

# Simultaneous catalytic removal of NO<sub>x</sub> and diesel soot particulate over perovskite-type oxides and supported Ag catalysts

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**Abstract:** A series of perovskite-type oxides and supported Ag catalysts were prepared, and characterized by X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The catalytic activities of the catalysts as well as influencing factors on catalytic activity have been investigated for the simultaneous removal of NO<sub>x</sub> and diesel soot particulate. An increase in catalytic activity for the selective reduction of NO<sub>x</sub> was observed with Ag addition in these perovskite oxides, especially with 5% Ag loading. This catalyst could be a promising candidate of catalytic material for the simultaneous elimination of NO<sub>x</sub> and diesel soot.

**Keywords:** perovskite-type catalysts; supported Ag catalyst; NO<sub>x</sub>; diesel soot

## Introduction

NO<sub>x</sub> and soot particulate emitted from diesel engines cause serious environmental hazards. Oxides of nitrogen contribute not only to the formation of acid rain but also to the formation of photochemical smog under the influence of sunlight. Fine soot particles can penetrate into lungs and cause a serious health hazard because of the presence of mutagenic polynuclear hydrocarbons on their surfaces.

Control of NO<sub>x</sub> emissions from diesel engines remains to be a challenging problem for both academic research and practical application. One possibility is the use of an exhaust gas recirculation system (EGR). But it is well known that EGR systems, while lowering the tailpipe NO<sub>x</sub> emission, could increase the particulate emission (Needham, 1989). The conventional three-way catalyst for gasoline-fueled engines cannot be applied under lean combustion conditions of diesel engines. Selective reduction of NO<sub>x</sub> by hydrocarbons has been investigated by many researchers (Mabilon, 1993; Petunchi, 1993; Konno, 1992; Li, 1992; Armor, 1995; Hamada, 1994; Shimizu, 1998; Burch, 1997; Yentekakis, 1999; Chen, 1999). Because of the low concentrations of unburned hydrocarbons in diesel exhausts, it is necessary to inject additional hydrocarbon. Selective reduction of NO<sub>x</sub> by ammonia (Ito, 1994; Hultermans, 1994; Long, 2000) is not practical because of the high cost of control systems required, and the handling and safety problems of ammonia. Selective reduction of NO<sub>x</sub> with soot particulate has been intensively investigated recently (Teraoka, 1995; Shangguan, 1996). It is attractive, because no external reducing agent is required. However, it is necessary to develop active catalysts for this reaction. Perovskite-type oxide (ABO<sub>3</sub>) catalyst have been studied. It was found that substitution of potassium at "A" sites significantly promoted the redox reaction between soot and NO<sub>x</sub> (Teraoka, 1995). The catalytic performance of ternary AB<sub>2</sub>O<sub>4</sub> spinel-type oxides depends on the metal cations. CuFe<sub>2</sub>O<sub>4</sub> turned out to be a superior catalyst for this reaction (Shangguan, 1996). Doping potassium to CuFe<sub>2</sub>O<sub>4</sub> was found to be effective in promoting the catalyst performance (Shangguan, 1998). The activity of potassium-bimetallic catalysts (such as KFe, KCo, KNi and KCu) for the reduction of NO<sub>x</sub> with carbon has also been studied. KNi catalyst gave the most interesting results (Luan, 1999). Ag/Al<sub>2</sub>O<sub>3</sub> catalyst has been reported to be

active for selective reduction of NO<sub>x</sub> with hydrocarbons (Tatsuo, 1993a; 1993b). But there is no report about perovskite-type oxide supported Ag catalysts for simultaneous catalytic elimination of NO<sub>x</sub> and soot.

In the present work, a series of perovskite-type oxides and supported Ag catalysts were prepared by co-precipitation methods, and their catalytic performance for this reaction was investigated in detail.

## 1 Experimental sections

### 1.1 Catalyst preparation

Perovskite-type  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Co}_{1-x}\text{M}_x\text{O}_3$  ( $\text{M} = \text{Fe}, \text{Ni}, \text{Cu}$ ) catalysts were prepared by co-precipitation. Required nitrates, in calculated amounts, were mixed thoroughly, followed by titration with sodium carbonate (the solution pH was kept in the range of  $10 \pm 0.5$ ). The precipitate was washed with de-ionized water, dried at 120 °C for 4h, and then calcined at 900 °C for 2h. Catalysts of perovskite structure were thus obtained. Silver-supported perovskite-type catalysts were prepared by impregnation in an aqueous  $\text{AgNO}_3$  solution, drying at 120 °C for 2h and calcination at 500 °C for 4h. All the catalyst loadings were expressed in weight percents.

