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## Special Feature: Automotive Exhaust Catalyst

*Research Report*

# In Situ Redispersion of Platinum Supported on Ceria-based Oxide for Automotive Catalysts

Yasutaka Nagai

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**ABSTRACT** In situ time-resolved X-ray absorption spectroscopy (XAS) was used to examine the morphological changes of Pt metal particles to elucidate the detailed and fundamental characteristics of three-way catalysts for the treatment of automobile exhaust. XAS analysis revealed unexpected phenomenon in the rapid and efficient oxidative redispersion of large Pt agglomerates on a ceria-based oxide support by splitting into smaller particles during rapid (ca. 60 s) redox cycling. This phenomenon does not occur in equivalent Pt/$Al_2O_3$ systems. The redispersion arises from the trapping of Pt species at sites on the Ce support that exhibit a much stronger Pt-oxide-support interaction than can be achieved on supports such as $Al_2O_3$. This redispersion process leads to a tangible potential for incorporation into an "on board" methodology for extension of car catalyst lifetime by suppressing or reversing the effects of metal sintering during operation.

**KEYWORDS** Automotive catalyst, Platinum, Redispersion, Sintering, Pt-support interaction, X-ray absorption spectroscopy

## 1. Introduction

The present automobile society is growing on a global scale, and vehicles have become a necessity to make our living more convenient and richer. However, vehicles have undesirable effects on the global environment and deplete natural resources. Recently, more stringent emission regulations have been imposed on the automobile industry, due to increasing demands of global environmental protection. Automobile companies are striving to develop effective systems for the purification of automobile exhaust. Consequently, there is a strong demand to produce more advanced automotive catalysts by technical innovation with requirements such as high catalytic activity, minimal use of precious metals, and increased longevity.

Supported precious metal catalysts such as the platinum (Pt) - rhodium (Rh) - palladium (Pd) system have been widely utilized in various industrial catalysts, and especially in automotive three-way catalysts (TWCs) to abate harmful gas emissions from gasoline-powered engines. These precious metals act as active sites to simultaneously purify harmful automotive exhaust such as nitrogen oxides ($NO_x$), carbon monoxide (CO) and unburned hydrocarbons (HC). The precious metal particles in TWCs are a few nanometers in diameter, and are dispersed on a support oxide. When the TWC is exposed to high temperatures (≥800°C), the highly dispersed metal nanoparticles agglomerate and sinter, which decreases the active surface area.(1-4) Sintering of the precious metal particles during operation causes a loss of catalytic activity (i.e. deactivation). Exhaust gases exiting from gasoline engines change quickly and dramatically during operation and the temperatures can rise transiently to around 1000°C, so that the exhaust gas composition itself fluctuates quickly between oxidative and reductive compositions. Hence, in situ dynamic observation of the sintering and redispersion (reversal of the sintering effects and regeneration) phenomena of precious metals in automotive catalysts is very important. Efficient Pt redispersion phenomena under rapid (ca. 60 s) redox cycling has not been reported to date.

In this study, we present the rapid and effective redispersion of Pt particles on ceria-based oxide supports during cyclic oxidative/reductive cycling through the application of in situ and time-resolved X-ray absorption spectroscopy (XAS) and transmission electron microscopy (TEM). In situ and time-resolved measurements are essential for investigation of the transient response of catalysts to changes in their working environment. In the present study, real-time observation of changes in the noble metal structure were made at the Pt $L_{III}$ edge using the fluorescence yield variant (Turbo-XAS (T-XAS)) of energy dispersive extended X-ray absorption fine structure (EXAFS) developed by Pascarelli et al. at ID24 at the European Synchrotron Radiation Facility (ESRF).(6) At present, the combination of low levels of Pt in catalysts with high levels of heavy, absorbing, elements such as Ce and Zr severely compromises the conventional transmission based in dispersive XAS analysis. In order to obtain a window into the structural-reactive behavior of the Pt in these systems with subsecond time resolution, the fluorescence variant must be adopted to realize Pt $L_{III}$ edge X-ray absorption near edge structure (XANES) observation on the required timescale.(5)

