Embedded Nanoporous Sensors for the Electrical Detection of Degradation Products, FY22

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Introduction

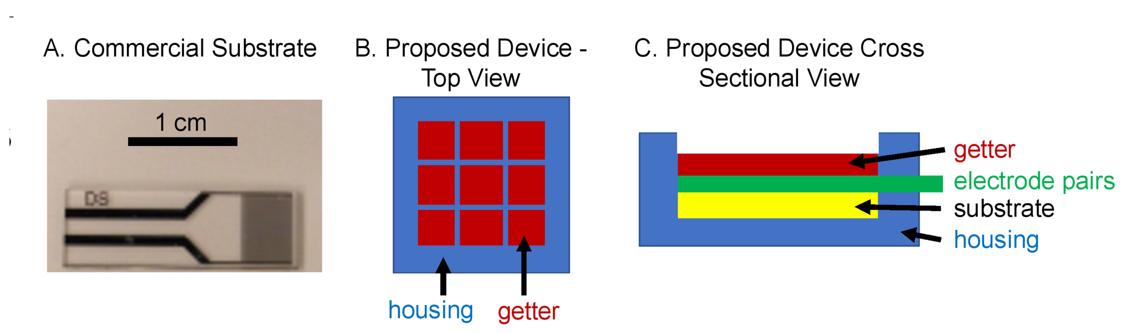
Advanced diagnostics for monitoring of degradation products have been called out as specific needs by both the JASON Defense Advisory Panel (JSR-97-320, 1998) and SNL.

An embedded sensor tuned to high specificity of target off gas molecules of interest will provide a unique dataset with which to characterize materials degradation, potentially providing a path to *in-situ* diagnostics monitoring; 'near zero' powered (<10nW) sensors for wide environment range.

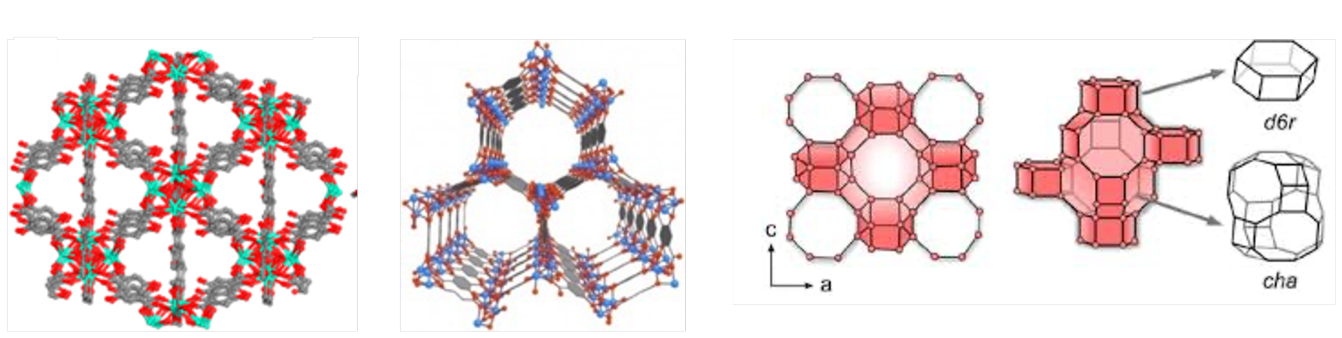
A&L project successes are leveraged to DoD collaborations offering an environment for maturing technologies that are relevant to the DOE mission space.

