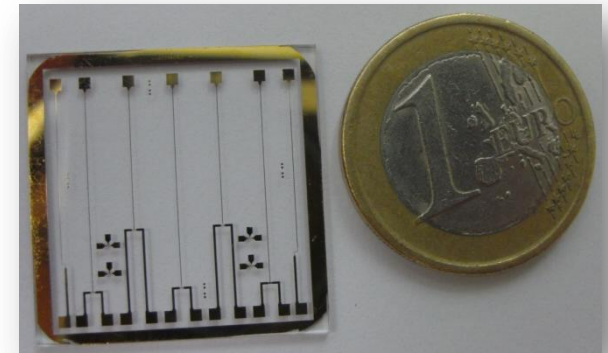


Room Temperature Sputtered Ta₂O₅ for Solid State Biosensors



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Outline

- **Biosensors**
- **EIS and ISFETs**
- **Oxide semiconductors, dielectrics and TFTs**
- **Production & characterization details**
- **Results**
- **Conclusions**

Biosensor applications

- **Clinical diagnostics stock**
- **Monitoring**
- **Pharmaceuticals & Drug Discovery**
- **Environment**
- **Food Control**
- **Biodefense**
- **Forensic / Genetic identification**
- **Research**
(medical, biochemical, biotechnological)

Electrochemical biosensors

Potentiometric biosensor, was invented by Clark in 1962

ISFET, was invented by Bergveld in 1970

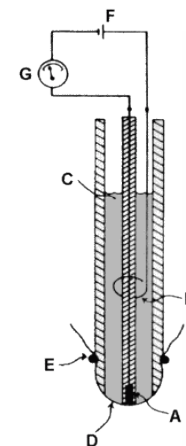
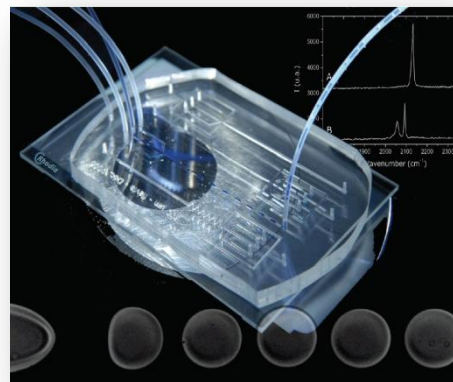
Among the types of biosensors that have been proposed, the ISFET has several advantages:

*label free
small size and weight
fast response
high reliability
low output impedance
on-chip & bio-integration
miniaturization
continuous monitoring (in-situ)
small volume samples*

Low cost

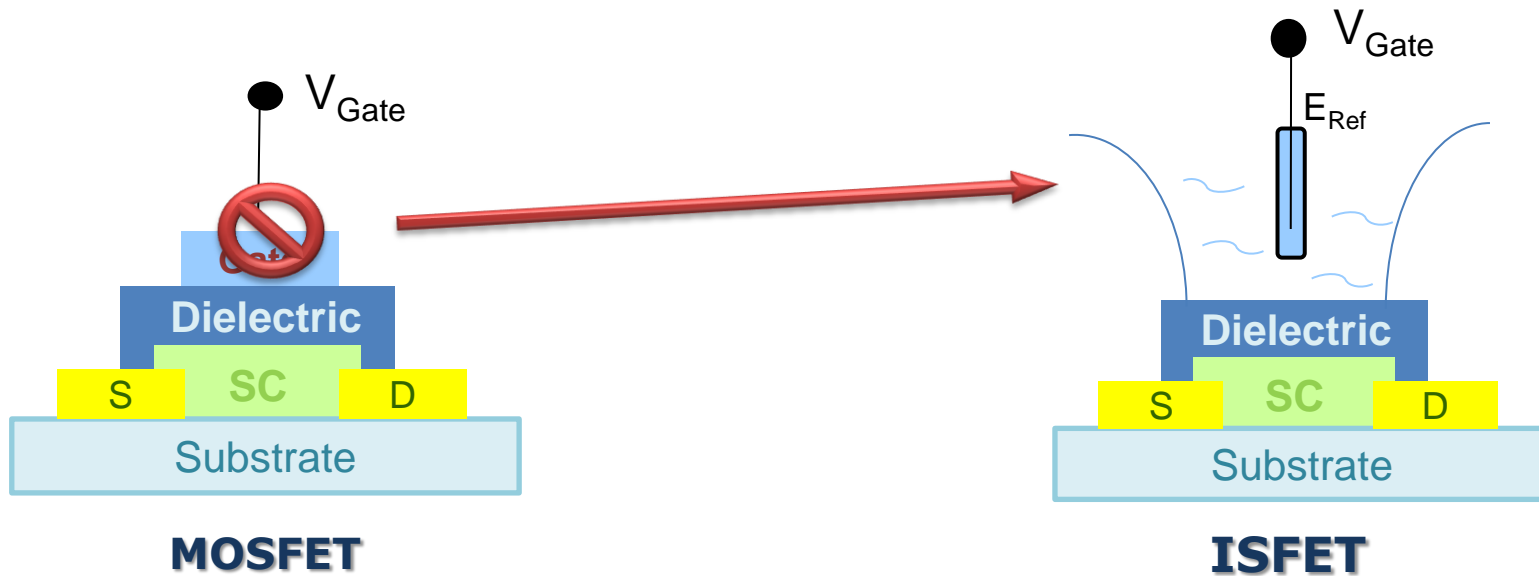


lab-on-chip applications



What is an ISFET?

(Ion Sensitive Field Effect Transistor)



MOSFET
Metal Oxide Semiconductor
Field Effect Transistor

ISFET
Ion Sensitive Field Effect Transistor

The gate electrode is substituted by a reference electrode (E_{Ref}) and a solution. Changes in the characteristics of the solution modulate the TFT channel conductance.

