Materials Properties & Associated Test Methods for Non-metallic Materials

ESA SME Initiative
Course Materials and Processes

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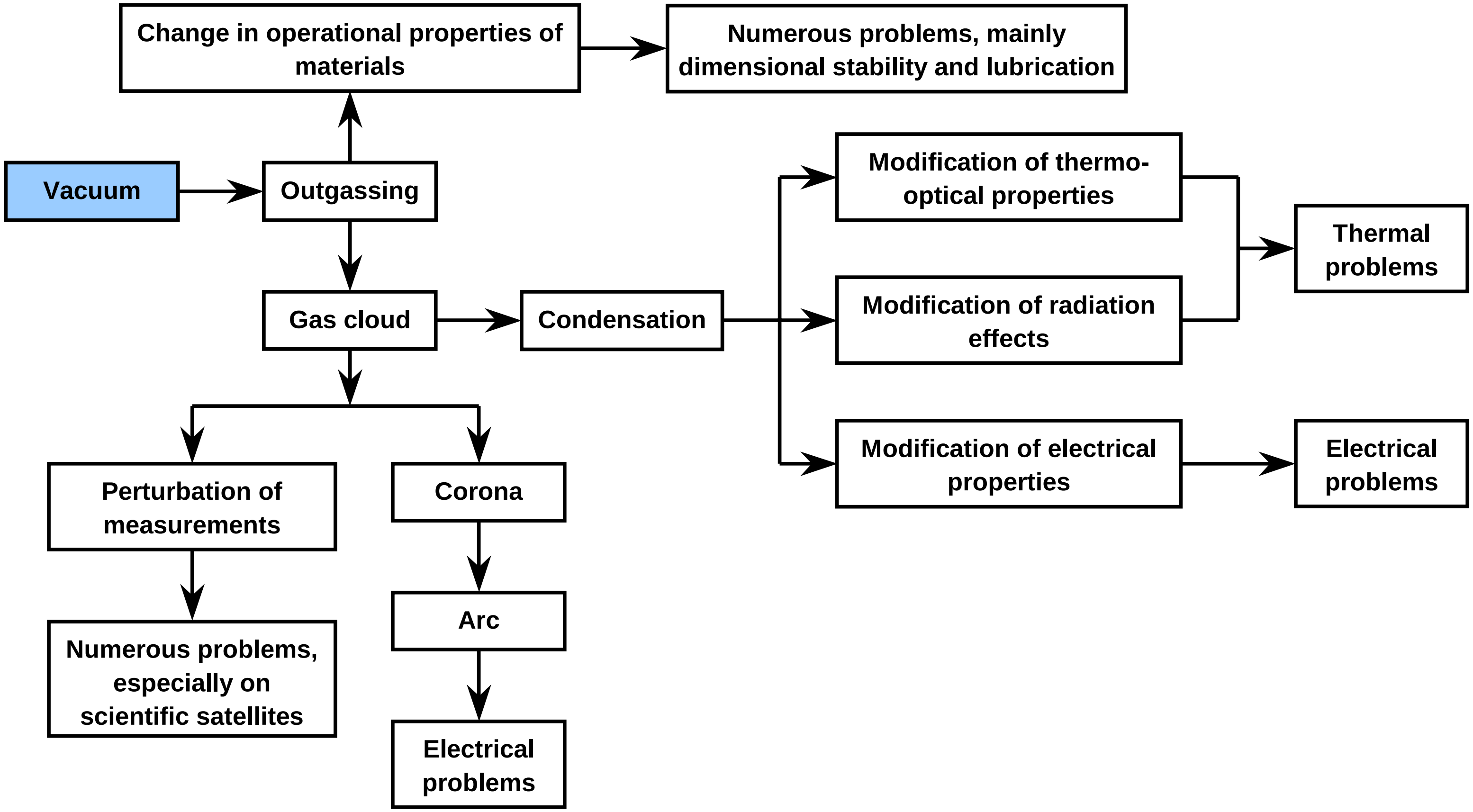
Overview

- Outgassing
- Infrared techniques
- Thermo-optical properties
- Thermal analysis
- Radiation
- Thermal cycling
- Atomic oxygen
- Failure analysis

Outgassing – Effects in Vacuum



Vacuum

Outgassing

Change in operational properties of materials

Numerous problems, mainly dimensional stability and lubrication

Gas cloud

Condensation

Modification of thermo-optical properties

Modification of radiation effects

Thermal problems

Modification of electrical properties

Electrical problems

Perturbation of measurements

Numerous problems, especially on scientific satellites

Corona

Arc

Electrical problems

Outgassing (ECSS-Q-70-02)

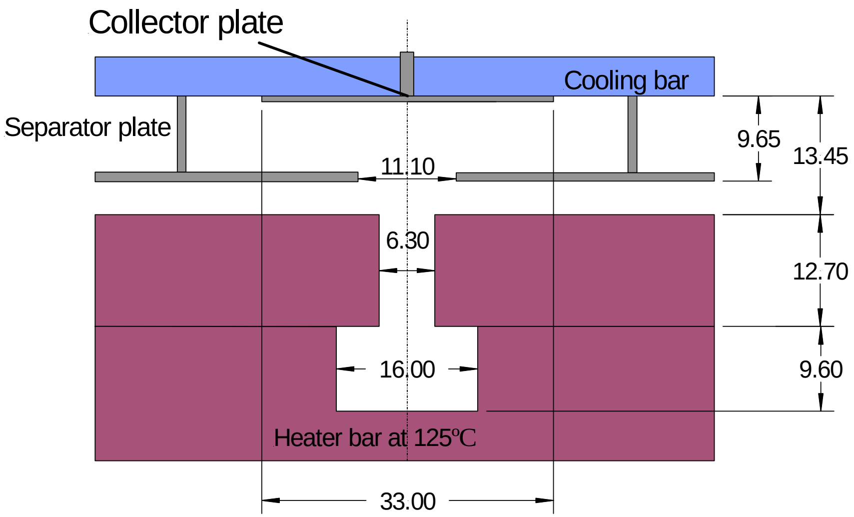
Thermal vacuum outgassing test for the screening of space materials