### 1.2 Evaluation of catalyst activity

Catalyst evaluation was performed in a fixed-bed quartz reactor by feeding a gas mixture of 600 ppm NO and 4.5% O<sub>2</sub> in N<sub>2</sub> into the reactor at a flow rate of 3 L/min. The catalyst and soot particles, in a definite weight proportion, were mixed with a spatula, and then the mixture was pelletized under a pressure of  $4 \times 10^8$  Pa, crushed and sieved. The catalyst/soot mixture was then placed in the quartz-tube reactor. The reaction temperature was raised at a rate of 3 °C/min from 200 °C to 540 °C. Gas analysis at the outlet was carried out with a GC of Model Digas 4000.

Diesel soot particulate used in this study was collected from the tail pipe of a Model 195T diesel engine (made in China).

### 1.3 Catalyst characterizations

X-ray diffraction (XRD) measurements were carried out on a Rigaku D/Max-Rc X-ray diffractometer with  $\text{CuK}_\alpha$  radiation. XPS measurements were conducted on a VG Scientific ESCA LAB 220i-XL system with  $\text{AlK}_\alpha$  radiation.

## 2 Results and discussion

### 2.1 Effect of the oxygen concentration on NO<sub>x</sub> conversion

5% Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> catalyst has been used for the study of the effect of oxygen concentration on NO<sub>x</sub> conversion. The results are shown in Fig. 1. The NO<sub>x</sub> conversion rate increased pronouncedly with

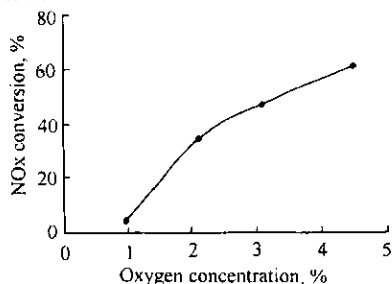


Fig. 1 Effect of the oxygen concentration on NO<sub>x</sub> conversions for 5% Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> catalyst at 450 °C

(Experimental conditions: NO (600 ppm), the weight ratio of catalyst to soot (20:1), S.V. = 71000h<sup>-1</sup>)

increasing oxygen concentration. It is contrary to the inhibition effect of oxygen observed in the NO<sub>x</sub> + C<sub>3</sub>H<sub>6</sub> reaction over Pt/BSA catalyst (Geng, 1992). However, our observation is consistent with Iwamoto's results (Iwamoto, 1994). We speculated that oxygen plays an important role in converting NO to NO<sub>2</sub>. NO<sub>2</sub> is more reactive towards soot particles.

### 2.2 Effect of NO concentration on NO<sub>x</sub> conversion

The NO<sub>x</sub> conversion rate *vs* NO concentration for 5% Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> is shown in Fig. 2. The rate was hardly affected by the variation of NO concentration. On the contrary, in the case of selective reduction of NO by propane on Al-pillared α-zirconium phosphate, NO<sub>x</sub> conversion was retarded, as NO concentration increased (Hernandez-Huesca, 2001). This

contradiction might be explained by the fact that different reducing agents and catalysts were employed. The reaction orders with respect to NO are different too.

### 2.3 Effect of the ratio of catalyst to soot on NO<sub>x</sub> conversion

NO<sub>x</sub> conversion as a function of the reaction temperature under different catalyst/soot ratios is shown in Fig. 3 for the 5% Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>Ni<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> catalyst. At temperatures lower than 420 °C, the conversion order  $R = 10:1 > R = 5:1 > R = 2:1 > R = 20:1$  was observed, where R represents the catalyst/soot ratio. At temperatures above 420 °C,  $C_{R=20:1}$  (NO<sub>x</sub> conversion with  $R = 20:1$ ) apparently increased and reached a maximum at ca. 500 °C. Later on  $C_{R=20:1}$  decreased because the remaining soot was not sufficient to reduce the continuously fed NO<sub>x</sub> stream in the reactor. Above 440 °C,  $C_{R=5:1}$  and  $C_{R=2:1}$  were obviously smaller than  $C_{R=20:1}$  and  $C_{R=10:1}$ . This may be explained by the reduced weight ratio of catalyst to soot. The contact between the catalyst and soot particles became worse too. This led to a decrease in catalytic activity for NO<sub>x</sub> conversion.

