

Performance of ITO, NiCr, and Pd thick film electrodes on lithium niobate under high-temperature operation

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Abstract

Reliable electrical contacts are essential for high-temperature piezoelectric devices based on lithium niobate (LiNbO₃, LN), yet the behavior of electrode materials under elevated temperatures remains insufficiently studied. Here we evaluate three practical electrode systems on bidomain LN (BLN) substrates: screen-printed Pd thick films, sputtered indium–tin oxide (ITO), and sputtered nichrome (80 wt.% Ni and 20 wt.% Cr). Bidomain LN crystals function as single-crystal piezoelectric bimorphs with well-defined bending resonances, enabling sensitive impedance-based detection of electrode degradation. Impedance spectra were recorded in 100 °C increments up to 800 °C, complemented by microstructural inspection, electrical resistivity measurements, and X-ray photoelectron spectroscopy. Pd thick films showed the highest stability, with unchanged impedance spectra after heating to 800 °C and cooling. Indium–tin oxide electrodes remained functional up to at least 700 °C, with possible operation at 800 °C. X-ray photoelectron spectroscopy results suggest gradual incorporation of Nb from the BLN substrate which may initiate slow degradation in ITO. NiCr electrodes showed degradation of electrical contact near 500 °C due to oxide formation, while intrinsic electrical degradation associated with loss of film continuity occurred above 600 °C. These results identify Pd as a robust but costly choice, ITO as a promising mid-range alternative, and NiCr as suitable only within a lower high-temperature window.

Keywords

lithium niobate, indium–tin oxide, palladium, nichrome, high-temperature electrodes, impedance

1. Introduction and background

Lithium niobate (LiNbO₃, LN) is a ferroelectric single crystal that maintains strong piezoelectric response up to near its Curie temperature (≈1140 °C; depends on composition). At room temperature it exhibits good electromechanical coupling, as well as stable elastic and piezoelectric constants, thus the use of LN below 300 °C is well established, e.g. in piezoacoustics [1]. This intriguing combination of a wide thermal operating window and high room-temperature electromechanical quality motivates studies of the possibility to use LN in sensors and actuators intended to operate above 300 °C, where many

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common piezoelectric single crystals and ceramics lose performance or depolarize.

The use of LN in piezoelectric devices for elevated temperatures is not novel and has been addressed in numerous reviews; the following are representative examples from recent years [2–7]. Across these reviews, three recurring limitations are highlighted for LN in high-temperature transducer applications: potential chemical decomposition, high electrical conductivity, and a strong pyroelectric effect at elevated temperature.

Chemical decomposition is typically discussed with reference to the LN solid-solution range [8] and to an English translation of a Russian monograph chapter [9]. These sources note that the congruent composition of LN is metastable within the Li_2O – Nb_2O_5 phase diagram and can partially decompose between about 300 °C and 900 °C under sustained thermal exposure. The most visible consequence in optical-grade crystals is whitening and opacity due to precipitation of secondary lithium niobate phases (LiNb_3O_8). Prolonged anneals at higher temperatures (>700 °C) can also degrade piezoelectric properties. For this reason, near-stoichiometric crystals are preferred because their compositions remain in the single-phase field over the diffusion-active temperature range. However, at high temperatures LN tends to lose Li_2O by out-diffusion, which drives the composition toward Li deficiency [10, 11] and can reopen decomposition pathways even in initially near-stoichiometric crystals. Mitigation strategies can include enclosing devices to maintain a steady Li_2O partial pressure and applying diffusion-barrier coatings to the exposed LN surfaces. Recent results indicate that well-engineered transducers fabricated on single-crystal LN can withstand at least 72 h at 750 °C, operate across multiple thermal cycles, and remain functional for months at 550 °C [12].

High conductivity in LN at elevated temperature arises primarily from Li^+ ionic transport with an activation energy of about 1.3 eV [13]. In oxygen-rich atmospheres this ionic contribution dominates the dc response over a wide high-temperature window, including 500–900 °C [14]. For low-frequency piezoelectric transducers the resulting conductivity provides a shunt path that screens piezoelectric charge and reduces sensitivity and Q-factor [3].

The study by Weidenfelder et al. [14] showed that the bulk conductivity of LN between 500 and 900 °C decreases as the Li_2O content approaches stoichiometry (at fixed oxygen partial pressure). This follows the defect chemistry of lithium-deficient, congruent crystals, where niobium antisites on lithium sites (Nb_{Li}) charge-compensated by lithium vacancies (V_{Li}) are prevalent; increasing Li_2O reduces both defect populations, which suppresses Li-vacancy-mediated ionic transport and small-polaron electronic conduction on niobium sites. Hence near-stoichiometric compositions are preferable to congruent material for high-temperature devices when low conductivity is required. An additional advantage is phase stability: near-stoichiometric LN lies in the single-phase field over

most of the high-temperature range relevant for diffusion, lowering the decomposition risk characteristic of congruent material. Brief excursions into adjacent two-phase fields should yield only minor secondary-phase fractions even after prolonged heat treatment and are unlikely to significantly alter the piezoelectric response in the frequency range considered.

Palatnikov et al. [15] further showed that, despite elevated conductivity, near-stoichiometric LN can be used in high-temperature piezoelectric resonators when the operating resonance frequency ω_{res} greatly exceeds the ionic-relaxation rate. Under this condition Li^+ ions, the dominant carriers in the studied temperature range, cannot follow the alternating field and strain, so screening of the piezoelectric charge is weak and the electromechanical damping from space-charge transport is small. As a result, conductivity produces only a modest reduction in Q-factor at the relevant resonance frequencies. The same principle was later demonstrated independently in an LN-based high-temperature acoustic-emission sensor operating up to 500 °C [16]. From the point of view of surface acoustic wave (SAW) devices the LN-based transducers showed relatively stable characteristics (SAW signal attenuation of 0.8 dB/100 °C) below 550 °C, while at approximately 600 °C the attenuation increases rapidly and at approximately 800 °C the frequency response is not detectable [1], however these experiments were conducted in nitrogen atmosphere, causing chemical reduction of LN at temperatures higher than 300–400 °C with the subsequent drastic increase of conductivity.

The pyroelectric response of LN can introduce substantial errors in high-temperature applications, especially sensors. Any temporal change in temperature or spatial temperature gradient generates a spurious electrical signal. At elevated temperature the increased ionic conductivity shortens the dielectric-relaxation time. This lowers the open-circuit pyroelectric voltage but does not suppress pyroelectric currents when the measurement circuit has finite input impedance, so the artifact can be indistinguishable from a piezoelectric signal. Mitigation includes minimizing crystal size and thermal gradients and using packages that limit rapid temperature fluctuations. A further option is to employ bidomain crystals, which exhibit intrinsically low net pyroelectricity because pyroelectric charges from oppositely polarized domains compensate each other [17].

Whereas many studies report the high-temperature properties of LN and propose device concepts, far fewer address how to make reliable electrical contacts that operate from room temperature to elevated temperatures. Yet electrode choice is a primary design lever for high-temperature piezoelectric devices. Contact materials should resist interdiffusion with LN and oxidation in the ambient over the intended service life; maintain strong adhesion and survive repeated thermal cycles without delamination despite coefficient-of-thermal-expansion (CTE) mismatch; retain low, stable resistivity; and provide predictable interfacial behavior (ohmic or intentionally

blocking). They should also tolerate Li_2O loss and related interfacial reactions, be straightforward to apply by sputtering, evaporation, or screen printing, and remain cost- and process-compatible with semiconductor manufacturing.