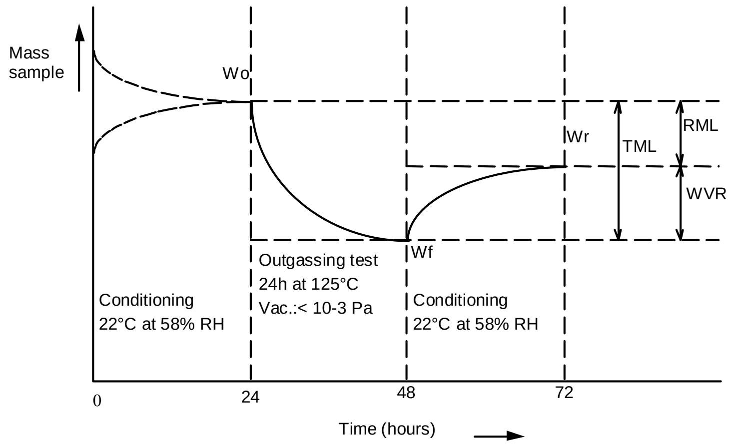
Purpose: Thermal vacuum test to determine the outgassing properties of materials proposed for use in the fabrication of spacecraft and associated equipment, for vacuum facilities used for flight hardware tests and for certain launcher hardware.

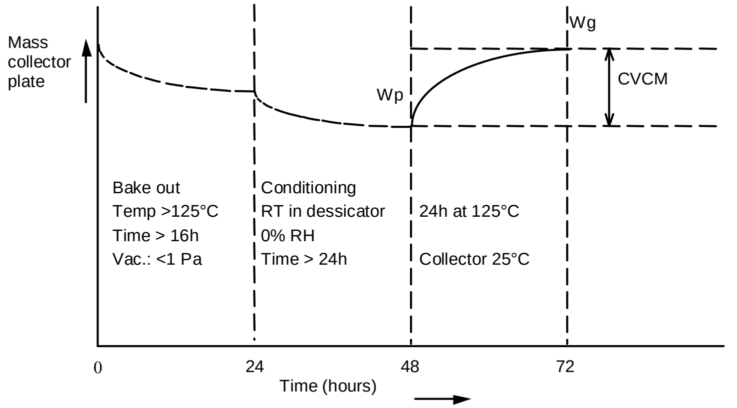
Determination of:

- Total mass loss (TML)
- Returned mass loss (RML)
- Collected volatile condensable material (CVCM)

Micro-VCM System I







TML	Total mass loss (< 1.0 % if water is a problem)
RML	Recovered mass loss (< 1.0 %)
CVCM	Collected volatile condensable material (< 0.1 %)

Micro-VCM System II

Dynamic Outgassing Test (VBQC) I

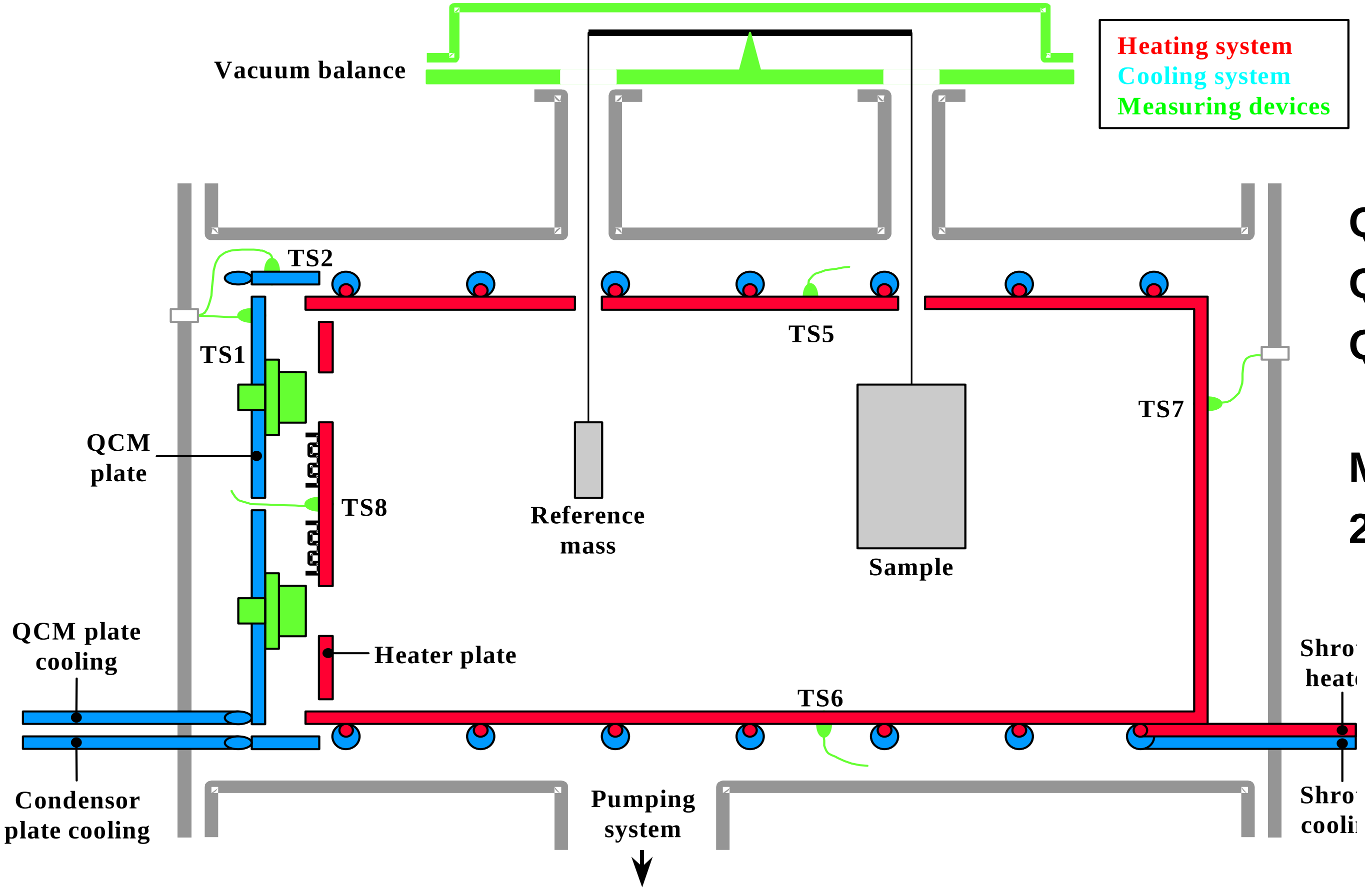
Quantification of outgassing and condensation of materials as function of temperature and time, to support mathematical models used for the prediction of molecular contaminant generation, migration, and deposition.

