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EFFECT OF COMPOSITION AND PREPARATION METHOD OF (Pd), Co, Ce, Zr - METAL-OXIDE CATALYSTS ON THEIR ACTIVITY IN THE REDUCTION OF NITROGEN(I), (II) OXIDES WITH CARBON MONOXIDE

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Among the studied (Pd), Co-, Ce-, Zr-oxide catalysts including catalysts deposited on structured cordierite support, the catalyst prepared by co-deposition of ceria and cobalt oxide is characterized by higher activity in reaction of reduction of nitrogen(I), (II) oxides (CO + NO, N_2O + NO + CO) compared with samples prepared by successive deposition. This may be caused by an increase in both mobility of surface oxygen and dispersity of components in the catalytic composition.

INTRODUCTION

One of the most important challenges for scientific and applied researches is reduction of nitrogen oxides (NO_x) in gas emissions mobile and stationary sources, as evidenced by the introduction of morestrict norms for emissions into the environment (EURO-V). The emission of nitrogen oxides NO_x (NO, NO_2 and N_2O) into the atmosphere is 30 million tons annually. Nitrogen oxides are involved into atmospheric processes that lead to depletion of ozone layer bringing about “greenhouse” effect and the formation of acid rain, smog [1]. One of the most efficient methods for neutralization of nitrogen oxides NO_x in exhaust gases is catalytic reduction with various reducing agents (CO and C_nH_m).

Platinum group metals (PGM) (Pt, Pd, Rh) in the composition of TWC catalysts (CO/NO/C_nH_m) are active for the reactions of CO + NO (N_2O). One the way for reducing of the PGM content is their partial replacement by other substances such as transition metal oxides with high mobility of surface oxygen [2, 3]. Rare earth oxide (REO) doping may increase the activity of transition metal oxide catalysts. It is known that the catalytic systems containing cobalt oxide (Co_3O_4) with REO (CeO_2, La_2O_3), which demonstrate high activity in oxidation of CO, hydrocarbons and in reduction of NO, may be used for purification of automobile exhaust gases [4–7]. The high catalytic activity of Co_3O_4 and CeO_2 mixtures is attributed to the formation of Co-O-Ce surface clusters during preparation [4, 8].

Now the main attention is focused on structured catalysts in the form of monolithic blocks, in particular, because of the wide selection of options for constructive solutions, low gas-dynamic resistance, easy placement in reactor [9].

This paper presents the results of study of the effect of composition of palladium-cobalt-cerium-oxide catalysts supported on zirconia as well as over structured honeycomb monoliths Pd/(Co_3O_4+CeO_2+(ZrO_2))/cordierite on their activity in the reduction of nitrogen(I),(II) oxides with carbon monoxide. Structure-sized characteristics (phase composition, dispersion) and redox properties of the composites Pd/CeO_2-Co_3O_4/cordierite (with different preparation methods) and their effect on the catalytic properties of metal-oxide catalysts were also studied.

EXPERIMENTAL

Monolithic ceramic blocks of synthetic cordierite ($2Al_2O_3 \cdot 2MgO \cdot 5SiO_2$) with a honeycomb structure and zirconia (specification 6-09-2486-77) were used as a catalyst support. The main characteristics of cordierite monolith were described in [10]. The formation of catalytic coating catalysts was carried by impregnation (in the case of cordierite – on moisture capacity) from aqueous solution of $(NH_4)_2Ce(NO_3)_6$, $Co(NO_3)_2 \cdot 6H_2O$, $Pd(NO_3)_2$ followed by drying in air at 110^0C and calcining at 600 °C. The

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multicomponent catalysts were prepared by the deposition of the active components in the following order: (1) cerium oxide, cobalt oxide (successive deposition, SD), palladium; (2) cerium oxide with cobalt oxide (co-deposition, CD), palladium. The following catalyst samples formed on cordierite and zirconia were prepared:
0.1 % Pd / 5 % Co_3O_4,
0.1 % Pd / 2 % Co_3O_4 + 3.5 % CeO_2 (CD),
0.1 % Pd / 2 % Co_3O_4 / 3.5 % CeO_2 (SD),
0.1 % Pd / 2 % Co_3O_4 + 2.7 % CeO_2 + 0.8 % ZrO_2 (CD).
Bulk samples with the same ratios and the same order of component deposition as for the supported catalysts were prepared to evaluate structural, dimensional characteristics and morphology of the catalyst surface by X-ray diffraction and transmission electron microscopy.

