Oxygen Diamond Sensors for Extreme Environments

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Objective

This project addresses features of **diamond technology** for the design of a **multifunctional amperometric oxygen sensor** for harsh environmental applications.

Diamond, doped with boron to make it quasi-metallic, has significant chemical and physical durability, and the widest electrochemical potential window of any material. These properties make it ideal for use in difficult conditions. In order to enhance the reduction of oxygen, the Pt NPs are attached - to the diamond electrode which act as catalysts for this reaction. Alternatively, localised graphitic-like regions were employed.

Pt-on-diamond disc microelectrode

Pt-nanoparticle-on-diamond disc microelectrodes were developed from holes in an SU-8 passivation layer on top of the boron-doped diamond covered with Pt.

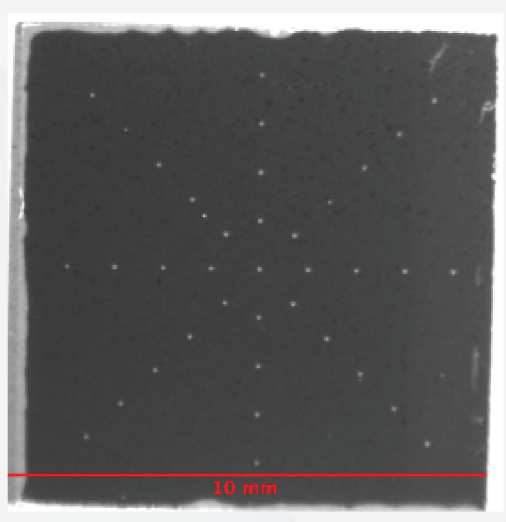

Fig. 1. *An array of Pt-nanoparticle-on-diamond disc microelectrodes[1]*

Potential Step Voltammetry in DI Water

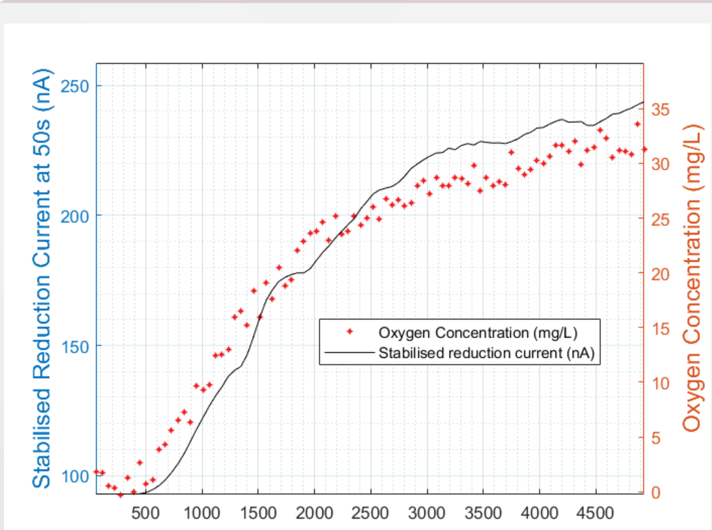

Fig. 2. Data for stabilized reduction current and the data for oxygen concentration, plotted over time. The reduction current is increasing with the increase of O2 concentration in the cell. The current increases from 90 to 240 nA.

