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Thermal analysis of the air-oxidation of high-purity Al foil

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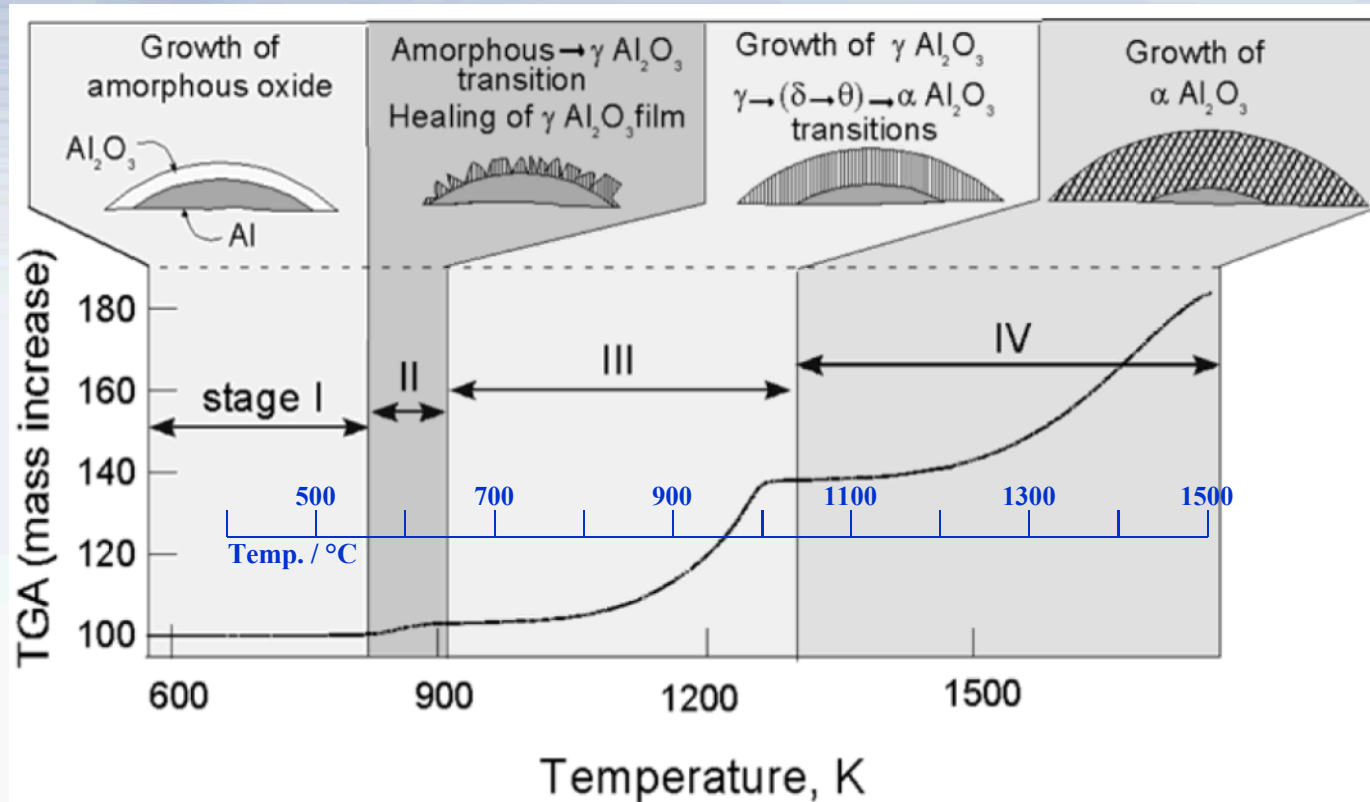
*Solid Propellant Fire Technical Interchange
Meeting
SNL-NM*

March 12th-14th, 2013



Introduction:

Transitions in Al_2O_3 layer on Al metal



Trunov, M.A., Schoenitz, M. and Dreizin, E.L., "Effect of polymorphic phase transformations in alumina layer on ignition of aluminium particles."

Combustion Theory and Modelling, **10**(4), 603-23, (2006).

