# Characterization of Sputter-Deposited Iridium Oxide Coatings for Medical Implants

**T. Loose, M. Frericks, T. Giesel, G. Herklotz**

**W.C. Heraeus GmbH & Co. KG**
**Hanau, Germany**

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Irox3-2000/Lupton 01e

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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.

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# 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

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# 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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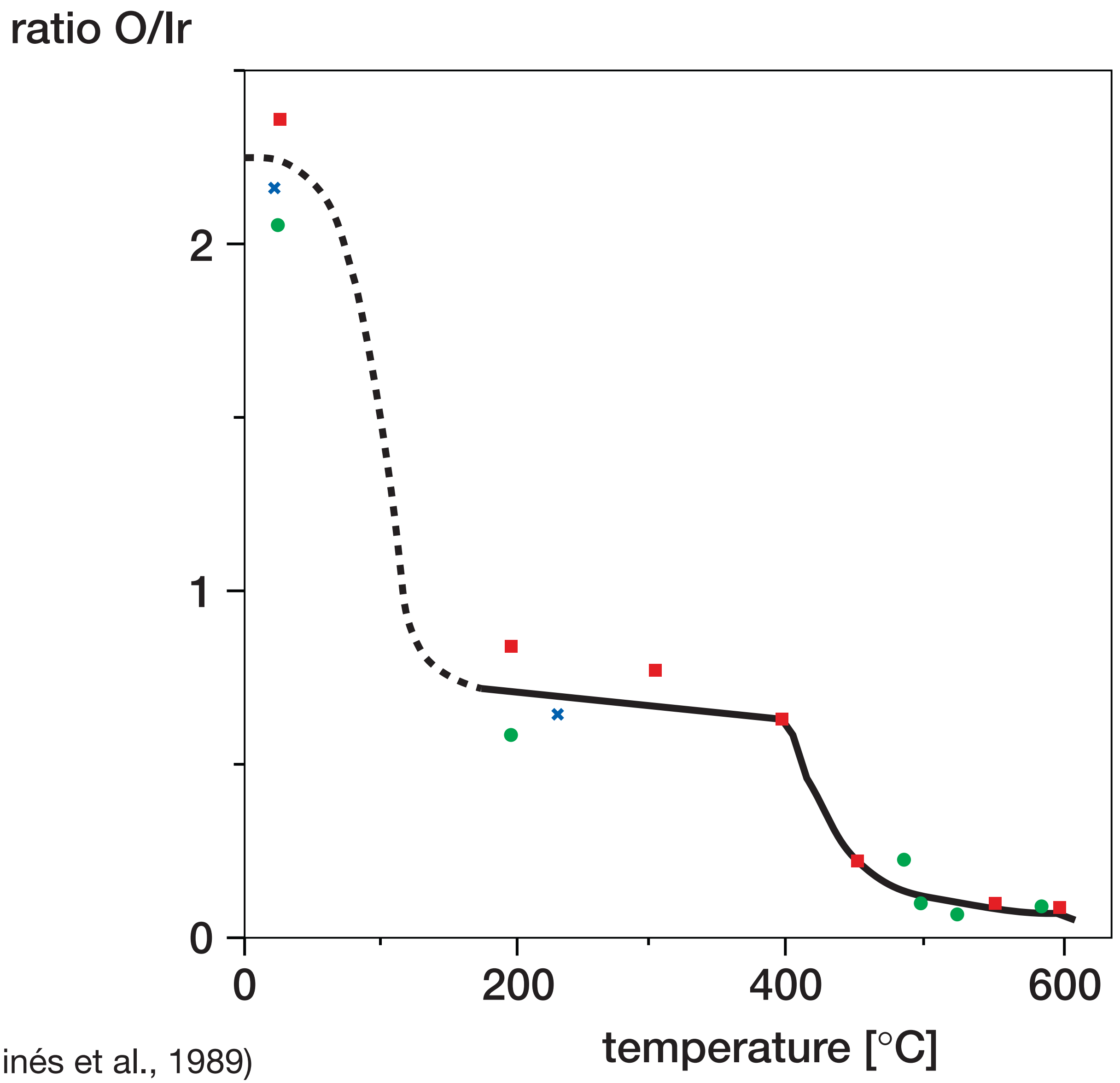
# Thermal stability of "dry" sputtered $IrO_x$ films in UHV