VBQC: Vacuum Balance Quartz Crystal

Standard temperature program: 5 steps of 25°C/24h

Results: Acceleration factors
Residence time
Activation energy
Long term prediction

Dynamic Outgassing Test (VBQC) II



QCM1 = - 25°C

QCM2 = - 50°C

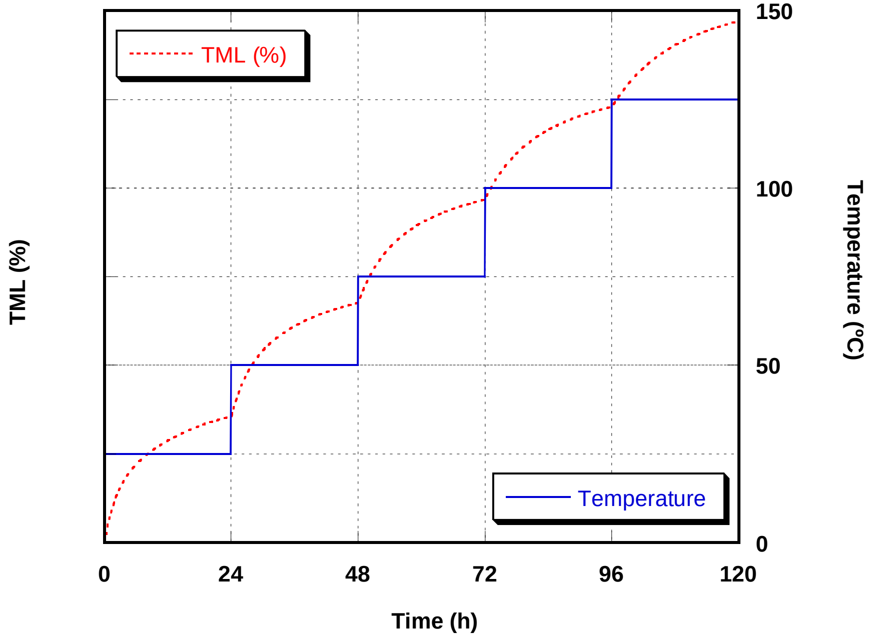
QCM3 = - 75°C

Material

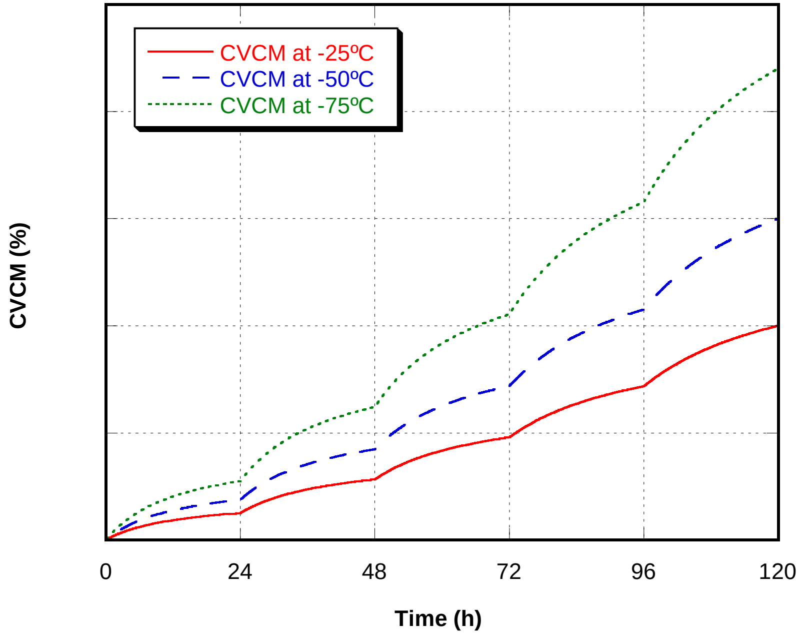
25 – 125°C

Dynamic Outgassing Test (VBQC) III

Outgassing and sample temperature profile



CVCM profile on quartz crystal microbalances



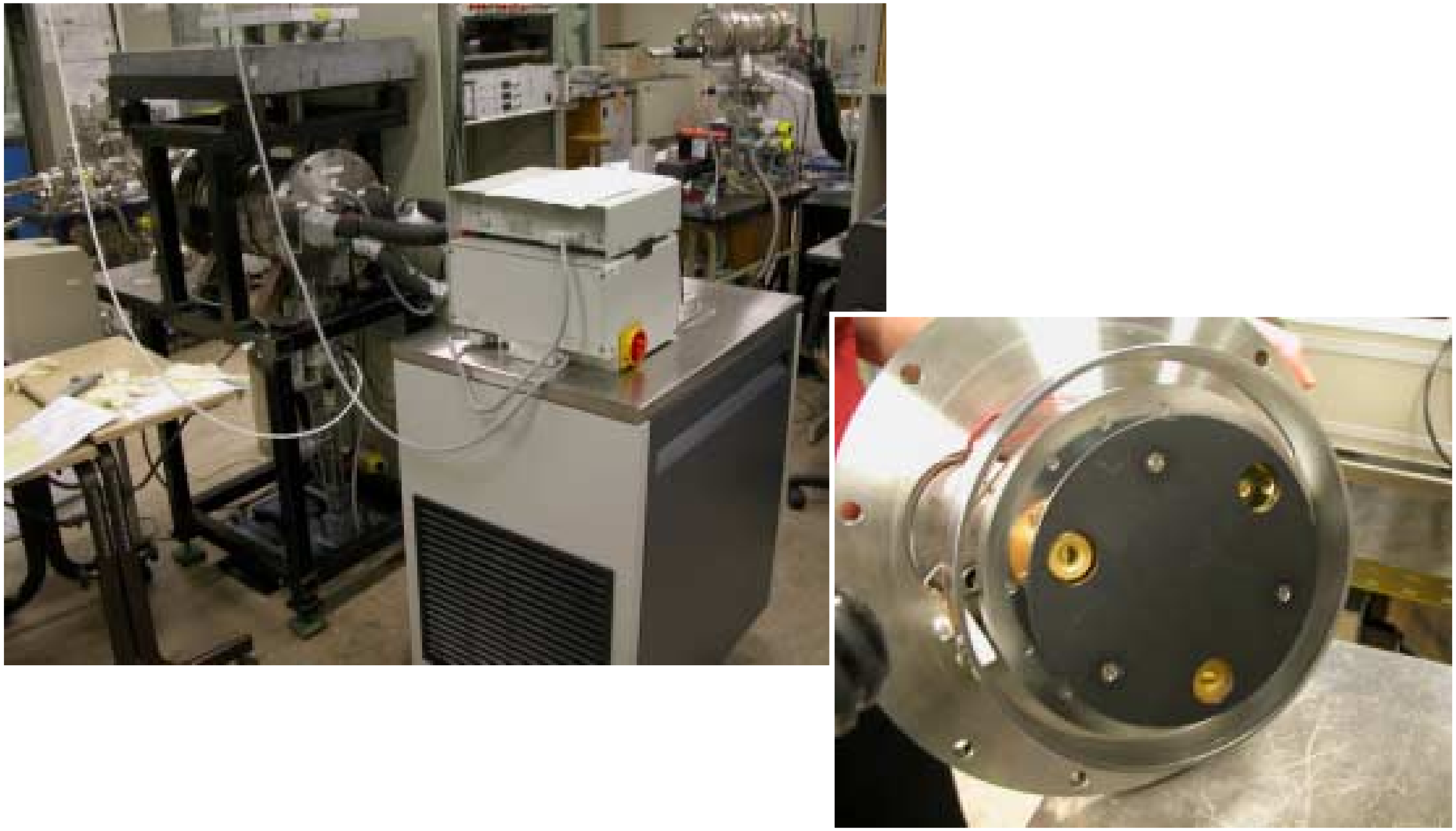
Dynamic Outgassing Test (VBQC) IV

- Results taken out of the Mathematical treatment consist of acceleration factor, activation energy, residence time - temperature dependency.
- Long term predictions with VBQC data is used within the temperature range of test.
- The TML or CVCM data are fitted period by period with a sum of 6 exponential equations.

$$W = \sum_{i=1}^{6} W_{0,i} \times \left(1 - e^{-t/\tau_{0,i}} \right)$$

- With $W_{0.i}$ the variables, affected with the time constant $\tau_{0,i}$, which are 50, 20, 5, 1, 0.5 and 0.1 hour.

Dynamic Outgassing Test (VBQC) IV



Infrared Methods for Identification of Outgassed Products

Infrared spectroscopy can be applied for a variety of investigations

- Identification of materials: Qualitative by identification of characteristic group frequencies and patterns, semi-quantitative via calibration curves using reference standards

  - Outgassing

  - Cleanliness and contamination control

  - …
- (Failure) investigation on materials: Monitoring of chemical and/or physical alterations by shifts/(dis)appearance of group frequencies
- Reflection properties of materials: Evaluation of specular and diffuse reflectance in the IR in combination with UV/VIS
- Thermo-optical Properties of Materials: Determination of emissivity from hemispherical reflectance measurements in combination with UV/VIS

Methods (ECSS-Q-70-05)

Direct Methods

