Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

Nickel nanoparticles grown by successive ionic layer adsorption and reaction method for ethanol electrooxidation and electrochemical quartz crystal microbalance study

Akshay Nandkishor Vyas[1], Mangesh Appasaheb Desai[1], Deodatta Moreshwar Phase[2], Rijuta Ganesh Saratale[3], Jalindar Dnyandeo Ambekar[4], Bharat Bhanudas Kale[4], Habib Mohiddin Pathan[1] and Shrikrishna Dattatraya Sartale[1,]*

[1]Thin Films and Nanomaterials Laboratory, Department of Physics, Savitribai Phule Pune University, Pune, India – 411 007
[2]UGC-DAE Consortium for Scientific Research, Indore, India – 452 001
[3]Research Institute of Biotechnology and Medical Converged Science, Dongguk University-Seoul, Ilsandong-gu, Goyang-si, Gyeonggi-do, 10326, Republic of Korea
[4]Centre for Materials for Electronics Technology (C-MET), Pune, India – 411 008

*Corresponding Author's Address:

Dr. Shrikrishna D. Sartale
Thin Films and Nanomaterials Laboratory,
Department of Physics,
Savitribai Phule Pune University,
Pune, India – 411 007
Email: sdsartale@physics.unipune.ac.in

Schematic of SILAR

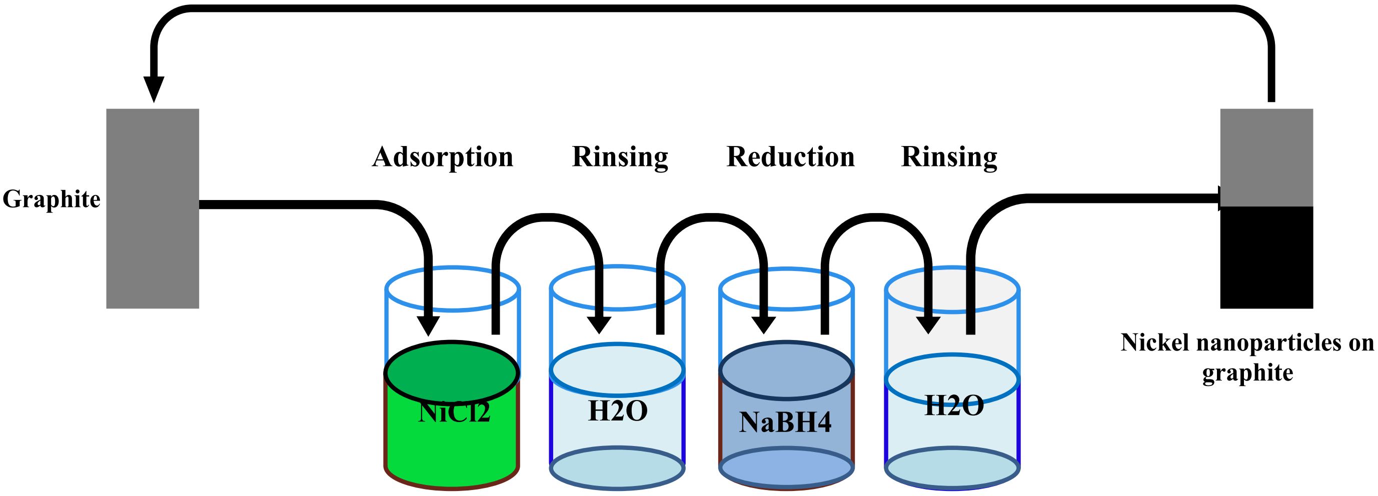

Figure S1 Schematic of one complete SILAR cycle

Use of sodium hypophosphite as reducing agent

We tried sodium hypophosphite as the reducing agent solution for growing nickel nanoparticles on graphite but it was observed that the onset potential for ethanol electrooxidation shifted towards more positive potentials after using sodium hypophosphite. The current density values were also less as compared to when sodium borohydride was used as reducing agent. As an example the behavior of nickel nanoparticles, prepared using 0.5 M $NiCl_2$, for ethanol electrooxidation is shown in figure S2.

