

Observation of Pd catalytic reactions using an in-situ gas reaction observation system that connects a transmission electron microscope (TEM) and a mass spectrometer (MS)

Product used: Mass Spectrometer (MS)

Introduction

By using a MEMS-Chip type sample holder, it has become possible to observe samples under conditions similar to those in which they are actually used, such as high pressure or underwater, even with general-purpose TEM. Furthermore, by combining it with gas analysis equipment such as MS, it has become possible to perform TEM observation and generated gas analysis simultaneously. In this MSTips, we will report on the reaction observation of palladium (Pd), an exhaust gas purification catalyst, by connecting JMS-Q1600GC to JEM-ARM200F equipped with an in-situ sample holder manufactured by Protochips.

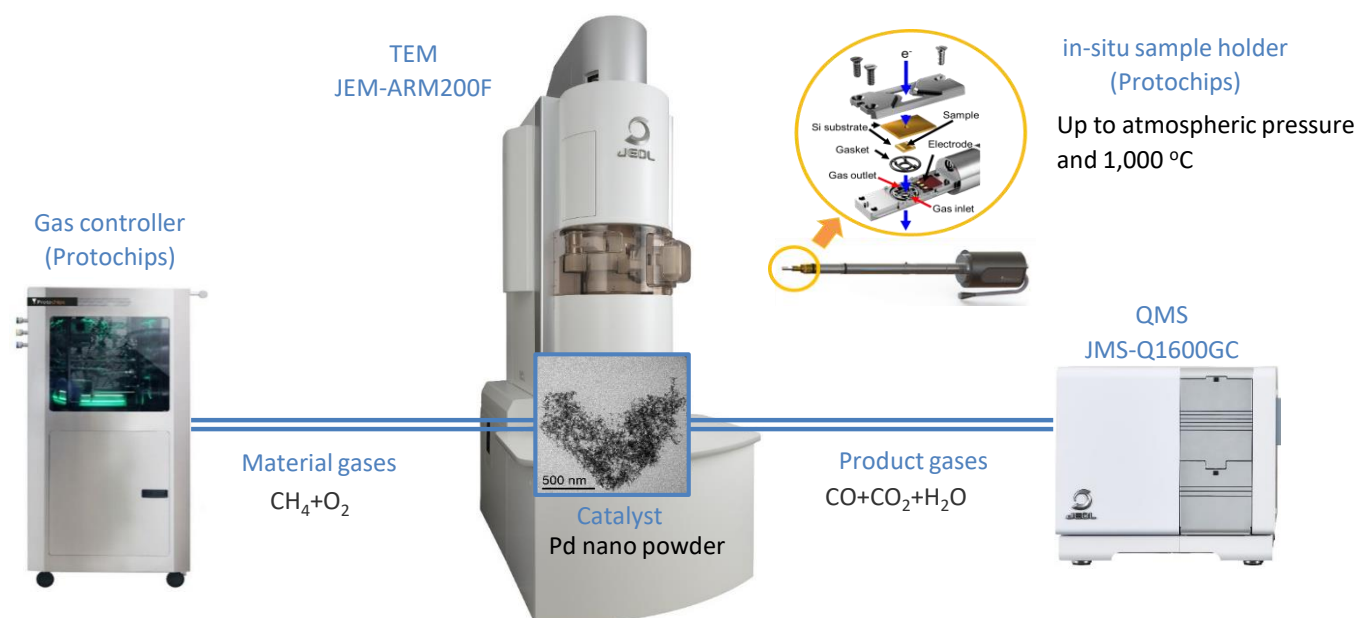


Figure 1 In-situ gas reaction observation system

Experiment

Commercially available Pd nanoparticles were used as the sample. Methane (CH₄) and oxygen (O₂) were supplied as material gases to the in-situ sample holder, and TEM observation and MS gas analysis were performed simultaneously while heating in the range of 25 to 960°C (holding and cooling as necessary). Table 1 shows the measurement conditions for each device.

