

Exploring stresses to develop functional nanoceramics by in-situ TEM sintering

Exploratory R&D projects in Emerging Technologies

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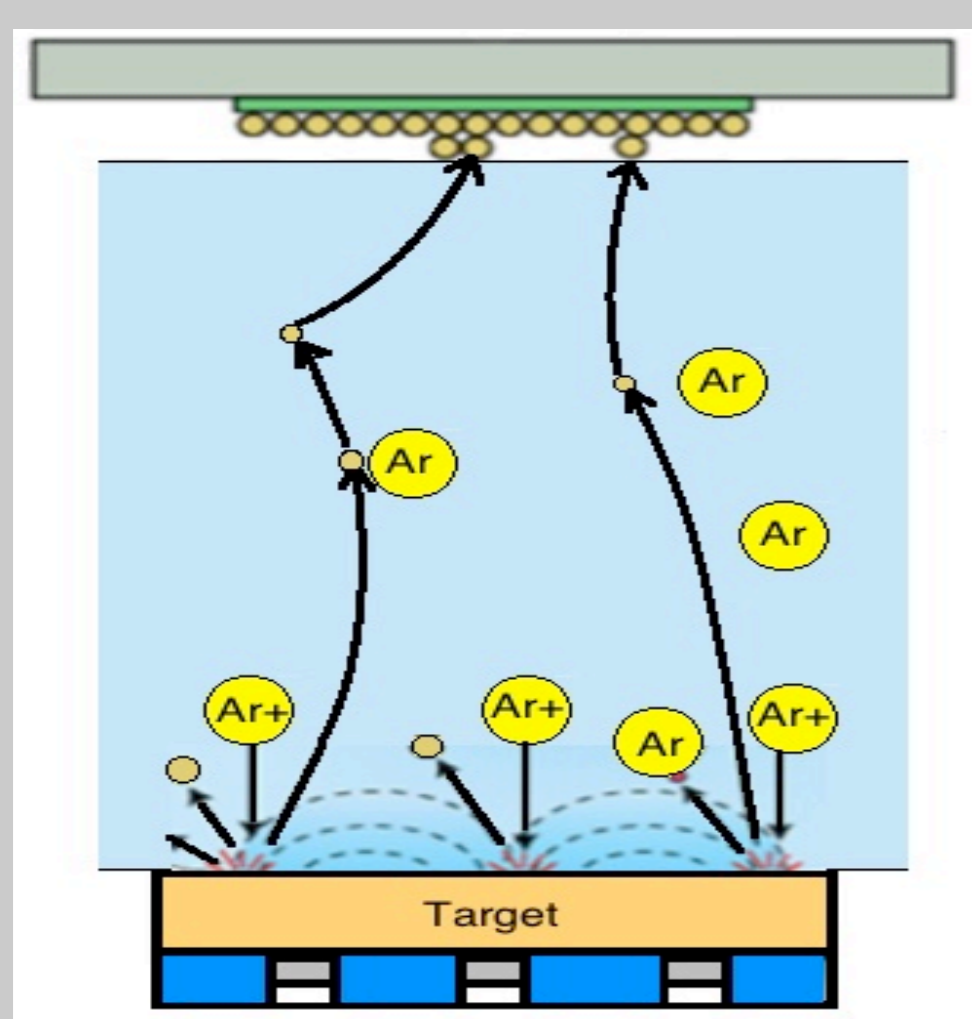
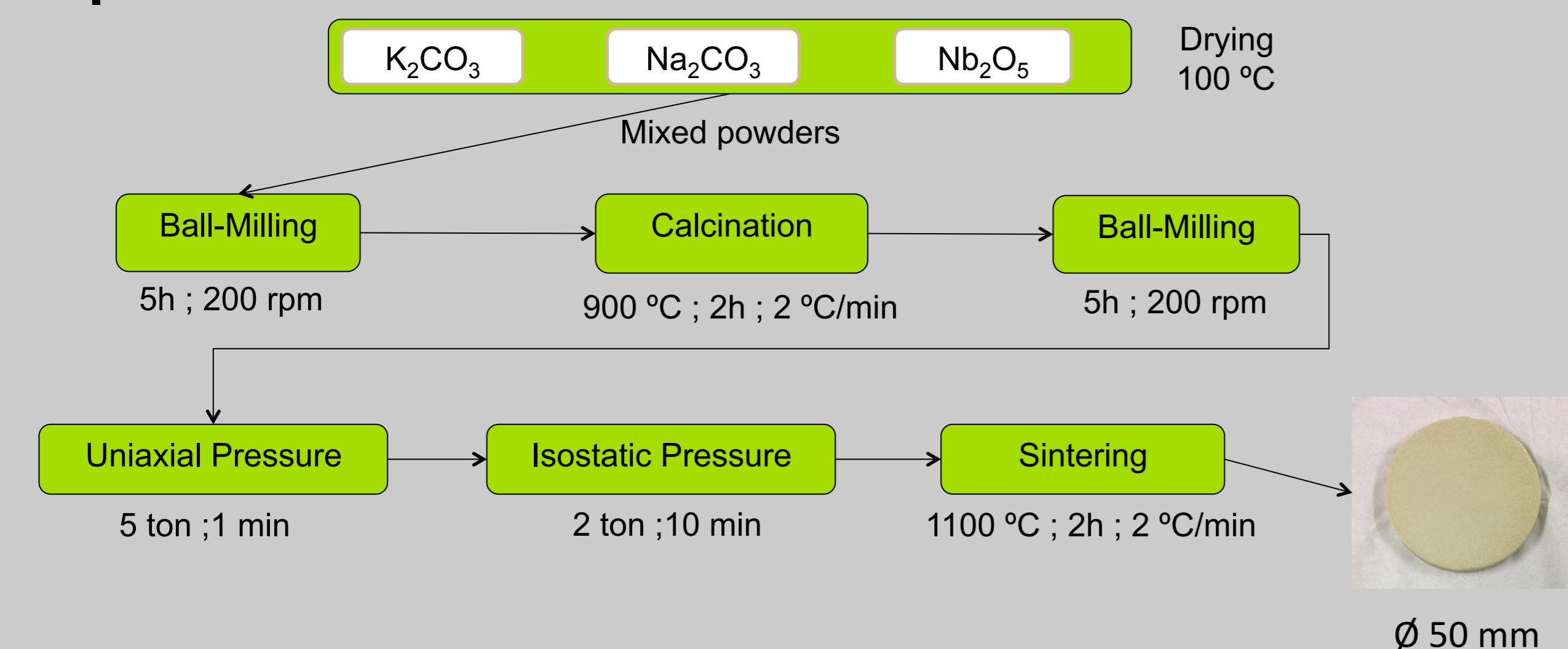
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Part I : Fabrication of KNN thin films by sputtering.

