HITACHI SCIENTIFIC INSTRUMENT TECHNICAL DATA STEM

SHEET NO. 1

SUBJECT: AN APPLICATION OF THE HD-2000 TO MATERIALS CHARACTERIZATION OF CATALYST

INSTRUMENT: THE HD-2000 SCANNING TRANSMISSION ELECTRON MICROSCOPE

CONTENTS:

1. INTRODUCTION

The HD-2000 is a new microscope which is easy to operate just like SEMs and performs as well as high-resolution TEMs. In addition, it allows high sensitivity elemental microanalysis. It has detectors for secondary electrons, transmitted electrons, and scattered electrons, all of which are useful for materials characterization.

In this Technical Data, we introduce an application to a catalyst (Pt) using the HD-2000.

Note:

SEM stands for Scanning Electron Microscope.

TEM stands for Transmission Electron Microscope.

STEM stands for Scanning Transmission Electron Microscope.

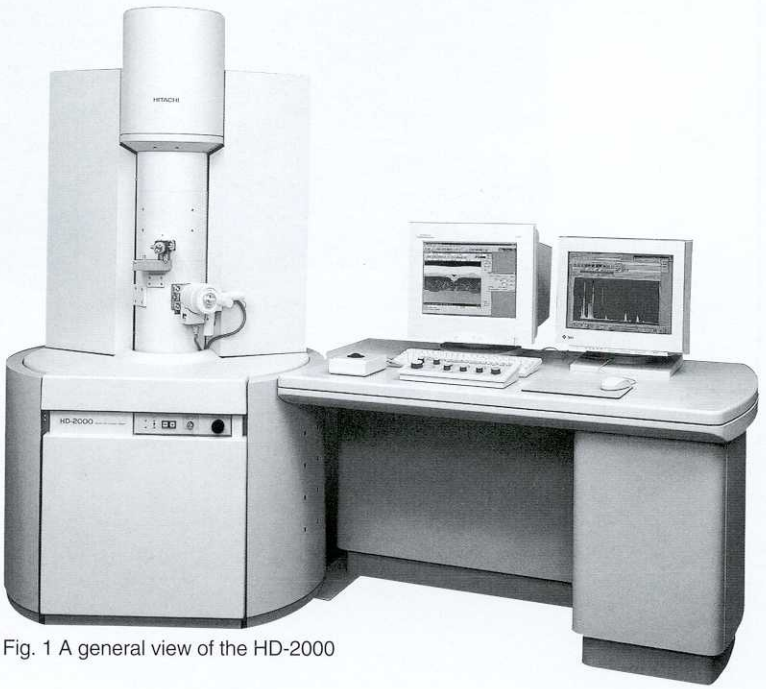

Fig. 1 A general view of the HD-2000

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2. INSTRUMENTATION

Fig. 1 shows a general view of the HD-2000. Fig. 2 shows a schematic diagram of the instrument. Signal detectors include one secondary electron detector and two transmitted electron detectors; one for Bright Field or BF STEM imaging and the other a High Angle Annular Dark Field or HAADF detector for collecting electrons scattered at high angles. The Bright Field STEM image exhibits image contrast similar to that available with a conventional TEM image. The HAADF STEM image exhibits image contrast which reflects atomic numbers and density of elements in the specimen. An energy dispersive X-ray (EDX) spectrometer is available at option. It allows elemental microanalysis using X-rays which are characteristic of the elements present in the specimen. In this Technical Data, we will show results obtained using a Vantage EDX system supplied by Noran. The HD-2000 and the Vantage combination allows a large solid angle of the X-ray detector which is about 0.3 sr (about 2.5 times that of conventional systems) and permits a high sensitivity elemental microanalysis.

3. SPECIMEN

The specimen is Pt particles on graphitized carbon. The specimen was crushed in a mortar, dispersed in an organic solvent and applied in drops onto a microgrid with supporting membrane.

