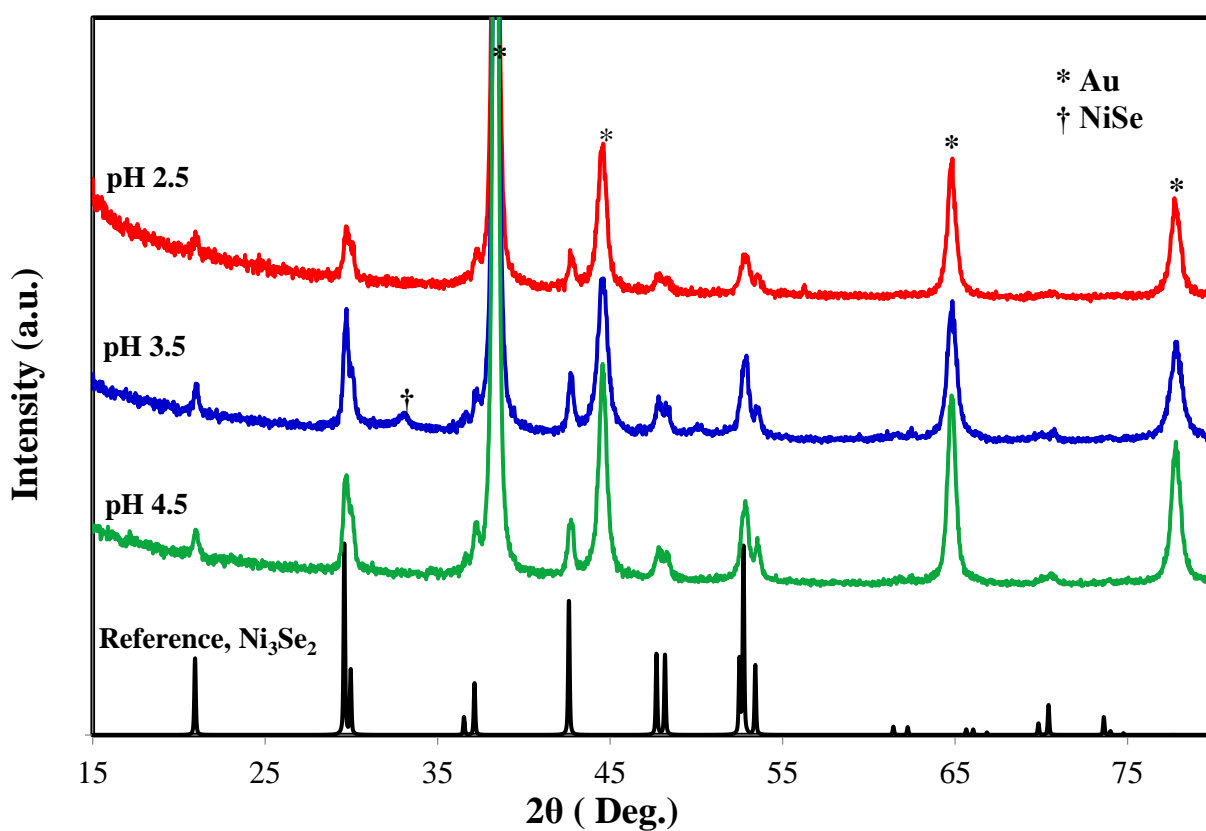
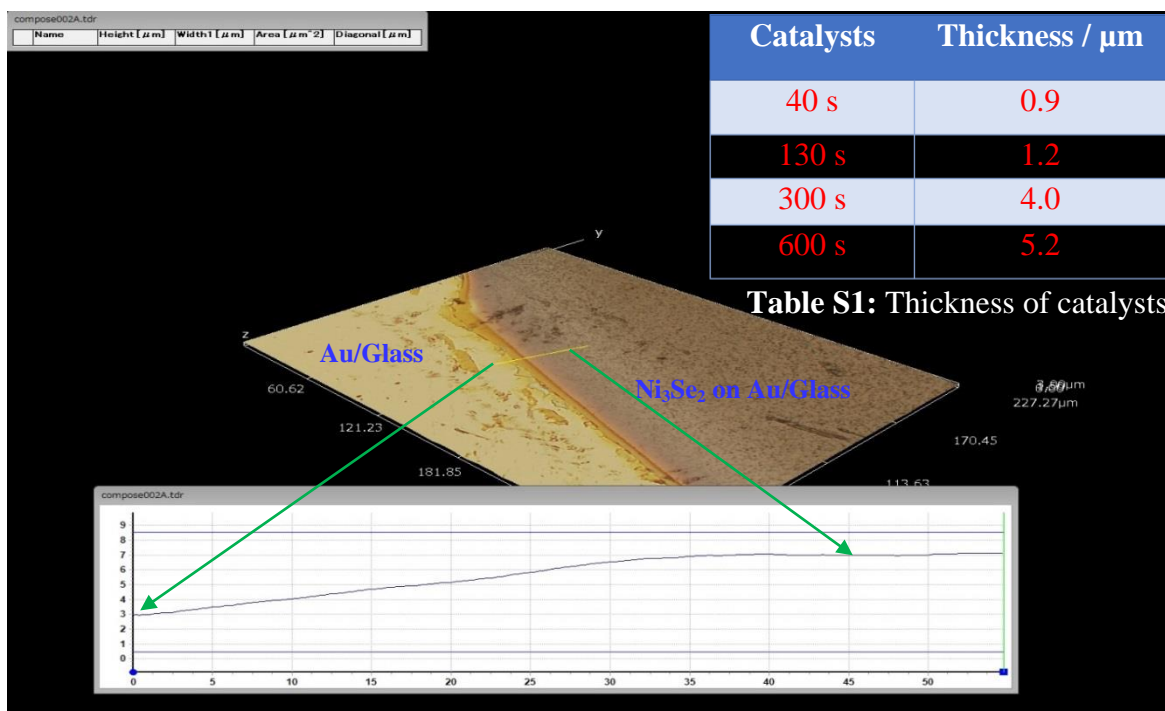


