B-AD-037

Metal dispersion rate analysis of Au-supported catalysts

Introduction

Au-supported catalysts are used in industrially important reactions such as selective oxidation, selective hydrogenation, and reforming reactions such as aqueous gas shift reactions, and are known to be very useful catalysts. The metal dispersion rate of metal supported catalysts are commonly evaluated by CO or H_2 pulse measurement. However, it is impossible to analyze the metal dispersion rate of Au-supported catalysts by using CO/H_2 pulse measurement method, because CO or H_2 are not adsorbed on Au at room temperature.
It has been reported that CO is chemisorbed on Au-supported catalysts at around -100°C[1].
We conducted the CO pulse measurement of an Au-supported catalyst at -100°C, and found that the Au metal dispersion rate can be evaluated successfully.
In this report, the procedure and the precautions for CO pulse measurement at low temperatrure for Au-supported catalysts are described.

Experiment

1. Measurement principle

For the basic measurement principle, please refer to CAT-APP-008 [Metal Dispersion].
Since CO is hardly adsorbed on Au-supported catalysts at room temperature, it is not possible to measure metal dispersion using the conventional CO pulse method. Therefore, the CO pulse method cannot be used to measure metal dispersion.

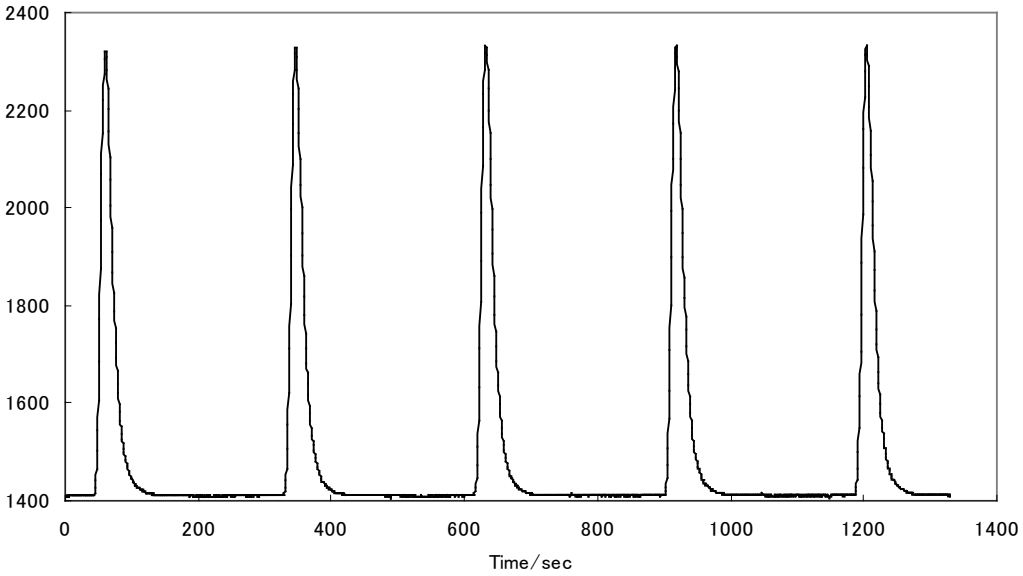
2. Measurement under normal conditions (50°C)

The metal dispersion rate measurement at 50**°C** was carried out under the following condition.

Pretreatment conditions

STEP	GAS	Flow rate(sccm)	Time(min)	Temperature(°C)
1	He	50	15	100
2	He	50	15	100
3	O_2	50	15	100
4	He	50	15	100
5	H_2	50	15	100
6	He	50	15	100
7	He	50	1	50
8	He	50	5	50

Measuring equipment : BELCAT II
Sample : 30.3wt% Au/C 50 mg
Measurement temperature : 50°C
Gas flow rate : 30 cm3/min (0°C, 1atm)
Pulse volume : Approx. 90µl (0°C, 1atm)



