Characterization of Ni-phases and their transformations in Fluid Catalytic Cracking (FCC) catalysts: Comparison of conventional versus boron-based Ni-passivation

Ioannis D. Charisteidis[1], Pantelis N. Trikalitis[2], Konstantinos S. Triantafyllidis[1,*], Vasileios Komvokis[3,*],
Bilge Yilmaz[3,*]

[1]*Department of Chemistry, Aristotle University of Thessaloniki, Thessaloniki, Greece*
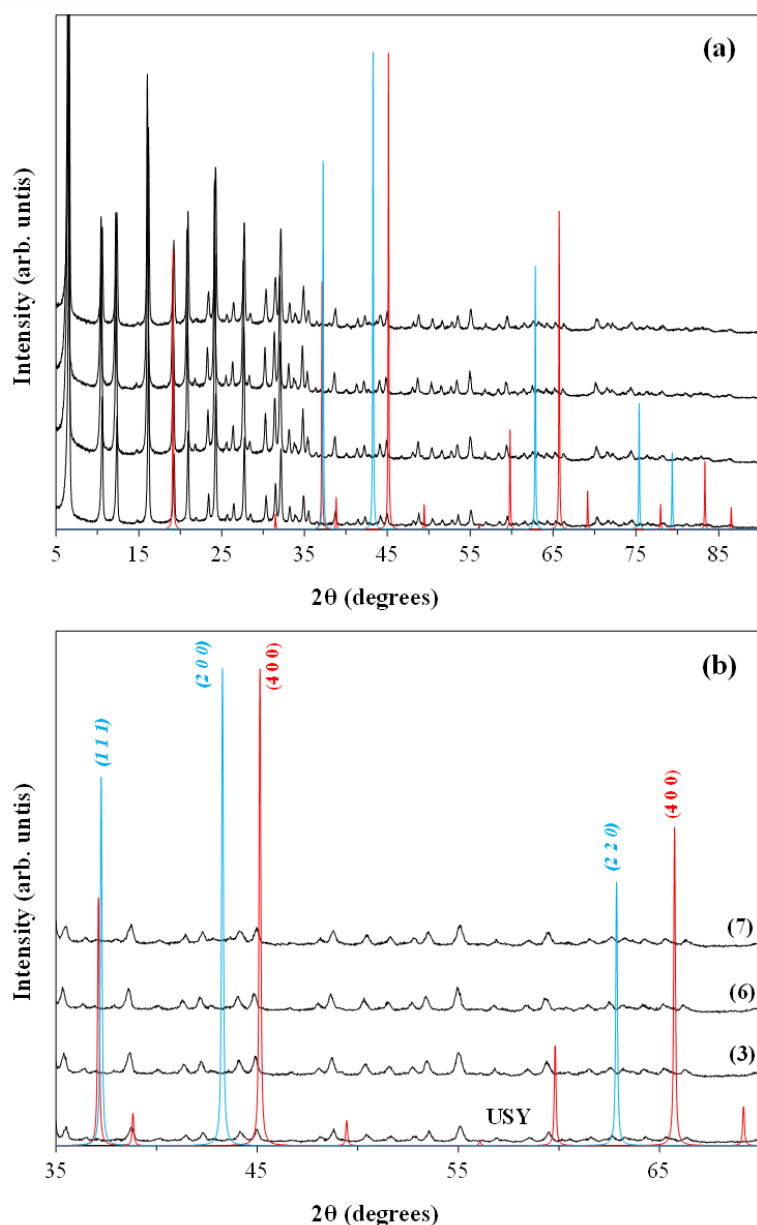[2] *Department of Chemistry, University of Crete, Voutes, 71003 Heraklion, Greece*
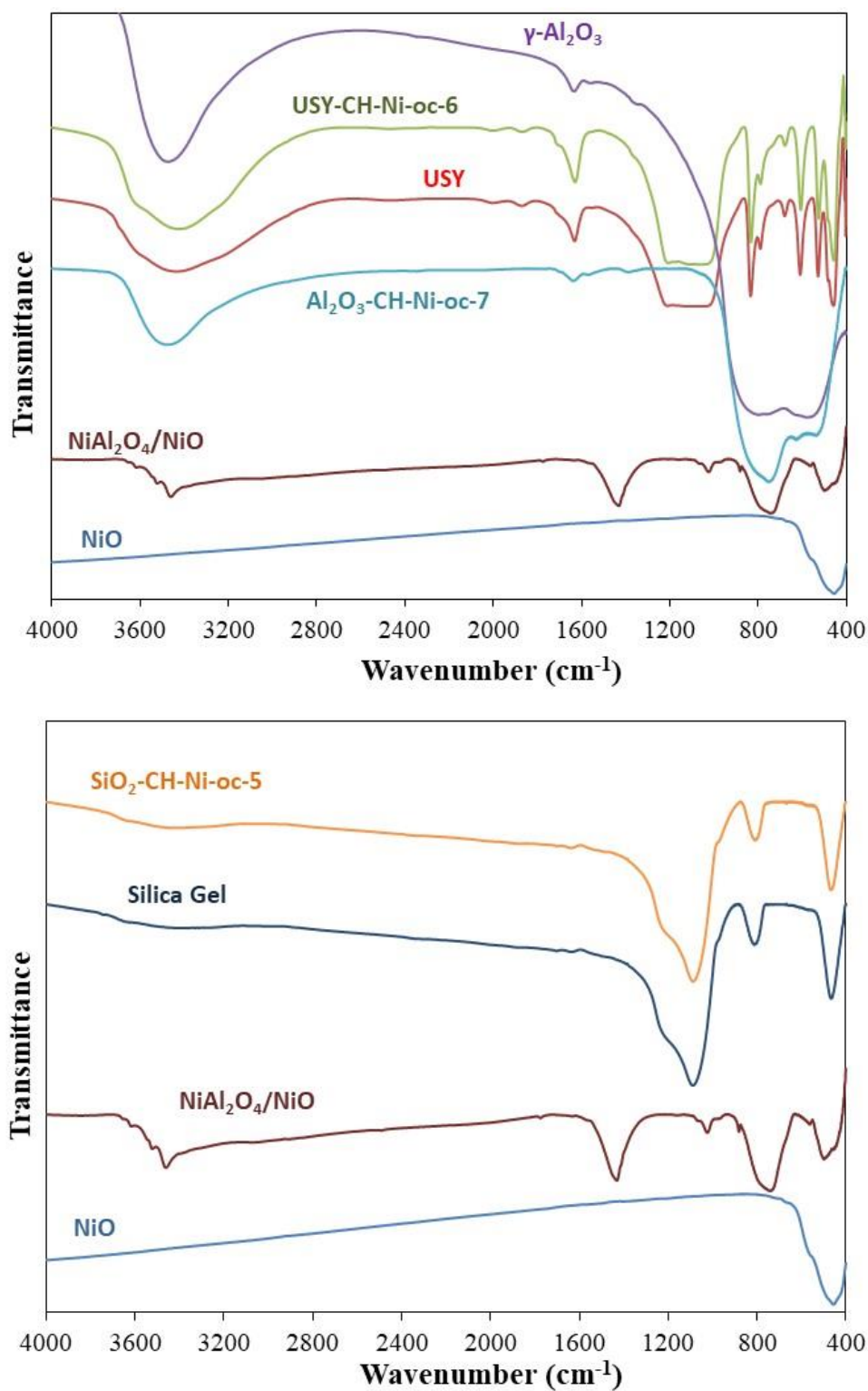[3] *BASF Corporation, 25 Middlesex/Essex Turnpike, Iselin, NJ 08830, USA;*
* Corresponding authors: ktrianta@chem.auth.gr; vasileios.komvokis@basf.com; bilge.yilmaz@basf.com