Our experiments for nickel nanoparticles growth also showed that sodium borohydride acts as a powerful reducing agent and reduces the nickel ions readily as compared to sodium hypophosphite. Reduction of nickel ions using sodium hypophosphite requires very high basic conditions (which could give rise to $Ni(OH)_2$formation) and comparatively high temperature. Moreover, the probability of presence of phosphorous in the final product giving Ni-P instead of pure nickel can also not be neglected. Considering these facts we decided to select sodium borohydride as a reducing agent for this study.

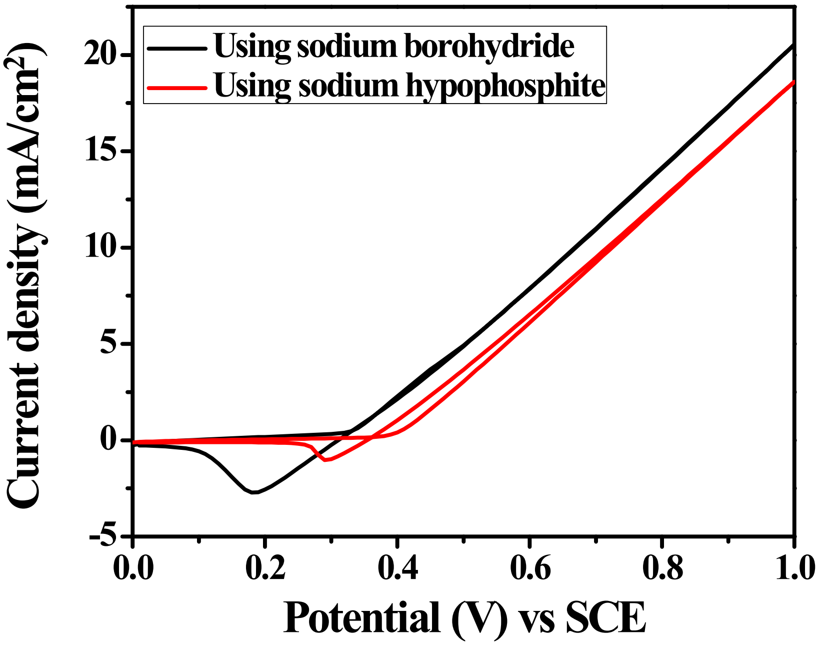

Figure S2 Comparison of cyclic voltammograms of nickel nanoparticles grown on graphite for two SILAR growth cycles using 0.1 M sodium borohydride and sodium hypophosphite reducing agents. 0.5 M $NiCl_2$ was used as nickel ions precursor. CVs were recorded in 0.5 M NaOH and 0.5 M ethanol solution at 50 mV/s scan rate.

XRD study of Ni nanoparticles

The XRD patterns of bare graphite and nickel nanoparticles grown by high (1 M) nickel chloride concentration and large number (~ 300) of SILAR growth cycles are shown below (Figure S3). It is evident from XRD of grown nickel nanoparticles that the intensity of peaks representing graphite becomes smaller and newer peaks corresponding to fcc nickel are emerged. The diffraction peak at ~ 44 degree corresponding to (111) plane of nickel exactly coincides with the diffraction peak of graphite. After growth of nickel nanoparticles in contrast to other peaks of graphite this peak becomes more prominent thus indicating the growth of nickel.