CO pulse chart

Adsorption amount (Vm)	:0.0039 cm^3/g
Metal dispersion rate(Dm):	0.0112% (%)
Metal surface area (Am (Sample)):	0.0091 m^2/g
Metal surface area (Am(Metal)):	0.0299 m^2/g
Average particle size (lm):	10397.5401 nm

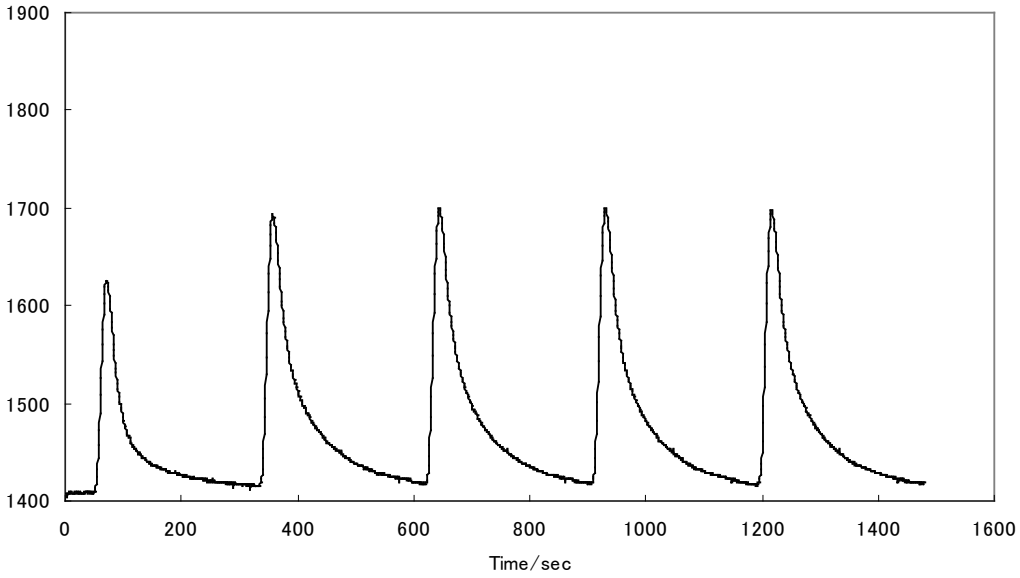
3. Measurement under low temperature conditions (-100°C)

The metal dispersion measurement at -100**°C** was carried out under the following condition.

Pretreatment conditions

STEP	GAS	Flow rate(sccm)	Time(min)	Temperature(°C)
1	He	50	15	100
2	He	50	15	100
3	O_2	50	15	100
4	He	50	15	100
5	H_2	50	15	100
6	He	50	15	100
7	He	50	1	-100
8	He	50	5	-100

Measuring equipment:	BELCAT II + CATCRYO II
Sample:	30.3wt% Au/C 50 mg
Measurement temperature:	-100°C
Gas flow rate:	30 cm3/min (0°C, 1atm)
Pulse volume:	Approx. 90µl (0°C, 1atm)



CO pulse chart

Adsorption amount (Vm):	2.3280 cm^3/g
Metal dispersion rate(Dm):	6.7516% (%)
Metal surface area (Am (Sample)):	5.4414 m^2/g
Metal surface area (Am(Metal)):	17.9586 m^2/g
Average particle size (lm):	17.2931 nm

Summary

CO pulse measurements on Au-supported catalysts at a low temperature of -100°C was conducted to investigate the possibility of evaluating the metal dispersion of Au-supported catalysts.
In literature 1), the measurements were performed at 142-179 K [-131 to -94°C], but it was found that measurements at -100°C were also possible.
In order to perform low temperature pulse measurement with our catalyst analyser [BELCAT II], the low temperature electric furnace [CATCRYO II] must be included.
For the analysis, the following parameters were used.

Au Parameters

Atomic weight:	196.967g/mol
Density:	19.30 g/cm^3
Cross-sectional area:	0.0870nm^2/atom

1)
F.Menegazzo,M.Manzoli,A.Chiorino,F.Boccuzzi,T.Tabakova,M.Signoretto,F.Pinna,N.Pernicone ,J.Catal.237(2006)431.

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