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Review and Analysis of Experimental Activities on the Demisability of Pressure Vessels

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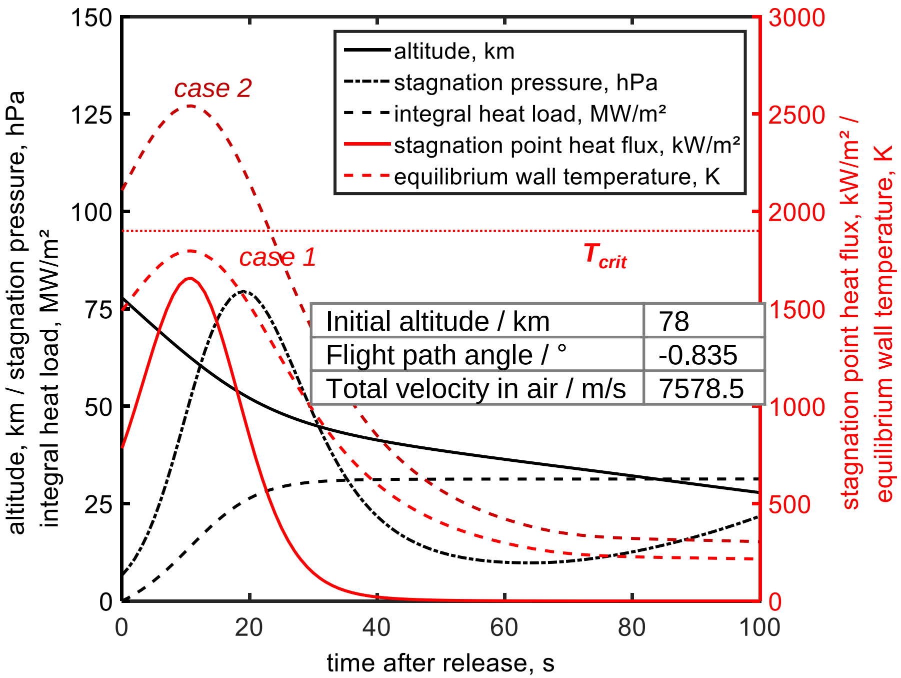
Motivation

- As is common knowledge by now, near-intact pressure vessels constitute the most frequently recovered type of post-re-entry space debris
  ➔ Tanks pose a significant on-ground casualty risk
- Two contemporary tank designs dominate: Monolithic Ti-6Al-4V (grade 5 titanium) tanks and Composite-Overwrapped Pressure Vessels (COPV)
- This presentation aims to provide an overview of relevant experimental activities on the material and component level
- We had initially over-estimated the general availability of published results so far
  ➔ IRS research is accordingly over-represented here

Source: NASA

Source: Space Safety Magazine

First things first: A "Quick-and-Dirty" Analysis of the Problem



Tank Volume / l	91
Mass / kg	6.4
Radius / mm	280
Material	Ti-6Al-4V
Solidus / K	1877
Liquidus / K	1933
Thermal conductivity (near melting pt.) / W/mK	27
Heat capacity (near melting point) / J/kgK	400
Enthalpy to melt $H_{demised}$ / kJ/kg	1500
Total surface emissivity (near melting point)	0.7 oxidat.

- Three simple and approximate demise criteria (similar to [Fritsche2007]) are coupled with post-break-up spherical titanium tank entry analysis with IRS code REENT
- **Conclusion**: Low ballistic coefficients coupled with large radii render a full demise ***often impossible*** especially considering the choice of materials:
  - ➔ Novel design approaches are essential
  - ➔ Alternative: Forced phys. disintegration?

Simulation result	Ti tank
Peak stagnation pressure / hPa	79.5
Peak stagnation point heat flux / kW/m²	1657
Critical temperature / K	1900
Radiative case 1: High conductivity, immediate th. equil.	
Peak equilibrium wall temperature / K	**1797**
Duration of "critical heat pulse" / s	**-**
Radiative case 2: Localised heating only *(→ holes)*	
Peak equilibrium wall temperature / K	**2542**
Duration of "critical heat pulse" / s	**23**
Calorimetric criterion *(no re-radiation → best case)*	
Integral stagnation point heat load / MJ/m²	31.3
$Q_{conv}/H_{demised}$	**0.80**

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Titanium Tanks: Experimental Activities

Experimental research activities relevant for Ti-6Al-4V pressure vessel demise have so far focused exclusively on material properties.

The following institutions have conducted directly relevant characterisation activities w.r.t. Ti-6Al-4V (list may be incomplete - publications are scarce):

- **PROMES-CNRS:** Emphasis on oxidation behaviour (e.g. oxide layer growth dynamics) in high-enthalpy oxidising environment (MESOX facility), pre- and post-oxidised emissivities
- **AAC** *(ESA-TRP "CoDM"):* Combined thermal and mechanical loads demise behaviour in Re-entry Chamber
- **IRS** *(ESA-TRP "CoDM"):* Transient material response / demise tests in high-enthalpy air flows (PWK1/4), pre- and post-test emissivities, preliminary characterisation of relative catalytic properties in PWK1 facility
- **DLR Cologne** *(ESA-TRP "CharDem"):* Transient material response / demise tests in L3K arc heater facility