(from Sanjinés et al., 1989)

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# 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 / $O_2$

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# Substrates used

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

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# Sputtering parameters

| Test series | Temperature | Power | Oxygen partial pressure |
|---|---|---|---|
| 1. | varied | 500 W | $2.7 \times 10^{-3}$ mbar |
| 2. | 435 °C | varied | $2.7 \times 10^{-3}$ 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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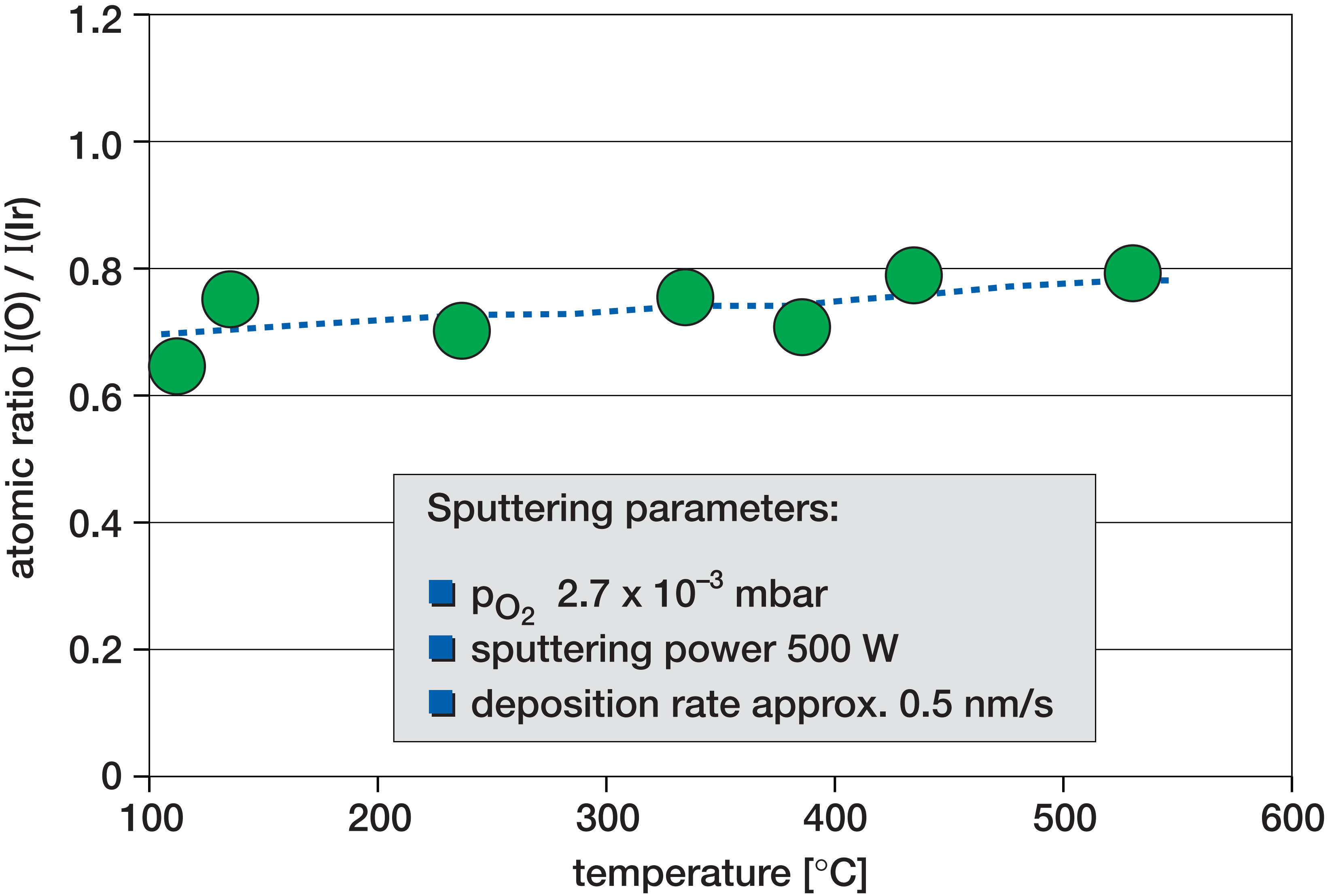
# Influence of substrate temperature on oxygen content




