



University of  
BRISTOL

School of Chemistry

Growth of Diamond-Coated  
Nanostructures for Electrochemical  
and Bactericidal Applications:

Appendix

Michael Clegg

17/3/16

Supervisor: Professor Paul May

Second Assessor: Professor David Fermin

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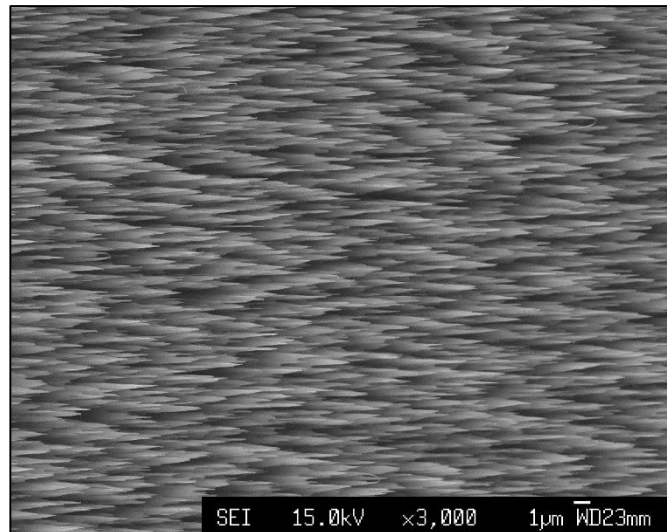
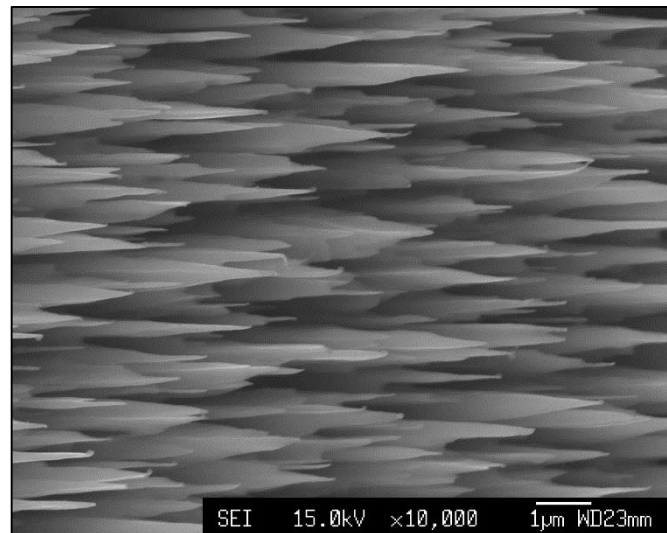
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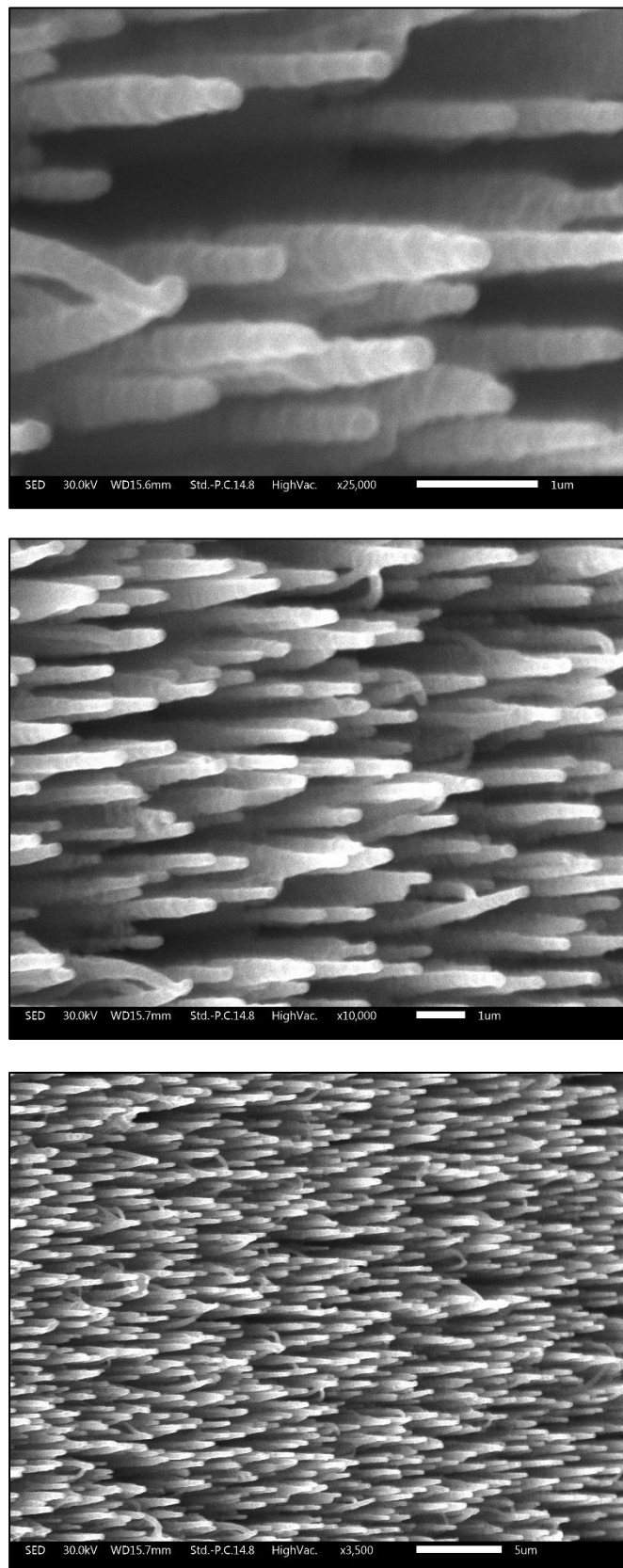
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# Scanning Electron Microscopy

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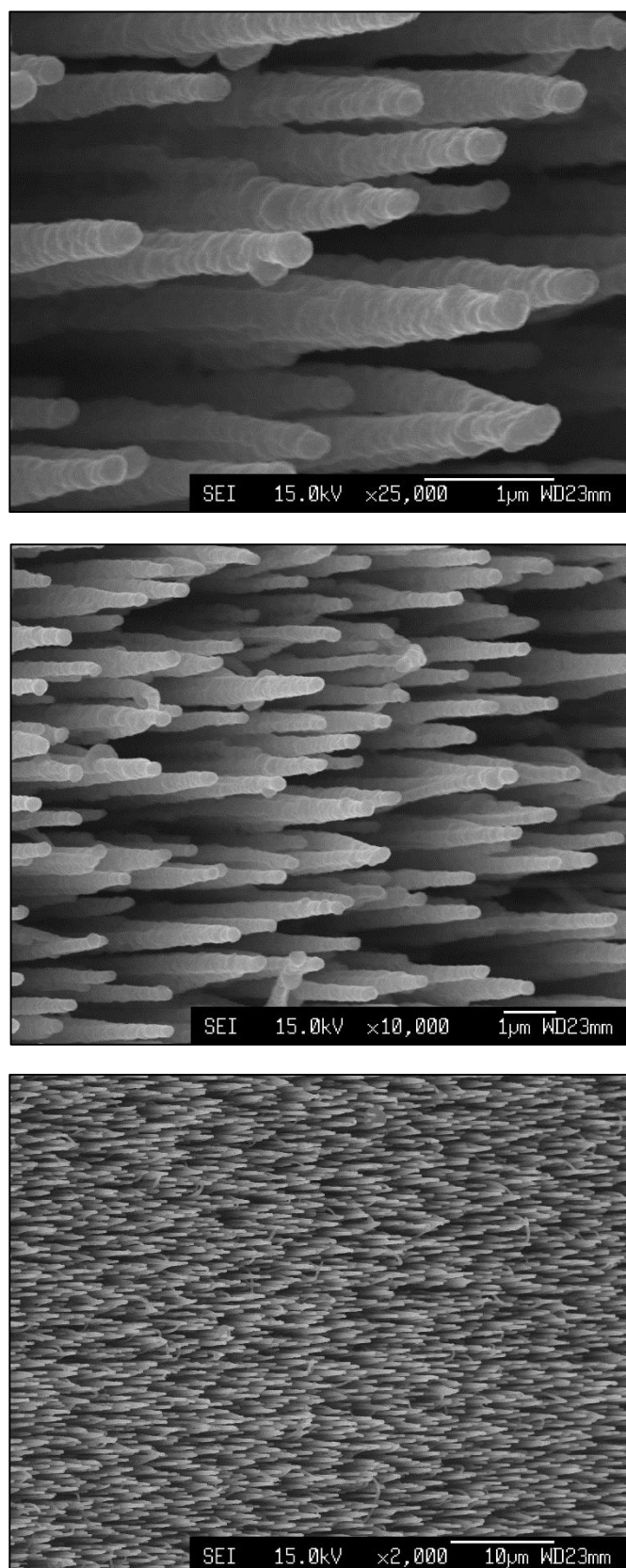


**Figure 1:** Scanning electron microscopy (SEM) images for uncoated large black silicon needles.

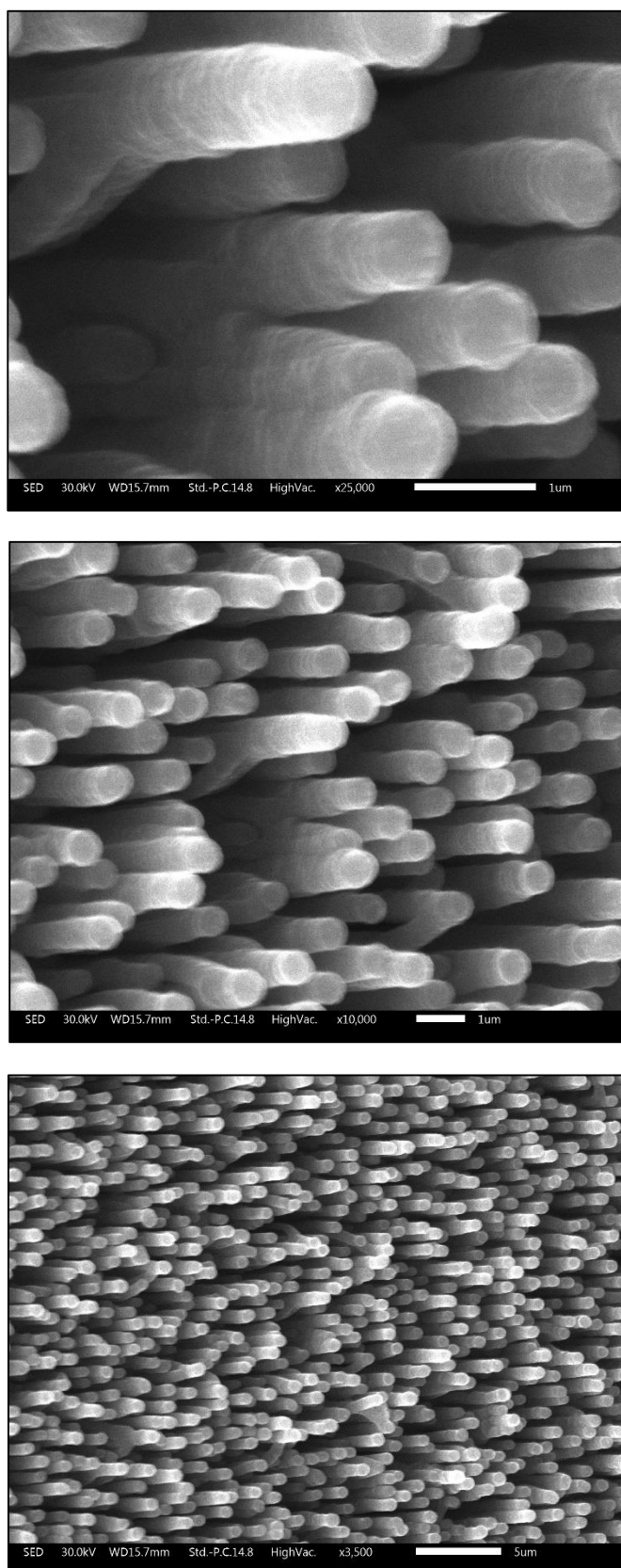


**Figure 2:** SEM images for large black silicon needles coated in a microcrystalline diamond (MCD) film grown for 20 mins after electrospray pretreatment.

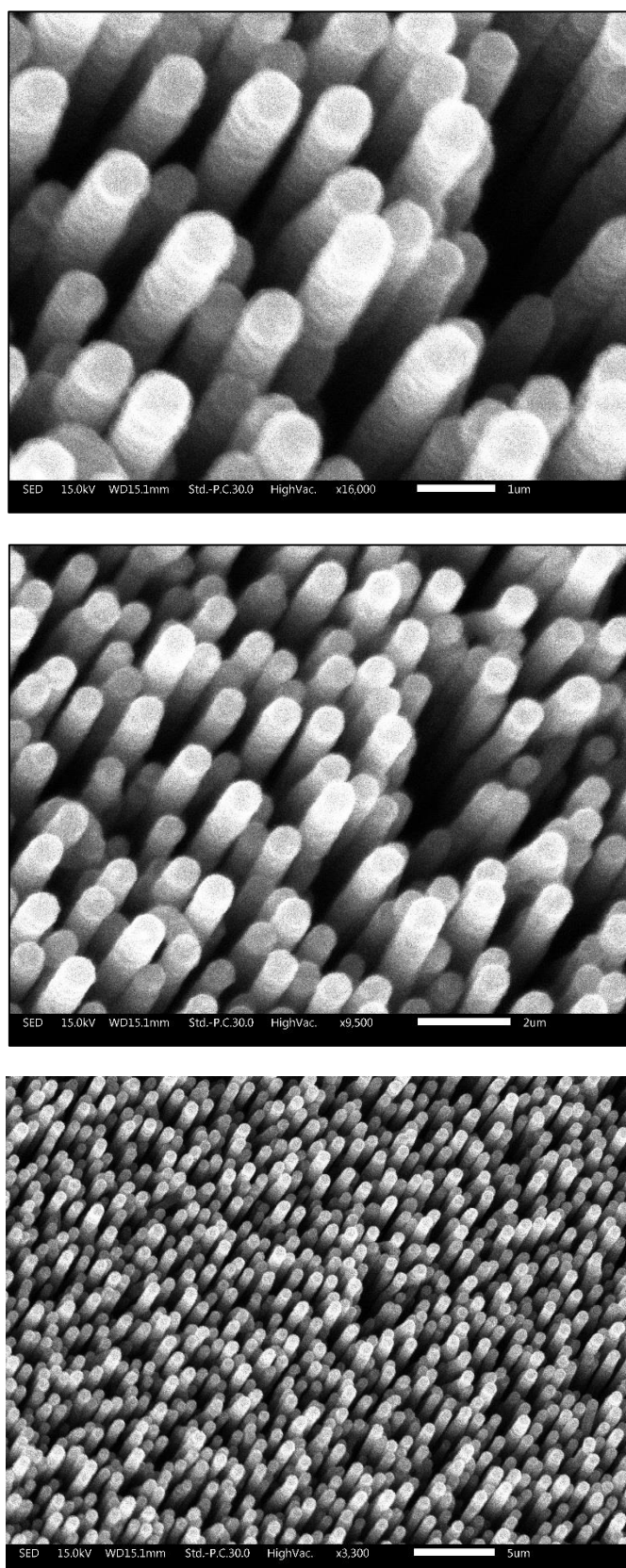




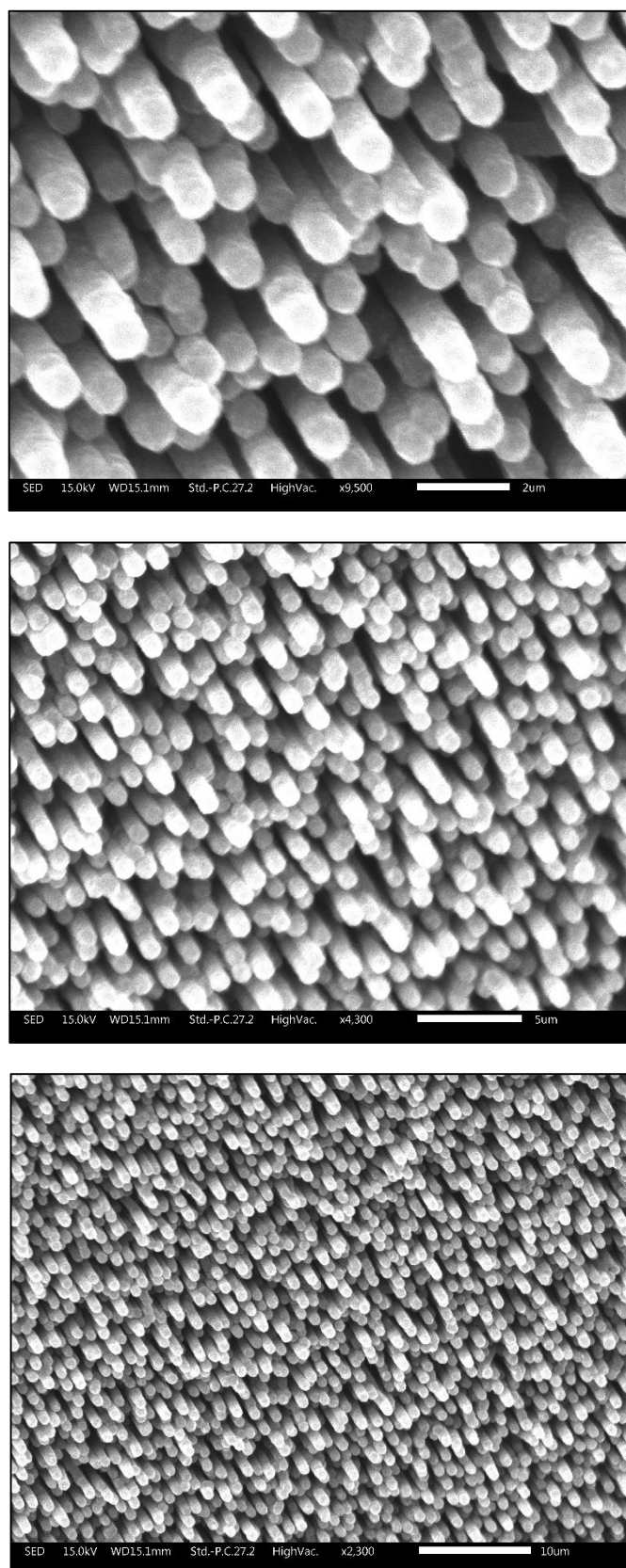
**Figure 3:** SEM images for large black silicon needles coated in a MCD film grown for 30 mins after electrospray pretreatment.



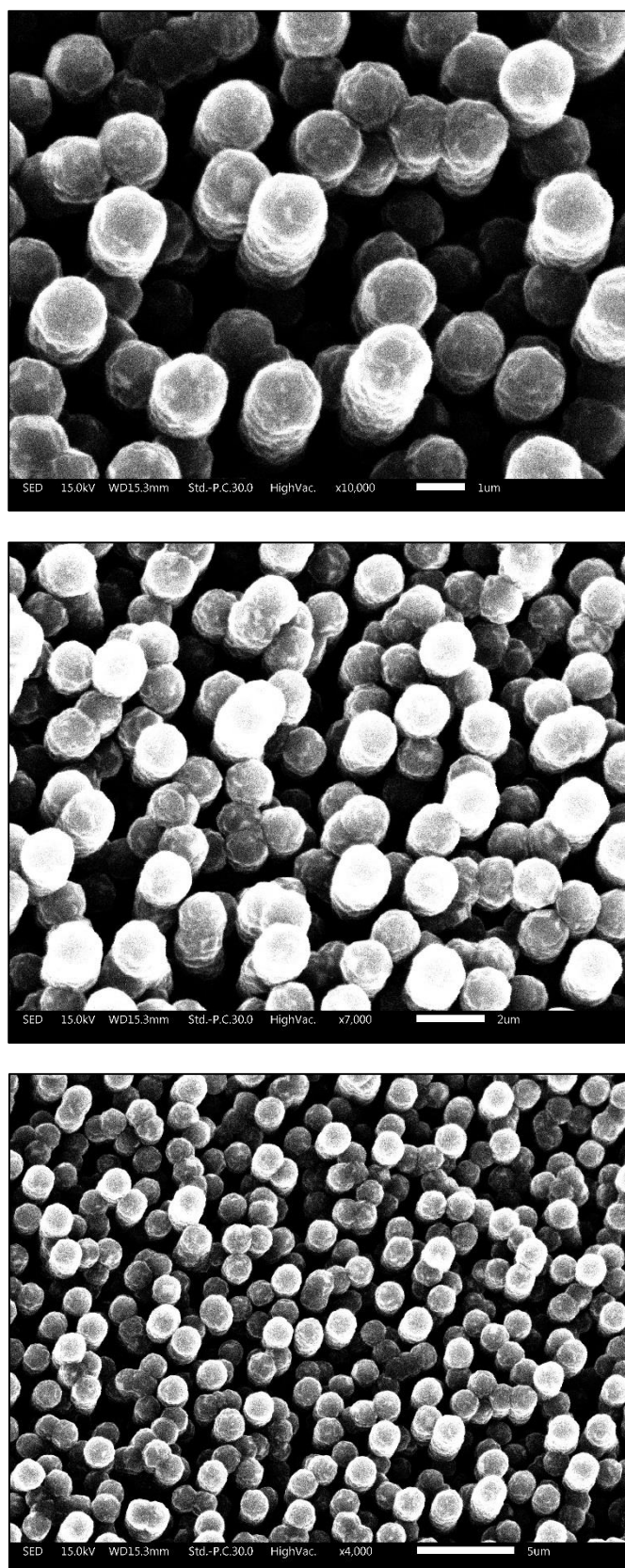
**Figure 4:** SEM images for large black silicon needles coated in a MCD film grown for 45 mins after electrospray pretreatment.



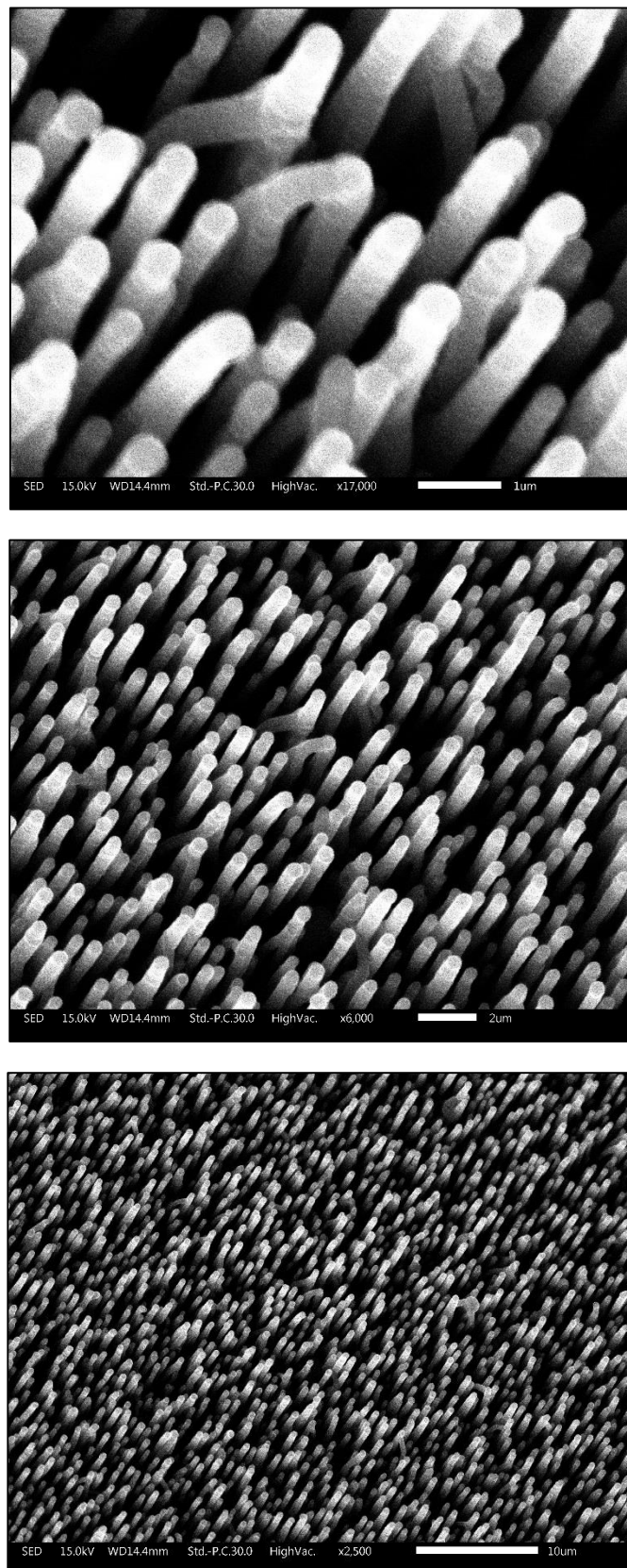
**Figure 5:** SEM images for large black silicon needles coated in a MCD film grown for 1 hour after electrospray pretreatment.



**Figure 6:** SEM images for large black silicon needles coated in a MCD film grown for 1 hour and 30 mins after electrospray pretreatment.

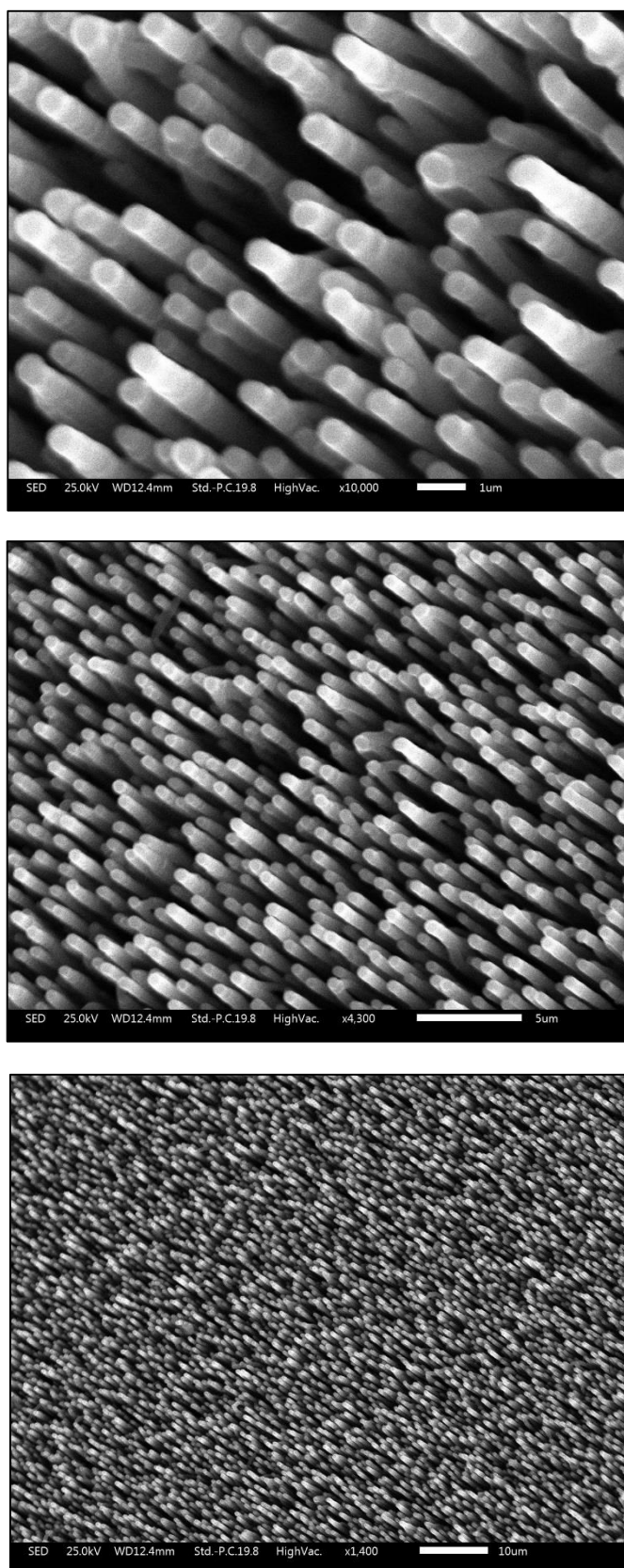


**Figure 7:** SEM images for large black silicon needles coated in a MCD film grown for 2 hours after electrospray pretreatment.

