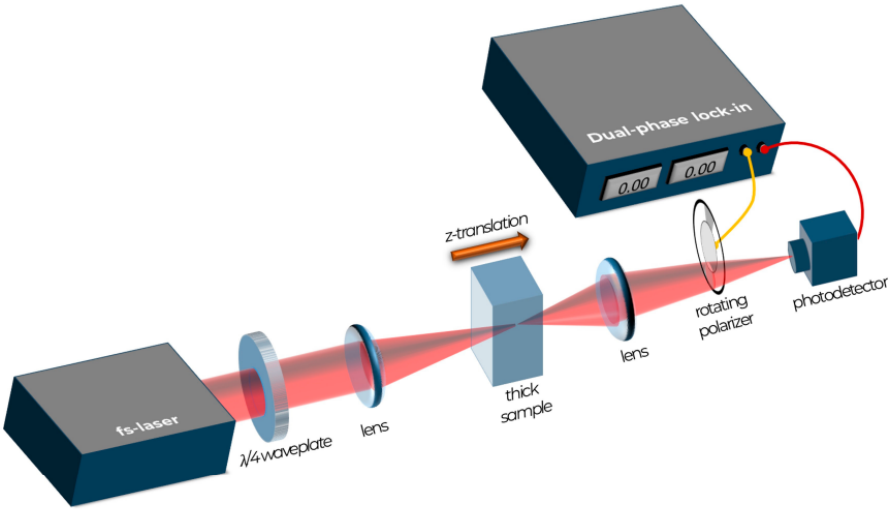

Figure 1. Schematic diagram of nonlinear ellipse rotation measurements using a dual-phase lock-in. Adapted from [11].

A dual-phase lock-in provides the in-phase (X) and in-quadrature (Y) signals. The angle is given by $< \alpha >_{lock-in} = arctan(Y/X)$ [5].

3. Results and Discussion

To determine the n_2 cross-section, NER measurements were performed along the 1 mm thickness depth of several chemically tempered aluminosilicate glass specimens (Gorilla® Glass precursor from Corning). The relative changes in NER shown in Figure 2 were derived by subtracting the signal data of the ion exchange samples from that of the untreated sample, which serves as a reference signal. The pattern is equivalent on the opposite side of the glass surface, as evidenced by previous work [11] with Gorilla® Glass. The relative NER signal rapidly increases to its maximum value between 20 and 50 μm, then gradually decreases towards the middle of the glass samples. As the treatment time increased (Figure 2), the overall relative nonlinear refractive index also increased, reaching its maximum at approximately 12 h of treatment.

According to previous studies, the nonlinear refractive index profile can be influenced by the potassium concentration [11], which exhibits relatively high polarizability. However, the EDS curves (Figure 3) show that potassium penetrates to much shallower depths (up to 80 μm). In Figure 2, the relative n_2 cross-section profile extends beyond the potassium penetration depth. While not directly matching the potassium distribution, this extended range is likely influenced by the residual stress profile within the glass. Indeed, as suggested by other works [19,20], the nonlinear refraction may follow the residual stress profile arising from compositional changes at the glass surface and stress relaxation [21]. Varshneya et al. [22] demonstrated that the stress buildup in chemically strengthened glass results from the balance in the stuffing effect due to the size difference between ions and structural relaxations within the glass network during IOX.

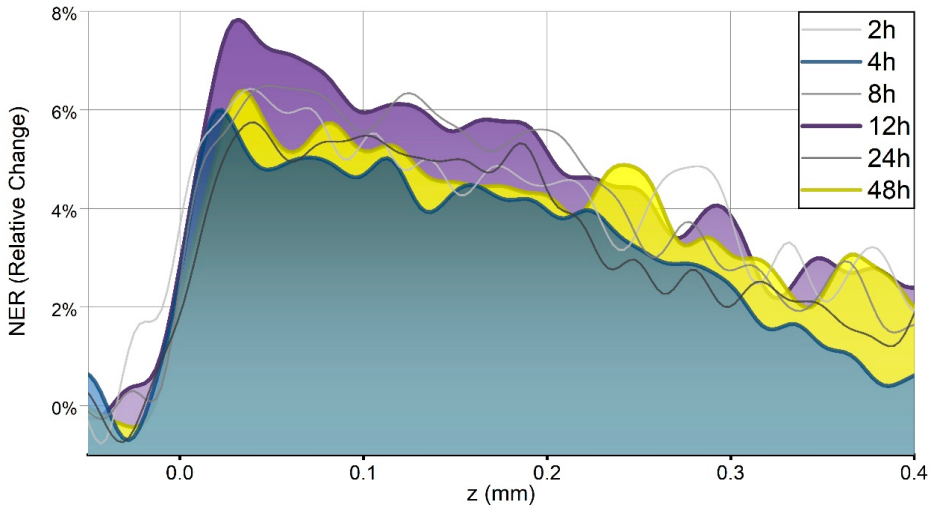

Figure 2. Nonlinear refraction changes as a function of depth [z (mm)] for different IOX treatment times. The areas below the curves represent the overall effect, peaking at 12 h.