## 2. Experimental

### 2. 1 Catalyst preparation and aging treatment

Pt/$Al_2O_3$ and Pt/ceria-based mixed oxide (Ce-Zr-Y mixed oxide, CZY) catalysts were prepared by the following methods. $Al_2O_3$ and CZY powders were used as support oxides. $Al_2O_3$ was supplied by Nikki Universal Co., Ltd. and has a $\gamma$-type crystal structure. CZY mixed oxide powders were prepared using a coprecipitation process with aqueous $NH_3$ and aqueous solutions of $Ce(NO_3)_3$, $ZrO(NO_3)_2$ and $Y(NO_3)_3$. The precipitate was dried at 110°C and calcined in air at 700°C for 3 h. The product contained 50 wt% $CeO_2$, 46 wt% $ZrO_2$ and 4 wt% $Y_2O_3$, and its crystal structure was cubic. 2 wt% Pt/$Al_2O_3$ and 2 wt% Pt/CZY catalysts were prepared by conventional wet impregnation of $Al_2O_3$ and CZY powders with $Pt(NH_3)_2(NO_2)_2$ aqueous solution. The impregnated powders were dried overnight at 110°C and calcined at 500°C for 3 h in air. These samples are referred to as fresh catalysts. Portions of the fresh Pt/CZY powders were aged in 3% $H_2$/He, $N_2$, or air at various temperatures in order to obtain specifically sized particles of sintered Pt.

### 2. 2 Characterization

The average particle size of Pt metal was measured using a CO pulse adsorption method.(7) The catalysts were pre-treated in a pure oxygen flow, and then pure hydrogen flow at 400°C. The reductive treatment with hydrogen reduces the Pt oxides to Pt metal. CO pulse adsorption was carried out under He flow at −78°C, at which temperature, the CO uptake on the ceria support was almost entirely suppressed, and CO was adsorbed only to the surface of Pt.(8) The average particle size was calculated from the CO uptake, assuming that CO was adsorbed on the surface of spherical Pt particles at CO/(surface Pt atom) = 1/1 stoichiometry.

Dynamic observation of the redispersion behavior of sintered Pt was conducted using in situ time-resolved Turbo-XAS (T-XAS) in fluorescence mode at ID-24 of ESRF (Grenoble, France). The experimental set-up for the T-XAS and for quick gas change from oxidative to reductive atmospheres is described elsewhere.(9) Incoming X-rays ($I_0$) and fluorescence X-rays ($I$) are simultaneously recorded by two detectors. The sample is placed in an in situ cell equipped with a rapid gas switching system, allowing alternation between oxidative and reductive gas flow over the sample. The cell was specially designed for fluorescence XAS detection at high sample temperatures (T max ca. 800°C), while minimizing dead volume.(10) Throughout the in situ experiment, the pellet samples were heated under cyclic oxidizing/reducing conditions. Gas mixtures of $O_2$ in He (150 cc/min) and $H_2$ in He (150 cc/min) were introduced alternately into the cell using a gas-actuated switching valve. Pt $L_{III}$ edge XAS spectra were collected every 1.1-6.0 s.

In order to investigate the local structure around Pt atoms under a static state, conventional EXAFS analysis was carried out at BL01B1 and BL16B2 of SPring-8 (Hyogo, Japan). The storage ring energy was operated at 8 GeV with a typical current of 100 mA. The EXAFS spectra at the Pt $L_{III}$ edge were measured using a Si(111) double crystal monochromator in fluorescence mode at room temperature and in air.

## 3. Results and discussion

### 3. 1 Analysis of Pt particle size using T-XAS

Firstly, a method to analyze the Pt sintering /redispersion was studied. **Figure 1**(a) shows Pt $L_{III}$ edge XANES spectra of the Pt/CZY catalyst under oxidizing (20% $O_2$)/reducing (3% $H_2$) atmospheres at 400°C. The $\Delta I$ denotes the difference between the white line peak height of the oxidized and reduced samples. $\Delta I$ did not change under cycling of oxidizing/reducing conditions at 400°C. The correlation between the various Pt particle sizes determined by the CO pulse method and $\Delta I$ is presented in Fig. 1(b). *ΔI* increases with the decrease in the particle size of Pt. A good linear correlation was obtained between the Pt dispersion (Pt particle size) measured by the CO pulse adsorption method and *ΔI* by the white line peak height of Pt $L_{III}$ edge XANES, which suggests that the Pt particles are oxidized only on the surface region, and that the portion of oxidized Pt atoms is proportional to the surface area of Pt particles. A detailed schematic illustration to aid in understanding the method and the linear correlation is presented in **Fig. 2**. In the case of small Pt particles, *ΔI* is large, because a large proportion of the Pt particle surfaces are exposed. However, in the case of large Pt particles, *ΔI* is small compared to that for small Pt particles. Therefore, using the correlation between the Pt particle size and *ΔI*, it is possible to track the change in Pt particle size during in situ redox cycling.