At room temperature, chromium deposited by magnetron sputtering or other common techniques is a frequent electrode choice for LN because it is inexpensive and typically forms a near-ohmic contact [18–22]. Chromium also effectively blocks lithium diffusion at room temperature [23]. Titanium can likewise exhibit near-ohmic behavior when the film is of low oxygen content; this is achieved with high-quality deposition such as electron-beam evaporation [24]. Platinum is a more expensive option but performs well from room temperature to at least 1000°C [25], and is widely used both as vacuum-deposited thin films [15, 26, 27] and as screen-printed pastes [14, 28]. For sputtered Pt, adhesion layers such as Ti or TiO_2 are commonly used to improve interfacial strength [1, 25, 29].

At elevated temperatures, platinum may appear to be the only practical electrode for LN devices [12, 25, 27]. Platinum is chemically inert, resists oxidation, and can mitigate lithium out-diffusion by limiting Li transport across the electrode/oxide interface over certain temperature ranges [30], which is relevant given LN's tendency to lose Li_2O at high temperature.

Although Pt has proven to be a robust electrode for LN at elevated temperatures in both fundamental and applied studies, it is not a panacea. Even this noble metal can degrade after prolonged exposure at high temperature, particularly above about $650\text{--}700^\circ\text{C}$ [4]. Reported degradation modes include grain growth and recrystallization, dewetting of thin films [31–33], and oxygen-assisted volatilization via formation of more volatile oxide species than the pure metal [34]. Strategies to raise the maximum working temperature include increasing Pt thickness, careful selection of adhesion layers, and using doped or composite coatings that combine Pt with additional stabilizing constituents [34–37]. These measures further increase the cost and complexity of Pt-based high-temperature electrodes.

From this perspective, lower-cost materials that can serve as high-temperature electrodes to LN single crystals within defined service windows, even if less stable than Pt, are of interest. In this work we evaluate three such options: magnetron-sputtered indium-tin oxide (ITO), a transparent conductor with reported high-temperature stability [38–40]; magnetron-sputtered nichrome (80 wt.% Ni : 20 wt.% Cr), known for survivability in air at high temperature; and a screen-printed palladium paste widely used industrially for poling (monodomaining) LN crystals. Although Pd is not generally a low-cost material, thick-film processing offers practical advantages over Pt for laboratory studies because robust conductive coatings can be produced without vacuum deposition, requiring only a firing furnace. This makes Pd pastes attractive for groups developing LN transducers without access to

sputtering equipment; the relative cost of Pd versus Pt will depend on commodity prices at the time of procurement.

To evaluate high-temperature electrode durability, we used bidomain LiNbO_3 (BLN) substrates. A BLN crystal is a LN plate with two thick, oppositely poled ferroelectric domains separated by a single domain wall. This architecture behaves as a series-configured piezoelectric bimorph bender while eliminating bonding layers and grain boundaries. Consequently, BLN devices are well described by the standard equations for an ideal bimorph [41, 42] and enable precise, sensitive piezoelectric transducers, including sensors, actuators, and energy harvesters [43–45]. In addition, the presence of strongly charged ferroelectric domain walls makes BLN a promising platform for memristive electronics [19].

Because a BLN crystal functions as a piezoelectric bimorph, its impedance spectrum shows a pronounced high-Q, low-frequency bending resonance and a corresponding antiresonance. By measuring the impedance over a frequency band that includes these features while incrementing the temperature from room temperature to 800°C , we determine for each electrode material a threshold temperature associated with the onset of irreversible degradation. This threshold is defined as a permanent change in the impedance spectrum observed after cooling the sample back to room temperature. After completing the impedance test at each temperature, electrodes of the same material deposited on reference samples and subjected to the same thermal treatments were examined by optical microscopy and four-probe conductivity measurements in order to identify degradation mechanisms and evaluate the consequences of heating. In addition, X-ray photoelectron spectroscopy (XPS) studies were performed on ITO and nichrome electrodes that had been placed in mechanical contact with pristine BLN crystals and annealed at 800°C , the highest temperature used in this study. These measurements made it possible to identify chemical interactions at the BLN–electrode interface and the processes that may contribute to electrode degradation. Based on all results, we suggest possible mitigation strategies and recommend allowable operating temperatures for each electrode material.

2. Materials and methods

A SAW-grade, single-domain LN wafer with a $Y+128^\circ$ crystal cut and congruent composition (the Roditi International Corp. Ltd.) was used to prepare samples. Rectangular bars were diced to $40 \times 4 \times 0.35$ mm for impedance-versus-temperature measurements and $10 \times 4 \times 0.35$ mm for optical microscopy, surface-conductance and XPS tests before and after heat treatment.

The bidomain ferroelectric structure in LiNbO_3 crystals was produced by the conventional Li_2O out-diffusion technique [10]. Because the starting wafer was single-side polished and the opposite face was ground,

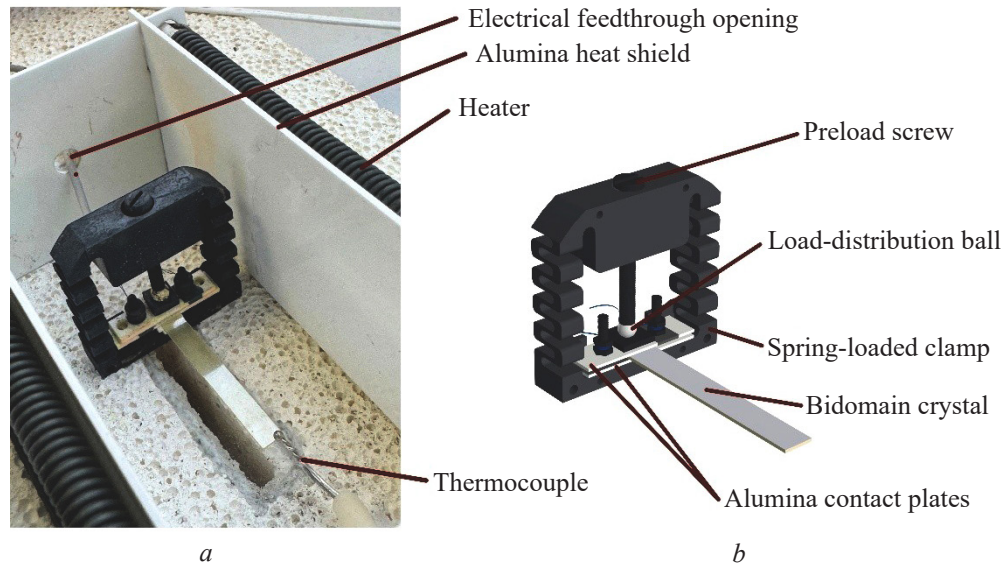


Figure 1. A photograph (a) and a schematic (b) of the clamp mounted on the brass holder

we polished the ground face of each sample after bi-domain formation to ensure comparable electrode–crystal interfaces on both faces. Polishing was performed on a Struers TegraPol-21 using a 2–3 μm diamond suspension.

The screen-printed palladium paste comprised palladium black and a lead-borosilicate glass frit doped with TiO_2 , dispersed in a turpentine-based organic vehicle. Before application, the paste was diluted with isopropanol to lower viscosity and enable thinner deposits. Excess paste was removed with a squeegee to form a thin, uniform film. The coated samples underwent a two-step thermal treatment: 140 $^\circ\text{C}$ for 12 h to dry, followed by 970 $^\circ\text{C}$ for 2 h to fire the layer. The fired Pd thickness was measured with a micrometer and was $\approx 28 \mu\text{m}$.