- IR transparent window (e.g. CaF2, ZnSe, Ge,…) is placed in situ near critical locations (e.g. sample holder, cold shroud) inside vacuum chamber and analysed prior and after the experiment. Immediate measurement important to avoid false results due to creep of certain substances.
- Direct reflection measurement of the contaminated surface.

Indirect Methods

The molecular contamination on a surface to be analysed is removed by a known quantity of a suitable solvent (*e.g.* $CHCl_3$). The solvent is collected in a Petri dish, after evaporation of most solvent transferred onto an IR transparent window, and subsequently analysed. Removal of contaminants can be performed by:

- Washing, rinsing
- Wiping
- Transfer in small solvent volume

Infrared Methods – Quantification I

Experience has indicated that the contaminants can be divided into four main groups with typical adsorption bands in the mid IR:

- Hydrocarbons (3000-2850, 1460, 1380 cm^{-1})
- Esters (1740, 1300-1050 cm^{-1})
- Methyl silicones (1260, 1130-1000, 860-740 cm^{-1})
- Phenyl silicones (1260, 1130-1000, 1125-1100, 860-740 cm^{-1})

Typical for human grease is the ester/acid doublet around 1735/1710 cm^{-1}.

Quantitative interpretation of spectra often complicated:

- Exact type of contaminant may be unknown
- Insufficient or unavailable material for calibration curve

Need to quantify relative to a known standard → semi quantitative

Infrared Methods – Quantification II

Typical calibration standards

- Paraffin oil
- Dioctyl phthalate
- Poly(dimethylsiloxane)
- Poly(methylphenylsiloxane)

Quantification based on Lambert-Beer's law

$$\boxed{Absorbance = \varepsilon_\lambda \cdot l \cdot c = \log\left(\frac{I}{I_0}\right) = f(mass)}$$

ε_λ Molar absorption coefficient at wavelength λ, $Lmol^{-1}cm^{-1}$

L Pathlength, cm

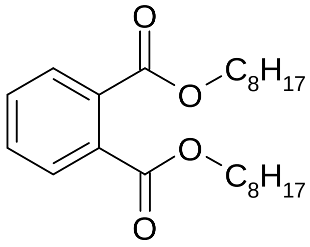
C Molar concentration, $molL^{-1}$

I/I_0 Ratio between transmitted and incident light

Calibration Standards 1

Esters:

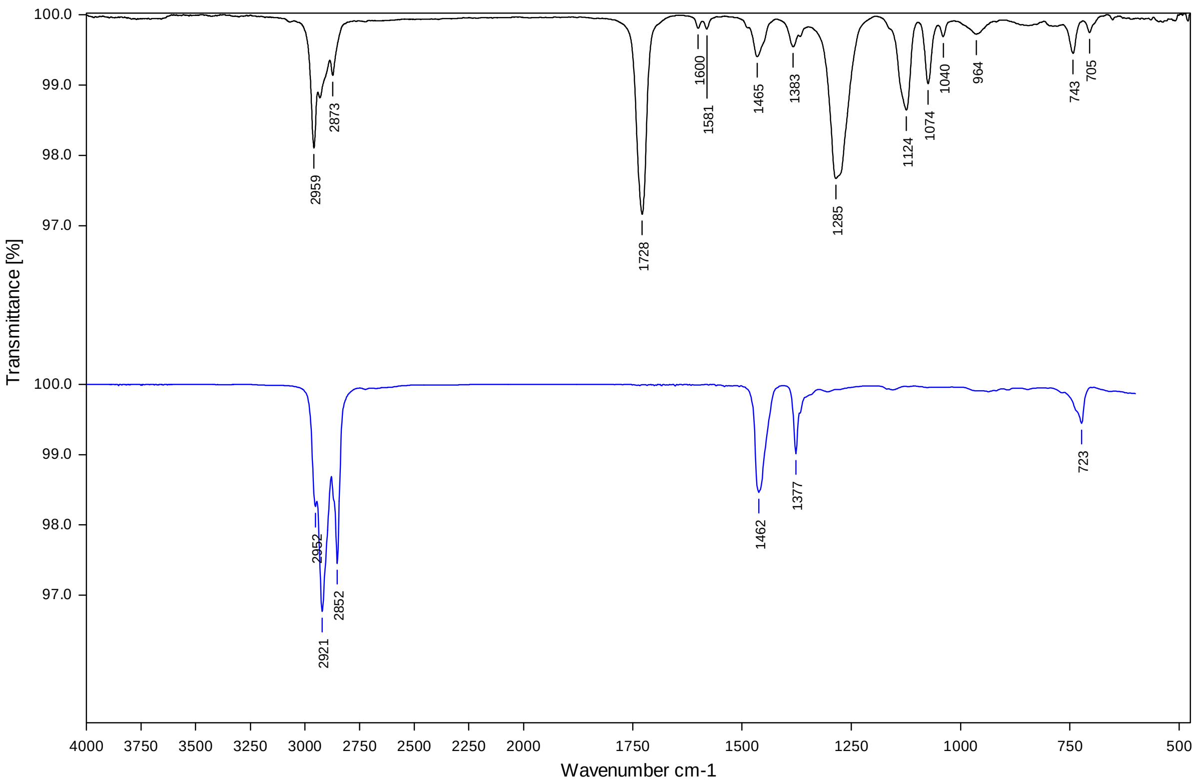
Dioctyl phthalate



Hydrocarbons:

Paraffin oil

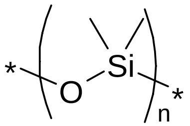
C_nH_{2n+2}



Calibration Standards 2

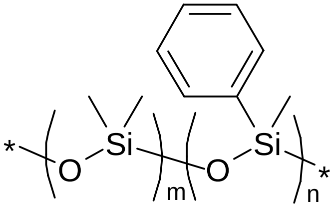
Methyl silicones:

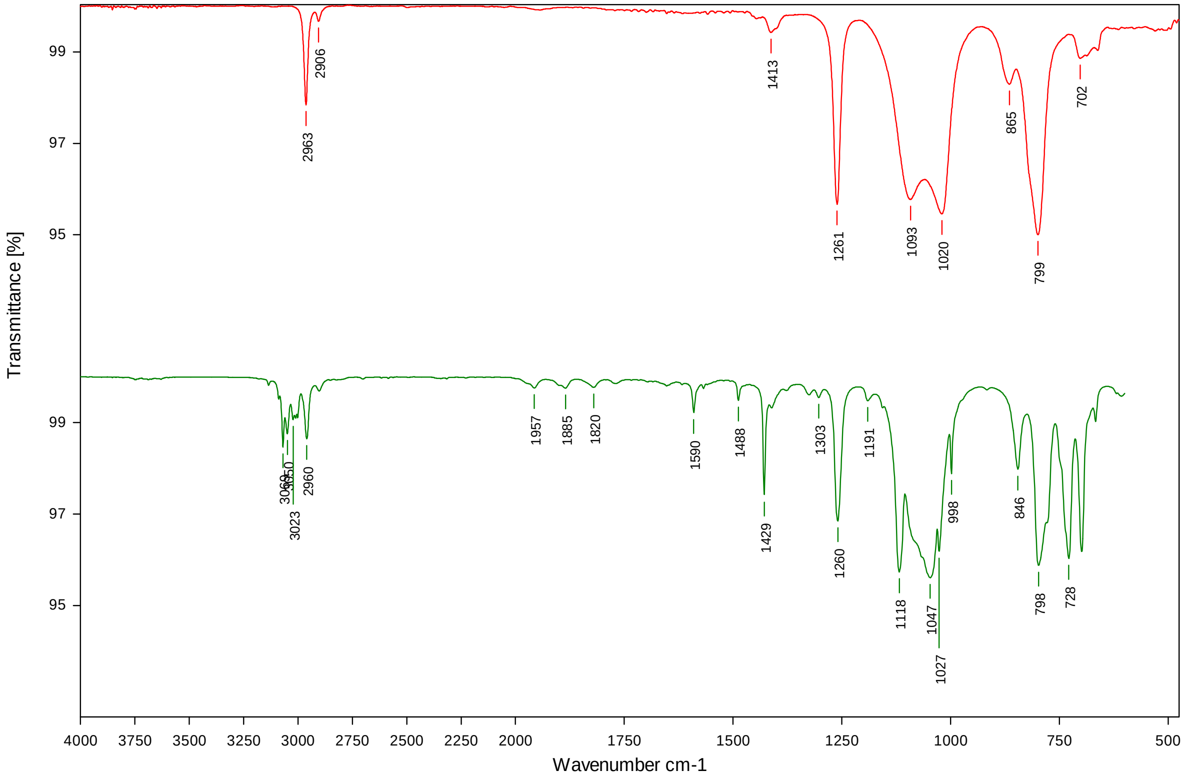
Poly(dimethyl-siloxane)