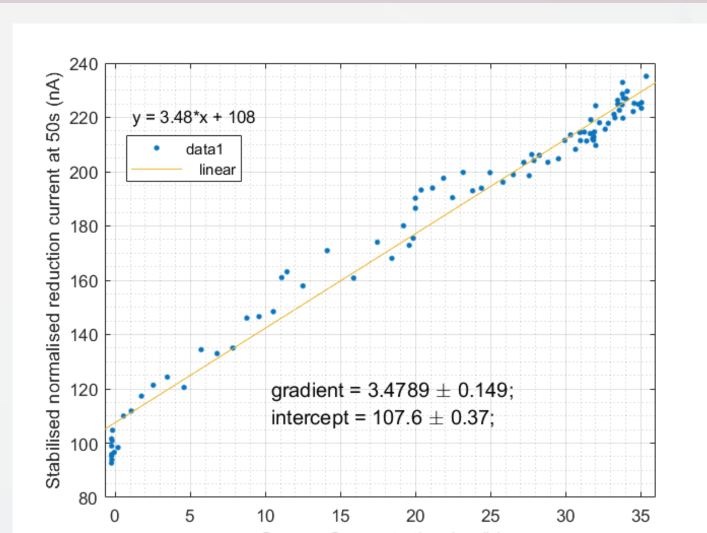

Fig. 3. Calibration Curve for Stabilised Reduction Current at Increasing O2 Concentrations. As it can be observed in the figure , the fit is fairly linear. The intercept can be interpreted as a background current. Its presence causes a rise in the error value at oxygen concentrations lower than 3-4mg/L, where there is a significant divergence from linearity. R-squared is 0.977.

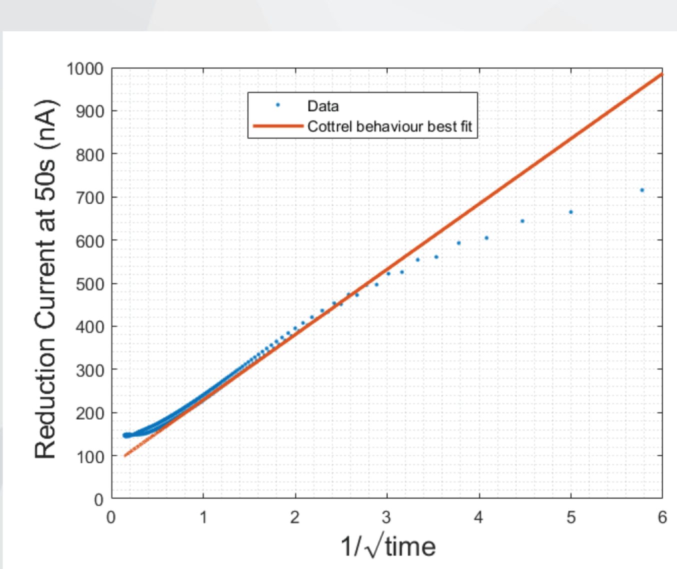

Fig. 4. Cottrel plot. The steady-state current plotted against 1/√*time*, for an oxygen concentration of 35 mg/L. At larger values of x there is a deviation from the Cottrell behaviour. This deviation happens from 1/√*time*=0.2 , which corresponds with 25 s within the cycle. The current deviates to a higher value than predicted by the Cottrel Equation because of the significant edge effect contribution.

Cyclilc Voltammetry

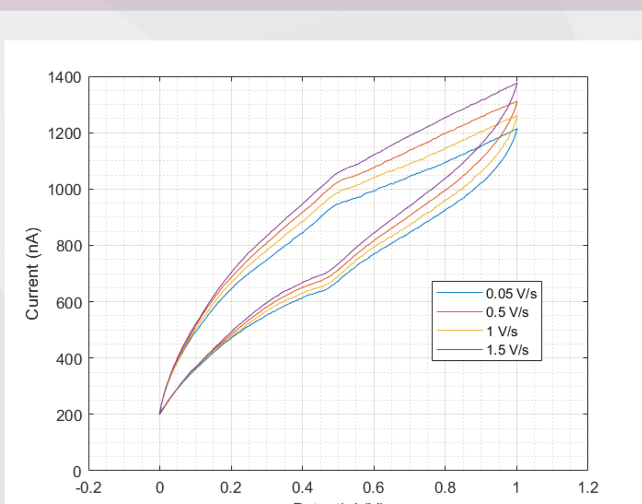

Fig. 5. Cyclic Voltammetry at 0.05V/s, 0.5 V/s, 1V/s, 1.5V/s. The current value is shown to be proportional to the scan rate, as the current increases with the increase of the scan rate.

