Ball-milling as an effective method for preparation and activation of catalysts[*]

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Conventionally, industrial catalysts are prepared by impregnaion, co-precipitation or grafting of one component onto a support. Mechanochemical preparation is technically simple and environmentally friendly. It can modify the defect structure and the electronic properties of catalytic materials, reduce particle sizes, increase the specific surface area and change the chemical state of constituent atoms. Mechanochemical treatment of the initial reagent impairs such properties that they remarkably influence the catalytic properties of the final catalyst. In this presentation, two examples are given to show advantage of this simple preparation and activation methods and how the mechanochemical treatment changes the microstructure, morphology, induce phase transition and increase the catalytic performance.

Tribochemical activation of V_2O_5 catalysts was carried out in a planetary ball-mill: 60 g of V_2O_5 together with six agate balls (1.5 cm diameter, 11g) were placed into an agate vessel (250 cm^3 volume). Milling was performed for up to 20 h at approximately 150 revolutions per minute. The most significant effect of the ball-milling is the increase of the specific surface area which increases from 7 m^2/g (precursor) to 32 m^2/g (after 10 h milling). Milling induces oxygen loss. V^{4+} and V^{3+} sites can be detected most likely distributed inhomogeneously on the surface of particles. Microstructural analysis reveals that the ball-milling process can be described as mechanical deformation followed by fine-grinding, amorphisation and reagglomeration [1]. Lattice defects are induced and the outer shell of most particles become amorphous. The defect structure at the surface and the mixture of V^{5+} and V^{4+} sites are believed to be the active sites of the V-containing calaysts.

$VOHPO_4.0.5H_2O$ (VHP) was activated to get vanadium phosphorus oxides $(VO)_2P_2O_7$ (VPO) that can be used for the selective oxidation of *n*-butane to maleic anhydride. Ball-milling change the initial needle-like particles in a blossom morphology. The particle size becomes smaller and the specific surface area increases. The VHP to VPO phase transition occurs after only 30 minutes milling in air, but the precursor changes firstly to an amorphous phase before it crystallises to VPO. This activation time is much shorter than that for the conventional chemical activation (usually followed in n-butan/N_2 mixture at high temperature for several days). The catalytic characterisation of mechano-prepared VPO reveals the same activity and selectivity as the VPO activated in vacuum or in butane/air at high temperature [2].

Reference:

1. D.S. Su, V. Roddatis, M. Willinger, G. Weinberg, E. Kitzelmann, R. Schlögl, and H. Knözinger: Catalysis Letters Vol. 74, 169 (201)
2. I Ayub, D.S. Su, R. Schlögl, A. Kharlamov, to be published

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