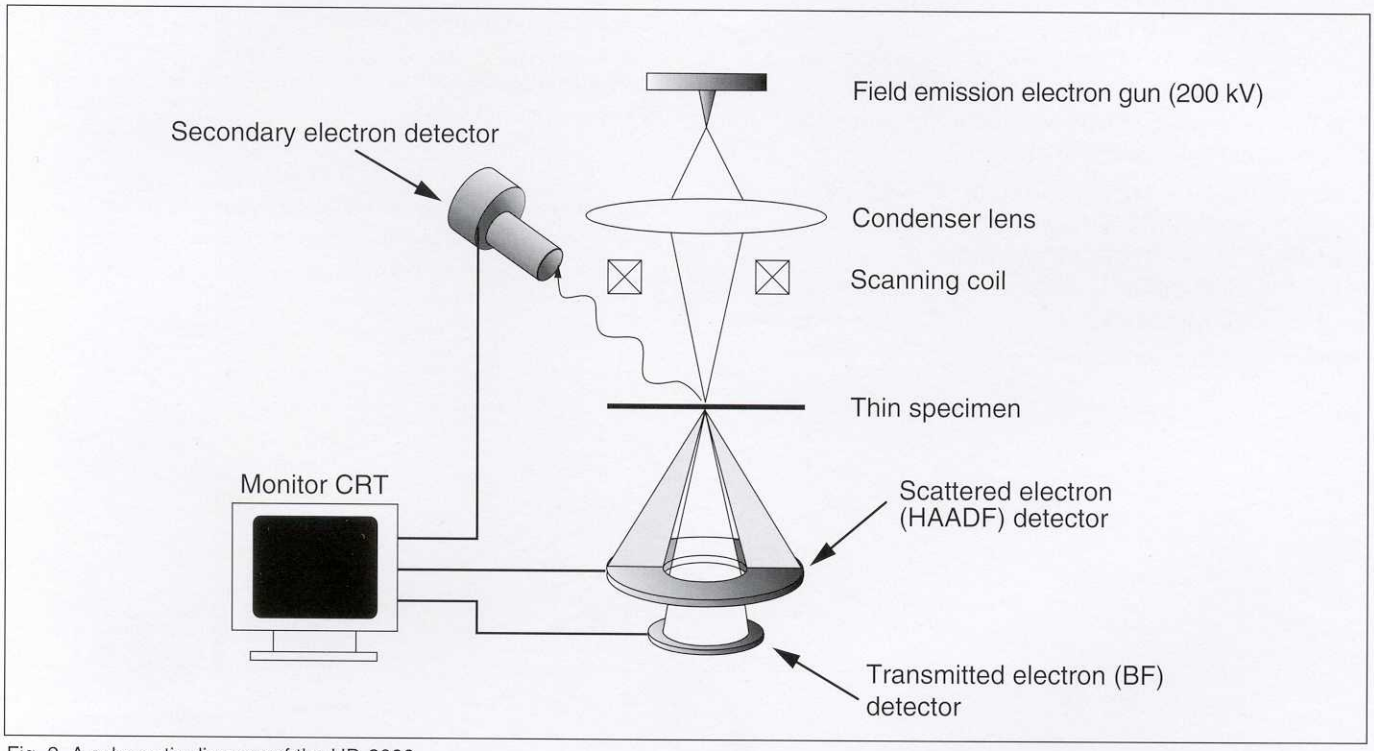

Fig. 2 A schematic diagram of the HD-2000

4. OBSERVATION OF DISPERSION OF CATALYST PARTICLES

4.1 Observation of Pt particles on carbon

Fig. 3(a) shows a secondary electron image of the specimen. This image shows 3D structure of carbon carrier and dispersion of Pt particles on the carrier also in 3D. The operating voltage of the HD-2000 is 200 kV which is about 10 times higher than most conventional SEMs. The HD-2000 exhibits secondary electron images like this one with contrast similar to the SEMs.

