## **†Electronic Supplementary Information (ESI)**

## Shape-Controlled Synthesis of Nanopyramids and Nanoprisms of Nickel Sulfide (Ni<sub>3</sub>S<sub>4</sub>)

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Fig. S1: TEM image of the  $Ni_3S_4$  nanoprisms showing a large area and the self-assembly of the nanoparticles on the TEM grid.



Fig. S2: TEM image of the  $Ni_3S_4$  nanopyramids showing a large area and the self-assembly of the nanoparticles on the TEM grid.



**Fig. S3**: UV-vis absorbance spectra of the Ni complexes formed in the presence of the capping ligands and dodecanethiol for (a) NiCl<sub>2</sub>·H<sub>2</sub>O and (b) Ni(acac)<sub>2</sub> in 1-octadecene at 120  $^{\circ}$ C.

Sample	OLM <sup>a</sup> (mL)	OLA <sup>b</sup> (mL)	DDT <sup>c</sup> (µL)	Solvent*	Edge Length(nm)	Yield	Time	Comments
1	0.8	0	120	BE	~10/5	Low	2 min	
2	1.2	0	450	BE	~50-70	Low	5 min	Mixed shapes
3	1.2	0	450	BE	~200	Low	35 min	
4	0.4	0.2	450	ODE	15.5	Low	2 min	
5	0.4	0.2	450	BE	8.1	Low	5 min	
6	0.6	0.2	450	ODE	16	High	2 min	
7	0.8	0.2	450	ODE	~16	High	2 min	Mixed shapes
8	0.8	0.2	450	BE	14.7		5 min	
9	0	0.6	450	ODE	17.2/6.5		5 min	Nanobars
10	0.4	0.6	450	BE	9.1	Low	5 min	
11	0.6	0.6	450	ODE	19.2	High	5 min	
12	0.8	0.6	450	BE	~15		5 min	Mixed shapes
13	1.2	0.6	450	BE	~16		5 min	Mixed shapes

Table S1. Reaction conditions to obtain Ni<sub>3</sub>S<sub>4</sub> nanopyramids in high yields.

a: oleylamine volume; b: oleic acid volume; c: 1-dodecanethiol volume.

\*All the reactions were carried out in 5 mL of solvent (ODE = 1-octadecene, BE = benzyl ether) in the presence of 120 mg of Ni $(acac)_2$ .