Thermal Analysis

Materials

- *Al foil; 99.998% (Johnson Matthey)*

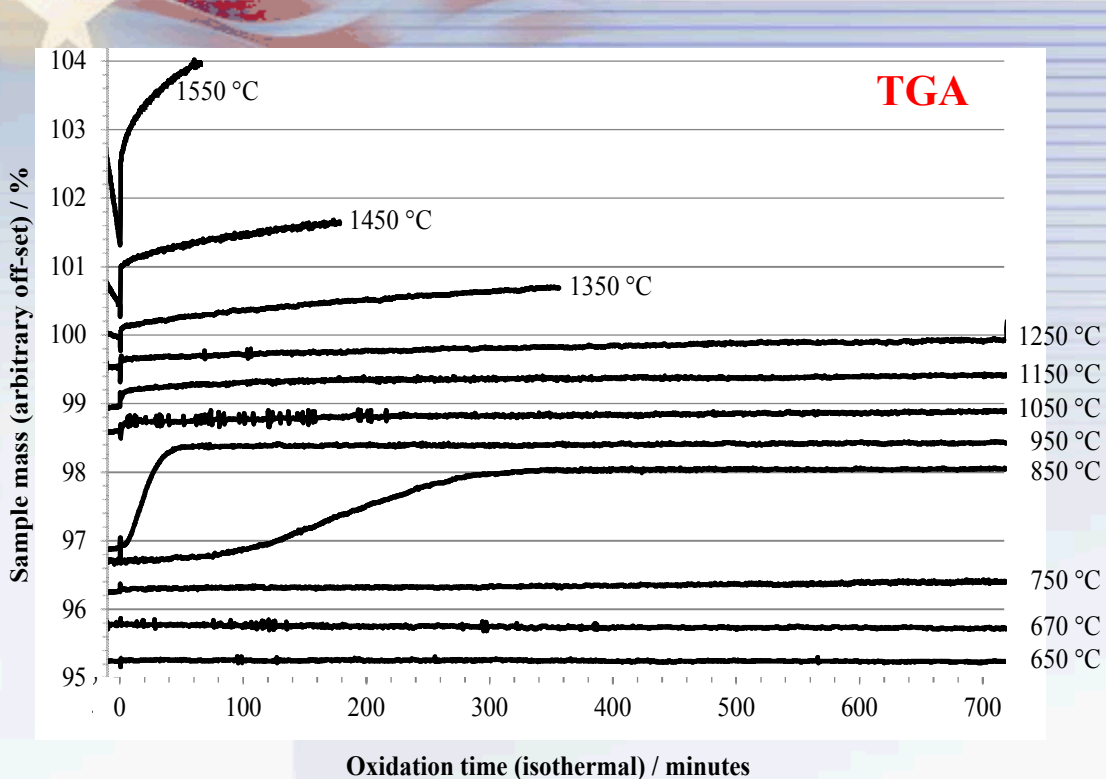
Instrumentation

- *Thermogravimetric Analysis (TGA)* Δ mass
- *Differential Scanning Calorimetry (DSC)* exo/endothrm
 - *Simultaneous TGA-DSC: Netzsch STA 449F3*