Phenyl silicones:

Poly(methyl-phenylsiloxane

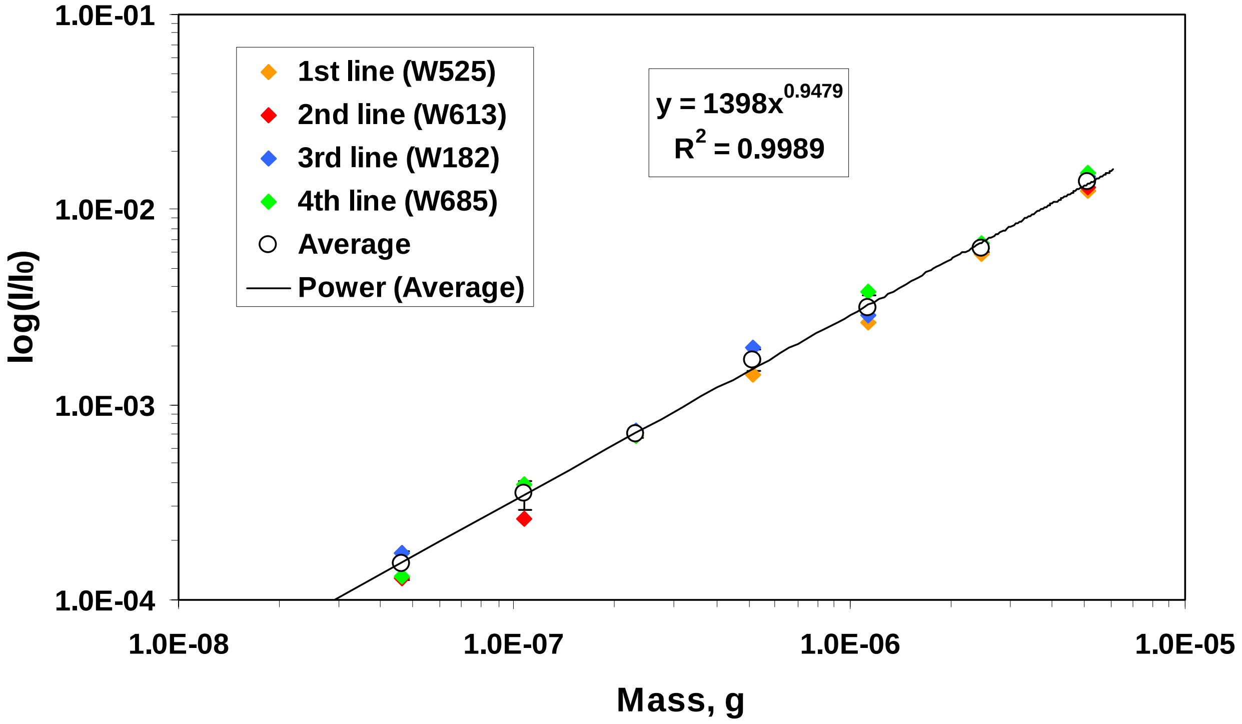




Typical Calibration Curve

Typical calibration curve: DOP for carbonyl band at 1735 cm^{-1}

Best curve fit: Power line



Thermo-Optical Properties (ECSS-Q-70-09)

Solar absorptance (α): Ratio of the solar radiant flux absorbed by a material (or body) to that incident upon it

$$\alpha_S = 1 - R_S \qquad R_S = \frac{\int_{\lambda_1}^{\lambda_2} R(\lambda) S(\lambda)\, d\lambda}{\int_{\lambda_1}^{\lambda_2} S(\lambda)\, d\lambda}$$

$R(\lambda)$ = Spectral reflectance

$S(\lambda)$ = Solar irradiance (λ_{max} = 0.45 µm)

λ_1 ~ 0.25 µm, λ_2 ~ 4 µm

Thermal emittance (ε): Ratio of the radiant intensity of the specimen to that emitted by a black body radiator at the same temperature and under the same geometric and wavelength conditions

$$\varepsilon_n(T) = 1 - R_n(T) \qquad R_n(T) = \frac{\int_{\lambda_1}^{\lambda_2} R(\lambda)_T\, BB(\lambda)_T\, d\lambda}{\int_{\lambda_1}^{\lambda_2} BB(\lambda)_T\, d\lambda}$$