Project Aim

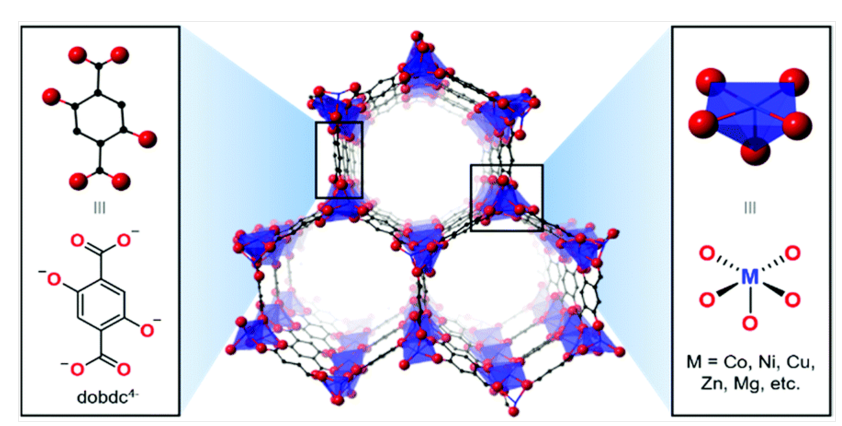
- Our aim is the development of a novel, robust, compact (~1 cm^3) embedded sensor for direct electrical detection of materials degradation products in nuclear weapons systems.
- The device should have low power requirements, exhibit stable and selective performance over a wide temperature range, and survive a variety of vibration conditions.
- The ability to selectively detect degradation products will introduce a new diagnostic capability to assess the health of nuclear weapons systems, providing confidence in the functional performance of the weapons system.
- We are interested in an integrated sensor, which will record degradation products during the sensor's lifetime.
- Project is divided into three main tasks:
  - Design of nanoporous getter materials
  - Materials integration
  - Electrical Testing



Nanoporous Getter Materials



Crystal structures of:
(1) RE-DOBDC MOF, (2) **MOF-74**, and (3) SSZ-13 zeolite



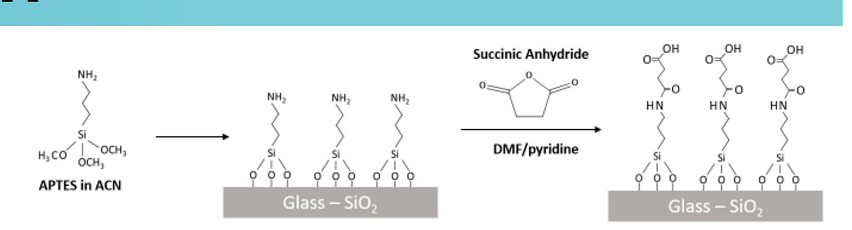
M-MOF-74; M = Ni, Co, Mg

Sava Gallis, et.al.,*ACS Appl. Mater Interfaces* **2019**, *11*, 43270.; Percival, et.al., *I&ECR*, **2021**, *60*, 14371.