Introduction and Motivation

The development of lead-free piezoelectric 2D/3D ceramics is considered a priority topic of research since 2005, in response to the European Union (EU) concern about the use of lead in electronic devices¹. Therefore, researcher efforts led to the search of alternative piezoelectric materials in order to replace Pb(Zr,Ti)O₃ (PZT). Sodium potassium niobate (KNN) is one of the most promising lead-free piezoelectric ceramic, owing to their good piezoelectric properties and high Curie temperature. However, the electromechanical coupling of KNN needs to be improved to produce competitive KNN-based materials. It is known that those properties are highly dependent on the texture and grain size^{2,3}, which can be affected by an applied stress or presence of internal strains developed during sintering, as is the case of ceramic thin films on rigid substrates⁴. In this work, we investigate the role of stresses on the microstructure of KNN thin films by in-situ sintering; for such, we are trying an innovative strategy of developing stresses in thin films during heating, using different thermal expansion substrates and this part of the work is concerned with the preparation of those 2D samples.

Experimental



Scheme of magnetron sputtering process

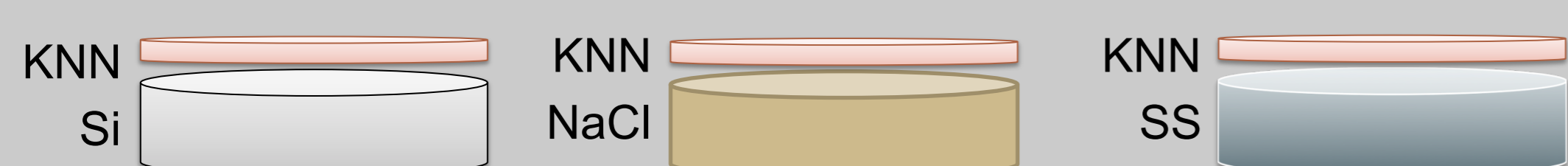
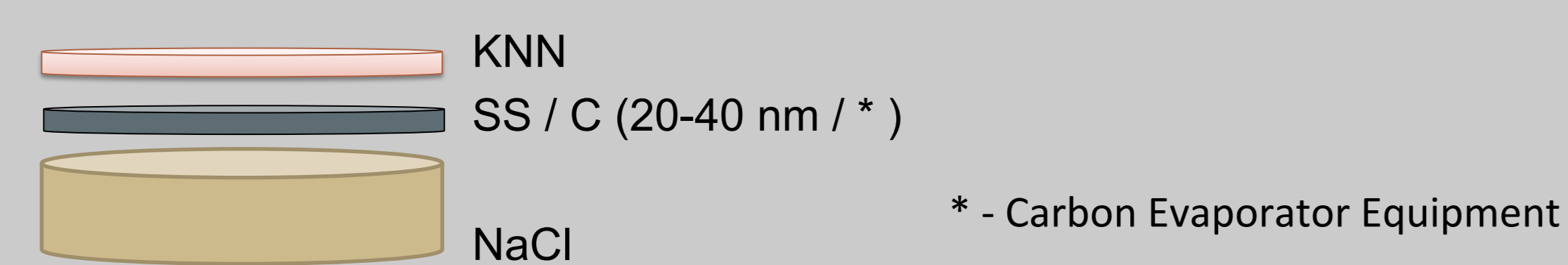


Magnetron Sputtering System.

Material	KNN	SiC	SS
Source	RF	RF	DC
Power (W)	100	80	200
DC-Bias (V)	190	190	—
Atmospheric Pressure (Pa)	0.5	0.55	0.3

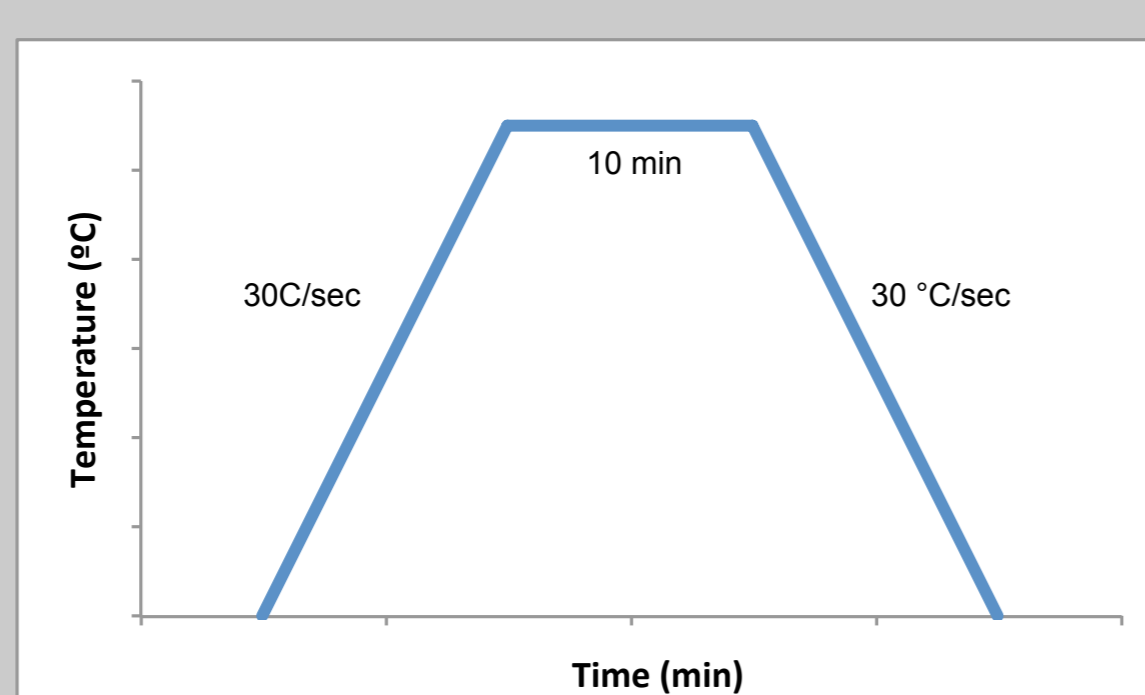
KNN – Sodium Potassium Niobate; SS – Stainless Steel