➔ **Due to the limited scope of this presentation, the following slides shall focus on the demise-relevant effects of surface oxidation on titanium**
(primarily studied at PROMES-CNRS and IRS – as far as I can tell)

Titanium Tanks: Role of Surface Oxidation for Demise

For any metallic bodies subjected to atmospheric entries, surface oxidation may influence their behaviour as follows:

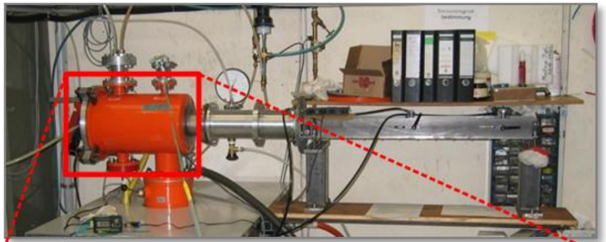
- **Emissivity:** An increased emissivity would increase heat dissipation
  → Less heat available to initiate and maintain destructive processes.

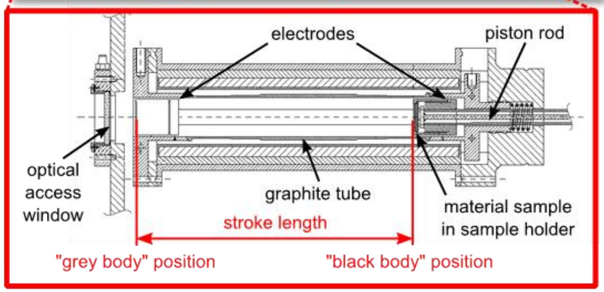
- **Catalycity:** Effective surface catalycities may change. Depending on this property, significantly varying degrees of additional heating may be released through recombination of dissociated air species.

- **Ablation behaviour:** Depending on the nature and (aero-)mechanical resilience of surfaces oxides, their formation could either contribute to demise by altering the primary mechanism for mass loss (from melt to “flaking”) ***OR*** inhibit it by providing a rigid thermomechanical barrier – more on that later!

Titanium Tanks: Surface Emissivity Measurements

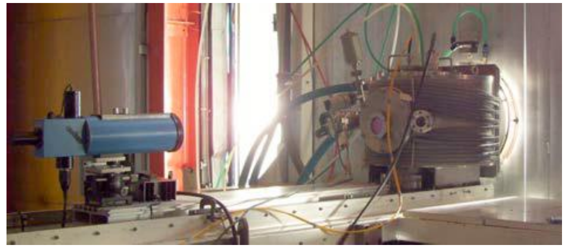
IRS Emissivity Measurement Facility (EMF)





- Measurement of temperatures and total emissivities using radiometer (0.4 to 8 µm)
- Rapid shift of sample between black body and grey body context, near-immediate comparison of measurements

PROMES-CNRS Moyen d'Essai et de DIagnostic en Ambiance Spatiale Extrême (MEDIASE)



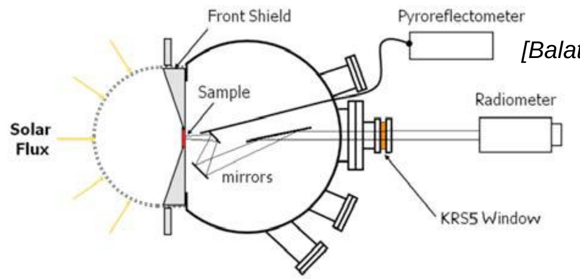
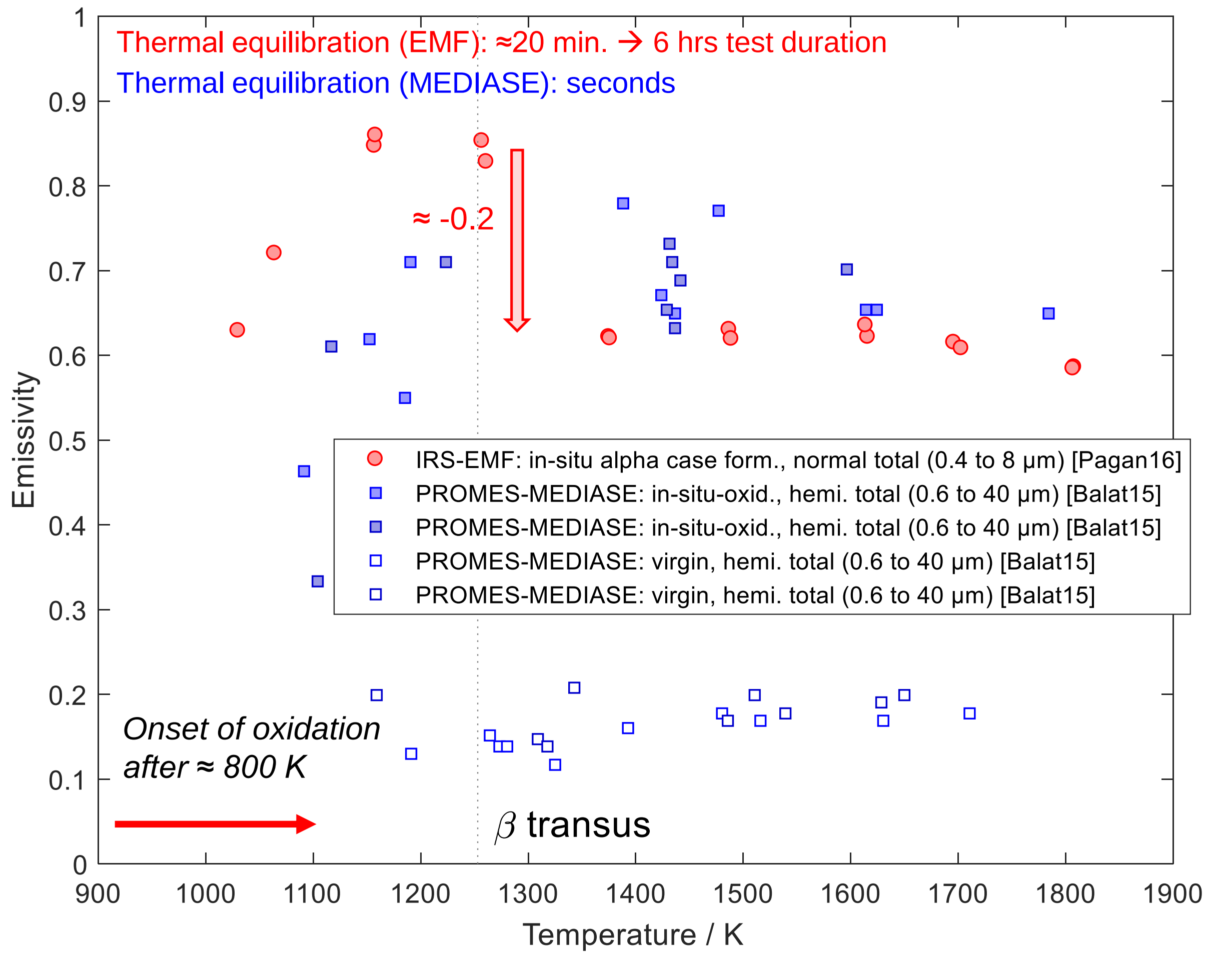


