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Characterization of Sputter-Deposited Iridium Oxide Coatings for Medical Implants

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Sputter-Deposited Iridium Oxide Coatings

- Why is IrO_x interesting?
- How can IrO_x coatings be made?
- Our own sputtering tests.
- Characterization of the coatings.

Why is IrO_x interesting?

- excellent compatibility with human body
- "electrode properties":
  - electrical conductivity similar to metal
  - redox reaction between Ir^{3+}/Ir^{4+} gives reversible electrochromic effect
  - $H_xIrO_2 \longleftrightarrow IrO_2 + x\ H^+ + x\ e^-$ $(x \sim 1)$
    colourless ↔ grey
  - transfer of current from electrode to tissue by Faradaic reaction rather than capacitance of Helmholtz electrical double layer
    → higher current densities

How can IrO_x coatings be made?

- sol-gel process

  fine IrO_2 powder precipitated from solution of iridium ethoxide by hydrolysis, oxidation and calcination

- thermal oxidation

  Ir-C films deposited by evaporation and oxidation in air 10 min / 250 °C

- anodic growth

  anodic oxidation of Ir metal films in 0.5-M H_2SO_4 by cycling potential between –0.25 and +1.25 V vs. SCE

- reactive sputtering

  sputtering from Ir metal target onto cooled substrates in high purity Ar + O_2

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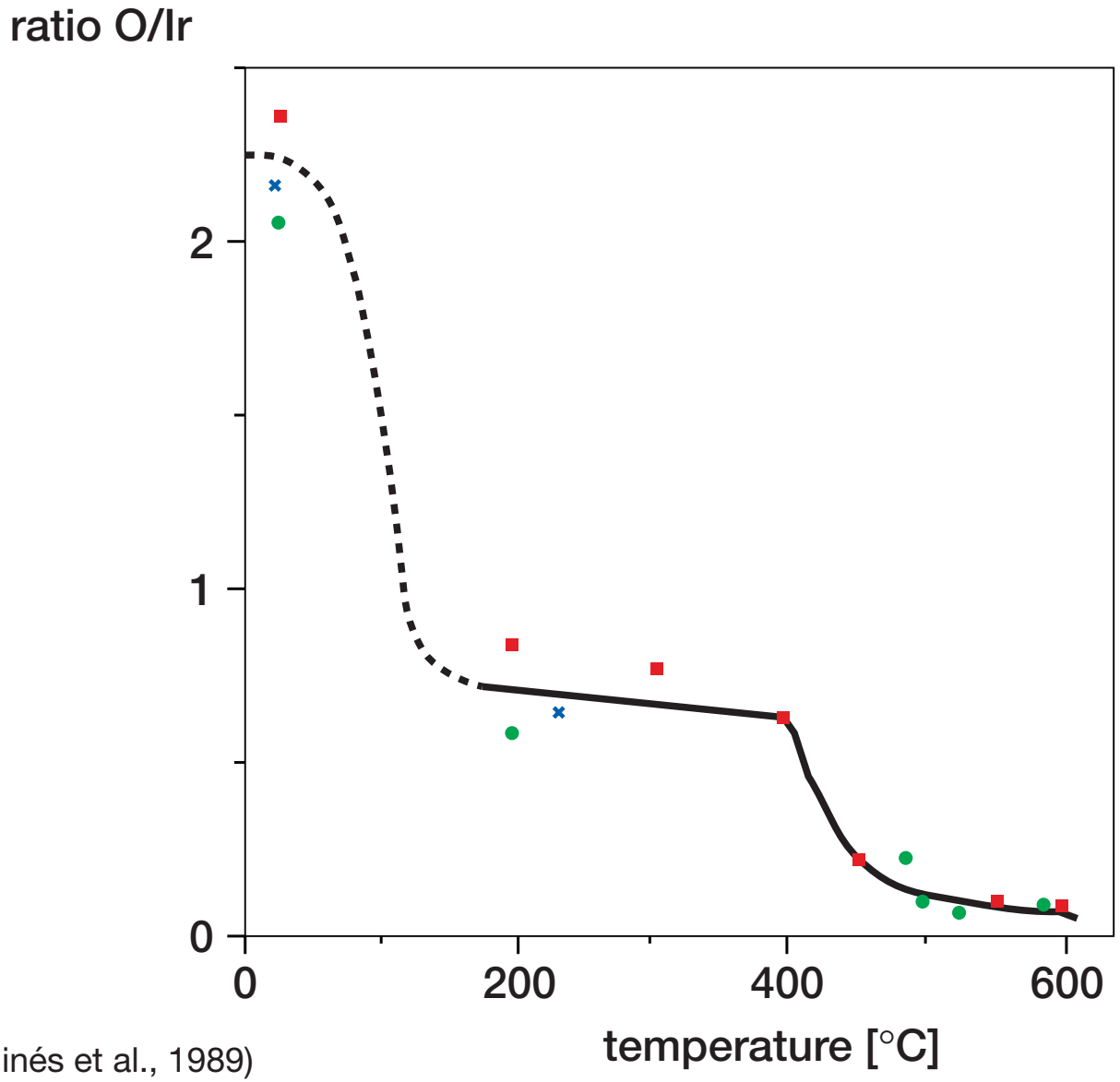
Thermal stability of "dry" sputtered IrO_x films in UHV