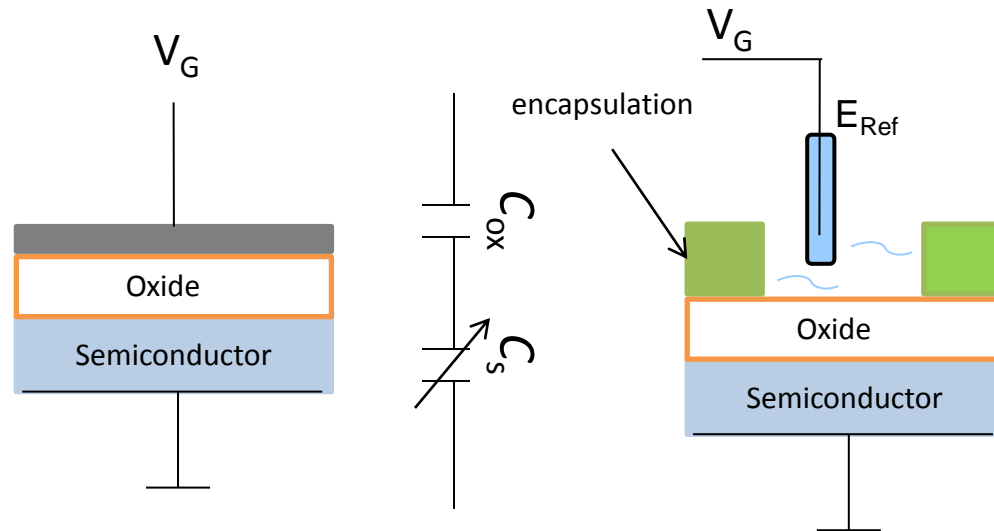
How does an ISFET work?

MIS/MOS vs EIS STRUCTURES

(Electrolyte-Insulator-Semiconductor)

The simplest field effect device is the MOS/MIS capacitor. The total capacitance, C is a series combination of the insulator capacitance (C_{ox}) and the semiconductor depletion layer capacitance, C_s .

$$\frac{1}{C} = \frac{1}{C_{ox}} + \frac{1}{C_s}$$

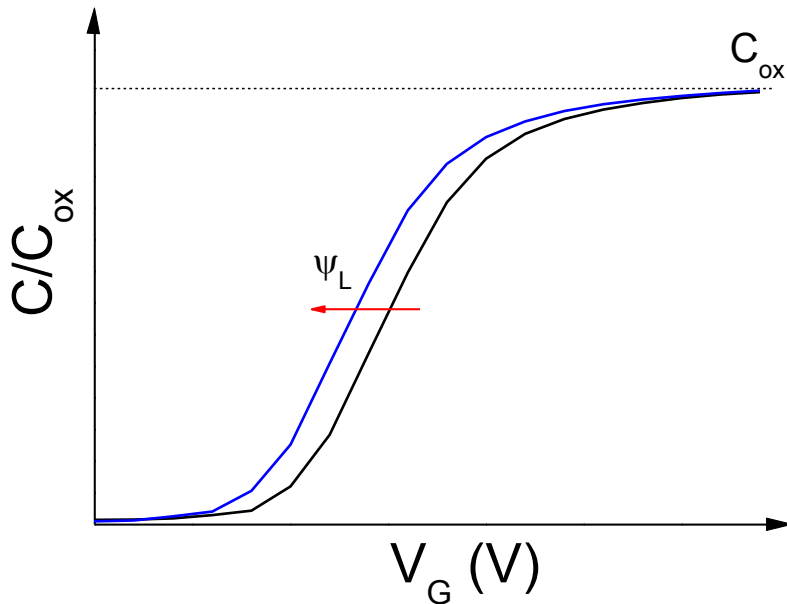


The layer set-up of the **EIS** sensor mimics the gate region of an **ISFET** with the advantage that no photolithographic process steps are necessary and the devices are fabricated with only one deposition.

How does an ISFET work?

EIS STRUCTURES

(Electrolyte–Insulator–Semiconductor)



Typical capacitance vs gate voltage

- The flat-band potential varies linearly with pH
- Different kinds of sensitive layers/membranes (SiO_2 , Si_3N_4 , Ta_2O_5 , Al_2O_3 , TiO_2 etc...)



pH sensitive Capacitor

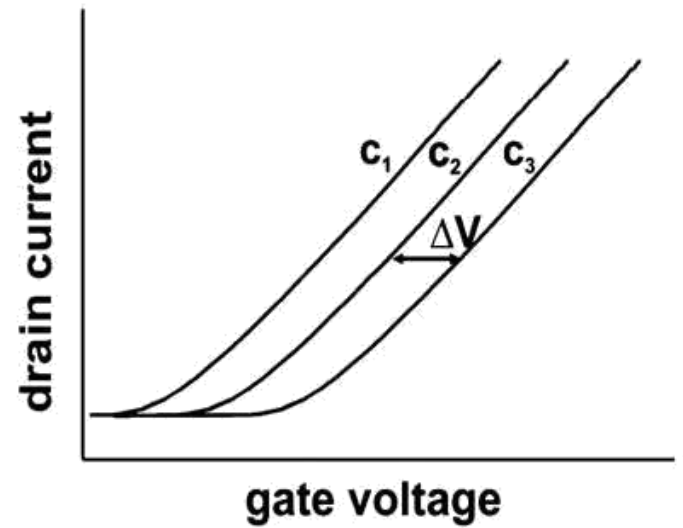
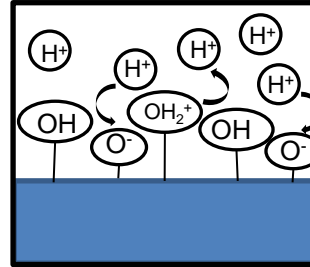
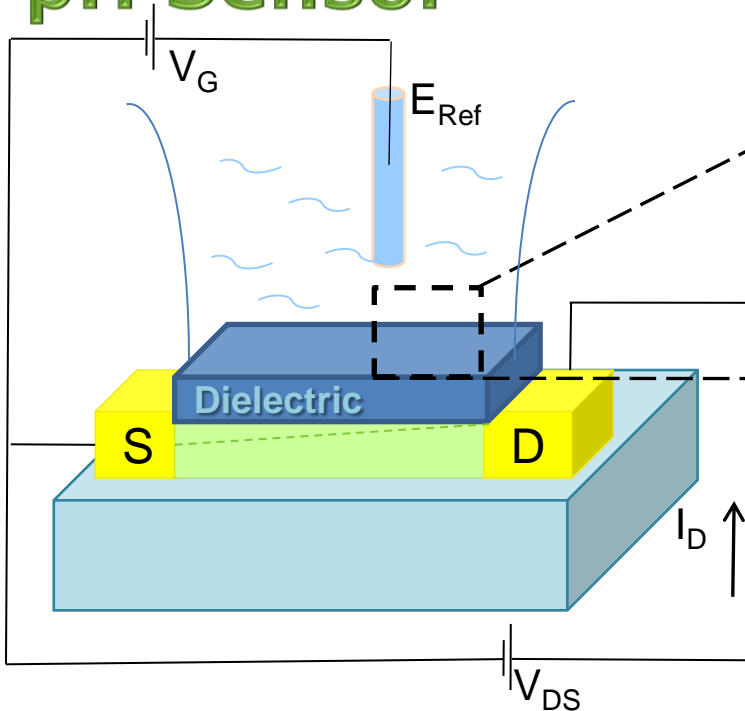
The pH of a solution is defined by:

$$\text{pH} = -\log[\text{H}^+]$$

where $[\text{H}^+]$ is the molar concentration of protons.