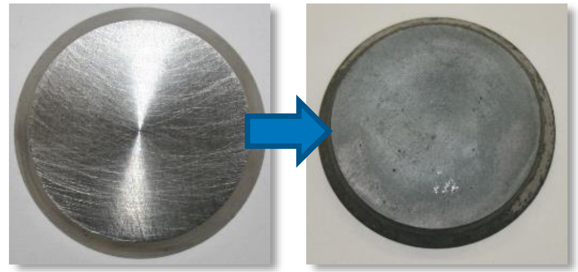
Image source: [Balat-Pichelin2015]

- Measurement of total emissivities using radiometer (0.6 to 40 µm)
- Separate black body reference calibration
- Separate measurement of temperatures using two-colour pyro-reflectometer

Titanium Tanks: In-situ Oxidation & Virgin Emissivities



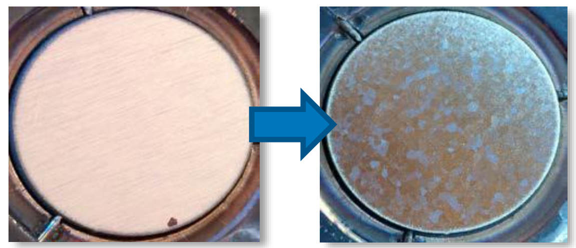
IRS sample [Pagan2016]**:**



(Alpha case formation through oxygen impurities in argon atmosphere)