## 3. 2 In situ dynamic observation of Pt sintering/redispersion

**Figure 3** shows the variation of the white line peak height of the normalized Pt $L_{III}$ edge XANES for the fresh $Pt/Al_2O_3$ catalyst under cyclical oxidizing/reducing conditions at 400-800°C. 4 or 20% $O_2$/He gas and 3% $H_2$/He gas were alternately introduced into the cell every 60 s. Under a reductive atmosphere, the supported Pt is in the metallic state and the white line peak is low; however, under an oxidative atmosphere, the surface of Pt particles can be oxidized, and the white line peak is high (inset of Fig. 3). The white line peak height of the reduced sample is constant; therefore, the height of the oxidized catalyst corresponds to *ΔI*. The *ΔI* for $Pt/Al_2O_3$ decreases as the temperature is increased, and then

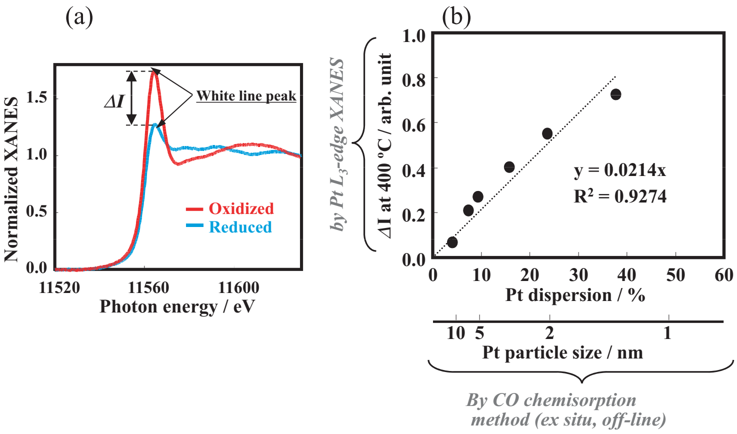

**Fig. 1** (a) Pt $L_{III}$ edge XANES of a Pt/CZY catalyst under oxidizing/reducing atmosphere at 400ºC. *ΔI* is defined as the difference in the heights of the Pt $L_{III}$ absorption edge between oxidized and reduced samples.
(b) Correlation between *ΔI* and Pt dispersion estimated by the CO pulse method for preliminarily aged Pt/CZY catalysts with a specific particle size of sintered Pt.