4.2 Observation of carbon structure and Pt particles attached on it

Fig. 3(b) shows a bright field image of Fig. 3(a) and a dark field image in Fig. 3(c), both of which are from the same field of view. The bright field image shows the particles on front and back of the carrier. The dark field image shows the carbon and Pt particles at much higher image contrast due to their atomic number and density differences. From this image we can learn the distribution of Pt particles without using the X-ray spectrometer.

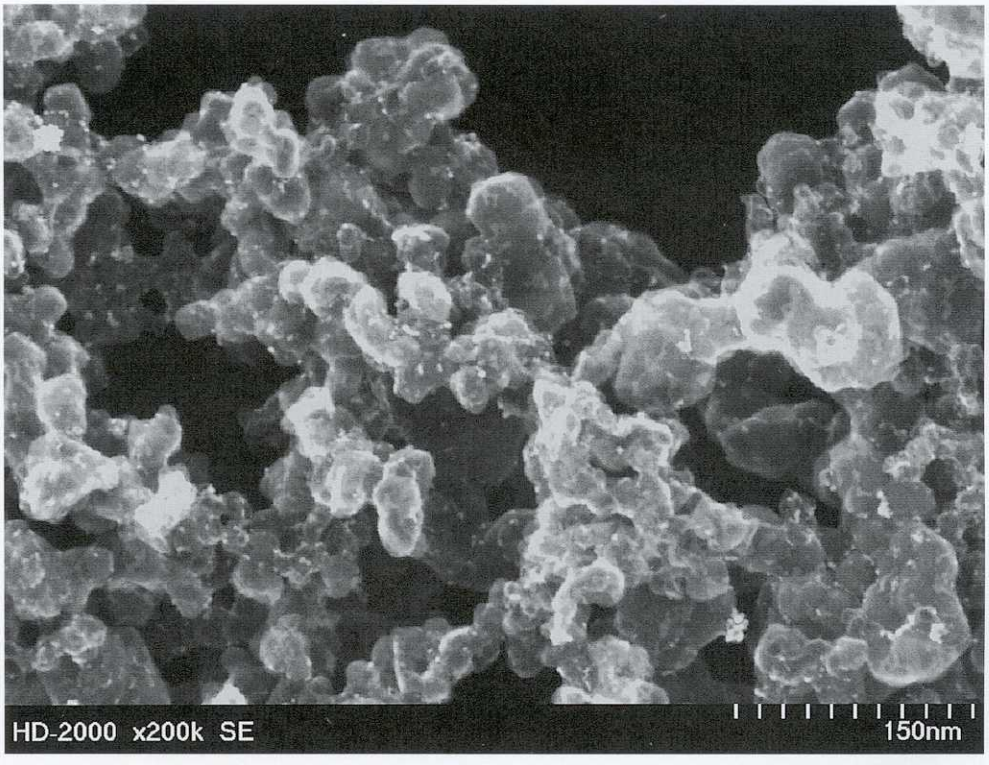

Fig. 3(a) Secondary electron image Accelerating voltage: 200 kV
Magnification: x200,000
Operation mode: Normal