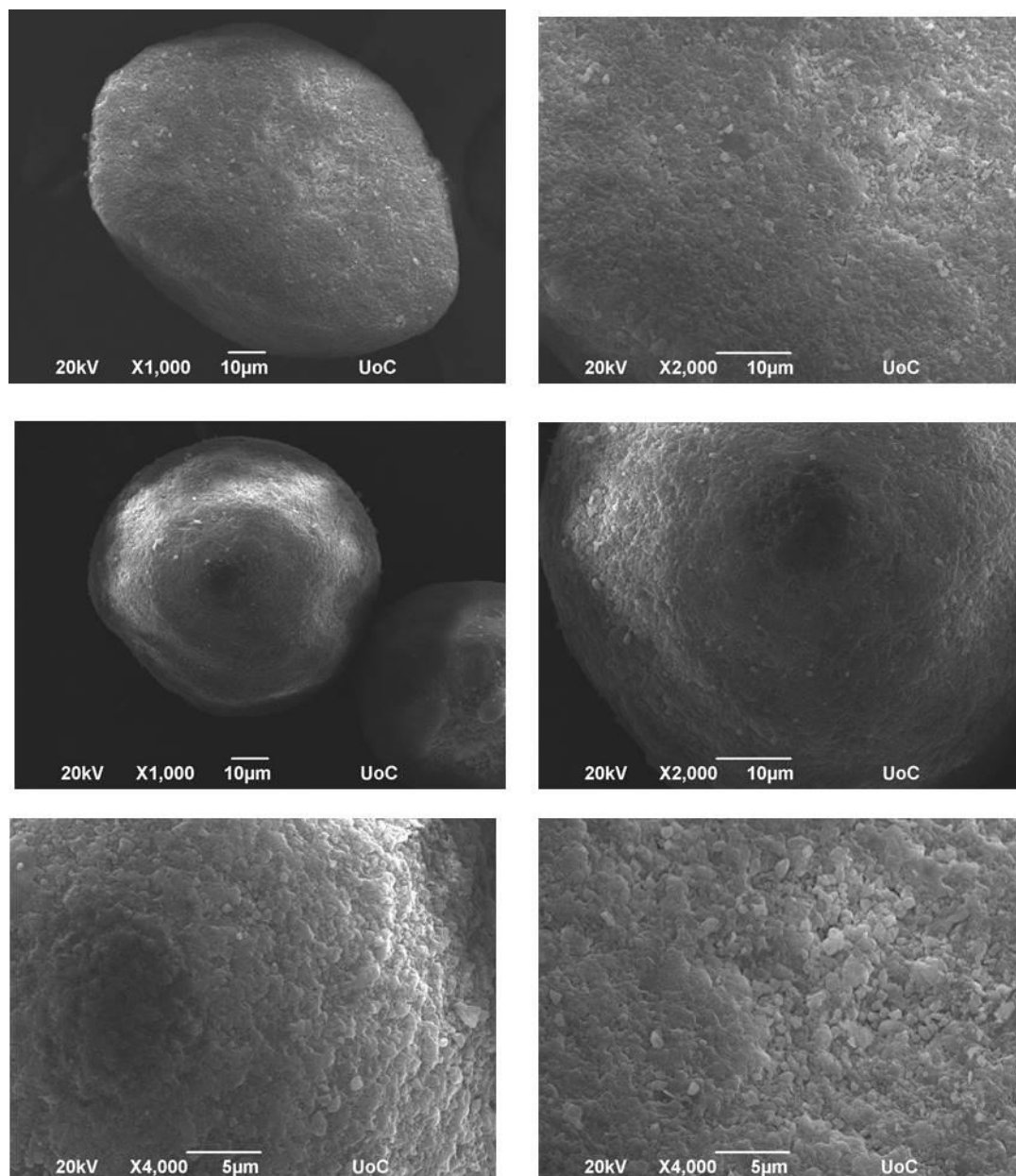## SUPPLEMENTARY MATERIALS

PXRD patterns

Figure S1. **(a)** PXRD patterns of USY-CH-Ni-oc-*X* samples, prepared via impregnation of USY zeolite with Ni octanoate solution diluted in cyclohexane (see experimental section); *X* : (3) 5000 ppm Ni, (6) 20000 ppm Ni, (7) 50000 ppm Ni, and **(b)** Enlarged view in the range 35-70º 2*θ*. The patterns of NiO (blue pattern) and NiAl$_2$O$_4$ (red pattern) are also shown for comparison.
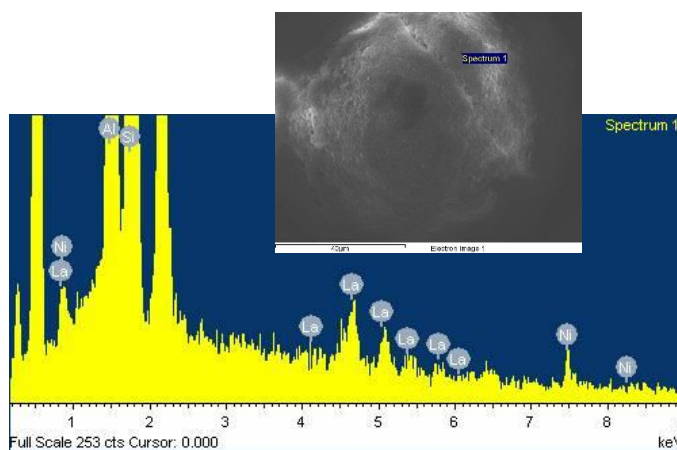