Nichrome and ITO electrodes were deposited by magnetron sputtering in a Sunpla 40TM vacuum system without intentional substrate heating. Nichrome films were grown by DC magnetron sputtering from an 80 wt.% Ni : 20 wt.% Cr target in Ar at 0.3 Pa, with a target power density of $\approx 10 \text{ W}\cdot\text{cm}^{-2}$. ITO films were deposited by radio frequency (RF) magnetron sputtering from a 90 wt.% In_2O_3 : 10 wt.% SnO_2 target in Ar/ O_2 (10 % O_2 in Ar) at 0.5 Pa, with a target power density of $\approx 8.5 \text{ W}\cdot\text{cm}^{-2}$. In both cases, the LN substrates were pre-cleaned by Ar ion-gun etching for 5 min. After sputtering, the ITO was annealed in air at 800 $^\circ\text{C}$ for 1 h. This short anneal did not degrade the film; the conductivity increased, consistent with crystallization and oxygen stoichiometry adjustment. Film thicknesses were measured by optical profilometry (Profil3D, Filmetrics) on step-edge references, averaging five locations: nichrome $\approx 1.0 \mu\text{m}$ and ITO $\approx 600 \text{ nm}$.

Bidomain LN crystals act as intrinsic bimorphs and exhibit a pronounced bending resonance–antiresonance pair in the impedance spectrum [41]. We therefore arranged the measurements to maximize the quality factor of this mode. Viscous gas damping cannot be eliminated, and operation in vacuum or at low oxygen partial

pressure would chemically reduce LN at high temperature. We thus focused on minimizing support losses by optimizing the clamping fixture.

In a cantilever configuration, dissipation increases when the clamp applies non-uniform pressure, allows slip, or when the clamped–free boundary is not perpendicular to the long axis of the crystal. To ensure uniform, secure fastening over the full temperature range, we designed a spring-loaded clamp that provides controlled preload and accommodates thermal expansion. The fixture was machined from AISI 321 stainless steel on a CNC mill.

We also needed to prevent fixture vibration excited by the bimorph near resonance. Even at low drive, displacement amplitudes become large close to resonance, so the fixture must have sufficient mass or be rigidly coupled to a massive anchor. Because the hot zones of most commercial furnaces are made of lightweight porous ceramics that offer little mechanical stiffness, we bolted the clamp to a heavy brass block with a long machined slot to accommodate the cantilevered crystal. This arrangement reduces parasitic fixture vibrations and improves thermal uniformity around the sample. A photograph and a schematic of the clamp mounted on the brass holder are shown in Fig. 1.

Impedance spectra of cantilever-mounted BLN bars were measured for all samples under identical mechanical boundary conditions. Each bar was clamped in a high-temperature fixture bolted to a brass holder and heated in a furnace; nichrome lead wires, insulated with high-temperature ceramic tubing, were attached before heating. The frequency dependence of the impedance was studied using a MFLI lock-in amplifier (Zurich Instruments) equipped with the MF-1A impedance option, recording the impedance magnitude $|Z|$, phase, and the instrument-reported parallel capacitance assuming a parallel resistor-capacitor equivalent circuit ($R \parallel C$). The sweep range was chosen to capture the first two bending resonances and anti-resonances: 10–3000 Hz for samples

with ITO and nichrome electrodes and 10–2000 Hz for samples with Pd-paste electrodes. Impedance spectra were recorded with a frequency step of 2 Hz and a test-signal amplitude of 300 mV.

Temperature was increased from room temperature to 800 °C in increments of 100 °C. After heating to each setpoint, the entire system was held at that temperature for at least 1 h to stabilize the sample temperature; the stabilization criterion was the absence of changes in the crystal's parallel capacitance larger than 10 % over a 10-min interval. Differences in total heating time between samples, including the stabilization period, did not exceed several minutes and were therefore insufficient to influence the electrode degradation process. After each high-temperature measurement the sample was cooled to room temperature and the measurement was repeated, and, in particular, after the 800 °C step a final room-temperature spectrum was recorded to assess irreversible changes. Irreversible electrode degradation was defined as a change in the shape of the impedance spectrum at room temperature compared to the previous room-temperature measurement for the same sample.

Electrode resistivity was measured before and after heat treatment using a conventional four-point probe. Tungsten-carbide tips were arranged collinearly over a span of 4 mm, with a spacing of 1.33 mm between adjacent electrodes. Five positions were tested along the long axis of each specimen. At each position, two measurements were collected with reversed current polarity to suppress thermoelectric offsets, and each measurement (one position, one polarity) was averaged over 100 rapid acquisitions. Sheet resistance was converted to resistivity using the measured film thickness. The reported resistivity for each electrode represents the average over all positions and both polarities.

Electrode micrographs were acquired with a ZEISS Axio Imager D1m optical microscope. Because the electrode area exceeded the field of view, representative regions displaying key features (such as cracks, pinholes, and color changes) were imaged.

X-ray photoelectron spectroscopy measurements were performed using a PHI 5000 VersaProbe II spectrometer with Al $K\alpha$ excitation ($h\nu = 1486.6$ eV), operated at 50 W with a 200 μm beam diameter. High-resolution (HR) spectra were acquired using a pass energy of 23.5 eV with a step size of 0.2 eV for the C 1s, O 1s, and Li 1s + Nb 4s regions, and a pass energy of 46.95 eV with step sizes of 0.2 or 0.4 eV for the Cr 2p and Ni 2p_{3/2} regions. Spectral fitting was carried out using a nonlinear least-squares procedure with Gaussian–Lorentzian or asymmetric Gaussian–Lorentzian line shapes. For Cr 2p and Ni 2p_{3/2}, multiplet splitting was accounted for using the Gupta–Sen theoretical multiplet model (GS multiplet) [46].

Because XPS probes only the upper few nanometers of a surface, direct access to the buried LN/electrode interface is difficult. Chemical etching or mechanical thinning would introduce substantial damage, and although ion-gun sputtering is gentler, it is slow and can still modify

the interfacial chemistry. Therefore, we adopted an alternative approach.

Each BLN sample bearing an electrode (ITO or nichrome) was stacked against a virgin BLN crystal with no coating and clamped with a force of ≈ 100 N. These assembled pairs were annealed at 800 °C for 1 h to promote interfacial interaction between the electrode surface and the opposing virgin LN. After annealing, the contacted surface of the electrode-coated BLN sample and the corresponding surface of the initially uncoated crystal were examined separately by XPS. Because the screen-printed Pd coating was relatively thick and had a high surface roughness, XPS measurements were not performed for this electrode type.

The choice of clamping force did not require high precision in this case. Excessive pressure would have resulted in film deformation and violation of the boundary conditions for impedance measurements. Variations in clamping force within acceptable limits could only modify the contact locally on the micrometer scale, whereas XPS measurements were performed over areas of several square millimeters, thereby minimizing the influence of clamping conditions on the measured chemical composition.

3. Results and discussion

Because DC I–V characteristics were not measured, we cannot classify the LN/electrode interfaces as ohmic or blocking, nor assess polarization or drift under constant bias. Under our AC test conditions, however, the impedance spectra showed no distinct nonlinearity at the analyzer's 300 mV sine excitation.

