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HIGH RESOLUTION ELECTRON MICROSCOPY OF NANOSTRUCTURE ON THE SURFACE OF COMMERCIAL FCC CATALYST

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Metal contamination on the FCC catalysts has been an issue in refinery industry for a long time. The morphology, phase distribution and nanostructure of poisoning metals on the surface of catalyst microspheres have strong effects on the performance of FCC catalyst. With the development of high resolution electron microscopy (HREM) technology, the nano structure on the surface of catalyst have been the subject of growing interest in recent years from both experimental and theoretical point of view. The common knowledge of the metal contamination is that there is an1-3 micron thickness iron rich layer or nodules which deactivate the catalyst [1-3]. The nodules have 1-2 wt% Fe content. However there are little high resolution electron microscopy data on the nature of Fe-rich structures. In this study, HREM and electron diffraction have been employed for the structure of these iron-rich features. It has been found that the actual thickness of iron rich layer under STEM is less than that found in SEM studies because of the elimination of the beam broadening effect. Most of the iron rich layers are amorphous structure embedded with different kinds of iron oxides. The formation of these nano structures is also discussed.

An Ultrathin sections 20-40nm, were sliced using a Leica ultramicrotome equipped with a diamond-knife and the sections were supported on lacey carbon film on Cu-grids. The sectioned samples were investigated in a HITACHI HD-2000 and H-9000 operating at 200 kV and 300 kV respectively. The structure images were recorded with a Gatan CCD-camera. Diffraction patterns from the same area as used for the HRTEM images were also recorded on the CCD, using the smallest condenser aperture and spot size of the microscope.

From Figure 1, we can see the thickness of the iron rich layer is around 3 um, while the same size microsphere under STEM shows a thickness about 0.6 um. This indicates the STEM would give more accurate measure of the thickness of the layer. Figure 2 shows the HREM image of the outer layer of poisoned FCC catalyst. It has the future of amorphous embedded with nano iron oxides.

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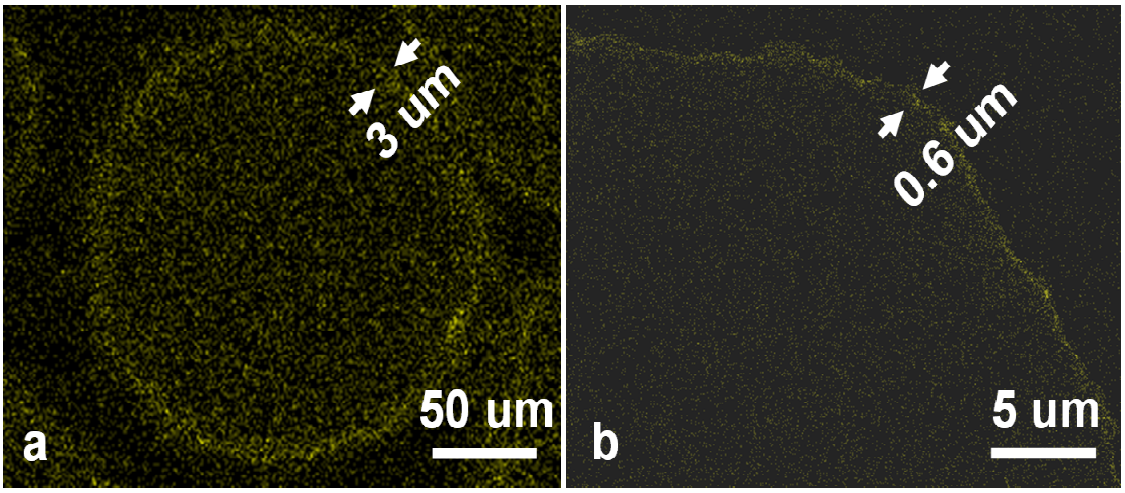

Figure 1 Comparison of the EDS mappings obtained under (a) SEM and (b) STEM

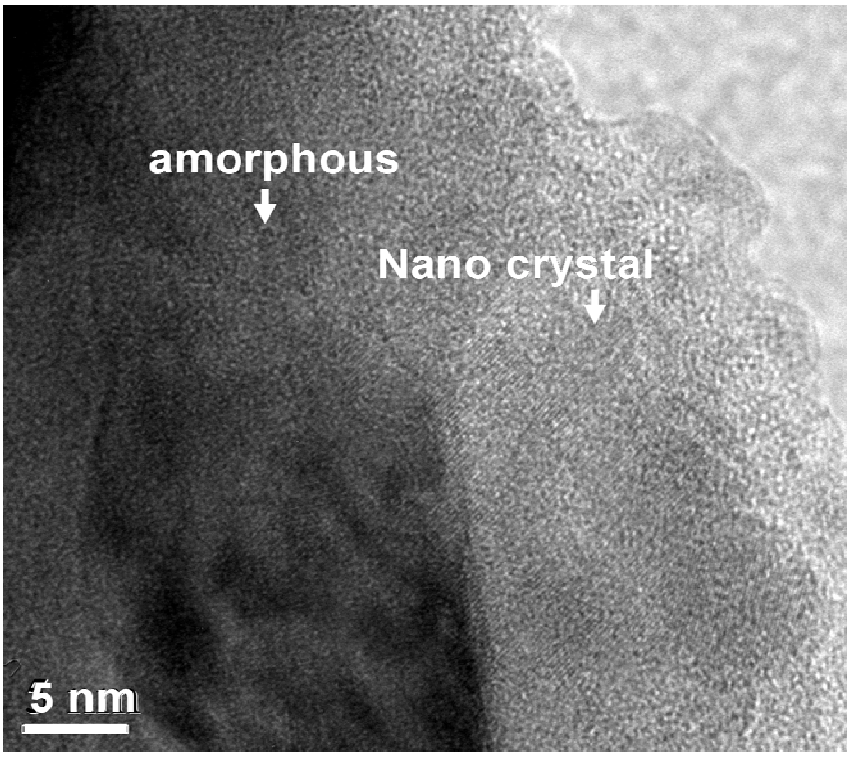

Figure 2 HREM images of the outer layer on FCC catalyst showing the embedded iron oxides in amorphous

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