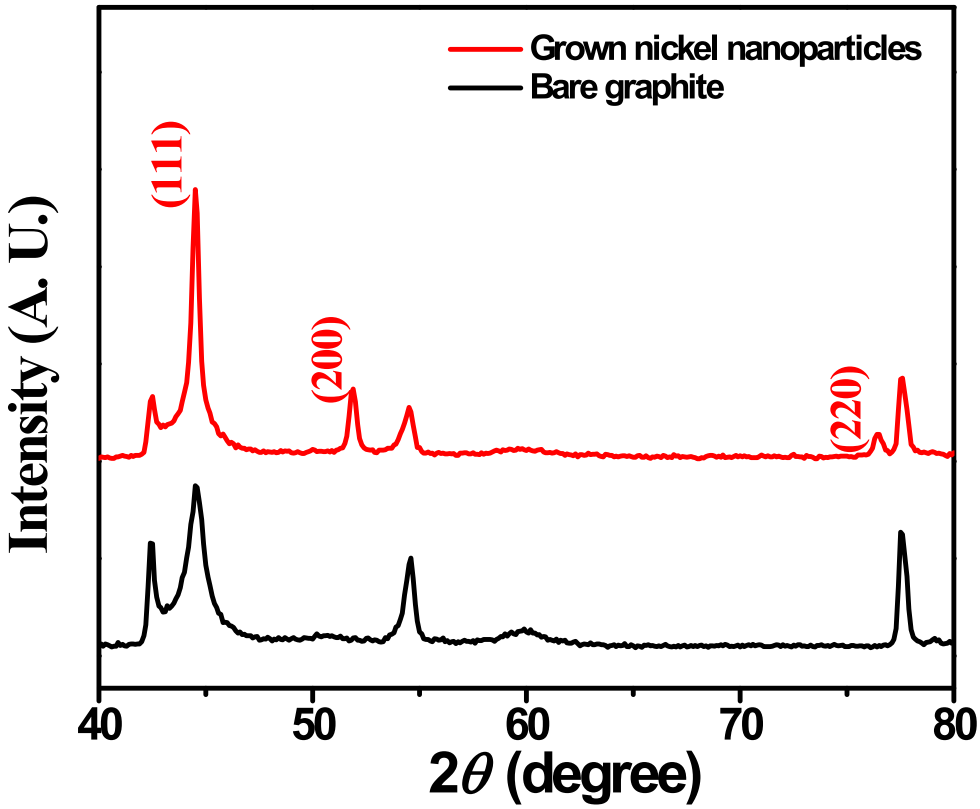

Figure S3 XRD of bare graphite and grown nickel nanoparticles.