(from Sanjinés et al., 1989)

Sputtering tests (current investigations)

- Sputter deposition system with turbo drag pump, magnetron and 10 kW DC power supply
- Ir metal target, >99.9 % purity, solder-bonded to water-cooled backing plate
- Substrates: thin-walled tubes (2 mm dia. x 0.15 mm wall) mounted on rotating holder; radiation heating
- Atmosphere Ar / O_2

Substrates used

- Platinum
- Titanium (grade 2)
- Stainless steel (AISI 316L)
- Stainless steel + adhesion layer

Sputtering parameters

Test series	Temperature	Power	Oxygen partial pressure
1.	varied	500 W	2.7×10^{-3} mbar
2.	435 °C	varied	2.7×10^{-3} mbar
3.	385 °C	500 W	varied
4.	110 °C	500 W	varied

Sputtered energy 300 kJ per test series.

Typical film thickness 0.3 µm

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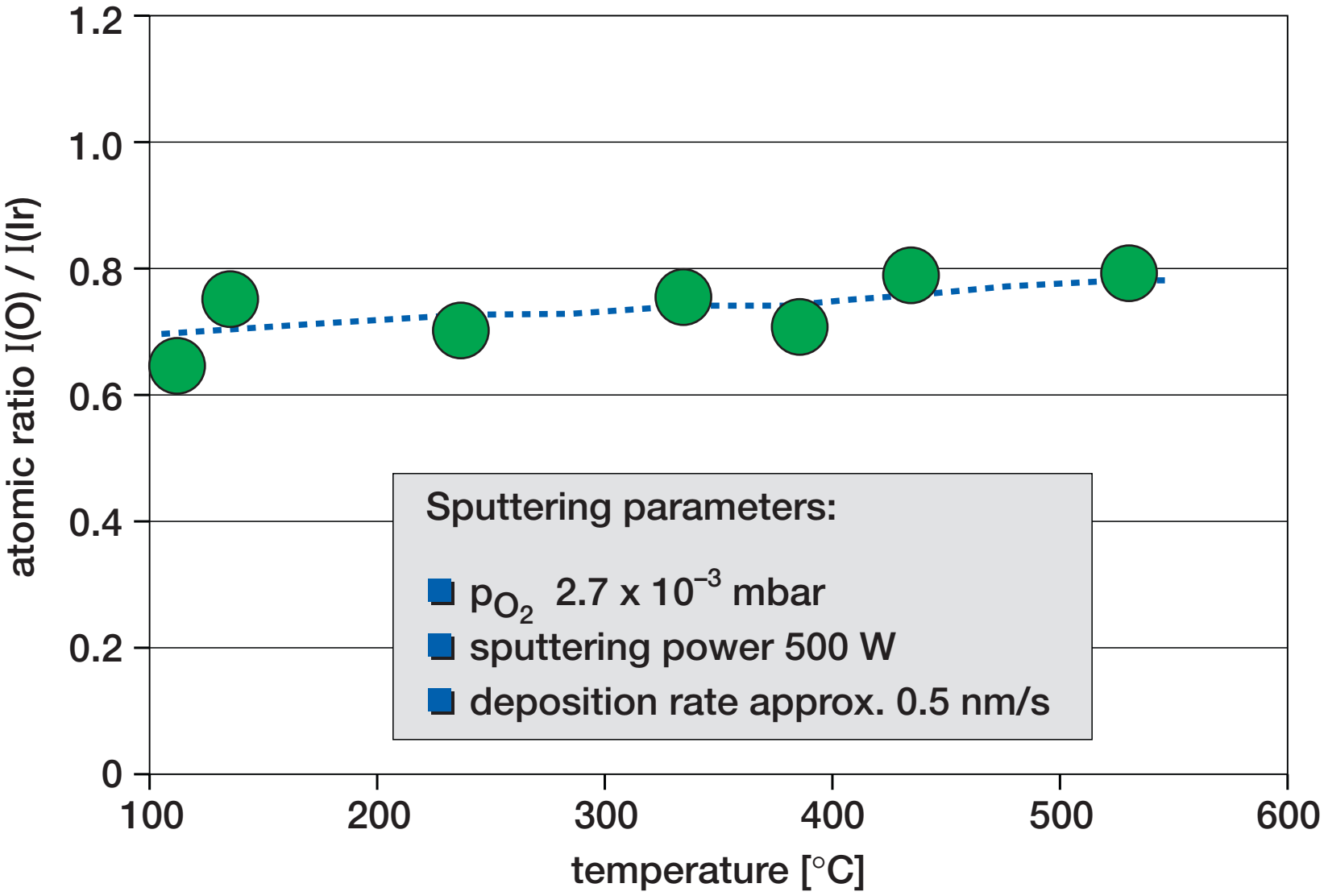
Influence of substrate temperature on oxygen content