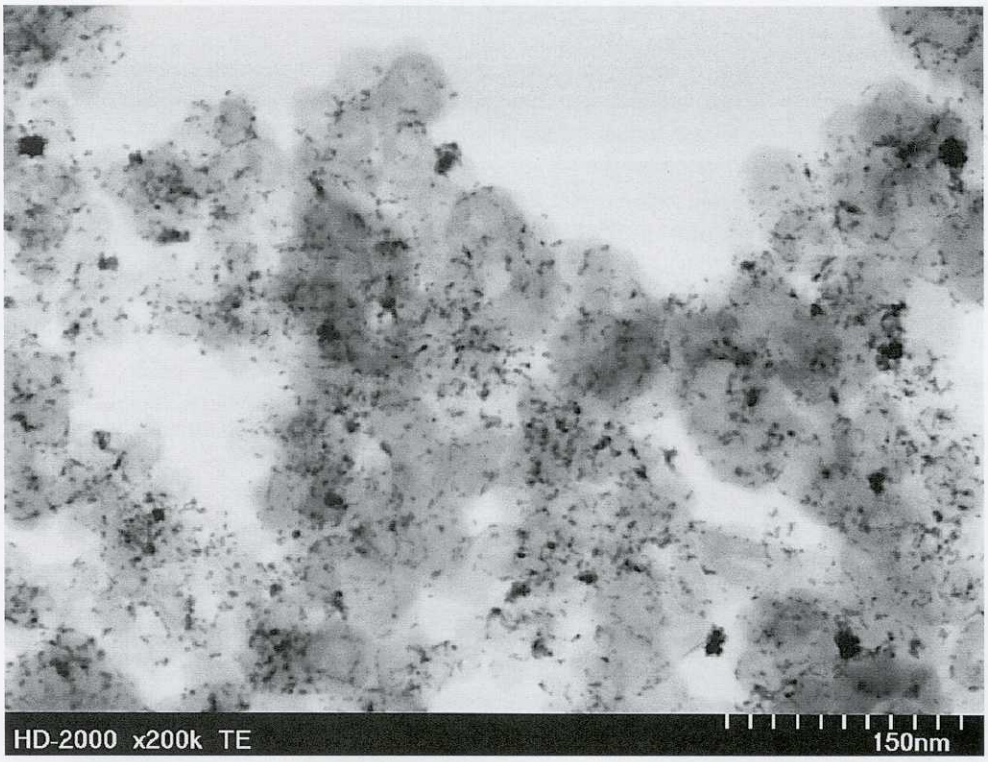

Fig. 3(b) Bright field image

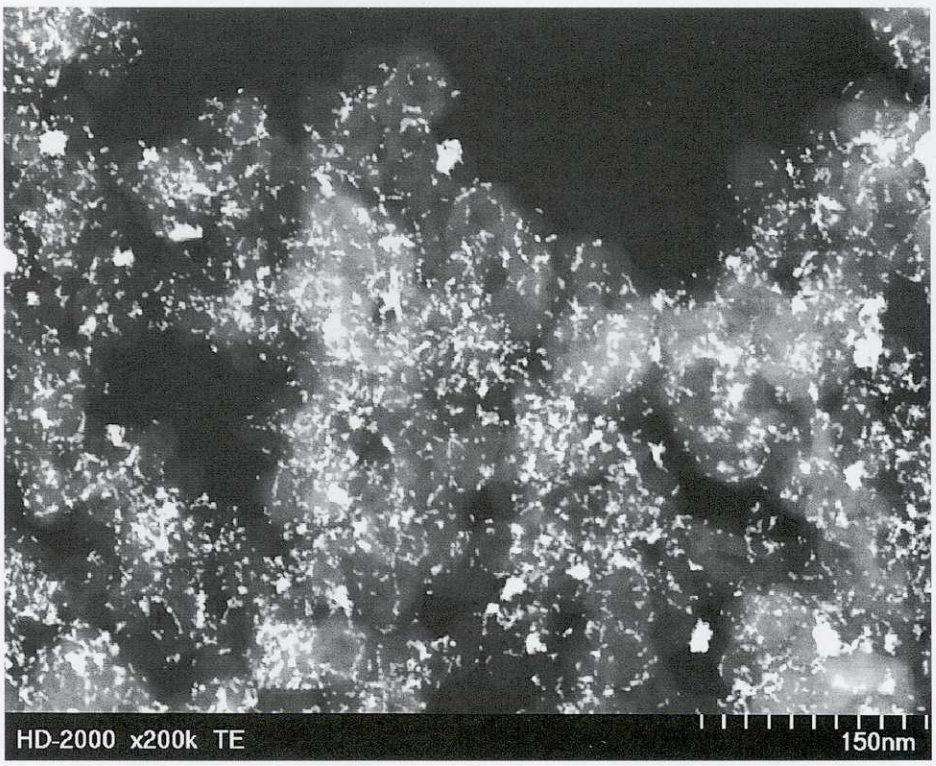

Fig. 3(c) Dark field image Accelerating voltage: 200 kV
Magnification: x200,000
Operation mode: Normal

5. MICROSCOPY OF FINE STRUCTURES OF CATALYST PARTICLES

Fig. 4 shows a high resolution image. Here, we can see crystal lattice spacing of 0.34 nm of graphitized carbon (002) and 0.23 nm of Pt (111) very clearly. High resolution microscopy shows crystal structures and orientations of the carrier and particles directly on the image.