Fired Pd thick-film electrodes provided the most stable AC response across the studied temperature range. In air, they tolerated stepwise temperature increases with frequency sweeps at each 100 °C increment, and after the highest-temperature step and subsequent cooldown, the room-temperature spectrum was essentially unchanged relative to the pre-heating baseline. The resistivity measured along the test specimen, averaged over five spatial positions, was $\approx 4 \cdot 10^{-4} \Omega \cdot \text{cm}$. No noticeable surface changes were observed after the complete sequence of measurements. For these reasons, the Pd thick film was used as the reference electrode for comparison with the sputtered ITO and NiCr films. Representative spectra for Pd are shown in Fig. 2a.

Indium–tin oxide electrodes also remained functional up to 800 °C, as indicated by their impedance spectra (Fig. 2b); however, after cooling, localized regions exhibited signs of delamination, although the films retained overall continuity. NiCr electrodes demonstrated the lowest thermal stability, with irreversible degradation occurring at 500 °C. Consequently, impedance spectra for Pd and ITO electrodes were collected over 100–800 °C, whereas for NiCr the measurement range was limited to 100–400 °C. The results for NiCr are presented in Fig. 2c.

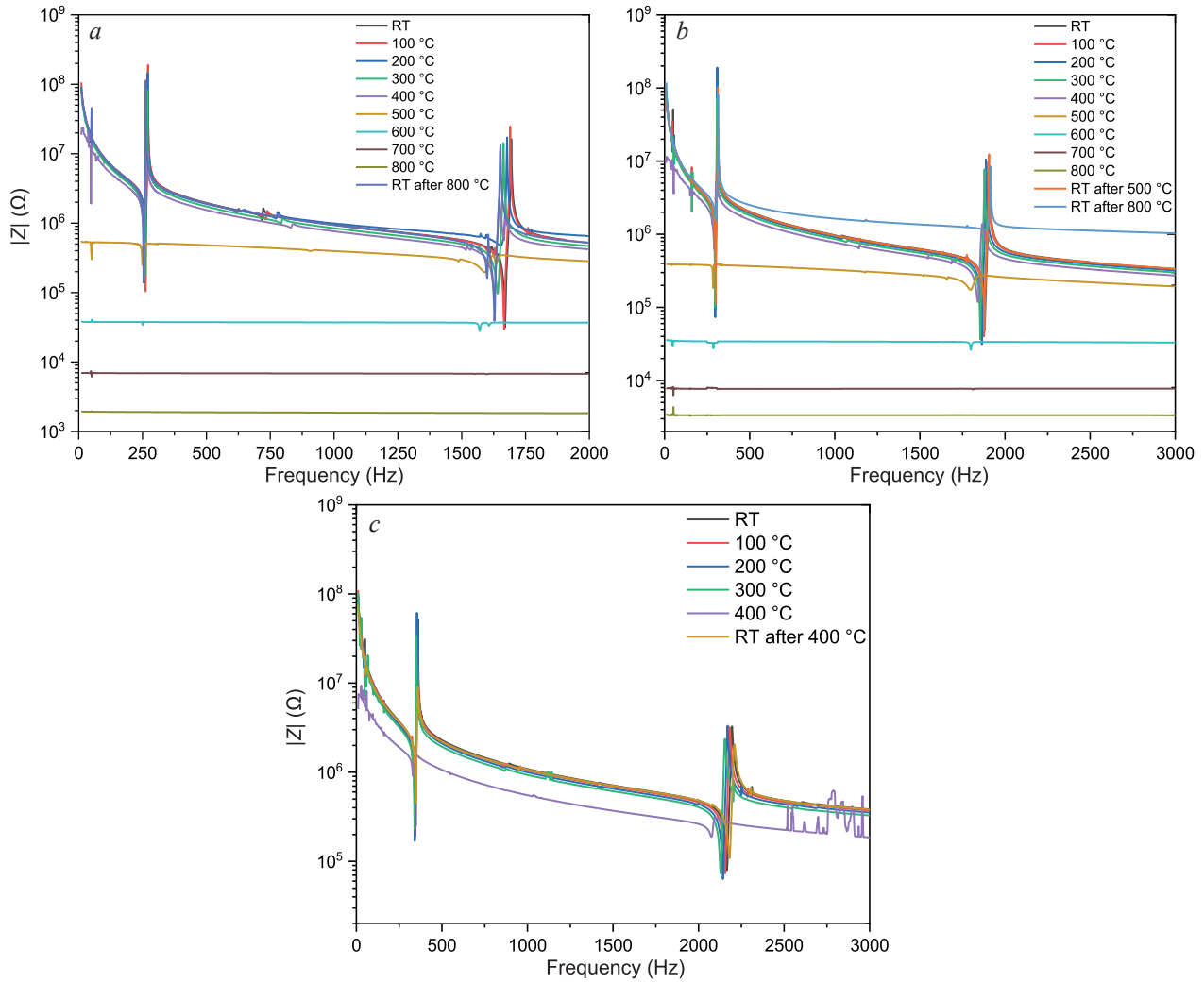


Figure 2. Impedance spectra of BLN-based piezoelectric bending transducers obtained at elevated temperatures with different electrodes: (a) Pd paste; (b) ITO; (c) nichrome

From the impedance spectra we observe that the mechanical and piezoelectric properties of the cantilevered BLN bimorphs remain effectively unchanged up to about 400 °C. In this range, the spectra exhibit a characteristic capacitive response with distinct peaks corresponding to the bending resonance and antiresonance. Increasing temperature within this lower-temperature interval produces only a slight shift in the resonance frequencies and a smooth decrease in the overall impedance magnitude across the measured band. Although the samples had nominally identical geometrical parameters, small differences in the effective clamped length caused minor shifts in the absolute resonance frequencies among the three specimens. These variations do not affect the qualitative conclusions of the comparative analysis. Because NiCr electrodes degrade at ≥ 500 °C, the high-temperature evolution of the impedance spectra is evaluated only for Pd thick films and ITO.

Pronounced changes appear between 400 °C and 500 °C, marking the onset of impedance degradation. The frequency dependence of the impedance magnitude $|Z|$ becomes weaker due to the rapid growth of the resistive contribution associated with the increasing conductivity

of the BLN crystals. The spectra evolve from a piezoelectric-oscillator signature with well-defined resonance and antiresonance to a predominantly resistive response, consistent with a sharp rise in parallel shunt conductance. It is important to note that, although the impedance resonance frequency coincides with the mechanical bending resonance, the same does not apply to the antiresonance. Therefore, no direct correspondence should be expected between impedance pattern or specific vibrational mode shape of the BLN crystal [41].

At 600 °C and above, the impedance magnitude becomes nearly frequency independent. This behavior is consistent with observations by Palatnikov et al. [15], who detected resonance–antiresonance peaks near 545 °C at frequencies three orders of magnitude higher. In our geometry, the bending mode provides a strong impedance feature, allowing the resonance to remain detectable at 500 °C despite rapid charge relaxation caused by the increasing crystal conductivity.

We first compare the impedance spectra obtained from the sample with ITO electrodes with the reference sample carrying Pd screen-printed thick-film electrodes.