Thin Films Deposition Design



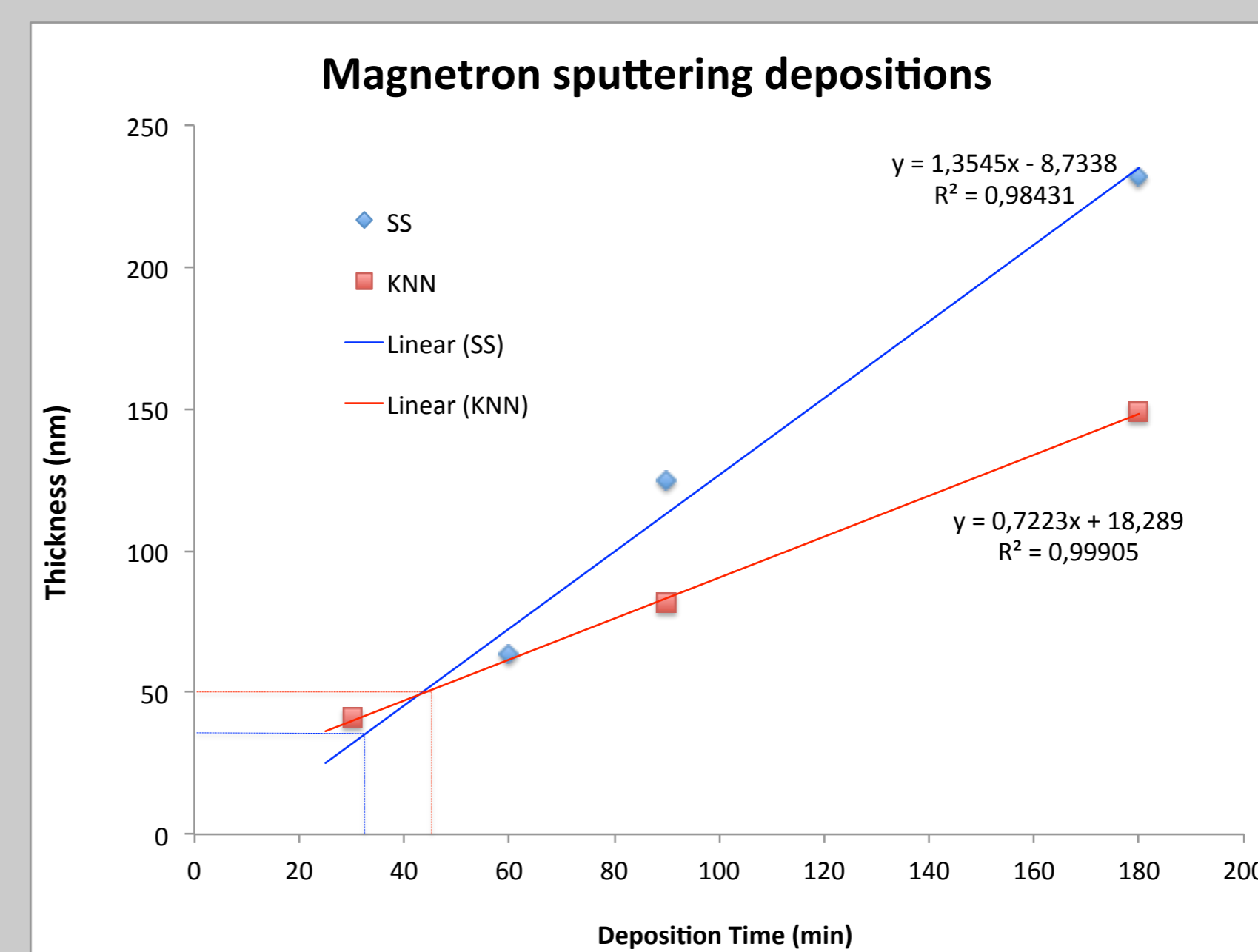
Thermal Treatments

KNN thin films were thermal annealed by Rapid Thermal Annealing system (RTA) [550;600;650;700] °C