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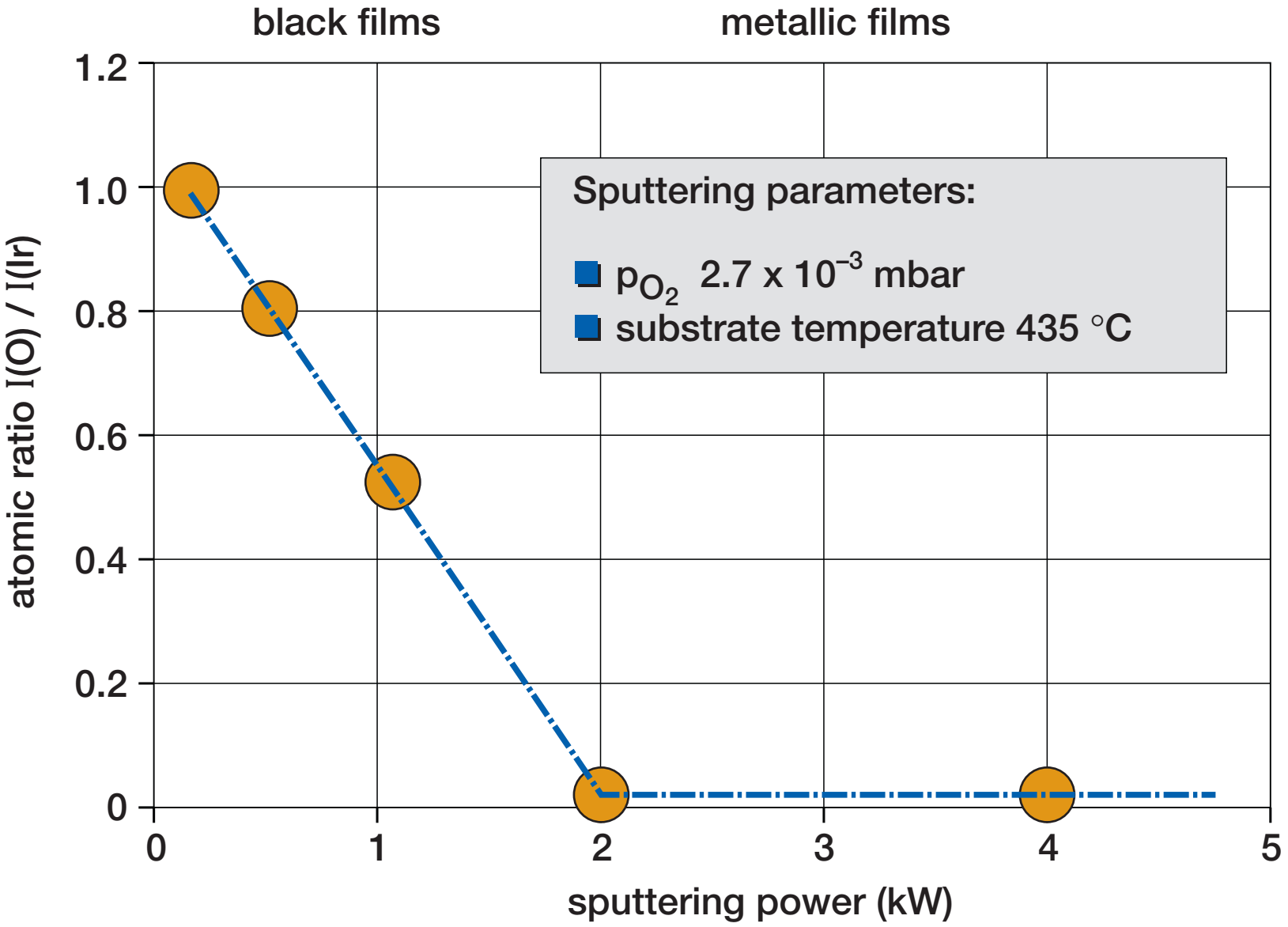
Influence of sputtering power on oxygen content