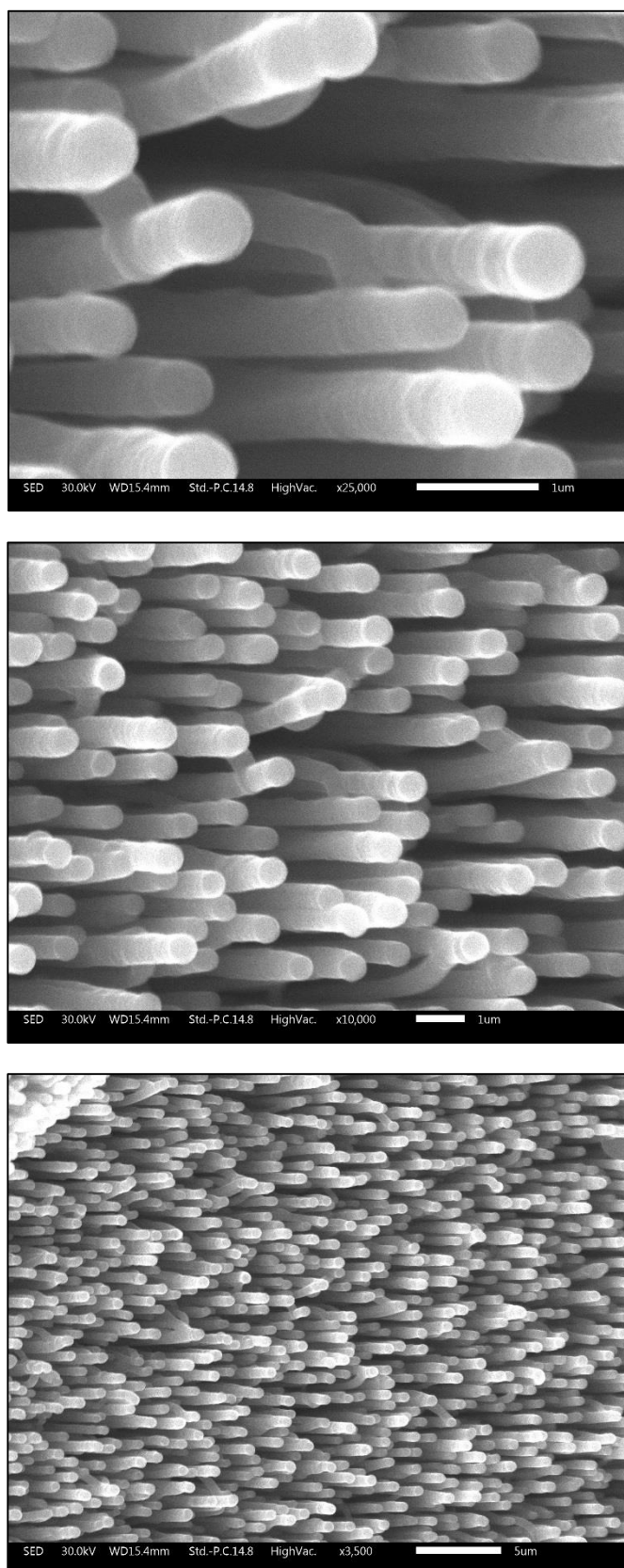


**Figure 8:** SEM images for large black silicon needles coated in a nanocrystalline diamond (NCD) film grown for 15 mins after electrospray pretreatment.



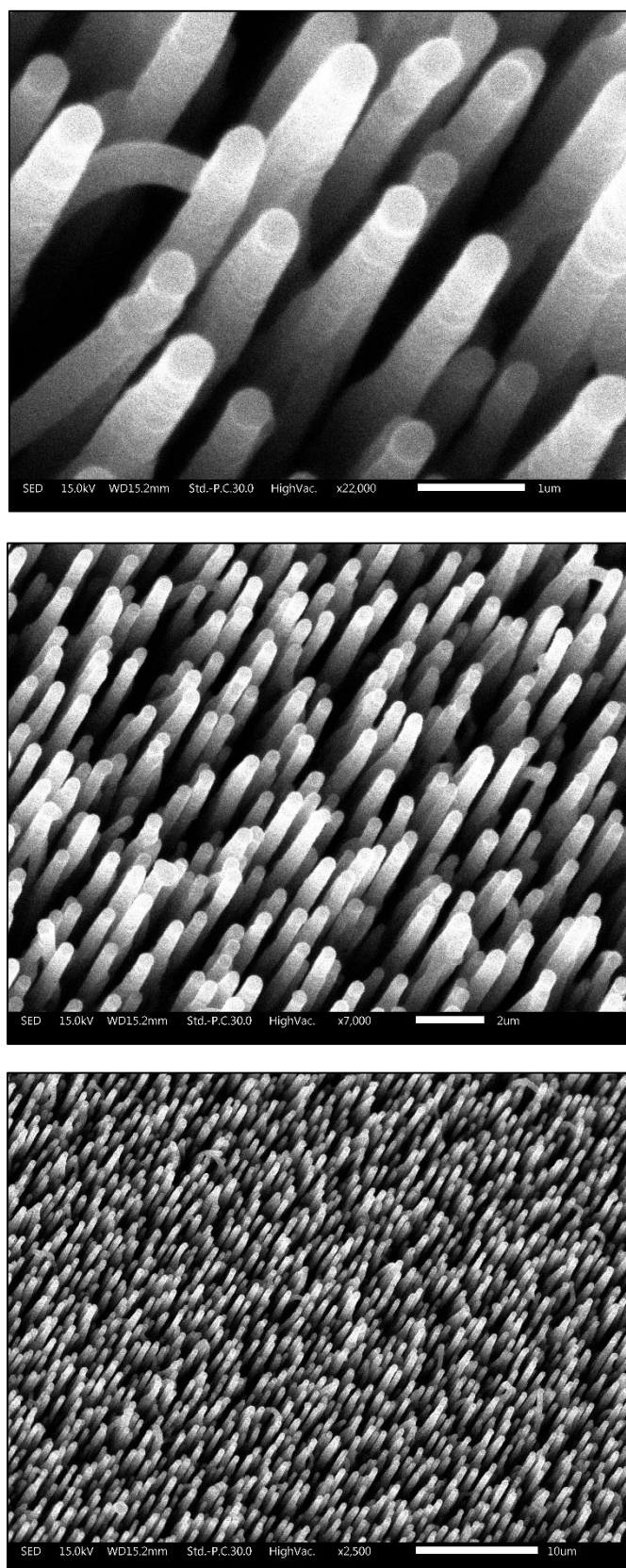


**Figure 9:** SEM images for large black silicon needles coated in a NCD film grown for 20 mins after electrospray pretreatment.

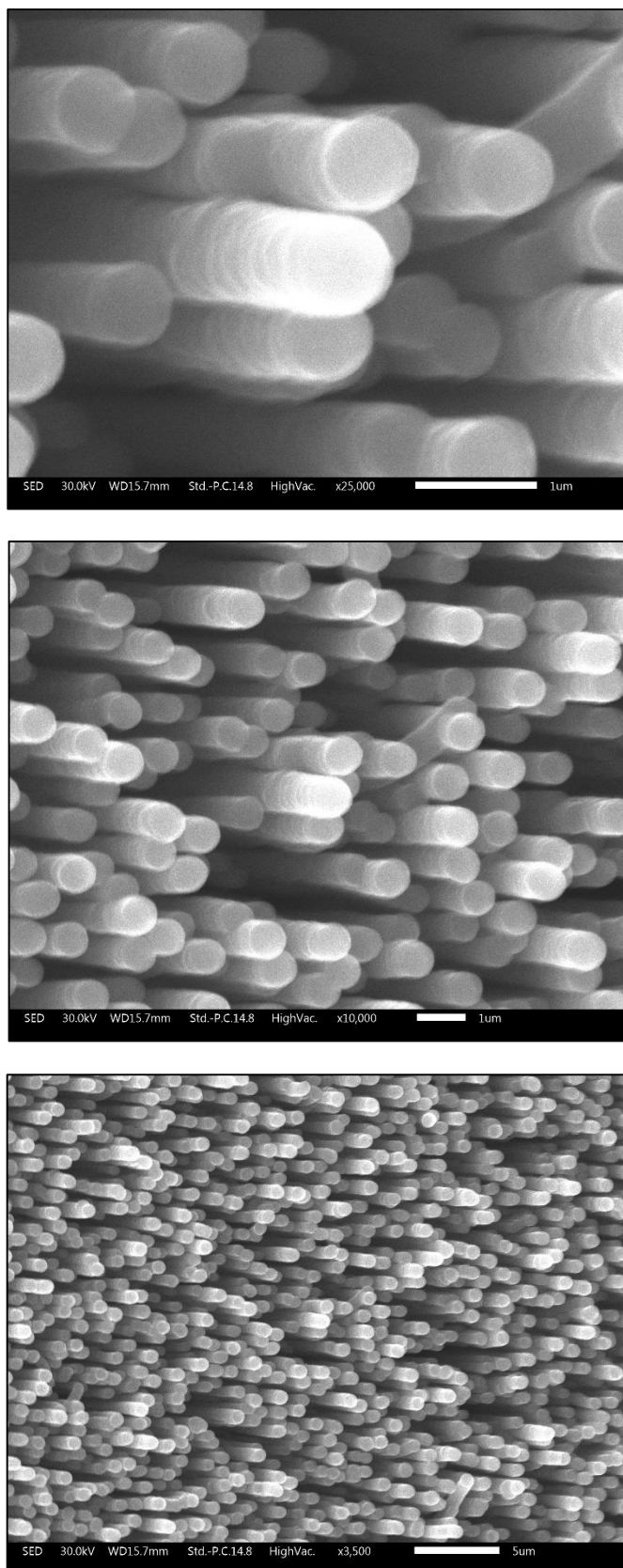


**Figure 10:** SEM images for large black silicon needles coated in a NCD film grown for 30 mins after electrospray pretreatment.

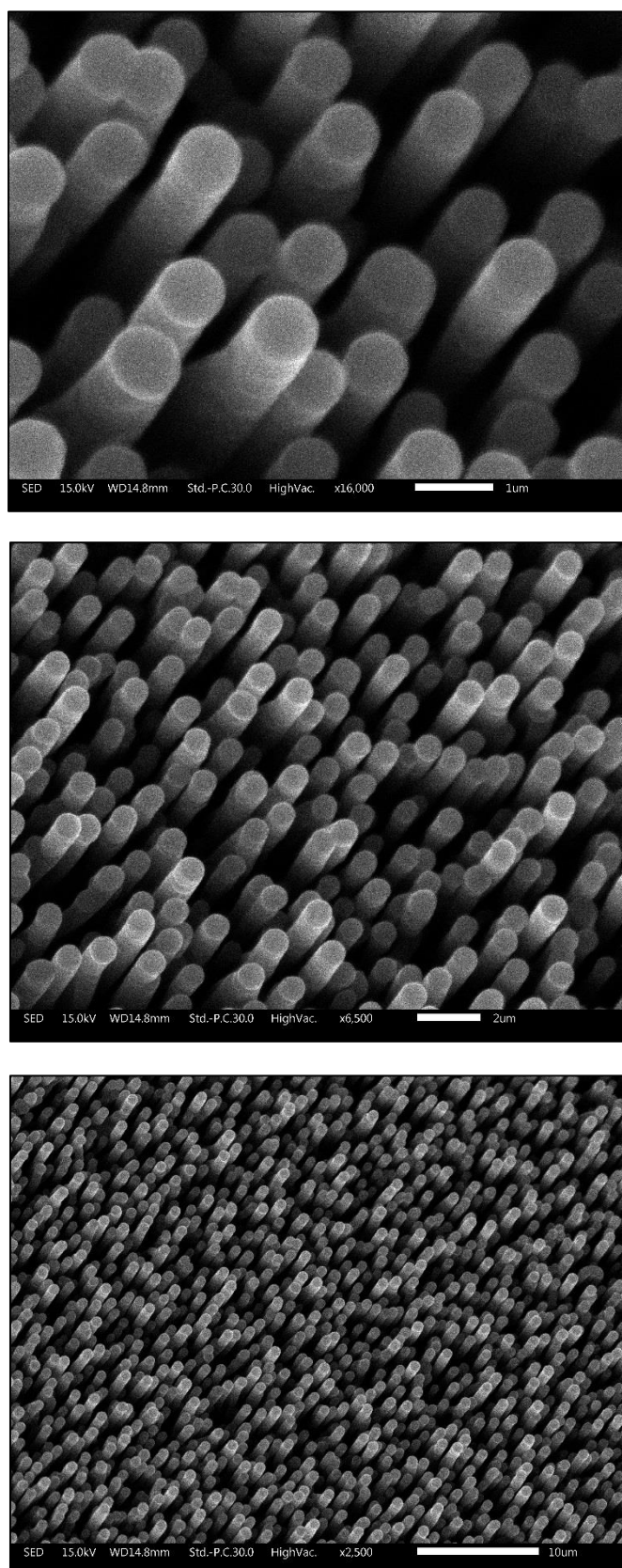




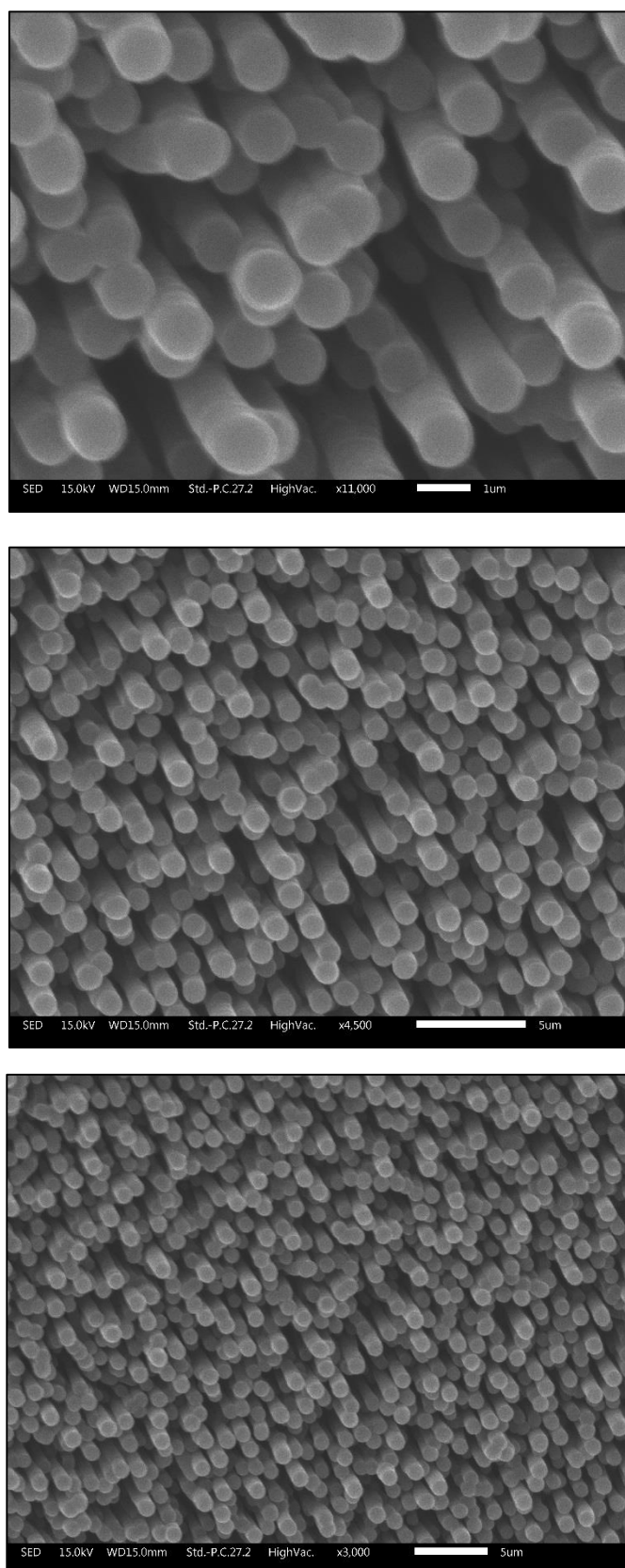
**Figure 11:** SEM images for large black silicon needles coated in a NCD film grown for 40 mins after electrospray pretreatment.



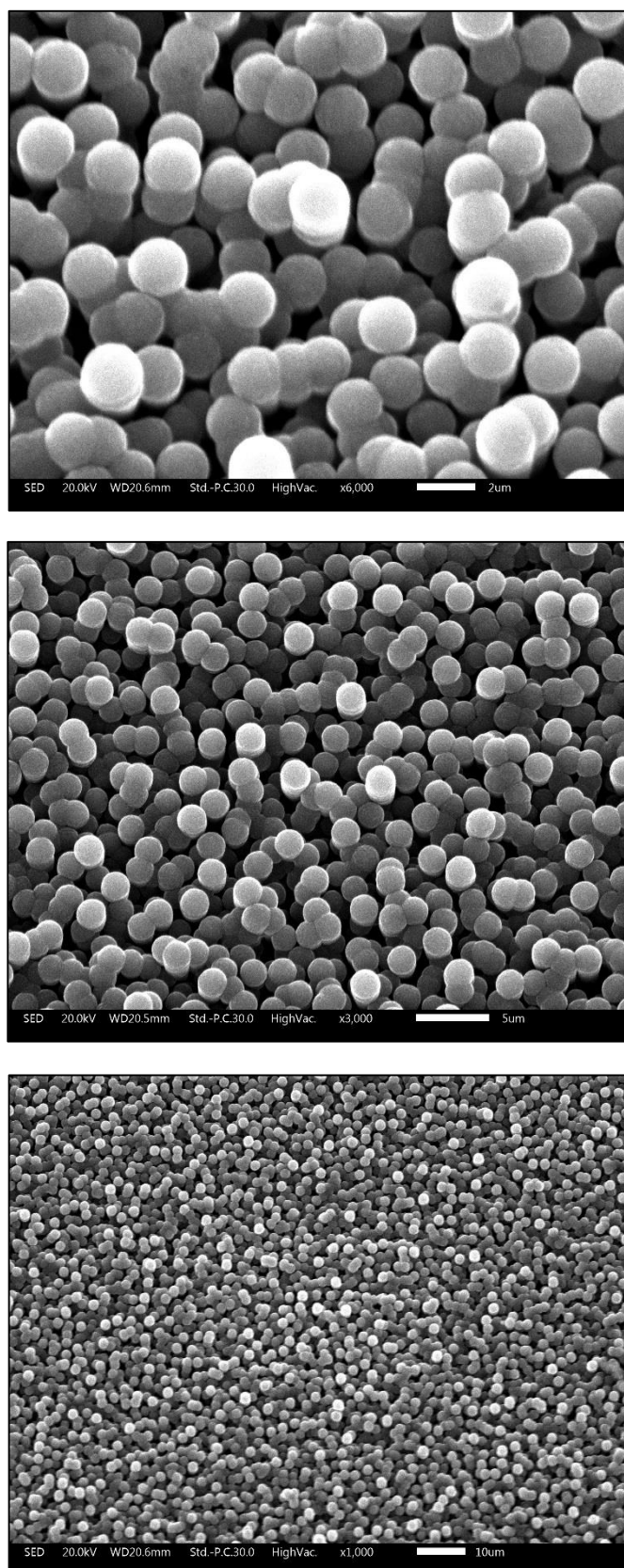
**Figure 12:** SEM images for large black silicon needles coated in a NCD film grown for 1 hour after electrospray pretreatment.



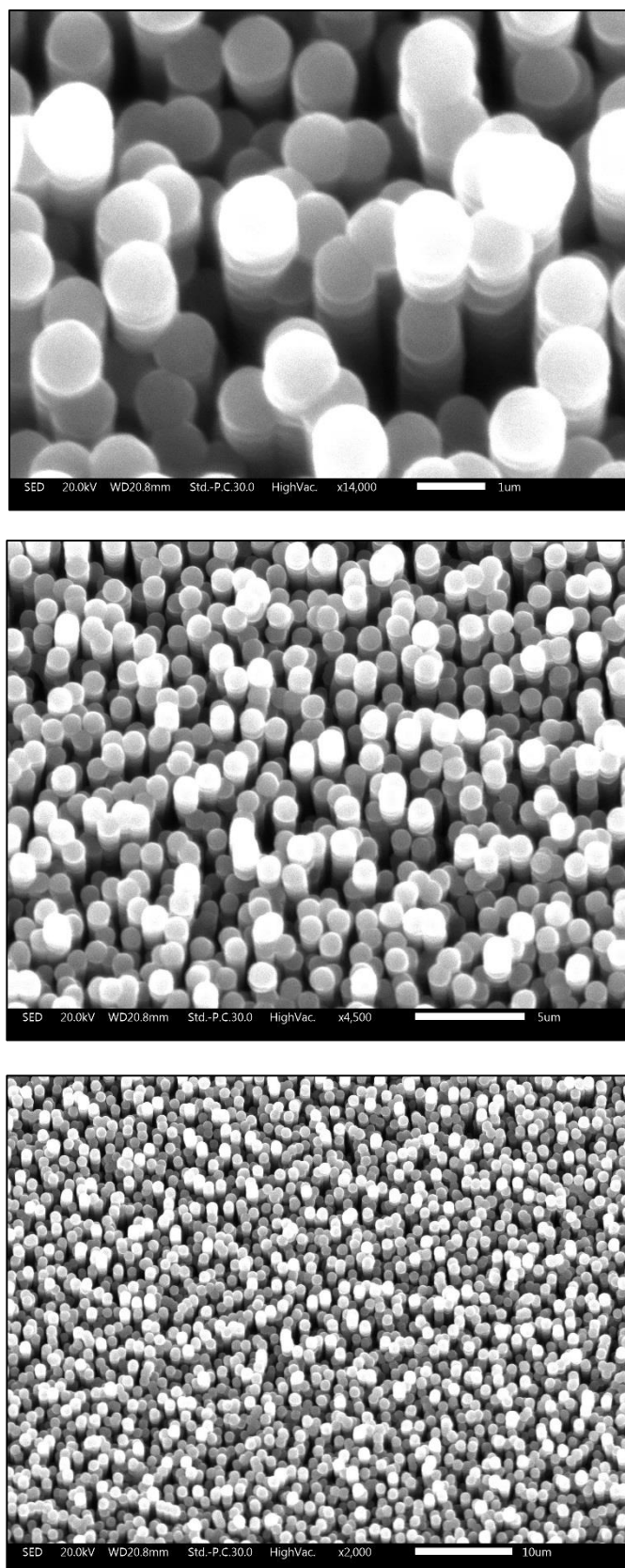
**Figure 13:** SEM images for large black silicon needles coated in a NCD film grown for 1 hour and 20 mins after electrospray pretreatment.



**Figure 14:** SEM images for large black silicon needles coated in a NCD film grown for 2 hours after electrospray pretreatment.

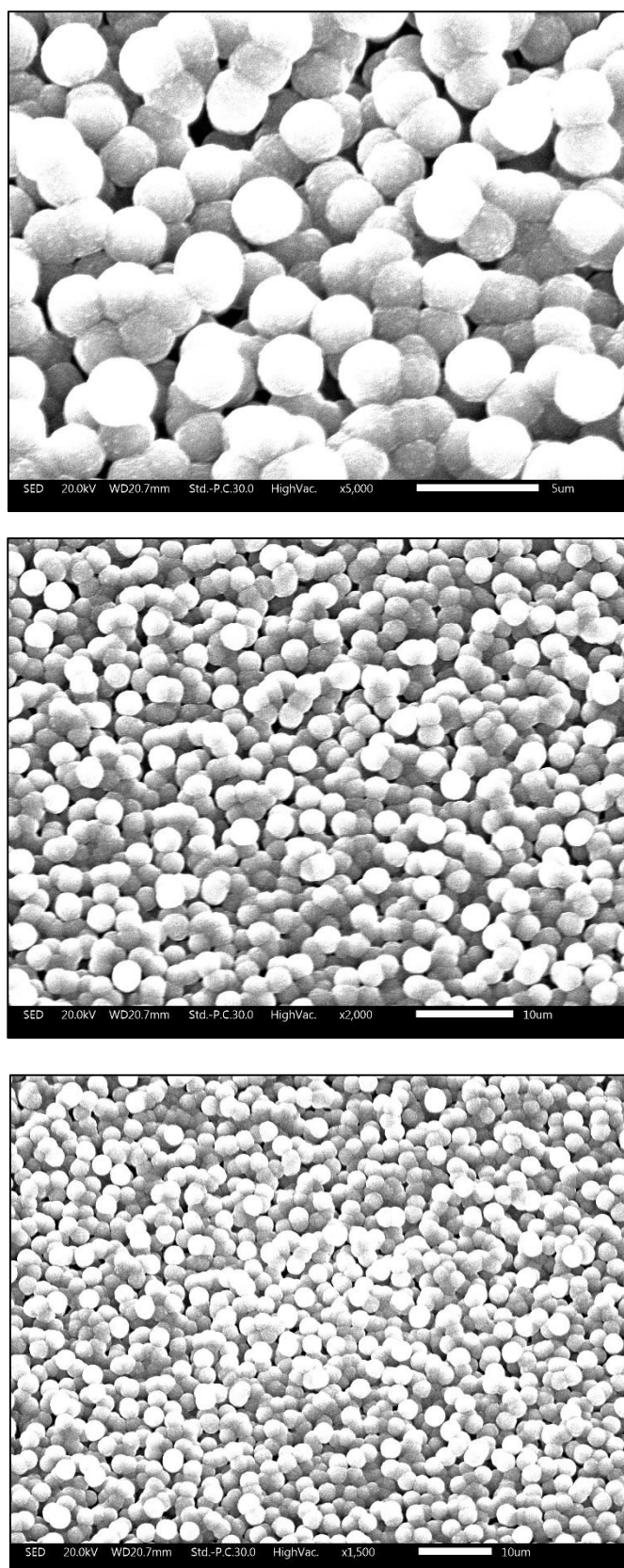


**Figure 15:** SEM images for large black silicon needles coated in a NCD film grown for 3 hours after electrospray pretreatment.

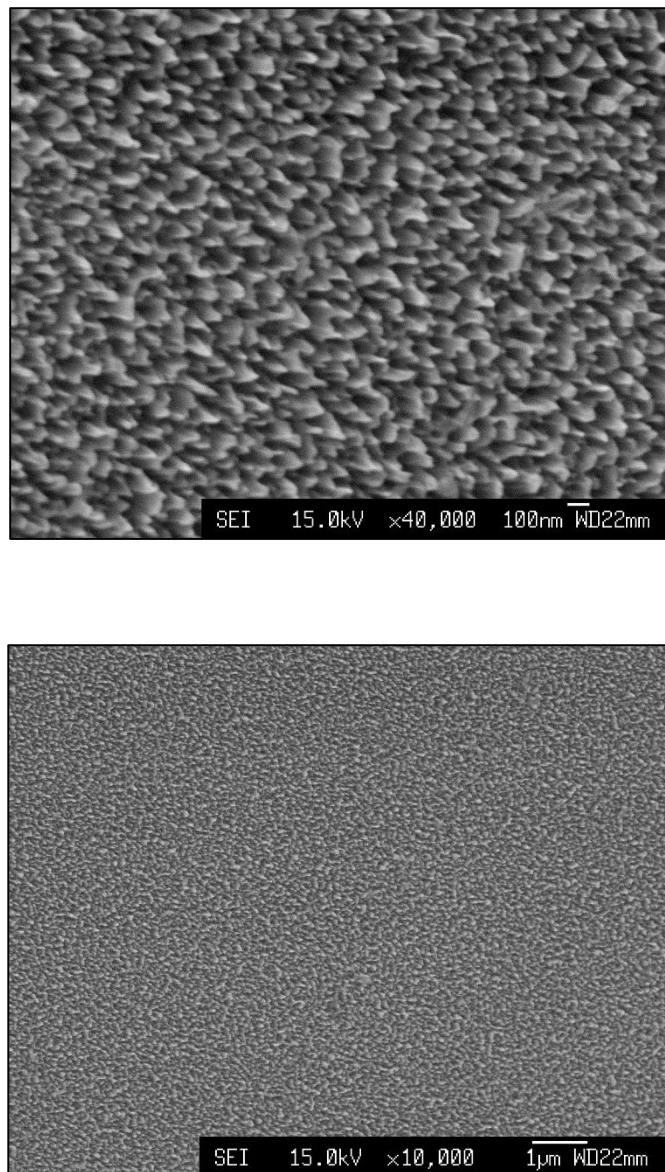


**Figure 16:** SEM images for large black silicon needles coated in a NCD film grown for 3 hours and 30 mins after electrospray pretreatment.





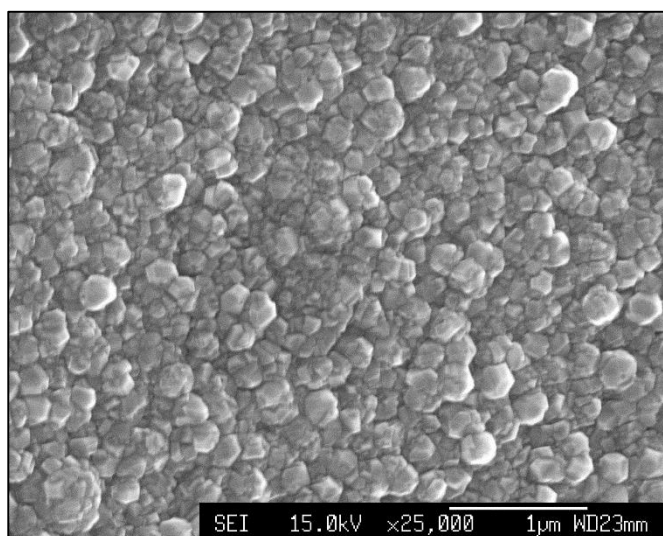
**Figure 17:** SEM images for large black silicon needles coated in a NCD film grown for 4 hours after electrospray pretreatment.