$R(\lambda)_T$ = Spectral reflectance at temperature T

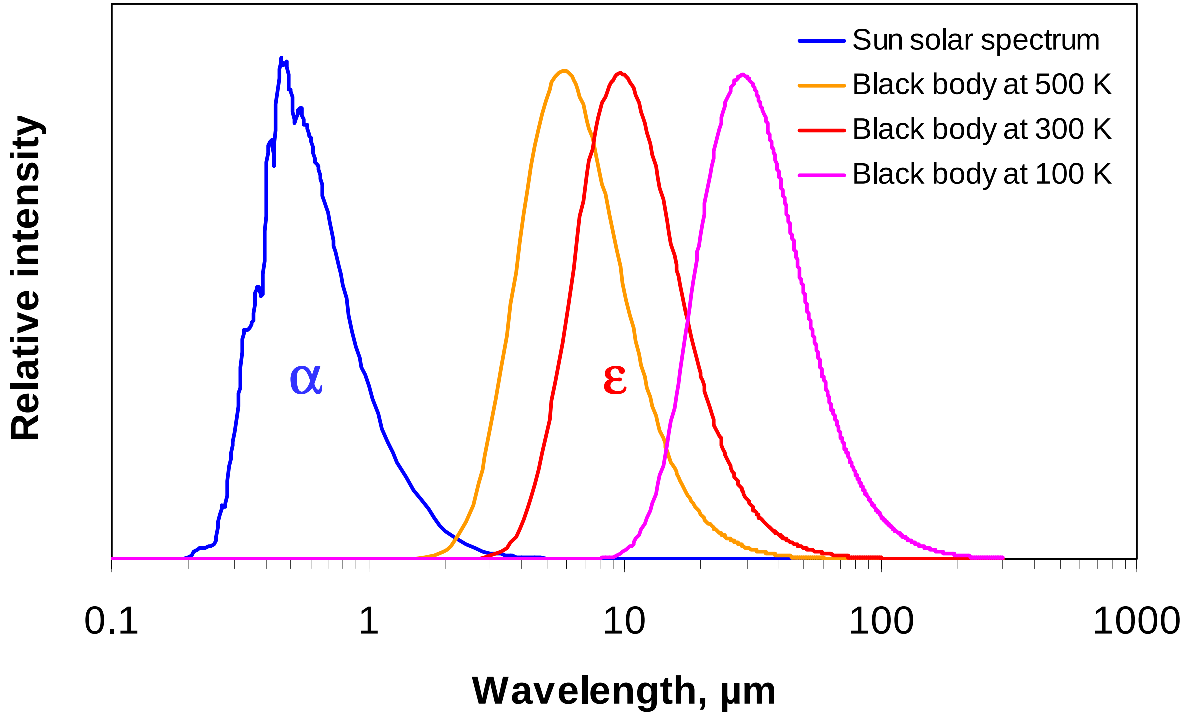
$BB(\lambda)_T$ = Black body at temperature T

25°C: λ_{max} = 9.7 µm, λ_1 ~ 2.5 µm, λ_2 ~ 40 µm

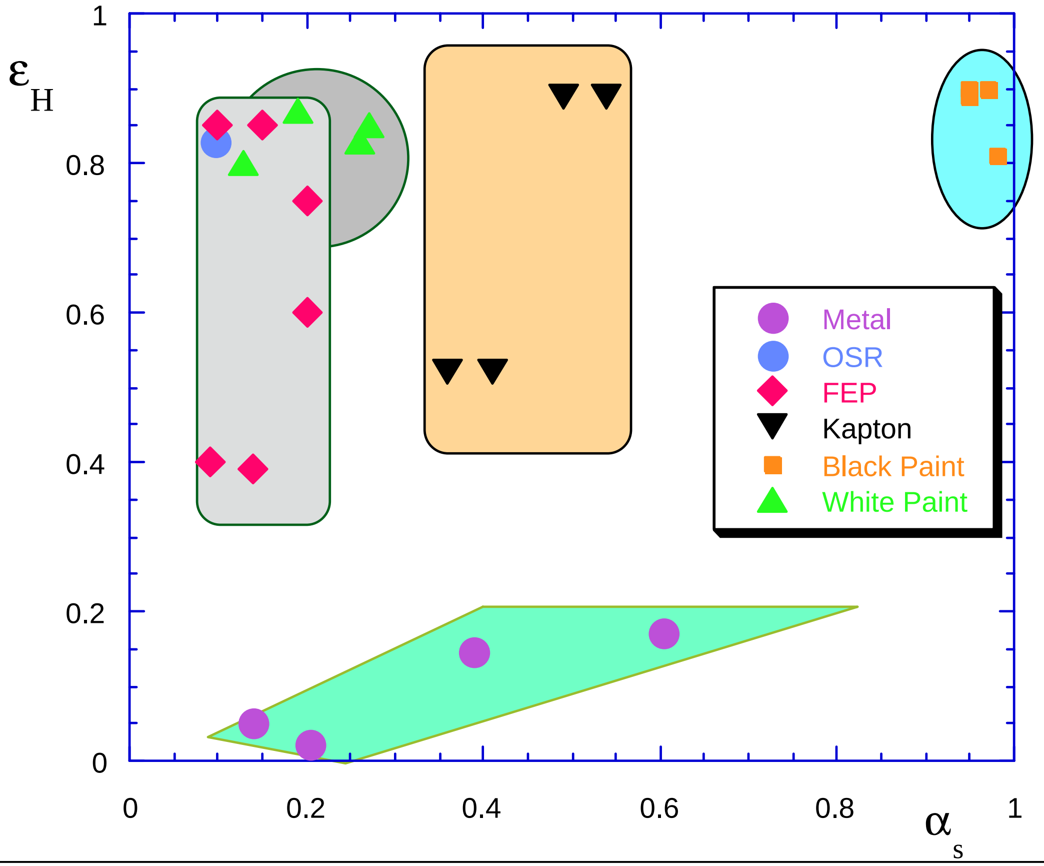
Thermo-Optical Properties (ECSS-Q-70-09)