# Nickel Selenide As High-efficiency Catalyst for Oxygen Evolution Reaction

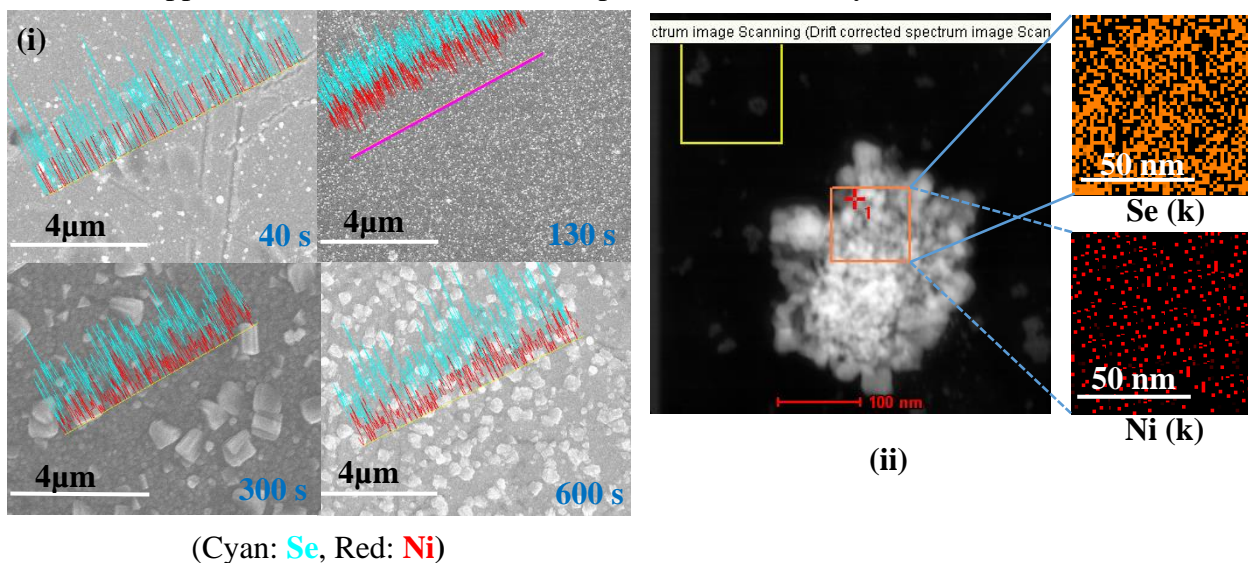
A. T. Swesi, J. Masud and M. Nath\*



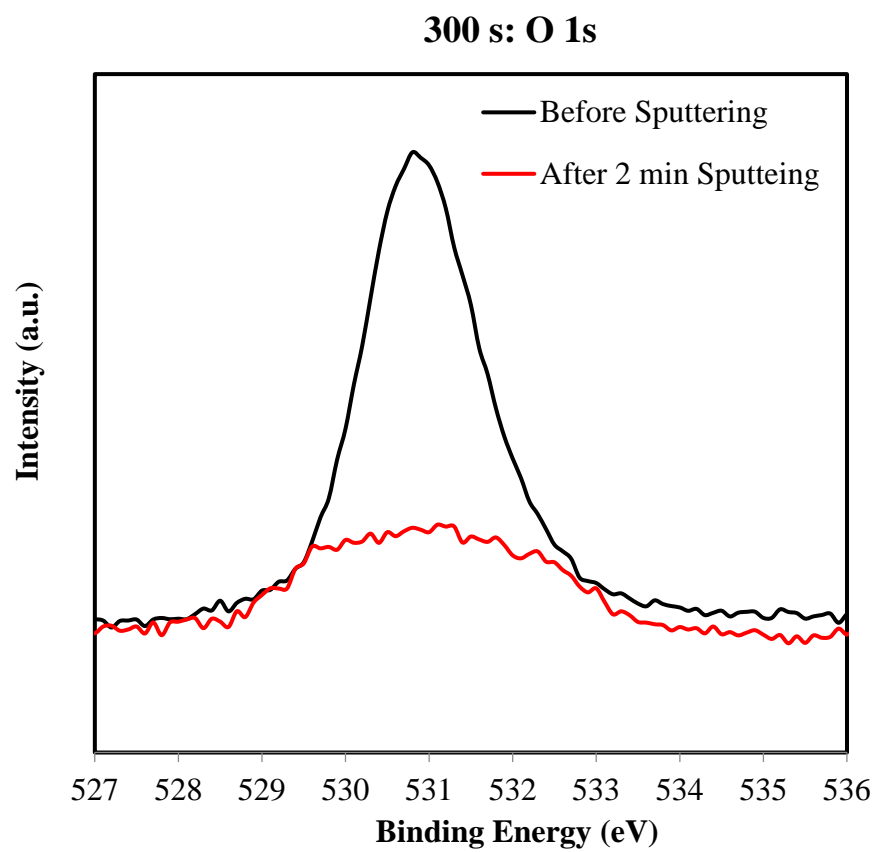
**Figure S1.** Pxr patterns of catalysts deposited for 300 s at different pH of the electrochemical bath.



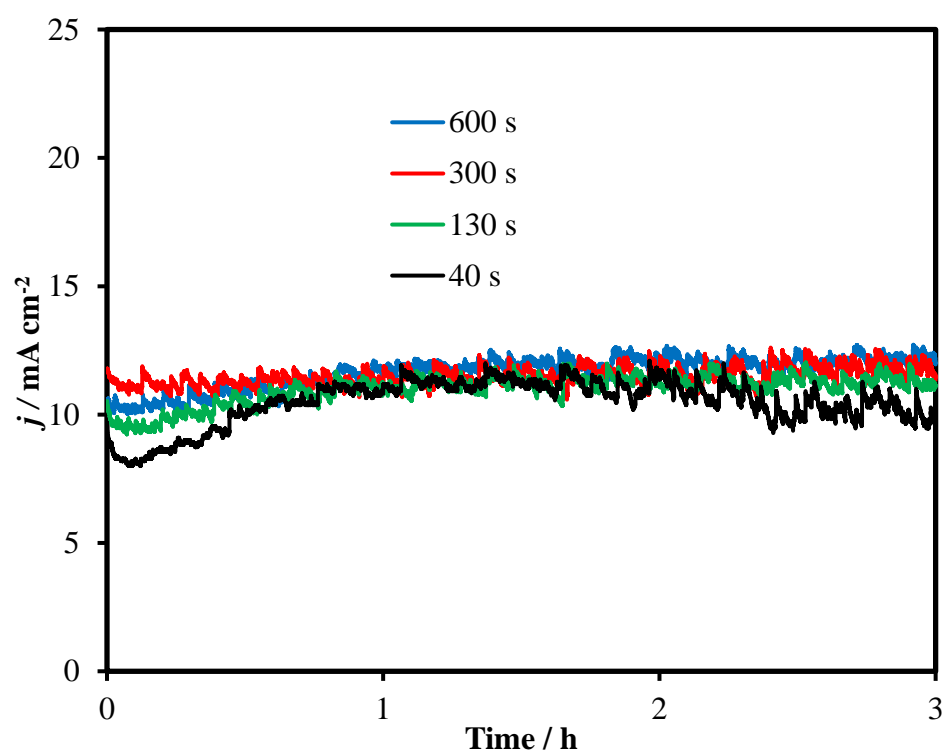
**Figure S2a.** 3D imaging and 3D profile of catalyst synthesized for 300 s. Table shows the thickness of catalysts that were synthesized for different time. The HIROX KH-8700 Digital Microscope, is the next generation system for the high precision of measurement and 3D profiling, was used to measure the approximate thickness of electrodeposited  $\text{Ni}_3\text{Se}_2$  catalyst on Au/Glass.