The catalytic activity of the samples was characterized by the conversion of N_2O and NO to nitrogen which was determined in a continuous-flow system with a gradientless quartz reactor under atmospheric pressure in the temperature range 150–400 °C. The following reaction mixtures were used (vol. %): N_2O – 0.2; NO – 0.2; CO – 0.2 and 0.8; the rest – helium. Gas hour space velocity was 6.000 h^{-1}. The sample (fraction 1–3 mm) was roasted prior to testing at 550 °C for 1 h. The structured catalysts were tested in reaction NO + CO in a flow reactor with gas hour space velocity 20.000 h^{-1} on a catalyst block fragment (8 mm in diameter × 13 mm in length with density 49 cells/cm^2, 0.3 g). The components and reaction products were analyzed by gas chromatography (Kristallyuks 4000M, Meta-chrom, Russia) with conductometer detector and CaA column (for N_2, NO, CO, O_2), Polisorb-1 column (for N_2O, CO_2).

The oxidation–reduction properties of the catalysts were studied by temperature programmed reduction with hydrogen (TPR–H_2) in a flow system with continuous chromatographic monitoring of the amount of hydrogen consumed in the reduction. The gas mixture contained was 10 vol. % H_2 in argon. The temperature was raised from 20 to 650 °C with the heating temperature rate 17 °C/min. The mass of catalyst was 0.5 g.

X-ray diffraction (XRD) patterns of the powder samples were recorded using diffractometer with monochromatized Cu-Kα radiation (nickel filter, λ = 0.15184 nm) (AXS GmbH D8 Advance diffractometer, Bruker, Germany).

The morphology of the sample surface was studied using transmission electron microscope (TEM) (Selmi TEM-125K, Selmi, Ukraine). The specific surface (S_{sp}, m^2/g) was determined chromatographically by thermal argon desorption on a GKh-1 instrument.

RESULTS AND DISCUSSION

The activity of the (Pd), Co-, Ce-, Zr-oxides catalysts, also over the monoliths with honeycomb structure, in reactions of nitrogen(I), (II) oxides reduction with carbon monoxide is presented in the Table.

Table. Catalytic activity of (Pd), Co-, Ce-, Zr-oxides catalysts in reactions of nitrogen(I) (II) oxides reduction by carbon monoxide (V = 6.000 h^{-1})

Catalyst (method of preparation)	**N_2O[NO] conversion, % / T °C ($T_{50\ \%}$) for indicated reaction mixtures:**		
	S_{sp}, m^2/g	**0.2 % N_2O + 0.2 % NO + 0.8 % CO**	**0.2 % CO + 0.2 % NO**
5 % Co_3O_4 / ZrO_2	6.7	83/400 (270) [82/250]	–
0.1 % Pd / 5 % Co_3O_4 / ZrO_2	5.7	85/350 (200) [99/200]	–
0.1 % Pd / 2 % Co_3O_4 + 3.5 % CeO_2 / ZrO_2 (CD)	5.8	85/300 (190) [99/160]	–
0.1 % Pd / 5 % Co_3O_4 / cordierite	1.5	78/350 (275) [99/300]	99/250 (230)
0.1 % Pd / 2 % Co_3O_4 + 3.5 % CeO_2 / cordierite (CD)	2.7	78/300 (180) [99/200]	99/155 (123)
0.1 % Pd / 2 % Co_3O_4 / 3.5 % CeO_2 / cordierite (SD)	2.2	–	99/200 (169)
0.1 % Pd / 2 % Co_3O_4 + 2.7 % CeO_2 + 0.8 % ZrO_2 / cordierite (CD)	3.5	75/325 (240) [99/260]	99/190 (130)

