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doi:10.1017/S1431927615002299 Paper No. 0150 Microsc. Microanal. 21 (Suppl 3), 2015

Direct Evidence of Basal-Bonded MoS_2 Cluster on Al_2O_3 Support

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Transmission electron microscopy (TEM) plays an important role in the characterization of heterogeneous catalysts, in particular on the MoS_2-based hydrodesulphurization (HDS) catalysts. Information about their structure, chemical composition, crystallite size and dispersion on the support is required in the new formulations to improve the catalytic performance in the HDS reactions.

MoS_2-based/Al_2O_3 HDS catalysts have been widely studied by TEM. The results reported have shown that the MoS_2 clusters are bonded to the Al_2O_3 support surface in two different morphologies and orientations [1,2]. These have been identified as basal-bonded MoS_2 layers and edge-bonded MoS_2 layers. However, many dispersion studies of the MoS_2 phase on Al_2O_3 support only have taken into account the edge-bonded layers than the basal-bonded layers because these are easier to observe by high-resolution transmission electron microscopy (HR-TEM). Therefore, the information obtained is still incomplete and conventional HR-TEM cannot give information about basal-bonded if it is thinner than the support. The development of new detectors as the annular dark field detector in the scanning transmission electron microscopy (STEM) can help to give this information. In this work, we present evidence of both morphology and orientations. Samples of MoS_2/Al_2O_3 were prepared by conventional impregnation methods using Al_2O_3 and Mo and P salts. After impregnation, the samples were sulfide with H_2S/H_2. Powders were dispersed in isopropanol and an aliquot was deposited on a copper grid with a film Lacey/carbon. The characterization was performed in a transmission electron microscope JEM2200FS.

TEM results at low magnification showed the typical morphology of the gamma-alumina, wrinkled nanosheets, see figure 1. At higher magnification can be appreciated in the HR-TEM image of figure 2a, the edge-bonded MoS_2 layers showing their different lengths and stacking number. There is not clear evidence of basal-bonded MoS_2 layer clusters. This can be due to the thickness of the sample in the analyzed region. Figure 2b shows a thin area of the support. Chemical analysis performed from in this area were detected the Mo, S and P characteristic peaks, in addition to the peaks corresponding to the Al_2O_3. The detection of Mo, S and P in this region strongly suggest the presence of the MoS_2 phase on the Al_2O_3. However, these were not observed a different focus. Only some lattice line was revealed by the change of focus. This result suggests that the thickness of the support is bigger than the basal-bonded MoS_2 layers, absence of edge-bonded layers in this region or the Mo, S and P sintered with the support. In order to give light to these questions, a STEM study in the sample was performed and annular dark field images were obtained. The intensity observed in the image is related to the atomic number. Heavier elements produce brighter contrast than the lighter elements. The annular dark field images show the edge-bonded MoS_2 layers and polymorph plates of basal-bonded MoS_2. Both cluster types are brighter in the image. HR-STEM of a basal-bonded MoS_2 cluster shows the hexagonal arrangement of the Mo atoms in the [0001] basal plane. Therefore, a direct evidence of the basal-bonded MoS_2 structure on the Al_2O_3 support was obtained with a STEM analysis.

References:
[1] Y. Sakashita and T. Yoneda, J. catal.,**185**, 1999, 487-495.
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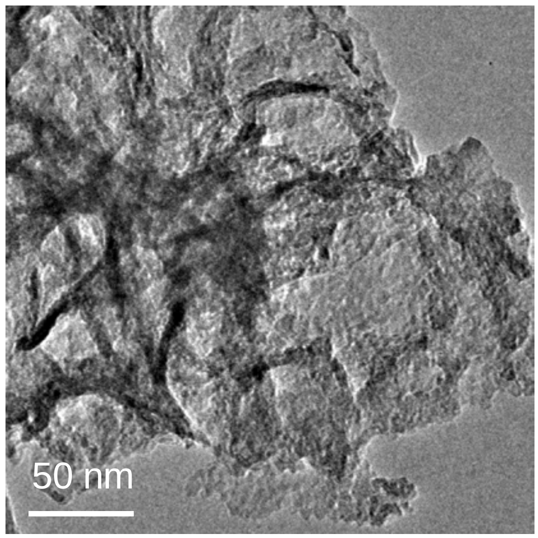
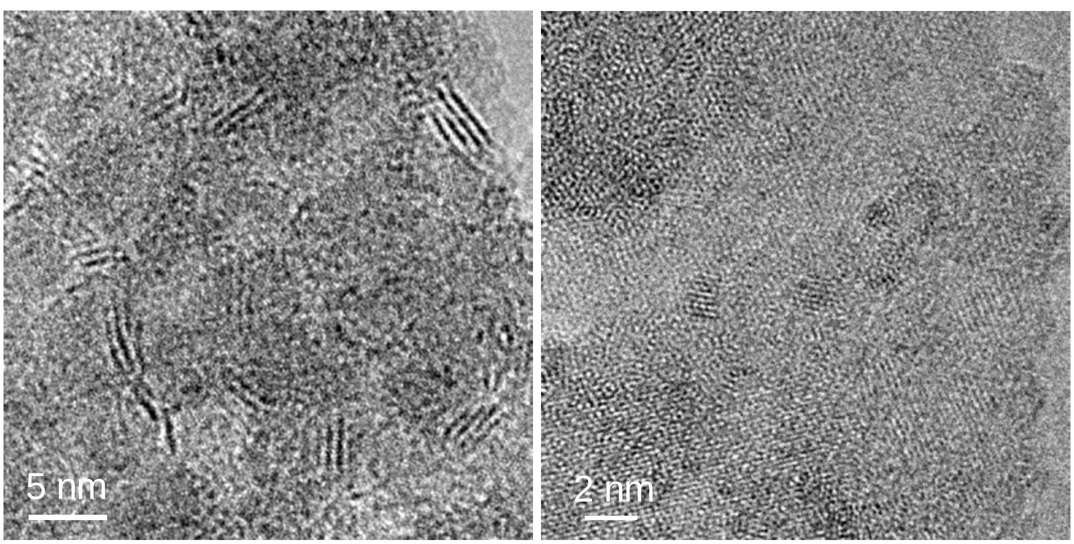


Figure 1. Bright field TEM image showing the wrinkled nanosheet of the gamma-alumina.



(a) (b)

Figure 2. HR-TEM images showing a) edge-based MoS_2 structures on the Al_2O_3 surface and b) some lattice lines in a thin region of the Al_2O_3 sheet.