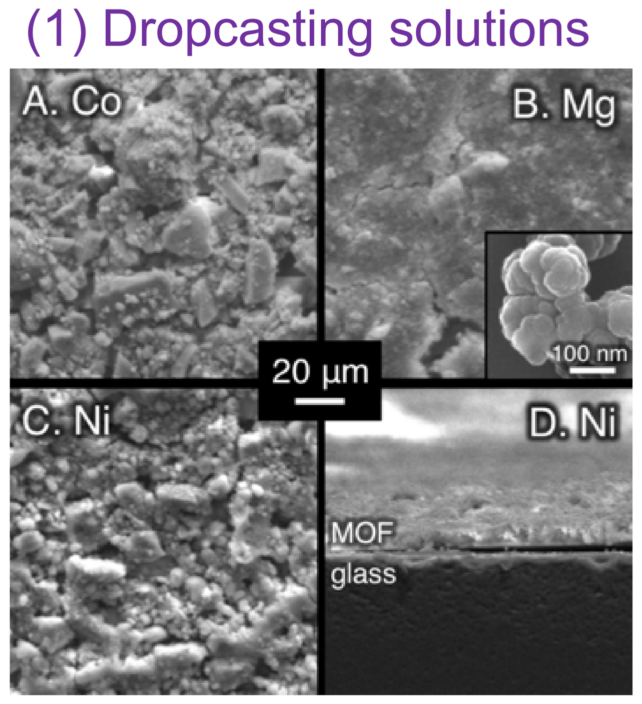
Materials Integration

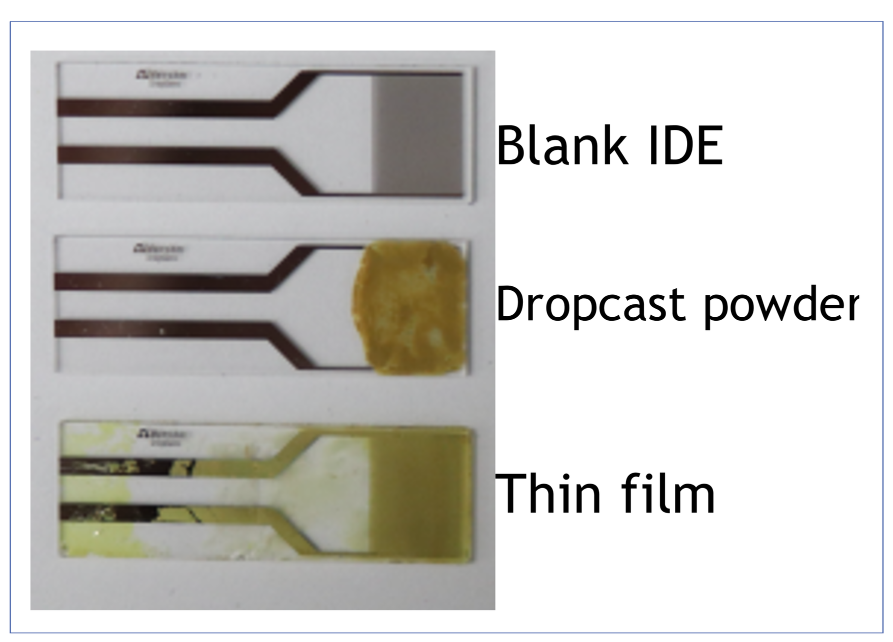
Henkelis, et.al, *Membranes*, **2021**, *11*, 176.

Thin film M-MOF-74 membranes fabricated by two methods:

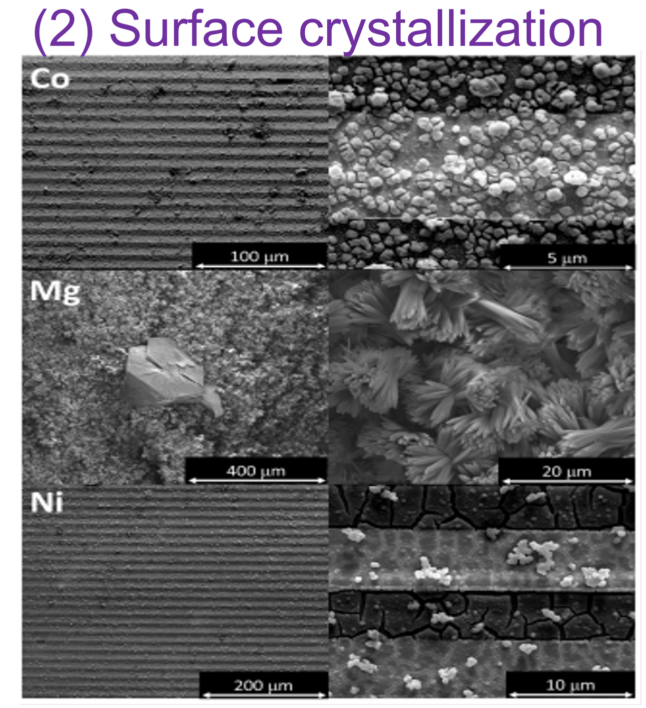


(1) Dropcasting solutions

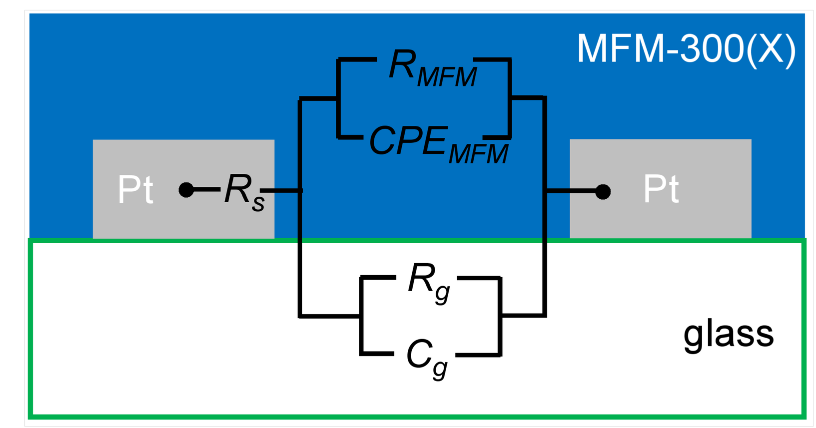




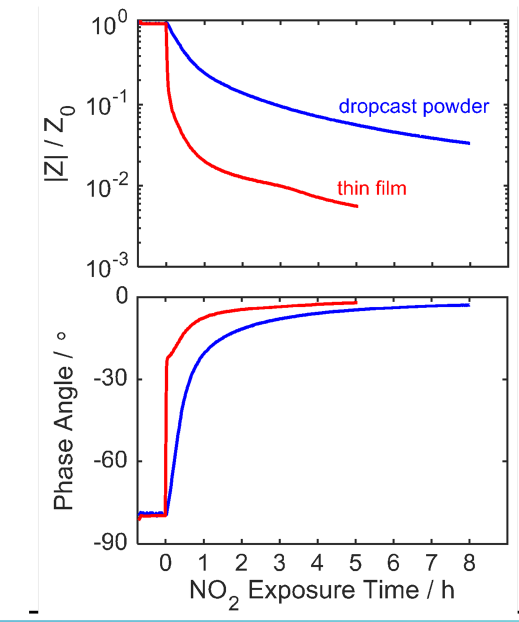
(2) Surface crystallization



Electrical Testing



An equivalent circuit fit to impedance data overlaid on a cross sectional schematic of the sensor

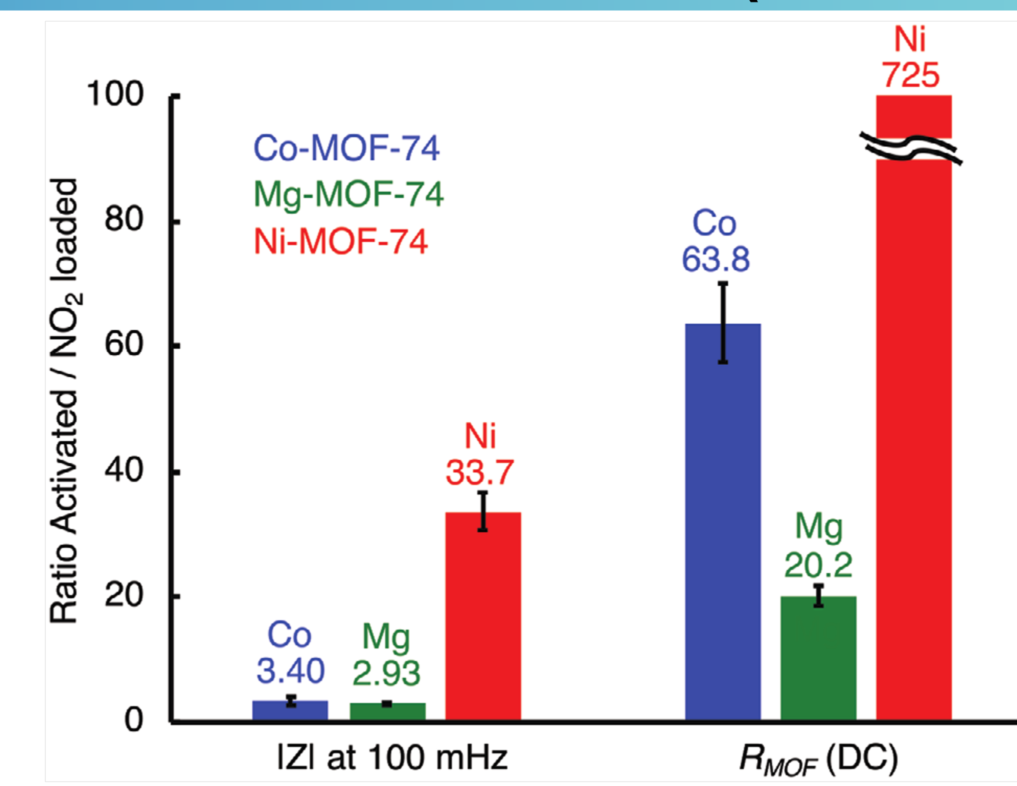


MOF/IDE direct electrical Impedance response of drop cast MOF film (blue) vs. crystallized thin film (red) MOF.

