Naturally self-assembled nickel nanolattice

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This electronic supporting information section consists of extra XRD to compare laboratory and synchrotron results, laboratory high resolution small angle X-ray scattering data, and transmission electron microscopy images.

1. Characterization

1.1 X-Ray diffraction

Samples were characterized using both the laboratory and synchrotron radiation X-ray diffraction (XRD) as in Figure S1. It is clearly seen that three peaks due to (111), (200) and (220) planes are visible in Laboratory XRD for 1ml and 3ml samples only, whereas they are seen in synchrotron XRD for all samples except 10ml sample. These results combined with SAED demonstrate clearly that the synchrotron data is superior to those of laboratory XRD and electron diffraction viz. SAED (Fig.2).

1.2 High resolution X-ray diffraction (Laboratory)

High resolution laboratory XRD was done using Cu K α radiation in the angle range 0.2-10° with a step size of 0.02°. This was basically done for ascertaining preliminary and final information that "Whether we can observe the nanolattice of nickel nanoparticles when dropcasted on a glass slide as seen in SAED or not (Figure 1a)." It is evident that such possibility prevails as indicated by the prominent four peaks (Figure S2). There is strong proof that nickel nanoparticles form a bulk 3D nanolattice as discussed in the text. The first peak near 20=0.36° was identified as the total external reflection from glass substrate (Figure S2). This was confirmed from similar experiment on blank glass slide that exhibits only one peak due to total external reflection.



Figure S1 A comparison of the X-ray diffraction data of the samples indicated for the laboratory and synchrotron radiation sources. Synchrotron data clearly reveals the peaks at (200) and (220) even for 8ml sample, which are otherwise not seen in laboratory XRD down to 3ml sample.



Figure S2 Grazing incident X-ray diffraction of (Cu K_{α}) laboratory source for 10ml sample drop-casted film on glass slide (black) and blank glass slide (red) as indicated. This confirms that the first peak near 0.36° is due to the total external reflection. Note: Angle of incidence was fixed at 0.25°.



Figure S3 Small angle X-ray scattering data of various samples. Samples with trioctylphosphine (combined with oleylamine also) surely exhibit peaks, indicating the formation of nanolattice while those with Olyelamine (OA), triphenylphosphine (TPP) or combined do not exhibit any peak evidencing that these surfactants do not support formation of nanolattice. This shows dominant role of trioctylphosphine in enabling the nanolattice formation; only one peak is seen in these SAXS patterns as the set up setting the incident X-ray angle had to be done below 0.5°, not like that in Figure S2. Had we chose smaller angle of incidence, more peaks would have been expected.



Figure S4 Representative Transmission Electron Microscopy images showing natural self-assembly. Natural self-assembly of (a) 10ml, (b) 8ml, (c) 3ml and (d) 1ml trioctylphosphine prepared nanoparticle samples for fixed 8ml oleylamine. Scales shown are for 100nm, 50nm, 100nm and 200nm, respectively.