Figure S2. FTIR spectra of representative Ni-impregnated materials: SiO_2-CH-Ni-oc-5 (1.5 wt.% Ni), Al_2O_3-CH-Ni-oc-7 (10 wt.% Ni), USY-CH-Ni-oc-6 (2 wt.% Ni). The spectra of bulk NiO, $NiAl_2O_4$/NiO, silica gel, γ-Al_2O_3 and USY zeolite, are also included for comparison.
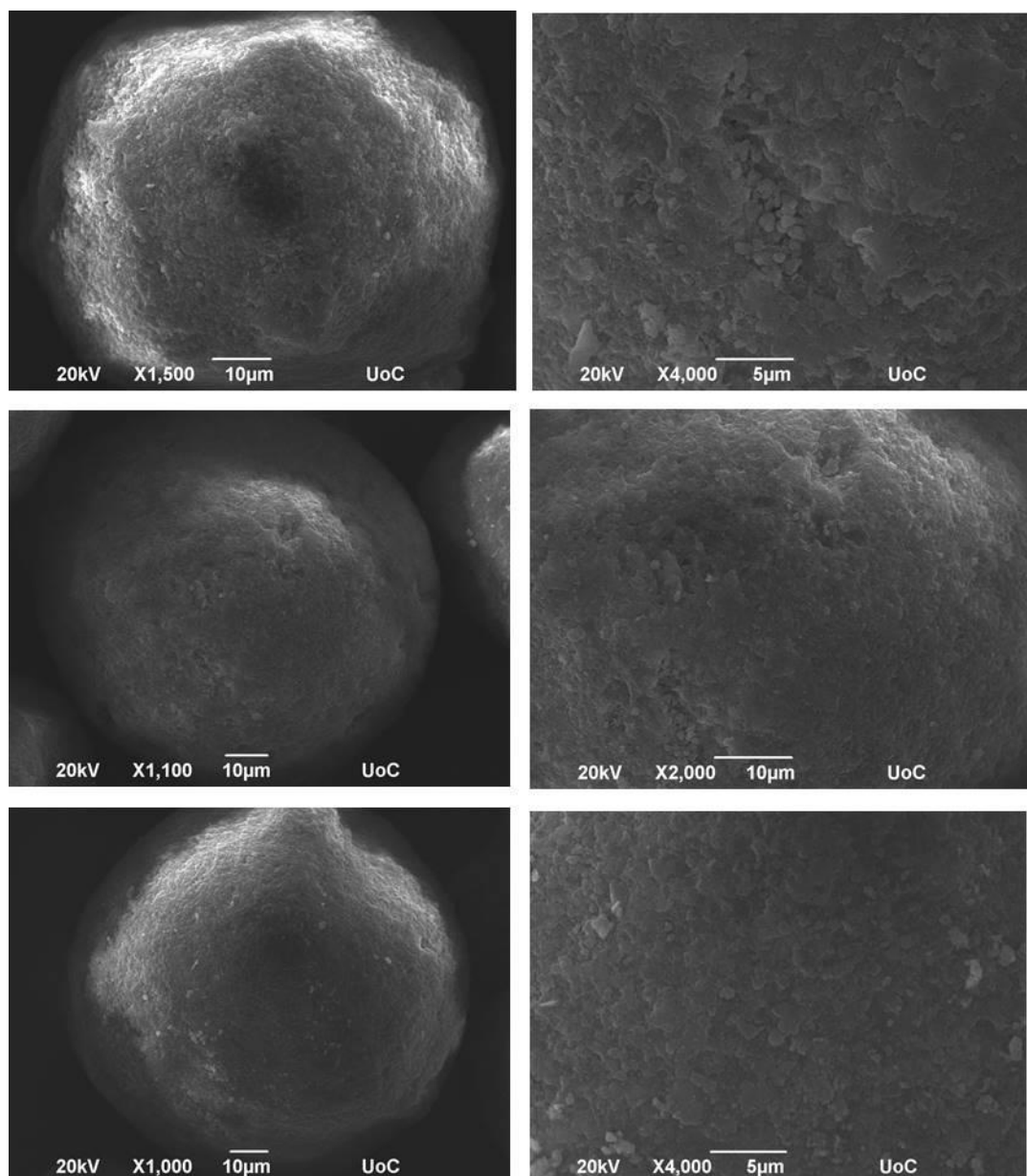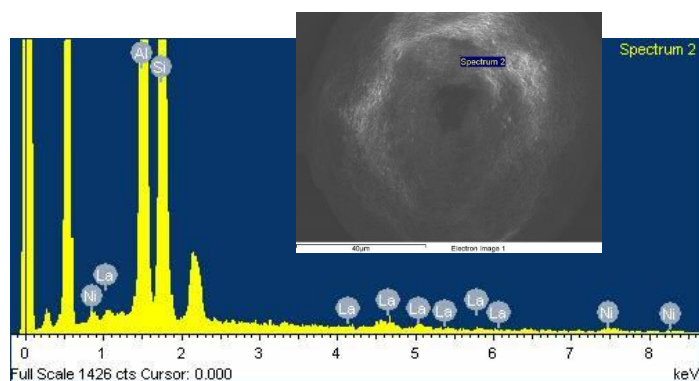
SEM images and point EDS microanalysis

Element	Atomic (%), average value	Range
Al K	37.3	33.4 – 43.4
Si K	60.9	55.7 – 65.1
Ni K	0.8	0.4 – 1.2
La L	0.7	0.2 – 1.1