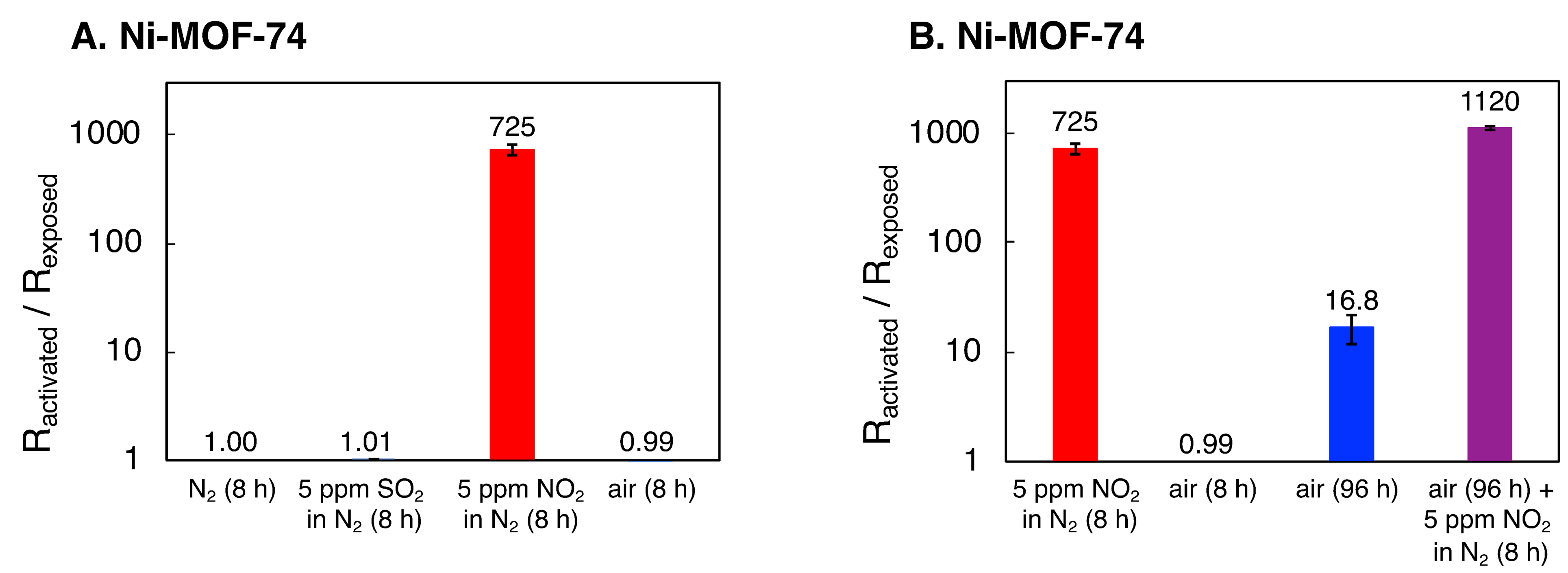
Faster kinetics and response from the thinner thin film version.

NO_x Detection, M-MOF-74 (M = Ni, Co, Mg)

Small, et.al, *Adv. Func. Mater.* **2020**, 2006598



Ratio of response as-activated to NO_2-exposed for (1) impedance magnitude at 100 mHz and (2) MOF DC film resistance for IDEs coated with M-MOF-74 (M = Co, Mg, Ni). NO_2 exposure was at 5 ppm NO_2 for 8 h at 50 °C.



Ratio of Ni-MOF-74 resistance (R_{MOF}) when exposed to different environments at 50°C; highly selective response towards NO_2. (A) 8 h exposures and (B) comparison of response of extended air-exposure combined with subsequent NO_2 exposure. "Air" is ambient atmosphere (25 °C 50% RH, 400 ppm CO_2, 21% O_2), then heated to 50 °C.

Conclusions

Combining tuned nanoporous materials (such as metal organic frameworks (MOFs) and zeolites) that are Highly selective with an electrical support, enables the fabrication of integrated sensors with sensitivity to target degradation product gases. The high selectivity of the material decreases interference from common and plentiful common 'air' component gases.

The proven 'low power' requirements for this sensor make it attractive to long term and multi-environmental conditions. Durability studies have shown films are in accordance with ASTM adhesion standards. Current successes enable on-going device developmental studies with DOD partners.

Acknowledgements

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