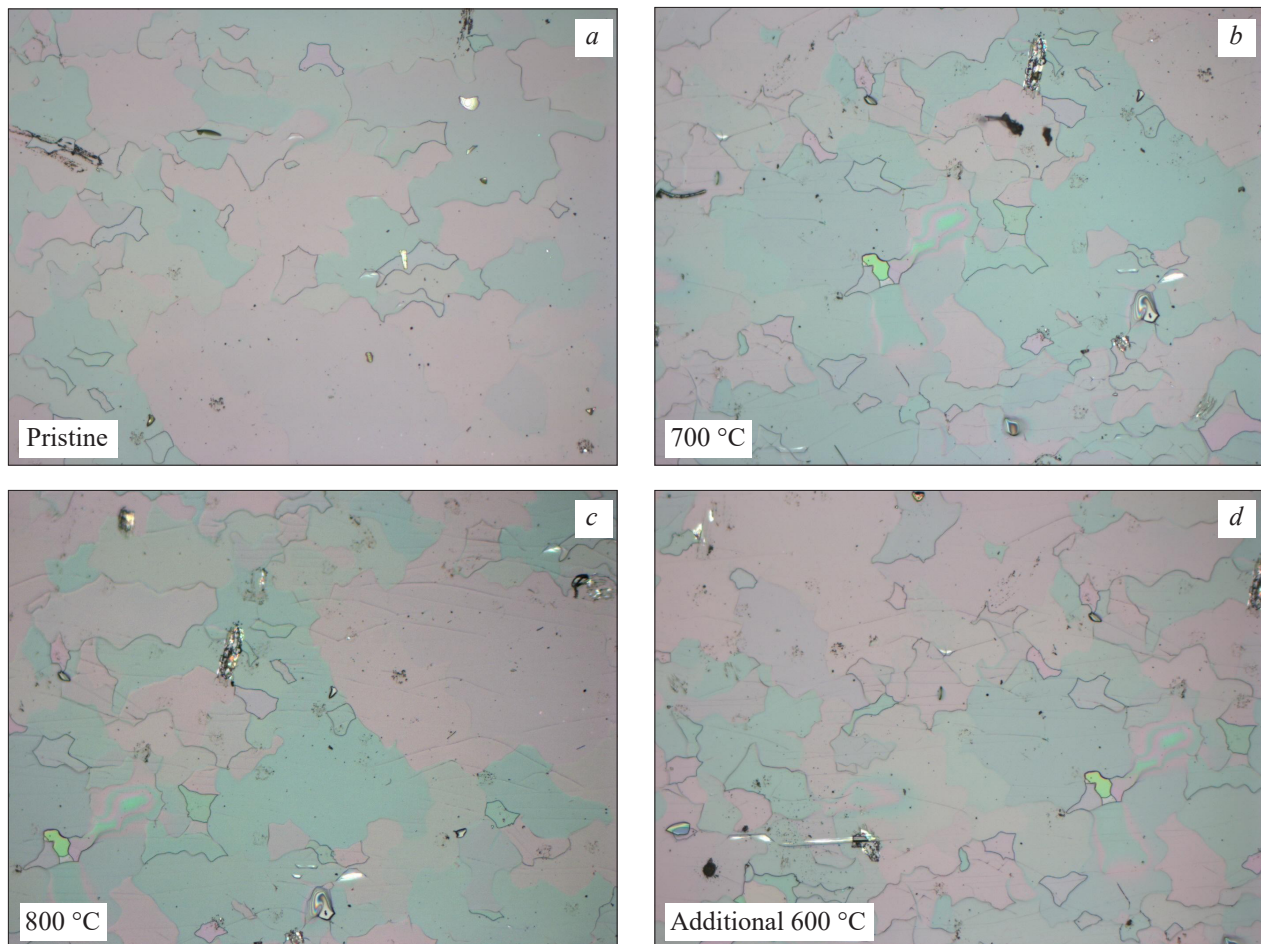


Figure 3. Surface of the ITO electrode for the following conditions: pristine state after the initial crystallization anneal at 800 °C (a); after annealing at 700 °C (b); after annealing at 800 °C (c); and after an additional 8 h anneal at 600 °C following the full sequence of thermal treatments from 100 °C to 800 °C (d)

At all temperatures up to and including 700 °C, the spectra are very similar. This agreement is important, because at 800 °C, where the dominant contribution to the impedance magnitude is the frequency-independent (active) conductance of the sample, the specimen with ITO electrodes becomes noticeably more resistive than the one with Pd thick-film electrodes. This change is likely an early indication of partial loss of film continuity. After cooling the sample to room temperature, the impedance magnitude increases at frequencies above the first bending resonance–antiresonance pair (300 Hz and 310 Hz). Such behavior is consistent with a reduction in the effective electrode area and can be interpreted as a combined effect of decreased capacitance due to stochastic perforation, including both physical voids and regions with strongly increased resistance, together with partial exfoliation of the electrode layer.

Micrographs of the ITO electrode surface do not show noticeable perforation, even after annealing at 800 °C. Figure 3 presents a sequence of micrographs obtained after several annealing temperatures. Although only a limited set of images is shown, the temperatures represented correspond to the most severe conditions applied to the crystal. In addition, after completing the full sequence of

thermal treatments from 100 °C to 800 °C, we carried out a further 8 h anneal at 600 °C in an attempt to accelerate degradation of the coating (Fig. 3d). No visually discernible changes were observed.

Similarly, the four-probe measurements do not show any evidence of ITO electrode degradation, even after an additional 8 h anneal at 600 °C following the full sequence of annealing processes up to 800 °C in 100 °C increments (Fig. 4). We therefore conclude that the degradation observed in the impedance spectra may originate from the appearance of localized low-conductivity regions in the film that could not be detected by the methods available to us. An alternative explanation is degradation of the electrodes on the rectangular bimorph sample used for impedance measurements, while the electrode on the smaller specimen used for conductance measurements remained stable. Such a difference could arise from variations in surface preparation prior to sputtering, because smaller samples are easier to clean in our facility. In either case, the ITO electrode demonstrates robust stability up to at least 700 °C, with possible stability extending to 800 °C.

The interaction between the ITO electrode and the BLN crystal at their common interface was studied by XPS. Before annealing, no technological contaminants

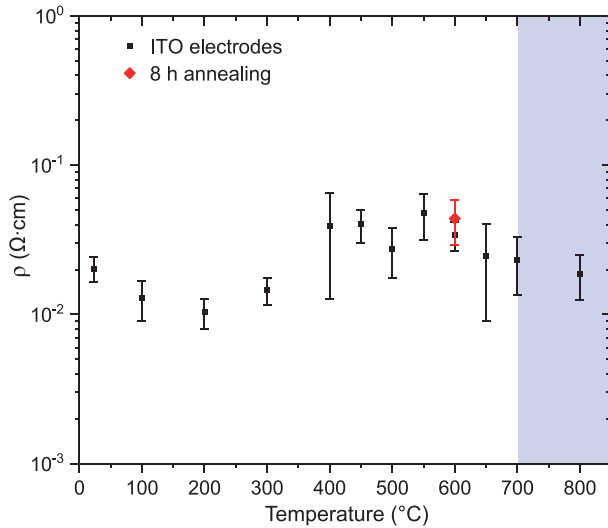


Figure 4. Resistivity of the ITO film as a function of prior annealing temperature, measured using the four-probe method. The diamond-shaped symbol corresponds to the resistivity measured after an additional long-term anneal at 600 °C performed after completion of the main annealing sequence

were detected on the ITO surface, apart from a small amount of carbon attributable to adsorbed hydrocarbons, most likely originating from laboratory solvents. The atomic fraction Sn/In was 0.088, which is consistent with widely used ITO compositions [47, 48]. After annealing in contact with a pristine, unelectroded BLN crystal for 1 h at 800 °C, this ratio remained essentially unchanged (Sn/In = 0.083), and a small amount of Nb (1.5 at.%) appeared at the surface, as shown in the wide-range XPS spectra in Fig. 5.