Figure S3. Representative SEM images and point EDS microanalysis data of Ni-loaded FCC catalyst: BLF-CAT-4 with 10,000 ppm Ni
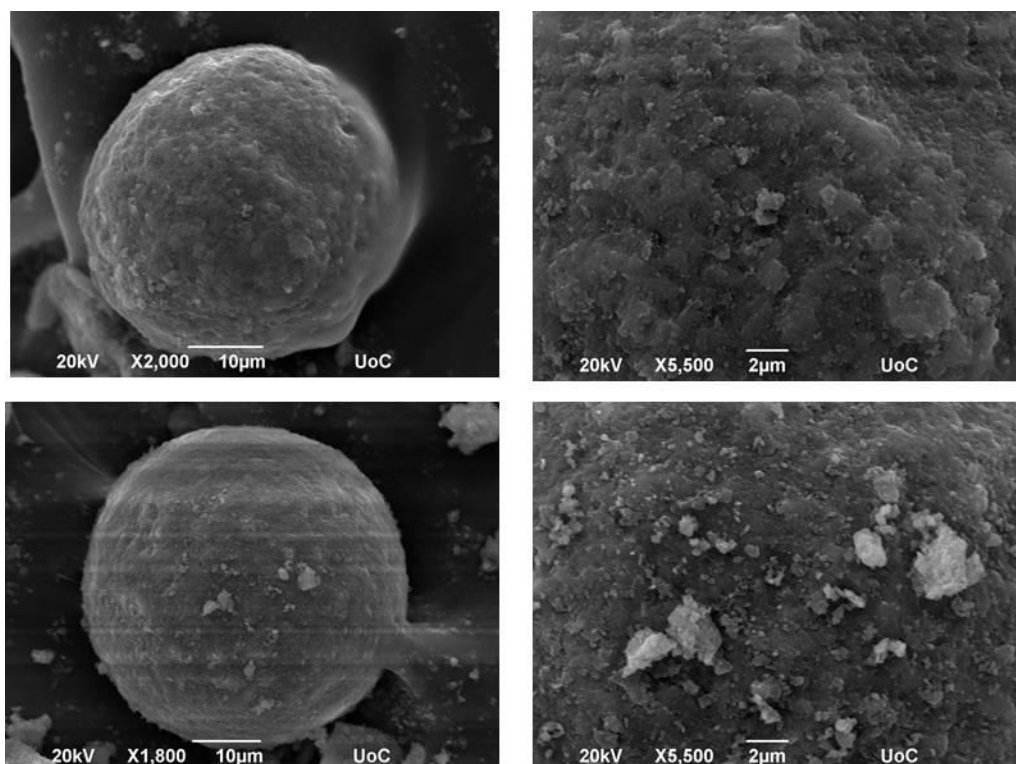
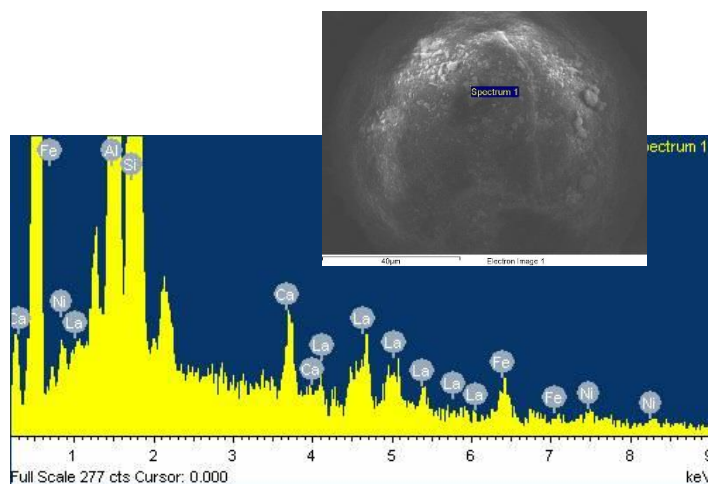| Element	Atomic (%), average value	Range
Al K	36.2	35.3 – 37.7
Si K	61.6	59.7 – 62.9
Ni K	1.0	0.7 – 1.2
La L	1.2	1.0 – 1.6

Figure S4. Representative SEM images and point EDS microanalysis data of Ni-loaded FCC catalyst: BLB-CAT-4 (containing also boron) with 10,000 ppm Ni
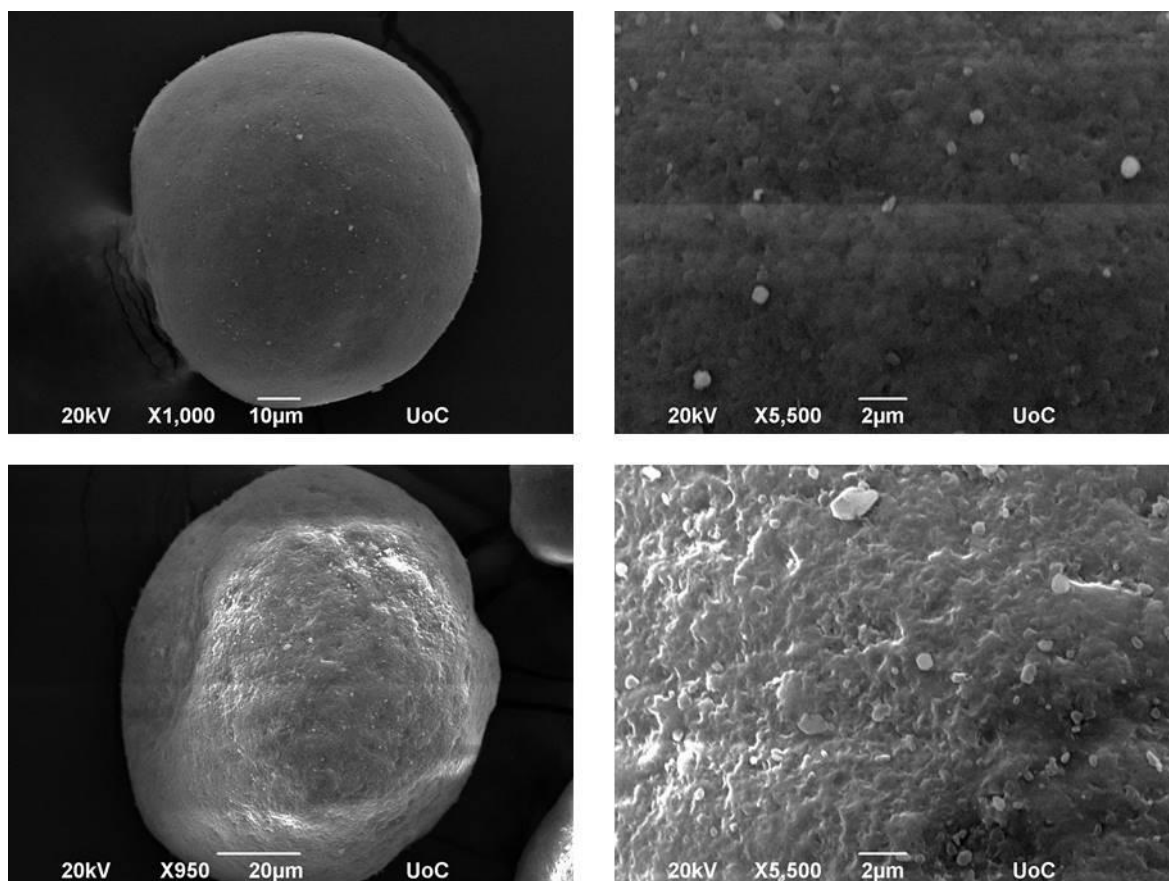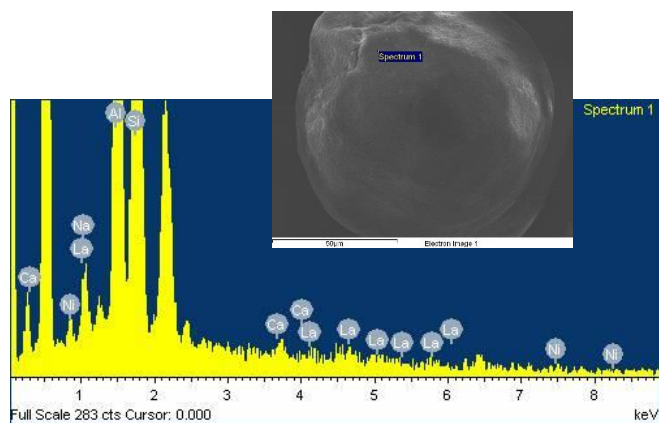
Element	Atomic (%), average value	Range
Al K	36.8	34.1 – 40.3
Si K	63.3	53.9 – 78.6
Ca K	2.5	1.4 – 5.8
Ti K	2.8	2.8
V K	0.6	0.6
Fe K	1.2	0.7 – 2.5
Ni K	0.7	0.3 – 1.3
La K	1.4	0.5 – 2.6
Mg K	3.1	1.3 – 6.3