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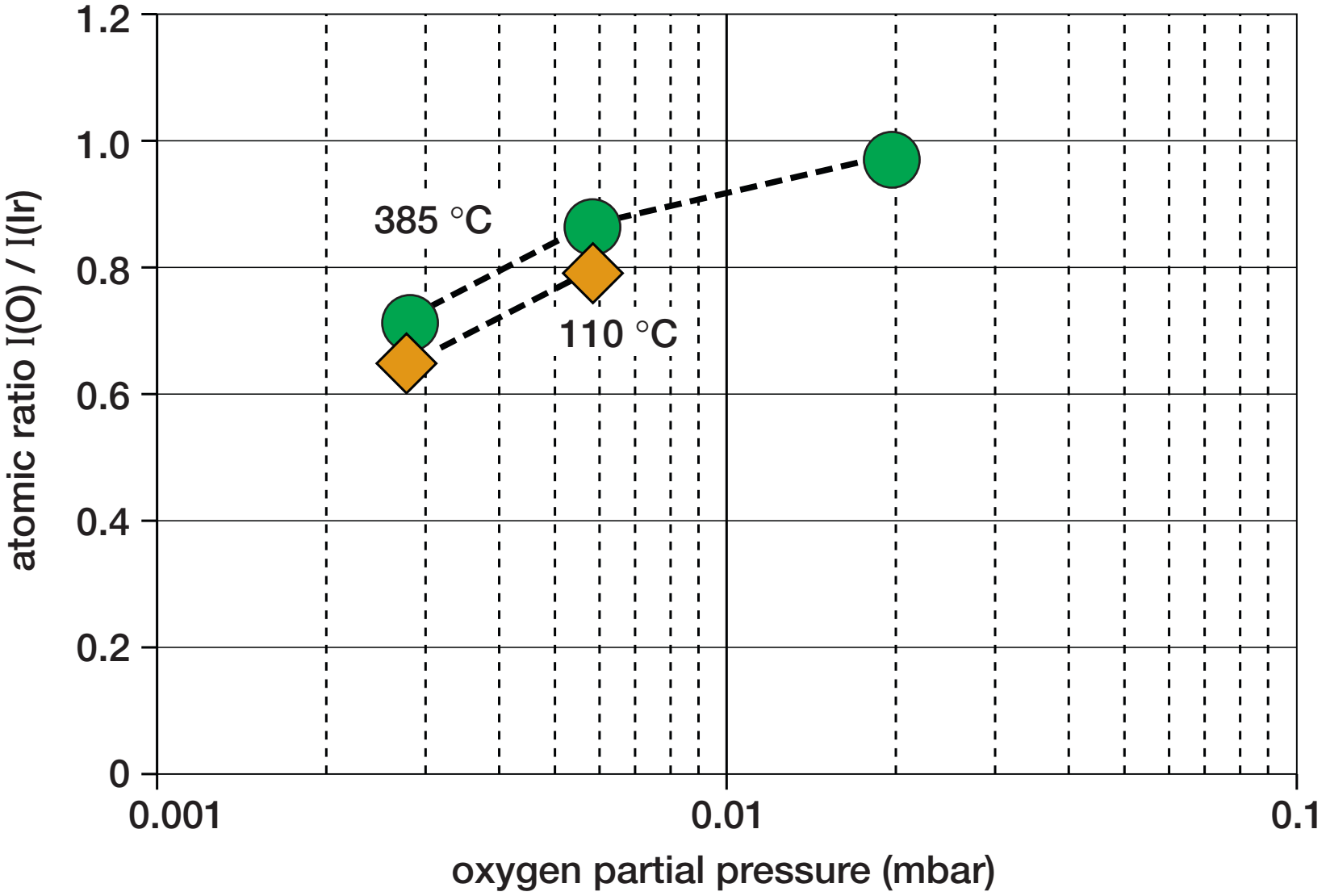
Influence of oxygen partial pressure on oxygen content



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Adherence tests on tube substrates

1. Adhesive tape test

   → pass = "good" (fail = "poor")

2. Tube squeezed to 0.9 mm

   + ultrasonic bath 20 s

   + examination 20 x

   → pass = "excellent"