Solar absorptance α → UV/VIS wavelengths

Thermal emitance ε → IR wavelengths



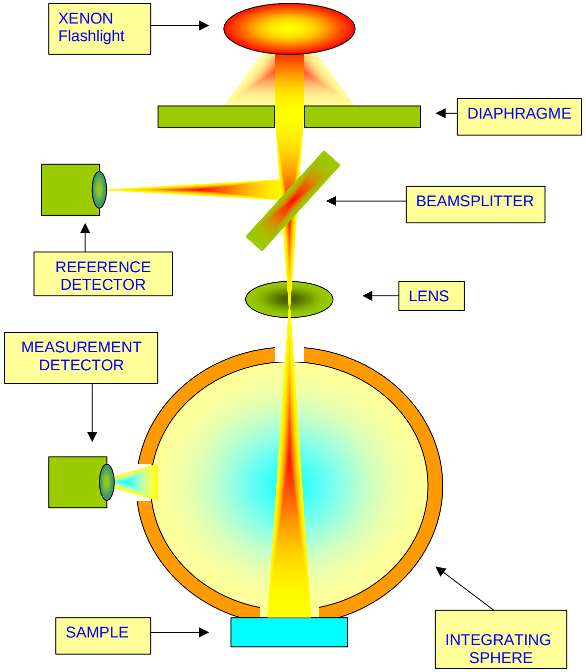
Thermo-Optical Properties of Coatings



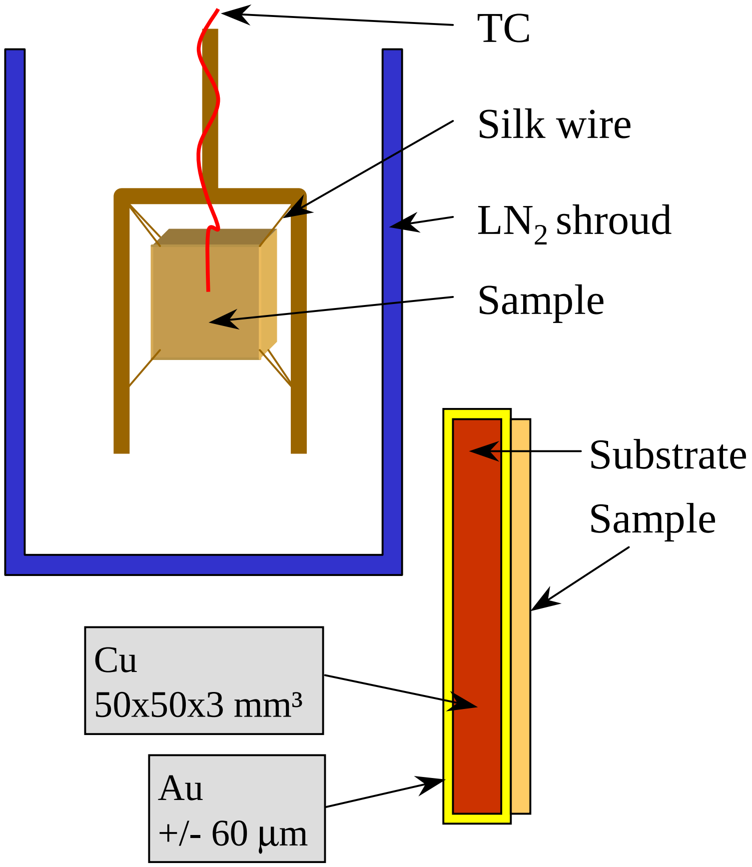
Determination of Absorptance

- Spectroscopic methods: Determination of total reflection spectrum in the UV/VIS/NIR spectral range with an integrating sphere accessory. Calculation of solar absorptance using standard solar spectrum from ASTM E490.
- Portable absorptance equipment: Integrating sphere with sample mounted on the wall, reflection of Xe light flash on sample, comparison between reflected light and reference beam, relative measurement (Xe lamp spectrum different from sun spectrum, detector has no spectral resolution).

ELAN Reflectometer EL 511



Determination of Emittance - Hemispherical Emittance



$$(mC)_T \frac{dT}{dt} = -\varepsilon\sigma S(T^4 - T_0^4)$$

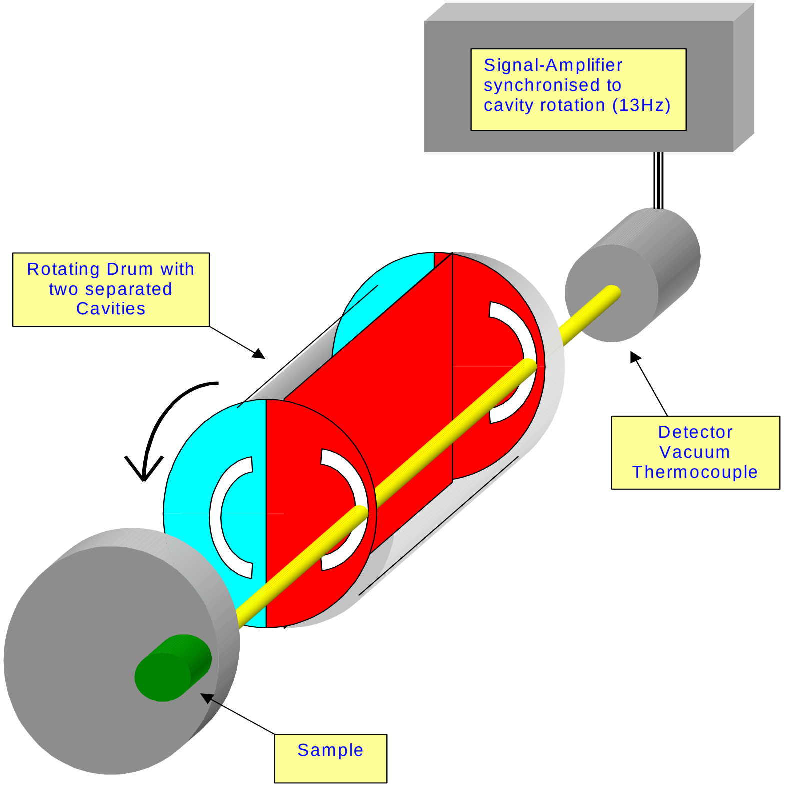
$$\text{with} \quad (mC)_T = M_{e,T}C_{e,T} + m_r C_r (1 + \alpha T + \beta T^2)$$

$$\varepsilon_e = \frac{\varepsilon S - \varepsilon_r S_r}{S_e}$$

ε : Emittance
σ : Stefan-Boltzmann constant
T : Temperature, K
T_0 : Temperature cold shroud. K
m : Mass
C : Specific Heat
S : Surface
t : time , e : sample , r : substrate

Determination of Emittance - Normal Emittance

- Reflection of heat from source at 80°C
- Consists of two semi-cylindrical cavities, rotating at 13 Hz.
  - One heated at 80°C
  - One stabilised at approximately RT
- Detector: Vacuum thermocouple
- Only reflected energy varies with the alternate irradiation by the two rotating cavities, and the detection amplifying system is made to respond only to the alternating signal.



Determination of Emittance - Spectroscopic Methods

Determination of total reflection spectrum in the IR spectral range with an integrating sphere accessory. Calculation of solar absorptance using black body (BB) radiation at specific temperature.