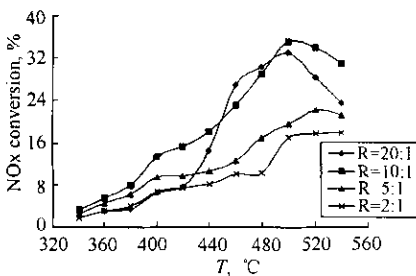


Fig. 3 NO<sub>x</sub> conversion as a function of the reaction temperature under different ratios of catalyst to soot for 5% Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>Ni<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> catalyst at 450 °C

(Experimental conditions: O<sub>2</sub> (4.5%), NO (600 ppm), S. V. = 71000h<sup>-1</sup>)

500 °C. As Duriez (Duriez, 1995) reported, perovskite-type oxide containing copper is active for the reduction of NO<sub>x</sub>, because copper plays a very important role. But in our case the observed activity of La<sub>0.6</sub>Ce<sub>0.4</sub>Cu<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> catalyst is comparatively low.

### 2.5 Catalytic activities of perovskite-type oxide supported Ag catalysts

Fig. 5 indicates the relationship between the activity of Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> catalyst and the Ag loading. Apparently, the activity of La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> catalyst was improved after the addition of 1% Ag. When the Ag loading was increased to 5%, the activity of Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> catalyst reached a

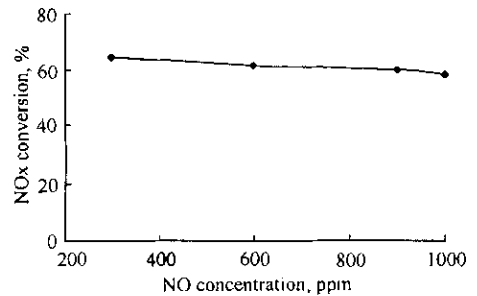


Fig. 2 NO<sub>x</sub> conversion as a function of NO concentration for 5% Ag/La<sub>0.6</sub>Ce<sub>0.4</sub>CoO<sub>3</sub> catalyst at 450 °C

(Experimental conditions: O<sub>2</sub> (4.5%), the weight ratio of catalyst to soot (20:1), S. V. = 71000h<sup>-1</sup>)

Catalyst evaluation was performed under the following experimental conditions: O<sub>2</sub> (4.5%), NO (600 ppm), weight ratio of catalyst to soot  $R = 20:1$ , S. V. = 71000h<sup>-1</sup>.

### 2.4 Catalytic activities of La<sub>0.6</sub>Ce<sub>0.4</sub>Co<sub>1-x</sub>M<sub>x</sub>O<sub>3</sub> (M = Fe, Ni, Cu) catalysts for the selective reduction of NO<sub>x</sub>

Fig. 4 shows the NO<sub>x</sub> conversion rate as a function of reaction temperature for La<sub>0.6</sub>Ce<sub>0.4</sub>Co<sub>1-x</sub>M<sub>x</sub>O<sub>3</sub> (M = Fe, Ni, Cu) catalysts. The activities of La<sub>0.6</sub>Ce<sub>0.4</sub>Ni<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> and La<sub>0.6</sub>Ce<sub>0.4</sub>Cu<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> catalysts cannot be detected at temperatures lower than 400 °C. La<sub>0.6</sub>Ce<sub>0.4</sub>Fe<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> catalyst is moderately active for the reduction of NO<sub>x</sub> in this temperature range. At temperatures above 400 °C, La<sub>0.6</sub>Ce<sub>0.4</sub>Ni<sub>0.5</sub>Co<sub>0.5</sub>O<sub>3</sub> catalyst became active and the NO<sub>x</sub> conversion reached a maximum at

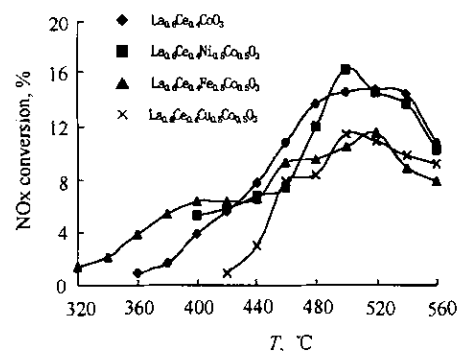


Fig. 4 NO<sub>x</sub> conversion as a function of reaction temperature for La<sub>0.6</sub>Ce<sub>0.4</sub>Co<sub>1-x</sub>M<sub>x</sub>O<sub>3</sub> (M = Fe, Ni, Cu) catalysts

maximum. The activities of various  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  catalysts almost remained constant, when the Ag was increased from 5% to 15%. In the view of cost reduction, the optimal Ag loading of this kind of catalyst was selected at 5%.