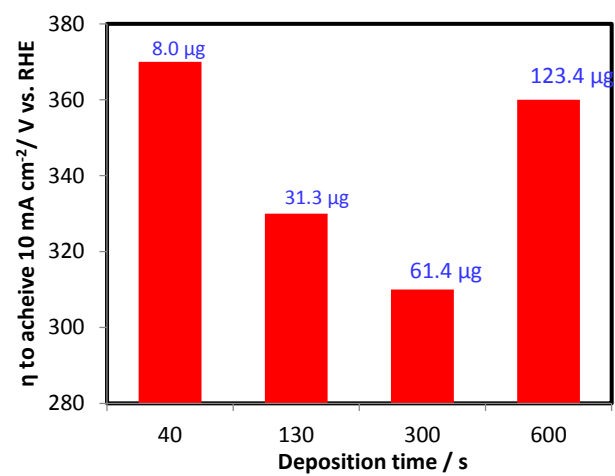
**Figure S2b.** (i) EDS line scanning of catalysts and (ii) HAADF image of electrodeposited  $\text{Ni}_3\text{Se}_2$  along with the elemental mapping of Ni and Se from a specific area within the cluster.



**Figure S3.** XPS spectra of O 1s peak obtained from the catalyst before and after 2 min sputtering.



**Figure S4.** Current transient recorded at a constant overpotential to achieve  $10 \text{ mA/cm}^2$ .

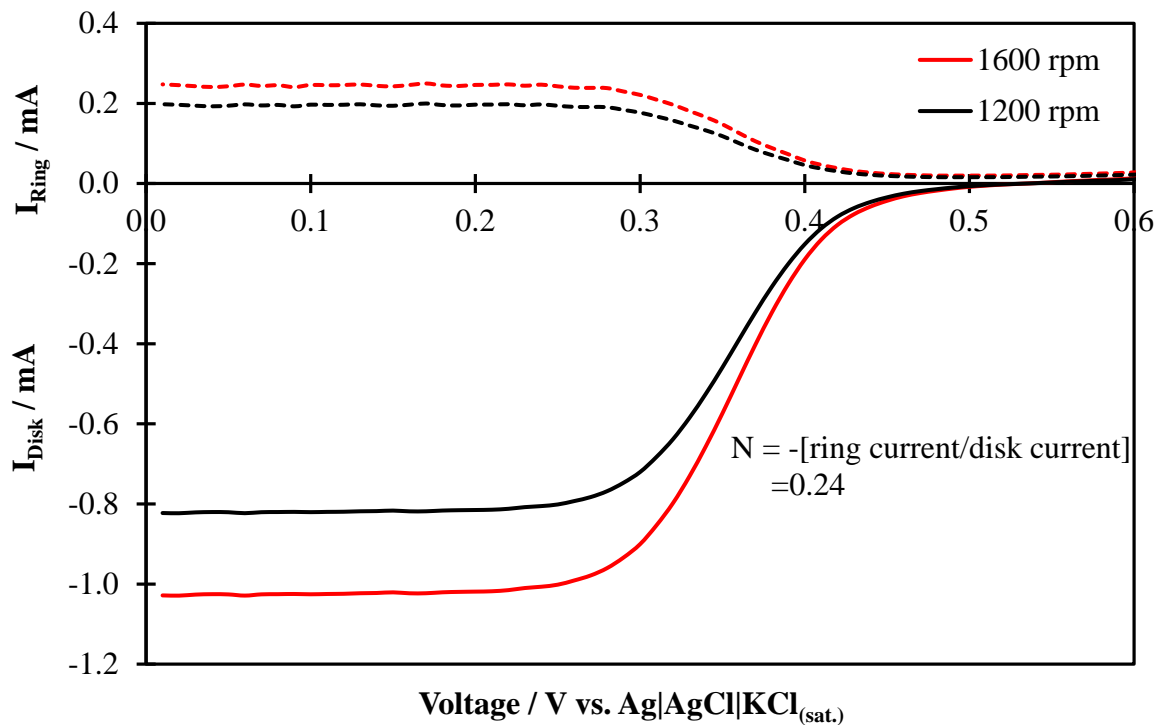


**Figure S5.** Overpotential required to achieve 10 mA cm<sup>-2</sup> for different deposition time of the catalyst. Total catalyst loading for different deposition times are shown on the top of the column.

