Thermal gravimetric analysis of lithium oxidation in air.

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ABSTRACT

Liquid lithium plasma facing components (PFCs) are being developed to handle long pulse, high heat loads in tokamaks. Wetting by lithium of its container is essential for this application, but can be hindered by lithium oxidation by residual gases or during tokamak maintenance. Lithium PFCs will experience elevated temperatures due to plasma heat flux. This work presents measurements of lithium reactions at elevated temperatures (298-373 K) when exposed to natural air. Cylindrical TZM wells 300 microns deep with 1 cm^2 surface area were filled with metallic lithium in a glovebox containing argon with <1.6 ppm H_20, O_2, and N_2. The wells were transferred to a hot plate in air, and then removed periodically for mass gain measurements. Changes in the surface topography were recorded with a microscope. The mass gain of the samples at elevated temperatures followed a markedly different behavior to that at room temperature. Increasing temperature from 25 to 150 degrees C results in a decrease in initial mass gain rate. In addition, samples at 100 and 150 degrees C diverged, with some samples rapidly gaining mass between 20 and 30 hours.

Thermal gravimetric analysis of lithium reactions with air

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GOAL

- **Characterize lithium passivation at elevated temperatures**

BACKGROUND

- Lithium plasma facing components (PFCs) are being developed to handle long pulse, high heat loads in tokamaks. However, lithium is chemically reactive and this can affect its performance. Previous results [ref.1] have shown that a lithium surface in a tokamak reacts with residual gases in minutes. Oxidation of the bulk requires interdiffusion of lithium and oxygen. At room temperature, this process slows oxidation by 5-6 orders of magnitude. Room temperature oxidation was characterized as shown in figure 1 [ref.2]. Markowitz et al., [ref.3] found rapid nitration between 170 - 180°C (figure 2).

- The purpose of the present project is to examine the bulk oxidation rate of lithium over extended time periods at elevated temperatures expected in a tokamak.

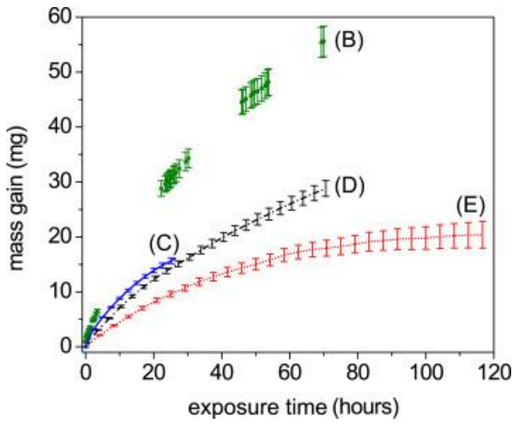
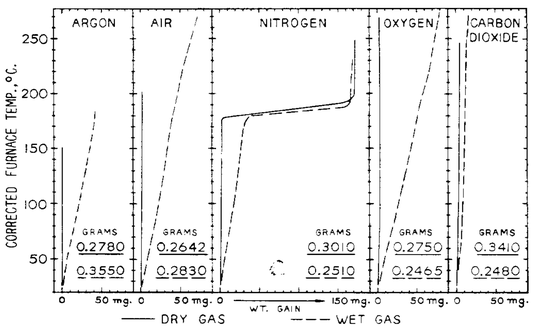


Fig 1 (above)- Hart et al. room temperature passivation
Fig 2 (below)- Markowitz et al. constant heating TGA



Method

- Lithium compounds have a higher mass than elemental lithium. Small samples of metallic lithium (~20 mg) in TZM wells were prepared in an argon glove box and exposed to 80°F and 50% relative humidity air while on a hot plate at temperatures up to 150°C. Approximately every hour the well was cooled, the increase in mass measured on a balance and the lithium surface imaged under a microscope, before returning the lithium to the high temperature hot plate.

Fig 3- Experimental setup

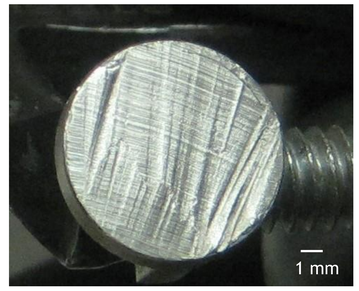
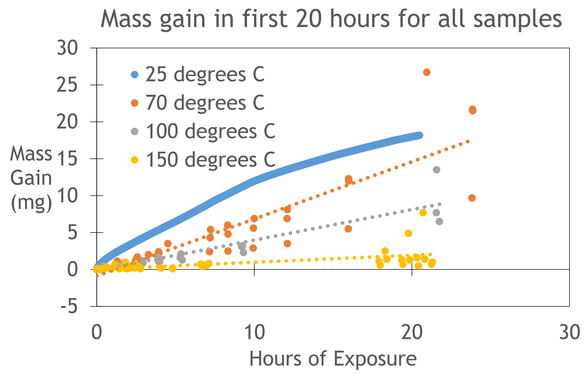