6. ELEMENTAL MICROANALYSIS OF FINE CATALYST PARTICLES

Fig. 5 shows an analytical result of the fine particles. Analysis points "a" and "b" are shown on the micrograph. An electron probe of about 0.3 nm was used. Acquisition time was about 10 seconds. With most conventional systems, an acquisition time of about 30 seconds or more has been required. During such a long acquisition time, fine particles do not stay stable so that it has been difficult to analyze chemical compositions of fine particles with conventional systems. The HD-2000 allows elemental analysis of these fine particles of about 0.5 nm within a short time. We have detected Pt signal of about 30 counts very quickly. We think that it is possible to analyze carrier particles qualitatively in a short time.

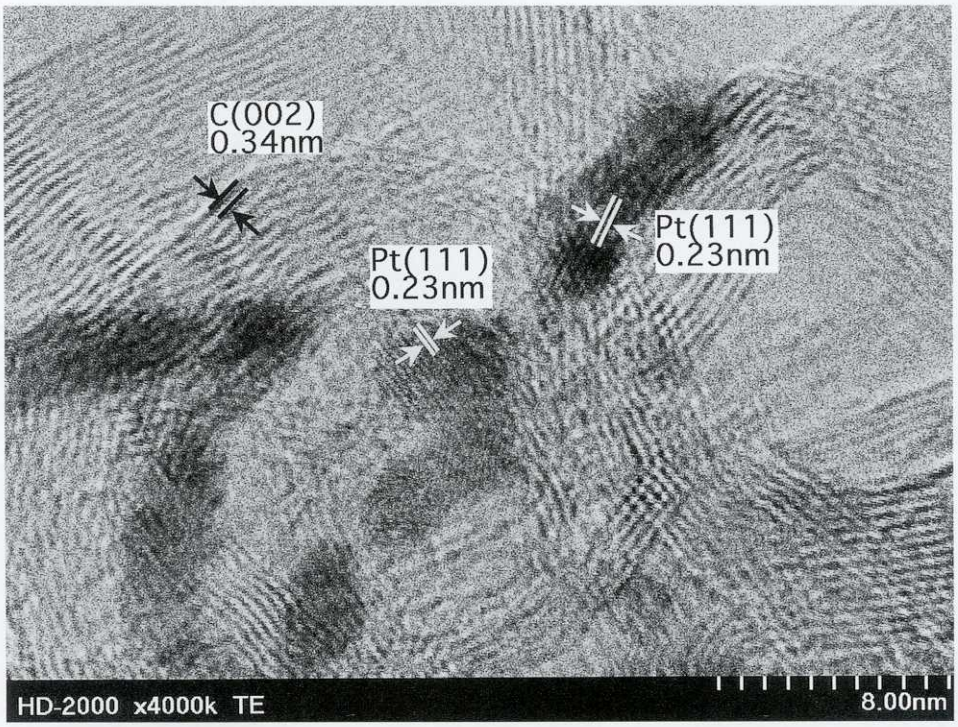

Fig. 4 High resolution image Accelerating voltage: 200 kV
Magnification: x4,000,000
Operation mode: Ultra high resolution