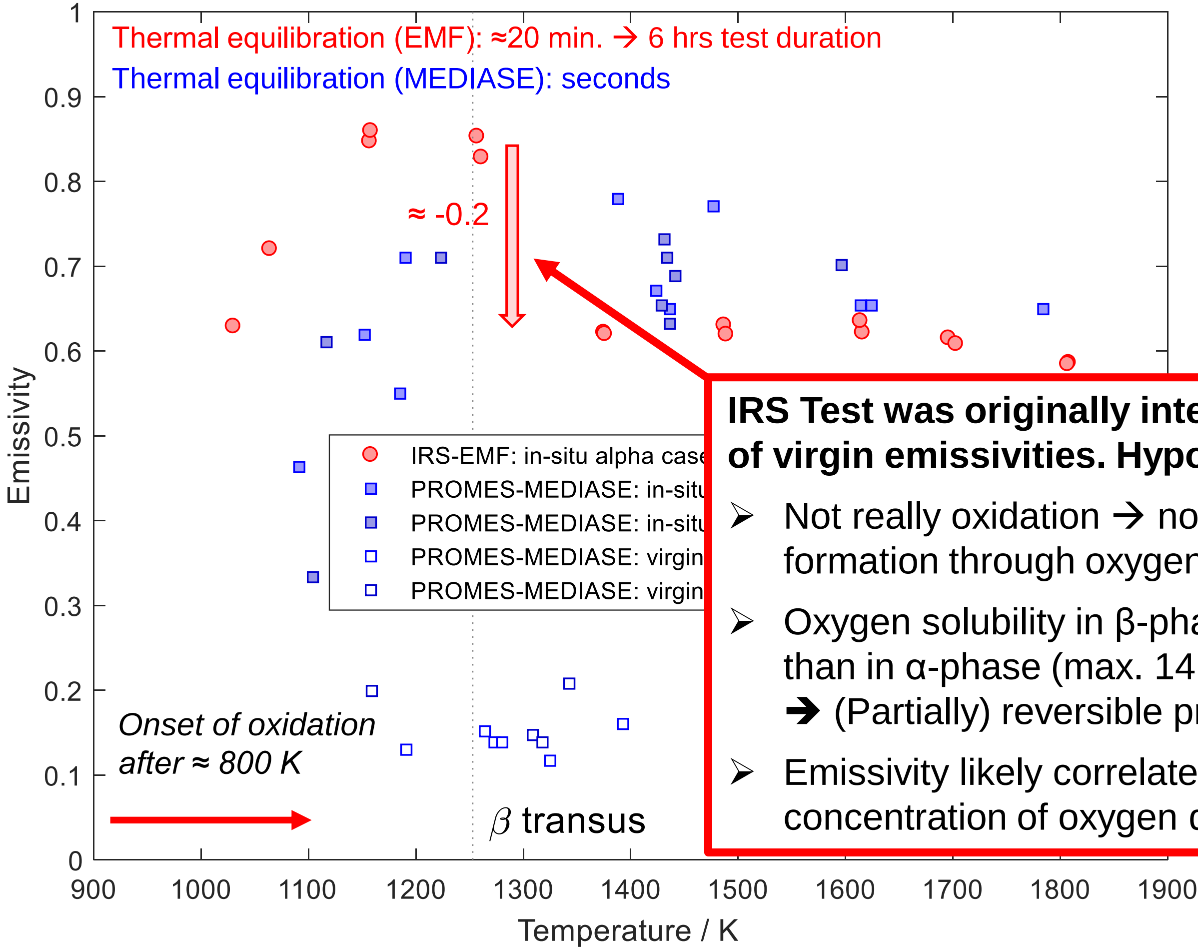
PROMES samples [Balat-Pichelin2015]**:**

Actual oxidation

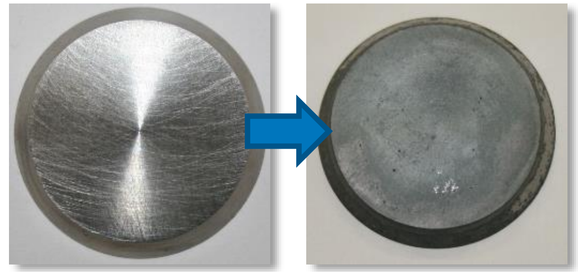


(virgin sample [Balat-Pichelin2015])

Titanium Tanks: In-situ Oxidation & Virgin Emissivities



IRS sample [Pagan2016]**:**



(Alpha case formation through oxygen impurities in argon atmosphere)

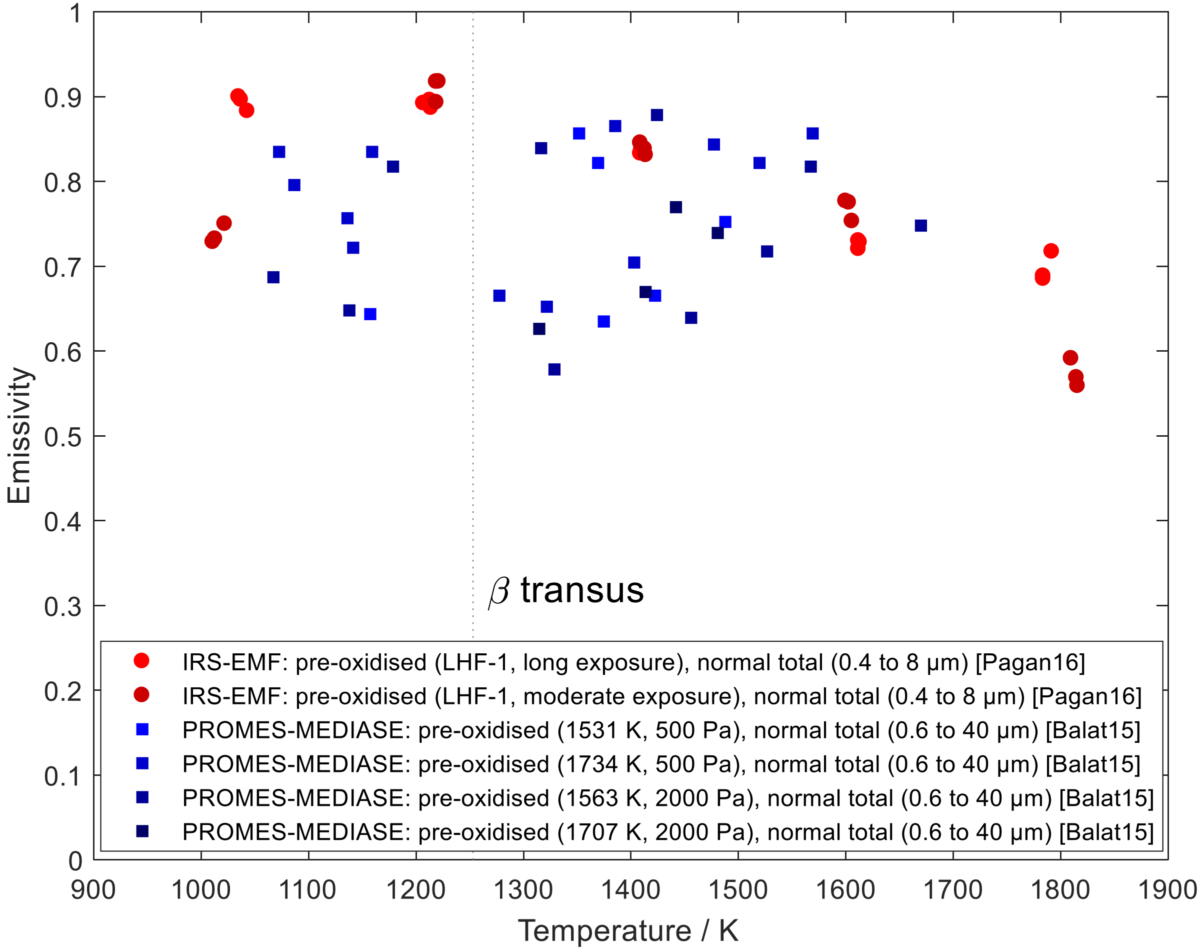
ichelin2015]:

IRS Test was originally intended for determination of virgin emissivities. Hypothesis:

- Not really oxidation → no TiO_2, but alpha case formation through oxygen enrichment by residues
- Oxygen solubility in β-phase (max. 8 wt.%) lower than in α-phase (max. 14.3 wt.%) [Sefer2014]
  ➔ (Partially) reversible process?
- Emissivity likely correlates directly to concentration of oxygen dissolved in bulk material

(virgin sample [Balat-Pichelin2015])

Titanium Tanks: Pre-Oxidised Emissivities



IRS samples [Pagan2016]:

(oxidised in IRS PWK1 facility at LHF-1 test condition at varying exposure times [Pagan2016, Pagan2015])

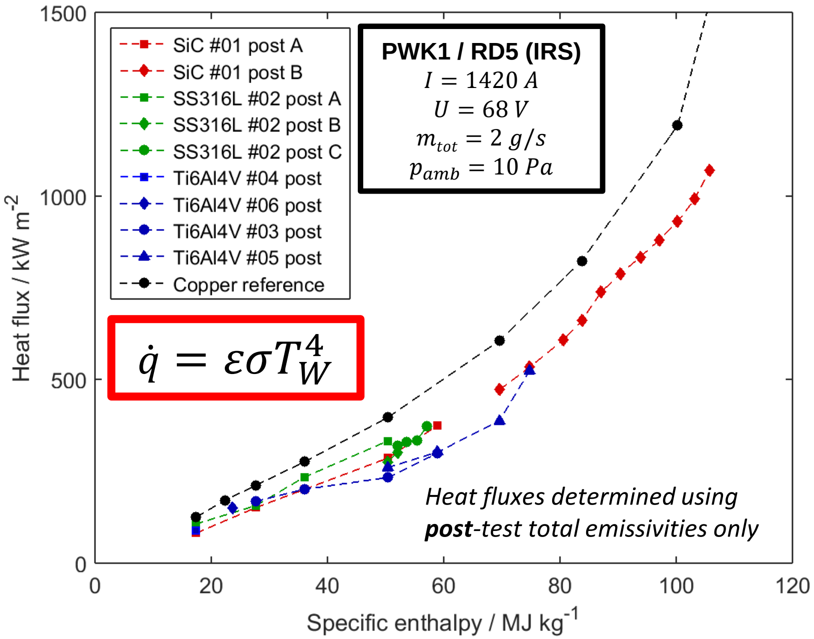
PROMES samples [Balat-Pichelin2015]:

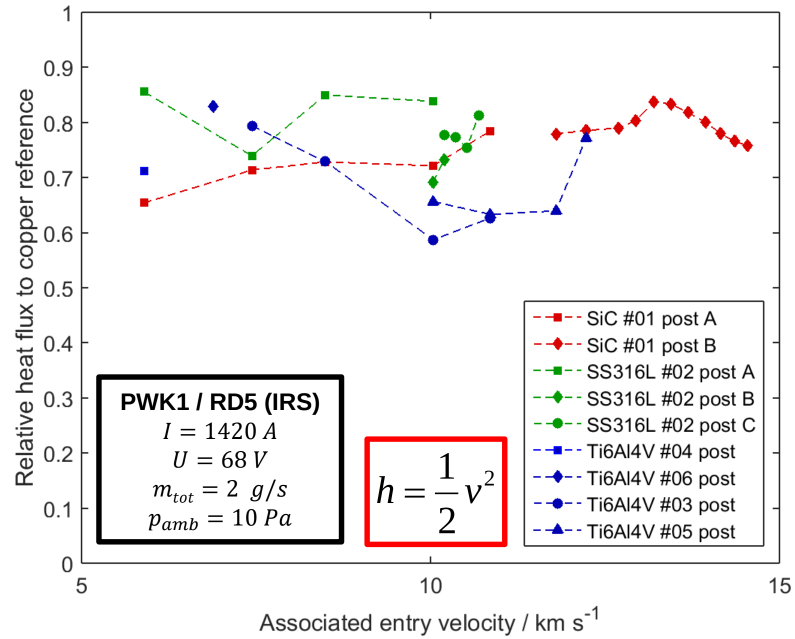
(oxidised in PROMES-CNRS MESOX facility [Balat-Pichelin2015])