Figure S5. Representative SEM images and point EDS microanalysis data of equilibrium FCC catalyst: B-CAT-2M (4327 ppm Ni)
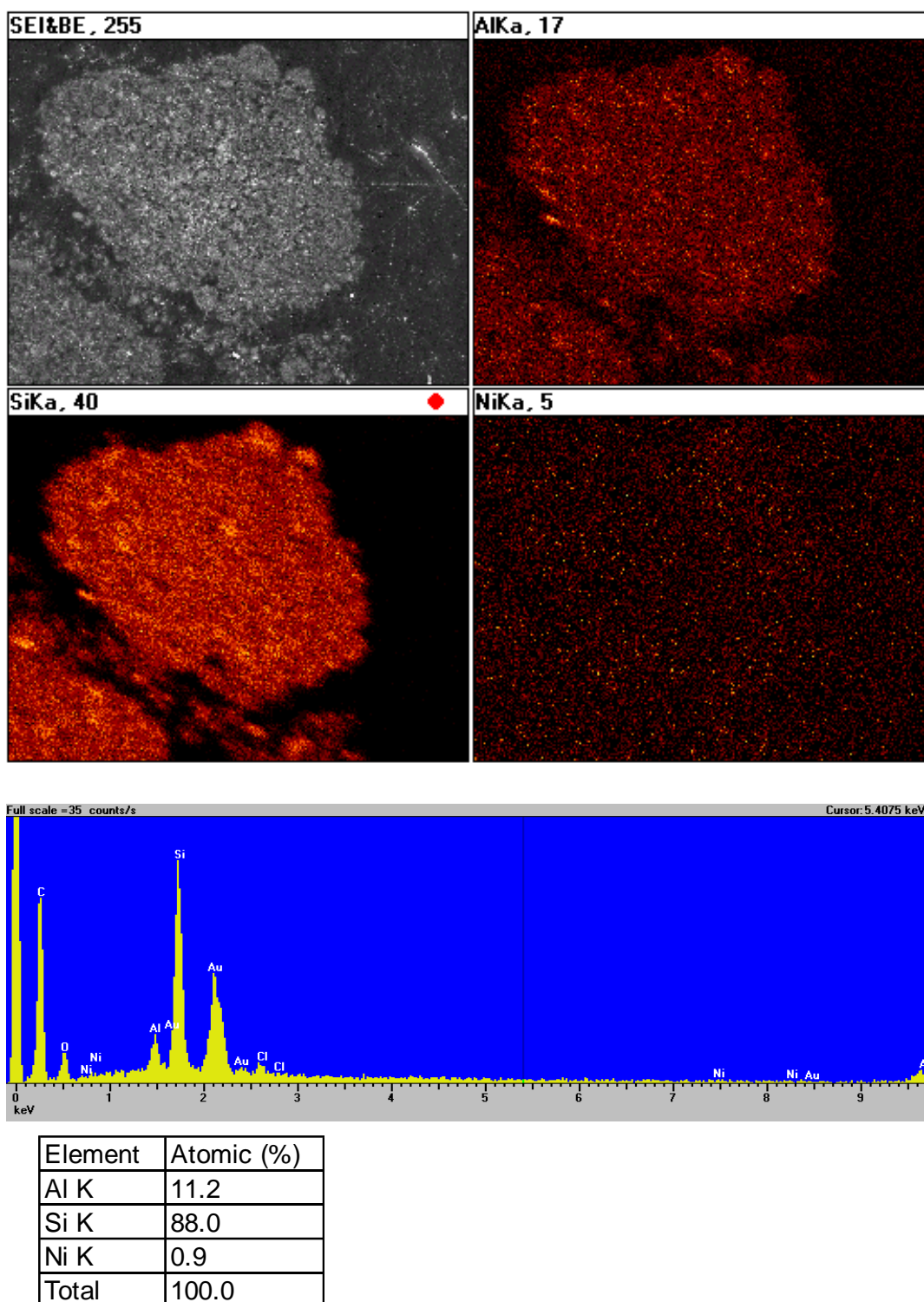
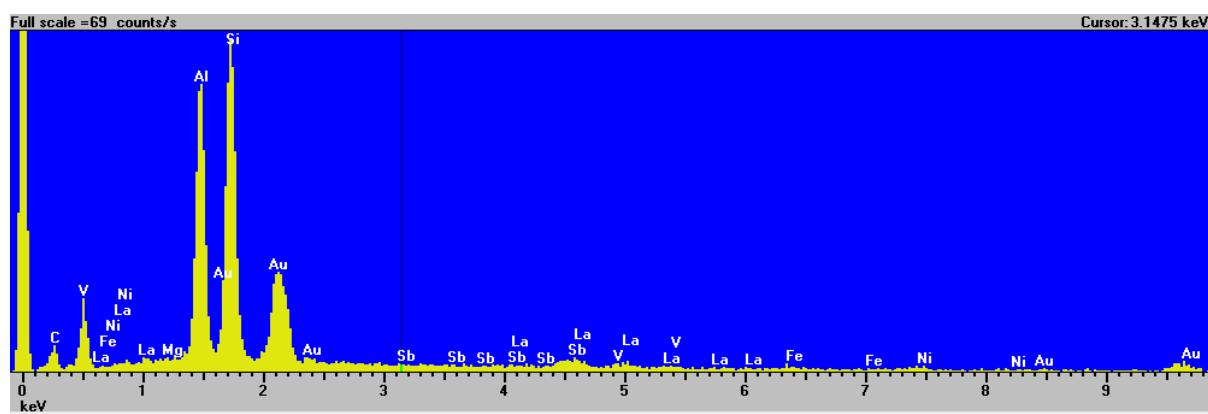