Table 1 Measurement conditions

Gas controller		TEM : JEM-ARM200F	
Gas mixture ratio	CH ₄ /O ₂ /He(dilution gas)=22/5/73	Accelerating voltage	200kV
Gas supply pressure	10kPa		TEM observation mode
			IDES Electron Dose Modulation
In-situ sample holder		MS : JMS-Q1600GC	
Temperature	25°C - 960°C, 1°C/sec rate	Ionization	EI+(70eV), 50µA
	* Held and cooled as necessary	Ion source temperature	100°C
		Acquisition mode	SIM, <i>m/z</i> 4, 15, 16, 18, 28, 32, 44

Results

Figure 2 shows the heating program for the in-situ sample holder (middle), TEM image (upper), and SIM chromatogram at m/z 44 (CO_2) (lower). At around 560°C , CO_2 generation was observed at the same time as fine vibrations of Pd particles. These behaviors stopped when cooled, and reappeared by heating again. Upon further heating, condensation of Pd particles was observed.

> [Click here for the movie](#)

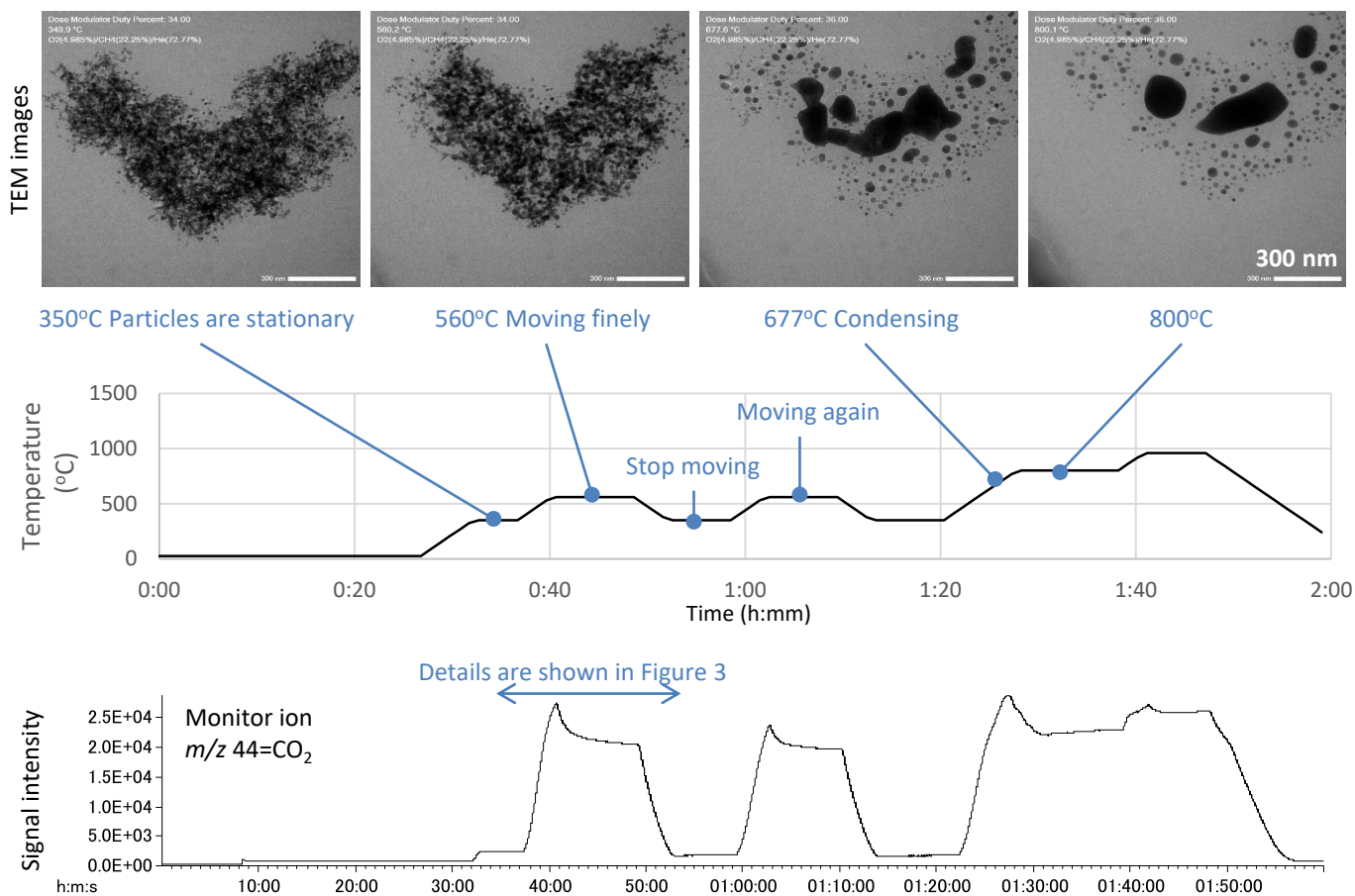


Figure 2 TEM images (upper), Temperature of in-situ sample holder (middle), SIM chromatogram at m/z 44 (lower)

Figure 3 shows the SIM chromatograms at each monitor ion between sample holder temperatures of 350→560→350°C. A decrease in methane (CH₄) and oxygen (O₂), which are material gases, and an increase in water (H₂O), carbon monoxide (CO), and carbon dioxide (CO₂), which are product gases, were observed.