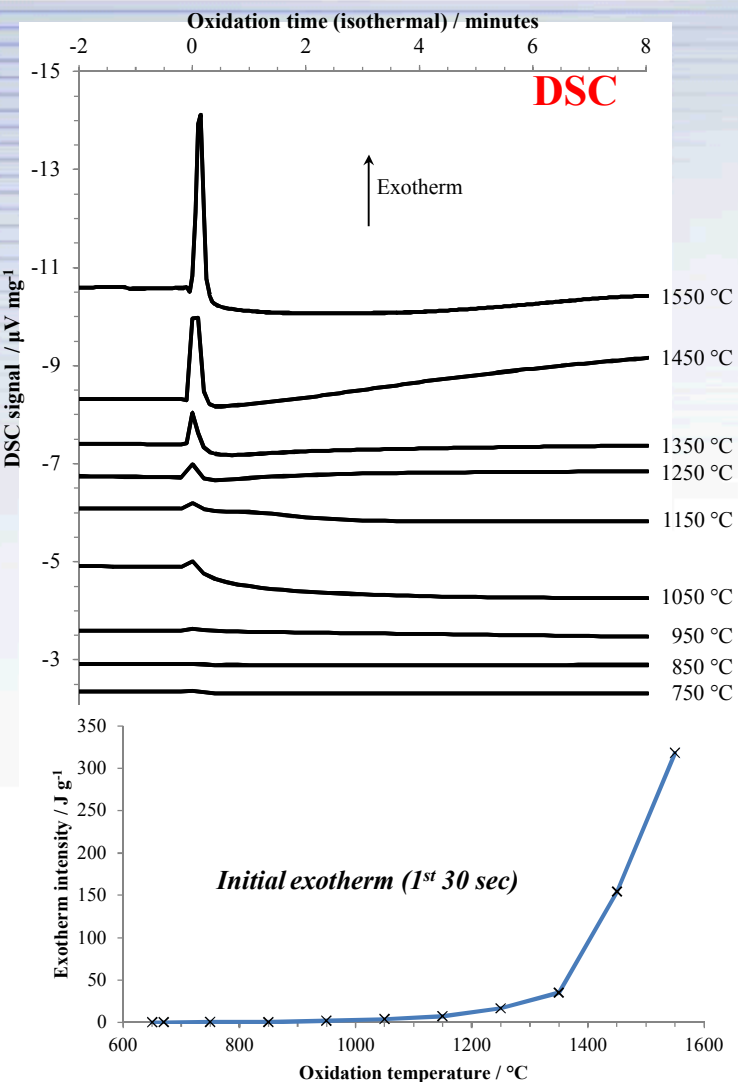
Procedure

- *~20 mg specimen (6 mm disc) in Al₂O₃ cup (6.6 mm o.d.)*
- *Ramp to desired oxidation temperature under Ar (inert)*
- *Allow system to equilibrate at temperature*
- *Expose to air, hold isothermally*

Oxidation of Al foil in air

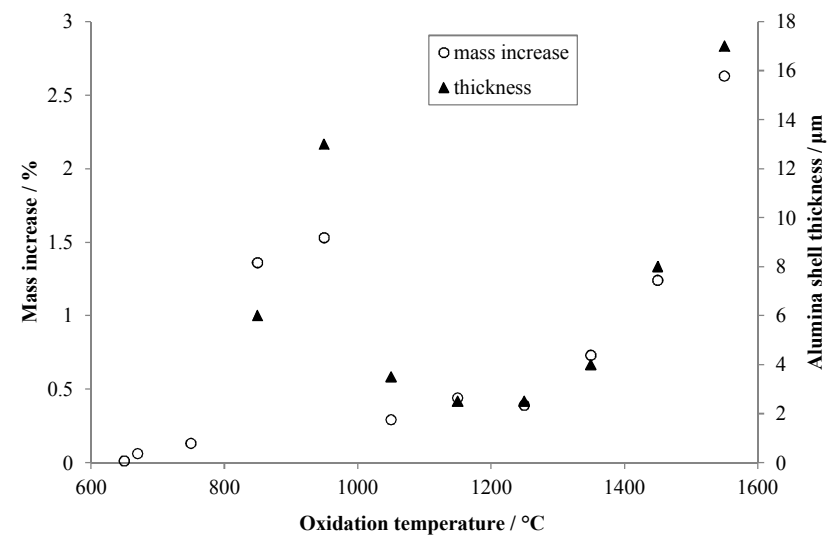
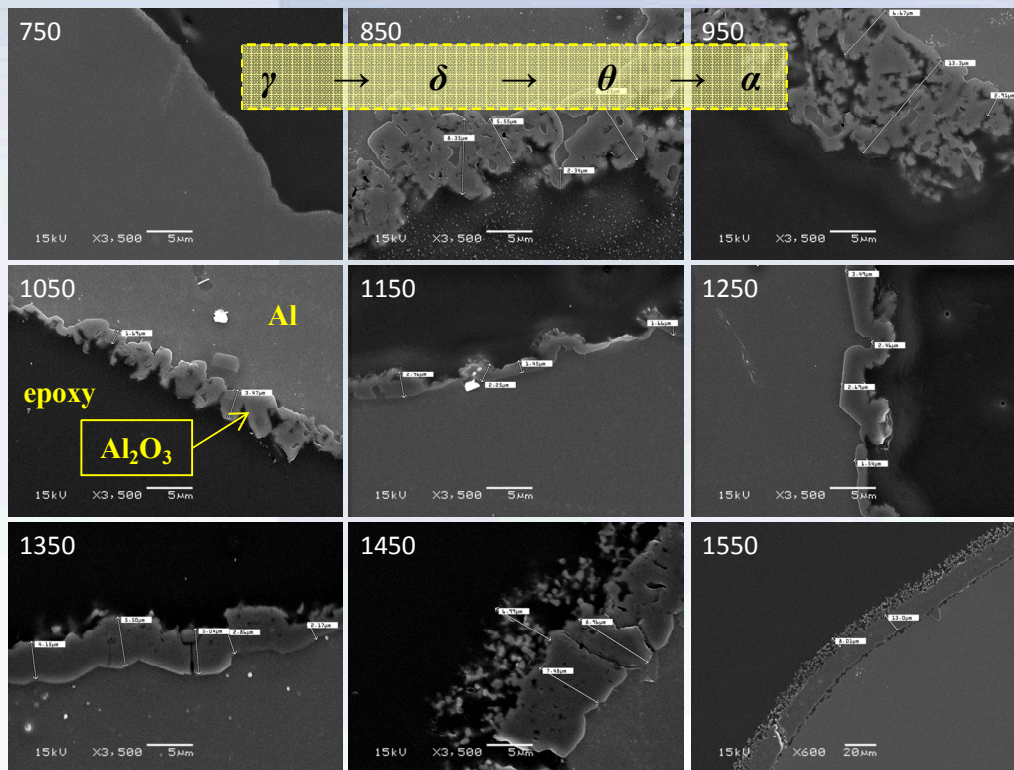