Titanium Tanks: Conclusions for Emissivity

- Clear positive correlation between surface oxidation and emissivity
  ➔ increased heat dissipation delays demise significantly!
- Same effect observed for alpha case formation (likely subject to O_2 concentrations and timescales), directly affected by beta phase transition
- Visually very diverse range of degradation / oxidation noted
- Similar results for pre-oxidised surface emissivities between both campaigns, identical ranges between 0.55 and 0.90
- Comparatively higher scattering of PROMES-MEDIASE test data possibly reflects considerable diversity of optical properties of surface oxide features (more samples tested)
  ➔ Recommended total emissivities (PROMES): virgin: 0.2 / oxidised: 0.75
- Low scattering of IRS-EMF data allows grouping and fitting of data points
  ➔ Recommended total emissivities (IRS): T-dependent function between approx. 0.55 and 0.90 (oxidised/degraded only!)

Titanium Tanks: Relative Catalycity (IRS)





- Three materials subjected to varying heat flux conditions → Measurement of surface temperature
- Normalising the individually determined heat flux densities to the cold wall copper (CuO) reference measurements indicates the effect of material-specific catalycity (full catalycity is assumed at approximately $\dot{q}_{fc} = 1.25\ \dot{q}_{CuO}$).
- Very little work has been conducted on catalytic efficiencies of aerospace alloys and their oxides → much remains to be learned, but results so far indicate that oxidised Ti-6Al-4V generally tends to exhibit a comparatively low catalycity [Pagan2015]

Titanium Tanks: Nature of Surface Oxidation: PROMES

- Extensive study on Ti-6Al-4V behaviour in oxidising plasmas in the PROMES-MESOX facility [Prévereaud2016]
- Measurement of oxide thickness growth rates at varying temperatures (see below)
- No aeromechanical effects, but assumed to remove continuously replenished flaky oxide layer → oxidation accelerating demise
- Oxide behaviour changes above melting point

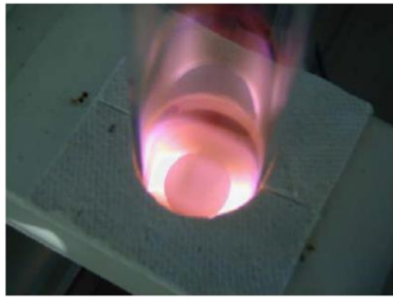
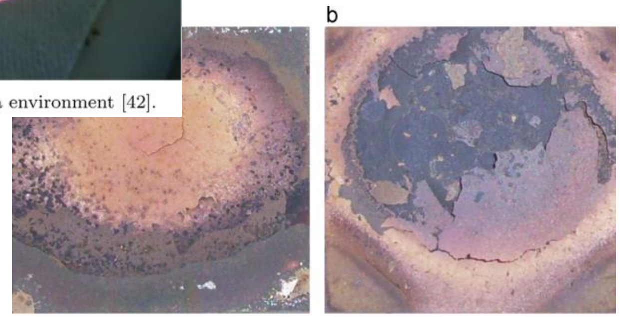


Image of air plasma environment [42].

Image source: [Prévereaud2016]



TA6V 11, 1600 K, 310 s. TA6V 6, 1800 K, 325 s.

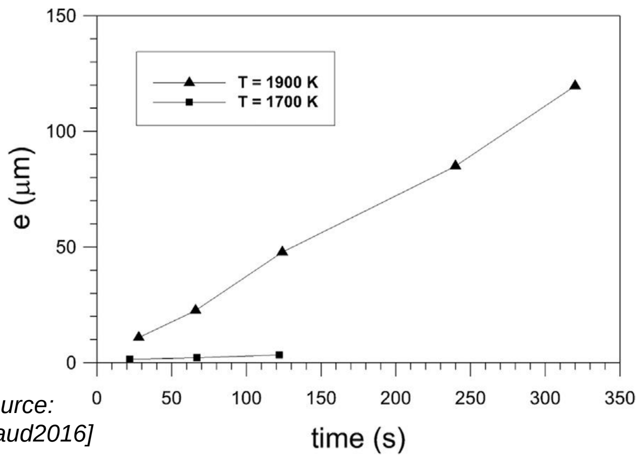


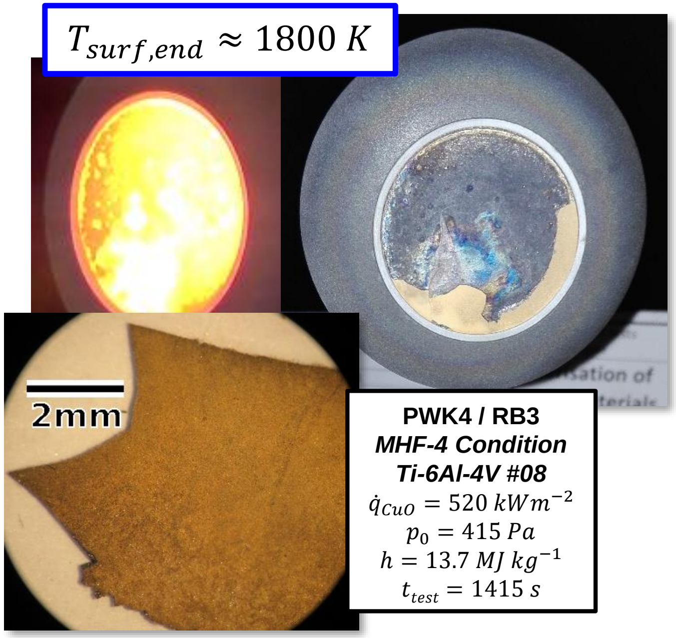
Image source: [Prévereaud2016]