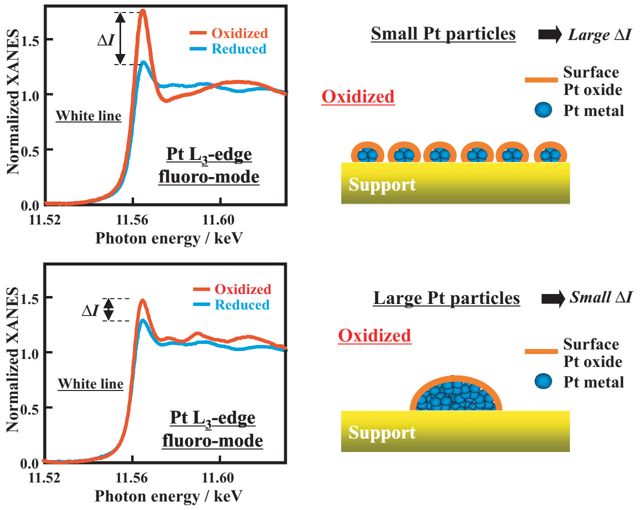

**Fig. 2** Schematic representations to aid understanding of the correlation between Pt particle size and *ΔI*.

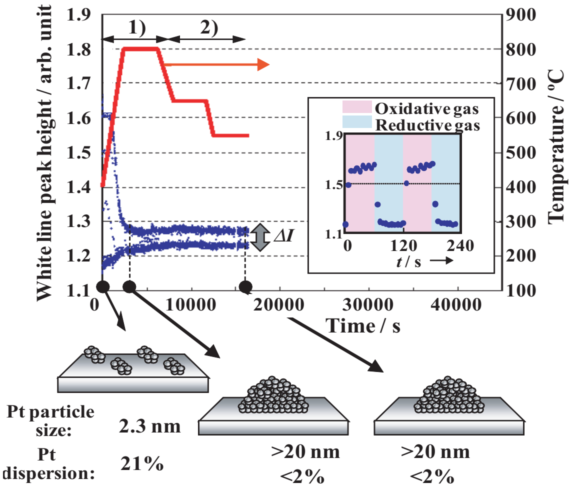

**Fig. 3** Temporal dependence of the white line peak height of the Pt $L_{III}$ edge XANES for fresh $Pt/Al_2O_3$ under oxidizing/reducing atmosphere at 400-800°C, and a schematic representation of the sintering/redispersion behavior. 1) 4% $O_2$/He and 3% $H_2$/He gases, 2) 20% $O_2$/He and 3% $H_2$/He gases were alternately introduced into the cell every 60 s throughout the measurement.

becomes constant as the temperature is decreased. Using the correlation between the Pt particle size and $\Delta I$ in Fig. 1(b), we observed that only facile sintering of Pt particles (>20 nm) occurred in Pt/$Al_2O_3$, and Pt redispersion was not observed at any temperature. In contrast, the Pt/CZY system exhibited significantly different behavior. **Figure 4** shows that $\Delta I$ for Pt/CZY decreases as the temperature is increased. Upon lowering the temperature, $\Delta I$ gradually increases. This indicates that the average Pt particle size becomes correspondingly larger or smaller as a function of the temperatures. The Pt particle size of the fresh catalyst increases from 1.8 to 4.4 nm during the temperature ramp from 400 to 800°C, but then decreases to 2.7 nm as the temperature is lowered to 550°C. It is interesting to note that the sintering/redispersion phenomena can be reversibly controlled using the sample temperature. During redispersion, the Pt particle size recovers from 4.4 to 2.7 nm at the first cycle, and then from 5.8 to 3.4 nm in the second cycle.

### 3. 3 Effect of initial Pt particle size on Pt redispersion

Next, we focused on the Pt redispersion process during redox cycling. The effect of the initial Pt particle size on the speed of Pt redispersion was investigated using pre-aged (sintered) Pt/CZY catalysts. **Figures 5** (a)-(e) show the variation of white line peak height for the preliminary sintered Pt/CZY samples. 20% $O_2$/He and 3% $H_2$/He gases were alternately introduced every 60 s at 600°C. The $\Delta I$ curve was simulated using the following exponential function:

$$\Delta I = \Delta I_f - (\Delta I_f - \Delta I_i)\exp(-t/\tau), \quad \ldots\ldots\ldots\ldots\ (1)$$

where $\Delta I_i$ and $\Delta I_f$ are the initial and final $\Delta I$, respectively, $t$ is time (in seconds), and $\tau$ is the time constant. The simulation results are also shown as orange lines in Fig. 5. Here, $1/\tau$ defines the speed of Pt redispersion; the correlation between $1/\tau$ and the initial Pt particle size (for particle sizes in the range of 5.2-8.2 nm) is plotted in Fig. 5(f). The result indicates that an increase of the Pt particle size decreases the speed of Pt redispersion.

### 3. 4 Effect of temperature and $O_2$ concentration on Pt redispersion

Additionally, the effect of temperature on the speed of Pt redispersion was investigated in the same way as

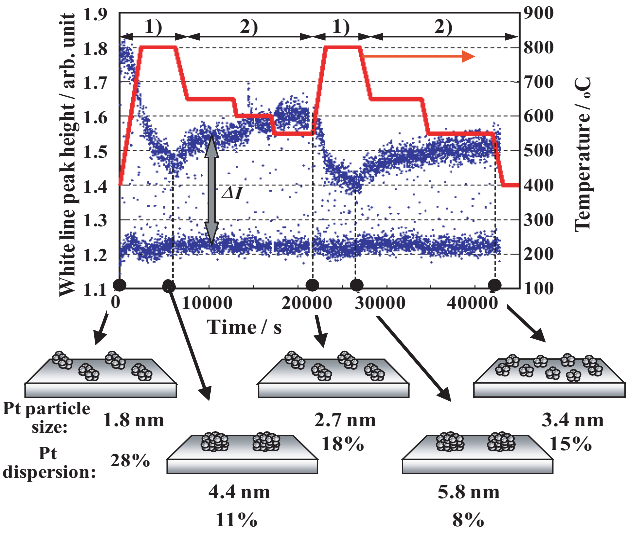

**Fig. 4** Temporal dependence of the white line peak height of the Pt $L_{III}$ edge XANES for fresh Pt/CZY under oxidizing/reducing atmosphere at 400-800°C and a schematic representation of the sintering/redispersion behavior. See Fig. 3 for experimental conditions.

