

***In situ* fabrication of chemical sensing arrays in extreme environments**

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Chemical and biochemical sensors commonly utilize detection mechanisms that rely on highly reactive chemical interfaces (e.g. enzyme, polymer, membrane, thin-films) to probe test environments for target analytes. This active sensing interface is frequently a consumable material that is prone to performance degradation during extended space missions and in extreme environments due to stress and aging. Compared to implementation on space missions or in extreme environments, this problem is less of a restriction in ground-based laboratories, where new sensors can be made as needed, or in industrial settings where components can be replaced.

To overcome this problem, we have developed a micromachined device for time-of-use *in situ* fabrication of electropositive metal sensor arrays for the detection of electrophilic trace gases in extreme environments. Our approach is based on the *in situ* thermal deposition of reactive metal thin-films onto electrodes that have been photolithography patterned on a sensor substrate. Fabricated using bulk Si micromachining, the microdevice consists of a silicon frame with suspended silicon nitride membrane seals. Once delivered to the planet's surface, a lithographically defined circuit on the inside of silicon nitride membrane is used to deposit an array of eight chemically sensitive thin-films on to the sensor substrate. After the film deposition is complete, a second circuit is used to rupture the silicon nitride membrane, exposing the sensing film to the environment.

Once deposited, the reaction of the target analytes with the thin-film sensors, as well as the identification of bulk vs. surface processes, can be measured by following, via impedance spectroscopy, the change in complex impedance of the sensor as a function of time. Our target analytes are low levels of reactive oxygen and hydrogen species including O₃, O•, H₂O₂, and •OH that are believed to be present in the near-surface atmosphere of Mars.

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SAXS and SANS as probes of sorption of CO₂ in the pore space of coal

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Sorption of gases in coal occurs inside the pore system where pore sizes are distributed over a wide range from less than 1 nm to hundreds of μm in diameter [1]. Both X-rays and thermal neutrons can penetrate millimeter-thick samples of coal, and their scattering pattern at small angles (SAXS and SANS, respectively) provides microstructural information in reciprocal (Fourier) space. These observations can be used to calculate the internal surface area, pore size distribution [2, 3] and the density of fluid contained in pores of specific sizes [4, 5].

As the pore size range accessible to small angle scattering techniques extends from ~1 nm to ~20 μm for SANS/USANS and from 0.5 nm to 1 μm for SAXS/USAXS, both methods are in principle suitable as non-destructive quantitative assessments of the micro- and nanopores in rocks. In applications involving sorbed gases, however, issues like incoherent scattering, detailed contrast considerations, instrumental beam size and intensity, etc., become important. It turns out that gas sorption averaged over the sample volume in pores in the size range from 5 nm to 20 μm can be conveniently studied with neutrons. In contrast, gas sorption in pores smaller than about 5 nm, including kinetics, at high spatial resolution (down to voxels ~500x10x10 μm³) is best studied using synchrotron X-rays [6].

In this presentation we will discuss general principles of SAXS/USAXS and SANS/USANS methods and show the results of a SAXS study of sorption kinetics of sub-critical CO₂ in a sample of coal.

[1] Radlinski (2006) *Reviews in Mineralogy & Geochemistry* **63**, 363-397. [2] Radlinski *et al.* (2004) *Coal Geology* **59**, 245-271. [3] Mares *et al.* (2009) *Coal Geology* **77**, 54-68. [4] Melnichenko *et al.* (2004) *Physical Review E* **69**, 057102-057105. [5] Radlinski *et al.* (2009) *Langmuir* **25**, 2385-2389. [6] Radlinski *et al.* (2009), *Coal Geology* **77**, 80-89.