The activities of 2%  $\text{Ag}/\text{Al}_2\text{O}_3$  and 6%  $\text{Ag}/\text{Al}_2\text{O}_3$  catalysts for lean NO reduction with  $\text{C}_3\text{H}_6$  were compared. High conversions of NO were obtained over a 2%  $\text{Ag}/\text{Al}_2\text{O}_3$  catalyst. In contrast, NO conversion rates were much lower than those over 6%  $\text{Ag}/\text{Al}_2\text{O}_3$  catalyst (Bethke, 1997).

The results of 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Co}_{1-x}\text{M}_x\text{O}_3$  ( $\text{M} = \text{Fe}, \text{Ni}, \text{Cu}$ ) catalysts are shown in Fig. 6. The activity of 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  catalyst is much higher than the other three catalysts. For the other three catalysts, under temperatures below  $440^\circ\text{C}$ , the following activity order was observed: 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Fe}_{0.5}\text{Co}_{0.5}\text{O}_3 > 5\%$   $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3 > 5\%$   $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Cu}_{0.5}\text{Co}_{0.5}\text{O}_3$ . Above  $440^\circ\text{C}$ , there is little discrepancy among their activities.

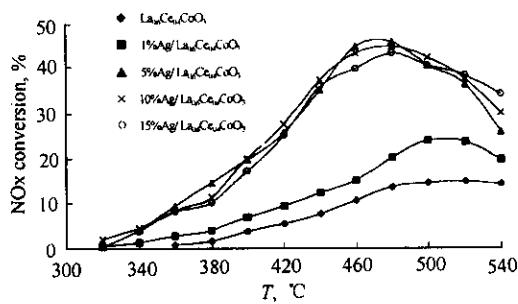


Fig. 5 Activities of various  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  catalysts for the reduction of NOx at different reaction temperatures

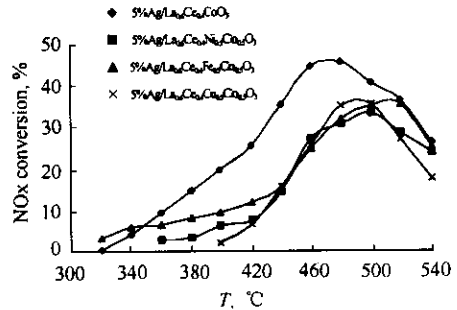


Fig. 6 Activities of 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Co}_{1-x}\text{M}_x\text{O}_3$  ( $\text{M} = \text{Fe}, \text{Ni}, \text{Cu}$ ) catalysts for soot oxidation at different reaction temperatures

## 2.6 Characterization of perovskite-type oxide supported Ag catalysts

Fig. 7 shows the XRD patterns of catalysts  $\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  and 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$ . Their diffraction peaks are similar to that of  $\text{LaCoO}_3$ , which is typical of a perovskite structure. There is no conclusive evidence for the existence of any crystalline phase for  $\text{Ag}^0$  and  $\text{Ag}_2\text{O}$  in the 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  catalyst. This means Ag is highly dispersed on the perovskite-type oxide support.

The surface structures of  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  and 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalysts were studied by the XPS technique, and the results are shown in Table 1.

The  $\text{Ag}(3d_{5/2})$ -XPS spectra of 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalyst are shown in Fig. 8, in which a main peak of 367.4 eV ( $BE$ ) was observed. The  $BE$  value of the main peak centered at 367.4 eV was consistent with that assigned to  $\text{Ag}^+$  ( $3d_{5/2}$ ) in the literature (Watanabe, 1996). There is no evidence for the existence of  $\text{Ag}^0$  ( $BE = 367.9$  eV). We found that most of the Ag surface species in  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Co}_{1-x}\text{M}_x\text{O}_3$  ( $\text{M} = \text{Fe}, \text{Ni}, \text{Cu}$ ) supported Ag catalyst was  $\text{Ag}^+$ . Highly dispersed  $\text{Ag}^+$  favors the adsorption-activation of NO on the catalyst (Meunier, 1999). Perhaps some of  $\text{Ag}^+$  doped onto the surface of  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Co}_{1-x}\text{M}_x\text{O}_3$  partially occupied the sites of  $\text{La}^{3+}$  due to their similar ionic radii, and thus, became stabilized by the perovskite lattice, which would contribute to the prevention of silver aggregation on the surface and enhance the stability of the catalyst. Fig. 9 shows the  $\text{Co}(2p)$ -XPS spectra for