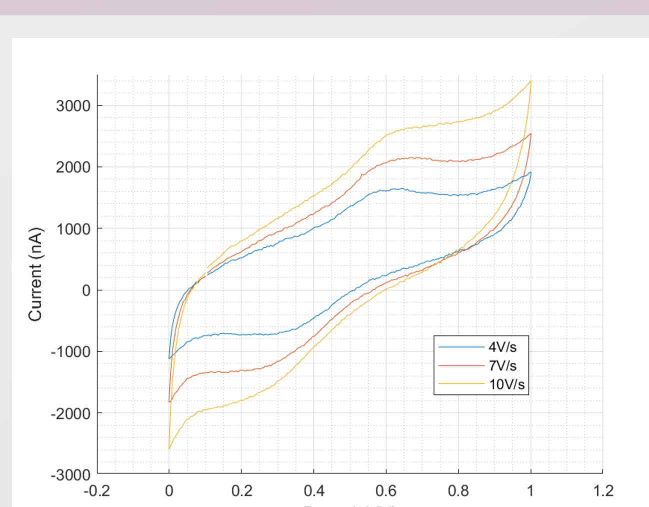

Fig. 6. Cyclic Voltammetry for 4V/s, 7V/s, 10V/ss. Increasing the scan rate restores the reversibility of the voltammogram.

3nm-Pt NPs on BDD

Nanoparticles have been formed through a **gas phase synthesis** process.

This method provides great control over the size of the NPs, as well as **high chemical purity** (organic solvents are avoided).

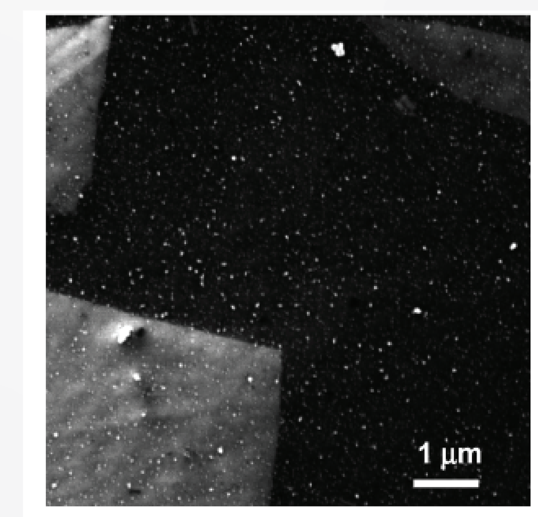

Fig. 7. FE-SEM images of a Pt NP-modified BDD electrode[2]

Potential Step Voltammetry in DI Water

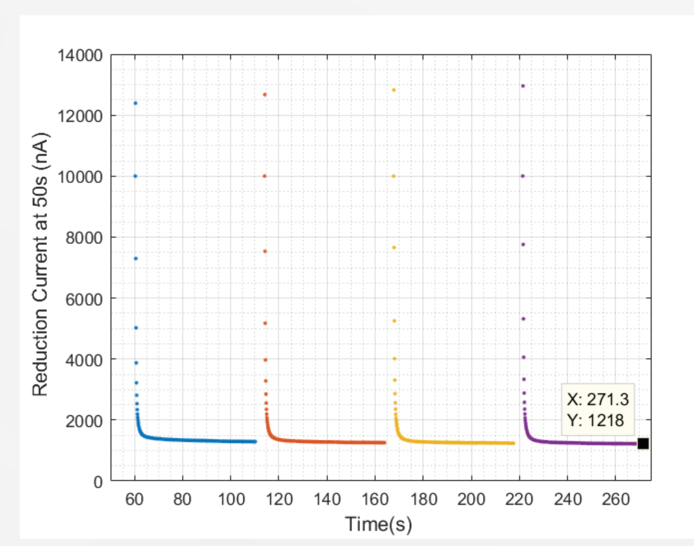

Fig. 8. Long term stability testing, first 4 measurement pulses. Reduction current of 1218 nA.