### Collection Efficiency:

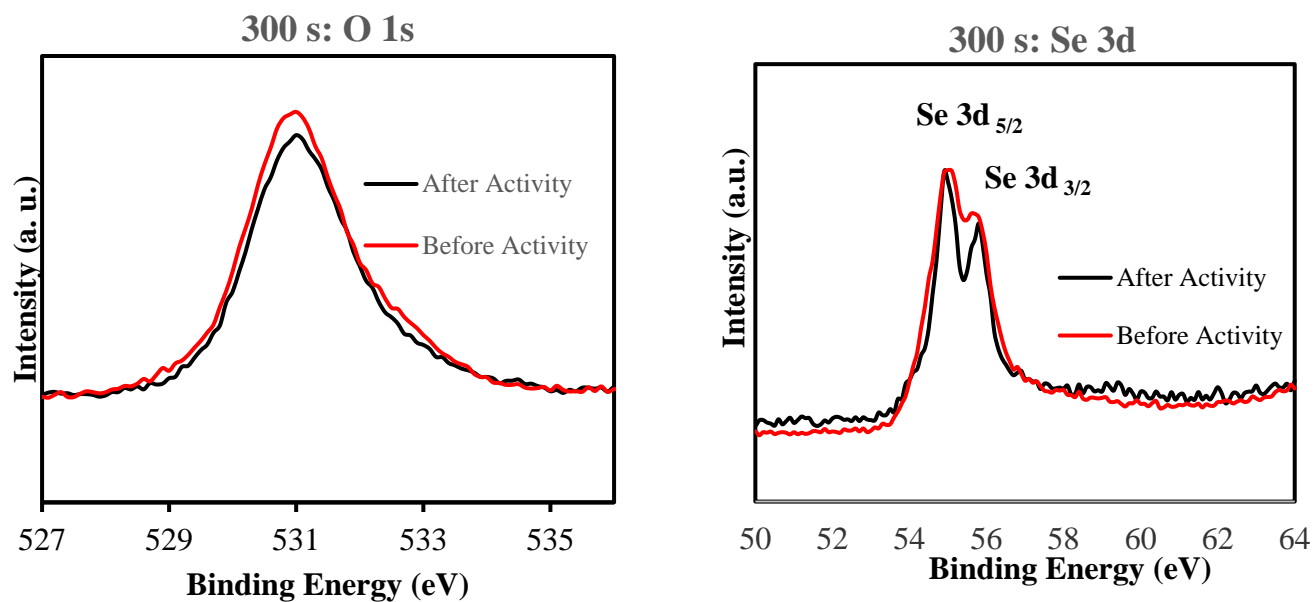
The measured ratio of the ring limiting current to the disk limiting current is known as the collection efficiency (N) of that RRDE electrode and it can be written as-