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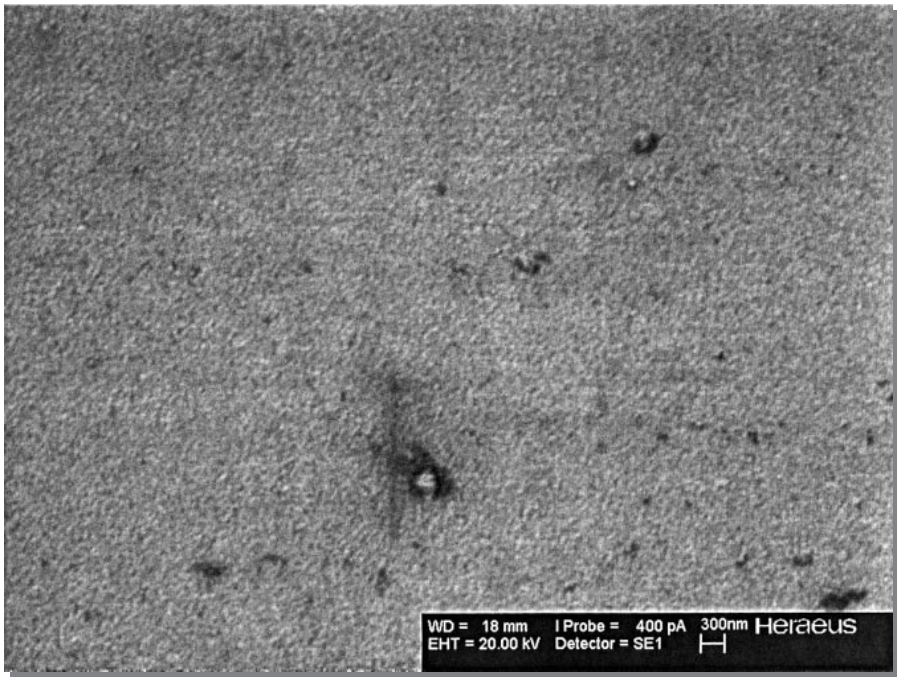
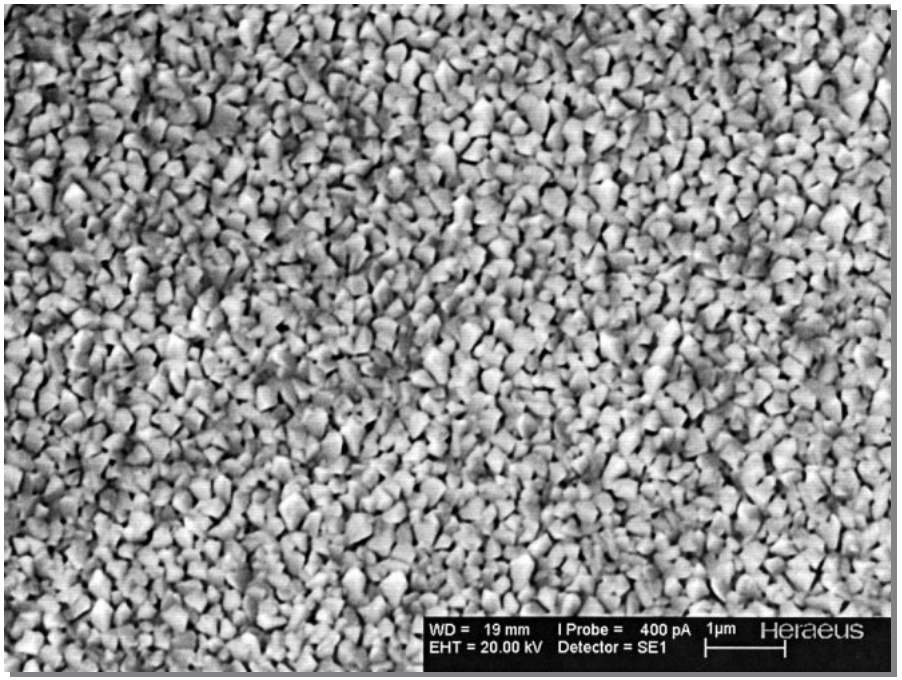
Adherence test results

	Number of samples tested		
Substrate material	poor	good	excellent
Platinum	0	12	3
Titanium	0	10	5
Stainless steel 316 L	12	3	0
Stainless steel 316 L + adhesion layer	5	6	4

IrO_x film morphology



film thickness 0.3 μm



film thickness 2 μm

Summary

- Reactive sputtering gives IrO_x films of mixed Ir / IrO_2.
- Oxygen content nearly constant ($IrO_{0.7}$) between 120 °C and 535 °C.
- Low growth rate essential for obtaining good Ir / IrO_x films but little effect on adherence.
- Adherence "good" to "excellent" on platinum and titanium.
- On stainless steel, metallic adhesion layer necessary.
- Good corrosion stability on stainless steel with adhesion layer.

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X-ray diffraction pattern of IrO_x coatings