The BLN surface after annealing is strongly Li-deficient, with a Li/Nb ratio of approximately 0.3, determined from the relative intensities in the Li 1s + Nb 4s region using the method of [49]. Such Li deficiency is common and can be observed even in thermally untreated crystals, because Li loss may occur during mechanical polishing with water cooling. More importantly, no diffusion of In or Sn into the BLN surface was detected. This observation indicates that the interfacial interaction is dominated by diffusion of Li and Nb from the crystal into the ITO film, while diffusion in the opposite direction is very limited. Given the high mobility of lithium, it is also reasonable to assume that Li can be incorporated into the ITO layer [50, 51], where it may adversely affect the electrical conductivity of the oxide [52]. However, because XPS is not well suited for detecting low concentrations of lithium, this conclusion remains tentative.

Because the relative concentrations of In and Sn remain almost unchanged and the oxygen concentration increases, we infer that the primary degradation mechanism of the ITO electrode on BLN is a slow deterioration of the conductive oxide due to incorporation of Li and Nb. Longer high-temperature annealing experiments, aimed at clarifying the later stages of this degradation process, will be the subject of future work.

The results obtained for nichrome, which showed the lowest corrosion resistance among the studied electrode materials, are unsurprising. The high corrosion resistance of this alloy in air arises from the formation of a Cr_2O_3 surface layer that is largely impermeable to oxygen and protects the underlying metal from further oxidation. In bulk nichrome components (e.g., heating elements), this process produces a chromium-rich oxide surface with a Ni-enriched metallic layer beneath it [53, 54]. The protective scale can reach several micrometers in thickness [55, 56]. Because oxidation is controlled by oxygen diffusion through Cr_2O_3 , even thin films may survive limited exposure at temperatures approaching 500 °C. However, a moderate-duration treatment,

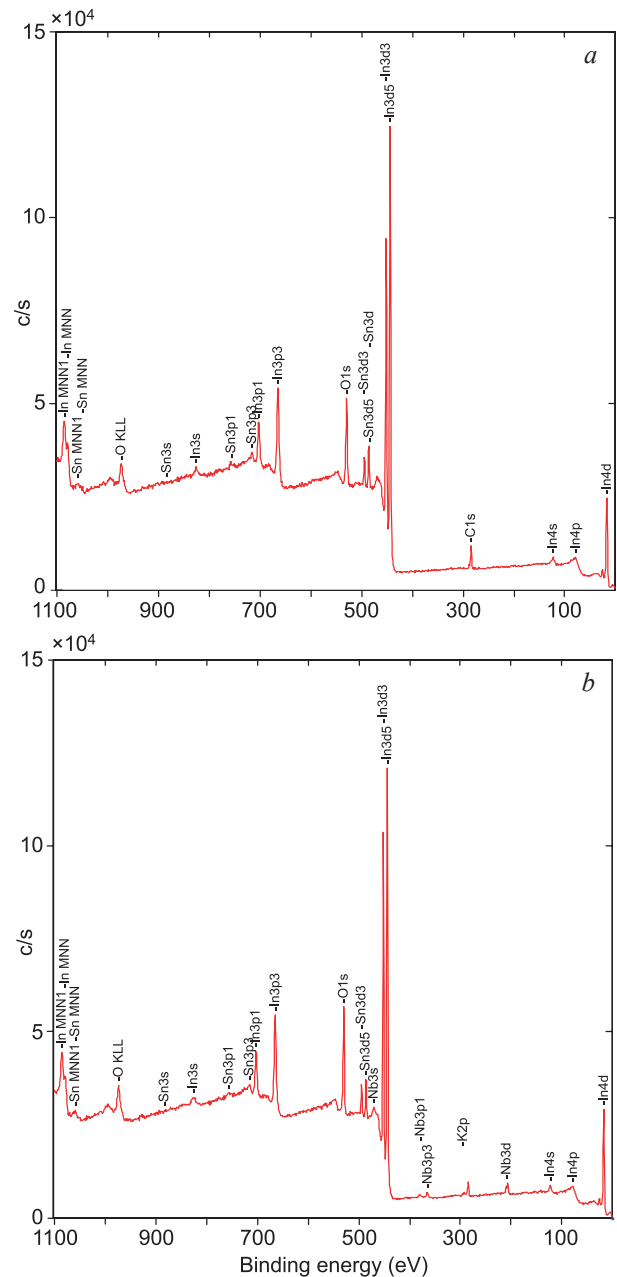


Figure 5. Wide-range XPS spectra of the ITO film before annealing (a) and after annealing in contact with a BLN crystal at 800 °C (b)

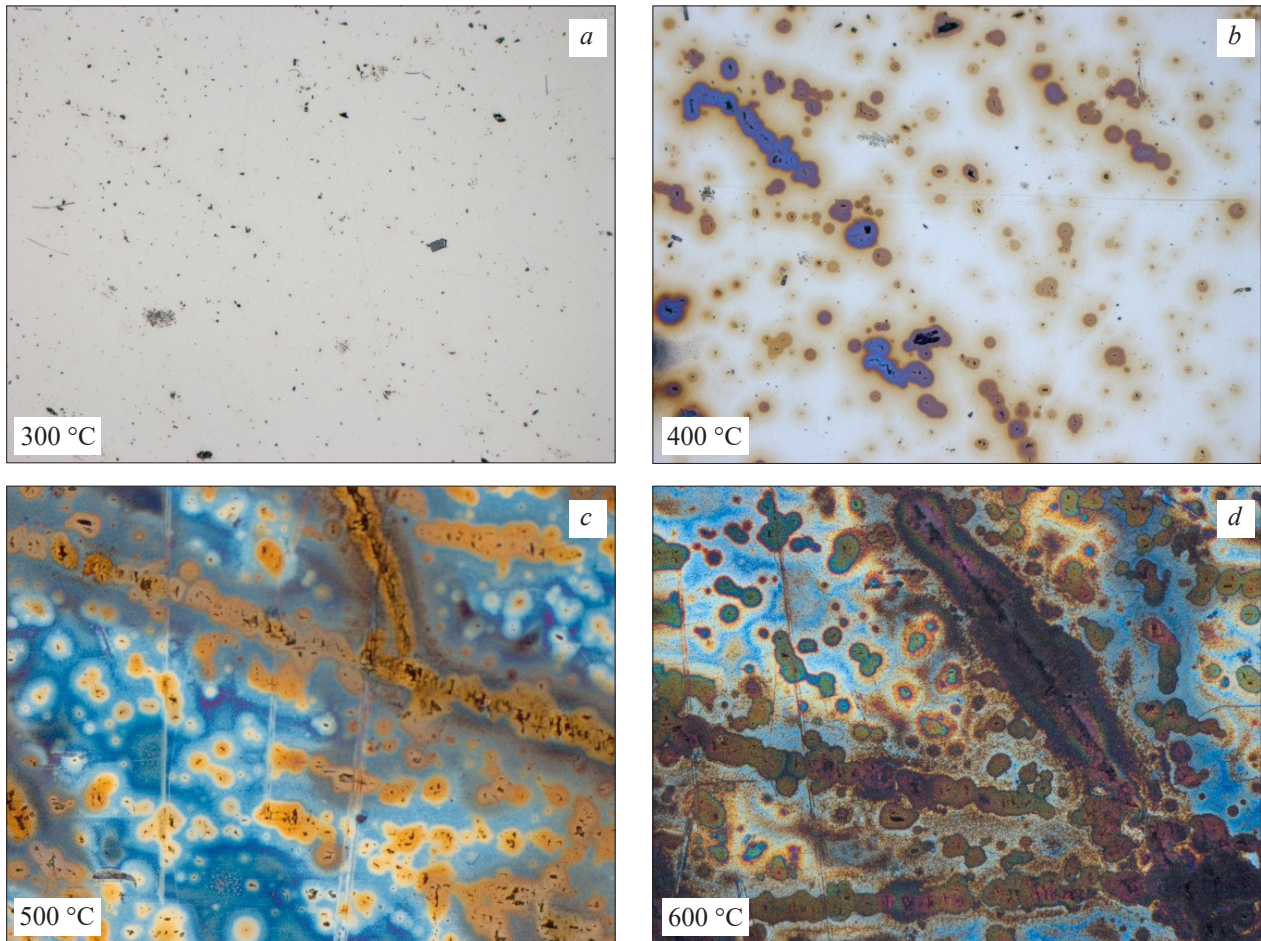


Figure 6. Surface of the nichrome electrode after annealing at 300 °C (a), 400 °C (b), 500 °C (c), and 600 °C (d)

