Progress on Insulator Coatings

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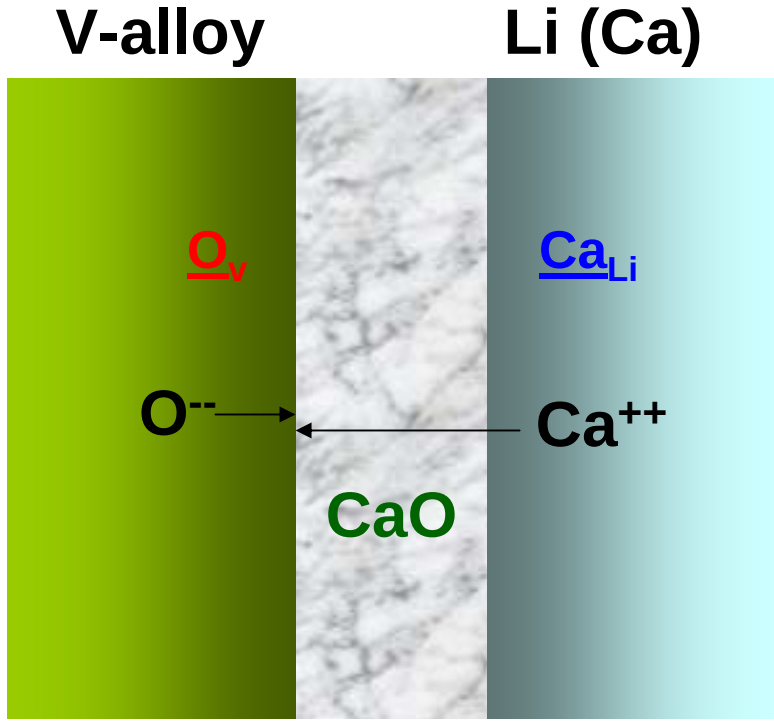
Emphasis on CaO coatings

- Ca in Li reacts with O in V-alloy at interface
  $\underline{Ca}_{Li} + \underline{O}_{V} \rightarrow \underline{CaO}$
- Add <u>O</u> to V-alloy surface
- Control Li chemistry
- If VO is formed on V-alloy
  VO + $\underline{Ca}_{Li} \rightarrow$ Ca-V-O
  Ca-V-O has low resistivity

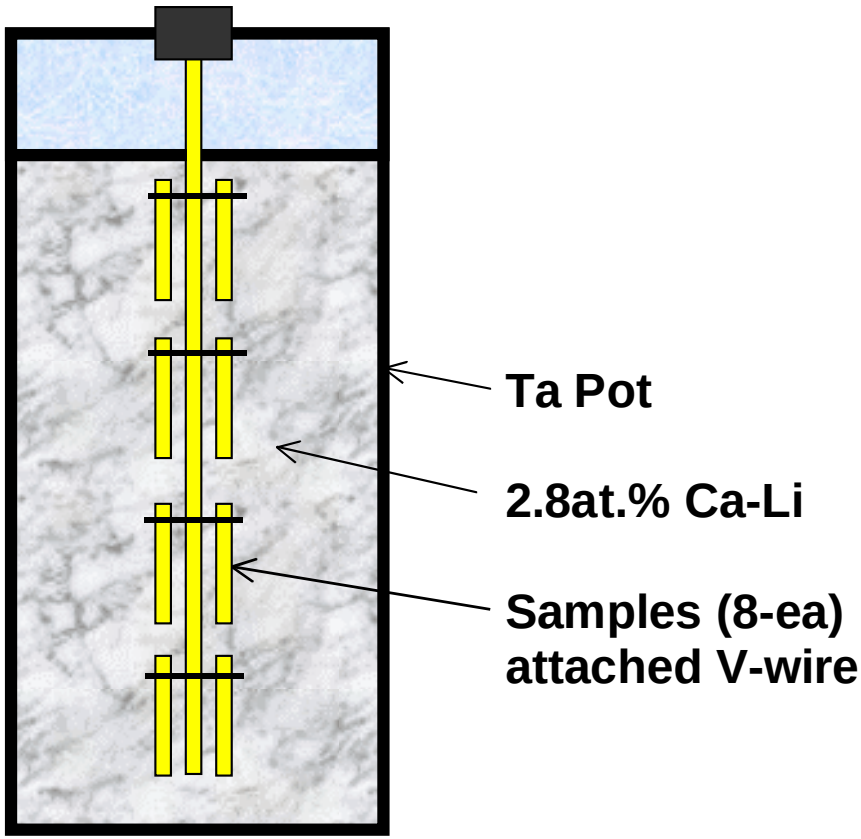


- a) Diffusion of <u>Ca</u> in CaO is higher than <u>O</u> in CaO
  b) Reaction controlled by Ca diffusion

