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Analysis report

Prepared by

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Results presented below were recorded for untreated BDD plate. Please note that the performance may be significantly improved when measuring chemically etched surfaces.

1. Surface morphology

1.1 Optical microscope analysis

Optical microscope pictures presented in Figure 1 shows that side A has higher amount of impurities which is expected to influence significantly the electrochemical properties (see electrochemical analysis later in this report).

a) b)

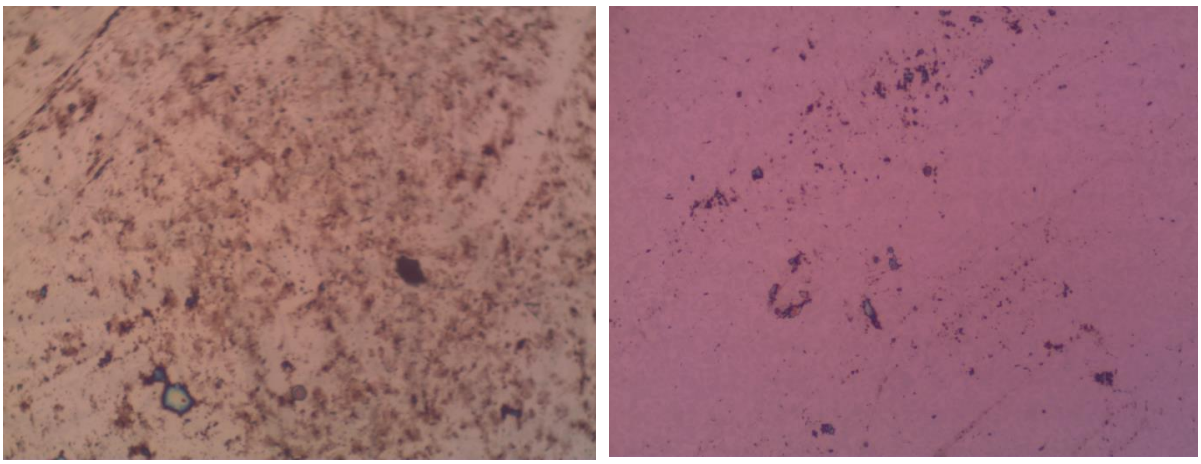

Figure 1. Pictures from optical microscope, magnitude 40x of a) side A and b) side B

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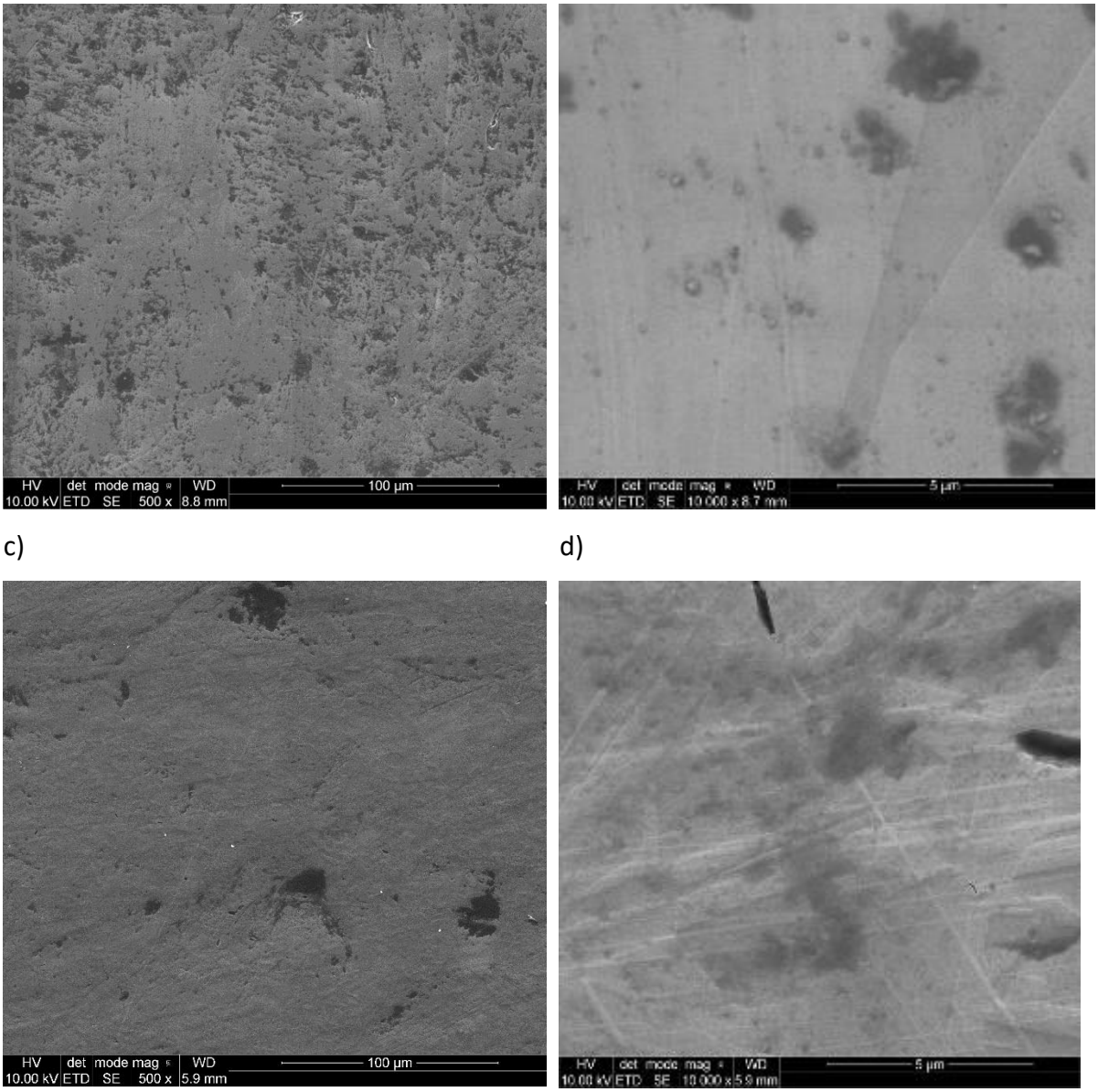
1.2 Scanning Electron Microscope analysis