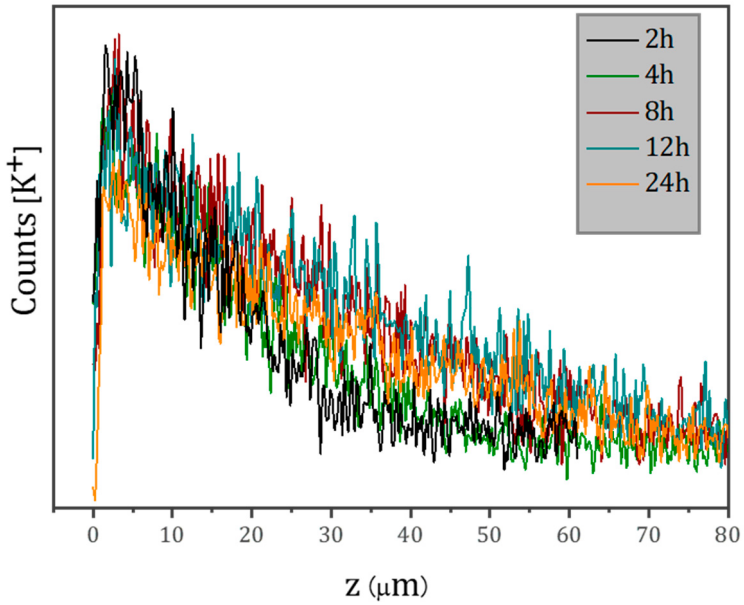

Figure 3. Potassium ion signal in chemically strengthened glass as a function of the depth [z (μm)], measured by Energy Dispersive X-ray Spectroscopy (EDS).

Compressive residual stress typically forms up to the ion penetration depth, while central tensile stress develops to maintain equilibrium. This tensile stress field stretches the glass network, altering bond lengths and angles. These structural changes can influence the electronic polarizability of the material, which is directly related to its nonlinear optical properties, and may extend beyond the potassium penetration depth, which aligns with the NER results.

Several studies, including one by Ragoen et al. [23], have shown that network modifications occur during the IOX process in silicate glass, leading to stress relaxation. Their Nuclear Magnetic Resonance (NMR) study on binary Na-silicate and ternary Na-Mg; Na-Ba; and Na-Ca silicate glass revealed that as the potassium content increases, Na–O bond distances shorten and Si–O bond angles widen. This allows the glass structure to better accommodate K+ ions, inducing stress relaxation. Additionally, the enhancement in mechanical properties is closely tied to the residual compressive stress induced during the ion exchange process in ION-XS glass [24]. This stress profile and the accompanying structural

relaxation play a critical role in increasing hardness and crack resistance, consistent with findings from other studies [23].

The data suggest that the extended NER cross-section profiles are due to glass network changes resulting from stress buildup and relaxation. Calahoo et al. [25] evaluated structural modifications in $30Li_2O{-}70SiO_2$ glass during the Li+ ↔ K+ ion exchange using micro-Raman spectroscopy. They found that variations in molar volume, linked to Si−O−Si bond angles, Si−O bond lengths, and Raman shifts, extended well beyond the ion penetration depth (up to three times). This observation is similar to the extended nonlinear optical signal seen in this study, suggesting significant network reorganization in both cases.

Further evidence for structural rearrangements beyond the ion exchange depth comes from Terakado et al. [26]. They developed a method to assess residual stress in chemically strengthened Gorilla® Glass using high-resolution micro-Raman spectroscopy, analyzing the Boson, D1, D2, and A1 peaks. Their findings matched the compressive stress profile obtained via the photoelastic effect in terms of stress magnitude and depth dependence (around 40 µm). Additionally, these authors observed local stress variations up to 130 µm in depth, which NER measurements may also be detecting.

Building on these observations, our study shows that the NER profiles are influenced by the stress profile in chemically strengthened glass. Previous studies have demonstrated that Vickers hardness is similarly affected by the residual compressive stress profile in such glass, as evidenced by both static and dynamic hardness measurements [27]. To further explore this connection, we analyzed cumulative NER values alongside Vickers hardness (Figure 4). Comparing these parameters, we substantiate the correlation between stress-induced structural changes and the observed hardness and nonlinear optical behavior.