How does an ISFET work?

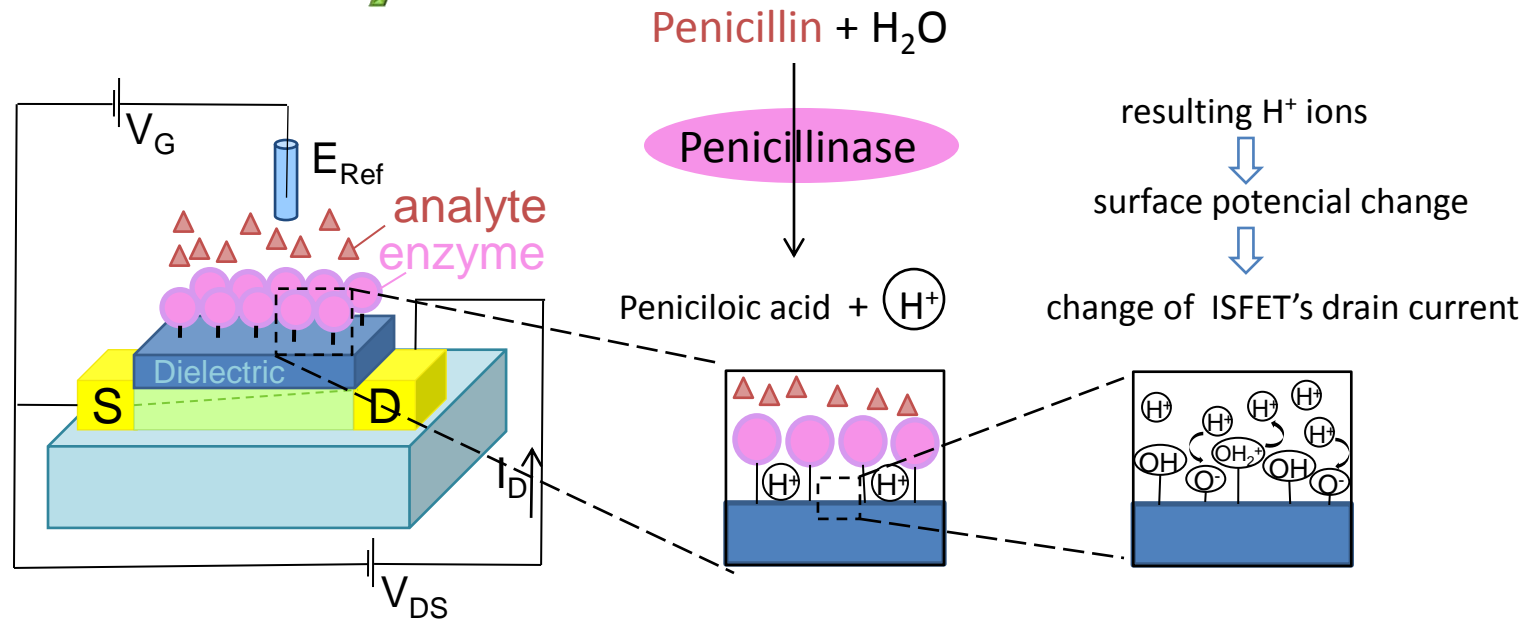
pH Sensor



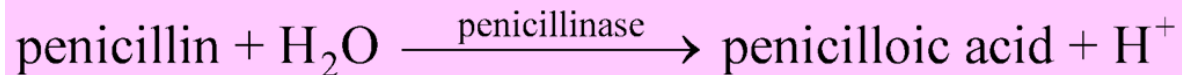
- ❖ The **Dielectric** – is amphoteric (accepts and releases protons)
- ❖ The surface potential varies with $[H^+]$
Threshold voltage shift
- ❖ The pH variation modulates the channel conductance

How does an ISFET work?

EnFET enzyme sensor



The corresponding enzymatic reaction is:



pH detection mechanism (acidic or basic product)

pH variation is directly correlated to penicillin concentration

Amorphous Oxide Semiconductor and Dielectrics

Layer	Materials	Method
Source/Drain	TCO ($\text{In}_2\text{O}_3:\text{ZnO}$)	RF Sputtering
Dielectric	SiO_2 , Al_2O_3 , Ta_2O_5 ...	RF Sputtering
Semiconductor	ZnO based ($\text{Ga}_2\text{O}_3:\text{In}_2\text{O}_3:\text{ZnO}$)	RF Sputtering

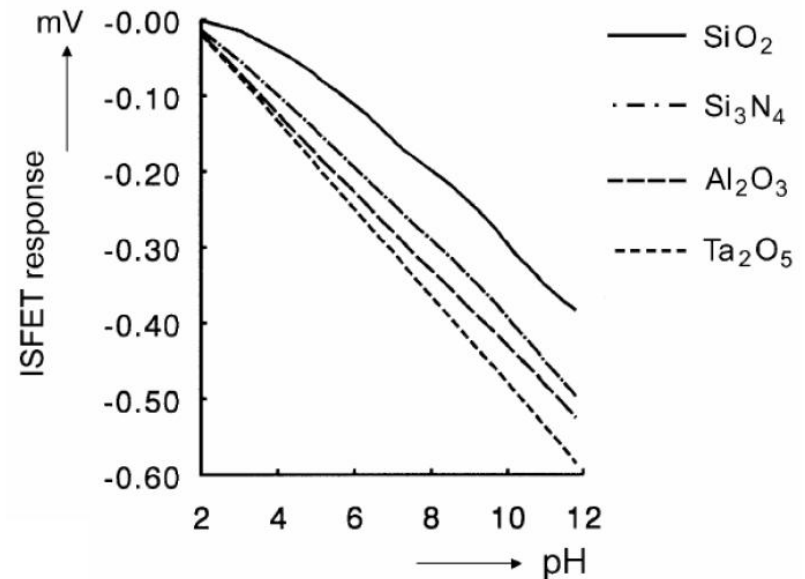
**Room Temp.
Production**

- ◆ The Dielectric sensitive layer is critical
- ◆ Ta_2O_5 is a promising material
high surface buffer capacity