Test Conditions

- Exposure at 600 C
  - 2.8 atomic percent Ca in Li
  - Exposure times to 747 h
  - Test coupons include both US and Japan heat
  - Extensive post exposure characterization
- Exposure at 700 C
  - 2.8 atomic percent Ca in Li
  - Exposure times to 425 h
  - 1000 h exposure in progress

Experimental Apparatus



Schematic for the Ca-Li pot made by Ta bucket inside the aluminized steel container. V-4Cr-4Ti samples attached on the V-4Cr-4Ti sample tree by V-wire and make space between sample tree and samples with small V-4Cr-4Ti spacers.

Test Procedures - 600 C

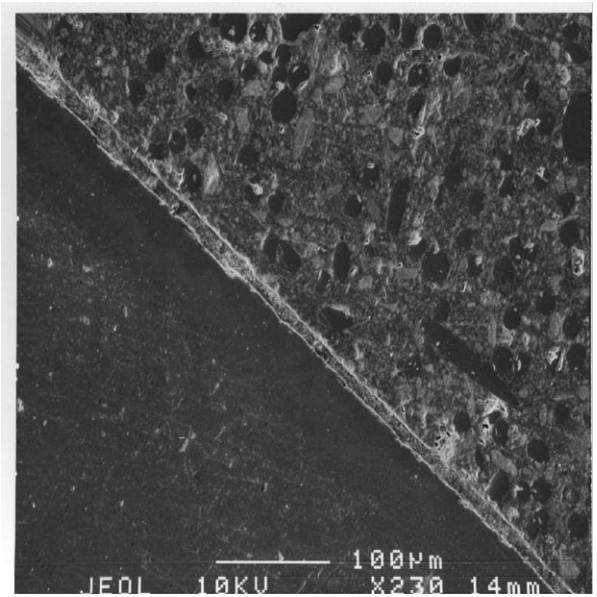
- Oxygen charging
  - Two methods -
    - Ar+10-100ppm O_2 @ 900-950C for 17 h
    - Cr/CrO exposure in sealed quartz tube @ 900-950C for 17 h
  - Oxygen content - 2000-10,000 wppm (6000 typ.)
- Exposure in LiCa
  - CaO coating forms in first 50-100 h
- Post exposure exams
  - SEM
  - EDS
  - Resistivity

Test Results - 600 C

- Uniform CaO coatings of 10-25 μm formed on both US and Japan heats.
- Extended exposures (625 h (J) and 747h(US)) showed intact coatings with very little change following the initial coating formation period.
- Resistance measurements in inert gas showed high resistivity to 700 C.
  - High resistance even after >10 thermal cycles

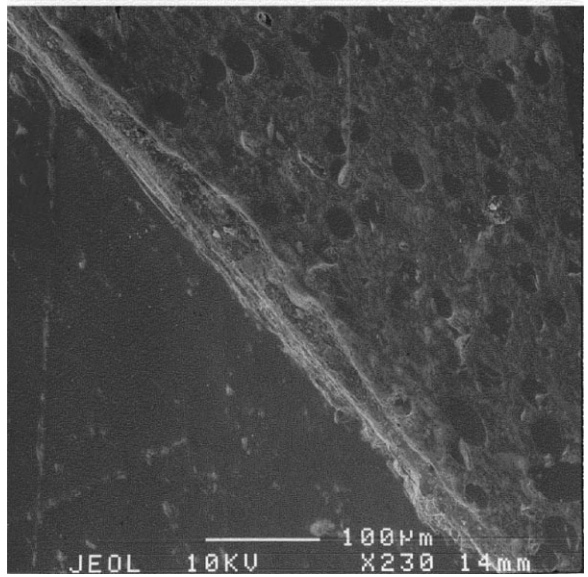
Coating Appearance after 747 h @ 600 C

Low oxygen in V



Sample # 29. Below 500 O-Wppm estimated. Left (or left down) is V-4Cr-4Ti, diagonal layer (~14 µm) is the in-situ formed scale, and right (or right top) is the sample mounting.