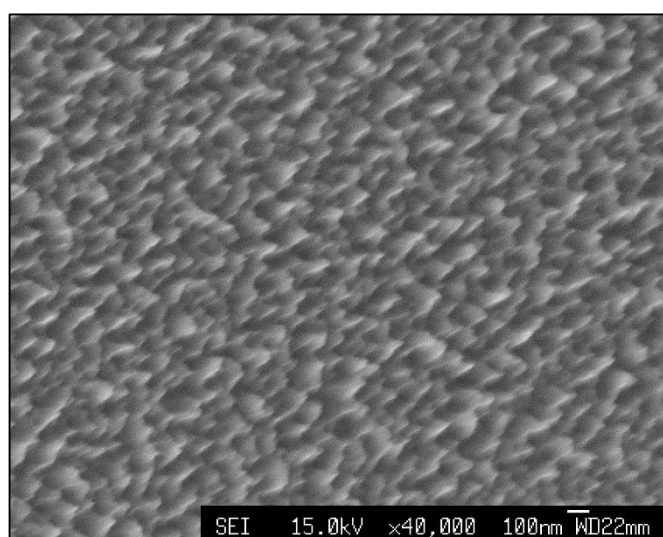
**Figure 18:** SEM images for uncoated small black silicon needles.



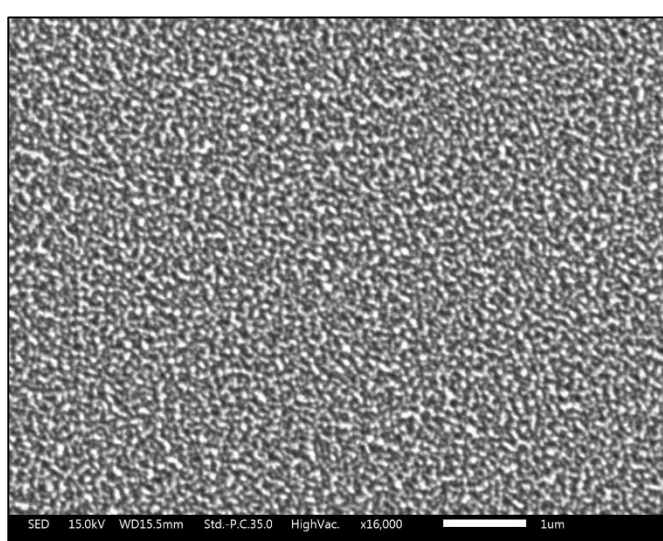
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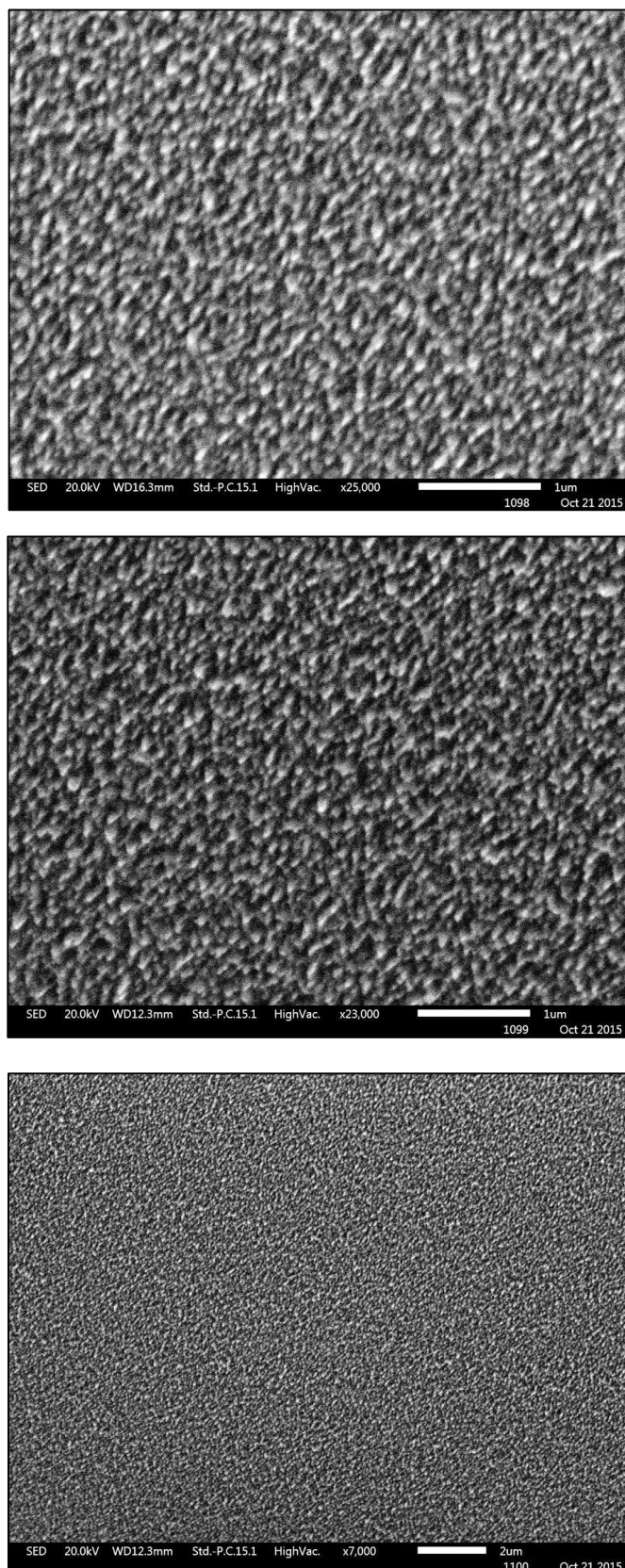
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c)

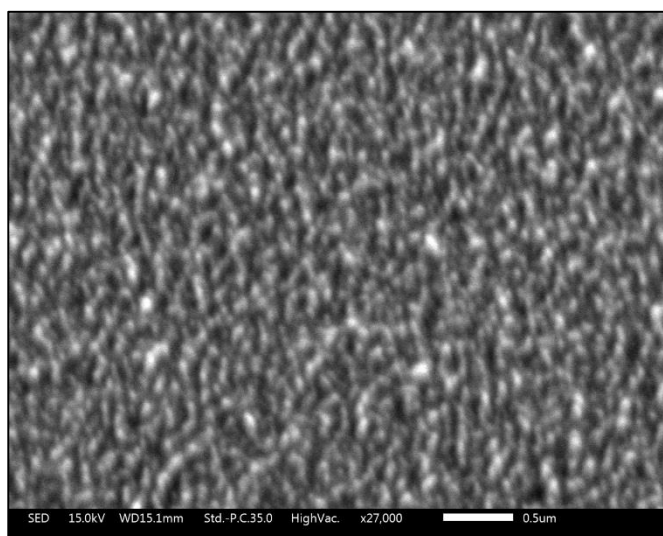


**Figure 19:** SEM images for small black silicon needles coated in a MCD film grown for a) 30 mins after electrospray pretreatment, b) 30 mins and c) 45 mins.

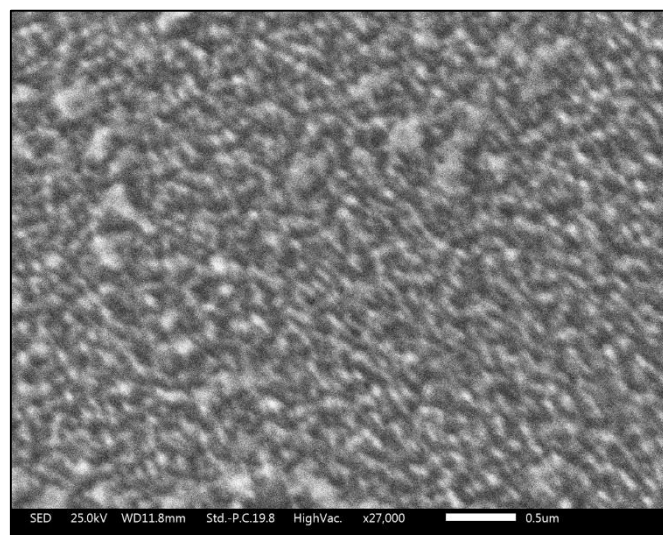


**Figure 20:** SEM images for small black silicon needles coated in a MCD film grown for 1 hour.

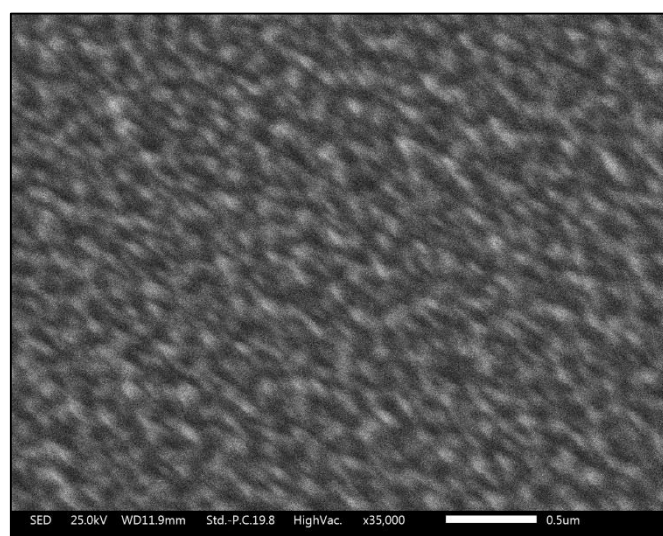
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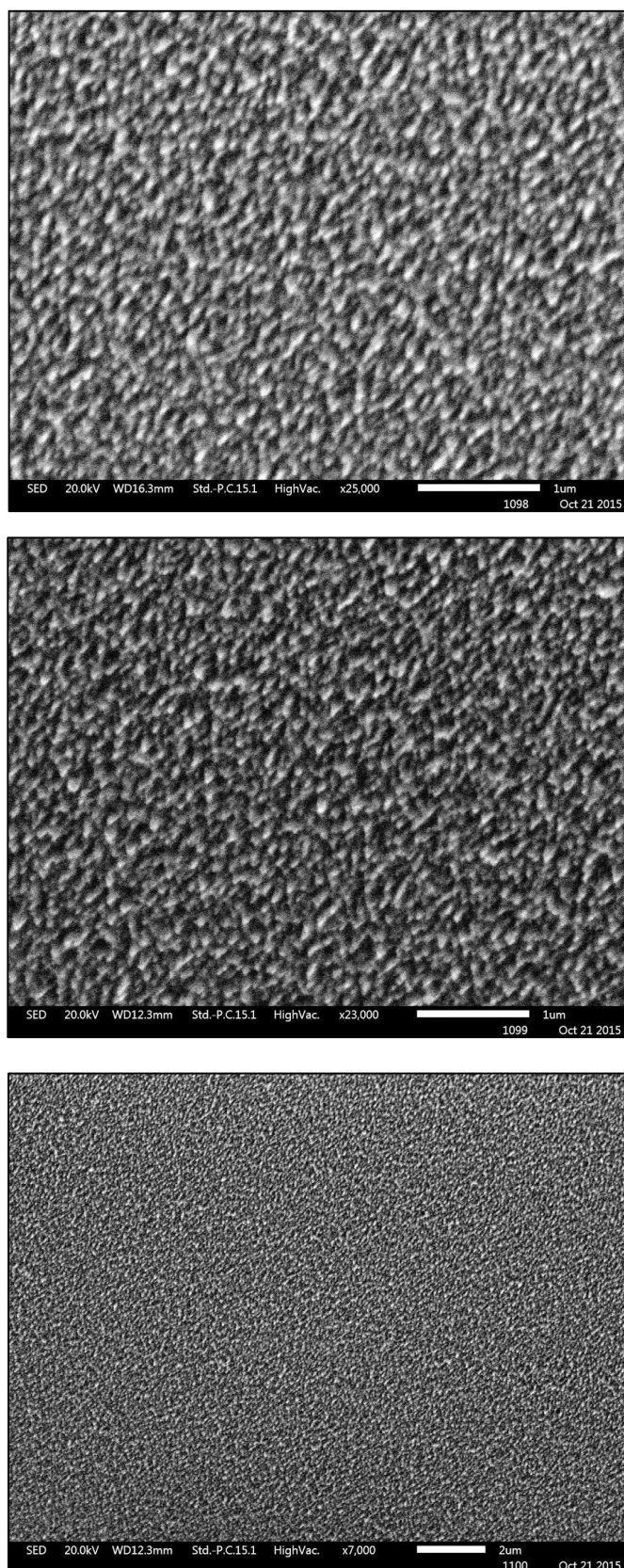
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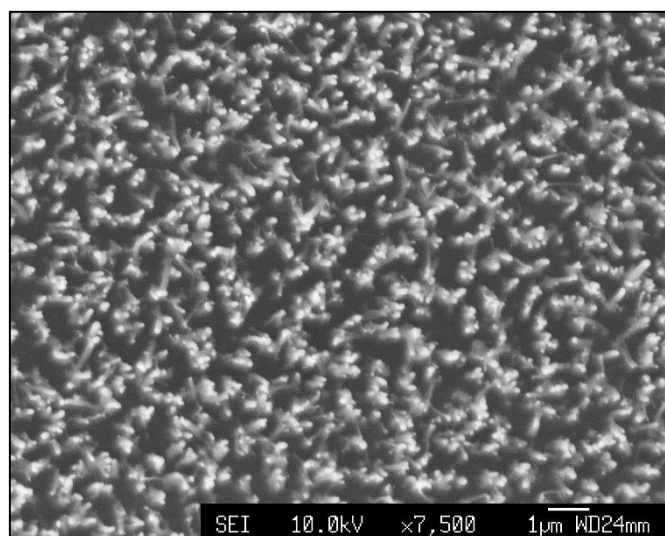
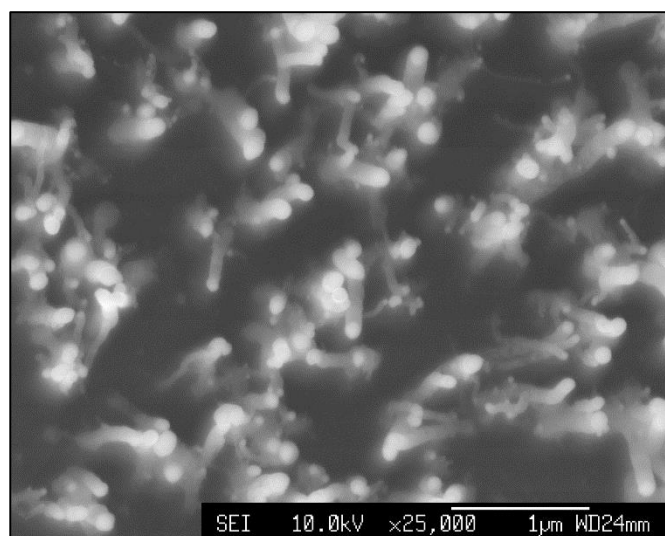
c)



**Figure 21:** SEM images for small black silicon needles coated in a MCD film grown for a) 1 hour 30 mins, b) 2 hours, and c) NCD film grown for 1 hour.

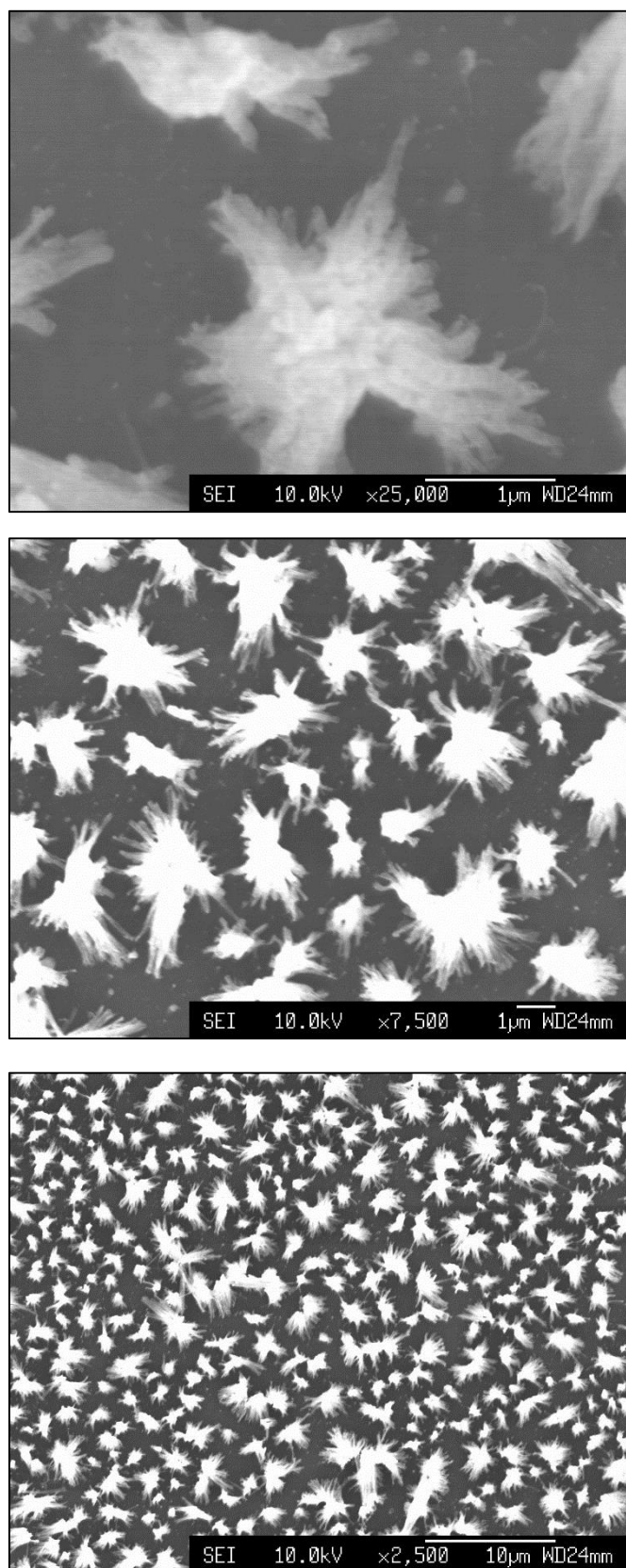


**Figure 22:** SEM images for small black silicon needles coated in a MCD film grown for 1 hour.

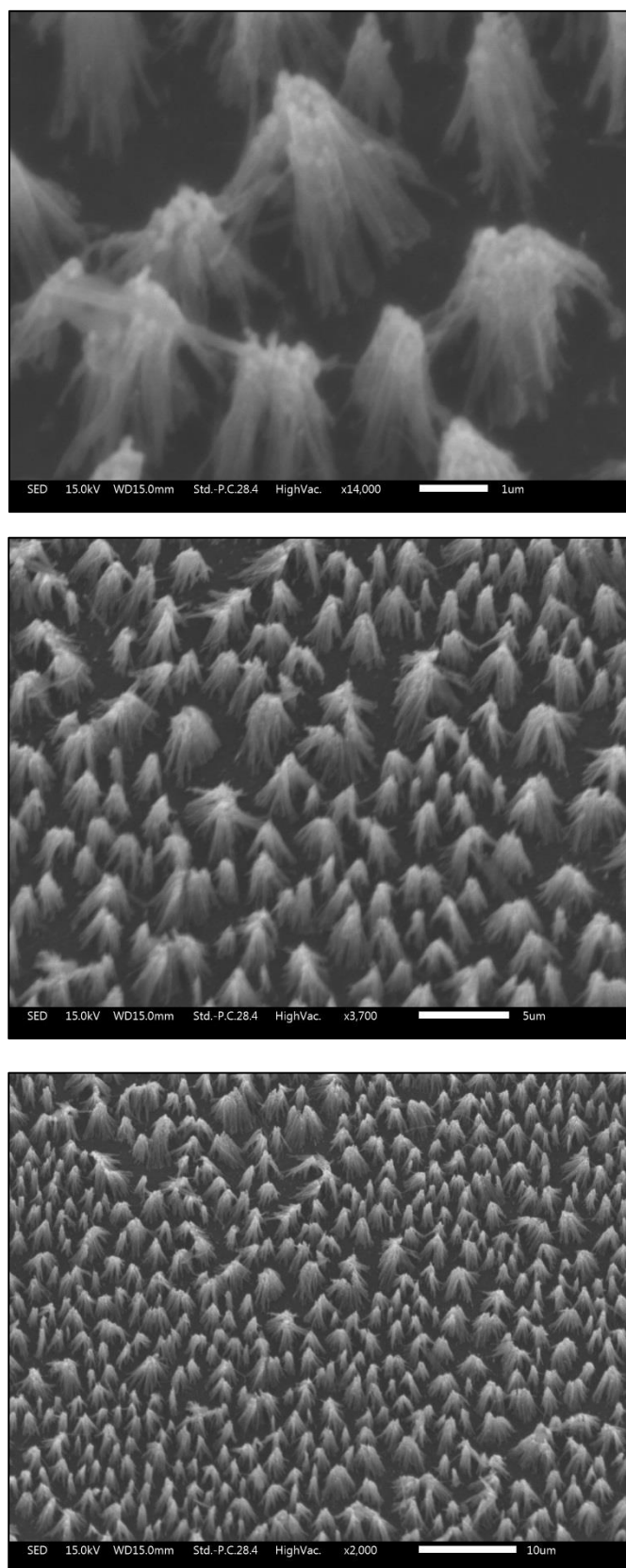


**Figure 23:** SEM images for vertically aligned carbon nanotubes (VACNTs) viewed from above.

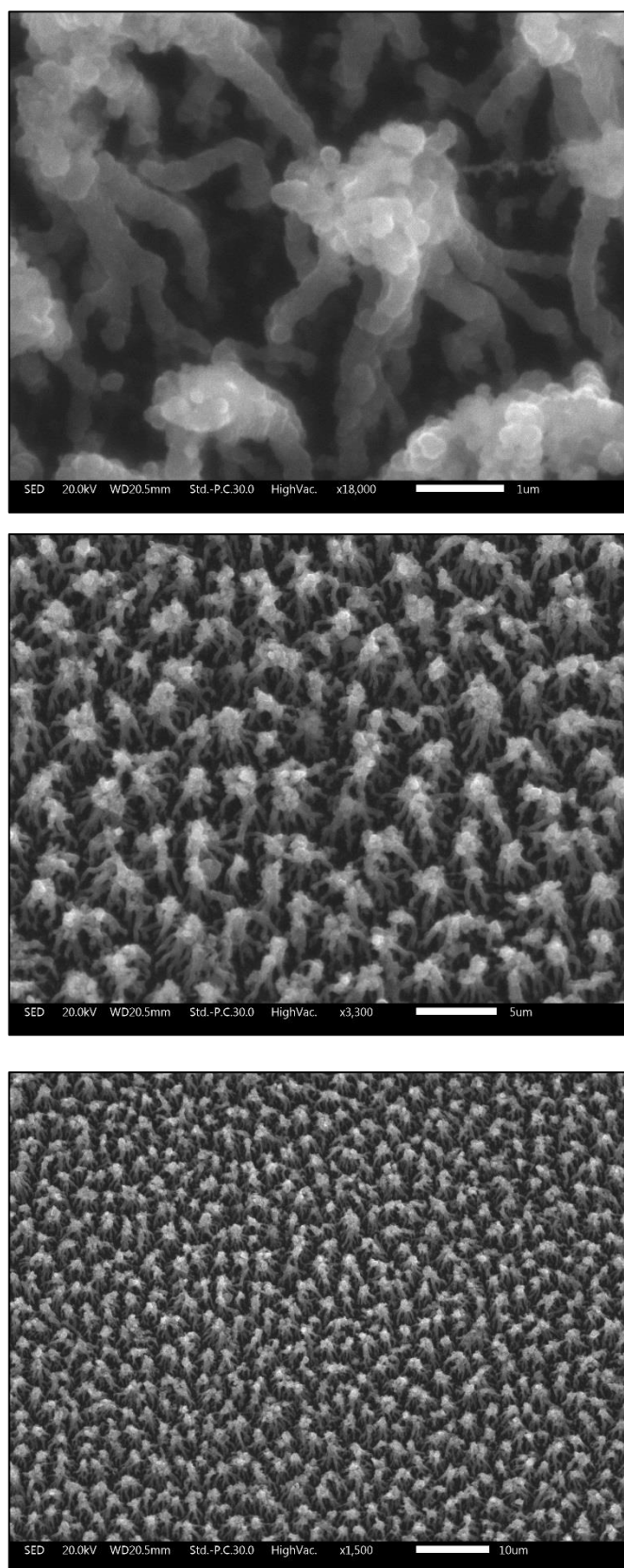




**Figure 24:** SEM images for VACNT teepees after electrospray pretreatment viewed from above.

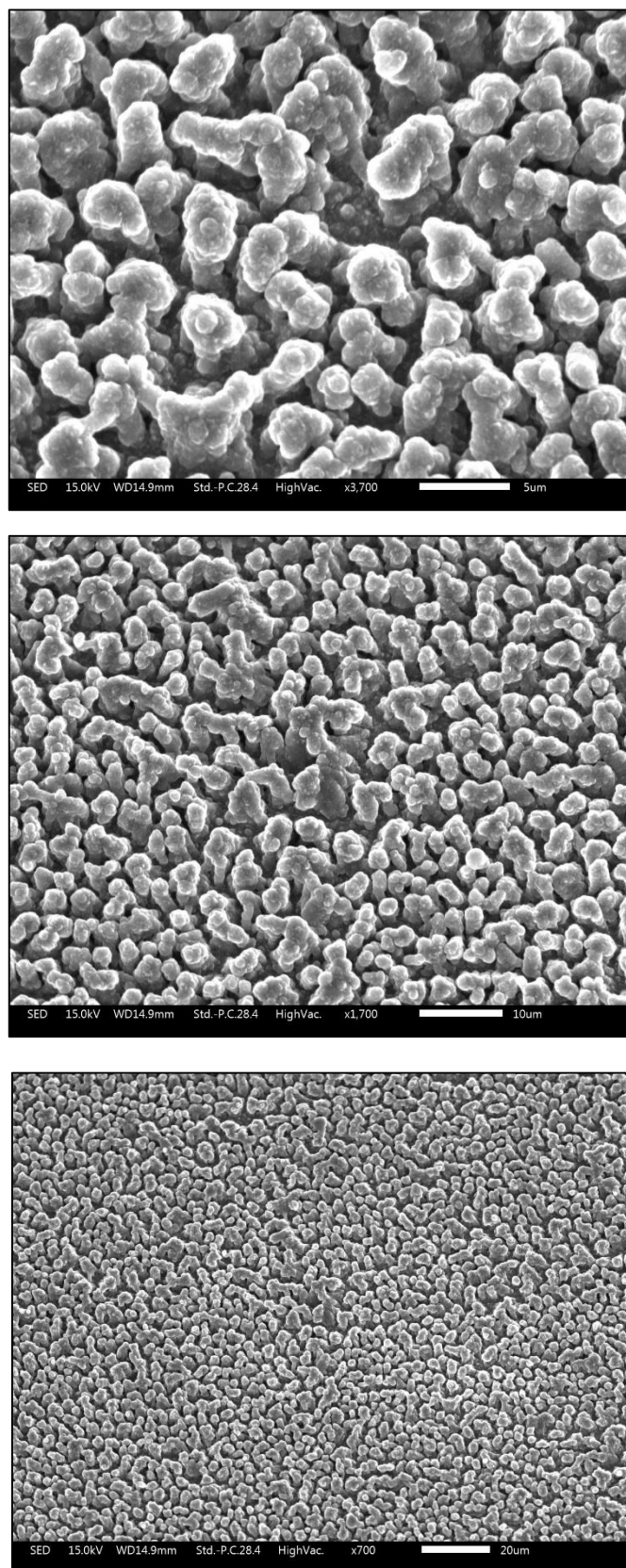


**Figure 25:** SEM images for VACNT teepees after electrospray pretreatment viewed from tilted angle.

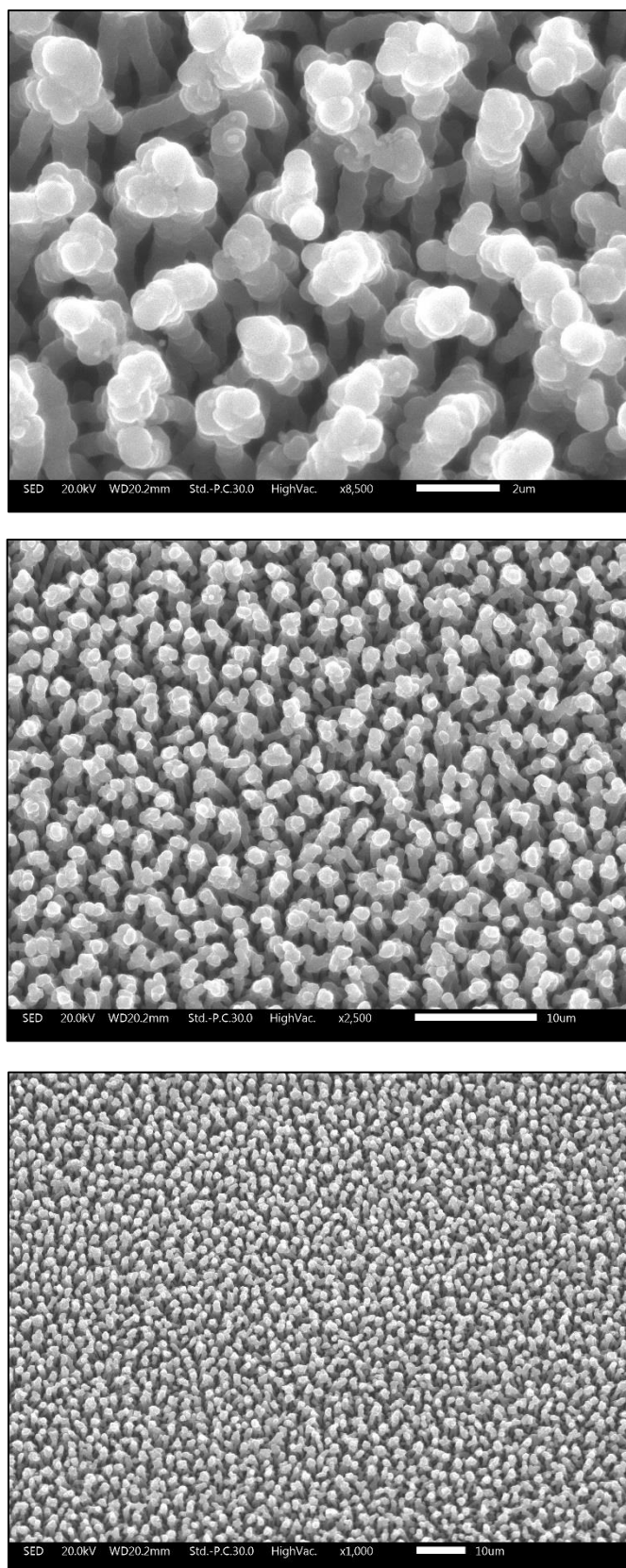


**Figure 26:** SEM images viewed from tilted angle for VACNT teepees coated in MCD film grown for 20 mins after electrospray pretreatment.