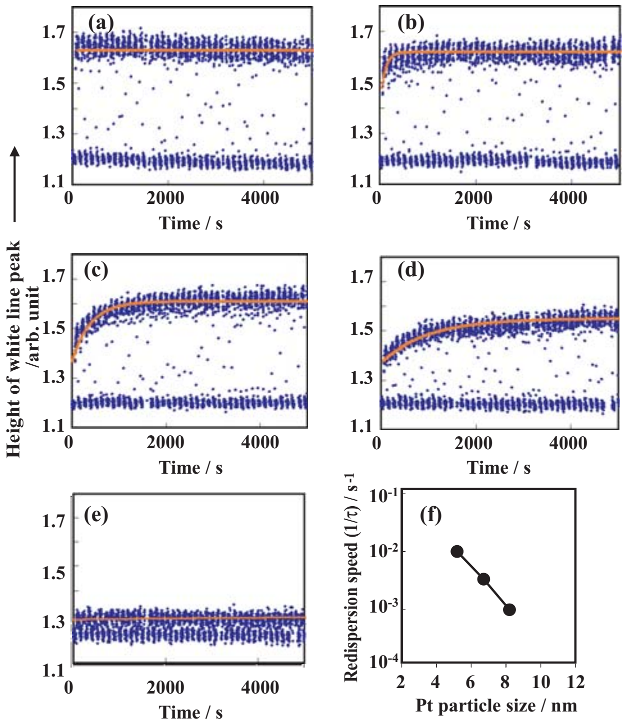

**Fig. 5** Temporal dependence of the white line peak height of Pt $L_{III}$ edge XANES for the pre-aged Pt/CZY with Pt particle sizes (by CO pulse method) of (a) 3.1, (b) 5.2, (c) 6.7, (d) 8.2, and (e) 12.2 nm, and (f) the correlation between the Pt redispersion speed ($1/\tau$) and the initial Pt particle size. 20% $O_2$/He and 3% $H_2$/He gases were alternately introduced into the cell every 60 s at 600°C.

that for Pt particle size, and the results are shown in **Fig. 6** (a). A pre-aged Pt/CZY catalyst (Pt particle size of 6.7 nm from the CO pulse method) was used for this experiment. 20% $O_2$ and 3% $H_2$ gases were alternately introduced every 60 s at 500, 600 and 700°C. The redispersion speed was calculated by the simulation fitting as in the preceding section. Increased temperature yielded an increased speed of redispersion, which demonstrates that Pt redispersion is an activated process. Furthermore, $\Delta I$ did not change under repeated redox cycling at 400°C, which indicates that high temperatures ≥500°C are required for Pt redispersion.

The effect of $O_2$ concentration on the Pt redispersion was also investigated (Fig. 6(b)). In the same way, the preliminarily sintered Pt/CZY catalyst (Pt particle size of 6.7 nm) was used and 3, 10 or 20% $O_2$/He gas and 3% $H_2$/He gas were alternately introduced into the cell every 60 s at 600°C. Fig. 6(b) shows that an increased rate of redispersion was achieved with increasing $O_2$ concentration up to 20%.