$$N = -\frac{i_{Limiting,Ring}}{i_{Limiting,Disk}} \quad (1)$$

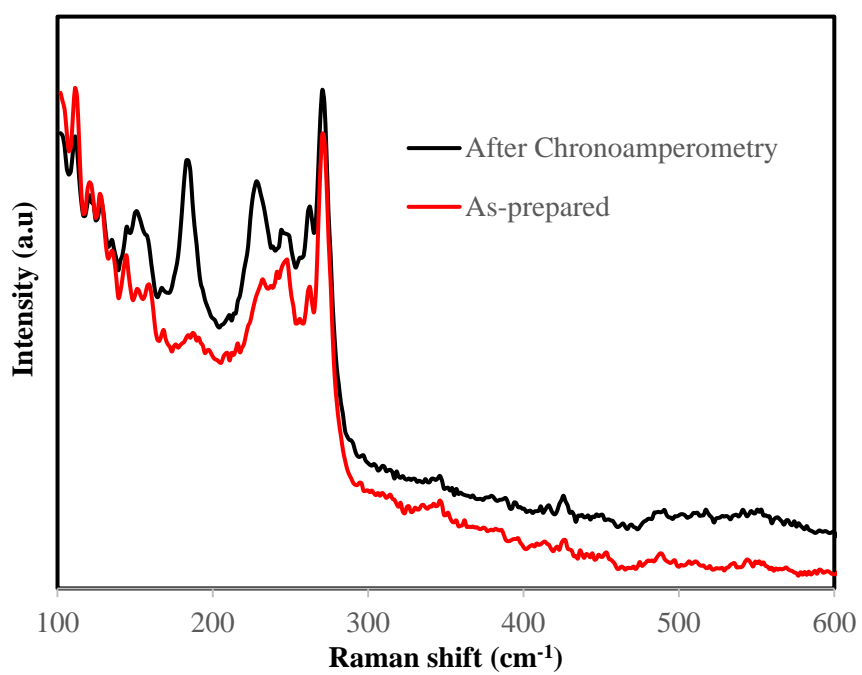


**Figure S6.** Rotating Ring-Disk voltamograms in N<sub>2</sub> saturated 0.001M K<sub>3</sub>Fe(CN)<sub>6</sub> containing 0.3M of KOH at 0.05 V s<sup>-1</sup> at different rotation rate. Ring potential was held on 0.7 V vs. Ag/AgCl which is sufficient to oxidize the product rapidly at ring. (Here ring current was shown as a potential of disk electrode)

At disk electrode, the reduction of ferricyanide (Fe(CN)<sub>6</sub><sup>3-</sup>) to ferrocyanide ((Fe(CN)<sub>6</sub><sup>4-</sup>) took place, on the other hand ferrocyanide is oxidized to ferricyanide at ring at positive potential (0.7 V vs. Ag/AgCl). The value of N was calculated for different rotation and it was found to be independent of rotation (0.24).