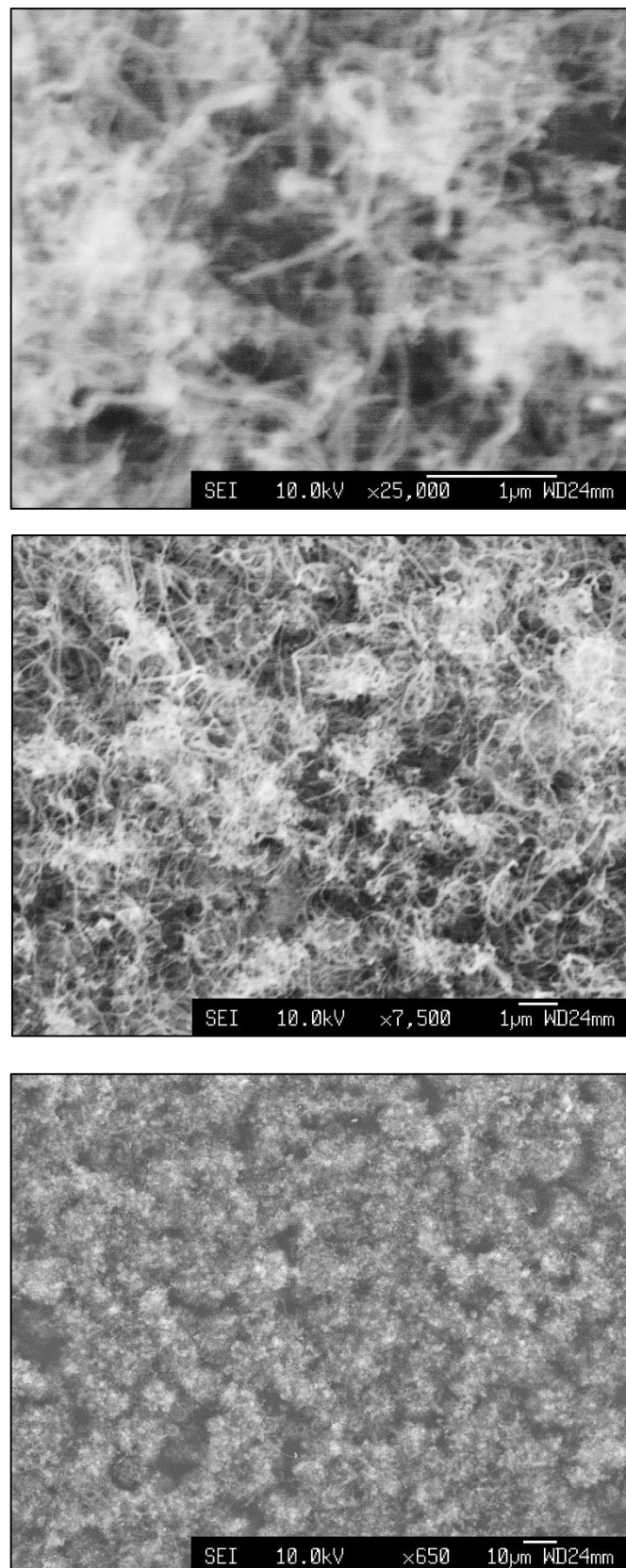




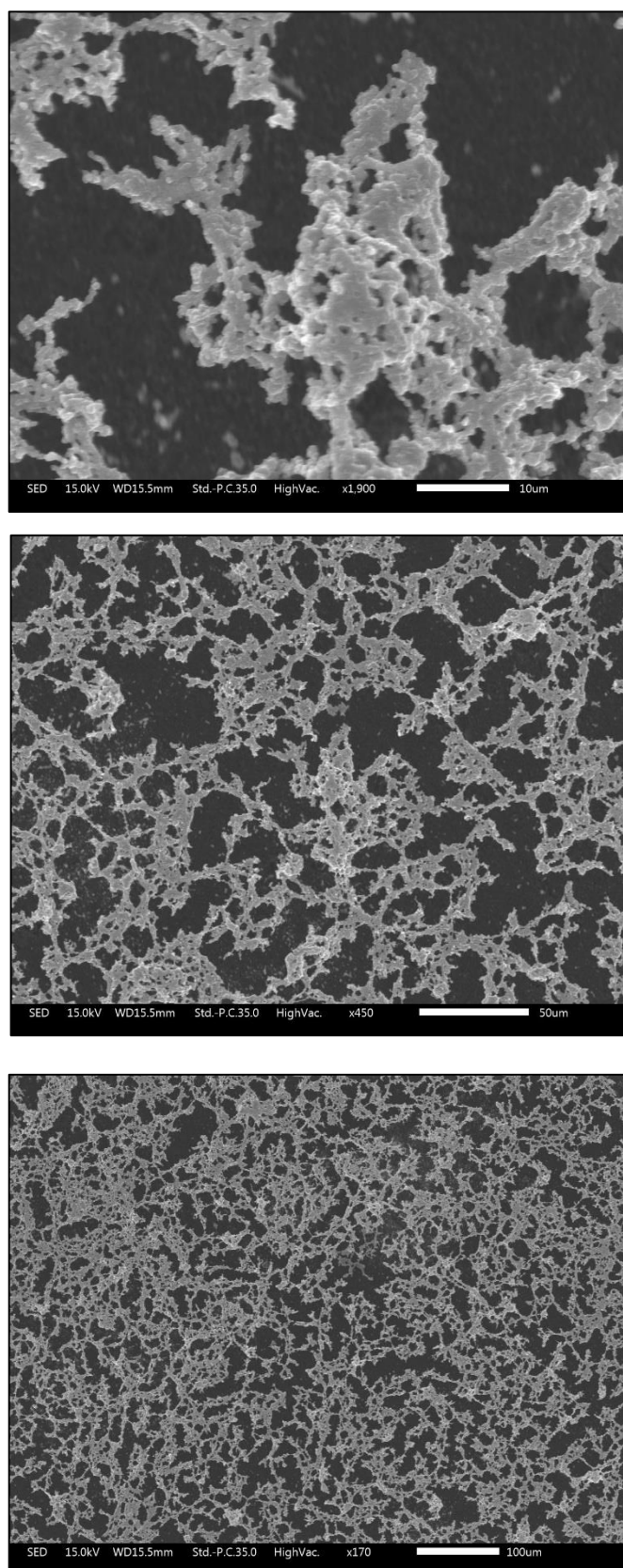
**Figure 27:** SEM images viewed from tilted angle for VACNT teepees coated in MCD film grown for 45 mins after electrospray pretreatment.



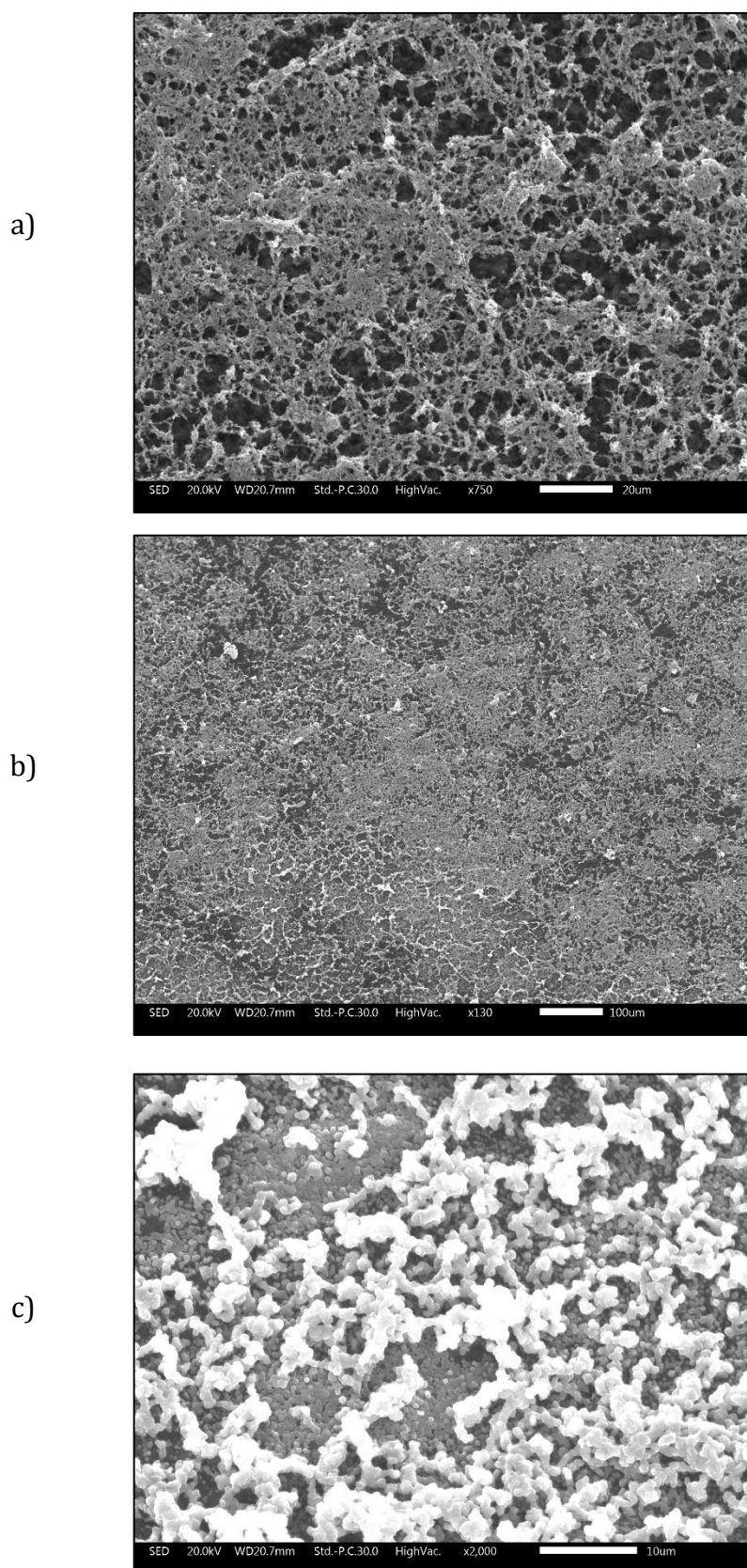
**Figure 28:** SEM images viewed from tilted angle for VACNT teepees coated in NCD film grown for 45 mins after electrospray pretreatment.



**Figure 29:** SEM images for uncoated carbon nanotube (CNT) webs.



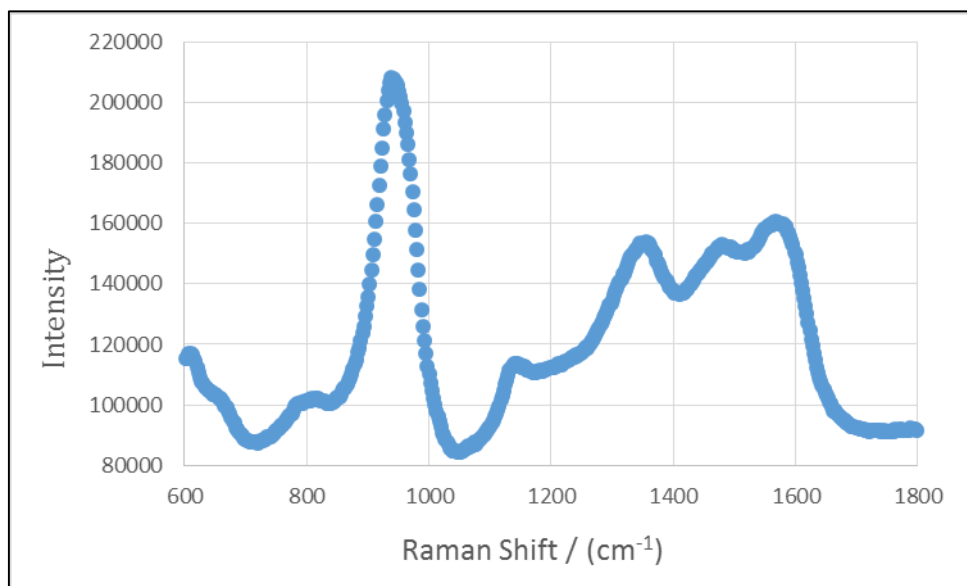
**Figure 30:** SEM images for carbon nanotube (CNT) webs coated in a MCD film grown for 30 mins after electrospray pretreatment.



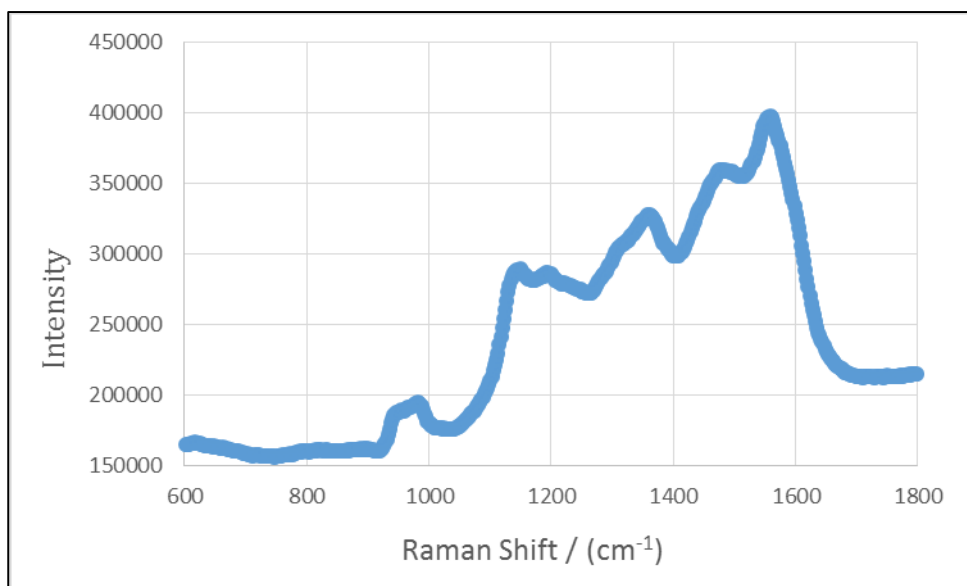
**Figure 31:** SEM images for carbon nanotube (CNT) webs coated in a MCD film grown for a) & b) 20 mins and c) NCD film grown for 45 mins, after electrospray pretreatment.

# Raman Spectroscopy

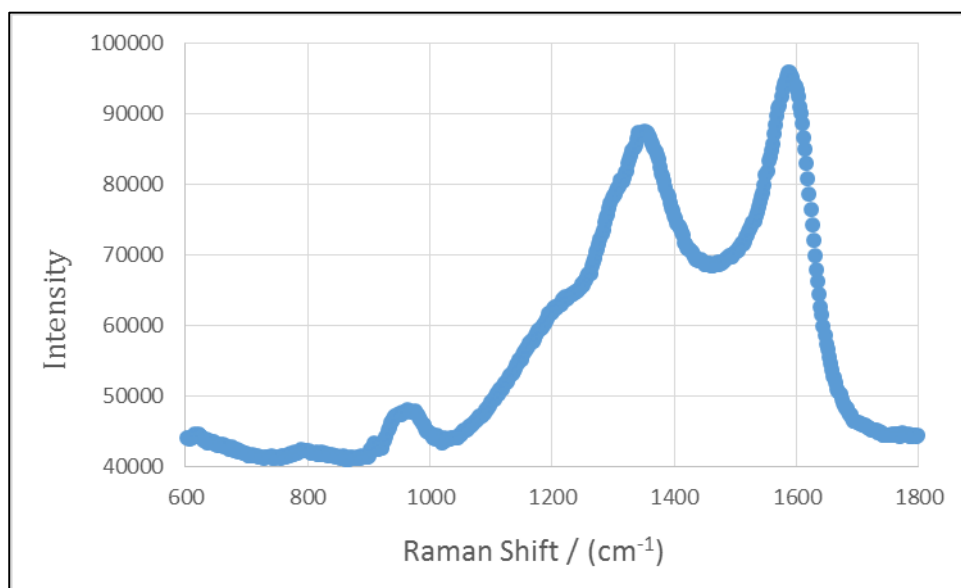
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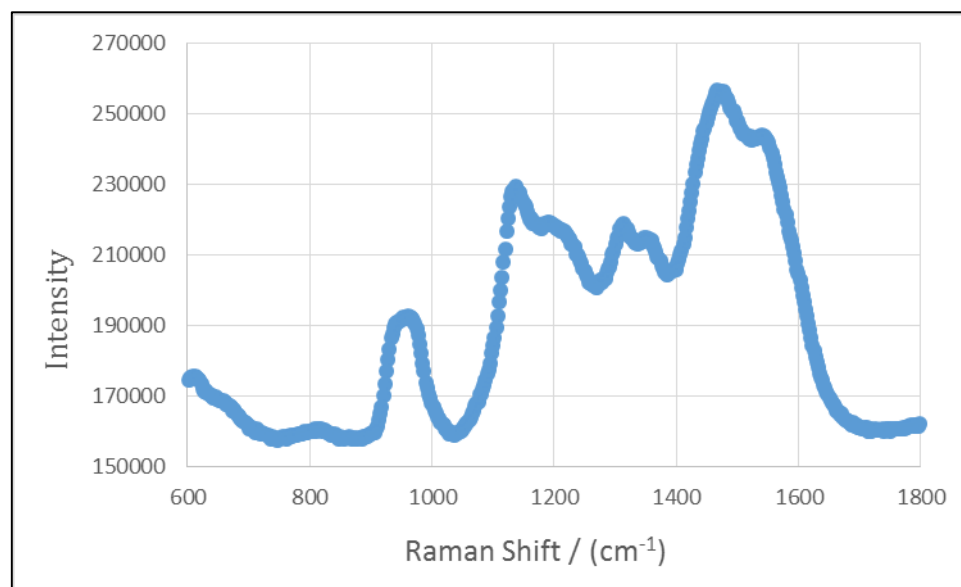
**Figure 32:** Raman spectra recorded for large silicon needles coated in a MCD film grown for 20 mins after electrospray pretreatment.



**Figure 33:** Raman spectra recorded for large silicon needles coated in a MCD film grown for 30 mins after electrospray pretreatment.

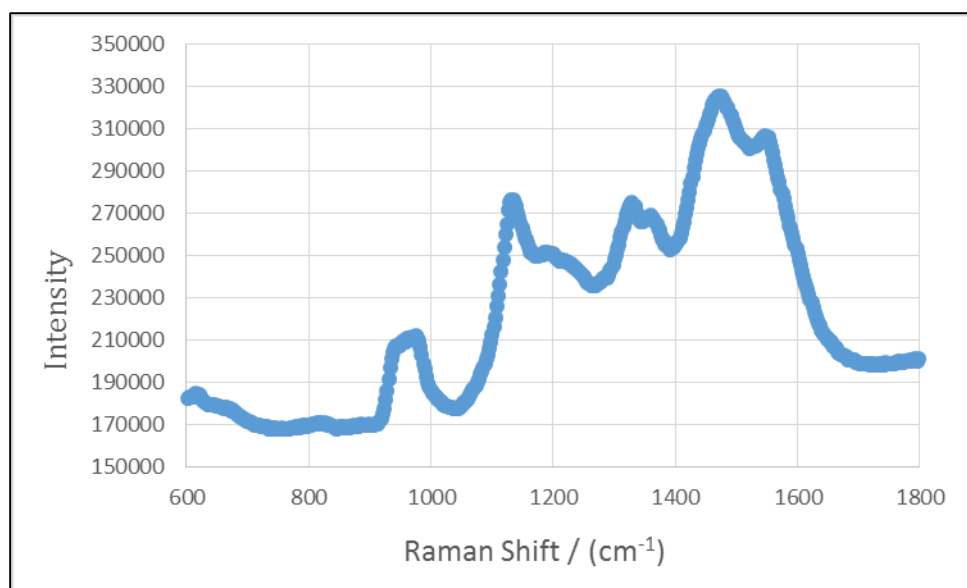


**Figure 34:** Raman spectra recorded for large silicon needles coated in a MCD film grown for 45 mins after electrospray pretreatment.

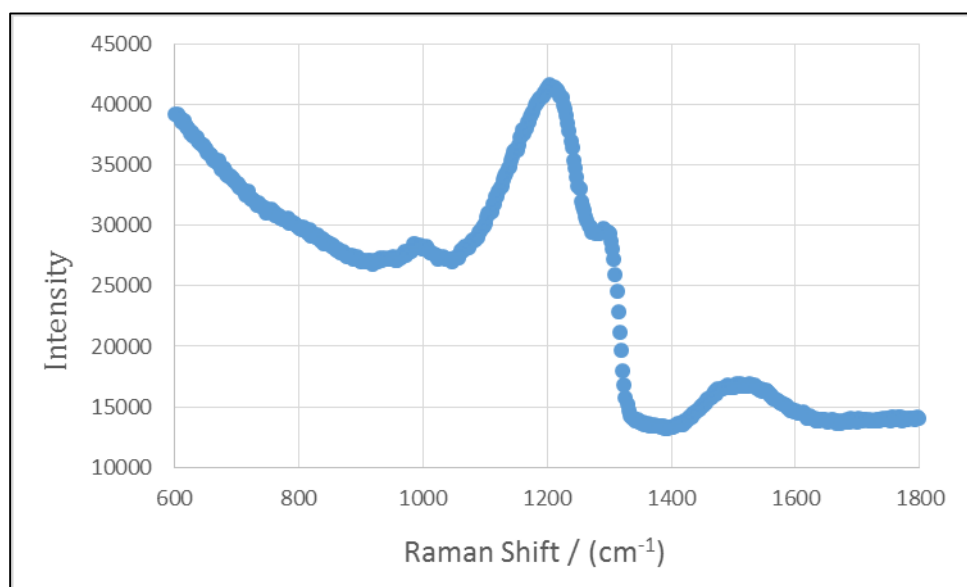


**Figure 35:** Raman spectra recorded for large silicon needles coated in a MCD film grown for 1 hour after electrospray pretreatment.



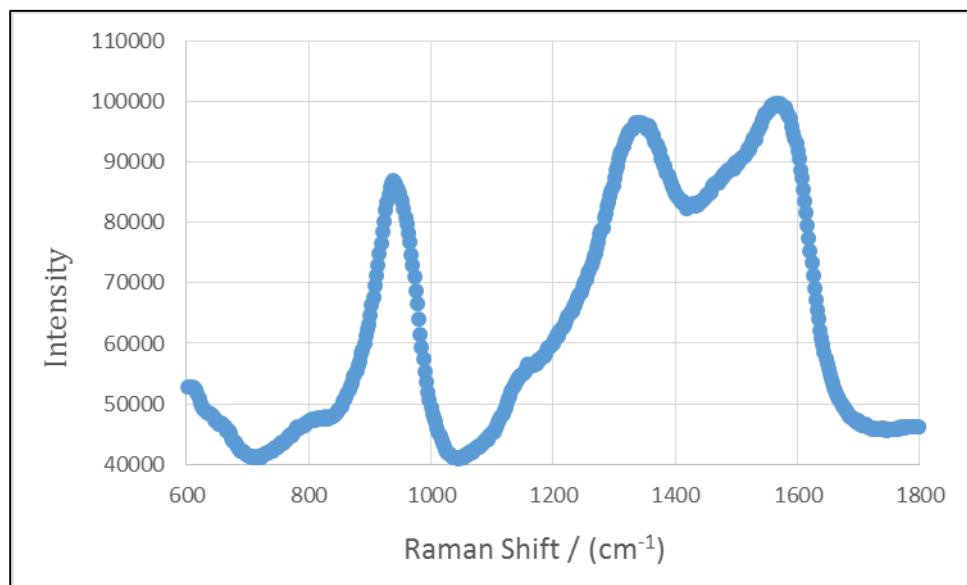


**Figure 36:** Raman spectra recorded for large silicon needles coated in a MCD film grown for 1 hour 30 mins after electro spray pretreatment.

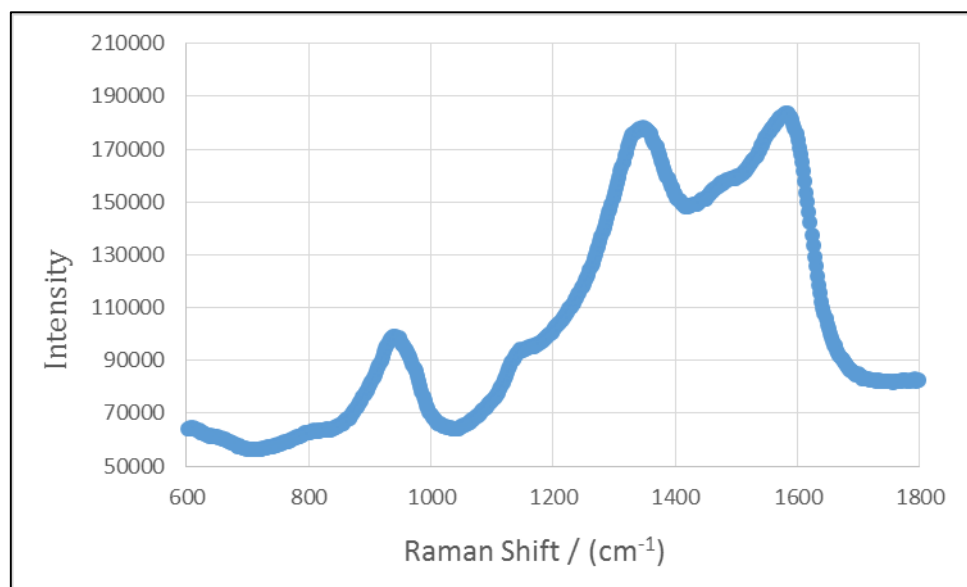


**Figure 37:** Raman spectra recorded for large silicon needles coated in a MCD film grown for 2 hours after electro spray pretreatment.