- Below 850 °C – little oxidation
- 850 to 950 °C – oxidation
- Above 950 °C – passivation
- Above 1250 °C – rapid increase in oxidation rate



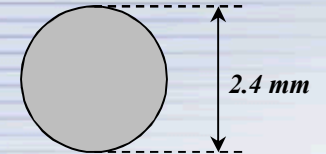
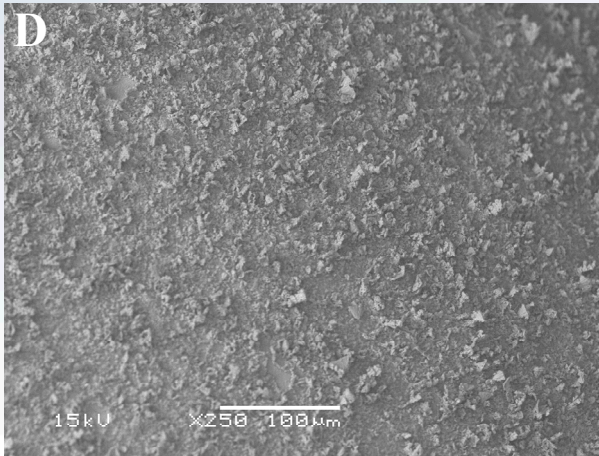
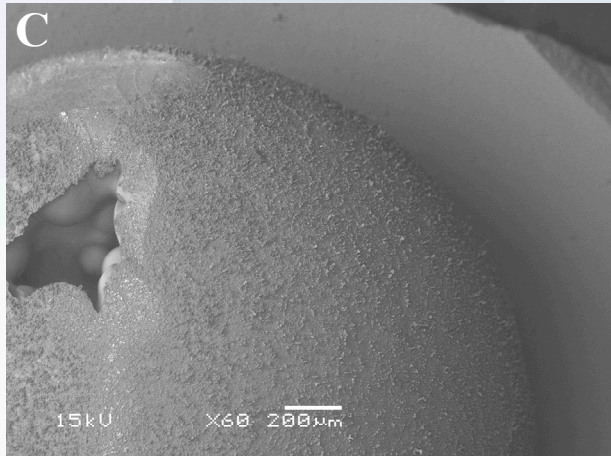
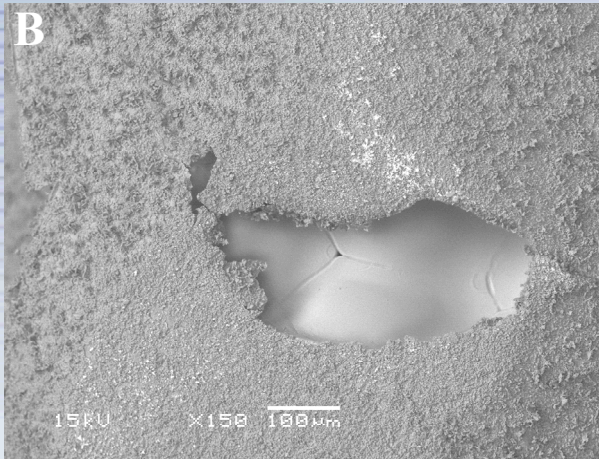
Oxide layer growth

Electron microscopy images of cross-sections of specimen
Oxidation temperature (°C) indicated

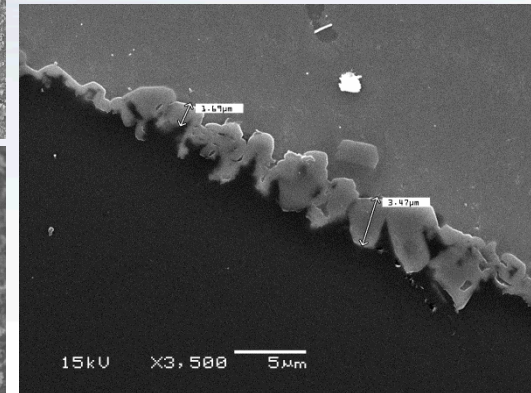


Below 1050 °C, foil topology persists.
At 1050 °C and above, spheroidal particle forms.

