

TITLE OF RESEARCH: STRUCTURAL CHARACTERIZATION OF DISPERSED METAL CATALYSTS

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RESEARCH SCOPE AND OBJECTIVES

The objective of the research project is to obtain a detailed structural and compositional characterization of dispersed metal catalysts in order to gain greater insight into metal-support interactions and the role of such interactions in determining catalyst activity, specificity and deactivation.

DESCRIPTION OF RESEARCH EFFORT

In the first year of the project, Analytical Electron Microscopy (AEM) and X-Ray Diffraction (XRD) characterization techniques have been developed to investigate the structural features of small metal particles (5-10 nm) in porous media. The systems investigated include Ni/silica, Ni/magnesium silicate, Au/silica and Co/ZSM-5. In the first phase of the project, bright and dark field imaging techniques have been emphasized with EDAX characterization. High quality images have been obtained in both bright field and dark field and earlier characterization results on three of the catalyst systems have been confirmed and extended. Particle size growth (sintering) at low temperatures is much greater in the Au/silica system, indicating a weak metal-support interaction. A more uniform, generally smaller particle size distribution, is observed in Ni/magnesium silicate compared to Ni/silica. Hydrogen chemisorption studies show that the metal surface area is higher in Ni/magnesium silicate, which also indicates a smaller metal particle size. The methanation turnover number is lower in Ni/magnesium silicate, however, indicating that a smaller fraction of the surface sites are active in Ni/magnesium silicate.¹ Detailed structural studies of cobalt particles in Co/ZSM-5 show that the particles contain a high concentration of basal plane stacking faults. Large nickel particles observed in Ni/silica show contrast effects which may be indicative of faults or twins.

FUTURE RESEARCH

Microdiffraction studies will be carried out on unsupported gold particles and particles supported in silica and a polymer matrix. The objective will be to determine if distorted microdiffraction patterns,

which have been obtained for Ni in both silica and magnesium silicate, are associated with support effects, the passivating oxide layer or strains in the metal particles due to surface tension effects.

Detailed characterization studies will be carried out on the metal particles and in regions of the support immediately adjacent to the metal particles in the next phase of the program. The large nickel particles that can be observed in the silica support will be carefully examined in order to determine whether internal structure details are associated with the type of lattice faulting which has been observed and characterized in cobalt particles in a ZSM-5 support.²

AEM and XRD characterization studies will be carried out on Ni/silica catalysts that have been reduced and sintered at temperatures between 500°C and 800°C. The objective will be to seek evidence for the existence of Ni-Si alloy particles which may result from a high temperature metal-support interaction. The effect of adding oxides such as chromium oxide and cerium oxide to the silica and magnesium silicate supports will also be evaluated in the next phase of the project.

REFERENCES

1. P. J. Reucroft, H. Parekh, P. Ganesan, S. N. Russell and B. H. Davis, *Applied Catalysis*, 3, 65 (1982).
2. A. G. Dhere, R. J. De Angelis, J. Bentley and P. J. Reucroft, *J. Molec. Catalysis*, in press.