SEM micrographs shown in Figure 2 confirms different morphology on side A and B, due to the processing of as-grown polycrystal (polishing and laser cutting).

a) b)



c) d)

Figure 2. SEM pictures of polished BDD surfaces of a, b) side A and c, d) side B

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2. Raman Spectroscopy

Raman spectroscopy revealed sp^3 carbon structure (σ bonds) which is shown in Raman spectrum (Figure 3 and 4) as a single diamond zone center optical phonon peak occurring at 1330 cm^{-1}. The line width is a qualitative measure of the crystalline quality of the film. The more defects, the shorter the phonon lifetime and the broader the line width. Other Visible bands correspond to boron doping at 548, 910, 1032 and 1211 cm^{-1}. The band located at 2464 cm^{-1} is attributed to BH isotopes. Estimated boron doping in side A is $1.409*10^{20}$ cm^3.

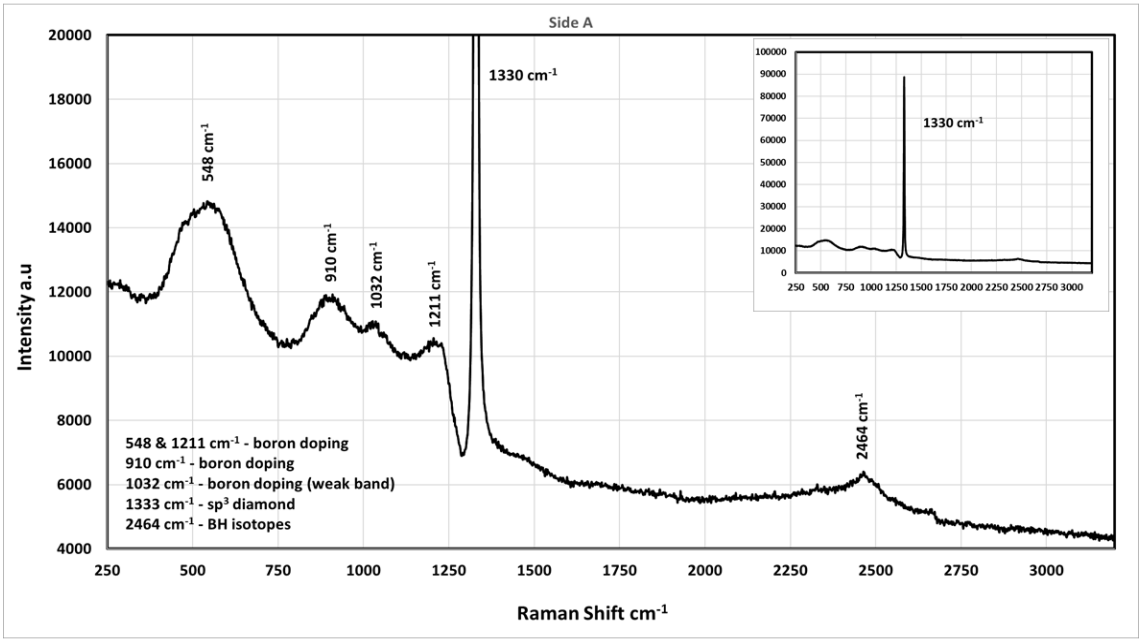

Figure 3. Raman spectra of side A with intensive diamond peak located at 1330 cm^{-1} (see inset) due to lower boron doping

Side B has a lot weaker sp^3 diamond peak and shifted to lover wavenumber values to 1321 cm^{-1} which corresponds to higher doping. Moreover, the Fano effect is visible (non-symmetric diamond peak). There are two strong bands attributed to boron doping located at 492 and

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1216 cm^{-1}, also a weak band at 995 cm^{-1} and BH isotopes band at 2429 cm^{-1} are visible. Estimated boron doping on side B is $4.45859*10^{20}$ cm^3

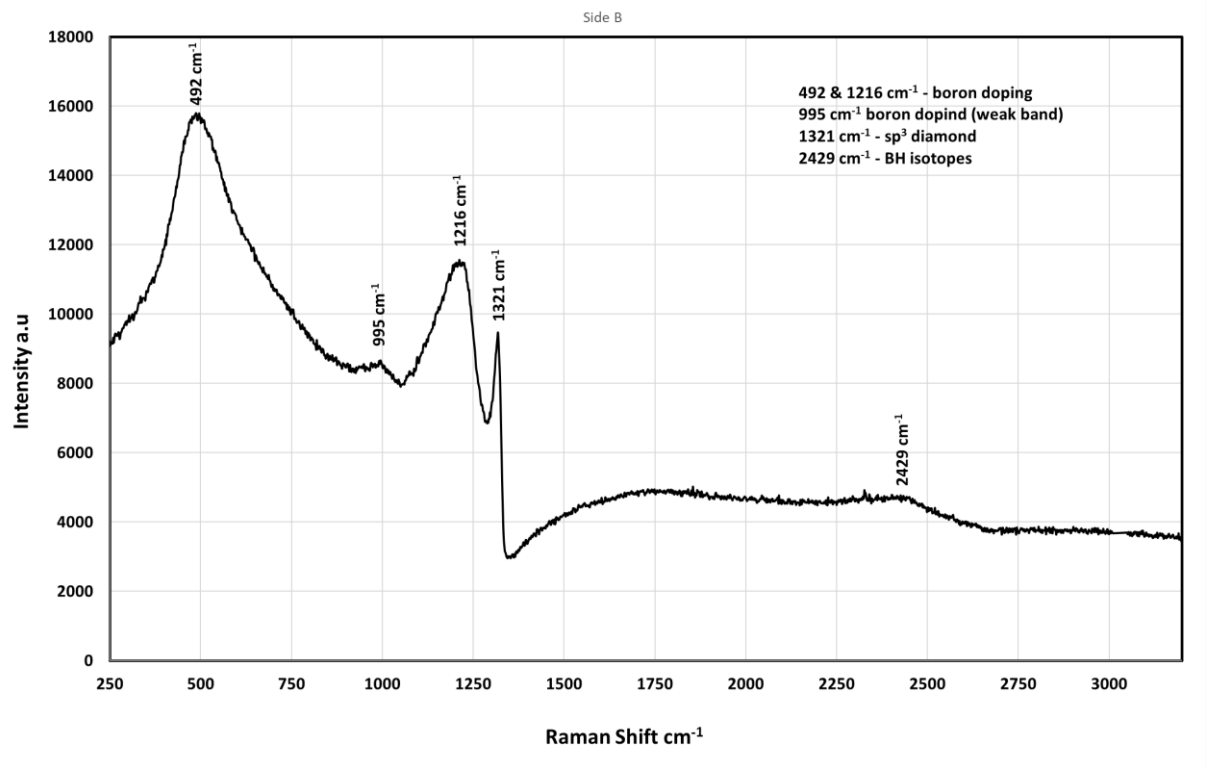

Figure 4. Raman spectra of side B

3. Electrical measurements

The resistivity of BDD plate was measured using Four Point Probe. The resistivity values corrected for thickness are:

Side A - 15.42 ohm·cm

Side B - 9.3 ohm·cm

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4. Electrochemical evaluation

4.1 Cyclic voltammetry

The solvent window defines the potential range over which an electrode can operate before the solvent is electrolyzed. Curves of electrochemical working potential window measured on both sides (Figure 5) show some capacitive effect attributed to non-uniform surface morphology. The potential window (ΔE) is around 1.6 - 1.7 V. However, this value may be significantly improved by the chemical etching of the surface before measurements.

a) b)

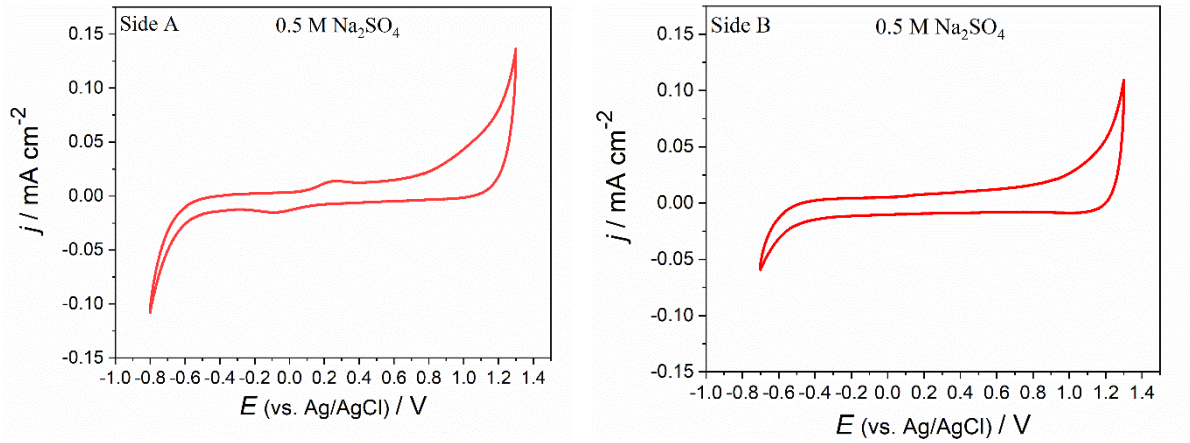

Figure 5. Curves of electrochemical working potential window for a) Side A and b) side B

Measurements of peak to peak separation for redox reaction (Figure 6) show decent reversibility in case of $K_3[FE(CN)_6]$ and very broad peak separation for $[Ru(NH_3)_6]Cl_3$.

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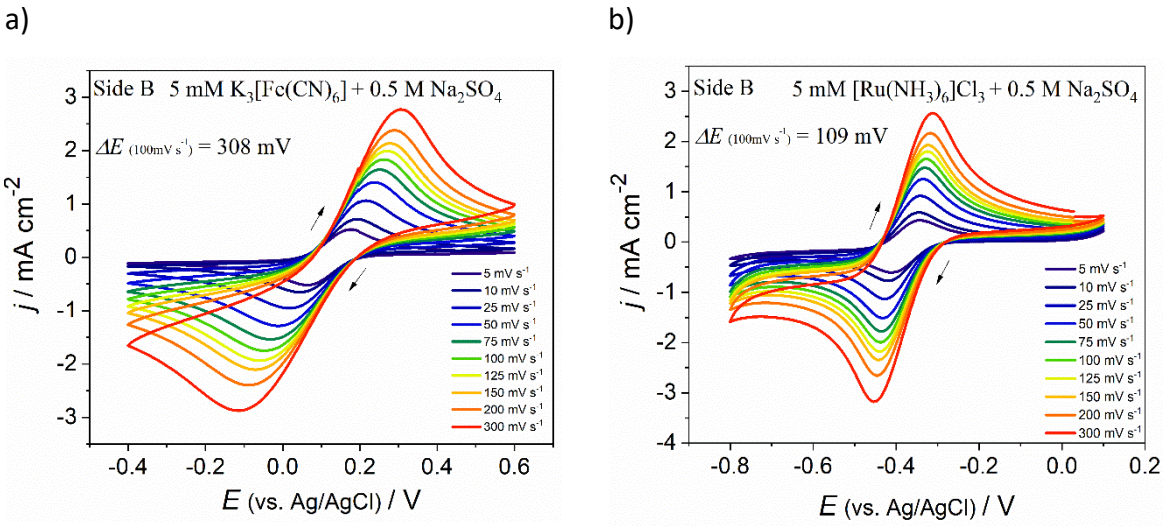