Sensitive layer optimization

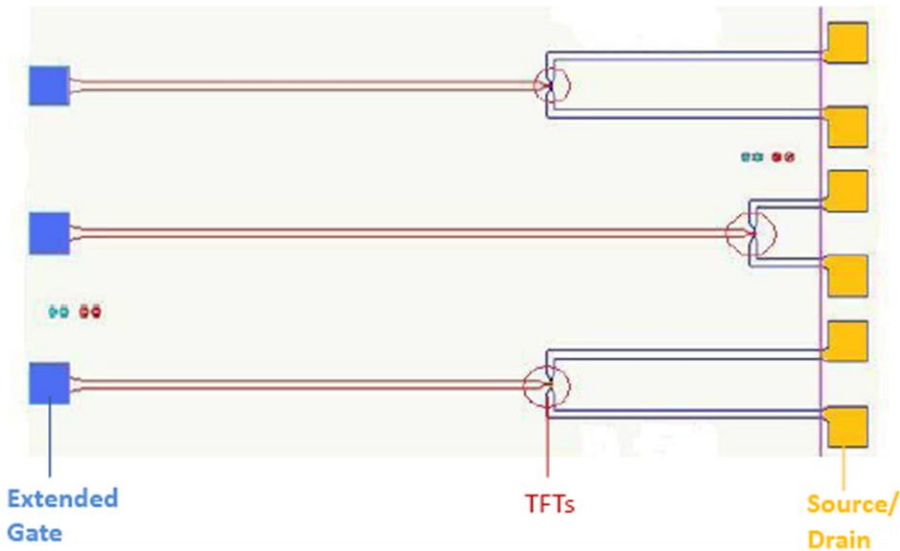


EIS structures



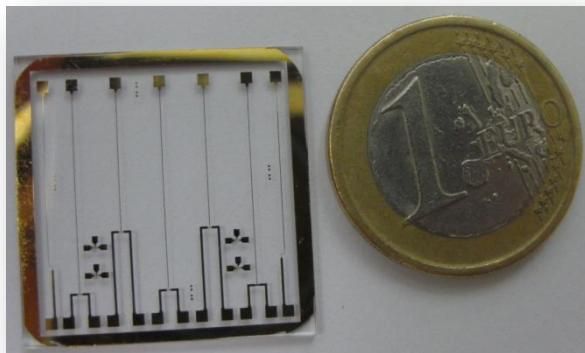
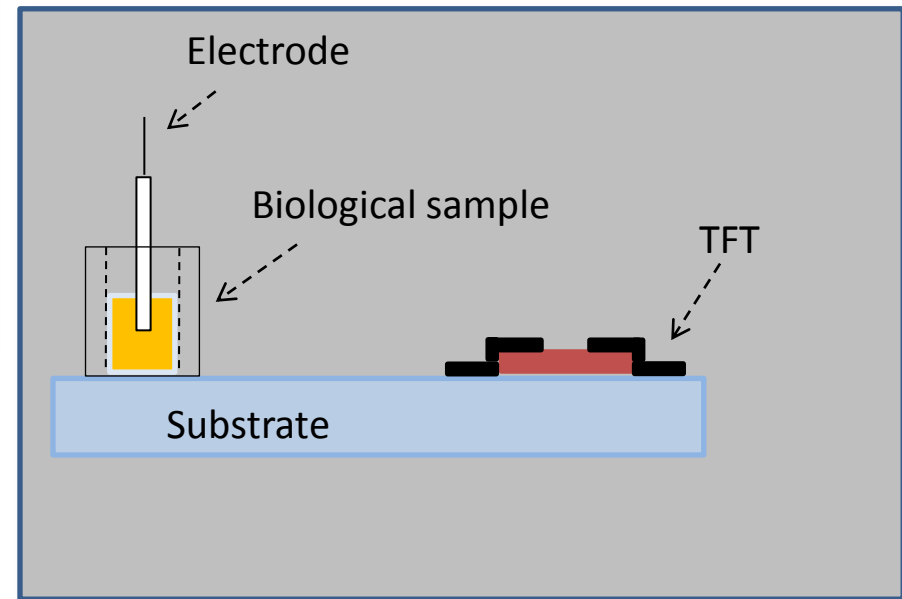
ISFET fabrication

Extended Gate Structure of ISFETs



Extended gate configuration:

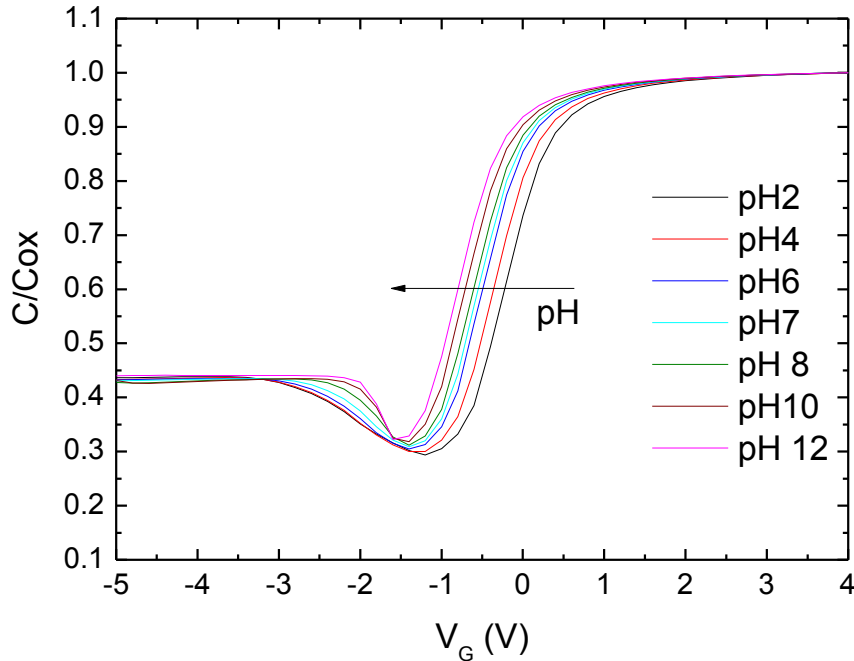
This approach allows an easy separation between the sensing area and the transistor in order to reduce any risk of leakage.