Conclusions [Prévereaud2016]**:**

- Oxide layer surface properties should be assumed (emissivities and slightly increased melting temperatures)
- Oxidation contributes to demise at sub-melting-point temperatures due to aeromechanical effects

Titanium Tanks: Nature of Surface Oxidation: IRS

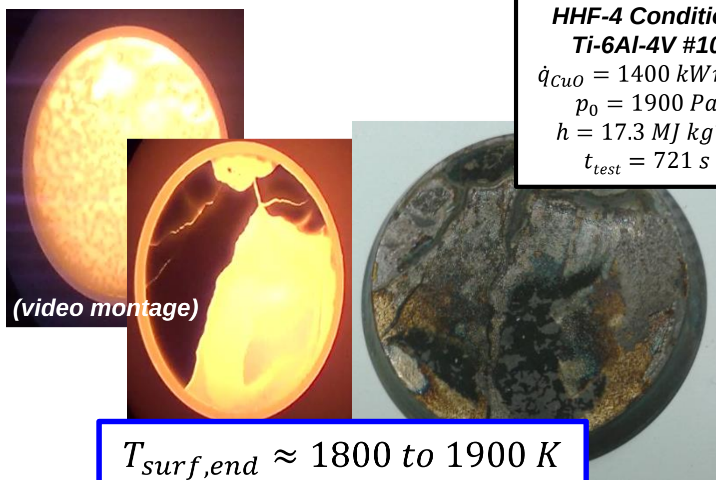
$T_{surf,end} \approx 1800\ K$



PWK4 / RB3
MHF-4 Condition
Ti-6Al-4V #08
$\dot{q}_{CuO} = 520\ kWm^{-2}$
$p_0 = 415\ Pa$
$h = 13.7\ MJ\ kg^{-1}$
$t_{test} = 1415\ s$

- ➢ No demise achieved despite according temperature ranges (long testing times)
- ➢ Liquid, but ***adhering*** surface film (→ solidifying to flaky oxides after shutdown!), likely V_2O_5 and rutile (?)
- ➢ Much more durable, degraded surface beneath (oxides / nitrides / alpha case?)
- ➢ Highly dynamic, reproducible temperature histories indicate intriguing effects of oxidation / degradation processes and beta phase transition

PWK4 / RB3
HHF-4 Condition
Ti-6Al-4V #10
$\dot{q}_{CuO} = 1400\ kWm^{-2}$
$p_0 = 1900\ Pa$
$h = 17.3\ MJ\ kg^{-1}$
$t_{test} = 721\ s$



$T_{surf,end} \approx 1800\ to\ 1900\ K$

Conclusions:

- ➢ Post-oxidation properties to be assumed
  ➔ Consensus with PROMES investigation
- ➢ Oxidation / degradation generally demise-prolonging despite (moderate) aeromechanical forces
  ➔ Differing interpretation than [Prévereaud2016]

COPV: Experimental Activities

The following institutions have or are conducting directly re-entry-relevant characterisation activities w.r.t. COPV demisability (list may be incomplete – publications are scarce):

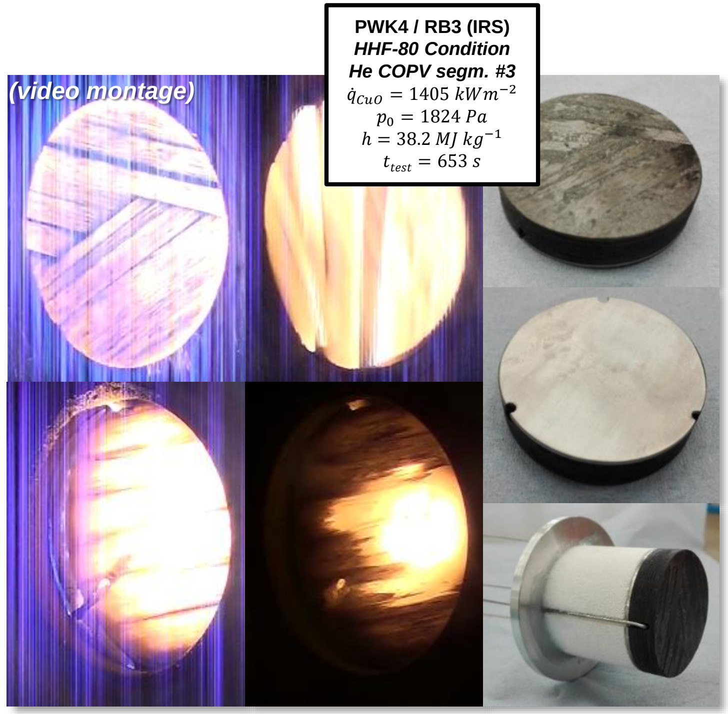
- **JAXA ARD Propulsion Group:** Ostensibly demisability-confirming laser- and arc heater ablation tests of JAXA-developed COPV variant with aluminium liner [Masuda2015].
- **AAC** *(ESA-TRP "CoDM"):* Combined thermal and mechanical loads demise behaviour in Re-entry Chamber.
- **IRS** *(ESA-TRP "CoDM"):* Transient material response / demise tests in high-enthalpy air flows of different variants of CFRP as well as COPV segments (PWK4 facility) [Pagan2017].
- **DLR Cologne** *(ESA-TRP "CharDem"):* Transient material response / demise tests in L3K arc heater facility of CFRP and COPV segments (referred to in [Lips2017].
- **VKI:** Experimental activities at component-level are planned/ongoing.

