Surface Preparation and Characterization of $LiNbO_3$ using LEED and AES

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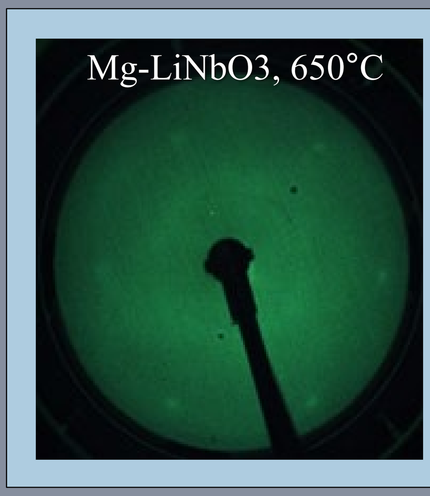
Introduction and Motivation

$LiNbO_3$ is a popular compound used for optoelectronic applications. Surface crystallography of $LiNbO_3$ single crystal wafers were measured using Low Energy Electron Diffraction (LEED) and Auger Electron Spectroscopy (AES) under thermal annealing in ultra-high vacuum conditions. In order to use $LiNbO_3$ in wafer-scale integration the surface characterization must be understood to avoid defect formation in epitaxially grown crystals. There is little existing data about $LiNbO_3$ surface crystallography. Measured samples were $LiNbO_3$ Z-cut categorized as: undoped optical grade $LiNbO_3$, stoichiometric $LiNbO_3$, Mg doped (5.0% mole) and Fe doped (0.07% mole). Thermal annealing was performed at elevated temperatures in the range from 200°C to 1000°C to clean surface and to improve surface crystallography.

Applications

OCI Vacuum Microengineering is specifically interested in $LiNbO_3$ for a possible energy storage applications and for wafer-scale integration. This material has also attractive properties as mixed ion-electron conductor for memistors and superconductors.



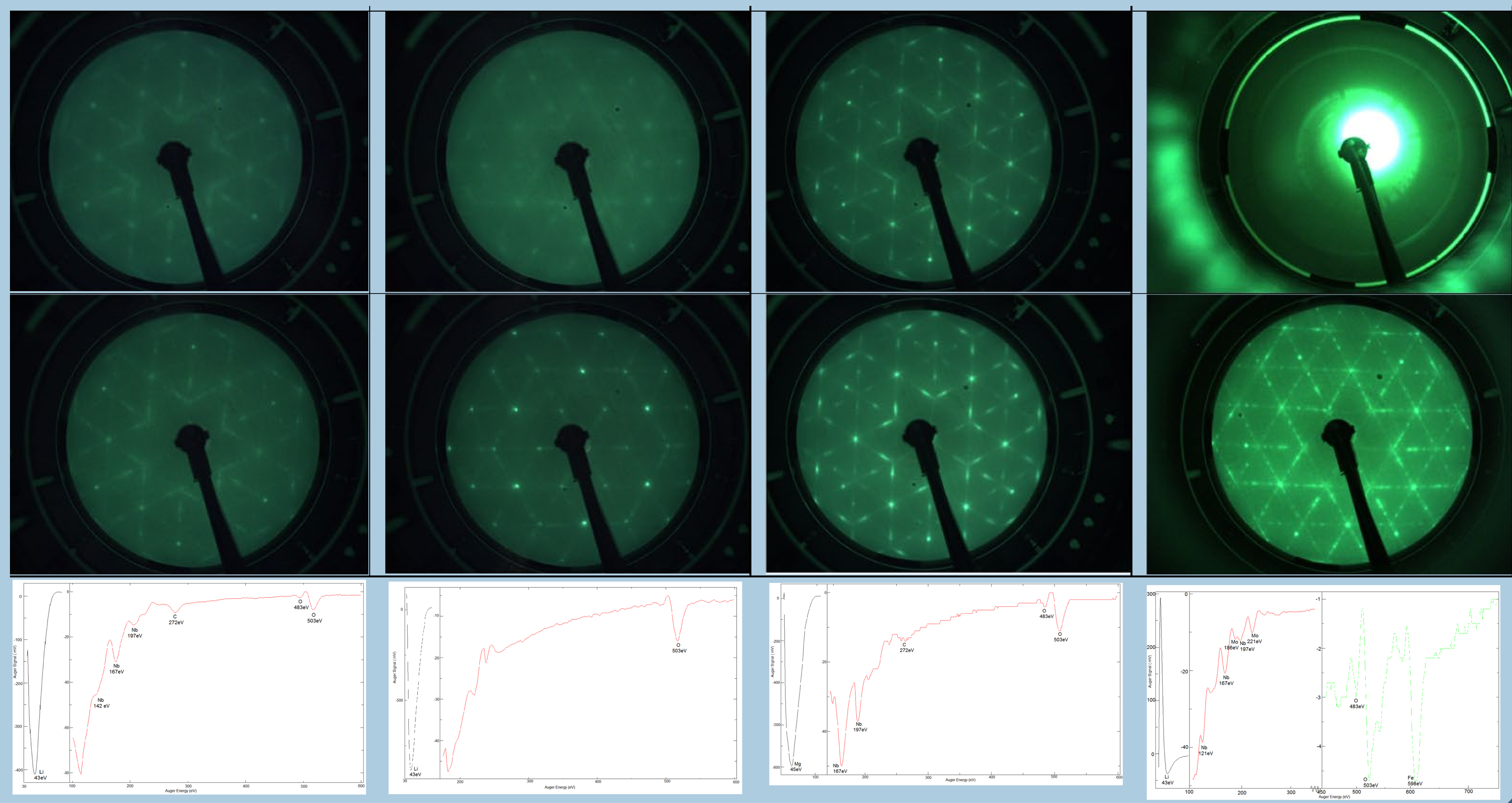
First sign of a LEED pattern at **650 °C on Mg-$LiNbO_3$**, exhibiting hexagonal ordered surface crystal structure at the lowest temperature. First sign of an ordered LEED pattern on the undoped $LiNbO_3$ sample it was at 700°C, for the stoichiometric sample 750°C.