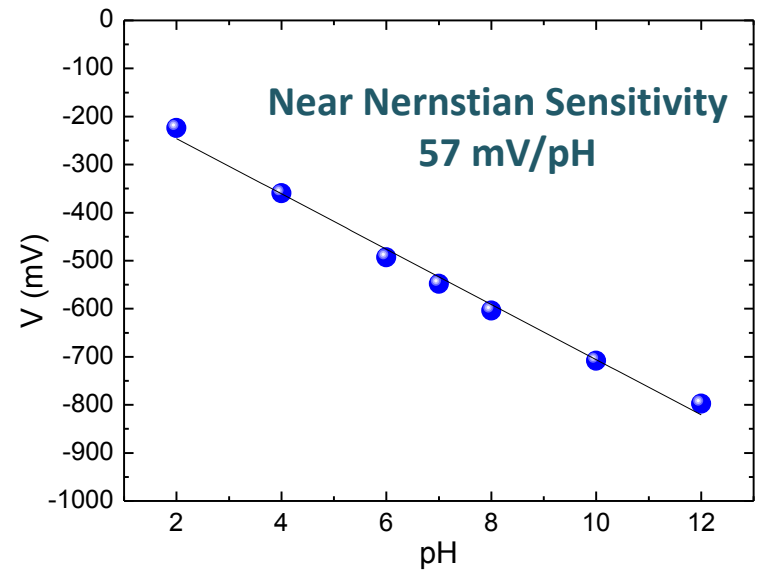
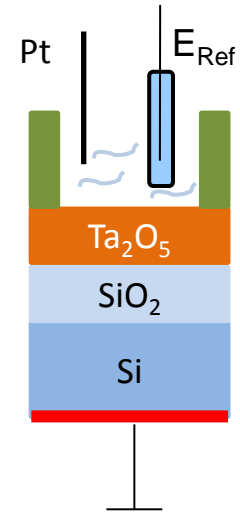
➔ ISFET produced @ CENIMAT

EIS pH Sensor Results

100 nm Ta₂O₅ through reactive RF sputtering

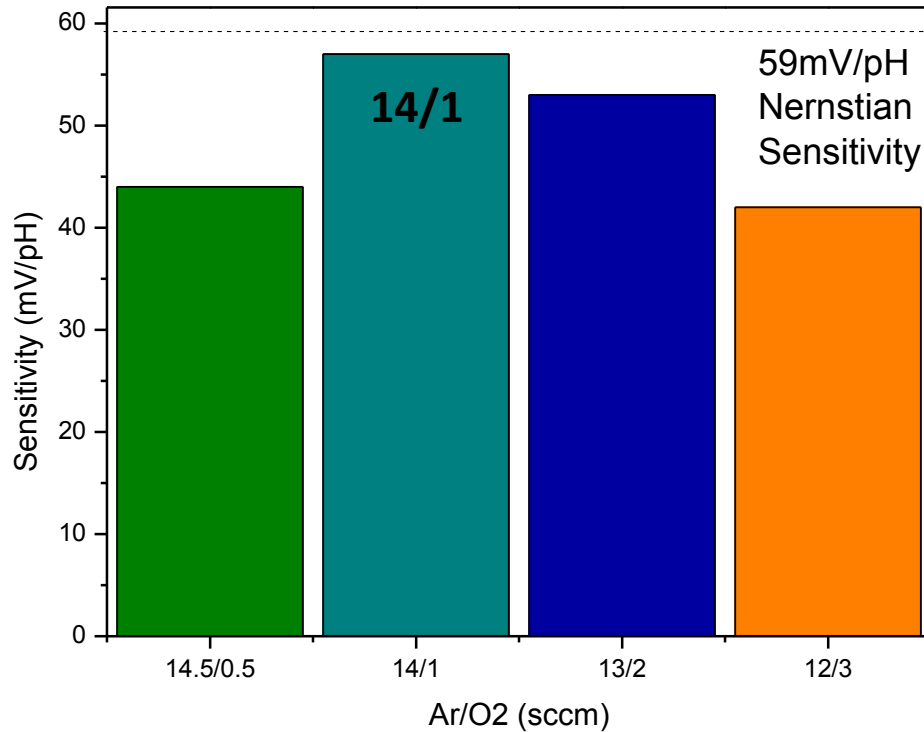


The flat-band potential varies linearly with pH

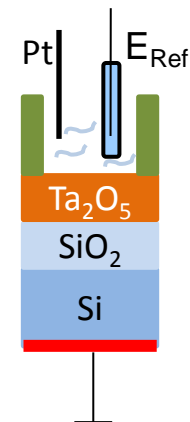


O₂ Partial Pressure

O₂ Partial Pressure influence on pH sensitivity
P_{dep}=0.3 Pa; P=150W; d=100nm

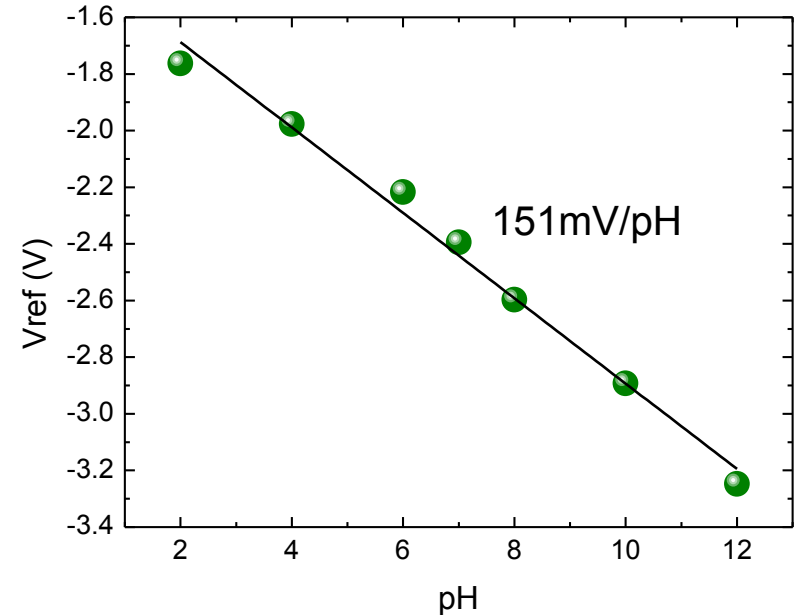
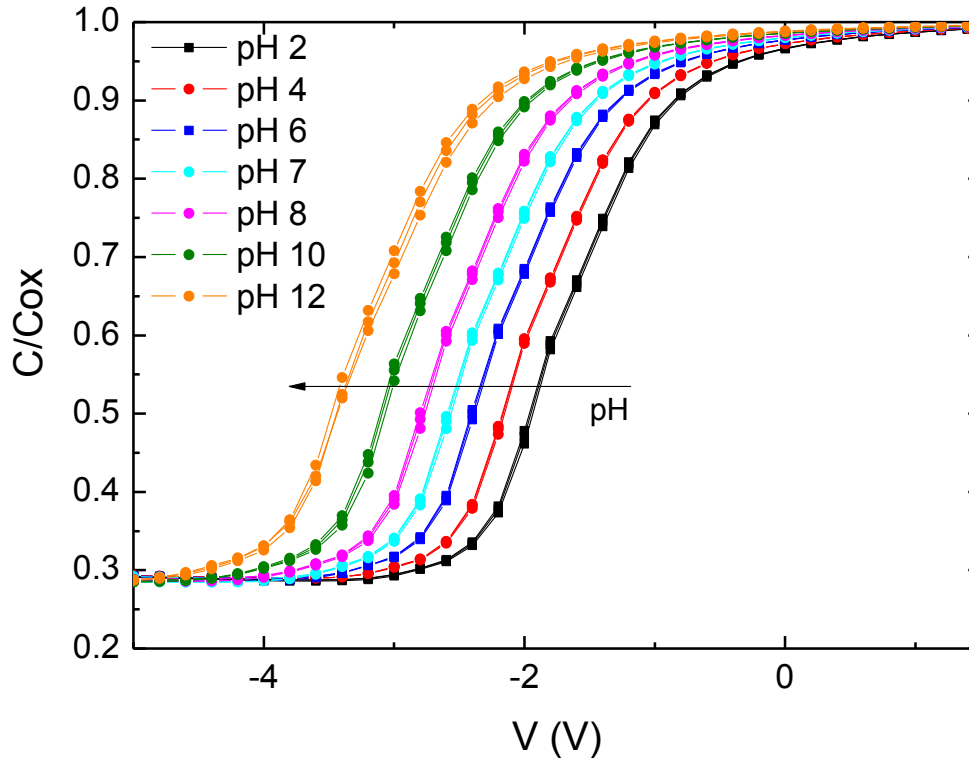