Irox3-2000/Lupton 09e

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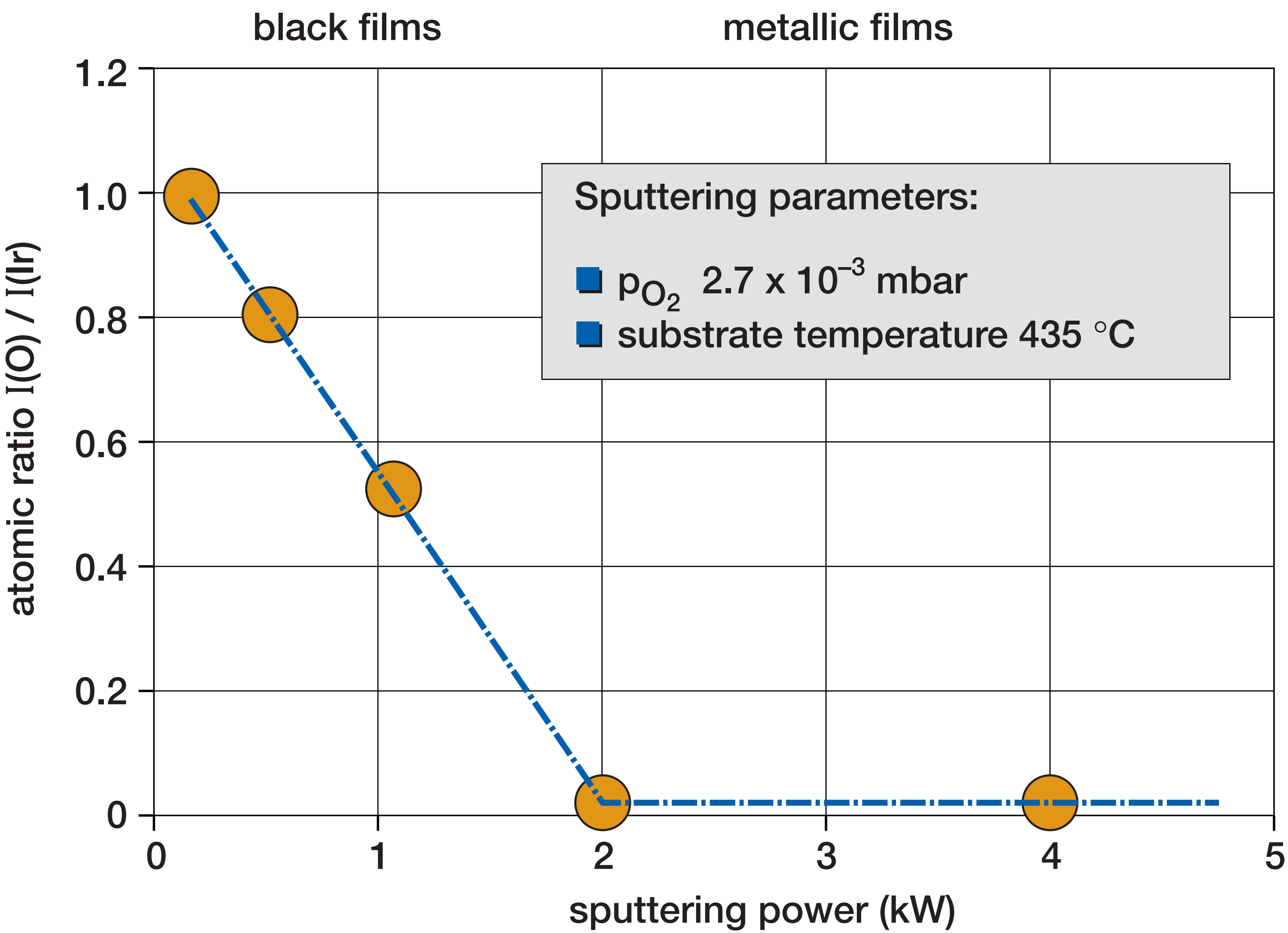
# Influence of sputtering power on oxygen content