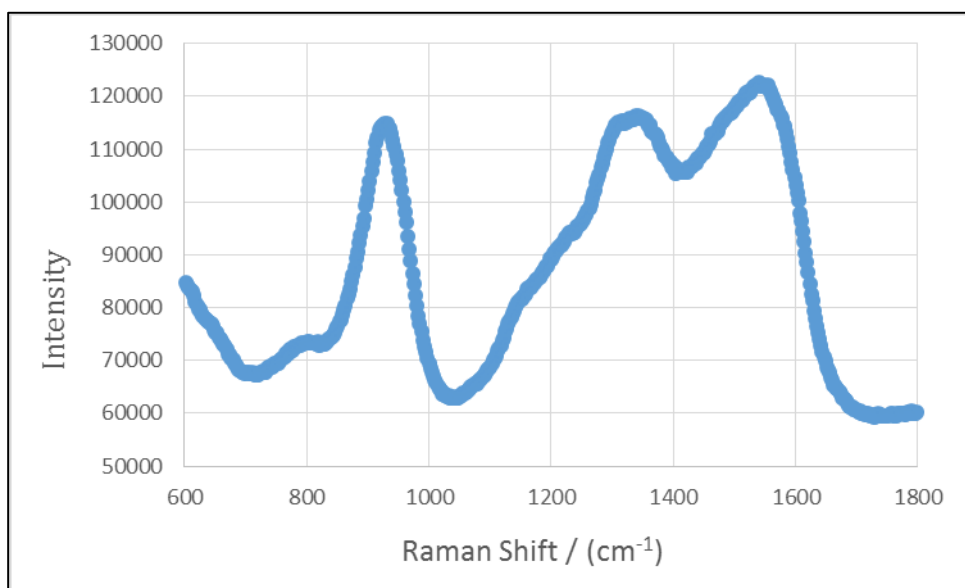




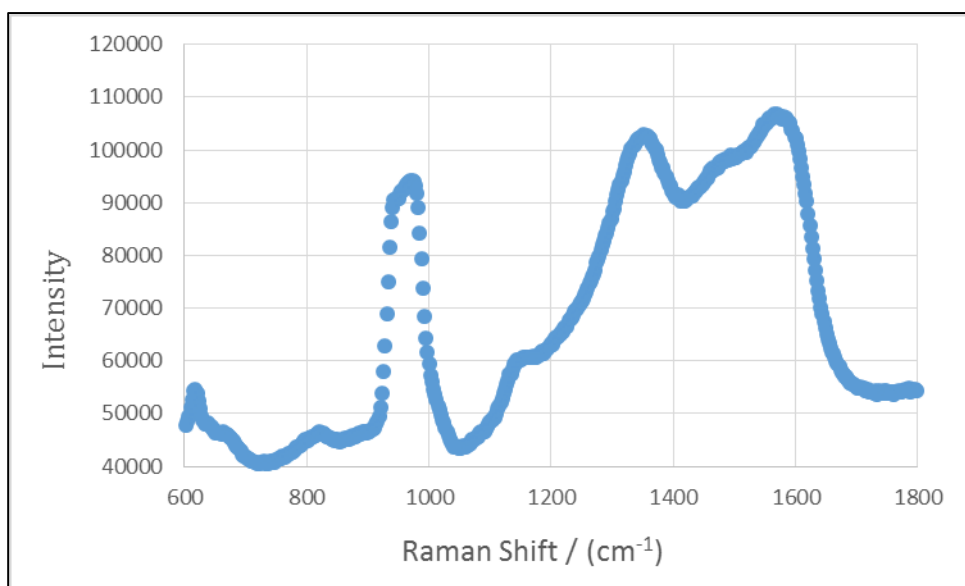
**Figure 38:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 15 mins after electrospray pretreatment.



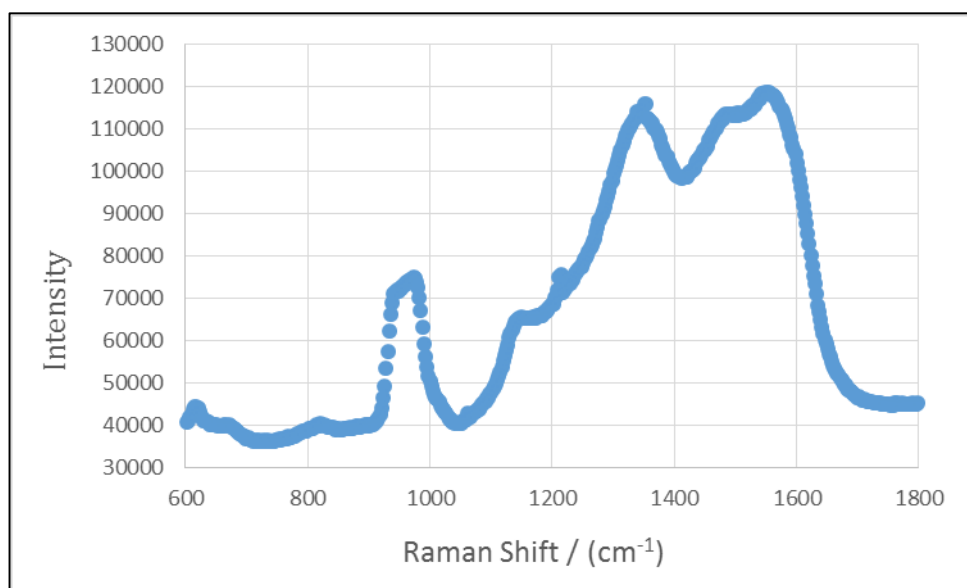
**Figure 39:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 20 mins after electrospray pretreatment.



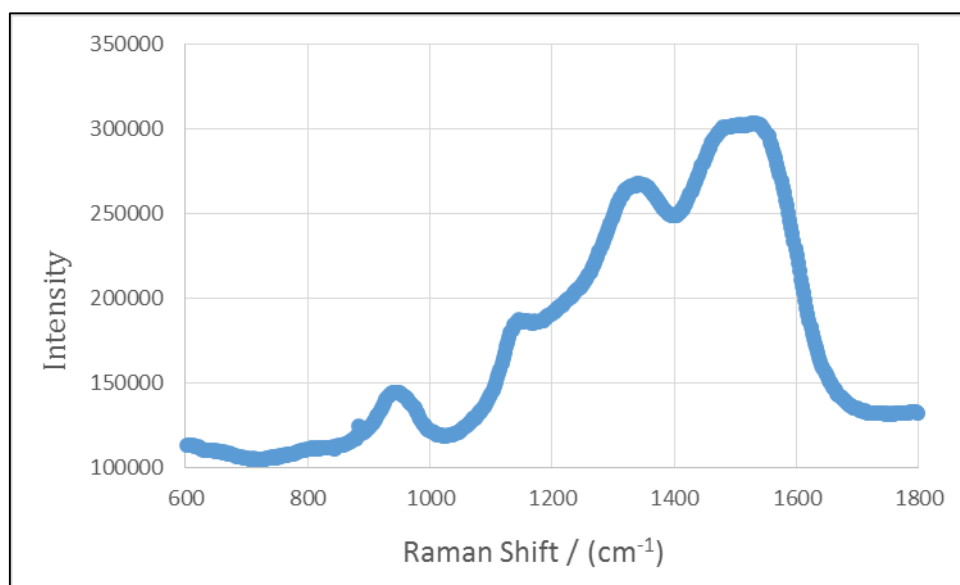
**Figure 40:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 30 mins after electrospray pretreatment.



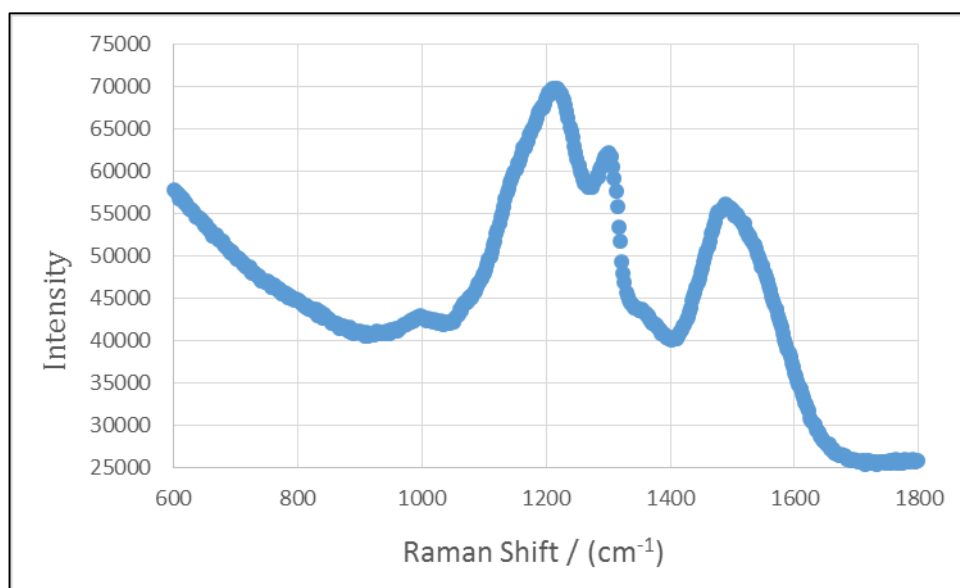
**Figure 41:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 40 mins after electrospray pretreatment.



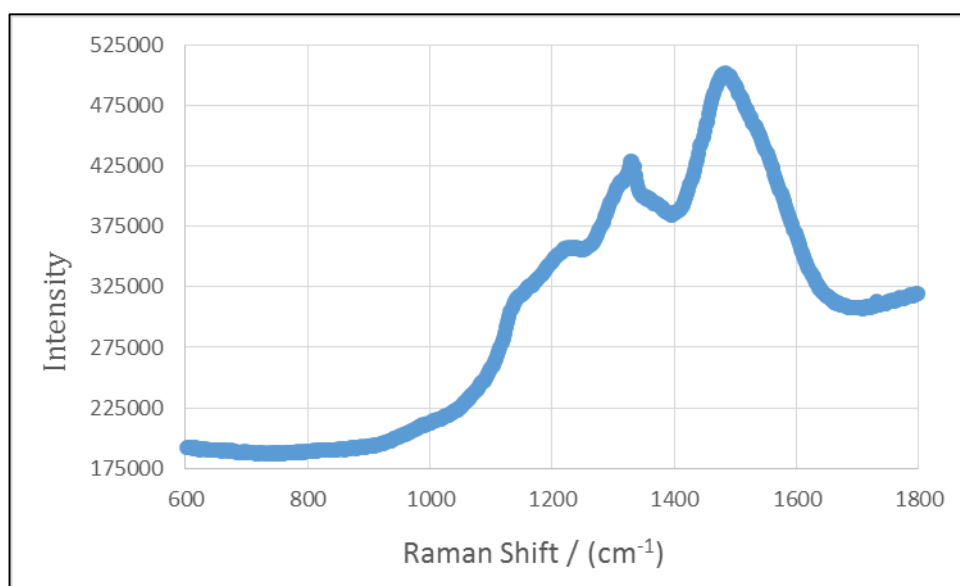
**Figure 42:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 1 hour 20 mins after electrospray pretreatment.



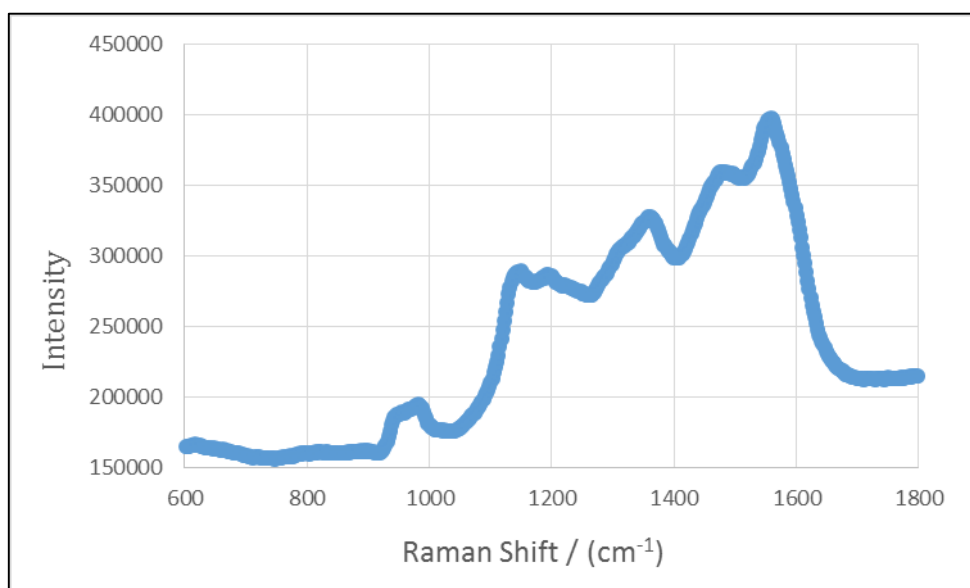
**Figure 43:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 2 hours after electrospray pretreatment.



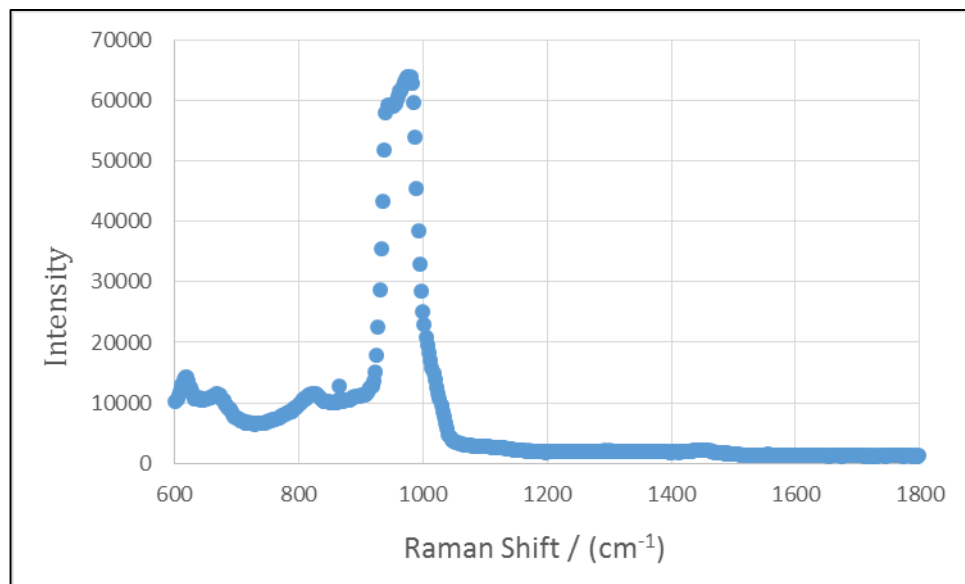
**Figure 44:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 3 hours after electrospray pretreatment.



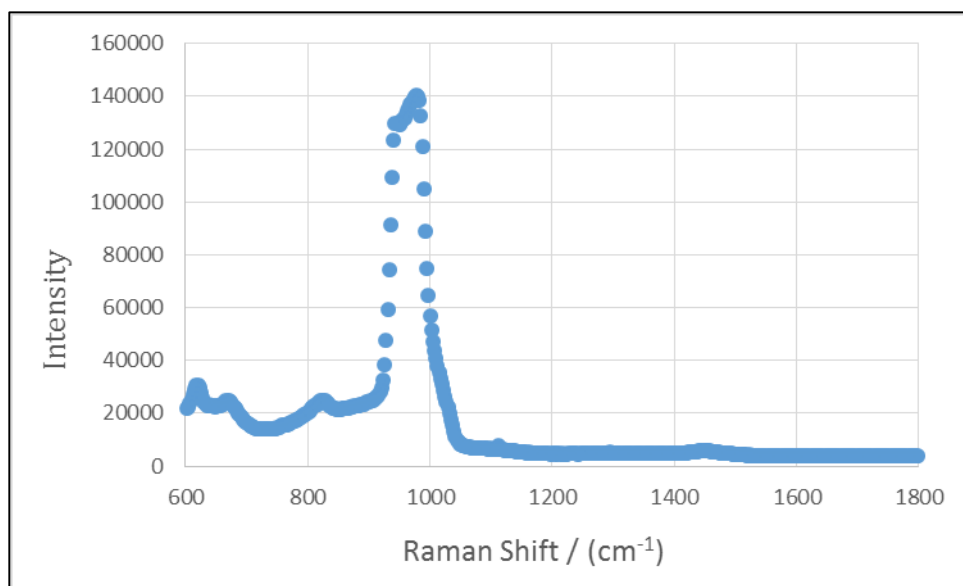
**Figure 45:** Raman spectra recorded for large silicon needles coated in a NCD film grown for 4 hours after electrospray pretreatment.



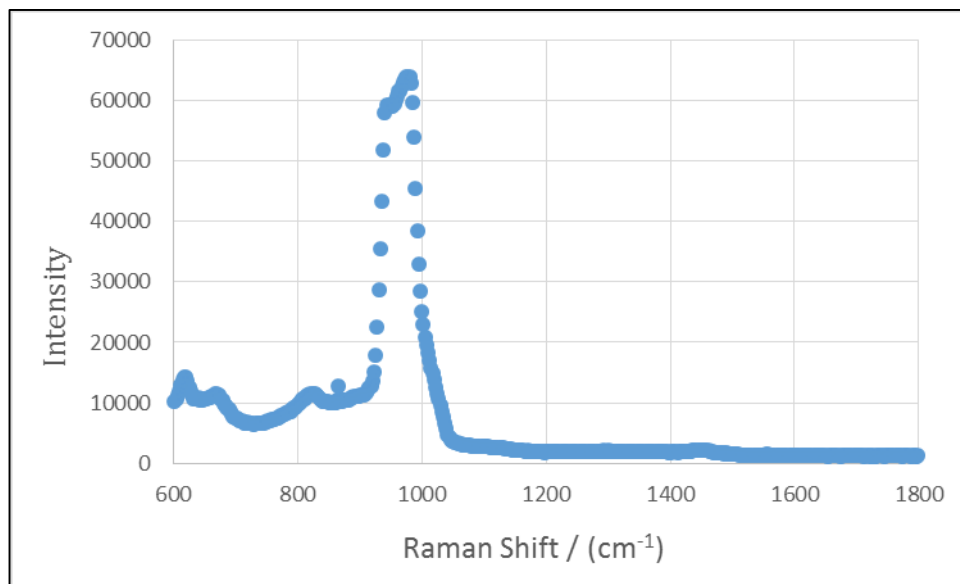
**Figure 46:** Raman spectra recorded for small silicon needles coated in a MCD film grown for 30 mins after electrospray pretreatment.



**Figure 47:** Raman spectra recorded for small silicon needles coated in a MCD film grown for 30 mins.

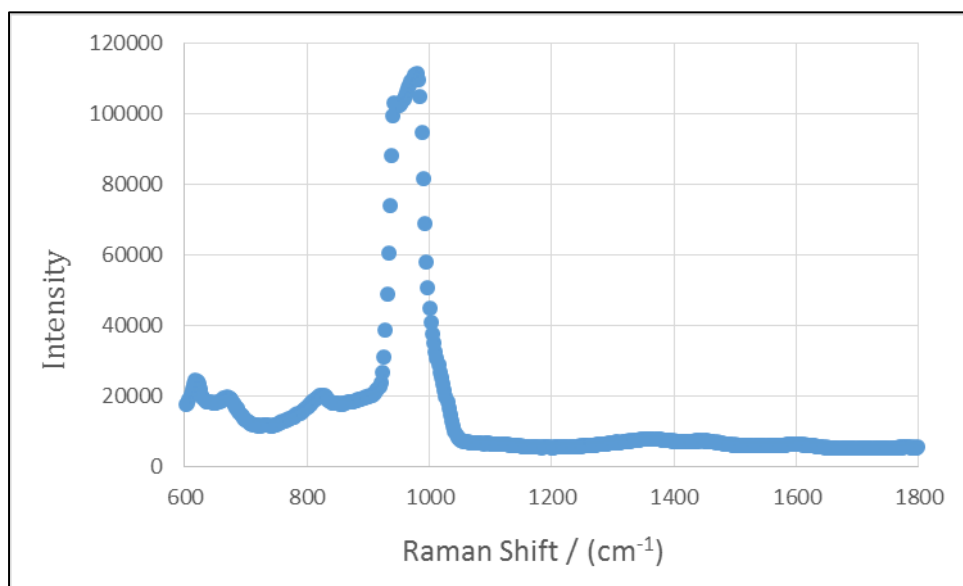


**Figure 48:** Raman spectra recorded for small silicon needles coated in a MCD film grown for 1 hour.

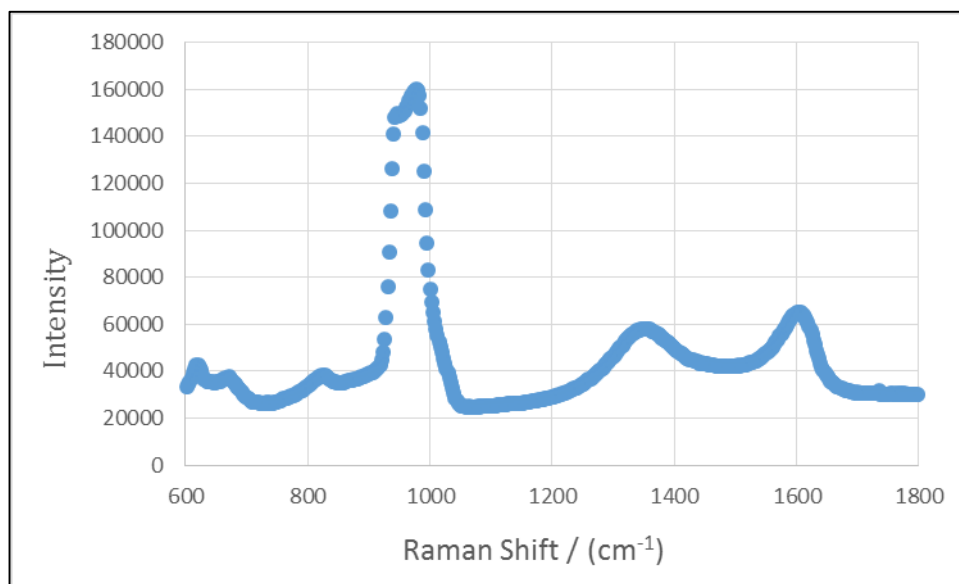


**Figure 49:** Raman spectra recorded for small silicon needles coated in a MCD film grown for 30 mins.

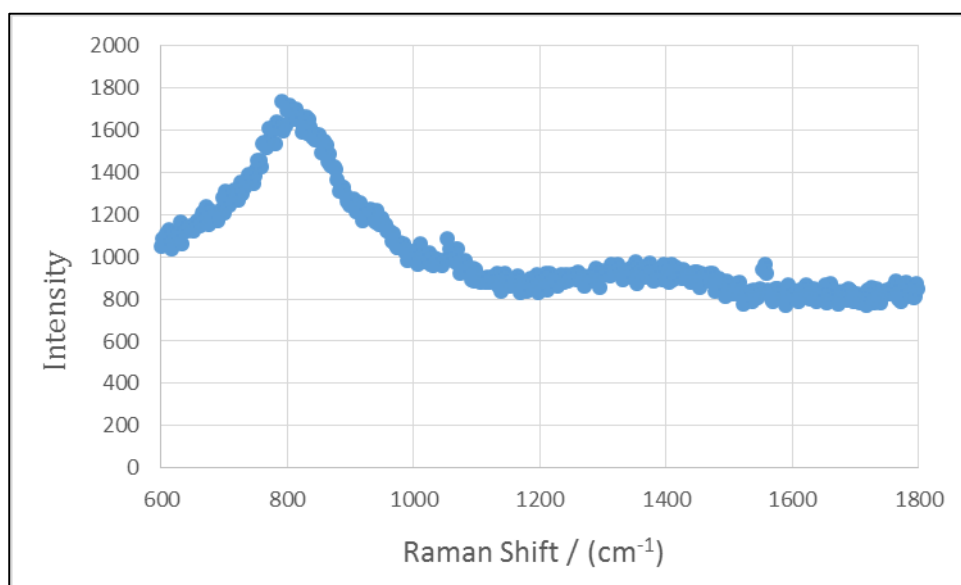




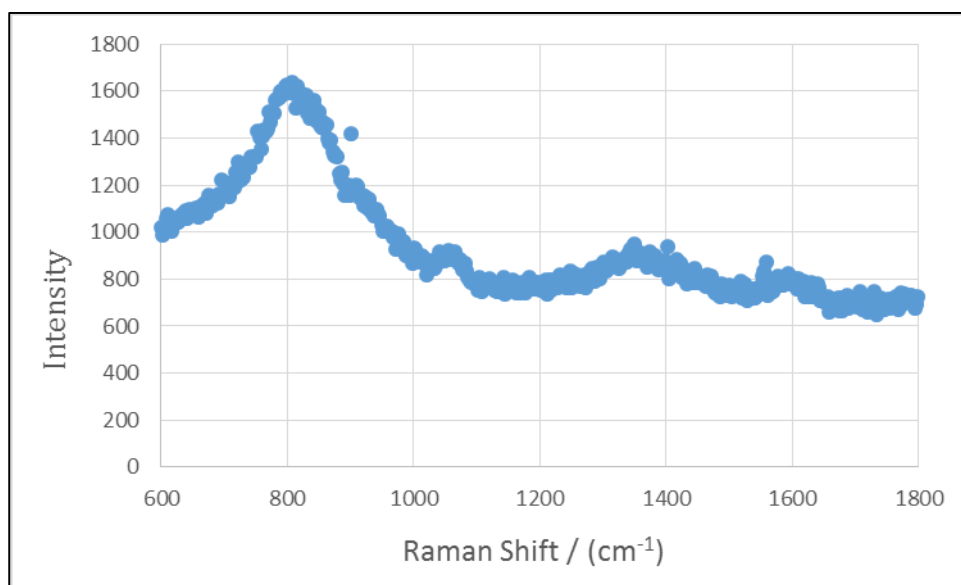
**Figure 50:** Raman spectra recorded for small silicon needles coated in a MCD film grown for 2 hours.



**Figure 51:** Raman spectra recorded for small silicon needles coated in a NCD film grown for 1 hour.

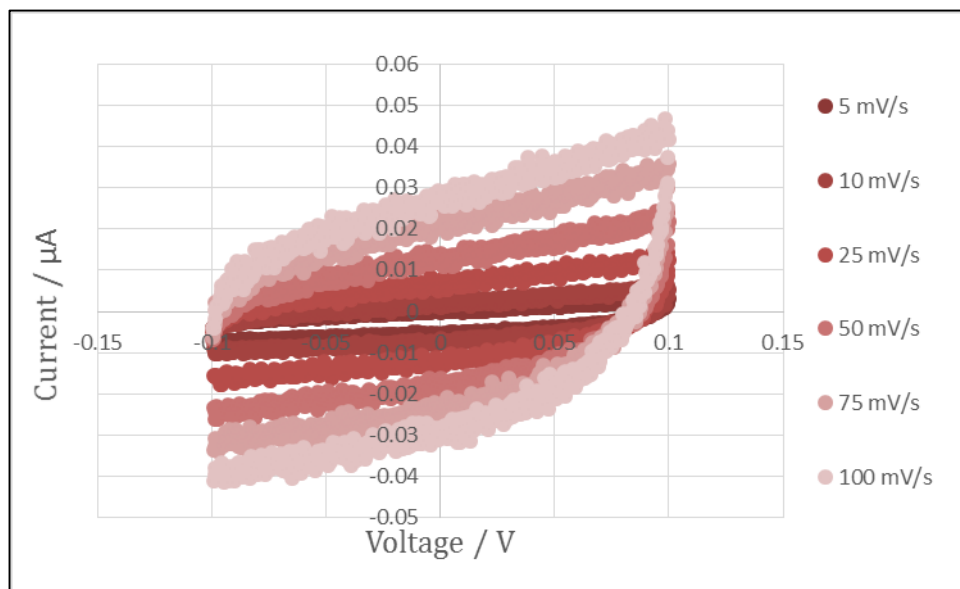


**Figure 52:** Raman spectra recorded for VACNT teepees coated in a MCD film grown for 20 mins after electrospray pretreatment.

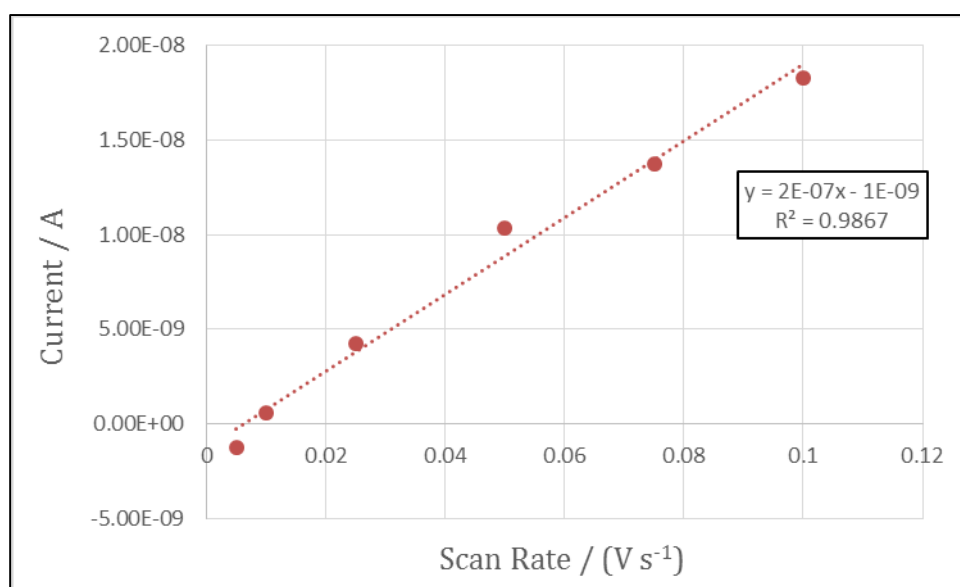


**Figure 53:** Raman spectra recorded for CNT webs coated in a MCD film grown for 20 mins after electrospray pretreatment.