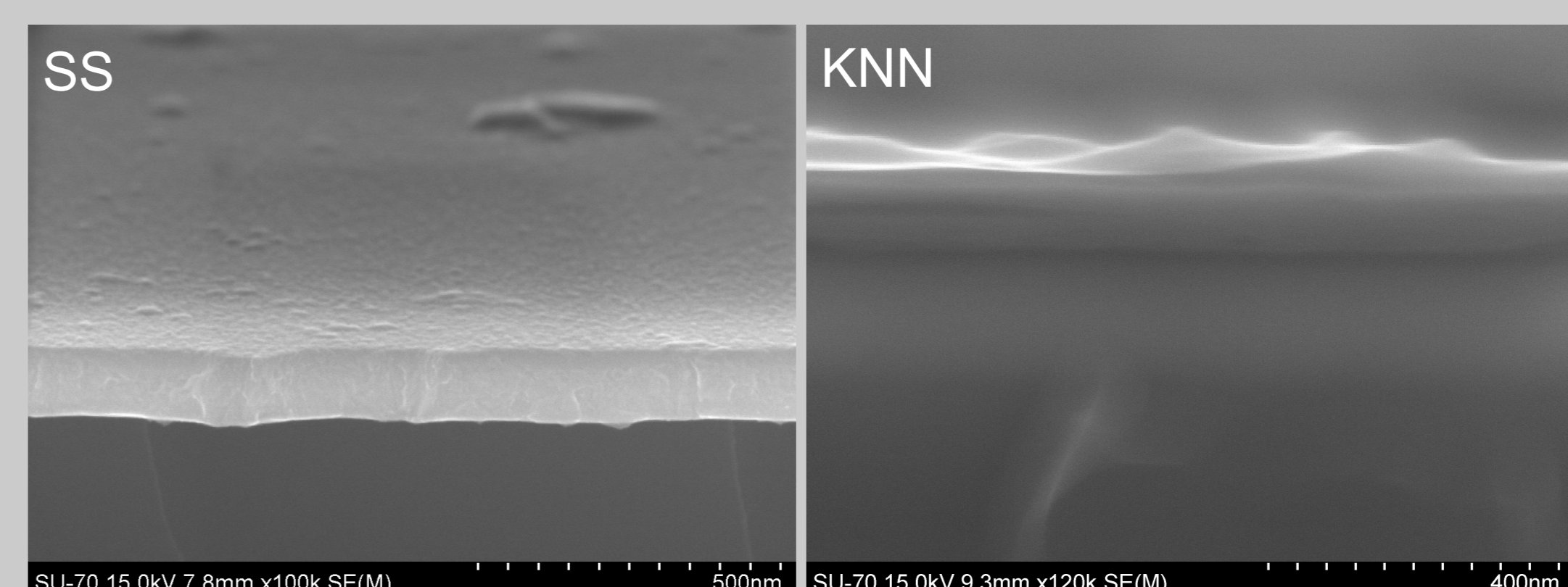


Results and Discussion

Thin Film Thickness Calibration



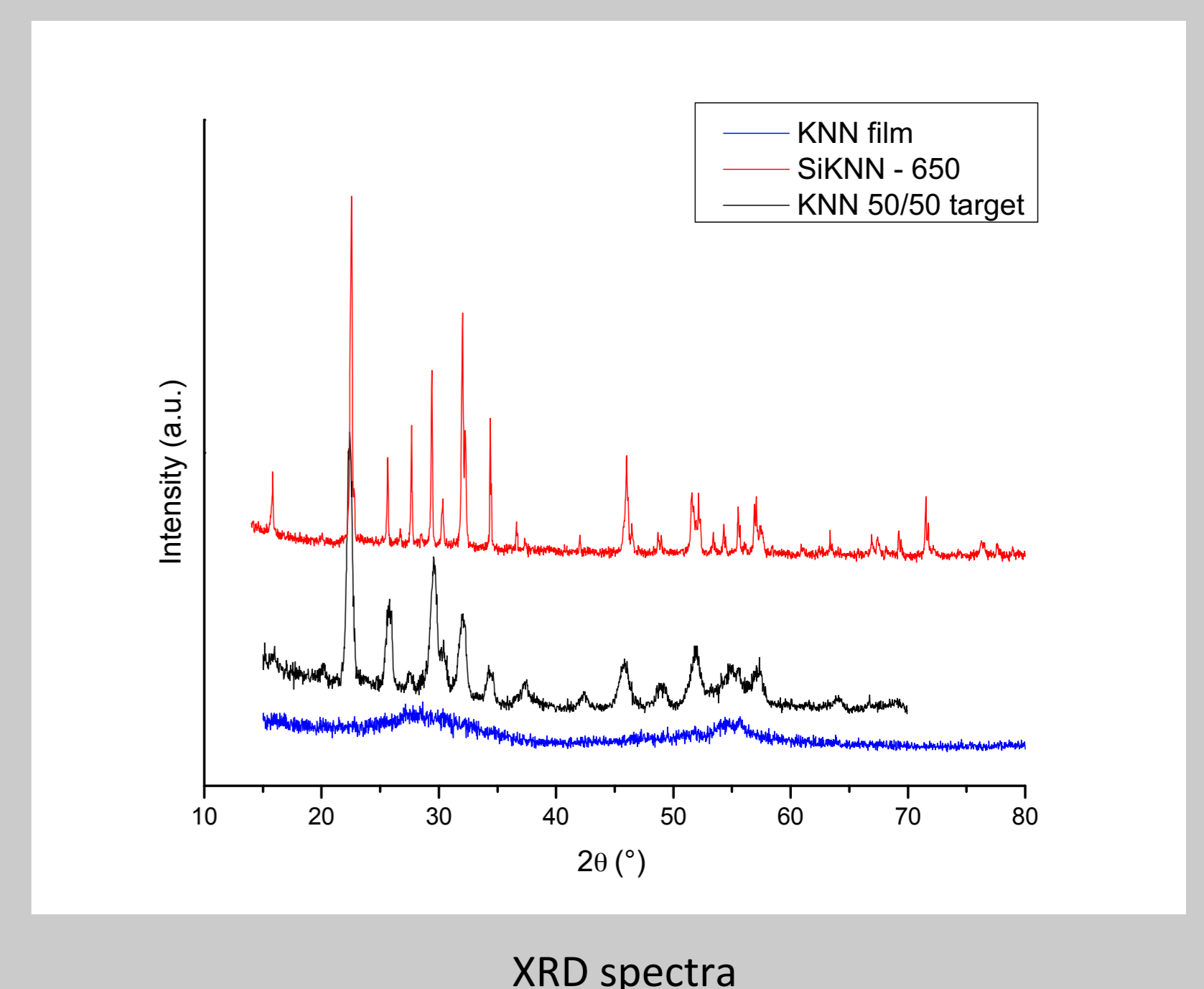
Calibration curves to determine the thickness dependence of the deposition time (thickness values determined by cross section SEM images)