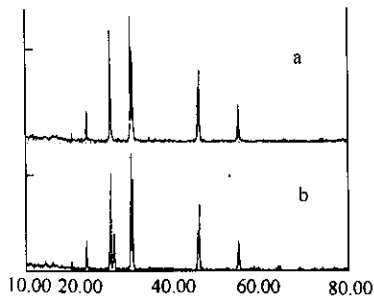
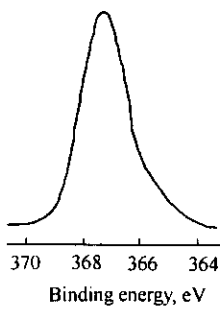


Fig. 7 XRD patterns of catalysts: (a)  $\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  and (b) 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$

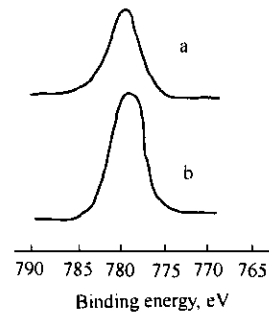
the  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  and 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalysts. In the XPS spectra of  $\text{Co}(2p)$  of  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalyst, the  $\text{Co}(2p)$ -XPS peak was centered at 779.7 eV (*BE*). After loading 5% Ag on  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$ , the  $\text{Co}(2p)$ -XPS peak shifted to 779.3 eV. Because of the low-valence of  $\text{Ag}^+$ , the content of  $\text{Co}^{4+}(2p)$  in the 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalyst was increased in order to keep the neutrality of electric charges. At the same time,  $\text{Ag}^+$  also induces some Schottky defects in the form of anionic vacancies, which would favor the adsorption-activation of oxygen on the functioning catalyst and the transport of the lattice- and surface-oxygen species. In addition, oxygen vacancies are often considered to be the preferential adsorption sites of NO (Teraoka, 1990). All these factors would contribute to the improvement of catalyst activities.

**Table 1** Binding energy (eV) of surface elements and surface chemical composition of  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  and 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalysts

Catalyst	B. E., eV					Content, %				
	La ( $3d_{5/2}$ )	Ce ( $3d_{5/2}$ )	Co ( $2p_{3/2}$ )	O ( $1s$ )	Ag ( $3d_{5/2}$ )	La	Ce	Co	O	Ag
$\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$	833.7	881.6	779.7	528.5	—	12.7	5.9	4.0	77.4	—
5% $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$	833.4	881.6	779.3	528.4	367.4	10.2	4.6	5.4	76.1	3.7



**Fig. 8**  $\text{Ag}(3d_{5/2})$ -XPS spectra of the 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalyst



**Fig. 9**  $\text{Co}(2p)$ -XPS spectra of catalysts: (a)  $\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  and (b) 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$

### 3 Conclusion

The catalytic activities of perovskite-type oxides and supported Ag catalysts for simultaneous removal of NOx and diesel soot particulate have been evaluated. The activities of the catalysts are pronouncedly improved by loading Ag on them. The optimal Ag loading is about 5%. Judging from the XRD characterization of  $\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  and 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{CoO}_3$  catalysts and from the XPS results of 5%  $\text{Ag}/\text{La}_{0.6}\text{Ce}_{0.4}\text{Ni}_{0.5}\text{Co}_{0.5}\text{O}_3$  catalyst, we can conclude that highly dispersed  $\text{Ag}^+$  played an important role in this reaction. On one hand, highly dispersed  $\text{Ag}^+$  favors the adsorption-activation of NO on the catalyst. On the other hand,  $\text{Ag}^+$  also induces some Schottky defects in the form of anionic vacancies, which would be favorable for the adsorption-activation of oxygen on the functioning catalyst and the transport of lattice- and surface-oxygen species. All these factors would contribute to the improvement of the activities of these perovskite-type catalysts.

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