Figure 6. Reversibility of the redox reaction process for side A

Reversibility of the redox reaction process for side B is slightly better (Figure 7), but generally both sides seem to be suitable for electroanalysis.

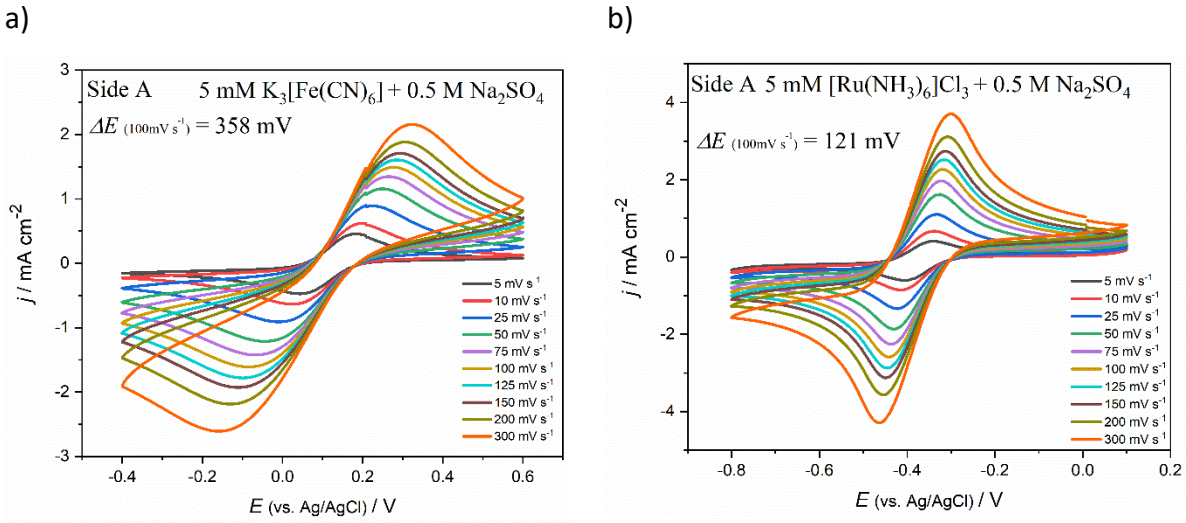

Figure 7. Reversibility of the redox reaction process for side B

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4.2 Electrochemical Impedance Spectroscopy

Nyquist plot shown in Figure 8 exhibits mainly diffused control of the reaction.

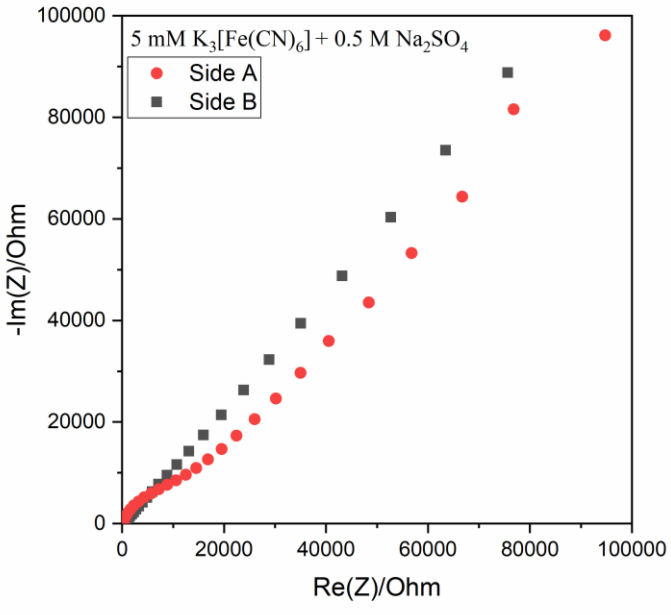

Figure 8 Nyquist plot of side A in 5mM K3[Fe(CN)6] + 0.5 Na_2SO_4