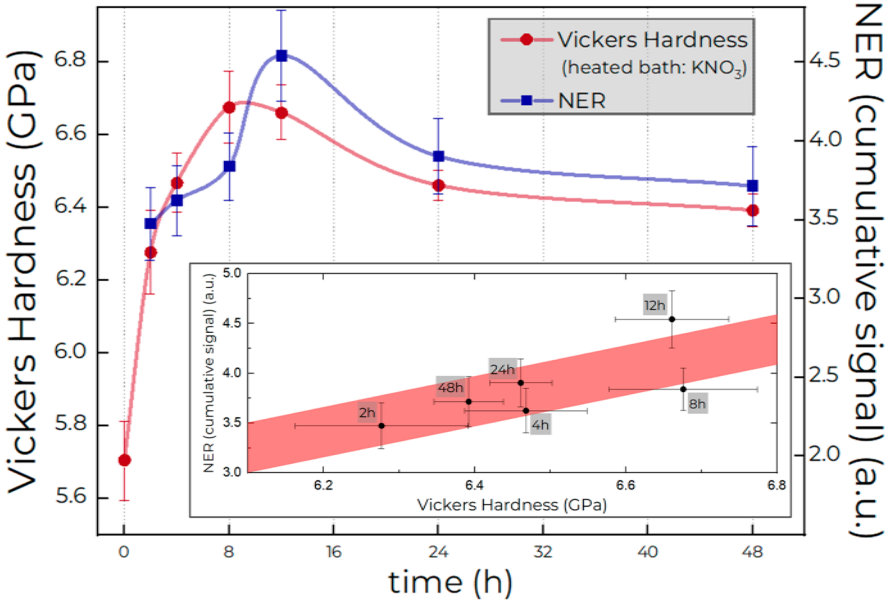

Figure 4. Vickers hardness (GPa) (red dots, left axis) and cumulative NER signal (a.u.) (blue squares, right axis) as a function of IOX treatment times (hours) for ion exchange in glass samples. The inset shows the positive correlation between mechanical and optical measurements, with the pink area representing the optimal linear fit.

Cumulative NER values were obtained by integrating the relative NER curves, representing the sum of optical physics contributions across the cross-section of the samples. The Spearman correlation analysis yielded $\rho = 0.771$ ($p = 0.072$), indicating a moderately strong positive monotonic relationship between the NER signal and Vickers hardness. While this

result does not reach statistical significance at $\alpha = 0.05$, likely due to the limited sample size ($n = 6$), the trend aligns with the observed physical linkage. The integrated NER profile showed a correlation coefficient $R^2 = (0.8 \pm 0.1)$ (see the inset of Figure 4) with the Vickers hardness profile for different IOX treatment times.

These analyses were initially performed on the same sets of chemically ion exchanged glass that were subsequently evaluated for Vickers indentation hardness. This sample selection ensured a direct comparison of mechanical properties across different experimental conditions within the same glass compositions.

Understanding glass strength is crucial for its applications, which currently require extensive destructive experimental testing. Our new approach enables hardness profiling through correlations with nonlinear optical measurements across the cross-section of ION-XS glass. This provides a foundation for developing rapid, nondestructive methods for assessing glass hardness. Additionally, we measured the NER signal in a soda lime heat-tempered glass. The signal extended up to $\sim$ 200 μm below the surface, which is shallower than in ion exchange glass, and exhibited a lower relative intensity. This suggests lower surface compressive stress and, consequently, reduced hardness in heat-tempered glass. Although qualitative because of differences in glass types and strengthening procedures, this comparison supports the validity of our method for detecting hardness variations in glass structures. However, the relative contributions of ionic species (e.g., K^+) and compressive stress to the NER signal remain an open question. Future studies will aim to decouple these effects to better understand their individual roles in the observed nonlinear response.

4. Conclusions

In this study, we introduced a novel, nondestructive testing (NDT) method for evaluating the hardness profile of ion exchange glass using nonlinear ellipse rotation (NER) measurements. By correlating the NER signal with the Vickers hardness data, we demonstrated that NER is a viable technique for assessing mechanical properties without causing damage. This approach represents a significant advancement in material characterization, offering a noninvasive alternative to traditional destructive testing methods. The results indicate that the NER signal extends beyond the potassium ion penetration depth, suggesting a relationship between the stress profile and hardness that aligns with findings from other studies on chemically strengthened glass.

Although further investigation is needed to fully understand the relationship between the nonlinear refractive index profile and stress-induced hardness variations, these findings open new avenues for developing high-performance glass materials. This study lays the foundation for future research to explore broader applications of NER in detecting stress irregularities and correlating them with other critical material properties. Ultimately, this technique could contribute to the design of stronger, more resilient types of glass, paving the way for innovations in both industrial and scientific applications.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/opt6030031/s1, X-ray Fluorescence (XRF); Differential Scanning Calorimetry (DSC); Vickers microhardness.

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Data Availability Statement: The experimental measurements, images, simulations, and calculations supporting the findings of this study are available from the corresponding author upon reasonable request. The datasets are stored in an institutional repository and can be shared without ethical, legal, or privacy restrictions.

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Conflicts of Interest: The authors declare no conflicts of interest.

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