Maximum sensitivity was obtained for 14/1 sccm Ar/O₂ gas flow



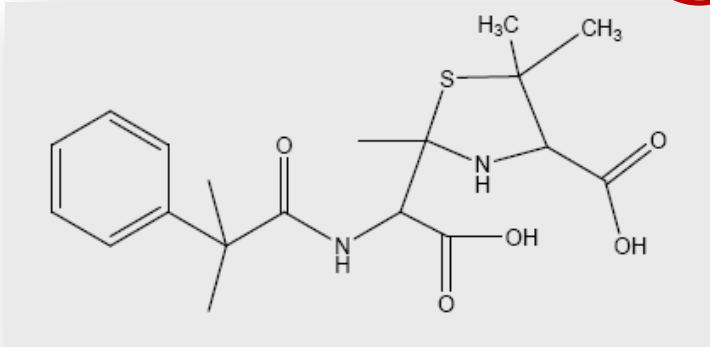
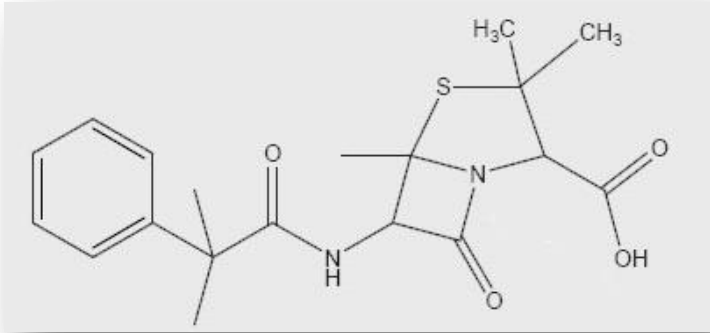
O₂ Surface Plasma Treatment

O₂ plasma (30W, 5') sample

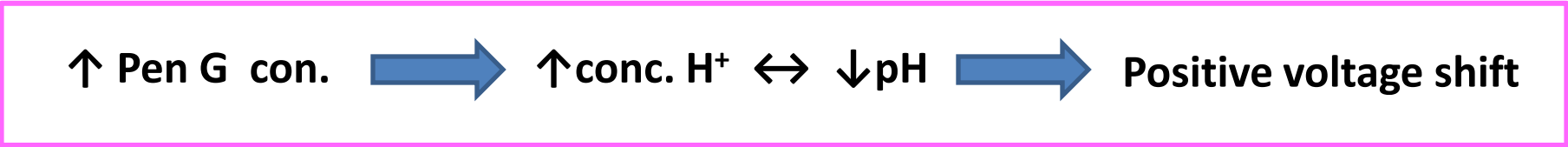


- ◆ Sensitivity was enhanced 2.5 times compared to untreated sample
- ◆ C-V curves shifted to more negative V
- ◆ Variability increase from 9% (untreated) to 12% (O₂ 30W 5min.)