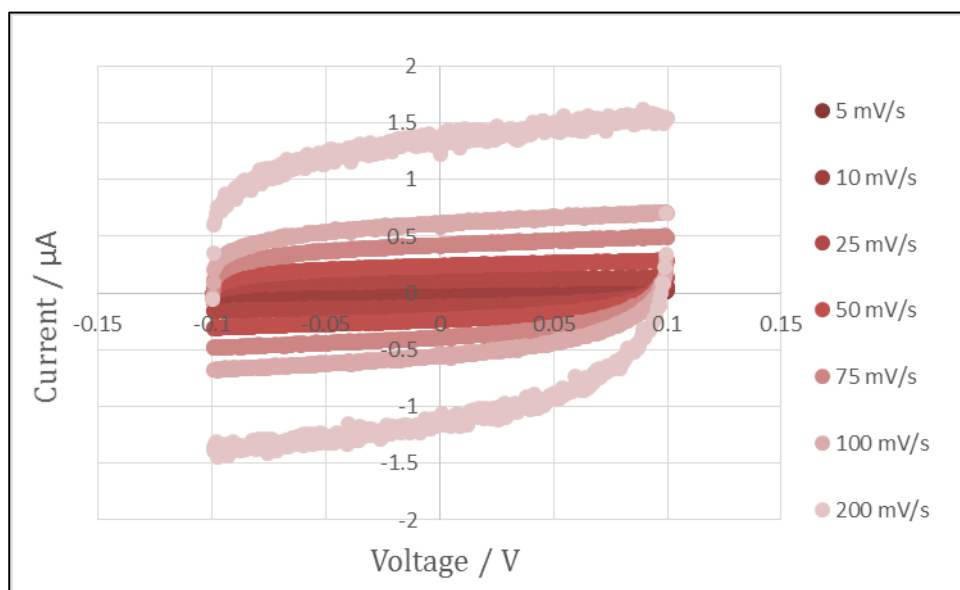
## Cyclic Voltammetry – $\text{KNO}_3$



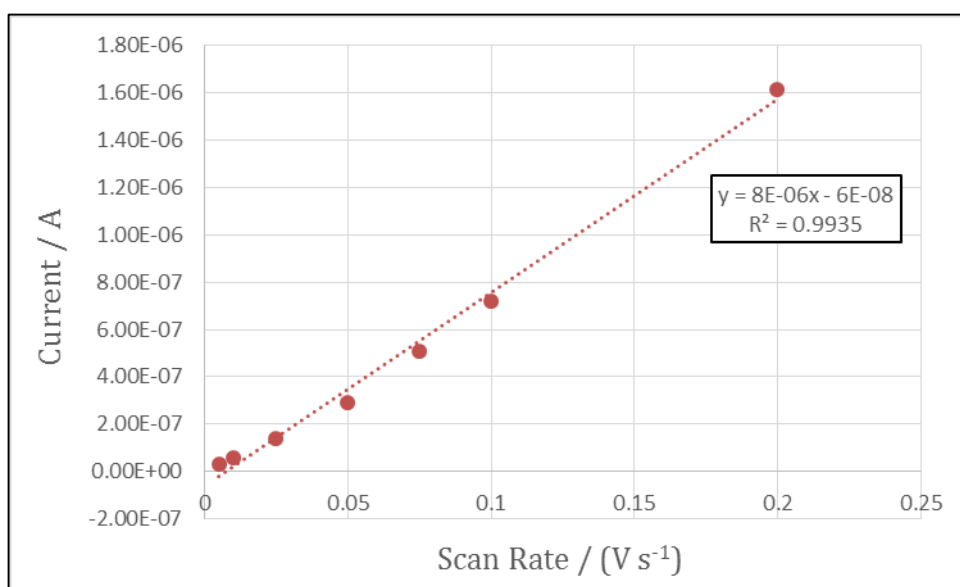
**Figure 54:** Cyclic Voltammograms (CVs) recorded for flat boron doped diamond sample using a 1 M potassium nitrate solution.



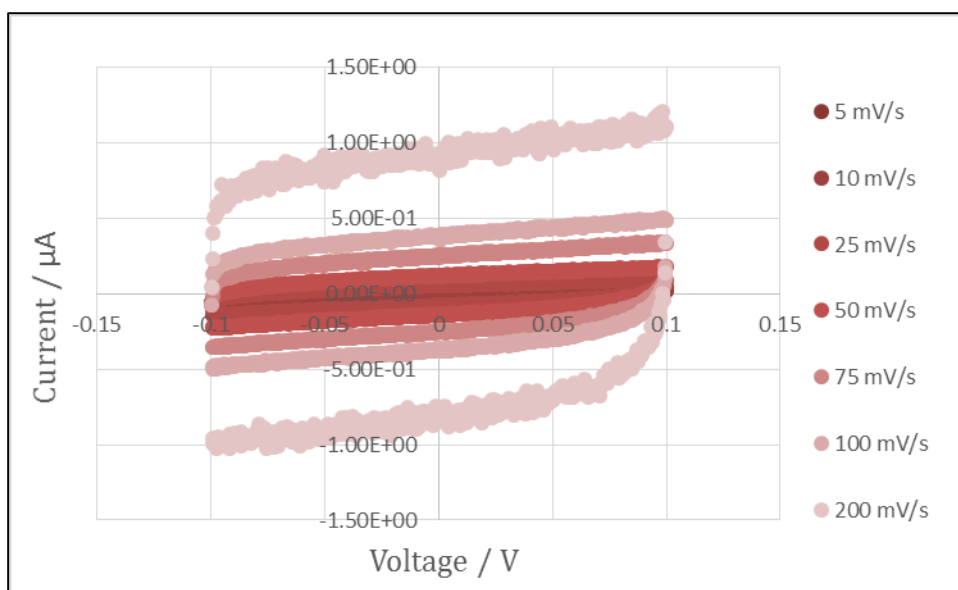
**Figure 55:** Plot of capacitive current vs scan rate for flat boron doped diamond sample.



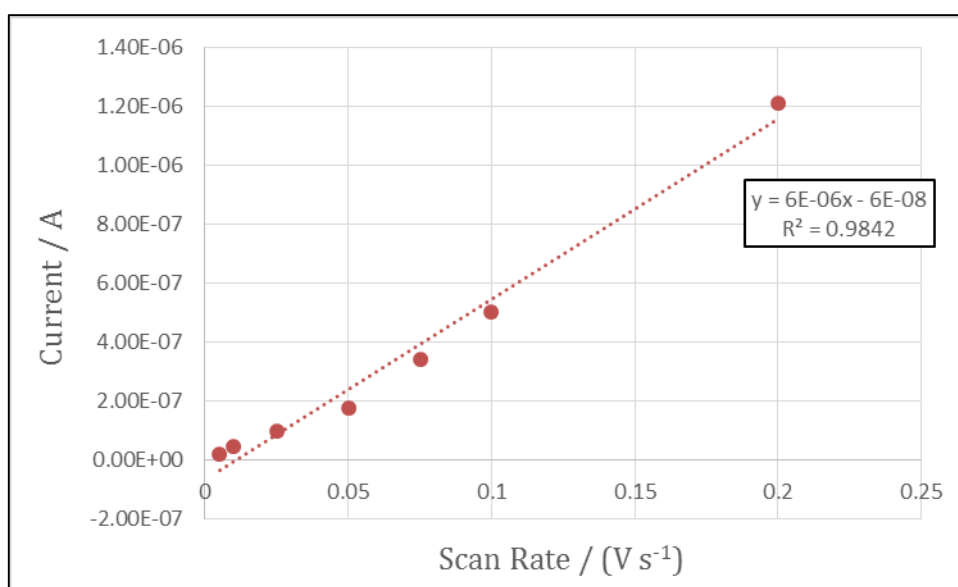
**Figure 56:** CVs recorded for large black silicon needles coated in a MCD film grown for 20 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



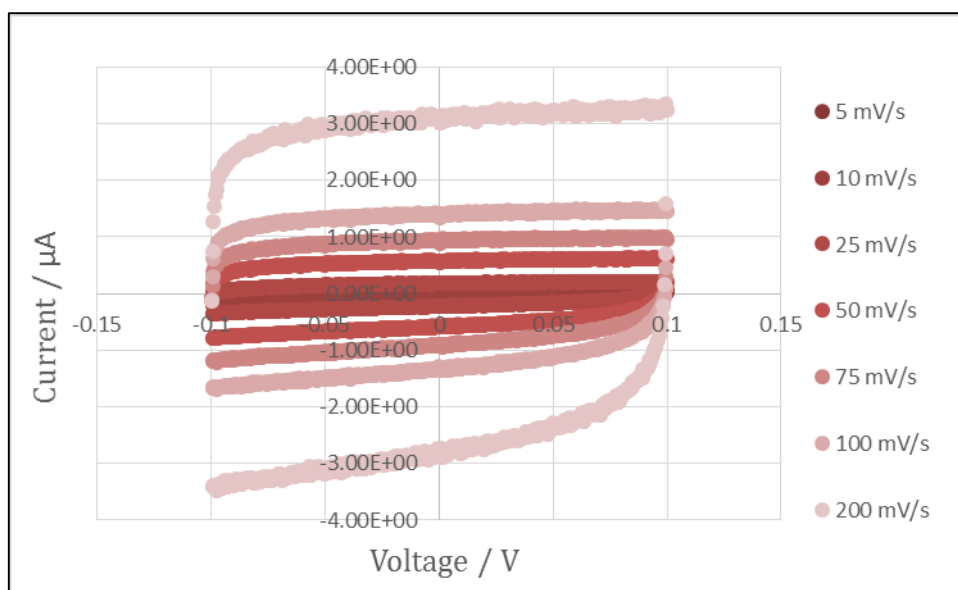
**Figure 57:** Plot of capacitive current vs scan rate for large black silicon needles coated in a MCD film grown for 20 mins after electro spray pretreatment.



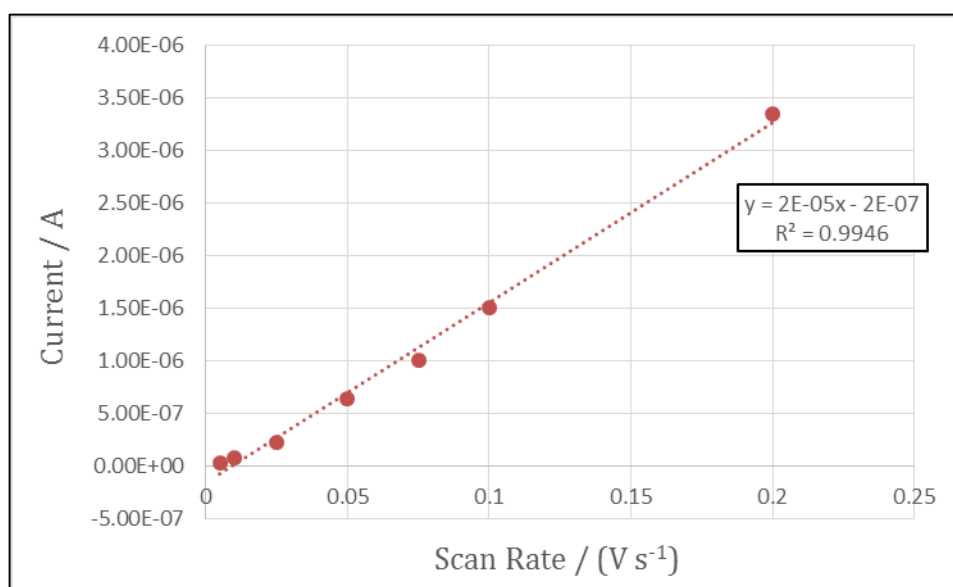
**Figure 58:** CVs recorded for large black silicon needles coated in a MCD film grown for 45 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



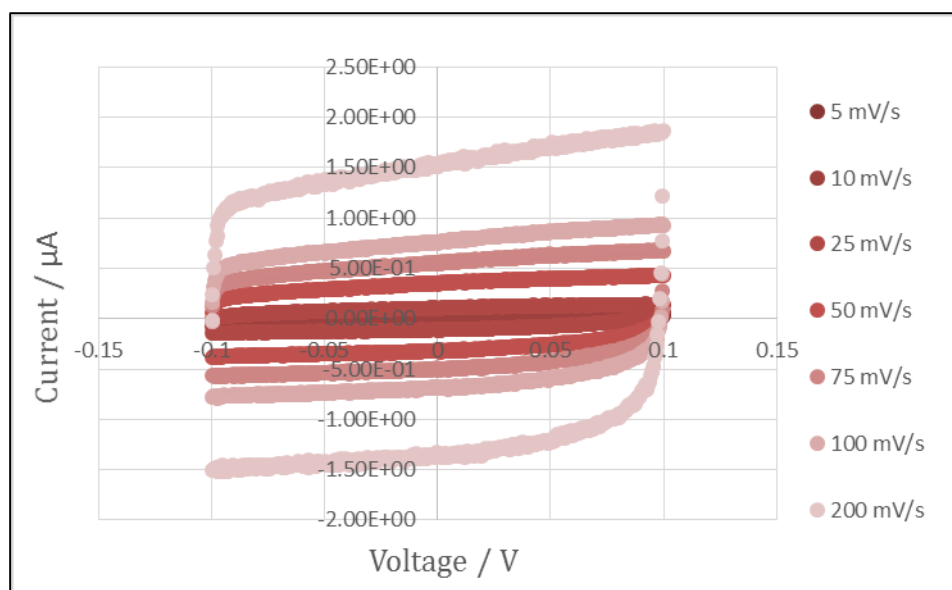
**Figure 59:** Plot of capacitive current vs scan rate for large black silicon needles coated in a MCD film grown for 45 mins after electro spray pretreatment.



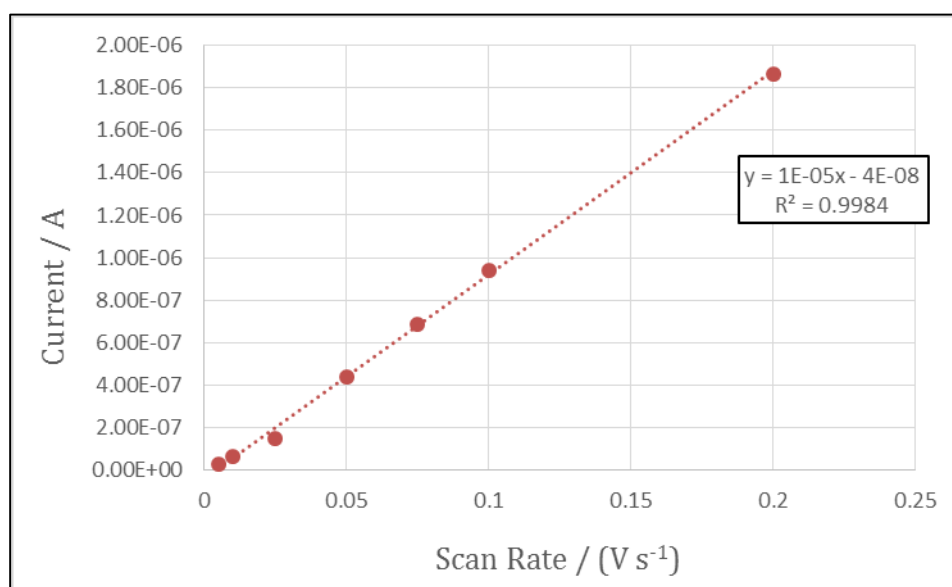
**Figure 60:** CVs recorded for large black silicon needles coated in a MCD film grown for 2 hours after electro spray pretreatment using a 1 M potassium nitrate solution.



**Figure 61:** Plot of capacitive current vs scan rate for large black silicon needles coated in a MCD film grown for 2 hours after electro spray pretreatment.

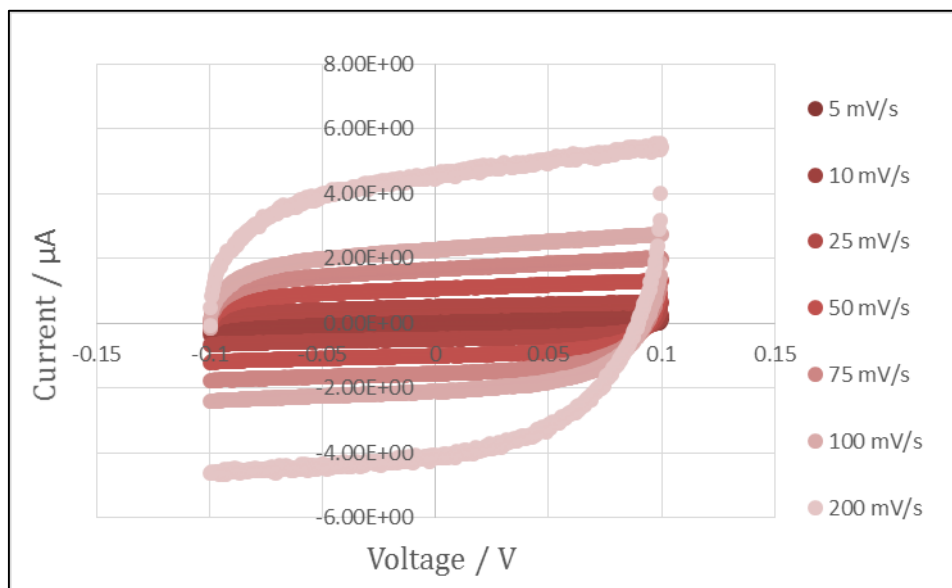


**Figure 62:** CVs recorded for large black silicon needles coated in a NCD film grown for 20 mins after electro spray pretreatment using a 1 M potassium nitrate solution.

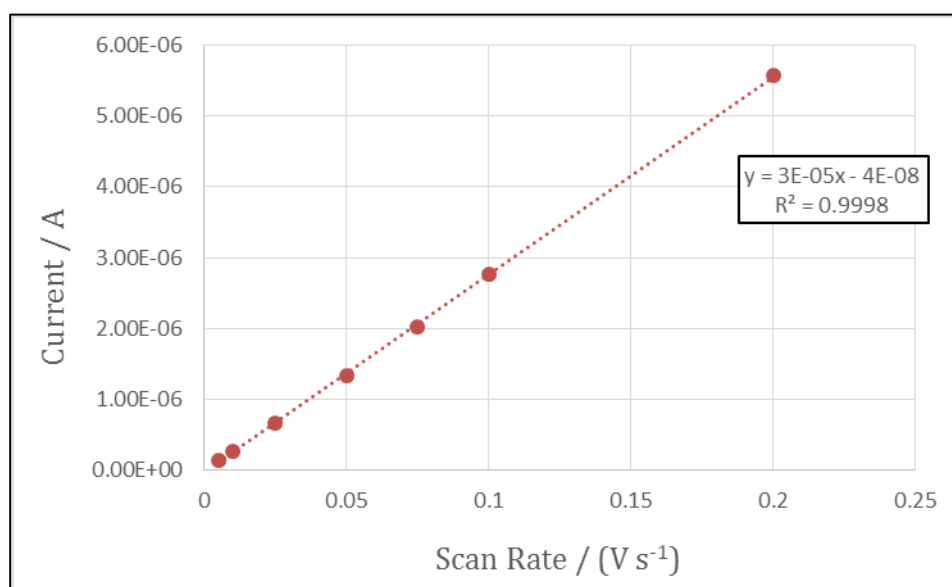


**Figure 63:** Plot of capacitive current vs scan rate for large black silicon needles coated in a NCD film grown for 20 mins after electro spray pretreatment.

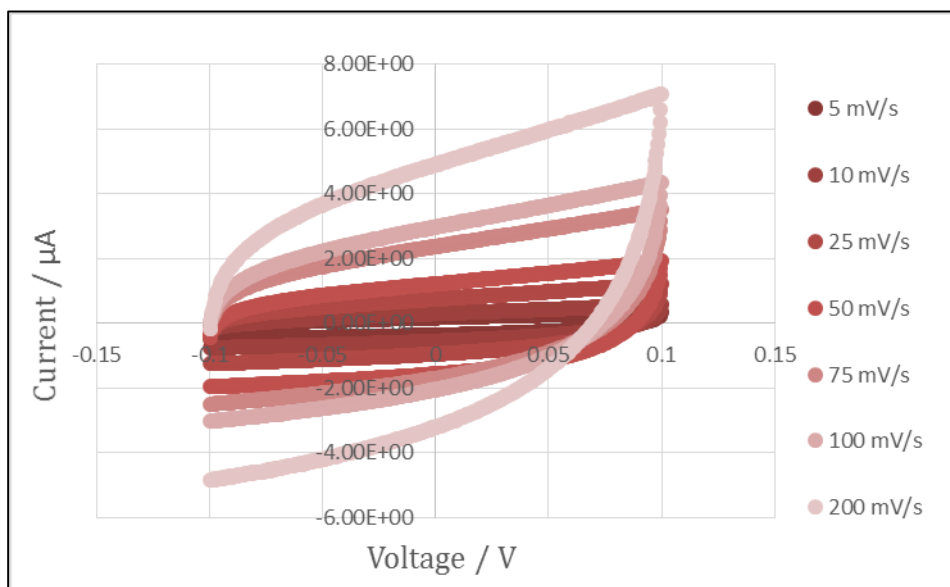




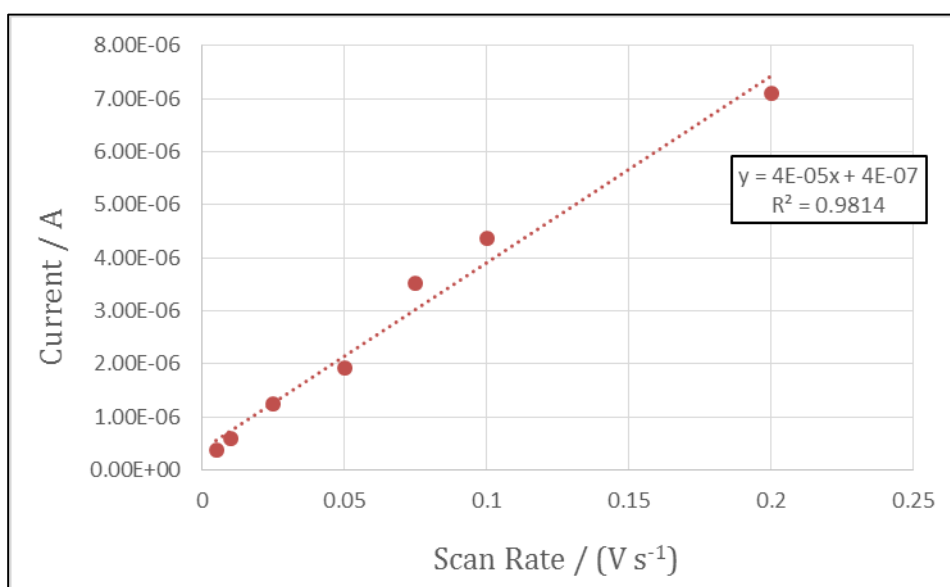
**Figure 64:** CVs recorded for large black silicon needles coated in a NCD film grown for 40 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



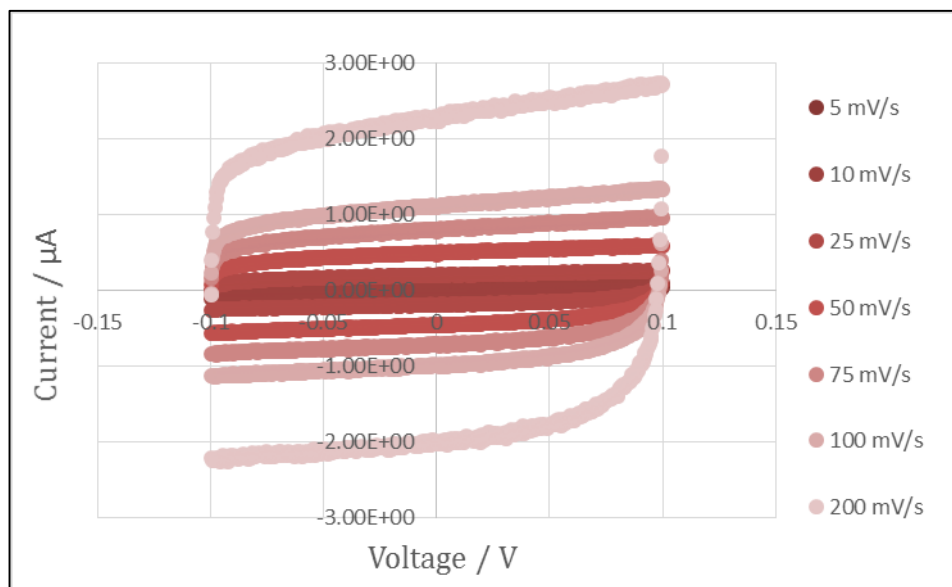
**Figure 65:** Plot of capacitive current vs scan rate for large black silicon needles coated in a NCD film grown for 40 mins after electro spray pretreatment.



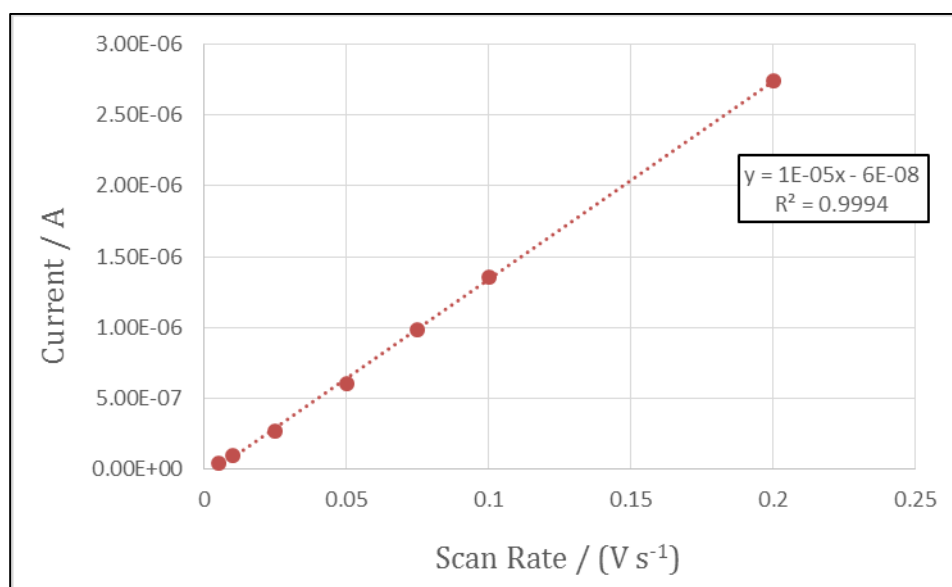
**Figure 66:** CVs recorded for large black silicon needles coated in a NCD film grown for 1 hour 20 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



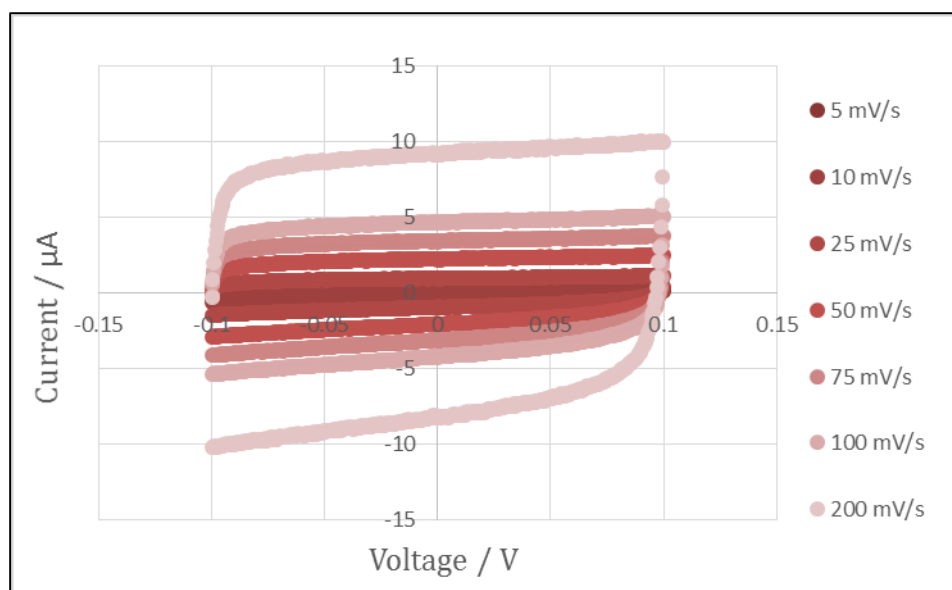
**Figure 67:** Plot of capacitive current vs scan rate for large black silicon needles coated in a NCD film grown for 1 hour 20 mins after electro spray pretreatment.