Irox3-2000/Lupton 10e

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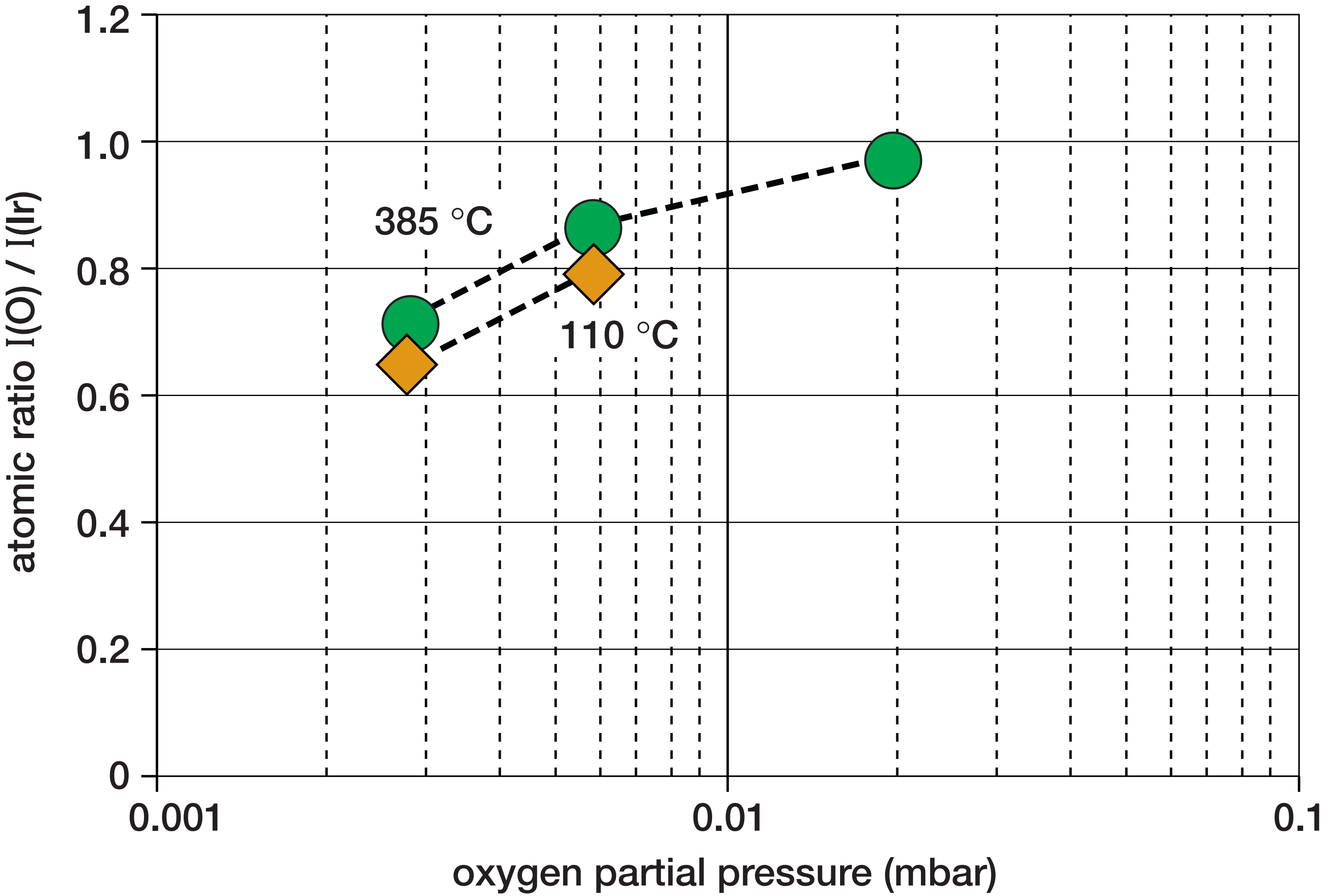
# Influence of oxygen partial pressure on oxygen content