### 3. 5 EXAFS analysis of Pt-CeO$_2$ support interaction

The effect of temperature and $O_2$ concentration on Pt redispersion implies that the driving force behind Pt redispersion on CZY is attributed to the Pt-support interaction created under an oxidative atmosphere at high-temperature. Therefore, the detailed structure of the Pt-support interaction was studied using EXAFS analysis. **Figure 7** shows Fourier transforms (FTs) of the Pt/CZY after oxidative and then reductive treatments, along with a schematic representation of the Pt-CZY support interaction. The fresh Pt/CZY sample was heated in air for 5 h at 800°C, and a portion of the oxidized Pt/CZY sample was then reduced using 5% $H_2$ ($N_2$ balance) at 400°C for 30 min. EXAFS measurements were carried out at room temperature in air. The FTs were performed on the Pt $L_{III}$ edge EXAFS spectra in the 3.0-16 Å$^{-1}$ region. In the oxidized Pt/CZY sample of Fig. 7(a), the first peak at 1.8 Å and the second peak at 2.8 Å could be assigned to Pt-O and Pt-O-Ce bonds, respectively.(11) In contrast, the oxidized Pt/$Al_2O_3$ sample (not shown) showed only an intense Pt-Pt peak, and no peaks derived from Pt-$Al_2O_3$ support interaction were evident.(11) These results lead to the conclusion that Pt atoms strongly interact with the CZY support under an oxidative atmosphere at high-temperature and form Pt-O-Ce bonds, as shown in Fig. 7(c). After the reductive treatment, the Pt-O peak of the oxidized Pt/CZY was decreased significantly, and the Pt-O-Ce peak almost disappeared. In addition, intense Pt-Pt could not be observed in the reduced Pt/CZY sample. These results suggest that the Pt-O-Ce bond breaks by reductive treatment and that Pt metal particles are highly dispersed on the CZY support. According to the results of the CO pulse method, the average size of Pt metal particles in the reduced Pt/CZY was estimated to be approximately 1 nm.

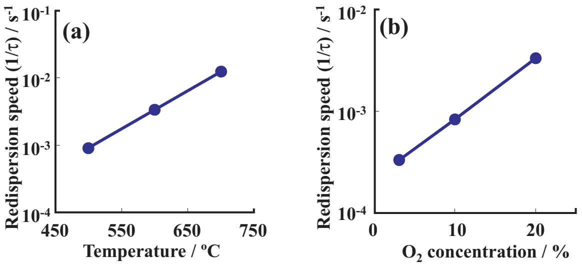

**Fig. 6** Effect of (a) temperature and (b) $O_2$ concentration on the Pt redispersion speed for preliminarily aged Pt/CZY (Pt particle size; 6.7 nm by CO pulse method) samples. (a) 20% $O_2$/He gas and 3% $H_2$/He gas were alternately introduced into the cell every 60 s throughout the measurement.
(b) 3%, 10% or 20% $O_2$/He gas and 3% $H_2$/He gas were alternately introduced into the cell every 60 s at 600°C.

### 3. 6 Pt-CeO$_2$ support interaction and Pt sintering/redispersion

The Pt sintering/redispersion mechanism is discussed according to the obtained results. Pt sintering

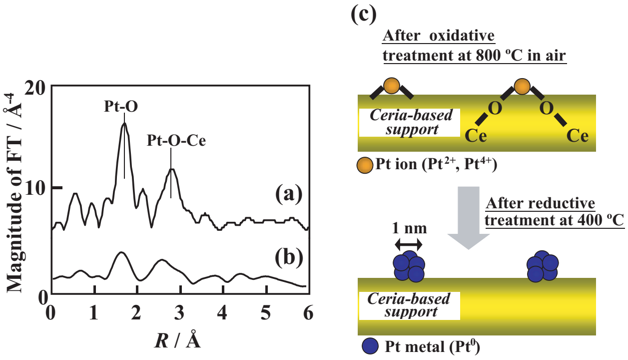

**Fig. 7** Fourier transformed k3x data of Pt $L_{III}$ edge EXAFS for Pt/CZY after 800°C treatment in (a) air and then (b) reduced Pt/CZY, and (c) a schematic representation of the Pt-CZY support interaction.

/redispersion behavior dependent on treatment and interaction with the Pt-O-support can be predicted by considering the following aspects of the thermodynamic equilibrium.

$$\mathrm{PtOx(s)} \underset{\textit{redispersion}}{\overset{\textit{sintering}}{\rightleftarrows}} \mathrm{Pt(s)+O_2} \quad \cdots\cdots\cdots\cdots\cdots\cdots (2)$$