$LiNbO_3$ | Stoich. $LiNbO_3$ | Mg- $LiNbO_3$ | Fe- $LiNbO_3$

Process: UHV Annealing at 800 °C

Process: UHV Annealing at 850 °C

Auger Electron Spectroscopy General Spectrum



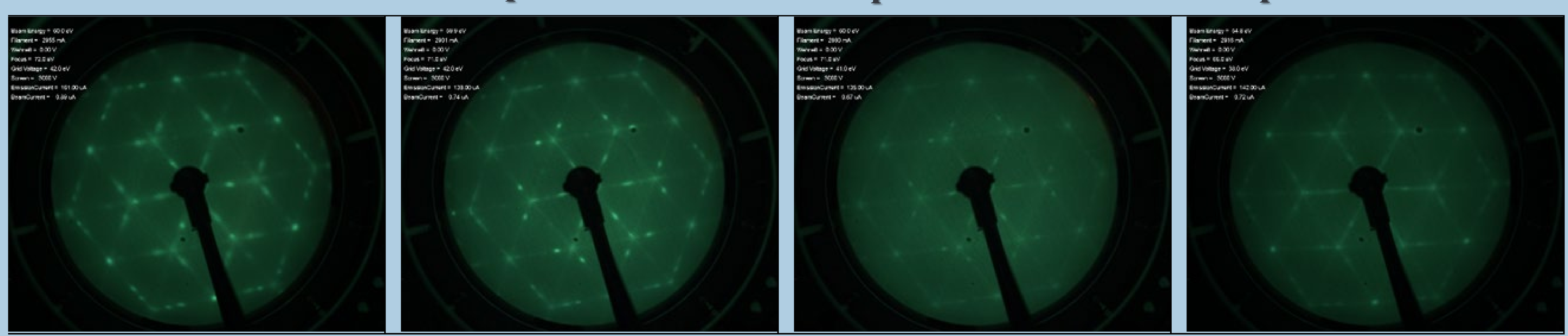
Magnesium Dopant induces long range order recrystallization on $LiNbO_3$ surface at lower temperatures than on undoped and stoichiometric surfaces.

Sample Charging

The electron beam induces a chagrining process when the surface electrons repel the beam electrons creating a unreadable LEED image as seen here with the **Fe- $LiNbO_3$** sample at a beam energy of 100 eV and after annealing at 800 °C. Charging was common in all LiNbO3 samples. Generally, all samples charged at beam energies below 45 eV.