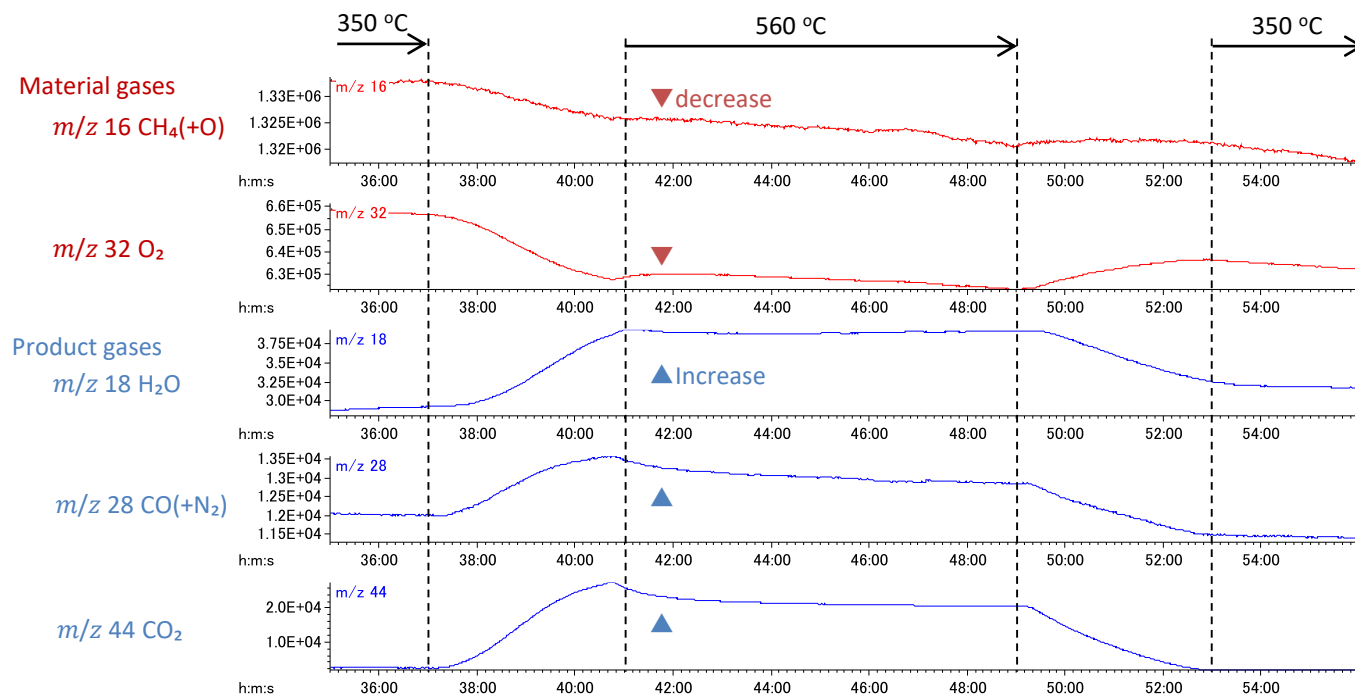


Figure 3 SIM chromatograms at each monitor ions

Conclusion

These results will be important in analyzing the mechanism of Pd-catalyzed reactions and optimizing conditions. The in-situ gas reaction observation system combining JEM-ARM200F and JMS-Q1600GC is expected to be useful in the analysis of highly functional materials such as catalysts.

Copyright © 2023 JEOL Ltd.

Certain products in this brochure are controlled under the "Foreign Exchange and Foreign Trade Law" of Japan in compliance with international security export control. JEOL Ltd. must provide the Japanese Government with "End-user's Statement of Assurance" and "End-use Certificate" in order to obtain the export license needed for export from Japan. If the product to be exported is in this category, the end user will be asked to fill in these certificate forms.

