Direct observation of industrial high performance Ziegler-Natta catalysts by scanning electron microscope

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Since the invention of Ziegler-Natta catalyst, more than 90% of the commercial manufacture of polyethylene and polypropylene is still based on modifications and improvements of the original ZN system. However, the mechanism of the polymerization with ZN catalysts has not yet been fully understood due to the complexity of the industrial supported catalyst system derived from the complicated interactions between the multicomponents. A great deal of efforts have been focused on characterization of the ZN system. Among a number of characterization techniques, electric microscope (EM) observation is the direct way in Ziegler-Natta catalyst characterization [1,2]. EM shows morphology of catalyst particles and energy-dispersive spectroscopic analysis of X-ray (EDS) gives element composition of them, and then material structure is clearly observed and the changes in different processes can be studied.

Our work presents a detailed morphological study of a TiCl4/MgCl2/SiO2 Ziegler-Natta catalyst for polymerization of ethylene. The secondary electronic image analysis was conducted on a FEI XL-30 Schottky thermal field emission environmental SEM. Element composition was conducted on a Thermo Noran Vantage ESI EDS. Figure 1 and Figure 2 show microphotographs of an industrial high performance catalyst and an unactive industrial catalyst sample respectively. Figure 3 gives EDS Area Scanning Spectrum of the above samples.

As a result, the high performance catalyst is found to be spherical-shaped particles with smooth surface and diameters range from 5 to 30um, compared with the unactive one with rough surface and diameters almost the same. The rough surface is spread with crystalline structure bulges. The bulges are very different from the active catalyst, containing MgCl2.4H2O crystal which is evidenced by XRD and EDS. EDS information also suggests that the unactive catalyst contain less Cl. It is possibly explained that Cl content decrease due to the reaction of TiClX with H2O or other materials, HCl is sent out. The catalyst is unactive with poor performance.

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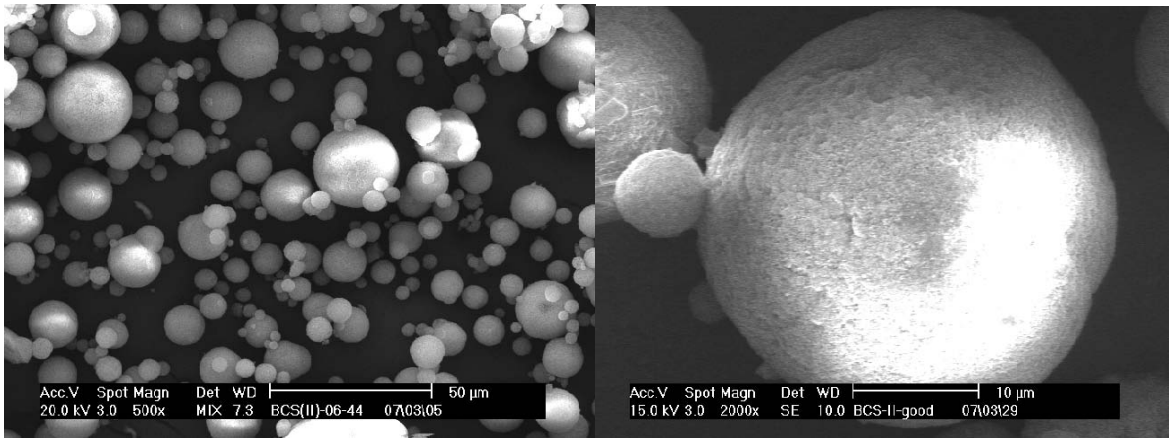

Figure 1. Surface morphology of active and unactive catalysts

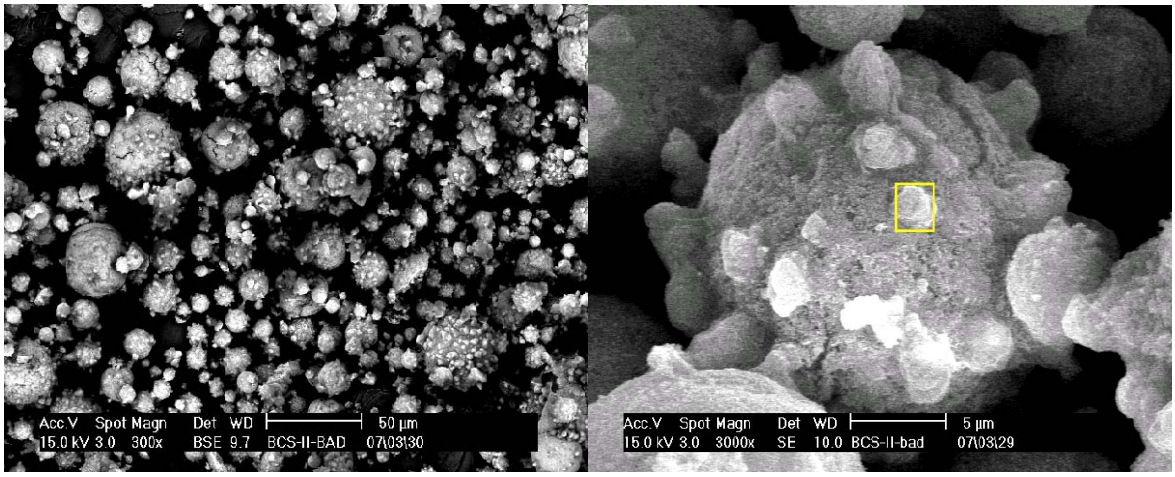

Figure 2. Surface morphology of active and unactive catalysts

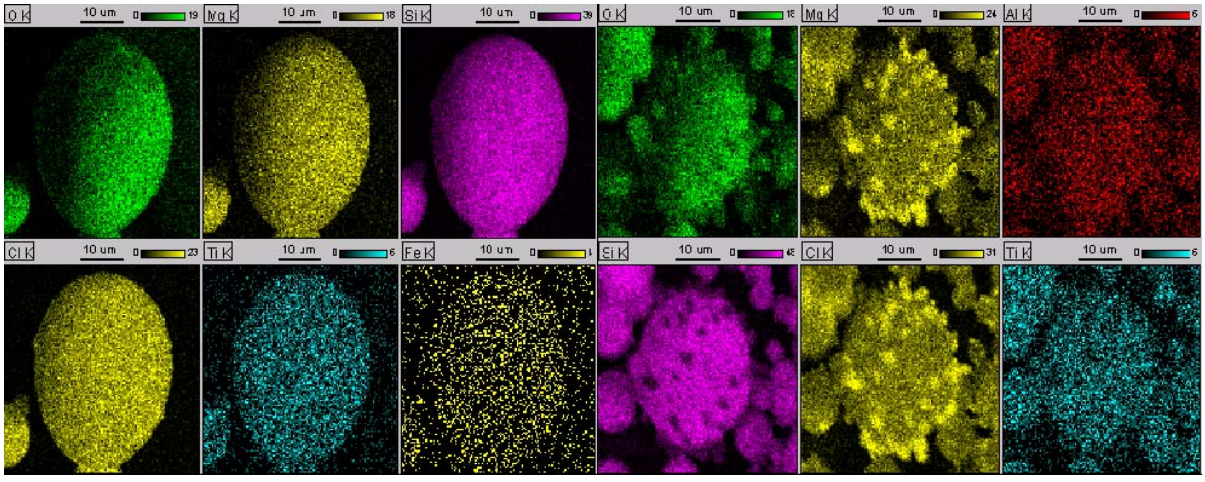

Figure 3. EDS Area Scanning Spectrum of the high and low performance catalysts

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