HRTEM observation of metal particles mobility on a $Pt/\gamma\text{-}Al_2O_3$ catalyst

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High resolution transmission electron microscopy analysis of Pt/Al_2O_3 catalyst prepared by deposition of platinum from mesitylene solution shows that the smallest metal particles on edges and corners migrate under the influence of the electron beam. Sintering is also promoted.

Keywords: microparticles; platinum catalysts; alumina support; electron microscopy

1. Introduction

Heterogeneous catalysis plays the most relevant role in the production of chemicals, and supported metals are key sectors in promoting important reactions of industrial interest [1]. A number of factors such as size and shape of particles and metal–support interactions [2,3] influence the catalytic performances of these systems. Consequently, research on catalyst preparations and pretreatments is very active [4,5], together with more and more accurate surface characterization analyses [6].

Among many physical techniques, high resolution electron microscopy gives detailed information on dispersion and morphology of metal particles [7] and on

their location on the surface of the support [8]. However, considerable energy is injected by the electron beam into the solid during the measurements, and this may well produce some consequences on the structure of the catalyst. Related to these topics, in this communication we report on some HRTEM observations of metal particles reorganization occurring under electron beam irradiation of a Pt/Al_2O_3 catalyst prepared under unusual conditions.

2. Experimental

Platinum was deposited on γ-Al_2O_3 powder (Rhône-Poulenc; SSA $\approx$ 200 m^2 g^{-1}) by impregnation at room temperature with a Pt/mesitylene solution obtained by melting at 233 K a solid matrix formally produced by cocondensation of platinum vapour and mesitylene at 77 K. The solvent was then eliminated at room temperature. The metal loading was 0.1 wt%. Details and advantages of this procedure are given in ref. [5]. For the TEM measurements the sample was ultrasonically dispersed in isopropyl alcohol and then deposited on a copper grid covered by a holey carbon film. Electron micrographs were obtained using a Jeol 2000 EX instrument (acceleration potential: 200 kV).

3. Results and discussion

TEM images show that the Pt particles are highly dispersed on the support, most particles having an average size of 8–10 Å. Some particles of ca. 20 Å diameter were also detected. Due to the reduced metal loading, the number of the observed particles was too low to allow a reliable statistical estimate of the size distribution. Upon increasing the beam energy to 25 pA cm^{-2}, the mobility of the platinum particles was promoted. After a short exposure to the electron beam (less than 10 s), metal atoms reorganized inside the Pt particles. Some particles appear as "vibrating drops", this behaviour being clearly visible in real time on the TV screen, in particular for the particles located on the corners of the microcrystals of alumina. Then the smallest metal particles start moving on the support, and two examples of the observed phenomena are shown in the figures. This phenomenon has been reported previously by Thomas et al. [9].

In the micrograph of fig. 1A two adjacent, though distinct, particles 20 and 4 Å in size are clearly visible, the largest exhibiting a quite well defined hexagonal shape. Under beam irradiation such two particles progressively merge, and after few seconds a single larger particle (22 Å in size) is produced (fig. 1B). Details of a different region of the sample, characterized by finely dispersed particles ($\sim$ 10 Å in size), are reported in fig. 2. The sequence of two micrographs shows that the effective movement of the Pt particle evidenced (see arrow) on the support, the distance covered being about 20 Å.

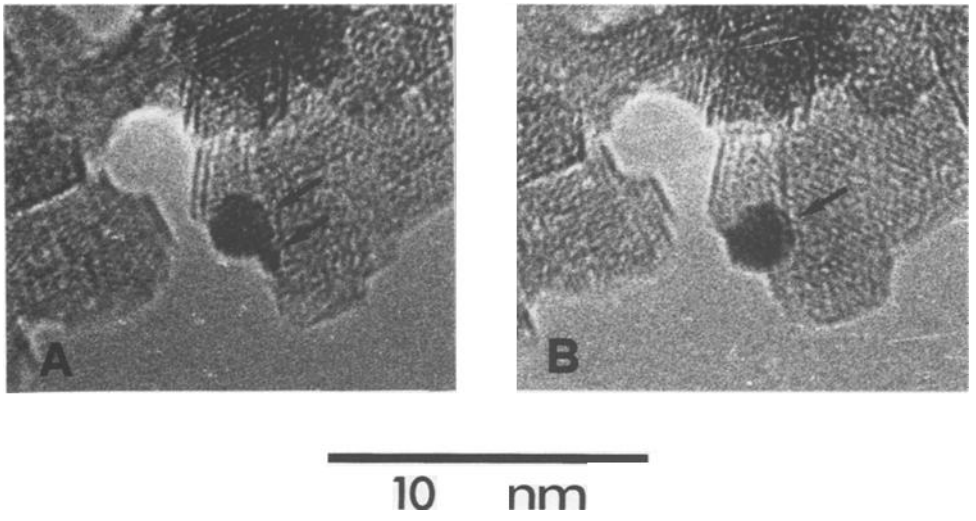

Fig. 1. HRTEM micrographs showing two metal particles of a Pt/Al_2O_3 catalyst merging under electron beam irradiation. (A) Immediately after image focusing; (B) after 10 s exposure to the electron beam.

Observation in real time indicates that metal particles move on the support with different rates, depending on the morphological features of the alumina surface. In particular, they move faster along the edges between the alumina microcrystals than on the flat surface of the platelets.

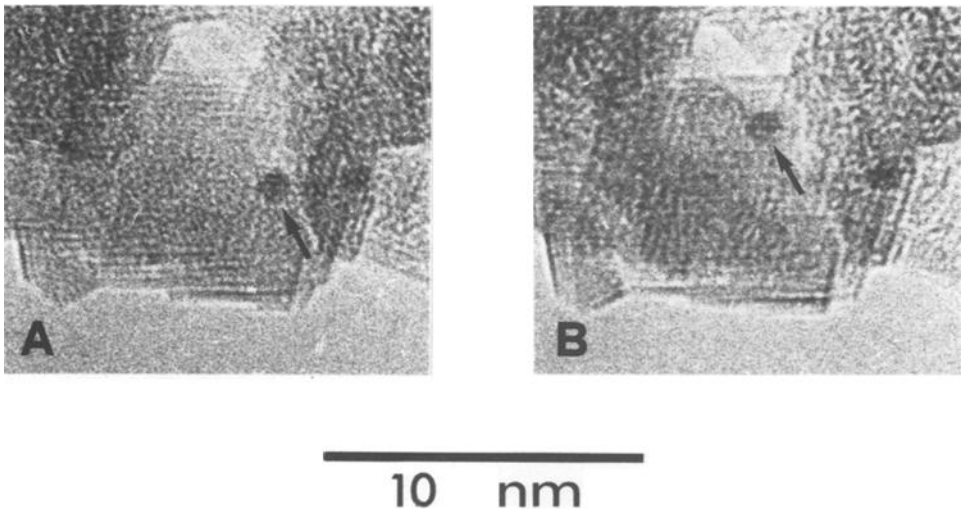

Fig. 2. HRTEM micrographs showing a metal particle of a Pt catalyst moving on an Al_2O_3 support under electron beam irradiation. (A) Immediately after image focusing; (B) after 10 s exposure to the electron beam.

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