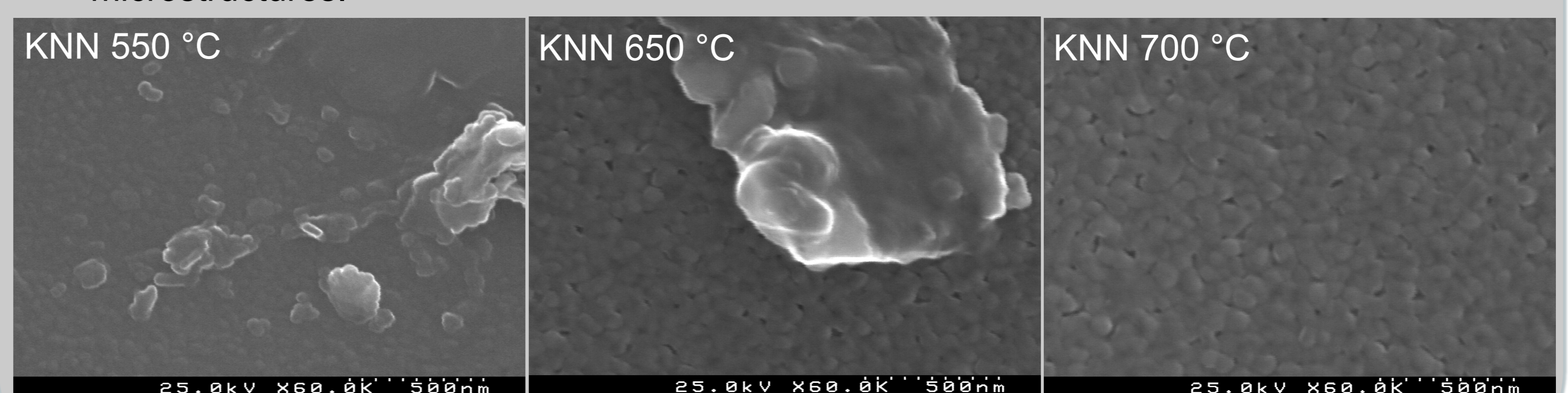
SEM images of SS and KNN thin films.

Characterization of the produced films

- The KNN thin films present uniform thickness ~50 nm and good adhesion on Si, NaCl, and SS substrates.
- The adhesion between multilayers thin films was also good, with exception for the SiC layer.
- The structural characterization of KNN thin films reveals two phases of KNN with different stoichiometry.
- Crystalline structure was achieved after RTA of KNN films at temperatures ~650°C. SEM images show high homogeneous and nanometric microstructures.



XRD spectra



SEM top view of KNN thin films after RTA.

Conclusions

Thin films of good quality were produced by sputtering. The KNN film presents good adhesion on the selected substrates. Two different phases were noticed in KNN thin films. The stoichiometry control is under research. RTA results show a nanometric structure of the KNN up to 700 °C.

References

- European Commission. *Off. J. Eur. Union* **2005**, No. 8, 22–142.
- Rafiq, M. A. Electromechanical properties of engineered lead free potassium sodium niobate based materials, University of Aveiro, 2013.
- Soares, M. R.; Senos, A. M. R.; Mantas, P. Q. *J. Eur. Ceram. Soc.* **2000**, *20* (3), 321–334.
- Huang, J.; Liu, J.; Li, Z.; Zhu, K.; Wang, B.; Gu, Q.; Feng, B.; Qiu, J. *J. Mater. Sci. Mater. Electron.* **2016**, *27* (1), 899–905

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