# 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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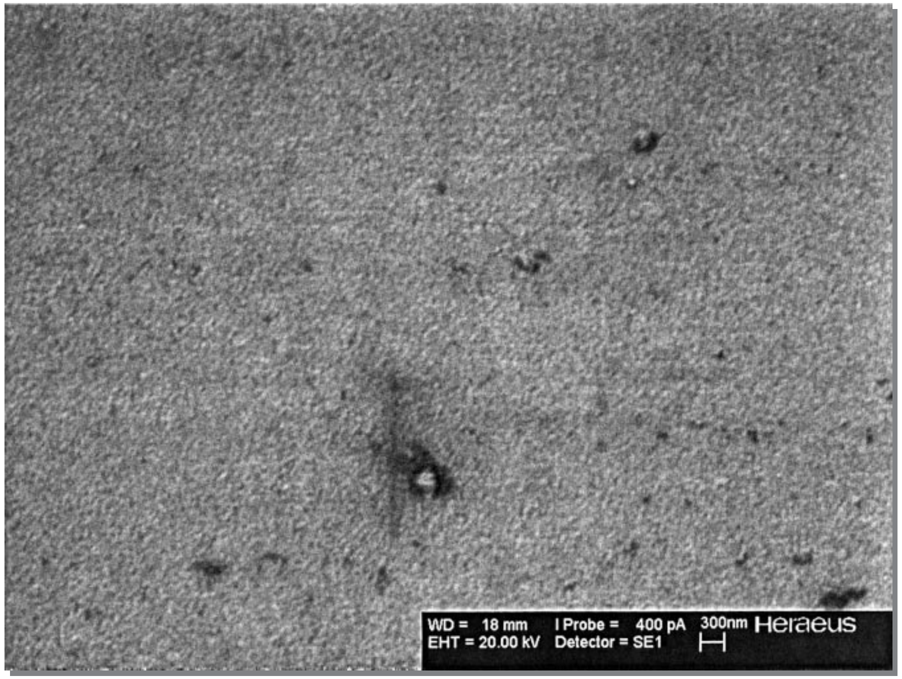
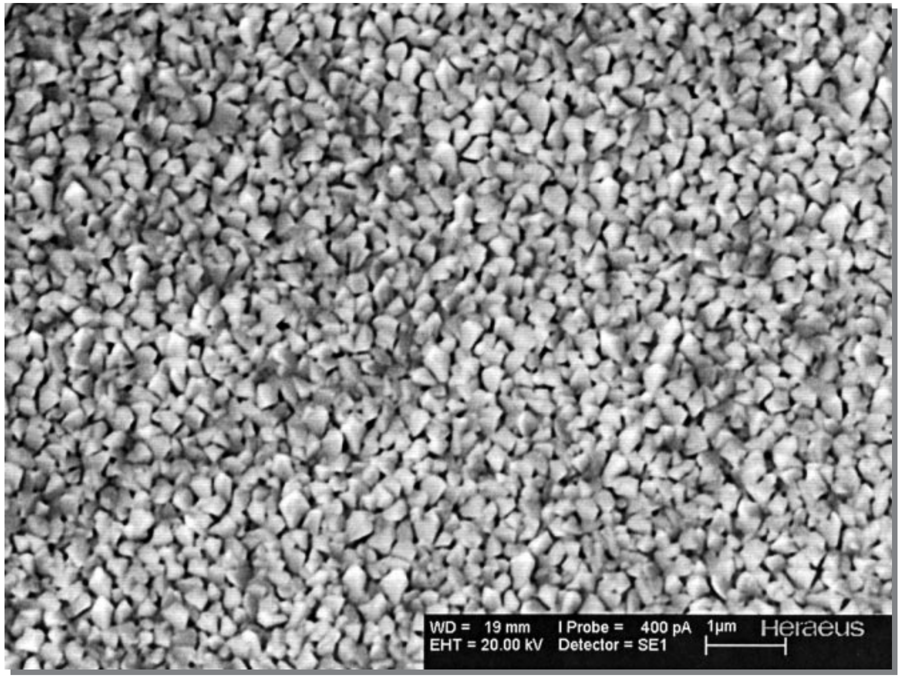
# Adherence test results

| Substrate material | Number of samples tested: 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.

# X-ray diffraction pattern of $IrO_x$ coatings