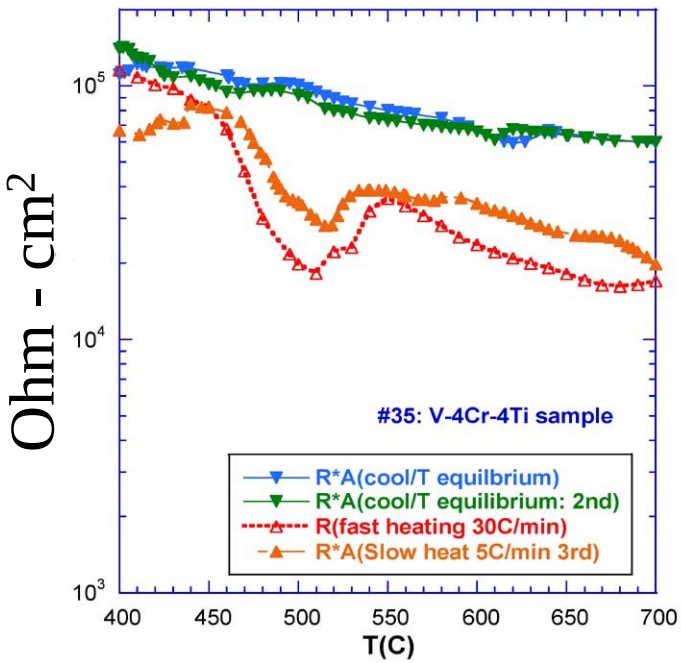
Typical oxygen in V



Sample # 46. 6,400 Wppm-O charged by Ar gas pursing at 700ºC for 18 h. Left down is V-4Cr-4Ti, diagonal layer (18 to ~33 µm) is the in-situ formed scale, and the right top is the mounting.

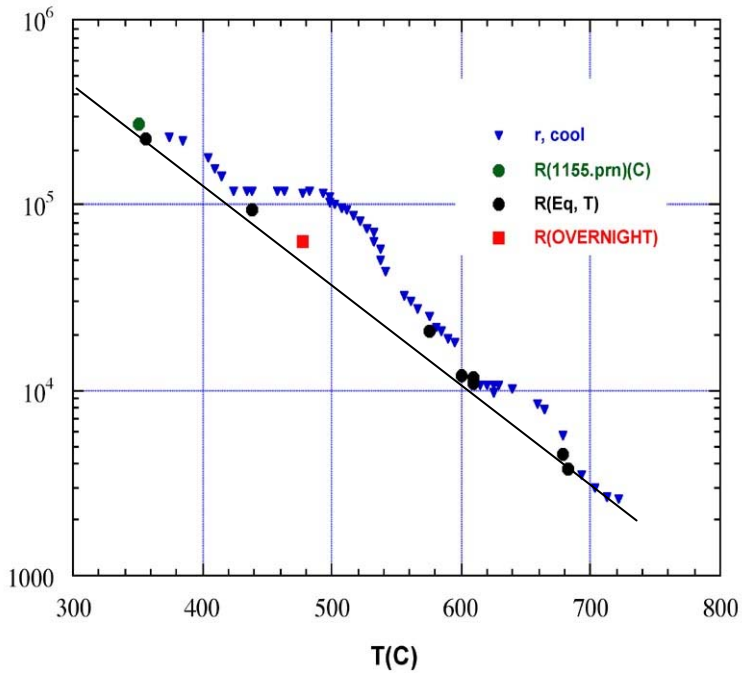
Resistance Following 600 C Exposure

US Heat



Electrical resistance times area vs. temperature in an inert atmosphere for the film formed on the V-4Cr-4Ti #35 (1431 O-wppm) after exposure in the 2.8at.%Ca-Li at 600C for 747.5 h.