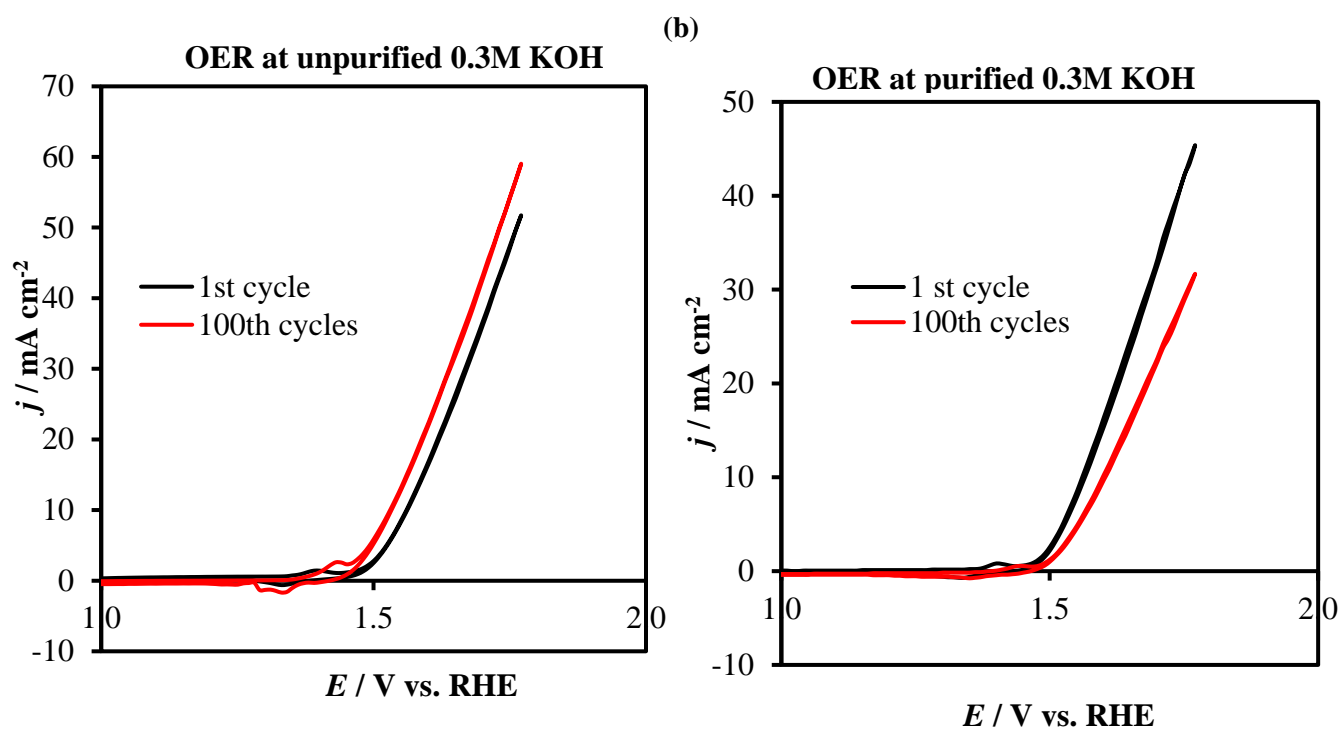
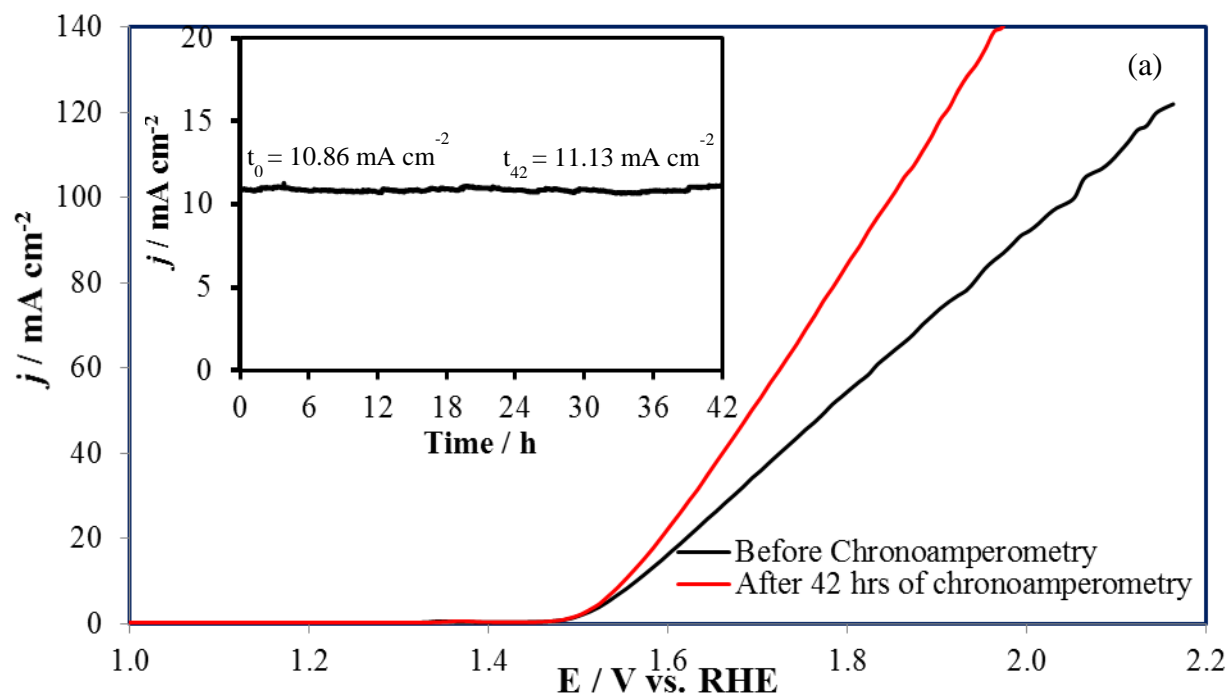