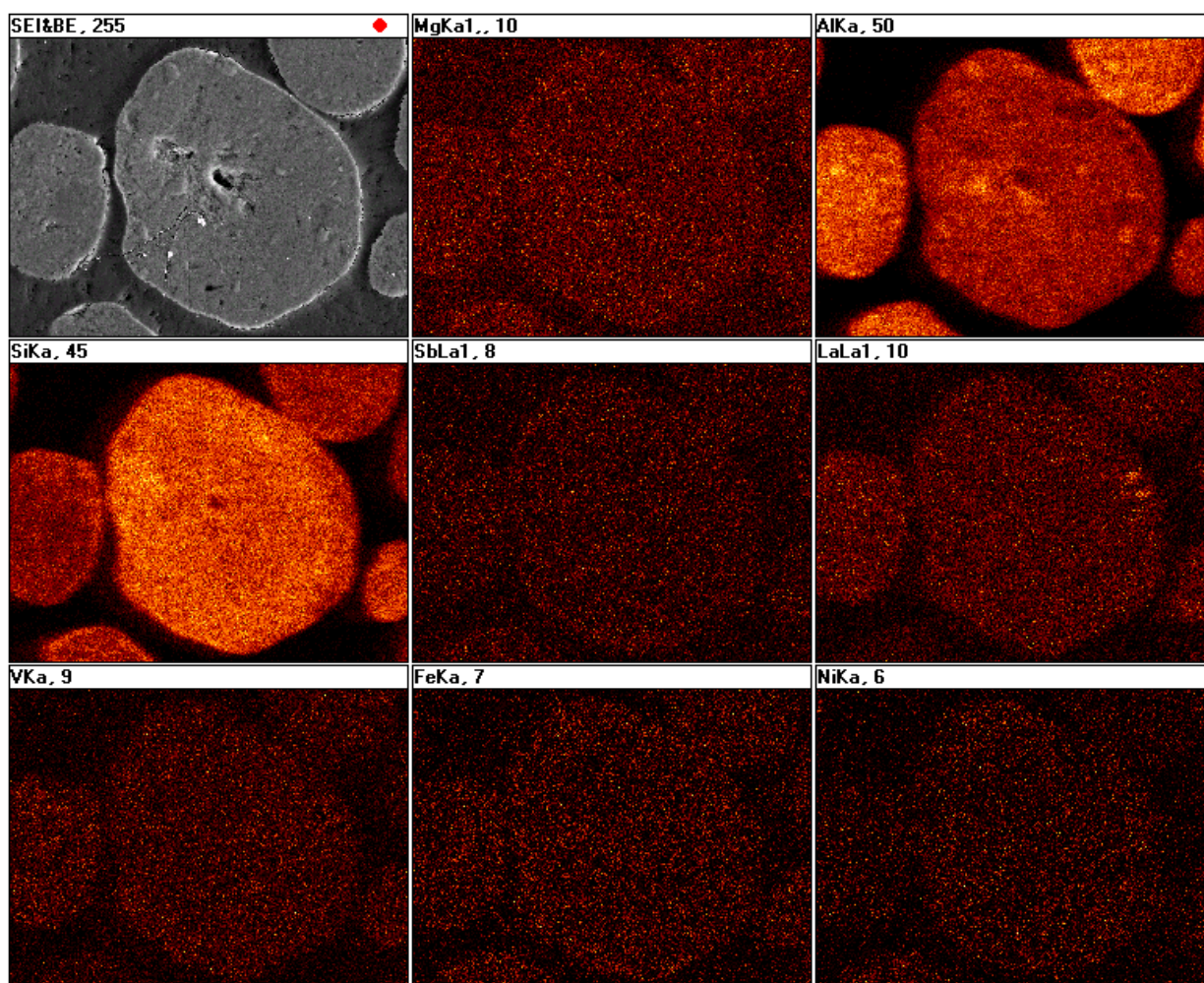| Element	Atomic (%), average value	Range
Al K	33.2	32.7 – 33.5
Si K	61.0	59.1 – 63.8
Ca K	0.5	0.5
Ti K	1.4	1.1 – 1.8
Fe K	1.4	1.3 – 1.4
Ni K	0.5	0.3 – 0.8
La K	0.4	0.1 – 0.7
Mg K	0.9	0.9