The cobalt-containing sample based on ZrO_2 exhibited high activity in the reaction N_2O + NO + CO: in the temperature range 250–270 °C the conversion of NO, CO and N_2O is 82, 90, and 50 %, respectively; the higher conversions of nitrogen(I) oxide (83 %) is achieved at higher temperature – 400 °C. Introduction of palladium (0.1 %) into 5 % Co_3O_4/ZrO_2 catalyst leads to an increase in its activity. High conversions of NO and CO (90 %) are achieved at lower temperatures (at ~160 °C) in the presence of cerium-containing sample $Pd/Co_3O_4 + CeO_2/ZrO_2$; 85 % conversion of nitrogen(I) oxide is achieved at 300 °C. The difference in temperatures of high conversion of nitrogen oxides under conditions of their combined reduction with carbon monoxide may be due to the competition of N_2O and NO molecules for an active site of the catalyst as was observed in the combined reduction of N_2O + NO with C_3–C_4 alkanes [11].

The activity of palladium-doped catalysts formed on the surface of cordierite in reduction of N_2O + NO with CO is changed in following order:

$Co_3O_4 – CeO_2 > Co_3O_4 – CeO_2 – ZrO_2 > Co_3O_4$.

At the most active sample of structured catalyst 0.1 %Pd/2 %Co_3O_4+3.5 %CeO_2/cordierite 60–78 % conversion of nitrous oxide and 99 % conversion of nitrogen monoxide is achieved in the temperature range 200–300 °C.

Decrease in NO and N_2O conversion on the structured catalysts in comparison with granulated, probablly, is related to some decrease in specific and consequently also an active surface of the samples (Table).

Modification of the palladium-cobalt-oxide structured catalyst by cerium oxide enhances its activity in CO + NO reaction. A more significant increase in catalytic activity is achieved by co-deposition of cobalt oxide and cerium oxide, in comparison with successive deposition of the components (Table).

The dependence of the catalytic activity on the chemical composition and the method of preparation (CD or SD), i.e. on the order of deposition of the components may be caused by generation of various phases during formation of the catalytic composition. Bulk samples were studied by XPA to elucidate this question (Fig. 1).

The diffraction patterns of the cobalt–cerium oxide compositions showed only finely dispersed phases of Co_3O_4 (spinel structure ($CoCo_2O_4$) (2θ = 18.95°; 31.35°; 36.90°; 44.90°; 59.40°; 55.90°; 59.40) and CeO_2 (fluorite structure) (2θ = 6.25°; 28.65°; 33.10°; 47.75°; 56.75°; 59.05°) with mean particle diameter 26 nm for Co_3O_4 and 14 nm for CeO_2 (co-deposition of the components), and 55 nm for Co_3O_4 and 14 nm for CeO_2 (successive deposition of the components). The mean particle diameter for pure cobalt oxide ($CoCo_2O_4$) is 44 nm. We should also note that the diffraction patterns of the cobalt–cerium oxide compositions show decreased intensity of the cobalt oxide peaks for compositions prepared by co-deposition of the components in comparison with compositions prepared by successive deposition of the components. Strong extinction characteristic reflexes that respond Co_3O_4 in the composition Co_3O_4-CeO_2 (patterns 2 and 3) can also indicate the formation of solid solutions. The formation of a bulk crystalline structure derived from oxides CeO_2 and Co_3O_4 proved impossible since the coordination numbers of the cerium and cobalt ions are different [12]. Such systems derived from Ce and Fe oxides were studied by Liu et al. [13] who found that the formation of solid solutions of $Ce_xFe_{1-x}O_2$ occured only for $x \le 0.2$ while a separate Fe_2O_3 phase was noted when $x > 0.2$. Hence, we may propose that the increase in activity of the Pd/Co_3O_4/cordierite catalyst after cerium oxide doping occurs as a result of an interaction of the finely dispersed particles of the active components on the phase boundary with formation of solid solutions which facilitates diffusion of oxygen from CeO_2 to Co_3O_4 [4, 6, 7].