such as in our case (≈ 1 h of heating followed by ≈ 1 h at the target temperature for stabilization), resulted in complete electrode failure. This indicates that when the sputtered nichrome layer is too thin to develop a continuous, mechanically stable Cr_2O_3 scale, its effective corrosion resistance in air is nearly absent.

Early stages of nichrome electrode degradation can be seen in the sequence of micrographs recorded after annealing at 300–600 °C in 100 °C increments (Fig. 6). Initial changes appear at polishing defects covered by nichrome, manifesting as localized discoloration. At 300 °C and 400 °C these regions remain small, and most of the film is still continuous, allowing the electrode to retain electrical connectivity. Between 400 °C and 500 °C, however, the degradation becomes abrupt: interference colors develop over much of the surface, indicating the formation of an oxide layer across the electrode. The presence of coloration suggests that a reflective metallic sublayer is still partially present. After annealing at 600 °C the film appears nearly black, consistent with extensive oxidation and the effective loss of metallic connectivity between the electrode and the BLN crystal.

To examine the effect of annealing duration on degradation, an additional sample was tested. This sample underwent a sequence of annealing steps with 50 °C temperature increments and intermediate cooling to

room temperature, similar to the procedure used for the previously reported data. However, instead of heating to 650 °C and above, the sample was subjected to a prolonged anneal of 8 h at 600 °C. After this treatment, four-probe measurements were performed, as indicated by the red diamond marker in Fig. 7. No significant increase in resistivity was observed, indicating that penetration of the oxide layer into the depth of the film proceeds slowly at this temperature and that the remaining metallic portion of the film remains electrically conductive.

Together, these observations indicate that magnetron-sputtered nichrome thin films can be used as electrodes for high-temperature LiNbO_3 applications up to about 500–550 °C, but reliable operation requires ensuring electrical contact through the Cr_2O_3 layer between the film and the external wiring or fixtures.

X-ray photoelectron spectroscopy analysis of the nichrome electrode surface before and after heat treatment in contact with a BLN sample helps clarify the chemistry of the film–substrate interaction. The spectrum of the pristine nichrome film shows an atomic ratio $\text{Cr}/\text{Ni} = 0.32$.

No technological contaminants were detected in the pristine nichrome film. Prior to heat treatment, a portion of the surface species was already oxidized (Fig. 8a and 8c): the $\text{Ni } 2p_{3/2}$ spectrum contains contributions from Ni^0 (45 %), Ni^{2+} in NiO , and $\text{Ni}(\text{OH})_2$, while the $\text{Cr } 2p$

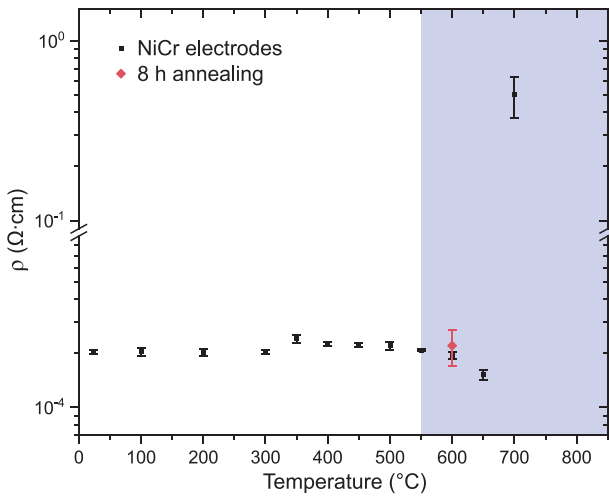


Figure 7. Resistivity of the nichrome film as a function of prior annealing temperature, measured using the four-probe method. The shaded area above 550 °C marks the approximate temperature range where the electrode transitions from a continuous to a discontinuous film. The diamond-shaped symbol corresponds to the resistivity measured on an additional sample that underwent the standard annealing sequence up to 550 °C, followed by an 8 h long-term anneal at 600 °C. Note that the shaded region includes temperatures at which the resistivity remains measurable by the four-probe method, while the impedance response is already strongly degraded (see discussion in the text)

spectrum contains Cr^0 (37 %) and Cr^{3+} associated with Cr_2O_3 . The presence of hydroxyl species is consistent with the results of Yong Kwon et al. [57] and can be attributed to exposure to moisture in air. The O 1s spectrum likewise includes components corresponding to oxides, hydroxides, and adsorbed oxygen-containing species. Here we need to note that the observable discrepancy between the model and experimental Ni $2p_{3/2}$ spectra before annealing is attributed to the difficulty of resolving multiple oxidized states of nickel.

Distinct changes appear on the nichrome surface after heat treatment in contact with BLN at 800 °C (Fig. 8b and 8d). Most notably, the atomic ratio Cr/Ni increases sharply to 6.2, in full agreement with the established corrosion-resistance mechanism of nichrome, in which a chromium-rich oxide scale forms while Ni is depleted from the surface region [53, 54]. The Ni $2p_{3/2}$ and Cr $2p$ spectra confirm that both metals are fully oxidized.

It is known that under oxidizing conditions with moderate humidity in air, NiO_x undergoes transformation during annealing with the formation of thermally stable NiOOH on the surface [58], which corresponds to trivalent Ni. After annealing in contact with BLN, we observe that most nickel is present in the form of NiOOH , with a smaller fraction detected as NiO . This observation is consistent with the results reported in [57], where NiO was also observed together with Ni_2O_3 . In the present case, however, the formation of Ni_2O_3 is unlikely because this phase decomposes at temperatures above approximately 600 °C.

Chromium is present predominantly as Cr_2O_3 , although Cr (VI) species are also detected; their appearance may be associated with reactions involving lithium out-diffused from the BLN crystal, leading to the formation of lithium chromate on the surface. Nb originating from the BLN substrate was also detected on the surface of the annealed electrode in the form of Nb^{4+} (NbO_2), suggesting that the near-surface layers of BLN partially dissociated during the heat treatment and mixed with the nichrome, forming a composite layer of transition-metal oxides.

Interestingly, although pristine lithium niobate surfaces are known to be Li-deficient in XPS measurements [49], and this deficiency is further enhanced by Li out-diffusion during BLN preparation, the BLN surface after heat treatment in contact with nichrome at 800 °C shows the opposite trend. Our measurements indicate an increase in the Li/Nb ratio to 1.1–1.2, determined from the relative peak intensities in the Li 1s + Nb 4s region. This apparent Li enrichment is accompanied by the incorporation of chromium, with a surface concentration of ≈ 3.5 at. %.