Figure S6. Representative SEM images and point EDS microanalysis data of equilibrium FCC catalyst: B-CAT-5M (5141 ppm Ni)

EDS elemental mapping of flat cross-sections

Element	Atomic (%)
Al K	11.2
Si K	88.0
Ni K	0.9
Total	100.0

Figure S7. SEM image and EDS elemental mapping of representative Ni-impregnated USY zeolite sample: USY-CH-Ni-oc-4 (10,000 ppm Ni)

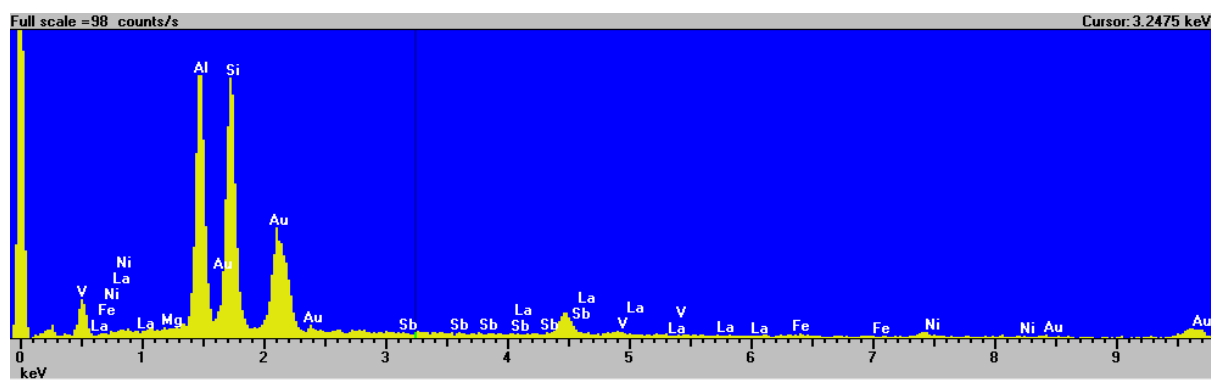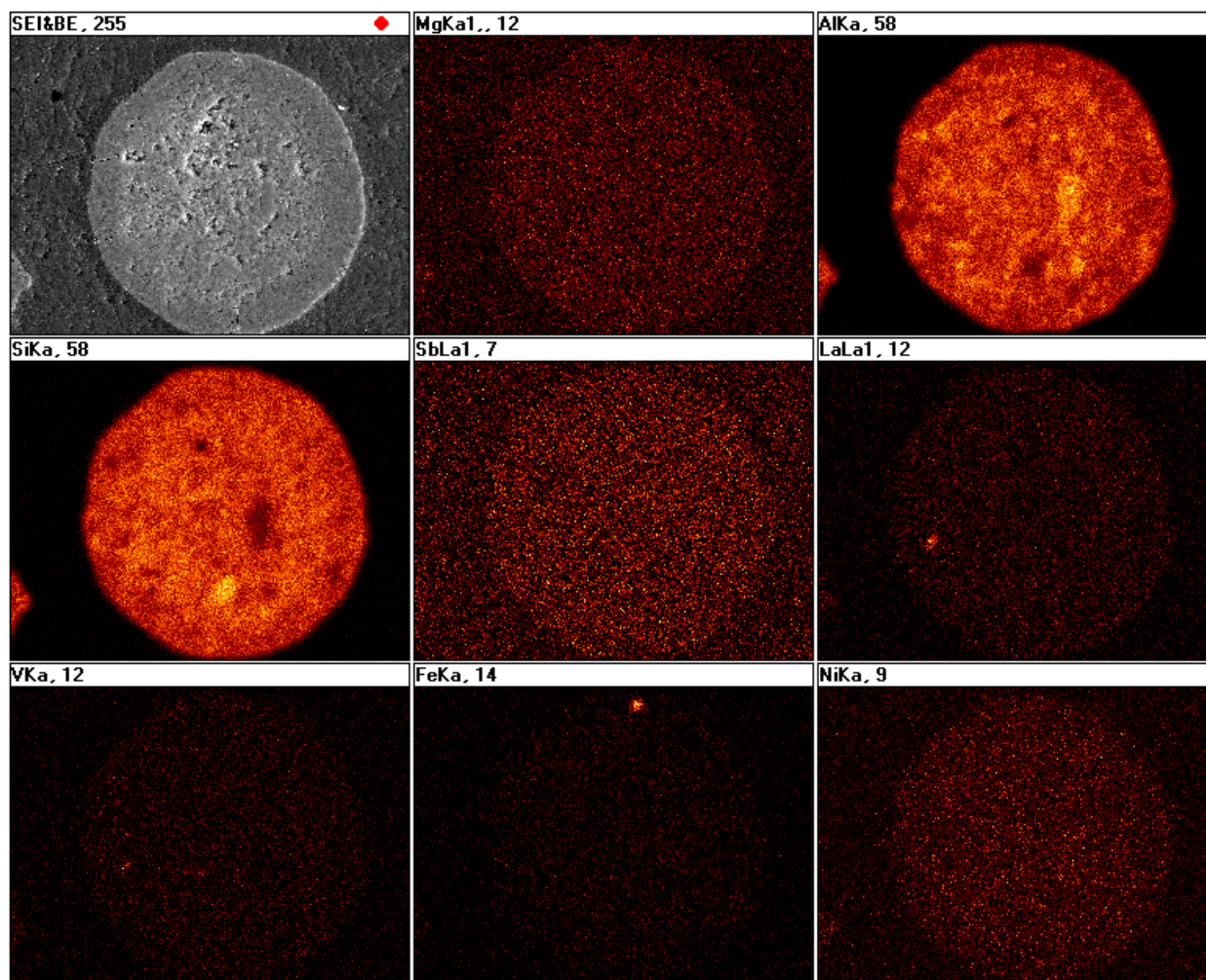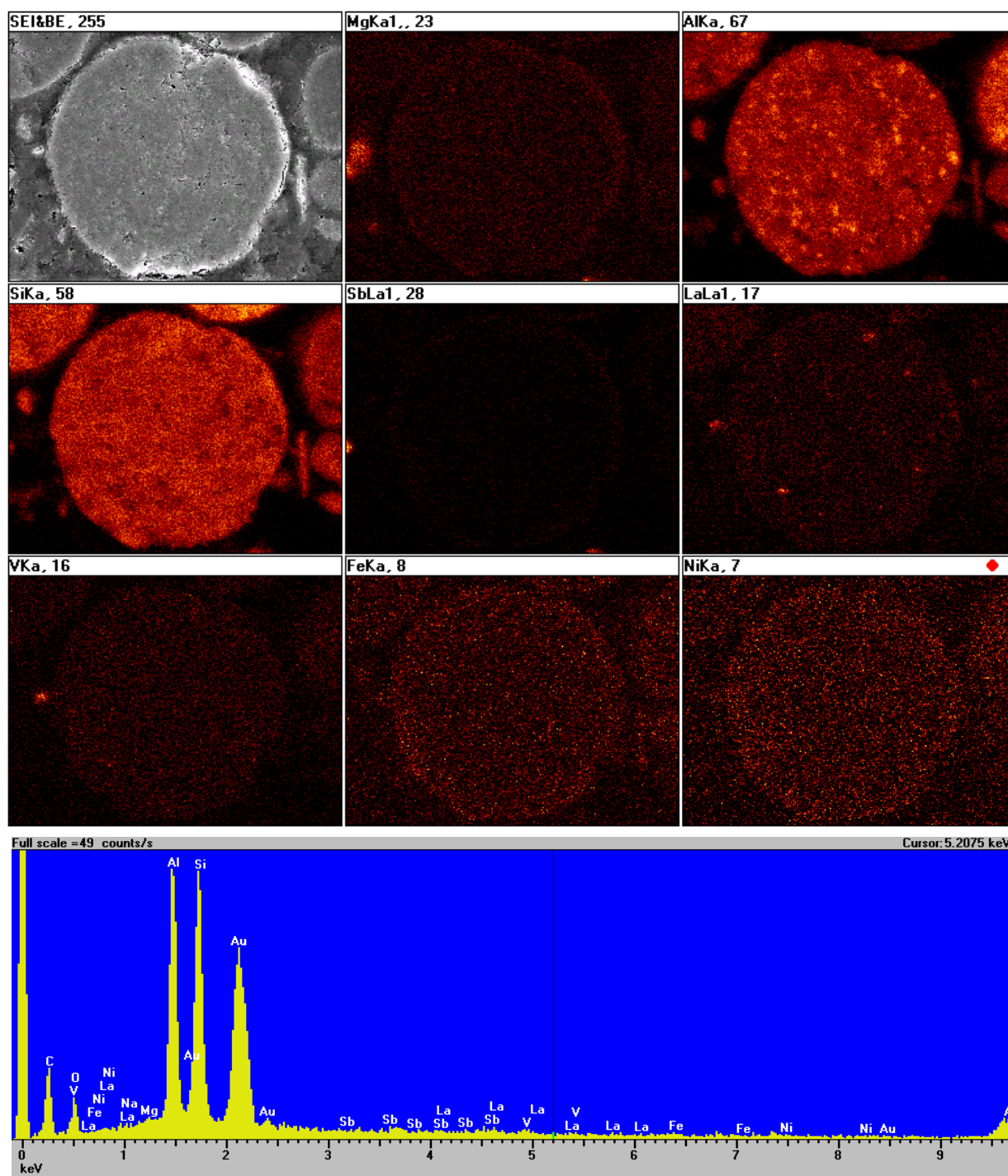| Element	Atomic (%)
Mg K	0.4
Al K	34.6
Si K	61.6
V K	0.1
Fe K	0.1
Ni K	2.7
Sb L	0.1
La L	0.4
Total	100