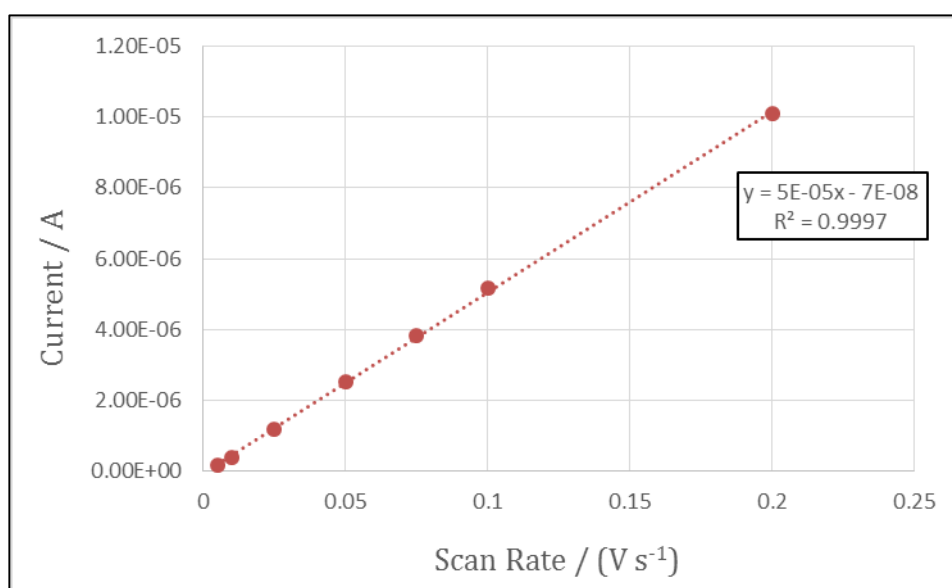
**Figure 68:** CVs recorded for large black silicon needles coated in a NCD film grown for 2 hours (Overgrown silicon needles) after electro spray pretreatment using a 1 M potassium nitrate solution.



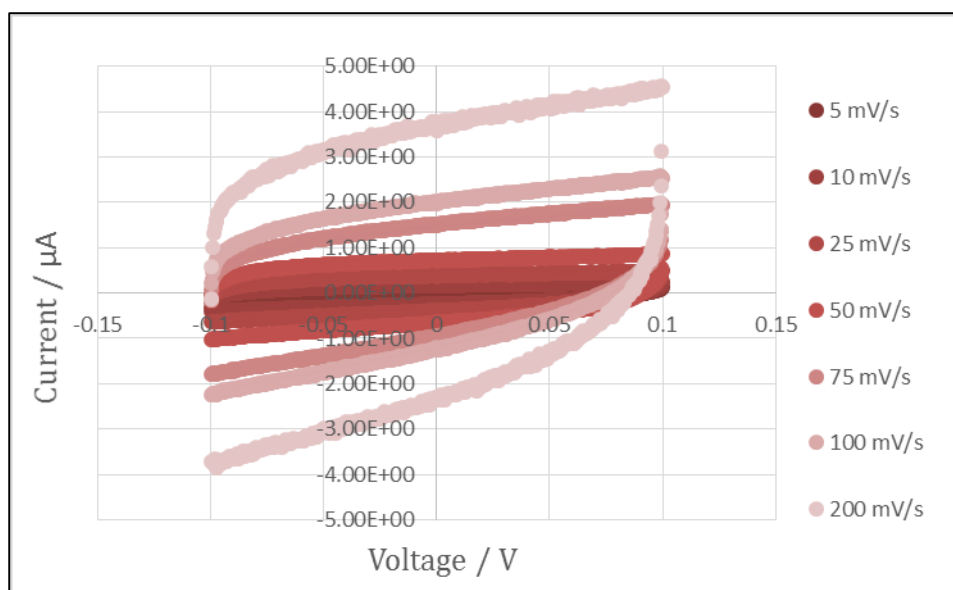
**Figure 69:** Plot of capacitive current vs scan rate for large black silicon needles coated in a NCD film grown for 2 hours (Overgrown silicon needles) after electro spray pretreatment.



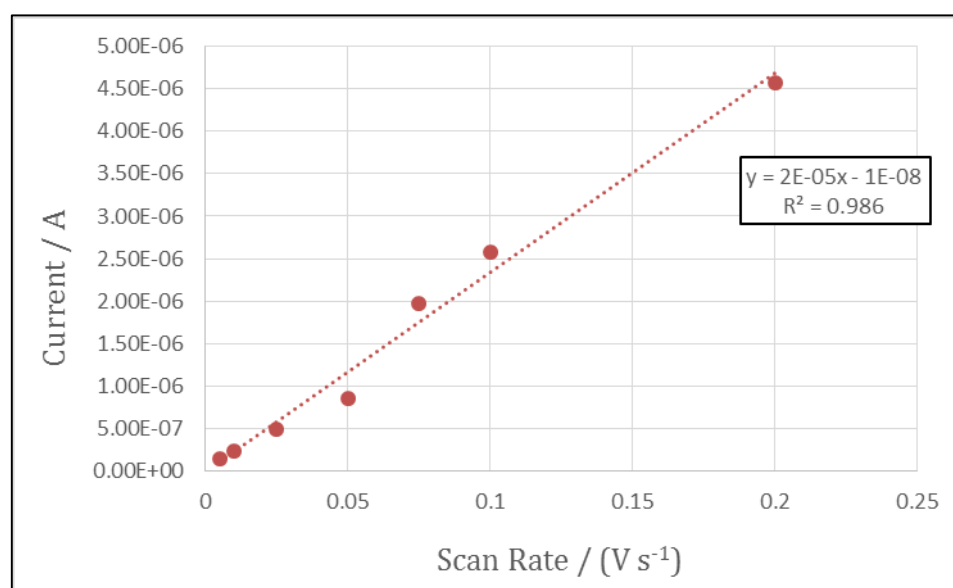
**Figure 70:** CVs recorded for large black silicon needles coated in a NCD film grown for 3 hours (Overgrown silicon needles) after electro spray pretreatment using a 1 M potassium nitrate solution.



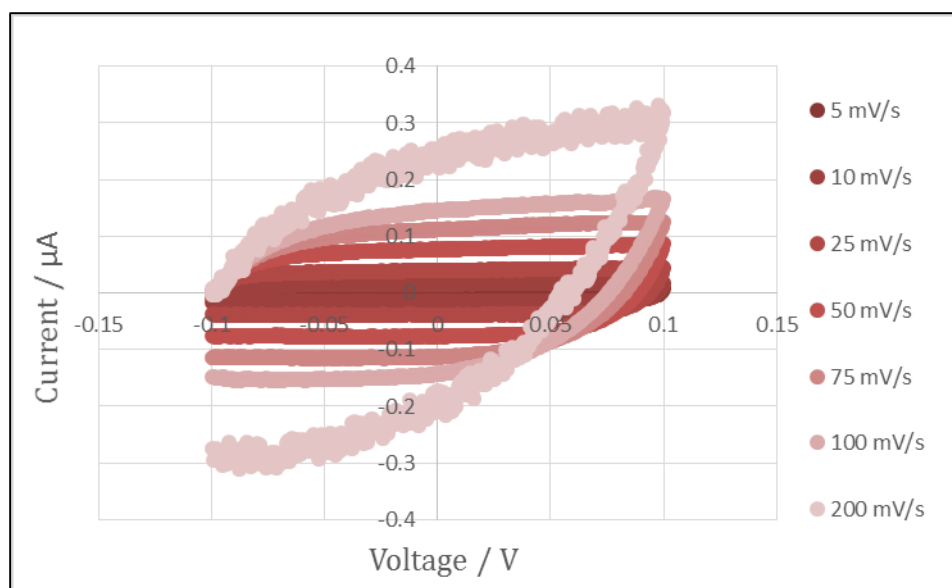
**Figure 71:** Plot of capacitive current vs scan rate for large black silicon needles coated in a NCD film grown for 3 hours (Overgrown silicon needles) after electro spray pretreatment.



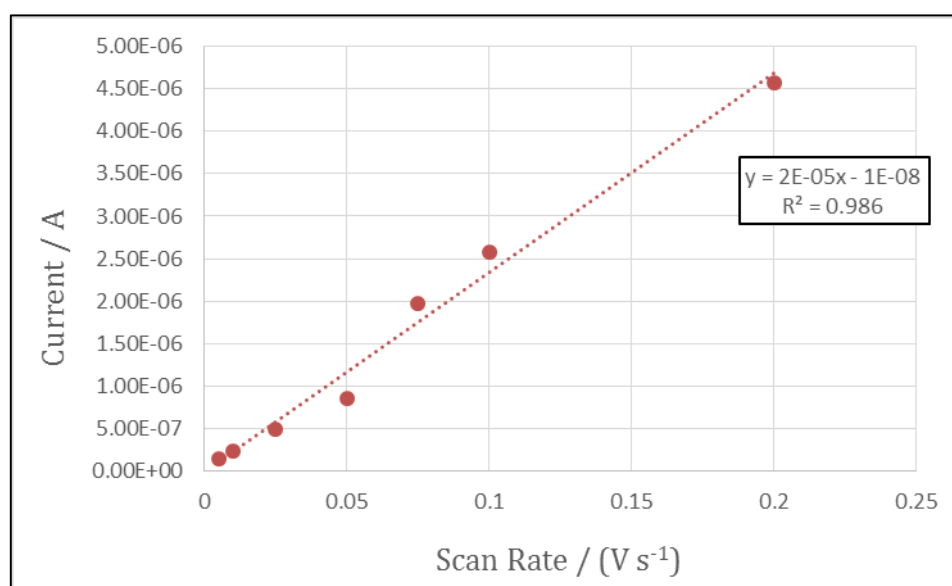
**Figure 72:** CVs recorded for large black silicon needles coated in a NCD film grown for 4 hours (Overgrown silicon needles) after electro spray pretreatment using a 1 M potassium nitrate solution.



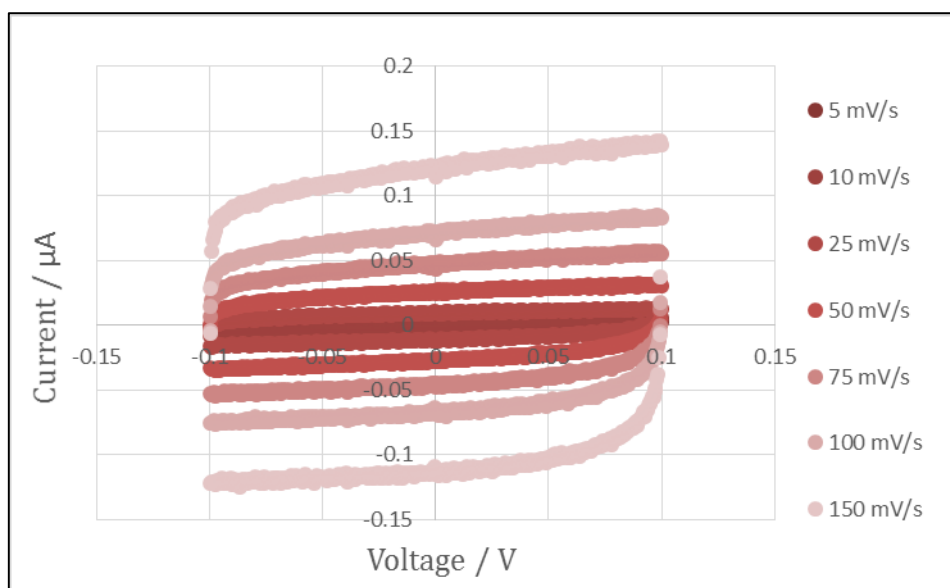
**Figure 73:** Plot of capacitive current vs scan rate for large black silicon needles coated in a NCD film grown for 4 hours (Overgrown silicon needles) after electro spray pretreatment.



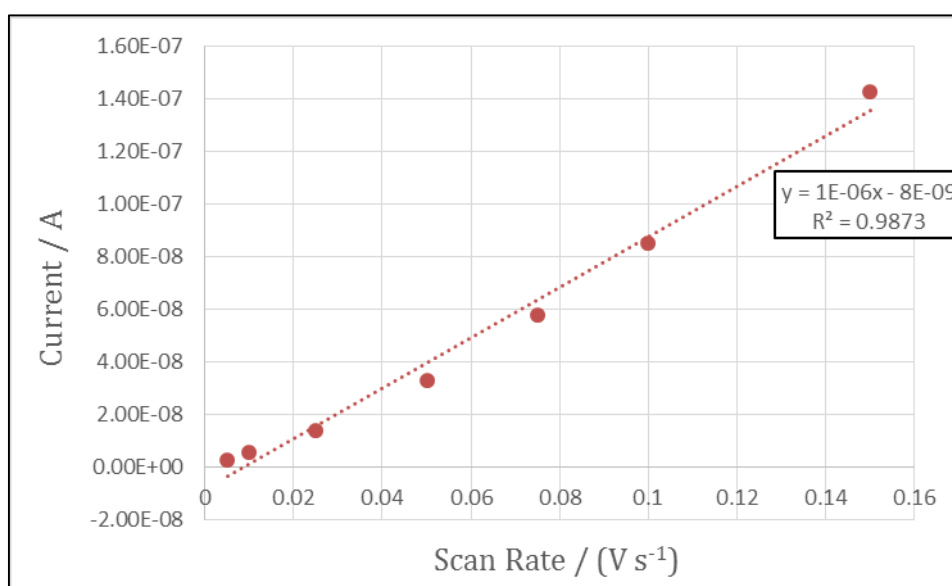
**Figure 74:** CVs recorded for small black silicon needles coated in a MCD film grown for 45 mins using a 1 M potassium nitrate solution.



**Figure 75:** Plot of capacitive current vs scan rate for small black silicon needles coated in a MCD film grown for 45 mins.

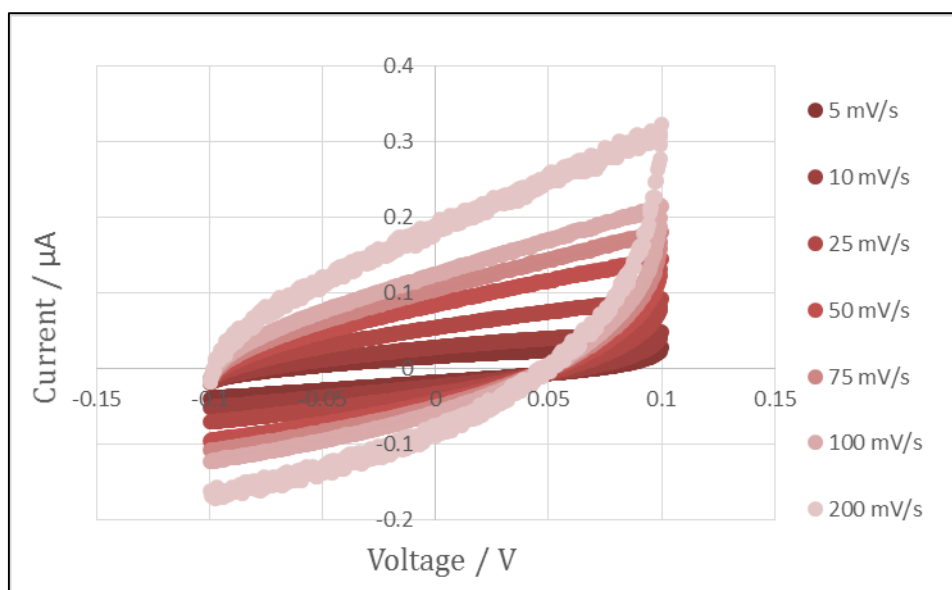


**Figure 76:** CVs recorded for small black silicon needles coated in a MCD film grown for 2 hours using a 1 M potassium nitrate solution.

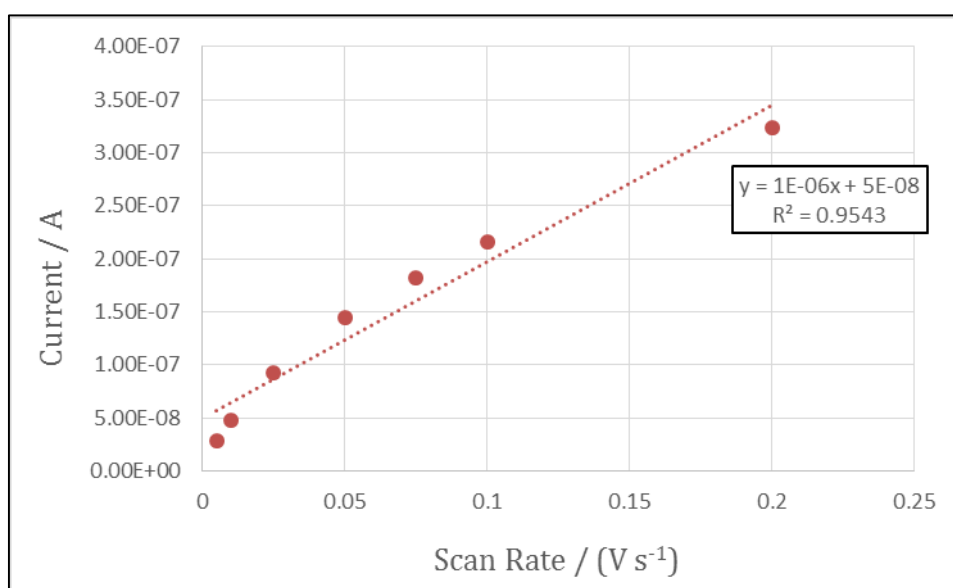


**Figure 77:** Plot of capacitive current vs scan rate for small black silicon needles coated in a MCD film grown for 2 hours.

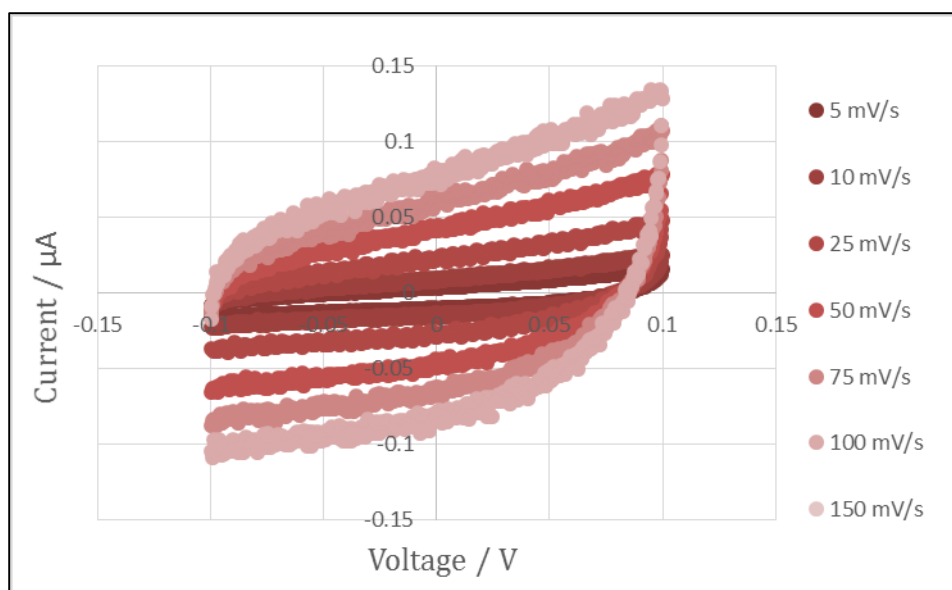




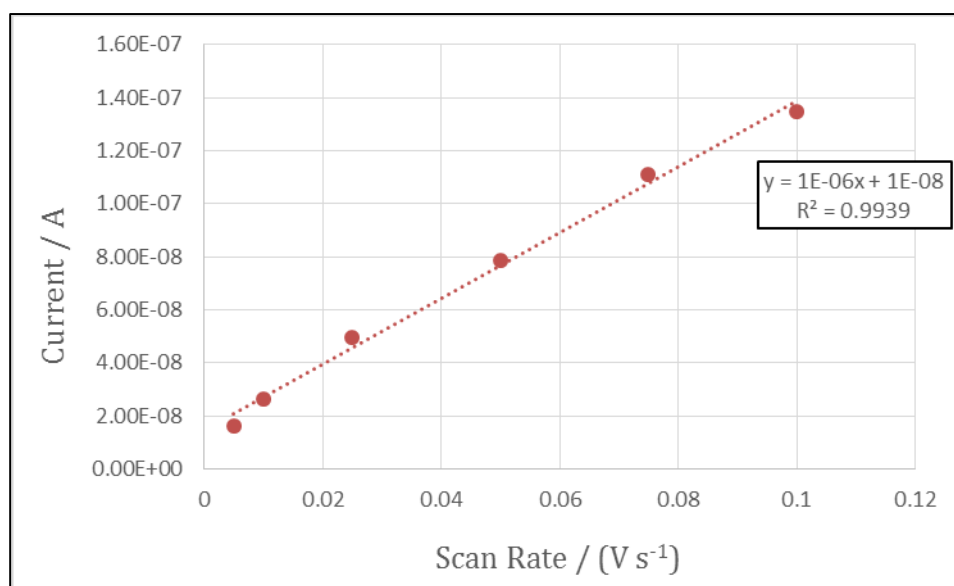
**Figure 78:** CVs recorded for small black silicon needles coated in a NCD film grown for 1 hour using a 1 M potassium nitrate solution.



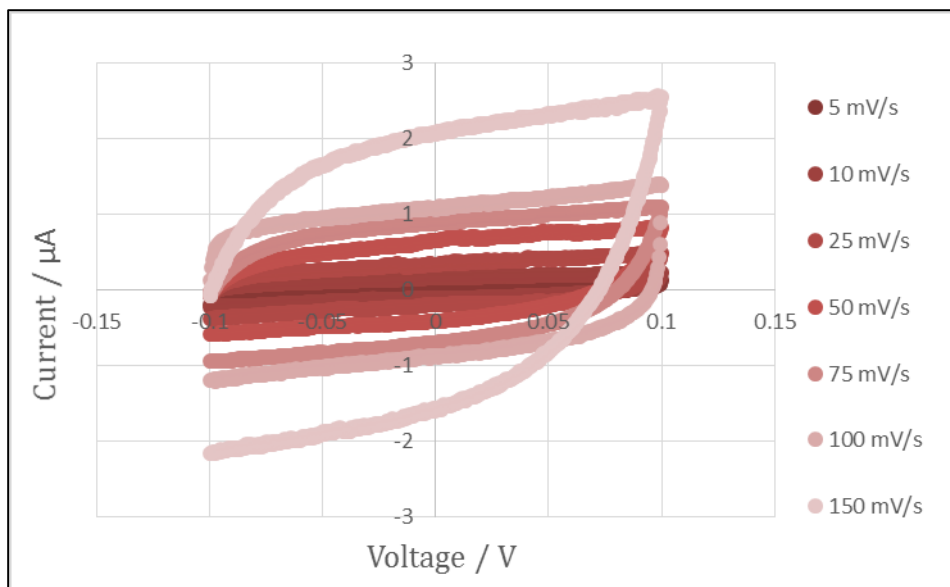
**Figure 79:** Plot of capacitive current vs scan rate for small black silicon needles coated in a NCD film grown for 1 hour.



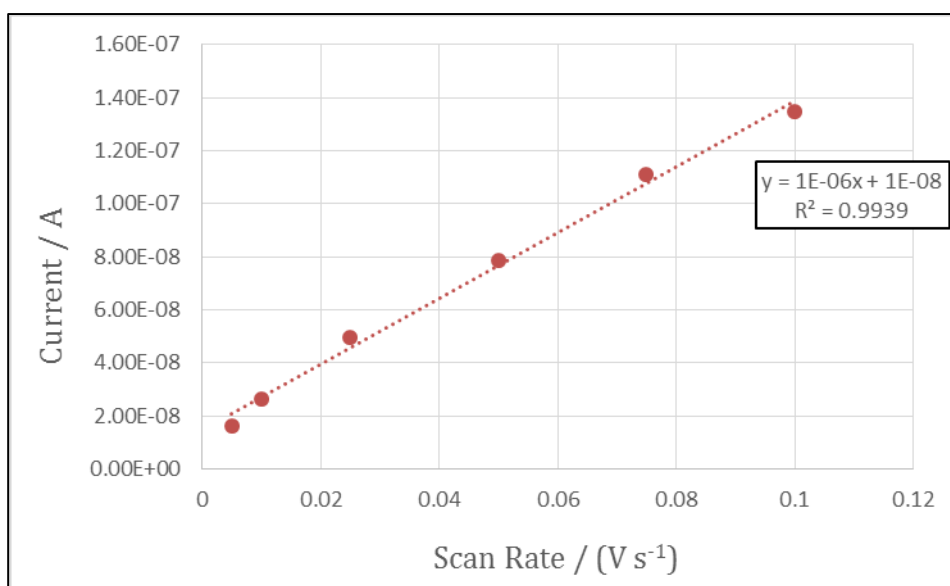
**Figure 80:** CVs recorded for VACNT teepees coated in a MCD film grown for 45 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



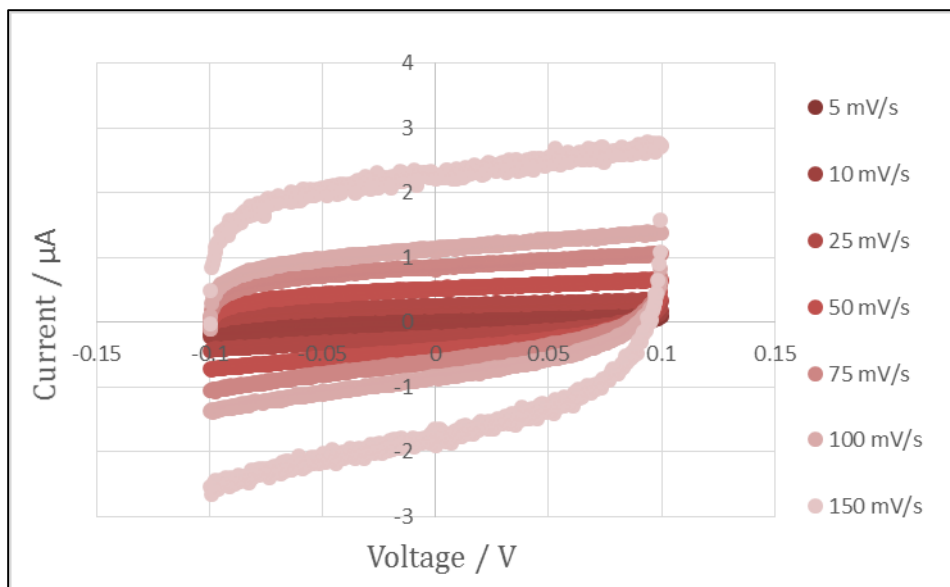
**Figure 81:** Plot of capacitive current vs scan rate for VACNT teepees coated in a MCD film grown for 45 mins after electro spray pretreatment.



