MESA+ INSTITUTE FOR NANOTECHNOLOGY
NanoLab Twente

TEM EXAMPLE

EFTEM in interdiffusion studies

INTRODUCTION

Electron Energy Loss Spectroscopy (EELS) is a technique in which the energy distribution of electrons that have exited the specimen is analysed. Part of these electrons may have lost energy by inelastic scattering effects, while other electrons may have lost no energy at all. By analysing the energy-loss events, in principle a wealth of chemical information can be derived from the specimen. Regarding the detection ability of chemical elements, EELS can be considered a complimentary technique to Energy Dispersive X-ray Spectrometry (EDX), since it is highly sensitive for the light elements.

Energy-Filtered TEM, also known as EFTEM, is a derivative technique of Electron Energy Loss Spectroscopy. EFTEM is an ideal tool for, e.g., chemical element mapping and analysis in a TEM in which the specimen is irradiated by a broad beam of high-energetic primary electrons so that images are obtained by parallel acquisition. The basic principle of EFTEM is that electrons of a specific energy are selected (or “filtered-out“), and after being transferred through the spectrometer they form an image. In this way, elemental mappings can be generated in a relatively fast and easy fashion (EFTEM image mode).

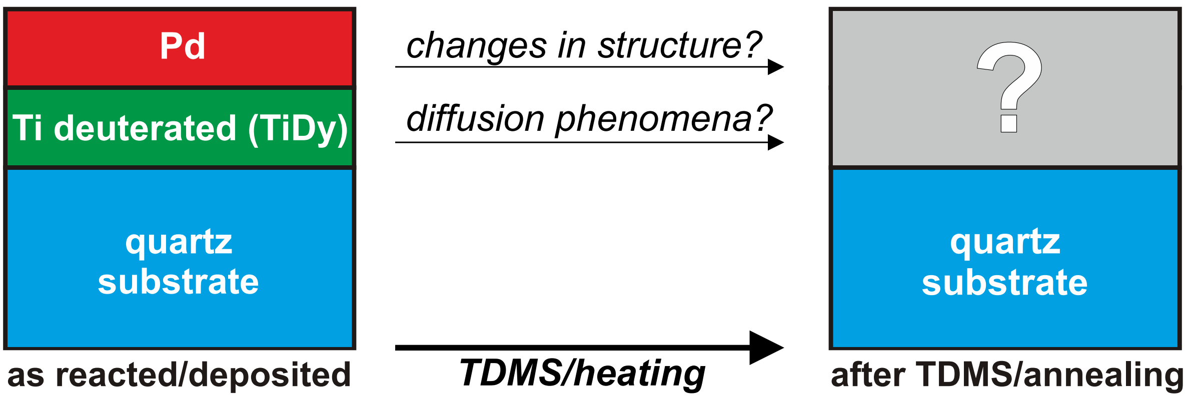
Summarising, EFTEM may be used as:
- **A mapping technique**. It creates elemental/chemical maps by forming images with inelastically scattered electrons.
- **A contrast enhancement technique**. It improves contrast in images and diffraction patterns by removing inelastically scattered electrons that produce background “fog”.

Our TEM is equipped with a so-called post-column GATAN Imaging Filter, model Tridiem. The filter is basically a GATAN Parallel EEL Spectromer with an energy-selecting slit after the magnetic prism and a CCD detector as final recording device.

For an easy overview of TEM, read our first Application Note “TEM EXPLAINED“. For thorough background information on the techniques mentioned above, EELS, EFTEM, EDX, TEM, may we refer to the excellent text book by Williams and Carter [1].

FORMULATION OF ANALYSIS PROBLEM

Ultrathin bilayer films of titanium deuteride and palladium, TiDy/Pd, have been investigated by Thermal Desorption Mass Spectrometry, TDMS [2]. As a result of TDMS-induced evolution of deuterium, it was important to know if structural changes and/or interdiffusion effects had taken place in these films:



ANALYSIS METHOD: ENERGY-FILTERED TEM

EFTEM was used since it allows a quick overview of the elemental distribution in the film of interest, before and after the TDMS/heating stage, while Bright-Field TEM imaging at the same time provides an insight into possible structural changes of the bilayer.

[1] “Transmission Electron Microscopy (A Textbook for Materials Science Basics, Diffraction, Imaging, Spectrometry)”, by David B. Williams and C. Barry Carter. Plenum Press, 2nd Edition, 2009: softcover: ISBN 978-0-387-76502-0