Potential scan rate variation

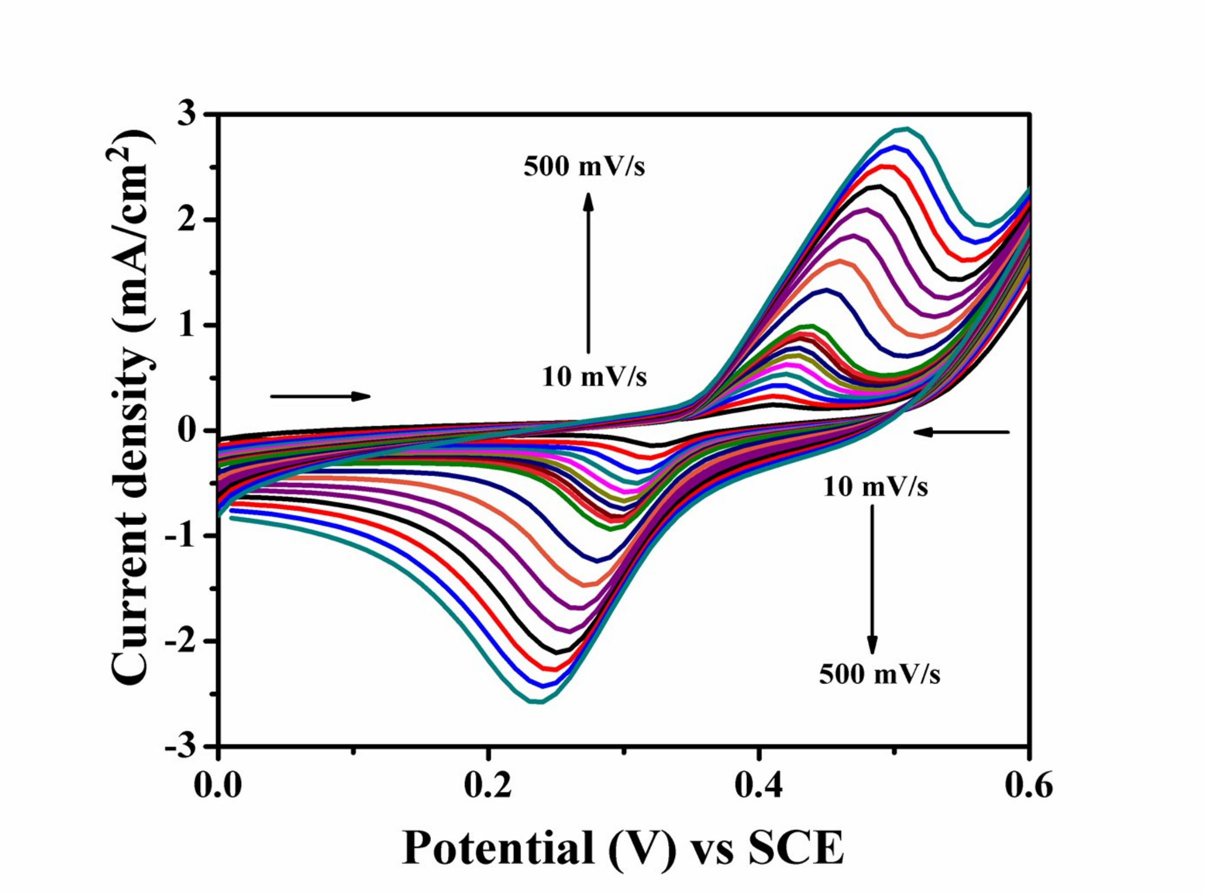

Figure S4 CVs recorded in 0.5 M NaOH solution with varying potential scan rates from 10 to 500 mV/s of nickel nanoparticles; grown on graphite substrates by taking two SILAR growth cycles for 0.005 M $NiCl_2$ concentration.

Nickel nanoparticles grown on gold coated quartz crystal

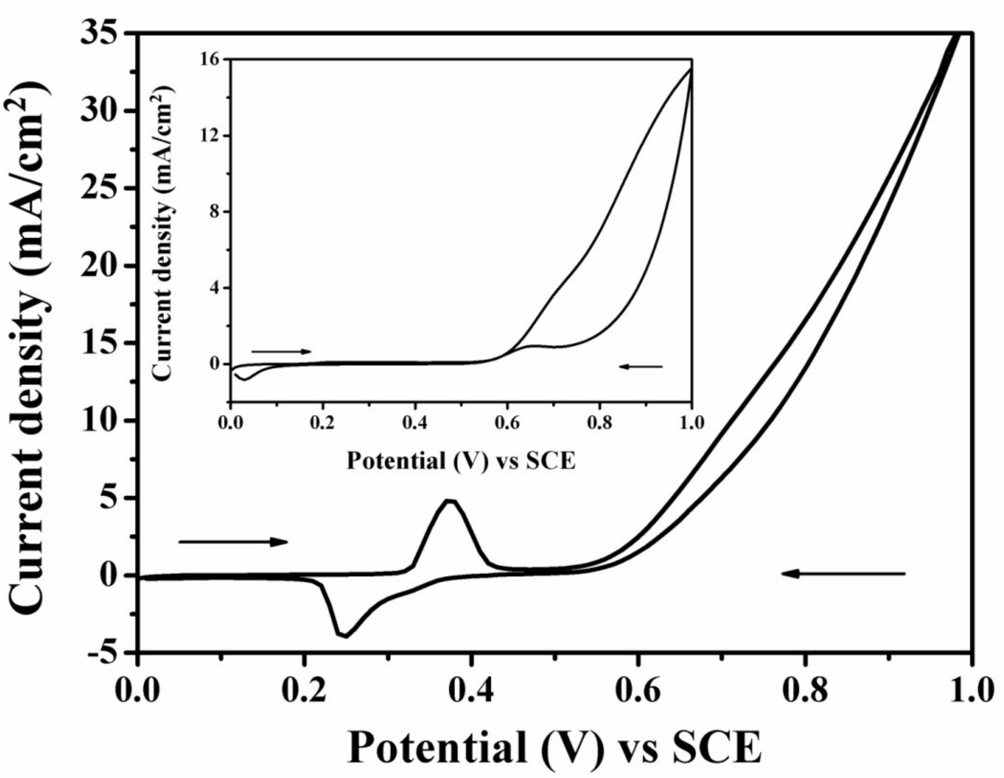

Figure S5 CV of nickel nanoparticles grown on gold coated quartz crystal by taking 1.0 M $NiCl_2$ concentration for 10 SILAR growth cycles in the presence of 0.5 M NaOH solution at 50 mV/s scan rate. (**Inset**: CV of bare gold coated quartz crystal in 0.5 M NaOH solution)