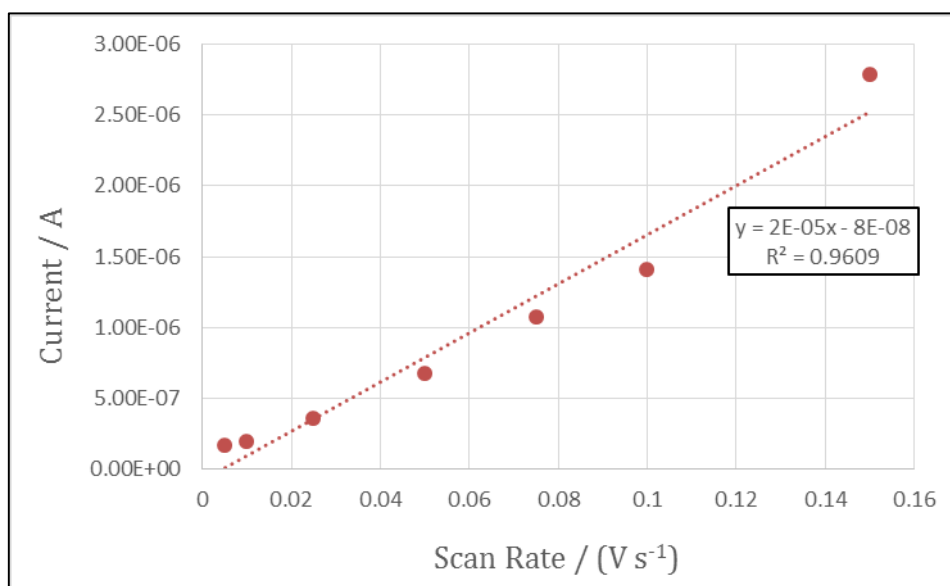
**Figure 82:** CVs recorded for CNT webs coated in a MCD film grown for 20 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



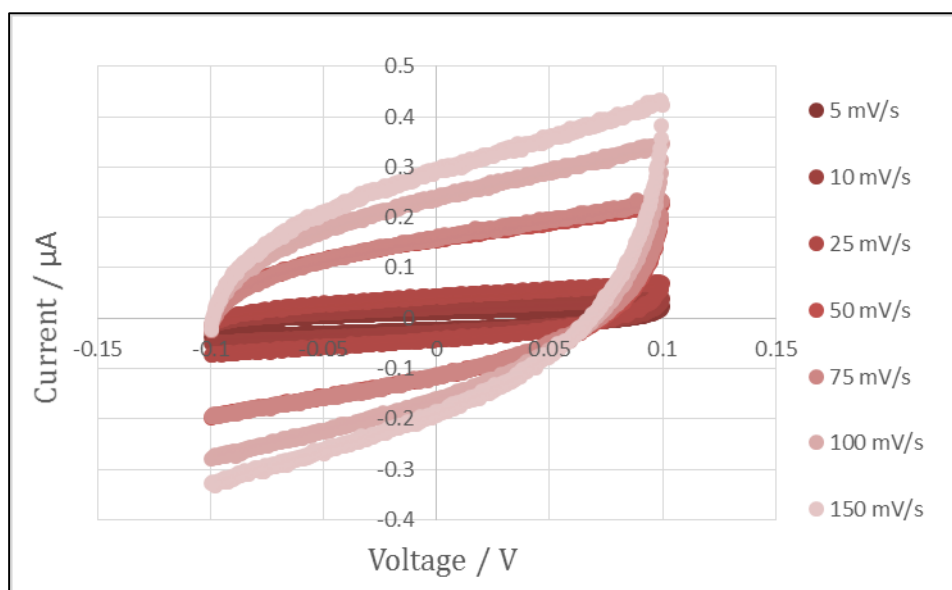
**Figure 83:** Plot of capacitive current vs scan rate for CNT webs coated in a MCD film grown for 20 mins after electro spray pretreatment.



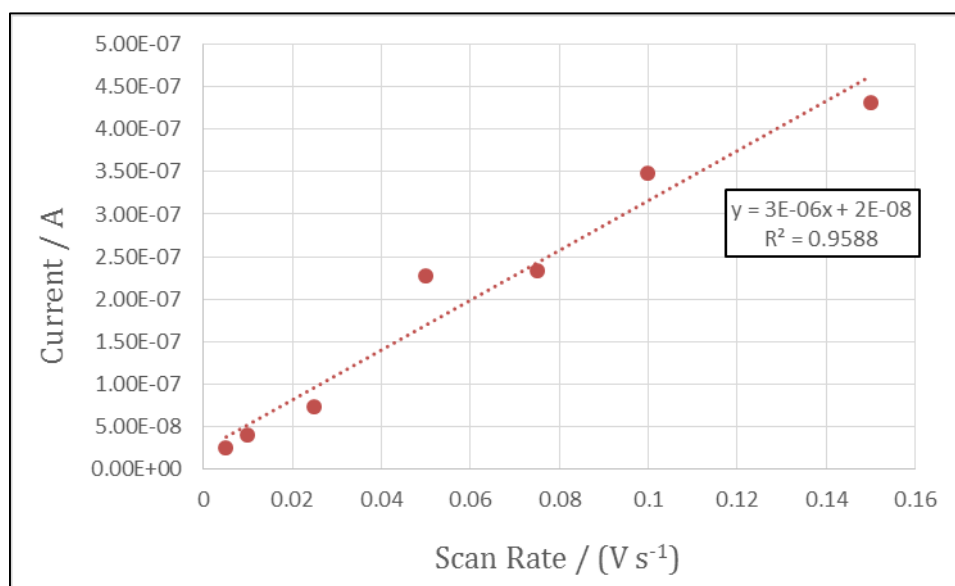
**Figure 84:** CVs recorded for CNT webs coated in a MCD film grown for 30 mins after electro spray pretreatment using a 1 M potassium nitrate solution.



**Figure 85:** Plot of capacitive current vs scan rate for CNT webs coated in a MCD film grown for 30 mins after electro spray pretreatment.

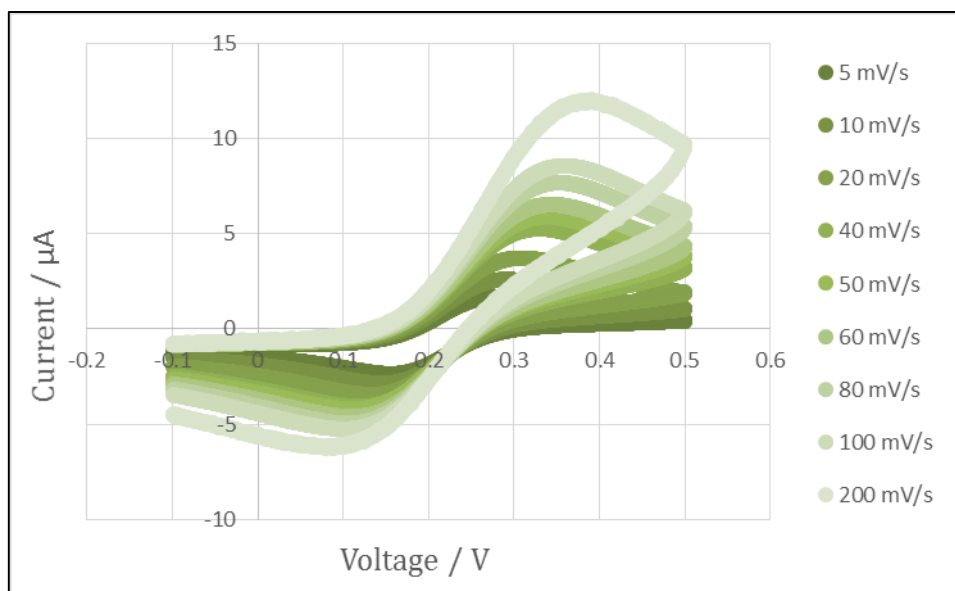


**Figure 86:** CVs recorded for CNT webs coated in a NCD film grown for 45 mins after electro spray pretreatment using a 1 M potassium nitrate solution.

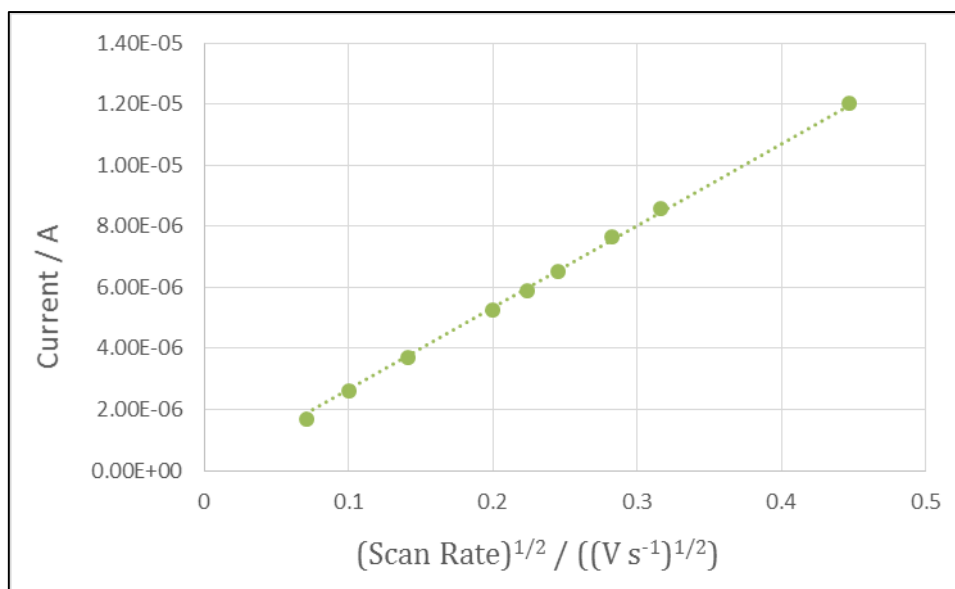


**Figure 87:** Plot of capacitive current vs scan rate for CNT webs coated in a NCD film grown for 45 mins after electro spray pretreatment.

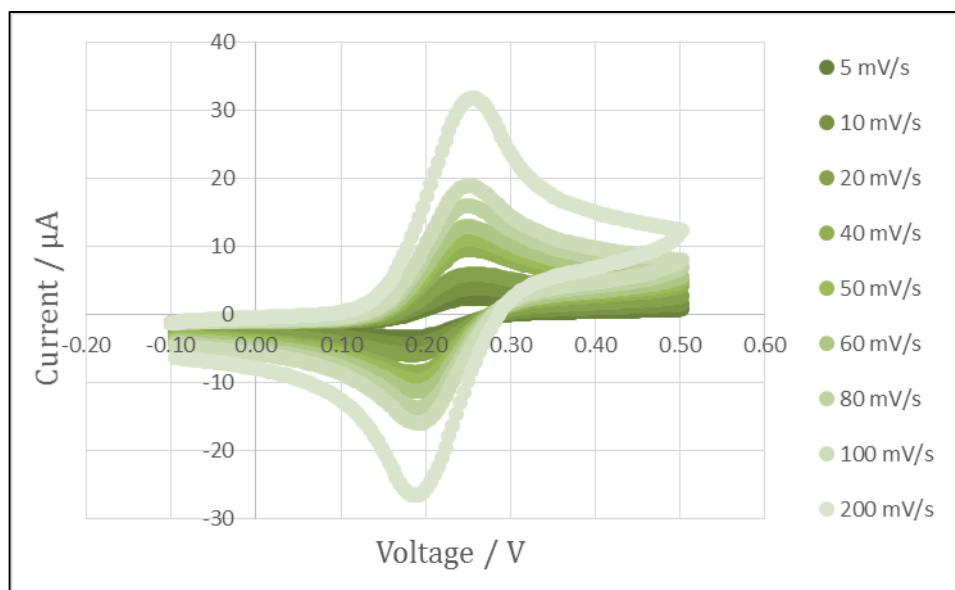
## Cyclic Voltammetry – $\text{K}_3[\text{Fe}(\text{CN})_6]$



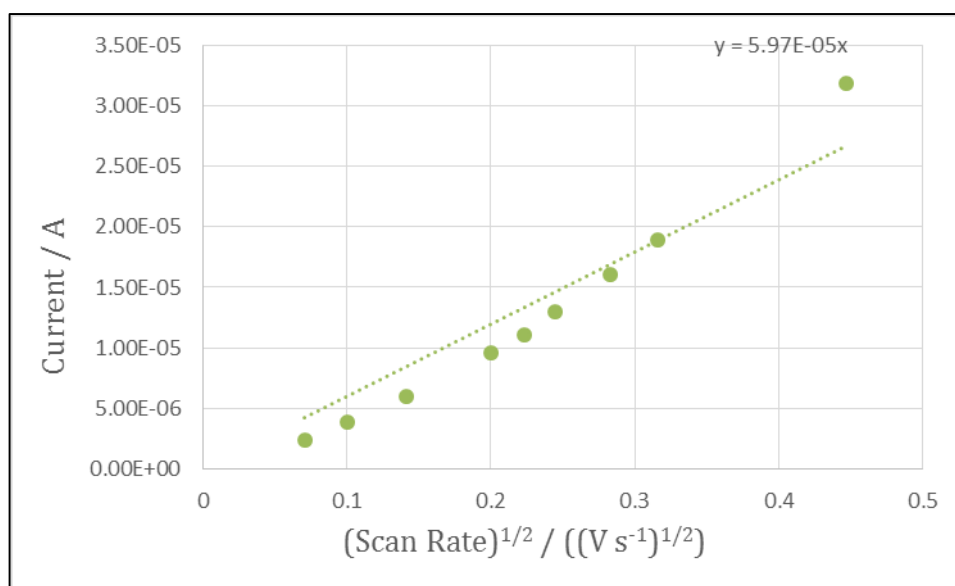
**Figure 88:** CVs recorded for flat diamond sample using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



**Figure 89:** Plot of peak current vs square root of scan rate for flat diamond sample using 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution CVs.

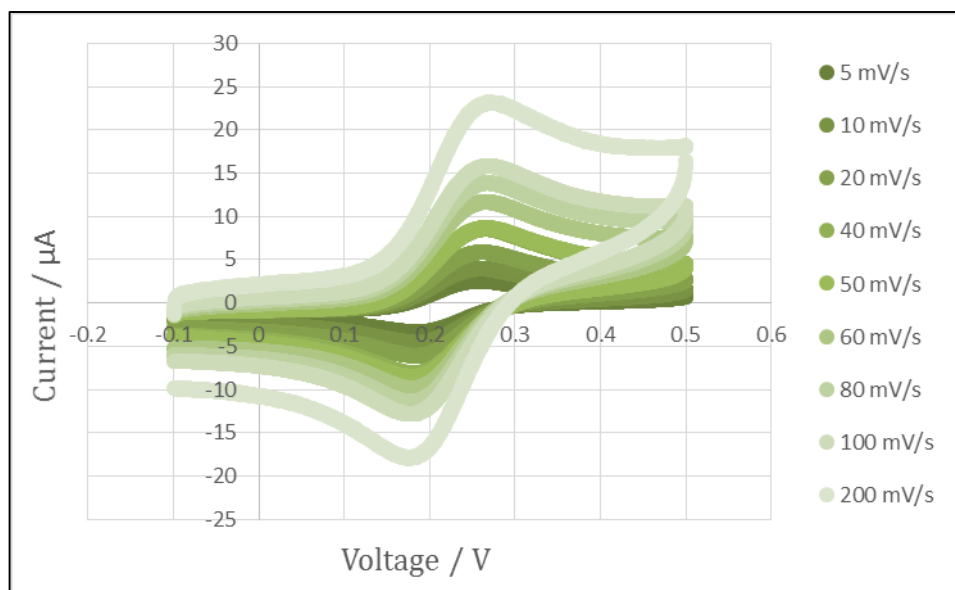


**Figure 90:** CVs recorded for large black silicon needles coated in a MCD film grown for 20 mins after electrospray pretreatment using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.

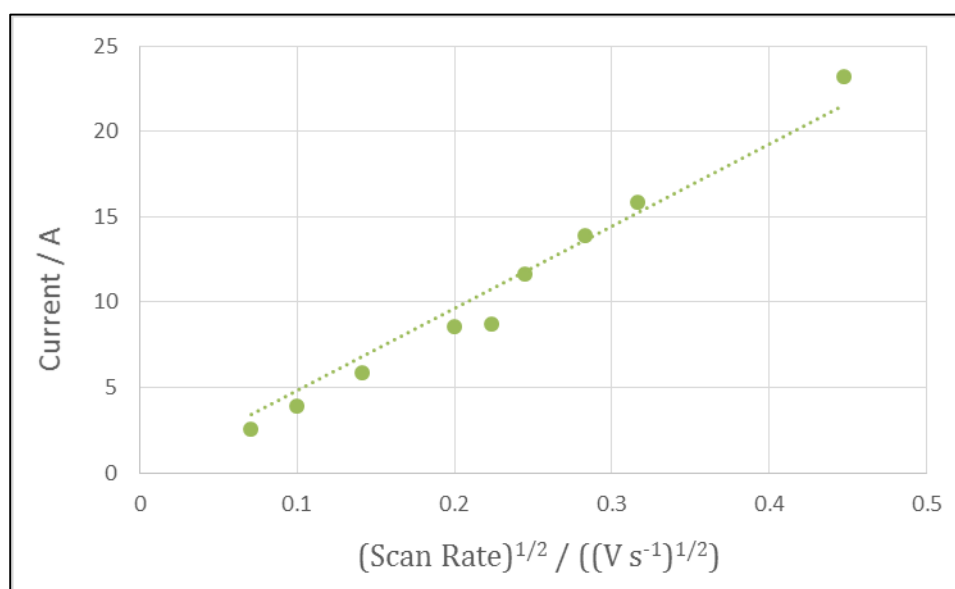


**Figure 91:** Plot of peak current vs square root of the scan rate for large black silicon needles coated in a MCD film grown for 20 mins after electrospray pretreatment using 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.

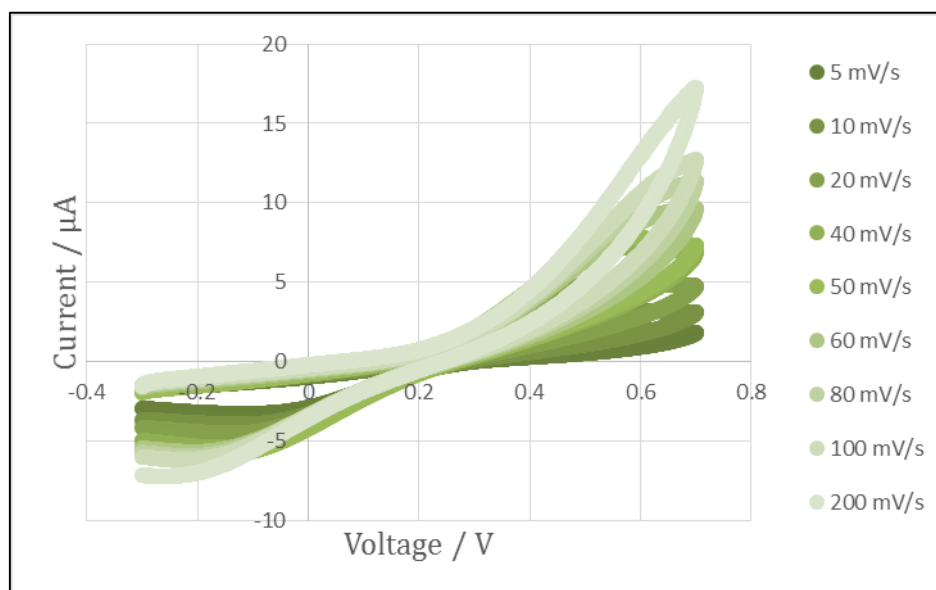




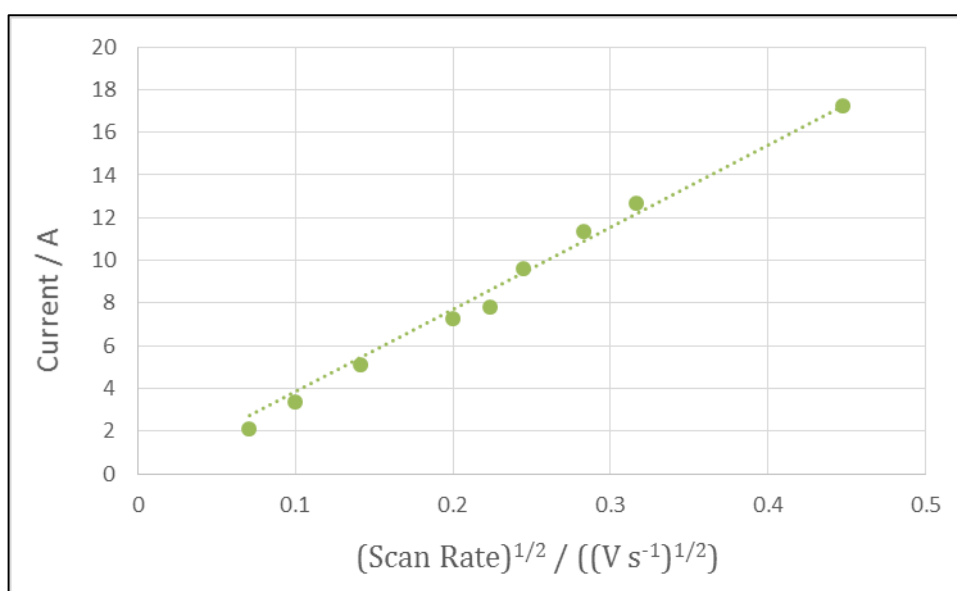
**Figure 92:** CVs recorded for large black silicon needles coated in a NCD film grown for 3 hours after electro spray pretreatment (overgrown needles) using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



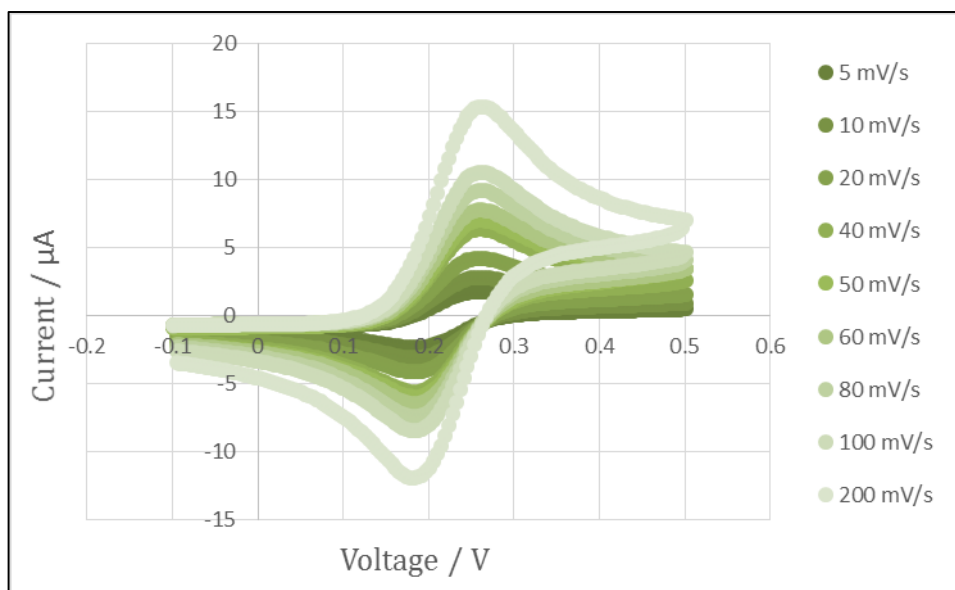
**Figure 93:** Plot of peak current vs square root of the scan rate for large black silicon needles coated in a NCD film grown for 3 hours after electro spray pretreatment (overgrown needles) using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution



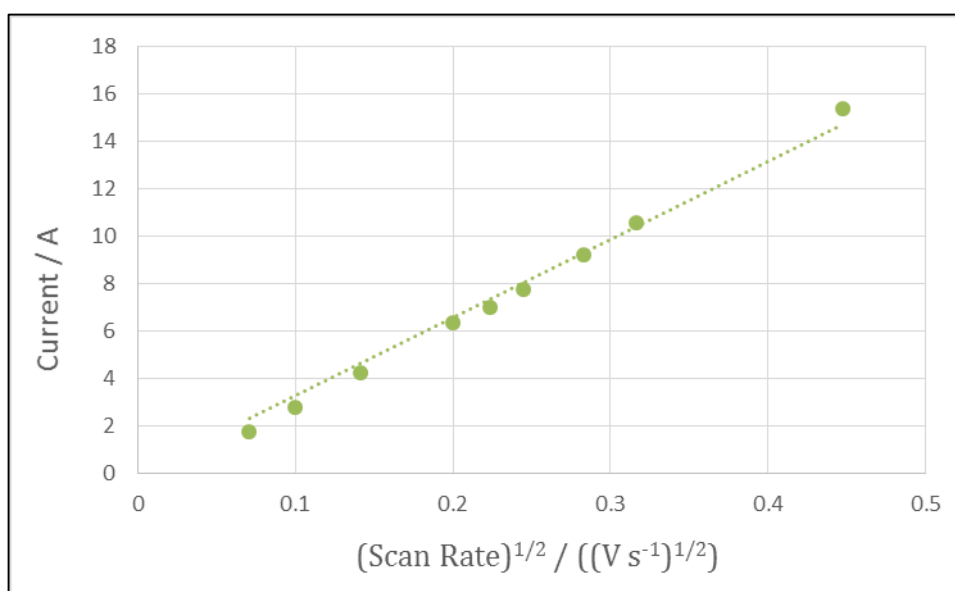
**Figure 94:** CVs recorded for small black silicon needles coated in a MCD film grown for 2 hours using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



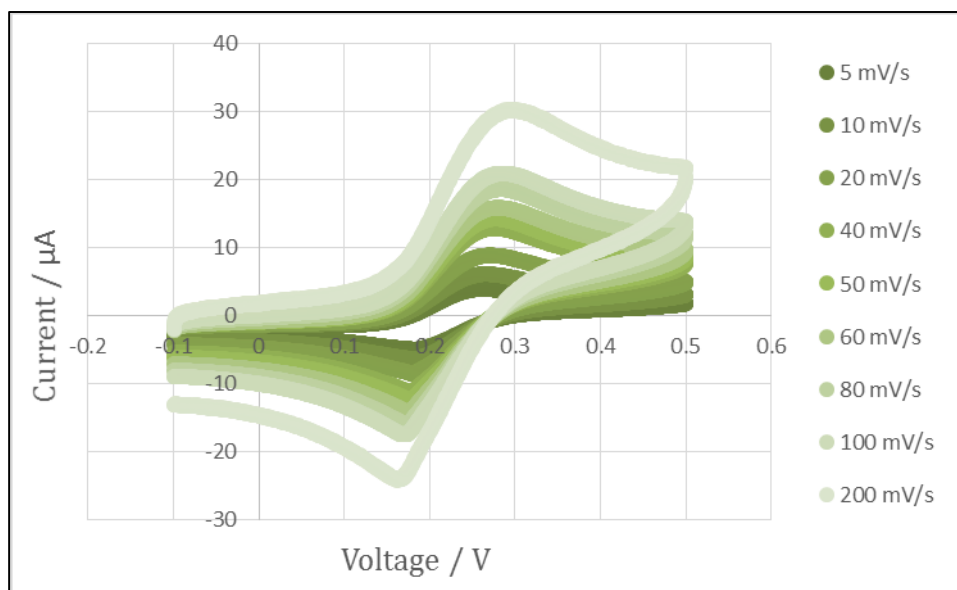
**Figure 95:** Plot of peak current vs square root of the scan rate for small black silicon needles coated in a MCD film grown for 2 hours using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



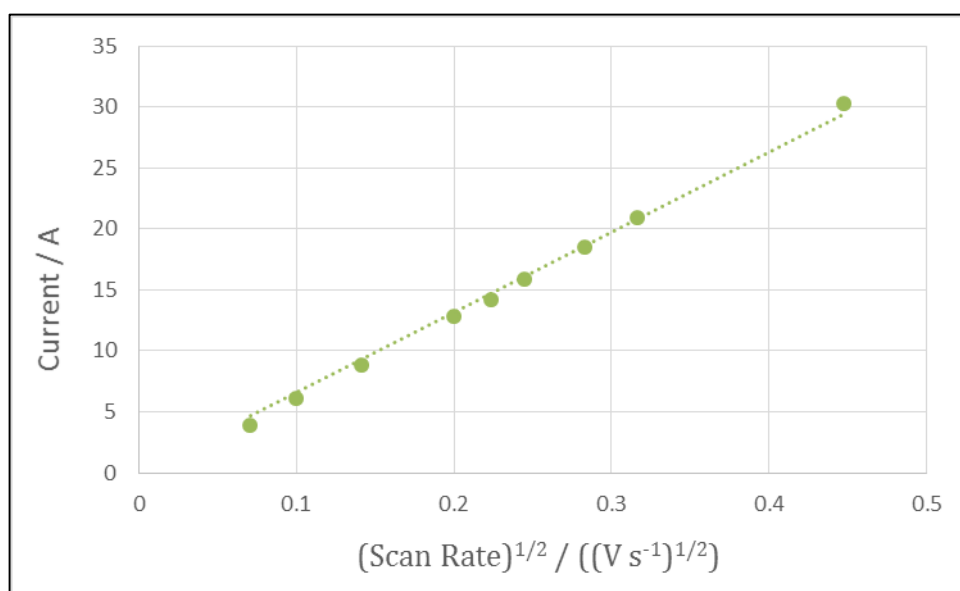
**Figure 96:** CVs recorded for VACNT teepees in a MCD film grown for 45 mins after electrospray pretreatment using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



**Figure 97:** Plot of peak current vs square root of the scan rate VACNT teepees in a MCD film grown for 45 mins after electrospray pretreatment using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



**Figure 98:** CVs recorded for CNT webs coated in a MCD film grown for 30 mins after electrospray pretreatment using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.



**Figure 99:** Plot of peak current vs square root of the scan rate for CNT webs coated in a MCD film grown for 30 mins after electrospray pretreatment using a 1 mM potassium ferricyanide with 1 M potassium nitrate supporting electrolyte solution.