Figure S8. SEM image and EDS elemental mapping of representative Ni-loaded FCC catalyst: BLF-CAT-4 with 10,000 ppm Ni
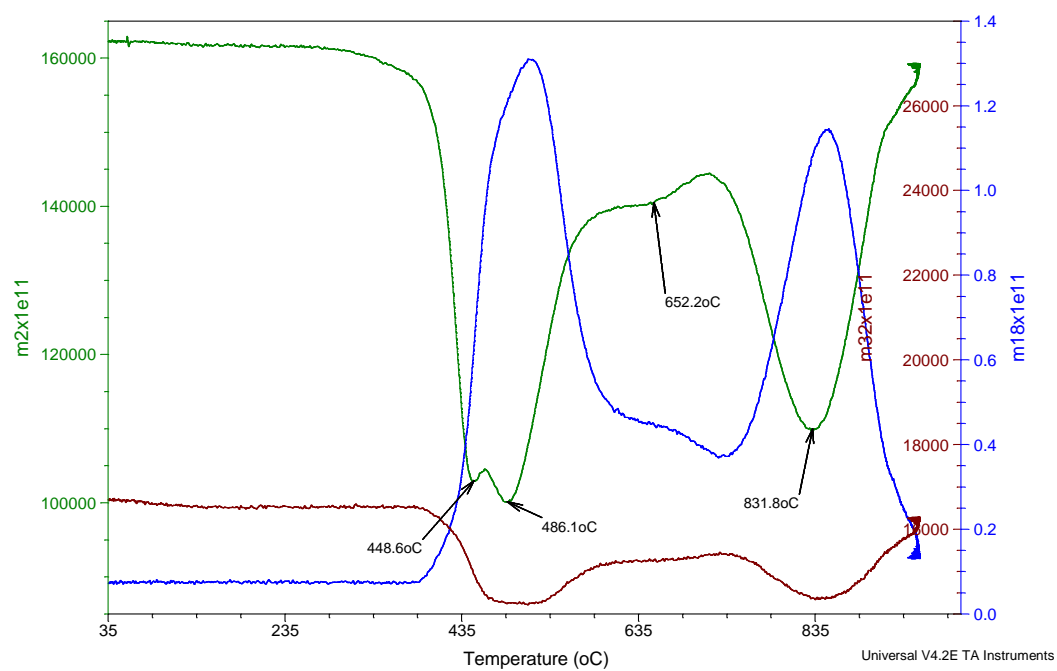
Element	Atomic (%)
Mg K	0.1
Al K	35.1
Si K	61.1
V K	0.2
Fe K	0.8
Ni K	2.5
Sb L	0.1
La L	0.1
Total	100

Figure S9. SEM image and EDS elemental mapping of representative Ni-loaded FCC catalyst: BLB-CAT-4 (containing boron) with 10,000 ppm Ni
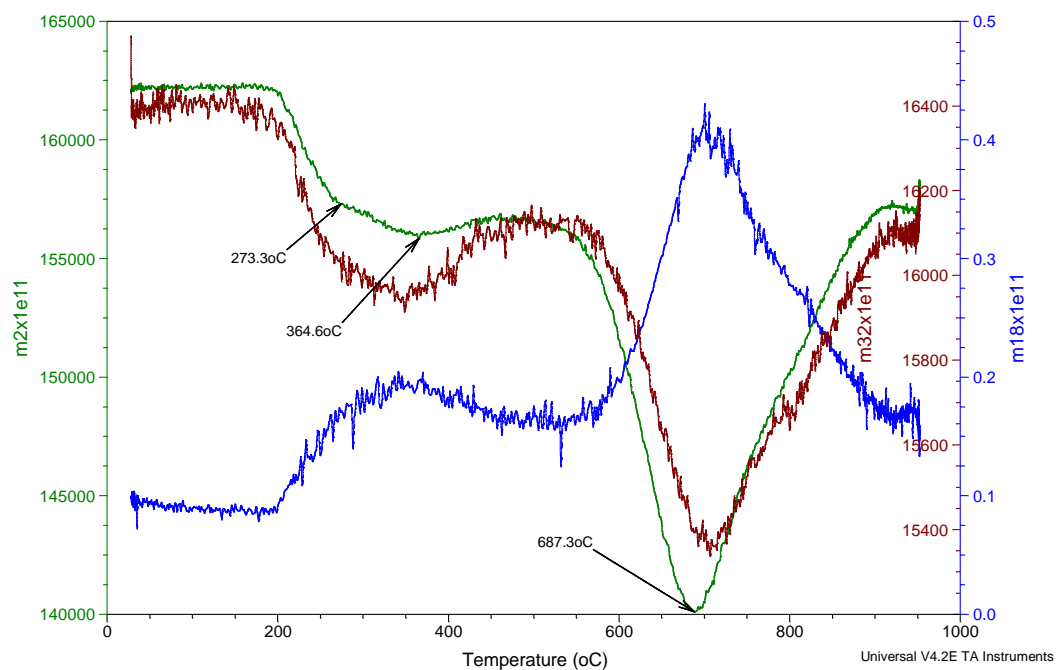
Element	Atomic (%)
Na K	0.1
Mg K	0.6
Al K	36.2
Si K	59.5
V K	1.1
Fe K	0.7
Ni K	1.2
Sb L	0.1
La L	0.5
Total	100

Figure S10. SEM image and EDS elemental mapping of equilibrium FCC catalyst: B-CAT-2M (4327 ppm Ni)

Temperature programmed reduction with H₂ (TPR-H₂)

Figure S11. TPR-H₂ profile of Al₂O₃-CH-Ni-oc-7 sample containing 200,000 ppm Ni

Figure S12. TPR-H₂ profile of USY-CH-Ni-oc-7 sample containing 50,000 ppm Ni