Helium ion microscopy and electron microscopy on high performance gas-atomised Raney-type nickel catalysts

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Nanostructured Raney-type Ni catalysts have been used in industry for the production of a wide range of chemicals since the 1920s.[1] In the EU supported project IMPRESS it has been shown that by using gas atomisation processing [2] high surface area particles with significantly increased catalytic activity in hydrogenation reactions can be produced.[2] Complementary structural investigations with helium ion microscopy and electron microscopy were carried out in order to link processing, structure and catalytic activity.

The Raney-type Ni catalyst particles were produced by gas-atomisation processing from undoped and doped Ni-Al alloys. Subsequently the particles were sieved into different size fractions. After activation by leaching with $NaOH_{aq}$ and prior to the structural investigations the samples were slowly passivated with oxygen.

The samples were investigated by helium ion microscopy (Zeiss Orion) in order to image the surface of the sample. Due to the outstanding imaging properties of the helium ion microscope, (i) very high depth of focus, (ii) the strong contrast as a result of the small excitation bulb and (iii) the sub-nanometer resolution it was possible to image the details on the surface of the particles on several relevant length scales, see Figure 1.

The inner microstructure of the particles was investigated by scanning electron microscopy (SEM) on embedded cross sectioned samples, using back scattered electron imaging and EDX mapping. Back scattered electron (BSE) images show the inner dendritc structure of the particles. The EDX maps show the elemental distribution, see Figure 2.

The inner nanostructure of the sample was investigated by TEM, see Figure 3a. The crystal lattice of the nanocrystalline mesopore walls was investigated by Cs-corrected high resolution transmission electron microscopy (HRTEM), see Figure 3b. The mesopore walls are nanocrystalline and the crystalline show monolayer steps on the surface. The HRTEM images show the sites for the catalytic reaction.

1. M. Raney: United States patent 1 563 587 (1925)
2. F. Devred A.H. Gieske, N. Adkins, U. Dahlborg, C.M. Bao, M. Calvo-Dahlborg, J.W. Bakker, B.E. Nieuwenhuys, Applied Catalysis A: General 356, 154-161 (2009)

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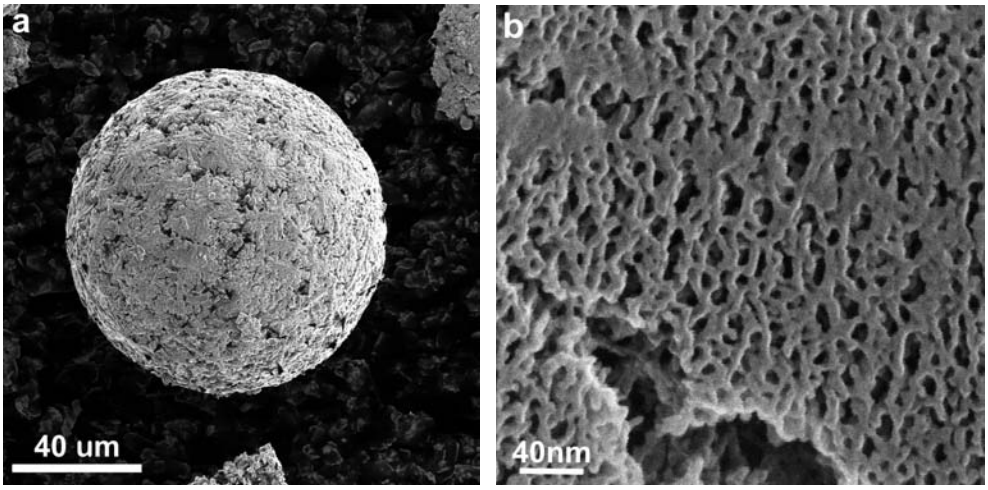

Figure 1. Helium ion microscopy images of a gas-atomised Raney-type Ni catalyst. a) The image shows the whole spherical particle. b) The image shows the mesopores on the surface.

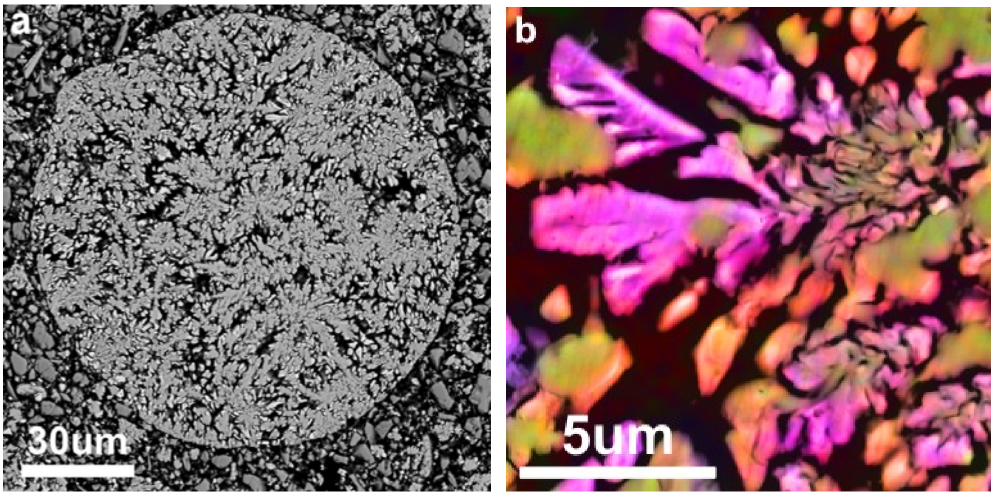

Figure 2. Cross section SEM images of a resinated catalyst particle. a) BSE image showing the inner morphology. b) EDX elemental map Ni – red, Al – green, dopant – blue.

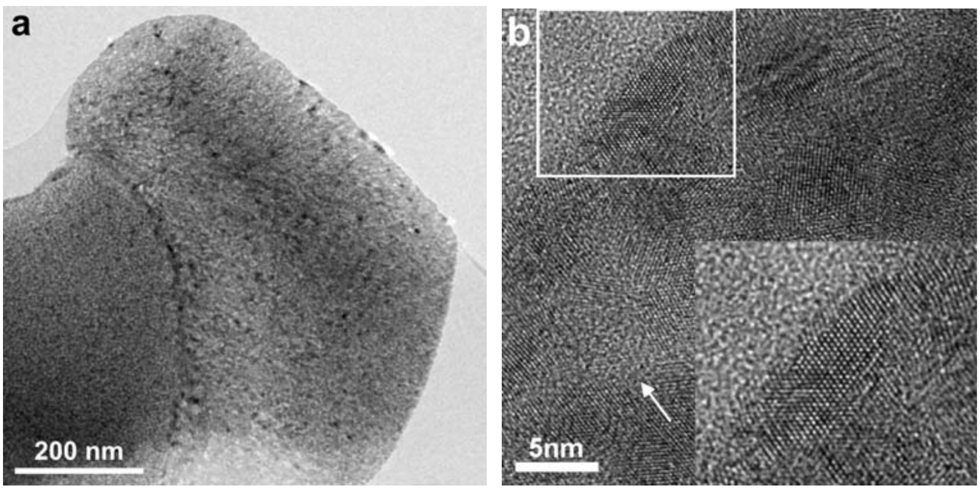

Figure 3. TEM images of resinated dendrite tips of an undoped sample. a) Bright field image of the a dendrite tip with the mesoporous layer on top. b) Cs corrected HRTEM image of the mesoporous layer showing the pores, see arrow, and the nanocrystalline mesopore walls.

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