One possible explanation is that chromium or nickel entering the near-surface region of BLN may slow Li out-diffusion, while the oxygen sublattice of LN readily participates in the oxidation of Cr and Ni, simultaneously reducing Nb to its intermediate oxidation state (IV). In the case of nickel, oxidation involving oxygen from the LN lattice, accompanied by a shift to the higher oxidation state (+3) with the formation of NiOOH , represents a plausible mechanism. Although these processes require further verification, the present results indicate that BLN and nichrome undergo chemical interaction at their interface that involves oxygen originating from the lithium niobate crystal.

Conclusion

This study evaluated three electrode materials for use on LiNbO_3 -based piezoelectric devices operating at elevated temperatures: screen-printed Pd thick films, sputtered ITO, and sputtered NiCr. The results reveal clear differences in thermal stability, degradation mechanisms, and practical applicability.

Pd thick-film electrodes demonstrated the highest robustness. Their impedance spectra remained stable throughout the entire temperature range up to 800 °C, and the room-temperature characteristics after cooling were indistinguishable from the initial state. Neither surface degradation nor significant changes in resistivity were detected. Although Pd is relatively expensive, its long-term stability, compatibility with simple firing processes, and absence of vacuum requirements make it an attractive option for laboratory prototyping and for devices intended to operate near the upper temperature limits explored here.

Indium–tin oxide electrodes also performed well and remained functional up to at least 700 °C, with possible

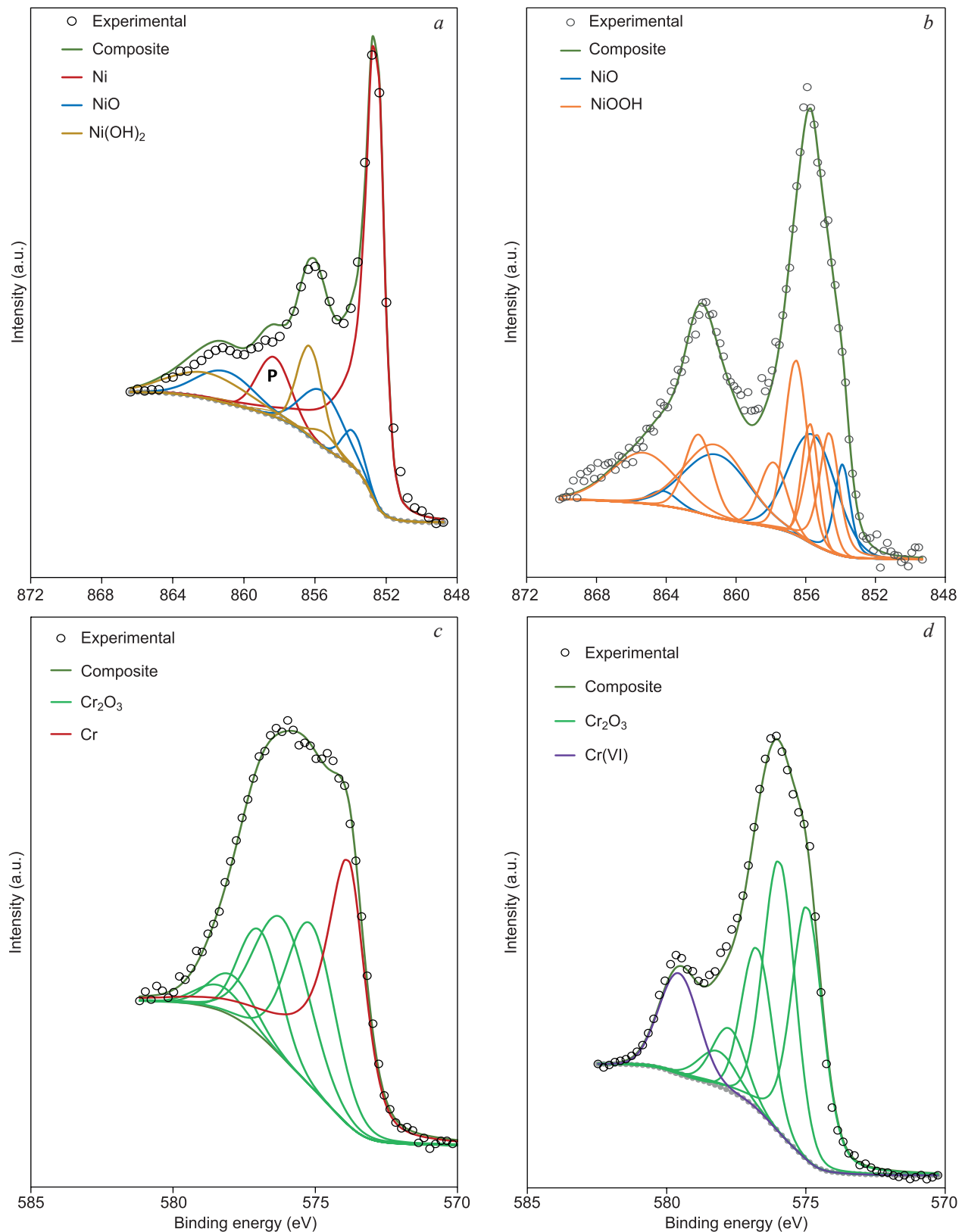


Figure 8. X-ray photoelectron spectroscopy spectra of Ni $2p_{3/2}$ before (a) and after (b) annealing in contact with the BLN crystal, and Cr $2p$ before (c) and after (d) annealing. In spectrum (a) assignment “P” corresponds to Ni⁰ plasmon peak

operation at 800 °C. The impedance spectra showed only subtle signs of degradation, and both optical micrographs and four-probe measurements indicate that the film remains largely continuous after prolonged heating. X-ray

photoelectron spectroscopy analysis suggests that degradation of ITO, when it occurs, may be driven by slow incorporation of Nb from the BLN substrate, accompanied by an increase in the oxygen content of the film. Given

the high mobility of lithium, it is also reasonable to assume that Li ions can diffuse into the ITO layer. These processes may gradually reduce the conductivity of ITO in localized regions. Despite these effects, ITO appears to be a promising lower-cost alternative to Pd for intermediate high-temperature ranges, particularly in applications where optical transparency, ease of sputtering, or compatibility with thin-film processing is required.

NiCr electrodes showed the lowest high-temperature stability. Although four-probe measurements indicated nearly unchanged resistivity up to 650 °C, impedance spectra and micrographs demonstrated clear degradation beginning at approximately 500 °C. X-ray photoelectron spectroscopy results confirmed rapid oxidation and formation of Cr₂O₃-rich layers together with Ni depletion, followed by chemical interaction between the electrode and BLN, including Nb incorporation and formation of transition-metal oxides. Thin sputtered NiCr layers lack the thickness necessary to form a protective, mechanically stable oxide scale. Nevertheless, because NiCr is inexpensive, easy to deposit by DC sputtering, and provides acceptable performance up to about 500–550 °C, it remains suitable for applications in this moderate temperature range, provided that reliable electrical contact through the growing Cr₂O₃ scale is ensured.

Overall, Pd thick films are the most stable option for the full high-temperature window studied, while ITO represents a promising and more economical alternative up to approximately 700 °C. NiCr electrodes are suitable for lower high-temperature ranges but degrade rapidly above 500 °C. These findings provide practical guidance for selecting electrode materials for high-temperature LiNbO₃-based sensors and actuators and point to specific interfacial mechanisms that should be addressed in future material optimization.

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