**Figure S7.** XPS spectra of O 1s and Se 3d peaks obtained from the catalyst before and after chronoamperometry (18h).

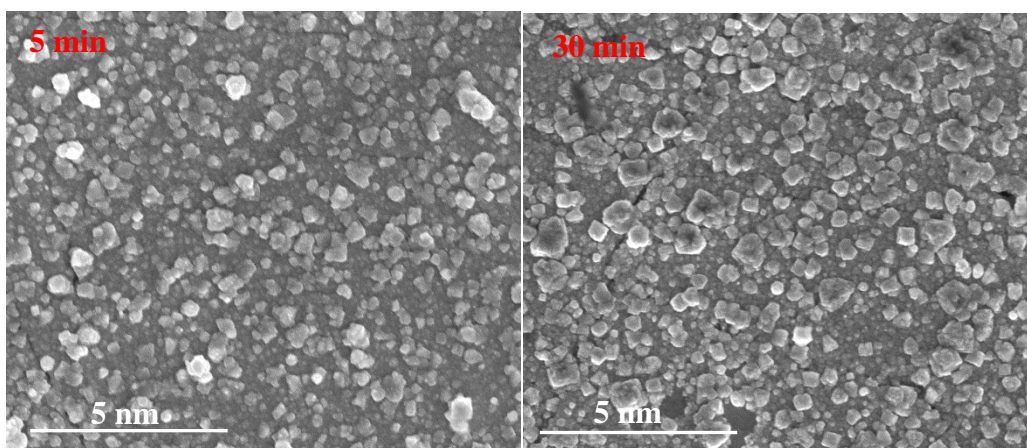


**Figure S8.** Raman spectra of  $\text{Ni}_3\text{Se}_2$  before and after chronoamperometry for 18h.

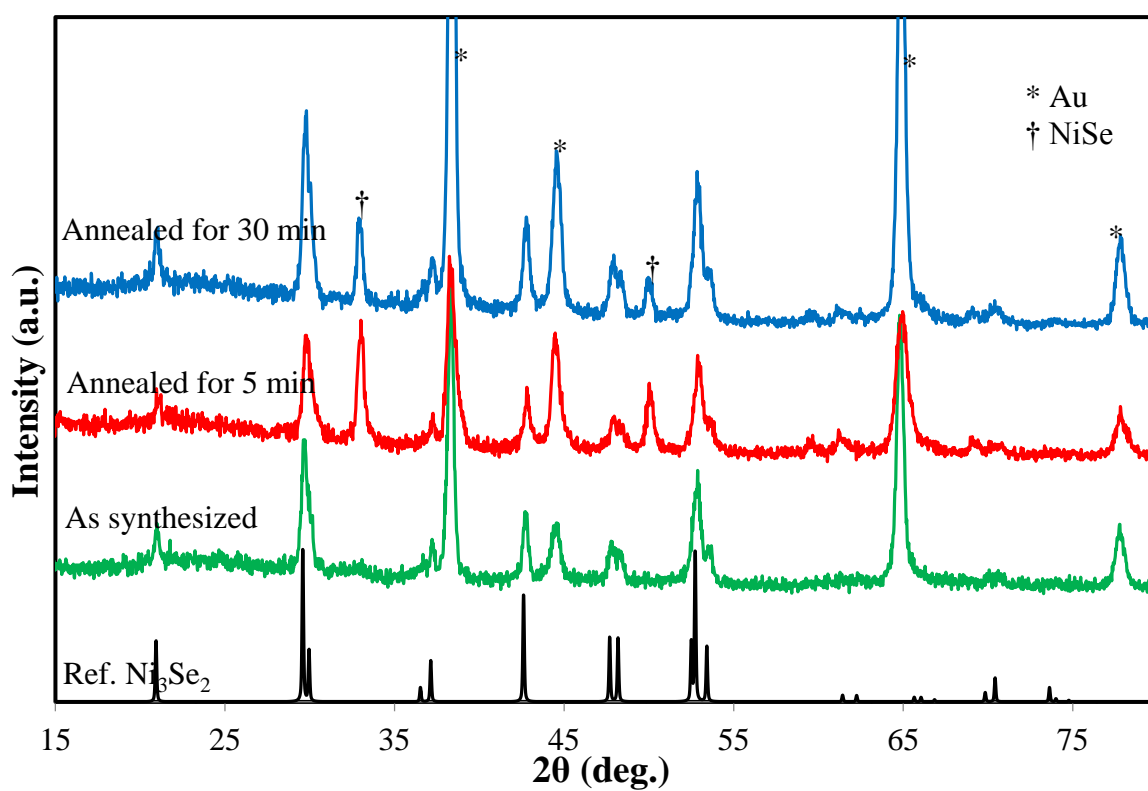




**Figure S9:** (a) Chronoamperometry at overpotential of 0.31 V (vs. RHE); comparison of (b) LSV before and after 42 h of chronoamperometry; and (c) cyclic voltammograms for OER of 1st cycle with 100th cycles in unpurified and purified 0.3 M KOH.



**Figure S10.** SEM images of the catalyst deposited for 300 s after 5 and 30 min annealing under  $N_2$ , respectively.



**Figure S11.** Pxd patterns of catalysts deposited for 300 s before and after annealing under  $N_2$ .

**Table ST2.** Variation of Ni:Se ratio as a function of annealing.

Catalyst synthesized for 300 s	Ni: Se
As prepared	1.32
Annealing for 5 min	1.43
Annealing for 30 min	1.93