EIS Penicilline Sensor

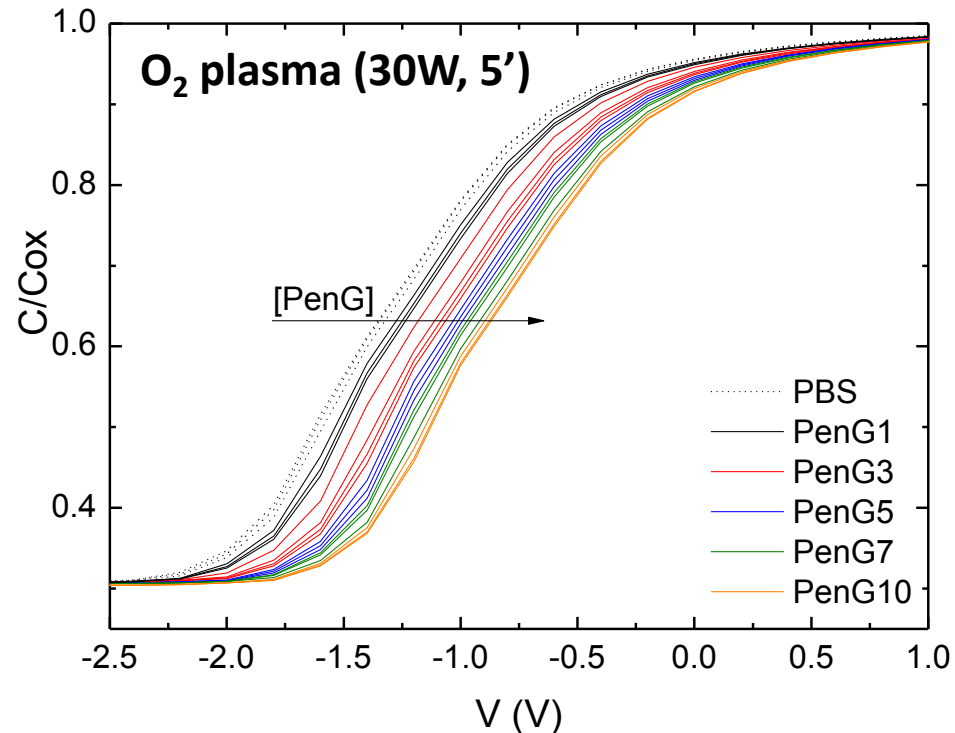
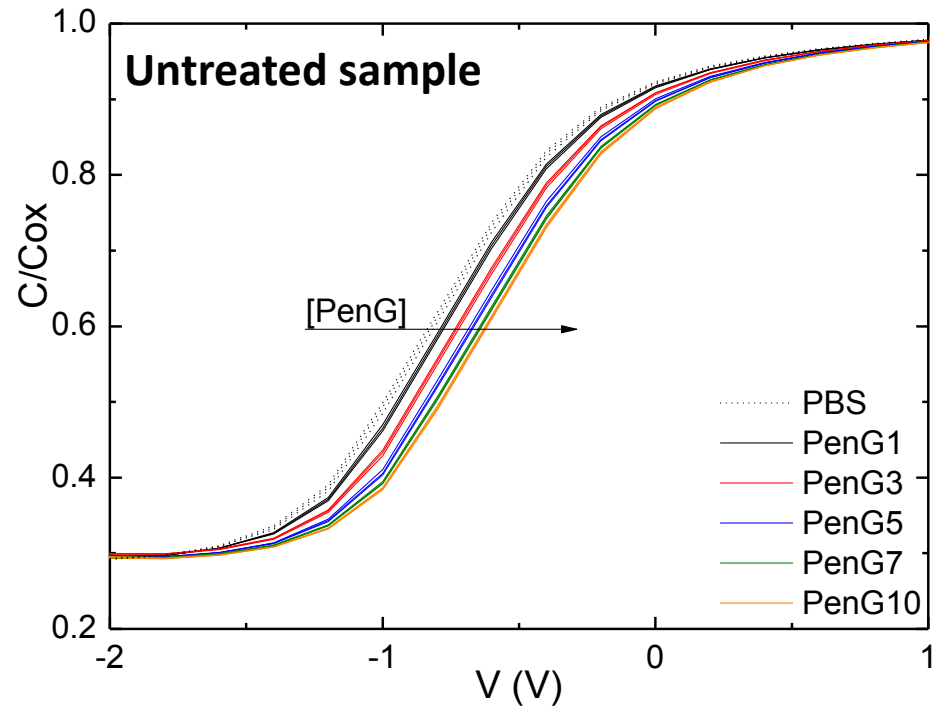


The enzymatic reaction induces a local pH change measured by the underlying EIS capacitor



Enzyme modified EIS sensors

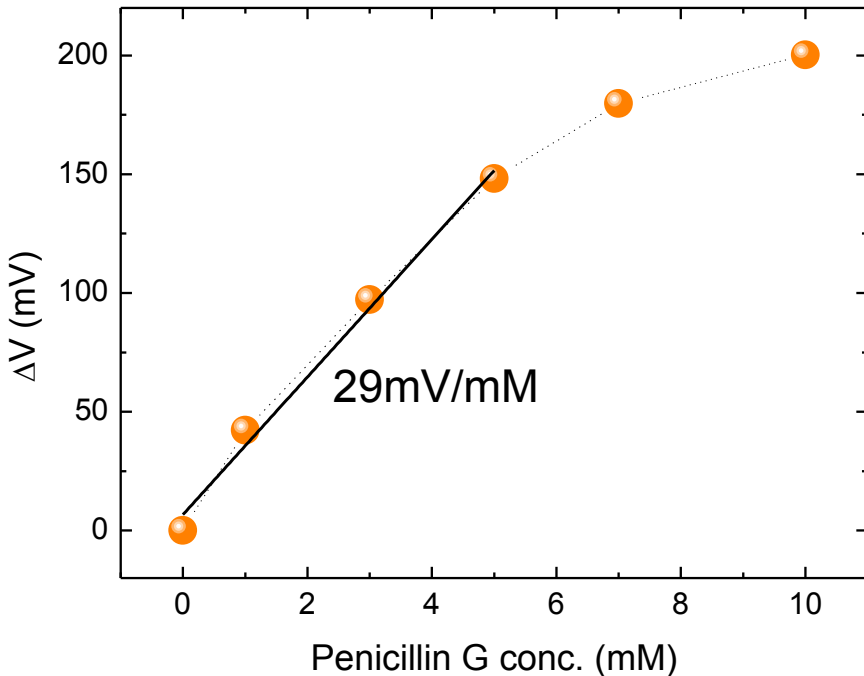
Immobilization of penicillinase on the oxide surface: physical adsorption
Buffer solutions (5 mM PBS, pH 7) with different concentration of penicillin (PenG)



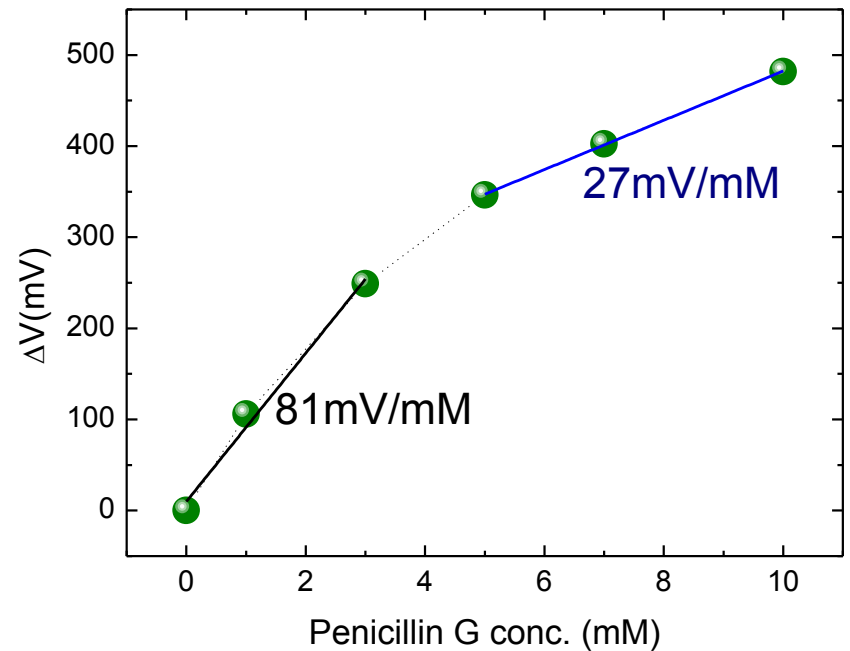
↑ Pen G con. → ↑ conc. H⁺ ↔ ↓ pH → Positive voltage shift