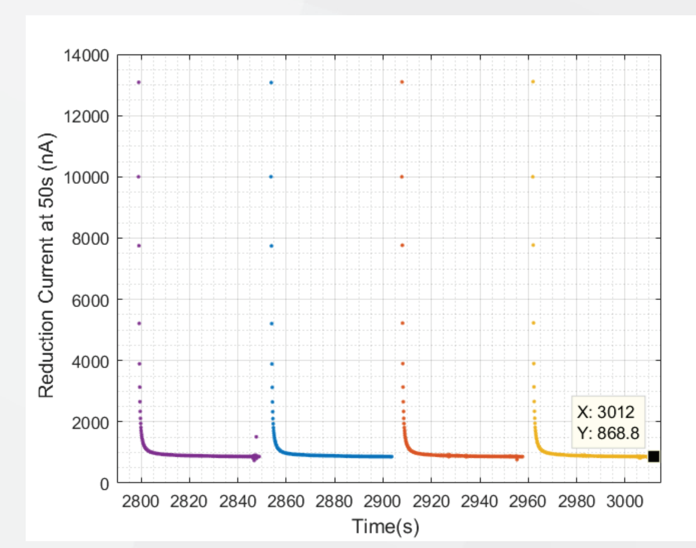

Fig. 9. Long term stability testing, after 50 minutes (same oxygen concentration). The reduction current has decreased in a proportion of 1.4%.

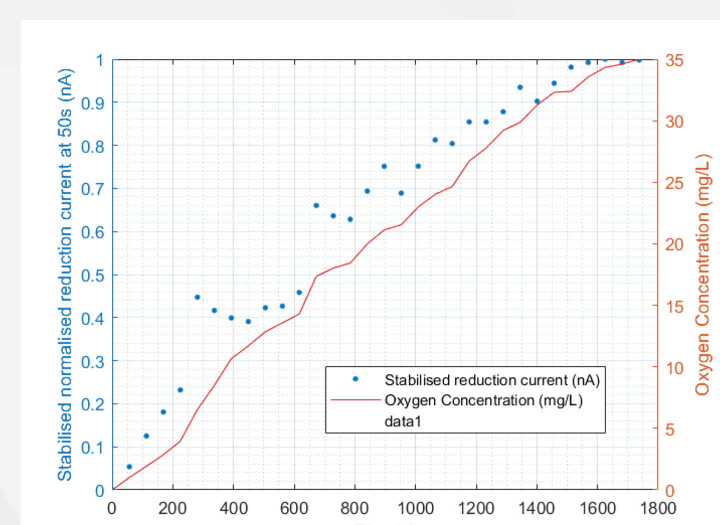

Fig. 10. The reduction current is increasing with the increase of O2 concentration in the cell. Greater errors are shown between 5-10mg/L and 10-15mg/L.

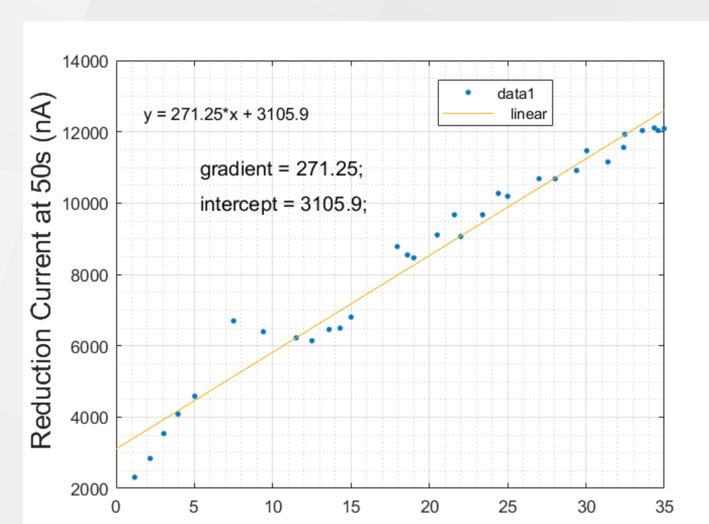

Fig. 11. Fit is less linear than for the microelectrode. Errors are greater at oxygen concentrations lower that 4mg/L. R-Squared is 0.967.