Japan Heat



R*A vs. T in an inert atmosphere for the in-situ coated in 2.8at.%Ca-Li for 623 h to the O-charged 5347.6 wppm (7D3) V-4Cr-4Ti (NIFS-II heat).

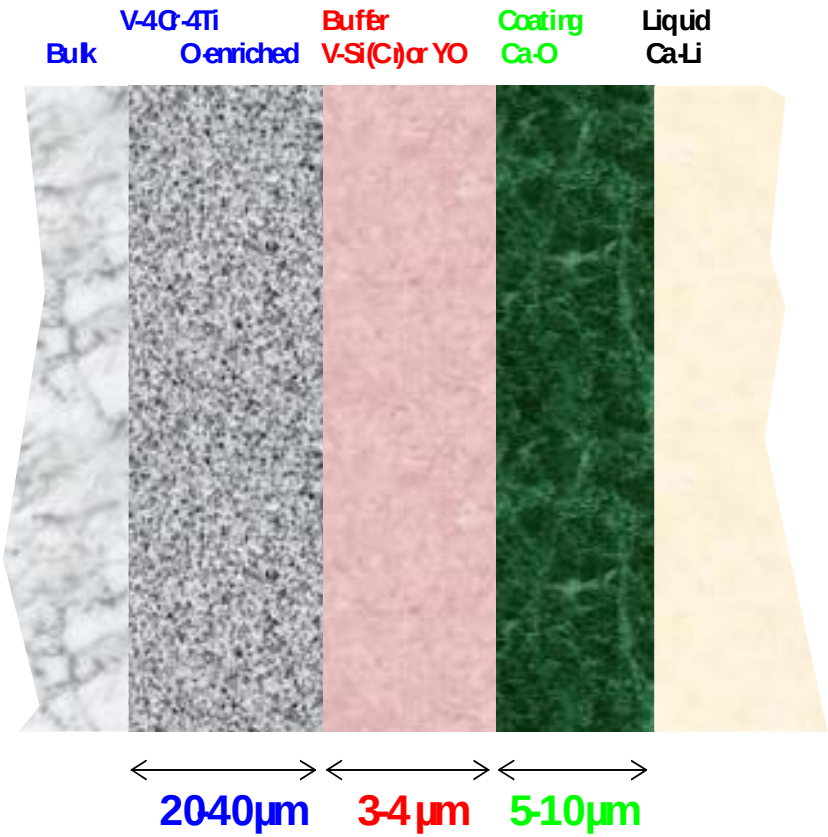
Test Results - 700 C

- Oxygen charging performed exactly as for 600 C tests.
- CaO coating forms in less time (~50 h) compared with 600 C tests.
- CaO coating is intact at 100 h, but dissolution of coating is observed after 425 h.
- Further work is required to understand kinetics of coating growth and dissolution plus effects of local chemistry at 700 C.

Initial Development of Yttrium Oxide Coating

- Vanadium samples were pre-charged with oxygen.
- A thin layer (~0.5 μm) of yttrium was vapor deposited on the vanadium.
- The samples were annealed at 750 C, forming yttrium oxide via a solid-state reaction.
- The resulting coating is extremely adherent.
- Samples were exposed to Li-2.8 at % Ca

Yttrium Oxide Coating



- Exposure to Li-2.8% Ca at 600 C resulted in formation of CaO over the Y_2O_3 film.
- The Y_2O_3 remained intact.
- Further work is planned

Future Work

- Study kinetics and chemistry effects at 700 C exposure.
  - Lower Ca level to 0.5 at %
  - Continue tests to 1000 h
- Continue development of other candidate coatings
  - Yttrium oxide
- Continue optimization of CaO fabrication techniques.

Conclusions

- CaO coatings perform extremely well during 600 C exposure to 747 h.
  - Little change in thickness following initial coating formation period.
  - High electrical resistance following exposure
- CaO coatings are observed to dissolve at 700 C after 425 h in Li-2.8%Ca.
  - Need better understanding of kinetics and local chemistry effects.
- Promising alternate coating candidates and fabrication procedures have been identified, and initial tests have been conducted.