

# Evaluation of corrosion attack for metals

Basic and more advanced techniques



# Two principal ways to quantitatively assess corrosion attack gravimetrically



WI – weight increase ML – mass loss = corrosion attack = standard method



# **Quantitative techniques**

- Mass Loss (ML)
  - Normal balance, standard 10 x 15 cm samples (outdoor)
  - Microbalance, small 1 x 5 cm samples (indoor)
  - Resistance sensors
  - Cathodic reduction (only copper and silver)
- Weight Increase (WI)
  - Normal balance, standard 10 x 15 cm samples (outdoor)
  - Microbalance, small 1 x 5 cm samples (indoor)
  - Quartz Crystal Microbalance (QCM)

### **Resistance sensors**





## **Quartz crystal sensors**



$$\Delta f = -\left(\frac{f_0^2}{N \cdot \rho_q}\right) \cdot \Delta m$$

 $\Delta f$  = change in frequency,  $f_0$  =resonance frequency

N = frequency constant (1670 kHz mm for a transversal wave in AT-cut quartz)

$$P_q$$
 = density (2,648 g/cm<sup>3</sup> for quartz)

 $\Delta m$  = mass change/area, assuming film growth on one side of the crystal



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Mass respons of Ag vs. time during a simulated malfunction of the air filter system in an electrical control room.



#### On-line corrosion monitoring of indoor atmospheres

#### Quartz crystal microbalance

• used in practice

#### Electrical resistance sensors

- not as common under atmospheric conditions
- metals subject to even corrosion only
- sufficiently sensitive for indicating changes
- mechanically vulnerable
  - affected by temperature
- affected by humidity
- affected by dust etc.
- signal disturbances

- slightly vulnerable
- unaffected by humidity
- unaffected by dust etc.
- large spread in results

Electrical resistance sensors considered most promising, improvement necessary for corrosivity classification use



Mass and relative humidity variations during outdoor exposure of gold, based on the quartz crystal microbalance.

### **Corrosion logger**



#### • Corrosion sensors







# More advanced techniques



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# Experimental techniques for analysis of indoor corrosion effects.

Technique Microgravimetry Cathodic reduction Quartz crystal microbalance Atomic force microscopy X-ray photoelectron spectroscopy Infrared reflection absorption spectroscopy Kelvin probe

#### Information measured

Mass change, sensitivity 10 mg/m<sup>2</sup> Mass of corrosion products, 5 mg/m<sup>2</sup> Mass change, 0.2 mg/m<sup>2</sup> Surface morphology Surface chemical composition

Surface chemical composition

Volta potential

## In-situ FTIR-microspectroscopy



Schematic drawing of the newly developed experimental set-up for *in situ* FT-IR microspectroscopy



#### Schematic illustration of the model experiment





### $350 \text{ ppm CO}_2$

< 5 ppm CO<sub>2</sub>

•A secondary spreading effect was observed outside the original water droplet, which was much larger at <5 ppm  $CO_2$  than at 350 ppm  $CO_2$ .

*Ex situ* FT-IR microspectra obtained at the secondary spreading area. (RH:  $80\pm2\%$ ; <5ppm CO<sub>2</sub>;Exposure time: 6 hours.)



•Hydroxyl ions(OH<sup>-</sup>) were formed in the secondary spreading area due to the cathodic reaction.



## Combined IRAS and QCM.



## Correlation between IRAS absorbance and mass of Cu<sub>2</sub>O, based on three techniques.





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# Mass change and surface species on Cu in humid air, $SO_2$ and $O_3$ - based on IRAS/QCM.



T. Aastrup and C. Leygraf, J. Electrochem. Soc, 147, 2543 (2000)

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