Enzyme modified EIS sensors

Untreated sample

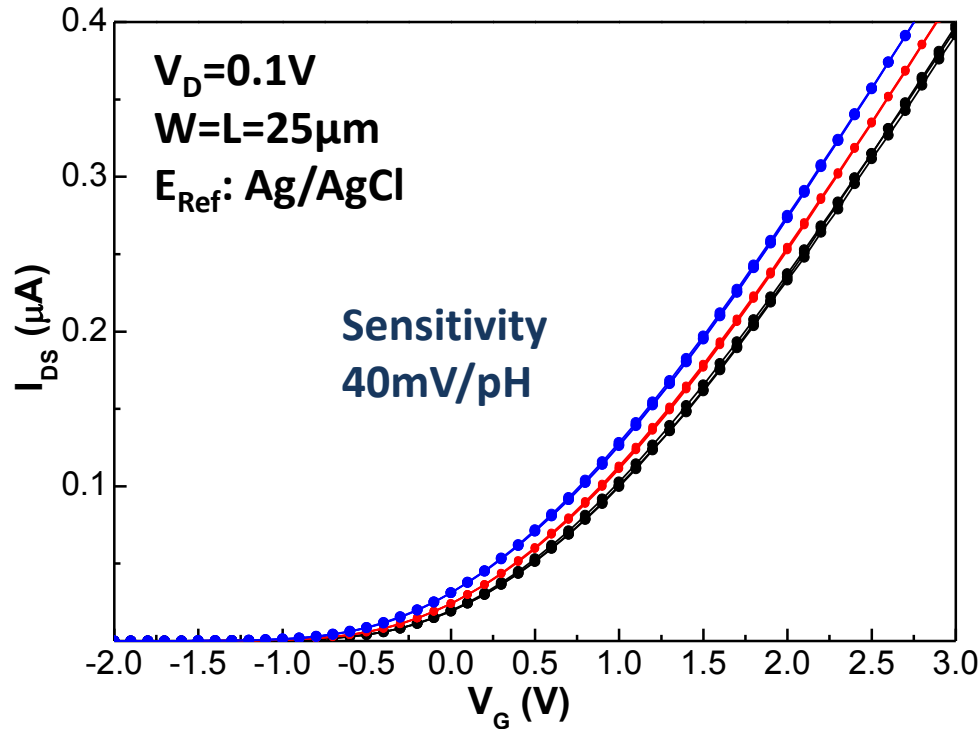


O₂ plasma (30W, 5') sample

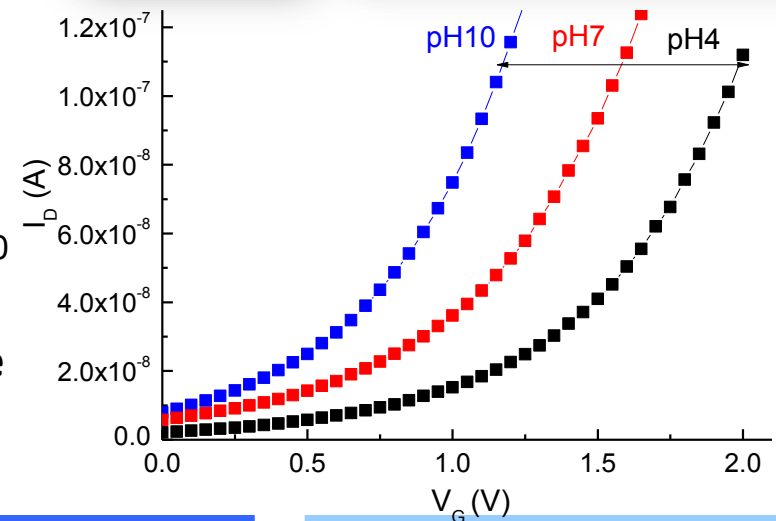
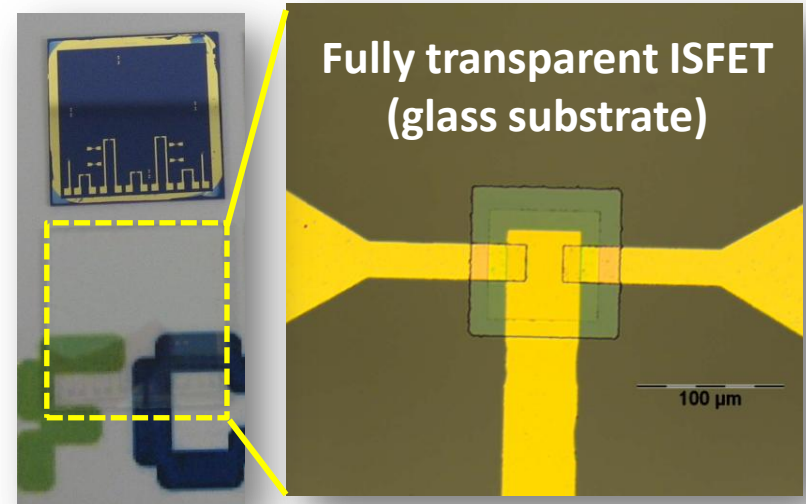


- ◆ Low PenG conc. Sensitivity enhanced 2.5 times as for the pH sensor
- ◆ High PenG conc. Sensitivity also improved

ISFET pH sensor results



Lower sensitivity than EIS structures due to the devices complexity



Conclusions

- We successfully develop by the first time extended gate ISFETs based on GIZO amorphous oxide semiconductors produced at room temperature by rf magnetron sputtering.
- Near Nernstian sensitivity of 57 mV/pH in a pH range of 2 to 12 was obtained for Ta₂O₅ EIS structures deposited with a 14/1 Ar/O₂ sccm ratio, with sensors being stable for at least 3 months.
- A sensitivity of 26 mV/mM to Pen G in a concentration range of 1 to 10 mM was achieved at room temperature for physically adsorbed penicillinase .
- Annealing and surface treatments on the sputtered oxides improve the sensitivity of the dielectric layer.

Thanks to:



PhD scholarship



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Jülich Campus / Biosensors Lab. and Prof. Schöning



For a Luso-British joint project with



THANK YOU FOR YOUR ATTENTION

QUESTIONS?