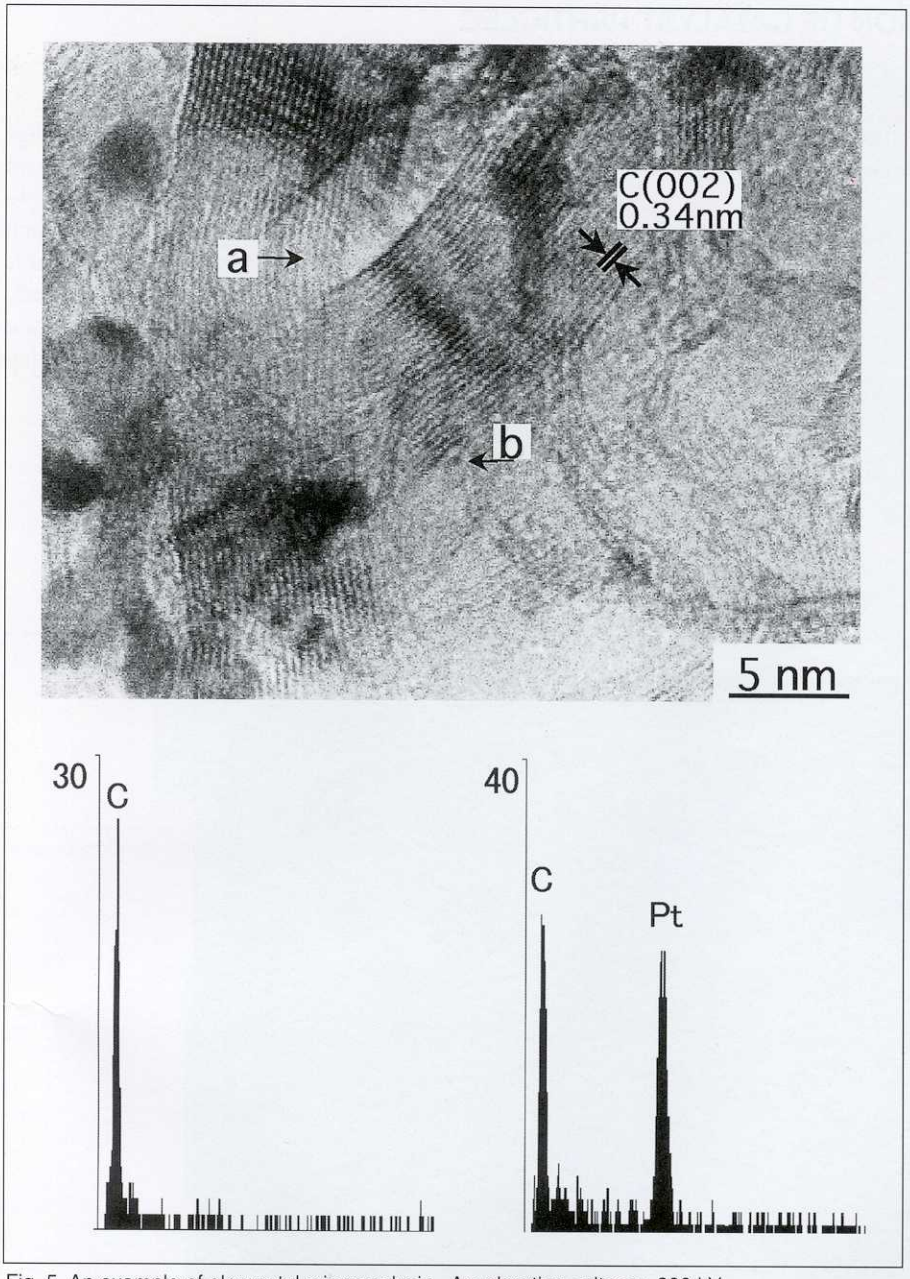

Fig. 5 An example of elemental microanalysis Accelerating voltage: 200 kV
Magnification: x4,000,000
Operation mode: High resolution

7. CLOSING

We have introduced an example of observation and analysis of Pt particles using the HD-2000. The HD-2000 allows secondary electron imaging, bright field STEM imaging, dark field STEM imaging and high resolution imaging, all of which are useful for materials characterization of catalyst particles. The HD-2000 also allows the use of an X-ray spectrometer. The combined system allows a high sensitivity microanalysis beyond the limits of the conventional system. It means that much smaller areas can be analyzed much more quickly. The HD-2000 should be found useful not only for catalysts but also many more advanced materials in the future.

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For further information, please contact your nearest sales representative.

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