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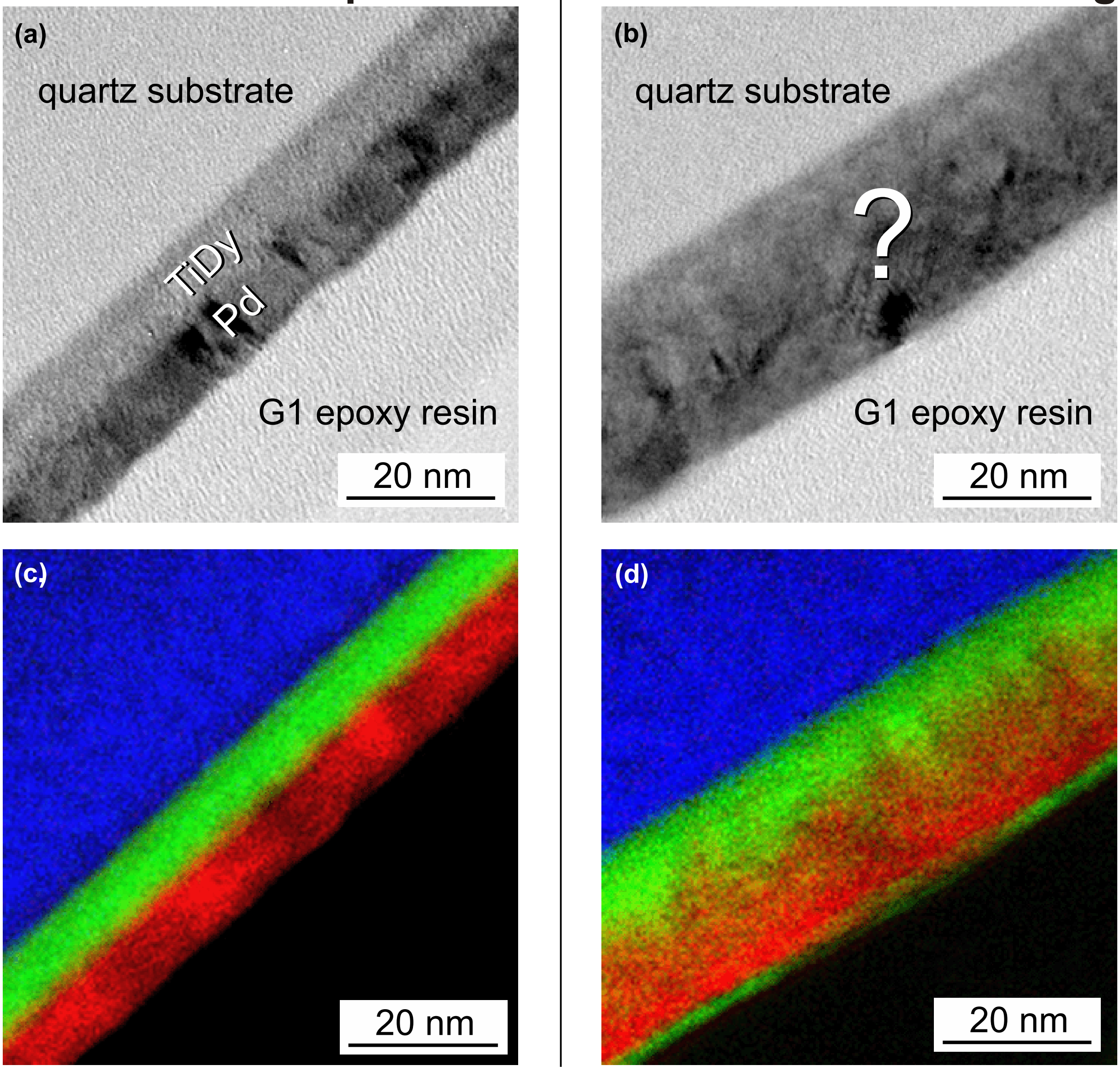
RESULTS ENERGY-FILTERED TEM AND BRIGHT-FIELD TEM

The most relevant results of the experiment before and after TDMS processing are condensed into the pictorial assembly below:

- The Bright-Field TEM images (a) and (b) indicate that significant structural changes must have occurred in the TiDy/Pd bilayer upon TDMS processing.
- Significant interdiffusion of Ti and Pd in the TiDy/Pd bilayer (compare c with d).
- Enrichment of Ti at the surface of the Pd film (d).

as reacted / deposited | **after TDMS / annealing**



*Displayed in the image gallery below are the Red Green Blue composite images of the ultrathin TiDy/Pd film taken before (c) and after TDMS (d). The RGB images are created by superimposing the elemental EFTEM maps of **Si** (blue), **Ti** (green), and **Pd** (red). The associated Bright-Field cross-sectional XTEM images of both films are presented on top of each column (a, b).*

CONCLUDING REMARKS

EFTEM provides direct visual evidence for an enrichment of Ti at the top of the Pd film surface, as a result of extensive interdiffusion of Ti and Pd within the Ti-Pd bilayer film. Corroborated by EDX and XPS, this result has been of tremendous importance to help understand the kinetics of deuterium desorption during TDMS processing [2].

[2] "Application of TEM and XPS in the interpretation of the kinetics of deuterium evolution from ultrathin TiDy/Pd films evaporated on quartz", W. Lisowski, E.G. Keim, Analytical and Bioanalytical Chemistry, Vol. 393, Nr. 8, April 2009, 1923-1929; ISSN 1618-2642.