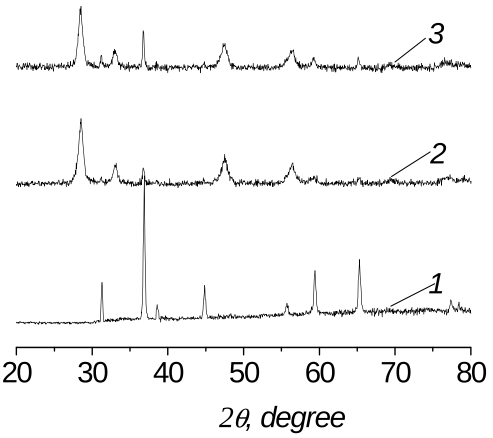


Fig. 1. XRD patterns of bulk samples of catalysts: *1* – Co_3O_4; *2* – Co_3O_4+CeO_2(CD); *3* – Co_3O_4/CeO_2(SD)

It is known that doping of cobalt oxide spinel structure with a small amount of cerium oxide (Ce/Co = 0.05) increases the surface Co_3O_4 and assists the reduction Co^{3+} to Co^{2+}, thus facilitating the desorption of surface oxygen – the limiting stage of nitrous oxide decomposition on the catalysts of this type [14]. Cobalt is stabilized on the separation boundary between Co_3O_4 and CeO_2 in an oxidation state higher than stoichiometric, there is an increase in the Co–O bond length at the sites of contact of the cobalt and cerium nanoparticles and the surface oxygen on the interface between of the oxide phases becomes more mobile than the bulk oxygen of the individual oxides [4].

According to IR spectroscopy data (Fig. 2), absorption intensity at 572 cm^{-1} (bond Co^{3+}–O [7]) for Co_3O_4+CeO_2 (CD) sample is higher than that for individual cobalt oxide. At the same time, one can see that absorption intensity at 663 cm^{-1} (bond Co^{2+}–O [7]) is decreased partially. This was not observed in the case of catalyst prepared by successive deposition of the components. So, preparation of the catalysts by co-deposition of cobalt and cerium oxides assist more intensive interaction and stabilization of Co^{3+} state which is more reactive in redox processes with oxygen.

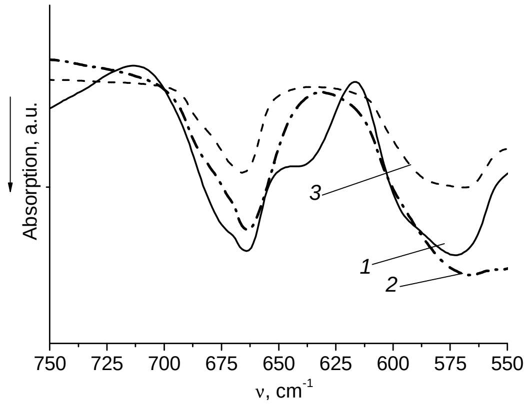


Fig. 2. IR-spectra of bulk samples of catalysts: *1* – Co_3O_4; *2* – Co_3O_4+CeO_2 (CD); *3* – Co_3O_4/CeO_2 (SD)

Thus, the interaction between CeO_2 and Co_3O_4 is more effective in the case of simultaneous introduction of Co_3O_4 and CeO_2 – active components of the catalyst.

Analysis of the TEM photographs of the bulk samples shown in Fig. 3 indicates that a more dispersed phase is formed upon the joint introduction of the components into the catalytic composition (precursors of cobalt and cerium oxides) than in the case of pure Co_3O_4 oxide (Fig. 3 *a*, *b*) while agglomeration of the Co_3O_4 and CeO_2 particles occurs upon the consecutive introduction of the components with subsequent sintering at 600 °C already during the catalyst preparation step. The stabilizing role of cerium oxide is due to localization of this oxide between the cobalt oxide microcrystals preventing agglomeration of the binary oxide composition [15]. Further reducing of the crystallites size of the catalyst components is observed at the partial replacement of cerium oxide by zirconium oxide (Fig. 3 *c*). Probably, zirconia prevents agglomeration of cerium oxide in the procedure of preparation and assists to increase the dispersion of the composition [16].