In Eq. (2), (s) indicates Pt species trapped on the support surface. The stability of PtOx(s) is dependent on the Pt-O-support interaction. The Pt(s) species can easily move on the support surface and will be captured by other Pt metal particles. In the case of Pt/$Al_2O_3$, the interaction between Pt and $Al_2O_3$ is weak; therefore, PtOx(s) readily decomposes to form Pt(s) species, and then Pt sintering occurs predominantly. Pt redispersion for sintered Pt particles (>20 nm) on the $Al_2O_3$ support did not occur under the conditions employed in this study. In the Pt/CZY catalyst, oxygen is adsorbed on the surface of sintered Pt particles to form mobile Pt oxide species. The Pt oxide species trapped on the ceria surface have sufficiently high stability, so that redispersion and sintering may occur reversibly. The PtOx(s) species can be stabilized at lower temperatures and higher concentrations of $O_2$, thereby providing conditions for effective redispersion to proceed. In contrast, the sintering of Pt particles is favorable under conditions of low $O_2$ concentration and high temperature. Higher temperature accelerates the Pt redispersion rate within the temperature where the PtOx(s) species is stable. In addition, large Pt particles decrease the Pt redispersion rate, even if the temperature and $O_2$ concentration are in the favorable range for redispersion, because the decreased surface area of large Pt particles significantly reduces the rate of PtOx(s) generation. Based on this mechanism, the sintering and redispersion of Pt in Pt/CZY could be reversible with the change of temperature, $O_2$ concentration and Pt particle size.

Finally, an overall scheme for Pt/CZY redispersion during cyclical redox conditions based on kinetic results from XAS is suggested in **Fig. 8**: Oxygen is adsorbed on the surface of large Pt particles and some mobile Pt oxide species form, migrate, and are trapped on the support surface through a relatively strong interaction with the Pt-O-support. Under reducing conditions, the Pt-O-Ce bonds on CZY break and Pt oxides are reduced to the metallic state, from which new small particles of Pt metal are formed. The formation/cleavage of the Pt-O-Ce bond on CZY was confirmed by EXAFS analysis. Pt redispersion proceeds by the cyclical process of Pt oxide migration from the surface of large metal particles and reduction of the Pt-O-support species.

## 4. Conclusion

In this study, modern XAFS techniques were used for dynamic in situ observation of automotive catalysts to investigate the Pt sintering/redispersion behavior in Pt supported catalysts. The following results were obtained:

a) There is a good correlation between the Pt particle size measured by CO pulse adsorption and *ΔI* measured by the white line peak height of Pt $L_{III}$ edge XANES. This technique allowed tracking of the changes in Pt particle size during in situ redox cycling.

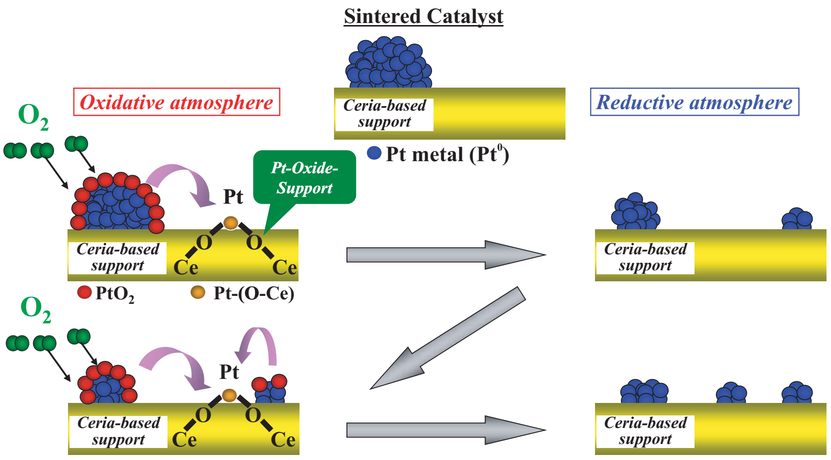

**Fig. 8** Schematic representations of Pt redispersion on a ceria-based support.

b) Only facile sintering of Pt particles (>20 nm) occurred in the Pt/$Al_2O_3$ catalyst during redox cycling at 400-800°C, and Pt redispersion was not observed at any temperature. In contrast, sintering and redispersion in Pt/CZY was reversible with the change of temperature, $O_2$ concentration and Pt particle size.

c) The driving force behind effective Pt redispersion on CZY is attributed to the strong Pt-ceria support interaction created under an oxidative atmosphere at high-temperature.

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Figs. 3-5
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Fig. 7
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**Yasutaka Nagai**

Research Field :
- Automotive catalyst

Academic Degree : Dr. Eng.

Academic Societies :
- Catalysis Society of Japan
- The Japanese XAFS Society

Awards :
- The 3rd Hyogo SPring-8 Award, 2005
- The Chemical Society of Japan Award for
- Technical Achievements, 2008