COPV: State of the Art

- Typical design: Thin Ti-6Al-4V-liner (in itself problematic → Al?) covered by thick CFRP overwrap
  ➔ Focusing on overwrap demise behaviour is prudent
- Initially considered more "demisable" than monolithic Ti tanks by many
- Processes (potentially) resulting in mass loss at LEO entry conditions:
  - **Charring / Pyrolysis:** Volume ablation, convective blockage through outgassing
  - **Combustion**: Primary surface ablation process below sublimation temperature
  - **Erosion / Spallation:** Aeromechanical removal of macroscopic particles
  - **Delamination:** Detachment and removal of entire filaments and layers through aeromechanical forces

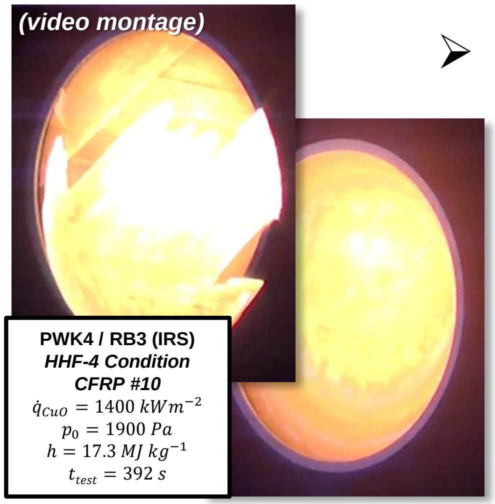
➔ **As experiments e.g. at IRS and DLR-K demonstrate, CFRP (and acc. COPV-overwraps) behaves much like an ablator, with laminate structure and behaviour as an additional complication!**

COPV: Question of Delamination



Question of overwrap's propensity to delamination in experiments and reality

- In arc heater experiments, laterally exposed samples may delaminate rapidly (or not!) depending on composition [Lips2017]
- Mechanical constriction (see below) and/or lateral shielding (see top right) of exposed edges can minimise delamination [Pagan2017]



- COPV post-entry recoveries (see bottom right) indicate delamination-resistance of overlaps

➔ No chance of demise unless structural integrity is compromised first!

COPV: Experimental Conclusions

- JAXA experimental investigation [Masuda2015] has indicated demisability of Al-COPV variant, however the tests were conducted on cut-out segments
- Demisability under actual entry conditions doubted in recently published analysis and review, referring to SCARAB simulations of equivalent re-entering COPV and behavioural evidence from DLR-L3K tests (CFRP and COPV segments) (“CharDem”) [Lips2017]
- Same conclusion reached by “CoDM”-consortium on account of observed behaviour in PWK4 tests of CFRP and COPV segments [Pagan2016]
- Different variants of CFRP show different propensities towards delamination (e.g. a JAXA-developed variant tested at IRS and UF3325 TCR variant tested at DLR-K), which could be exploited for D4D if overwrap integrity were compromised


CFRP EX1515/M55J (HHF-4 cond. [Pagan2017])


CFRP T60% IM7/PEEK-150 (MHF-4 cond. [Pagan2017])


“JAXA-CFRP” (MHF-4 cond. [Pagan2017b])

Three distinct CFRP variants tested at IRS with varying demise behaviours and delamination propensities [Pagan2017, Pagan2017b]

Summary

General issues with tank demisability:

- Low-ballistic coefficients restrict heating rates – could even prevent demise of large monolithic Al tanks (see [Lips2017])!
- Both monolithic Ti tanks and COPV are made of very resilient materials
  ➔ They will simply not demise unless broken up physically
- *Possible solutions:* Forced pre-fragmentation, overwrap dissection

Ti-6Al-4V tanks:

- Precise nature, dynamics and effects of surface oxidation (+ nitridation?) remain intriguing and somewhat disputed, but experimenters appear to consent on importance (ablation behaviour, optical properties, aerothermochemistry)
  ➔ Greater focus on metal surfaces needed in experimentation and modelling
- *Alternatives:* Use of “rapid melters” (e.g. Al)

COPV tanks:

- Recent activities have confirmed charring-TPS-like behaviour of CFRP overwraps
- Delamination could play a significant role in demise, but suppressed by overwrap context
  ➔ Demise investigations with representative overwrap structure would help
- *Alternatives:* Return to more degradable compounds (glass fibres? → Favourable demisability test results for FMLs such as GLARE), overwrap dissection

THANK YOU!

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