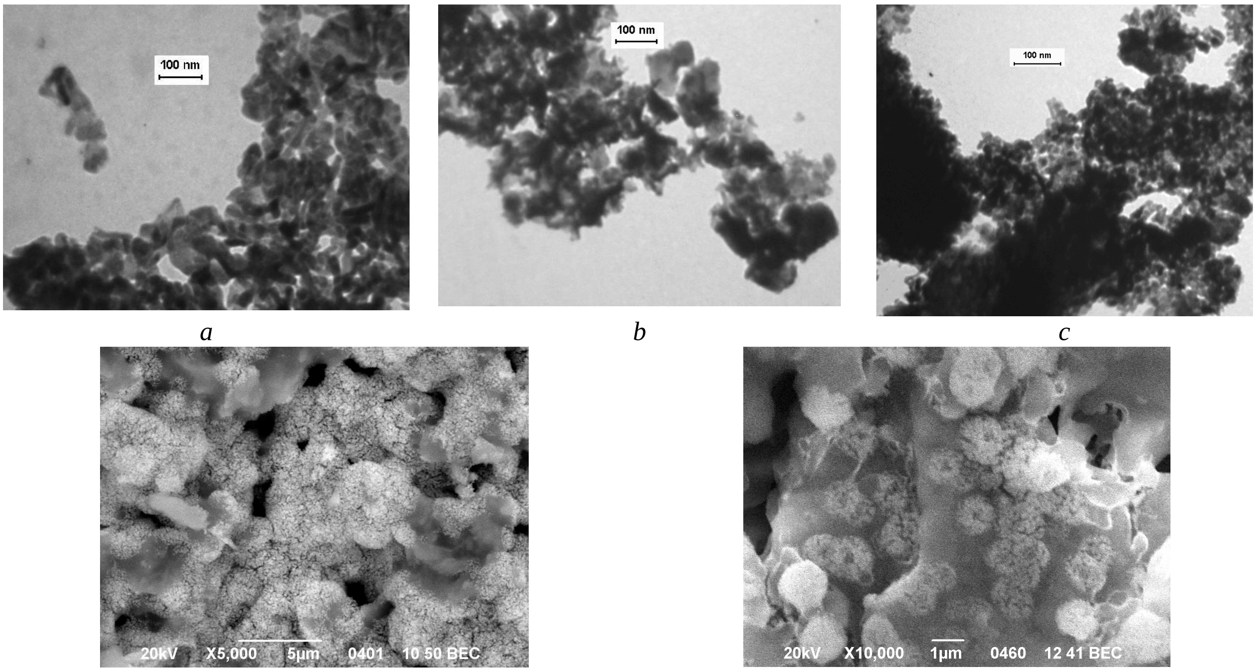
The results of a SEM study indicated a rather homogeneous distribution of the Pd/Co_3O_4 and Pd/(Co_3O_4+CeO_2) compositions on the cordierite surface. Formation of ring-like structures of the active components is observed for 0.1 %Pd/(Co_3O_4+CeO_2)/cordierite (Fig. 3 *e*). This may be taken as an argument to support the hypothesis that the components form a homogeneous structure containing a mixture of pure phases. These results are in accord with the XPA results and literature data [6, 7].

The values of the specific surface of the catalyst samples deposited on a structurized cordierite support and bulk samples indicate high dispersion of the active phase in the case of co-deposition of Co_3O_4 and CeO_2 (ZrO_2) components (Table).

The mechanism for the reduction of NO with carbon monoxide over platinum group metals, in particular over palladium, involves a step featuring the dissociative adsorption of nitric oxide (II) [17]. Bol'shakov et al. [2] have shown that palladium in the Pd-Co/γ-Al_2O_3 catalyst provides for spillover of the oxygen formed onto cobalt oxide where it reacts with carbon monoxide. As a consequence, oxygen passivation of the Pd-Co/γ-Al_2O_3 catalyst is overcome and high activity in the reduction of nitrogen oxides is achieved. Whereas modification of the palladium–cobalt oxide catalyst with cerium oxide increases activity in the reaction NO + CO, we may also assume that the addition of CeO_2 to the Pd–Co_3O_4 catalyst facilitates spillover of oxygen onto the cobalt oxide surface due to an increase in the number of oxygen vacancies on the separation boundary of the Co_3O_4 and CeO_2 phases and a reduction in the bonding energy of the bond of oxygen to the surface.

Platinum group metals are characterized by low metal-oxygen binding energy [3], so, oxygen is rapidly desorbed from the surface of such metals as a result of the reaction that occurs at lower temperatures.