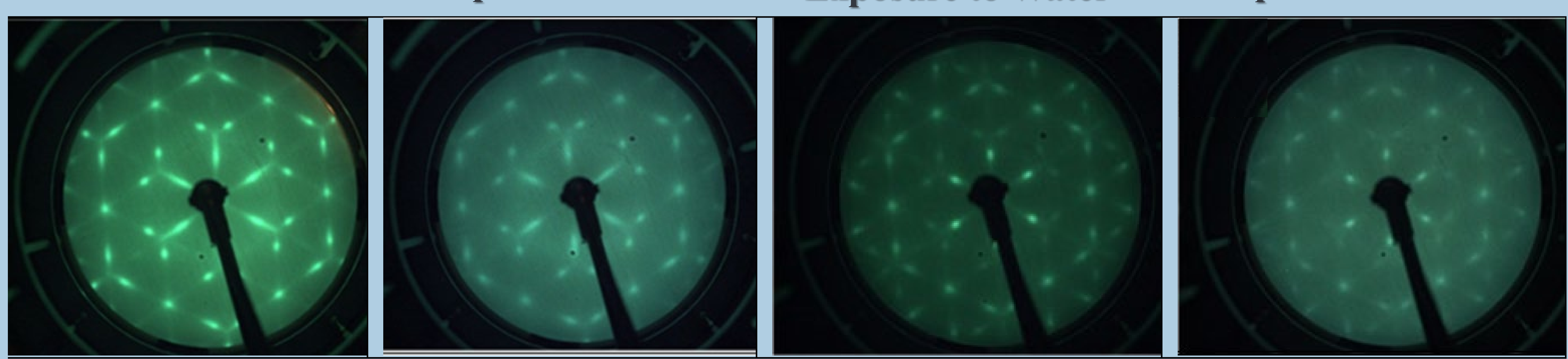
Crystalline Structure Exposure Tests on $LiNbO_3$

UHV Anneal 875°C | Exposure to Air | Exposure to Water | Exposure to Alcohol



Crystalline Structure Exposure Tests on Mg-$LiNbO_3$

UHV Anneal 875°C | Exposure to Air | Exposure to Water | Exposure to Alcohol



Both the $LiNbO_3$ and the Mg-$LiNbO_3$ had no initial LEED pattern before annealing. These samples were annealed in the UHV environment to recover the reconstructed surface crystallography. This crystallography was preserved after exposure to air, water and alcohol with only minor changes.

UHV annealing can create stable surface crystallography on $LiNbO_3$ samples suggesting improvements can be made to the electrochemical polishing processes to improve stability of the surface over time.

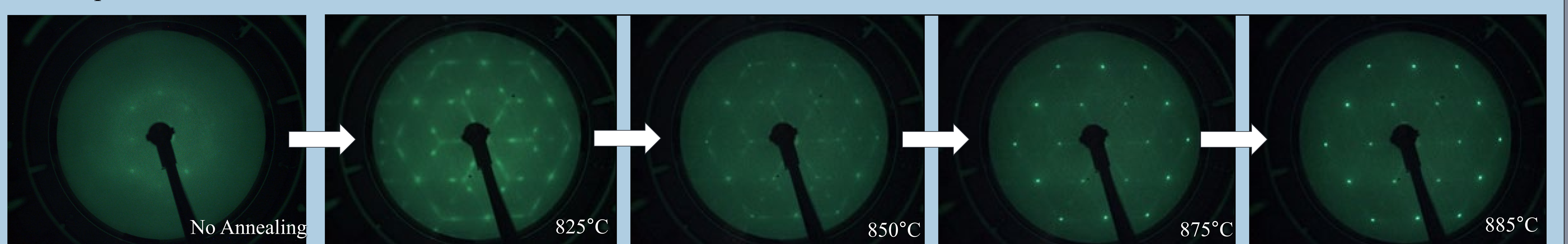
Electrochemical Polishing

The surfaces of LiNbO3 used in this experiments were electromechanically polished with surface roughness of 5 Angstrom in commercial process. However, this process is not creating surfaces that shows LEED pattern. In order to get LEED pattern the samples require to be annealed in UHV to temperatures above 700 °C. After that UHV annealing the samples were exposed to the air, water and alcohol for 5 minutes. The LEED pattern was still observed after the exposures. This suggest that the commercial electromechanical polishing process is not fabricating well defined surface crystallography.

Stoichiometric LiNbO3 demonstrate structural phase transition at temp. over 875 °C to recrystallize to bulk terminated structure with no reconstruction.

Stoichiometric $LiNbO_3$ Phase Transition

The observed phase transition with increasing annealing temperatures will be the topic of a future study at OCI Vacuum Microengineering! Before any annealing there is a LEED pattern, after annealing a LEED pattern with reconstruction can be seen. After annealing past 875 °C a LEED pattern without reconstruction can be seen.



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