Reactivity of carbon deposits on noble metal catalysts

DEFECTS IN Si-DOPED GaAs GROWN BY MOCVD

A.J. Bons, X. Tang* and F.W. Schapink

*Laboratory for Metallurgy, Delft University of Technology, Delft, The Netherlands; *Department of Experimental Solid State Physics, Faculty of Science, University of Nijmegen, Nijmegen, The Netherlands*

GaAs epitaxial layers grown by Metal-Organic Chemical Vapour Deposition (MOCVD) have been doped with Si by adding silane (SiH_4) to the reactant gases. Thermodynamic calculations predict that Si-precipitates will form when the silane mole fraction exceeds 1×10^{-6}. In order to check this prediction, a set of samples grown with 6.2×10^{-8}, 6.3×10^{-7} and 5.8×10^{-6} mole fraction silane has been studied with TEM, to see whether precipitates have been formed. Both plan-view and cross-sectional specimens have been studied using a Philips EM400T operating at 120 kV. Defects have only been observed in the samples grown with 5.8×10^{-6} mole fraction silane. These defects are small dislocation loops (diameter ca. 30 nm) lying on {110} planes. Such dislocation loops are the typical precipitates in GaAs heavily doped with Si during bulk growth or by ion implantation. This is the first time such precipitates have been observed in MOCVD-grown layers. It is concluded that the TEM observations support the theoretical prediction that Si-precipitates are formed when the silane concentration exceeds a mole fraction of 1×10^{-6}.

CRYO-SCANNING ELECTRON MICROSCOPY OF THE SHOOT APEX OF CELERIAC (APIUM GRAVEOLEUS VAR. RAPACEUM)

R. Booij, F. Thiel*, D.J.J. Meurs and A. Boekestein*

*Centre for AGrobiological Research (CABO) and *Technical and Physical Engineering Research Service (TFDL), Wageningen, The Netherlands*

In vegetable crops grown for the vegetative parts of the plant (bulbs, leaves, roots), generative parts like flowers are undesirable. The risk of bolting of celeriac is high under field conditions. With cryo-scanning electron microscopy it is possible to examine the very early stages of the morphological changes in the shoot apex induced by environmental conditions like temperature and photoperiod. Cryofixation in nitrogen slush was used to maintain the surface morphology of the apex in a reliable and quick way. The frozen-hydrated specimens were then transferred to the preparation chamber of the EMSCOPE SP2000A system for cryosputtering with gold. Finally the specimens were transferred to a JEOL 35C scanning electron microscope equipped with a cold stage. The most characteristic developmental stages have been used to analyse the effects of environmental factors on flower induction.

MODERN DEVELOPMENTS IN ELECTRON MICROSCOPY OF METALS

G. Boom

Department of Applied Physics, Materials Science Centre, University of Groningen, Nijenborgh 18, 9747 AG Groningen, The Netherlands

In electron microscopy of metals, and non-metals alike, there is a growing interest in interfaces between grains, between phases, and in composites between the matrix and embedded material, if possible on the atomic scale. There is also increasing interest in materials of 5-fold symmetry, sometimes in very small grains. Diffraction experiments on small areas are then needed for identification. Determination of elemental composition on images on the atomic scale have been reported, both with electron energy-loss spectroscopy and X-ray microanalysis. Especially in atomic-resolution microscopy, image simulations are needed. In total, modern electron microscopy involves not only super-microscopes, but also computer systems, preferably coupled to high-speed image analyzers.

REACTIVITY OF CARBON DEPOSITS ON NOBLE METAL CATALYSTS

R.J. Boom, A.D. van Langeveld and J.W. Niemantsverdriet

Laboratory of Inorganic Chemistry and Catalysis, Eindhoven University of Technology, Eindhoven, The Netherlands

Carbon deposits form an essential ingredient of a catalyst. They influence the activity, selectivity and stability of catalysts in industrially important reactions such as naphtha reforming. The

reactivity of this carbon toward oxygen is of practical interest, because deactivated catalysts are regenerated by oxidation. Here we use Auger spectroscopy (AES), secondary ion mass spectrometry (SIMS), and temperature-programmed reaction spectroscopy (TPRS) to show that oxidation provides a sensitive test for the reactivity of carbon on noble metals.

Carbon deposits, prepared by exposing the metals to ethylene at different temperatures, were characterized with AES and SIMS. The temperature at which the carbon reacts with oxygen depends critically on the hydrogen content of the deposit and on the substrate: carbon on iridium is significantly more reactive toward oxygen than on platinum. The oxidation temperatures of carbon on alumina-supported Pt and Ir catalysts as determined in a catalytic reactor are in good agreement with those measured on the metal foils in UHV.

QUANTITATIVE X-RAY MICROANALYSIS OF VOLUTIN GRANULES IN ACINETOBACTER

E.M. Bouw, A. Boekestein and M. Deinema*

*Technical and Physical Engineering Research Service and *Department of Microbiology, Agricultural University, Wageningen, The Netherlands*

Strains of Acinetobacter are able to accumulate large amounts of polyphosphate which is present in the so-called volutin granules. The bacterial cells, containing many granules, were cryosectioned in an LKB ultratome III cryo-ultramicrotome and subsequently transferred to a carbon evaporation device. Good preserved sections containing recognizable parts of the bacteria were analyzed in the cold stage of a Philips EM400T scanning transmission electron microscope at 80 kV accelerating voltage and 24° tilt angle. Quantification was achieved using cryosectioned standards composed of gelatin, glycerin, KH_2PO_4 and water. Both standard and specimen sections were freeze-dried prior to analysis in order to improve peak-to-background ratios. To calculate mass fractions of P in the volutin granules, the continuum-normalisation method of Hall was followed, using an extraneous background correction by measuring the background intensity on the supporting film in the vicinity of the sections. Measured values of P in the granules varied from 30 to 35%, which is in agreement with analyses using other methods and which supports the hypothesis that the volutin granules are almost entirely composed of pure polyphosphate.

INTEGRATED IMAGE ANALYSIS AND ELECTRON ENERGY LOSS SPECTROSOCPY IN EM

W.C. de Bruijn, C.W.J. Sorber, T. de Jong and J.B. van Dort*

*AEM Unit, Clin. Pathol. Inst. Erasmus University, PO Box 1738, Rotterdam; *Hogeschool West-Brabant, Lab Onderwijs, Etten Leur, The Netherlands*

The combination, Zeiss EM 902 microscope with an IBAS-2000 image analysis system, allows morphometrical quantitation and integration of images from element-related electron energy loss (EELS) with cell-structure-related inelastically scattered electron populations (ESI).

Contrast thresholding has been applied to such spectroscopical images to obtain appropriate and objective segmentation. In glutaraldehyde-fixed cells, chemical elements in two cell components have been morphologically analyzed: one intrinsic element and a cytochemical reaction product.

It is shown that for the intrinsic nuclear phosphate and for a barium-containing reaction product in the RER (from an arylsulfatase reaction) a volume fraction can be estimated.

The use of an ion-exchange bead type of Bio-standard (Polaron, UK) has recently been advocated for elemental concentration estimations, in X-ray microanalysis. It has been shown that a constant ratio, representing the relative concentration (R_x) of these standards, could be obtained.

For each type of standard a proportionality constant has been calculated, that relates R_x to the standard's mean (C_x) concentration value, estimated by neutron activation analysis. For quantitative EELS analysis the constancy of R_x is investigated for standards containeither Ca, Co, Ni, Fe or Cu. Special attention is given to deliberately introduced section thickness variations, to show that, within limits, the R_x value is independent of section thickness.