TPR-H_2 experiments were carried out in order to study the redox properties of the catalysts; the results are displayed in Fig. 4.



a *b* *c*

d *e*

Fig. 3. TEM (*a–c*) and SEM microphotographs (*d*, *e*) of catalyst samples: (*a*) Co_3O_4; (*b*) Co_3O_4+CeO_2; (*c*) Co_3O_4+CeO_2+ZrO_2; (*d*) 0.1 %Pd/Co_3O_4/cordierite; (*e*) 0.1 %Pd/Co_3O_4+CeO_2/cordierite

The introduction of palladium leads to a shift in the reduction onset temperature and maximum on the TPR-H_2 curves toward lower temperatures. The TPR-H_2 spectrum shows a new low-temperature peak (180 °C) due to an increase in the mobility of surface oxygen while the concurrent increase in the area under the TPR-H_2 curves indicates an increase in the number of active sites. A low-temperature peak in similar systems has been observed by Luo et al. [4] for the TPR of catalysts by carbon monoxide. The onset temperature and temperature for maximum reduction rate for the Co_3O_4+CeO_2/cordierite catalyst are 20 and 80 °C, respectively, lower than those for the Co_3O_4/cordierite sample. The values for T_{1on} and T_{1max} for Pd/Co_3O_4+CeO_2/cordierite are 55 and 60 °C, respectively, lower than those for the sample lacking the CeO_2 modifying additive. This result indicates a higher mobility of oxygen at the interface between of the cerium oxide and cobalt oxide phases and accounts for the increase in the activity of the 0.1 %Pd/(Co_3O_4+CeO_2)/cordierite catalyst in the oxidation–reduction reactions involving nitrogen oxides(I), (II) studied.

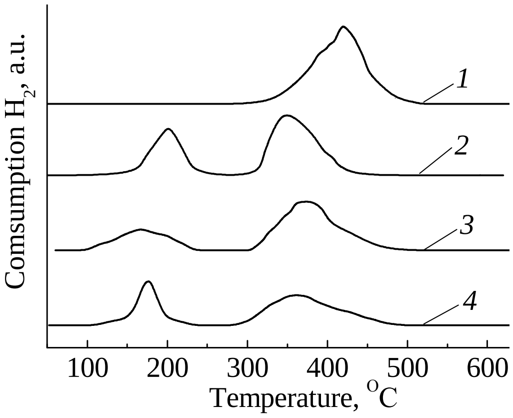


Fig. 4. TPR-H_2 profiles of the cordierite-supported catalysts: *1* – Co_3O_4; *2* – 0.1 % Pd/5 %Co_3O_4; *3* – Co_3O_4+CeO_2 (CD); *4* – Co_3O_4/CeO_2 (SD)

CONCLUSIONS

Modification of the palladium–cobalt oxide catalysts with cerium oxide enhances its activity in reaction of nitrogen(I), (II) oxides reduction with carbon monoxide and caused by increasing the mobility of surface oxygen of catalyst. The most significant increase in activity for the catalyst prepared by co-deposition of ceria and cobalt oxide takes place due to the more disperse components in the catalytic composition according to the results of XRD, TEM and SEM.

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Вплив складу і методу приготування (Pd), Co, Ce, Zr-метал-оксидних каталізаторів на їх активність в реакціях відновлення оксидів азоту(I), (II) монооксидом вуглецю

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В реакціях відновлення оксидів азоту(I),(II) CO (CO+NO, N_2O+NO+CO) серед вивчених (Pd), Co-,Ce- Zr-оксидних каталізаторів, зокрема на структурованих носіях з кордієриту, більшу активність виявив каталізатор, отриманий шляхом одночасного нанесення оксидів церію і кобальту, що обумовлено зростанням рухливості поверхневого кисню і дисперсністю компонентів каталітичної композиції.

Влияние состава и метода приготовления (Pd), Co, Ce, Zr-металл-оксидных катализаторов на их активность в реакциях восстановления оксидов азота (I), (II) монооксидом углерода

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В реакциях восстановления оксидов азота(I),(II) CO (CO+NO, N_2O+NO+CO) среди изученных (Pd), Co-,Ce-,Zr-оксидных катализаторов, в частности на структурированных носителях из кордиерита, большую активность проявил катализатор, приготовленный путем одновременного нанесения оксидов церия и кобальта, что обусловлено увеличением подвижности поверхностного кислорода и дисперсности компонентов каталитической композиции.