Graphitic microchannel array electrode (GMC)

A cylindrical region of intrinsic diamond was laser processed, with the surface showing **graphitic material** as a result of the cutting process.

The microchannels were written into the sample through **laser graphitisation** by Oxford Lasers. Metal contacts were added onto the electrode and annealed to create a **stable ohmic connection**.

The **sp2 regions** promote the electrocatalytic reduction of oxygen.

Potential Step Voltammetry in DI Water

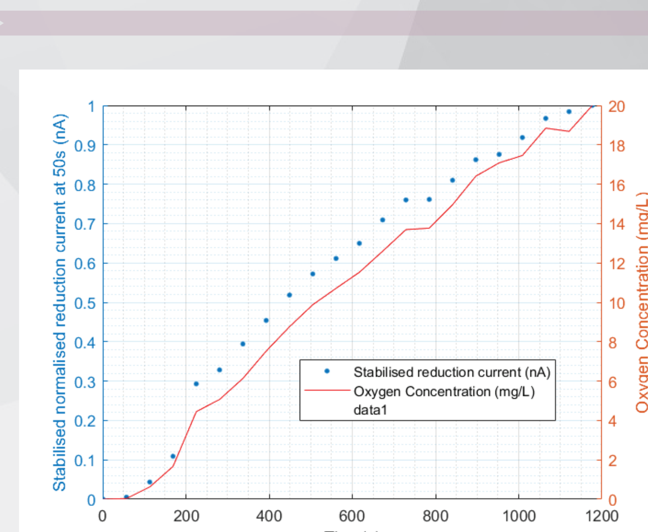

Fig. 12. The reduction current is increasing with the increase of O2 concentration in the cell. Greater errors are shown only below 2mg/L, which is a great improvement from last sample.

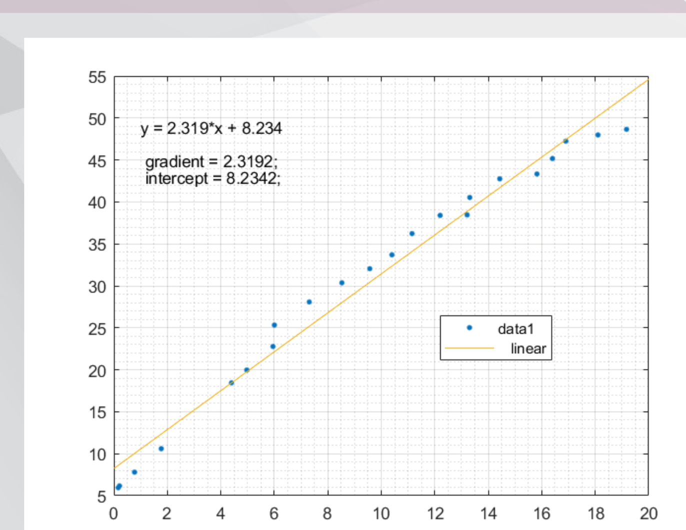

Fig. 13. The fit shows a strong correlation of reduction current on dissolved oxygen concentration. The starting reduction current for 0mg/L is of 5.9 nA. R-squarred is 0.991.

References

1. Moors, R. J. Diamond and sp2 carbon for green energy applications (2019).
2. Hutton, Laura, et al. Amperometric Oxygen Sensor Based on a Platinum Nanoparticle-Modified Polycrystalline Boron Doped Diamond Disk Electrode (2007).

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