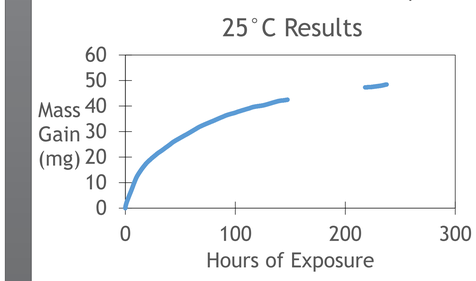
Fig 4- Representative metallic lithium sample (pre exposure 70°C Well 2)

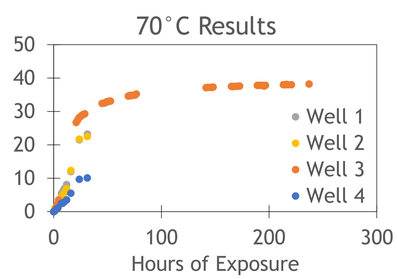
Results

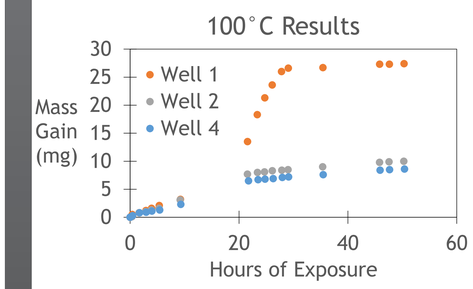


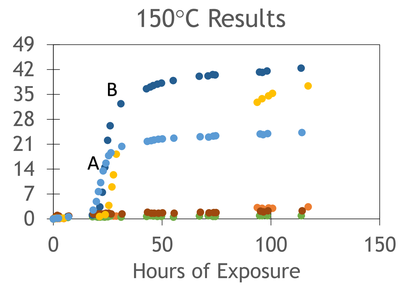
- Increasing temperature decreases the initial mass gain rate of lithium exposed to humid air (left)

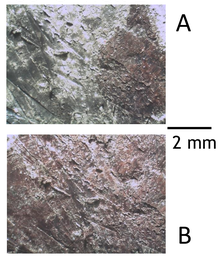
- Divergent behavior occurs between 100 and 150°C. Well 1 at 100°C and Wells 2 and 4 at 150°C diverged from the other samples at these temperatures and experienced rapid mass gain between 20 and 30 hours of exposure. These results are shown below.







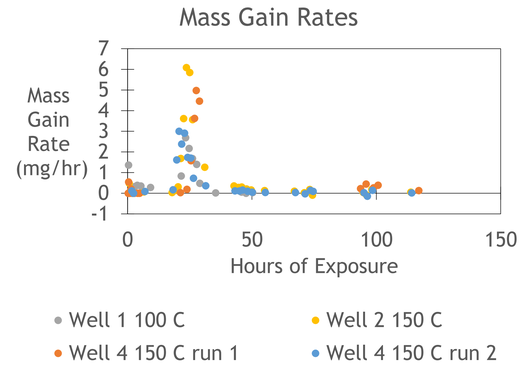




- Rapid mass gain at 150°C between 20 and 30 hours correlated with the formation of a reddish surface possibly indicative of lithium nitride as displayed by points A and B on the 150°C results graph and the above photos.

Results Continued

- Average mass gain rates between measurements for the 3 samples that experienced rapid mass gain between 20 and 30 hours are displayed below.



Conclusions

- The Initial rate of bulk lithium oxidation in humid air is lower at elevated temperatures up to 150°C
- After 20 hours some samples experience rapid mass gain
- Further analysis including X-ray photoelectron spectroscopy and Raman spectroscopy is in progress

REFERENCES

1. Skinner et al., J. Nucl. Mater 438 (2013) S647.
2. Hart et al., J. Nucl. Mater. 468, (2016) 71.
3. Markowitz et al., J. Chem. Eng. Data (1962) 586.

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Acknowledgements

Special thanks for invaluable technical assistance to L. Buzi, D. Clark, A. Dominguez, T. Holoman, R Kaita, B. Koel, T Kozub, R Majeski, D. Ortiz, G. Smalley, X. Yang, and PPPL staff.

This work was made possible by funding from the Department of Energy for the Summer Undergraduate Laboratory Internship (SULI) program. This work is supported by the US DOE Contract No. DE-AC02-09CH11466.