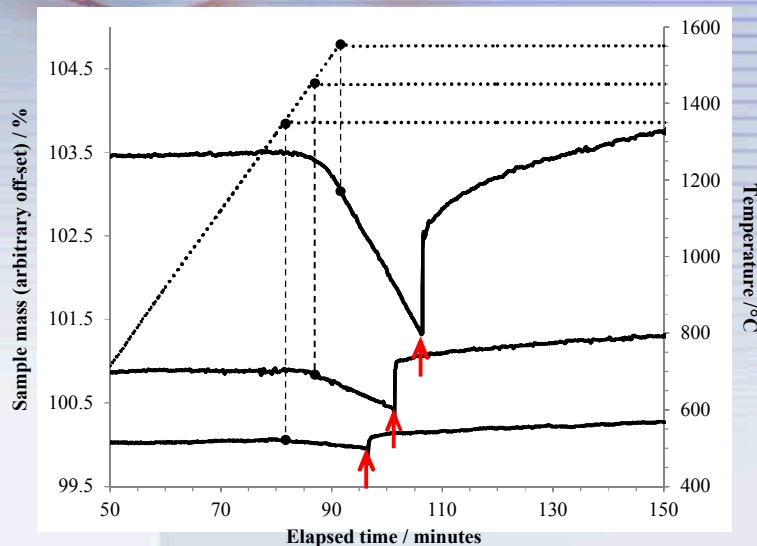
Alumina shell after oxidation at 1050 °C



Cross-section

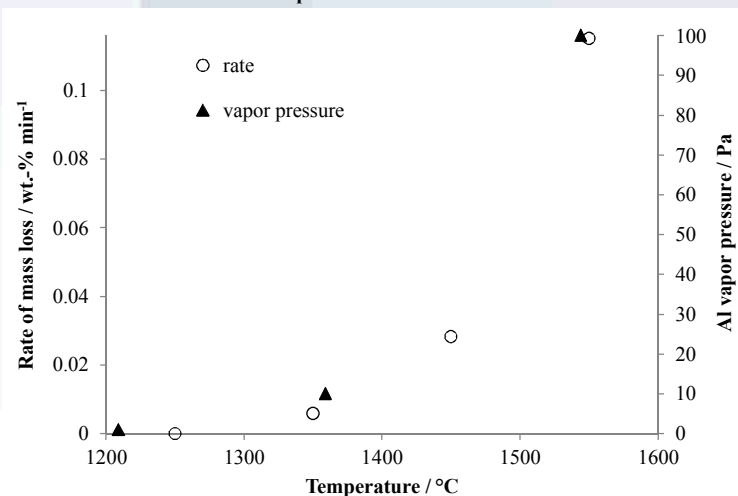


Al volatility at high temperature



Volatility noticeable at 1350 °C and above.

↑ = introduction of air



Measured rate of vaporization proportional to published Al vapor pressure.

Al vapor pressure data taken from:

http://www.knowledgedoor.com/2/elements_handbook/vapor_pressure.html.

Summary & conclusions

- **High-purity aluminum foil heated under inert atmosphere and exposed to air at desired temperature**
- **Non-linear dependence of oxidation on temperature**
 - Below 850 °C minimal oxidation occurred
 - At 850 °C oxidation occurred after “induction period”
 - At 950 °C oxidation occurred with no “induction period”
 - Above 950 °C passivation occurred (oxide barrier)
 - Above 1250 °C oxidation rate increased rapidly
- **Degree of oxidation small in all cases (low surface area specimen)**
- **Oxide layer strength overrides Al liquid surface tension below 1050 °C**
- **Observed oxide shell thickness (SEM) correlates with mass increase**
- **Al/Al₂O₃ spheroidal particle shows partial hollowing after 1050 °C oxidation**
- **Al volatility under inert gas measureable at 1350 °C and above**