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$Sr_{1-x}K_xTiO_3$ catalysts for diesel soot combustion

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ABSTRACT

Strontium titanates partially substituted with potassium ($Sr_{1-x}K_xTiO_3$, $x = 0.1–0.5$) were prepared by sol–gel citric method. The obtained materials were characterized by XRD, S_{BET} and cyclohexanol (CHOL) decomposition measurements, their catalytic activity in soot combustion was determined. It has been shown that substitution of strontium by potassium lowers temperature of soot ignition temperature by 270 °C in a 10% O_2 in Ar atmosphere and ca 400 °C in a 1500 ppm of NO_2, 10% O_2 in Ar atmosphere for $Sr_{0.2}K_{0.2}TiO_3$. The potassium promoting effect is discussed in terms of surface basicity and creation of oxygen vacancies where molecular oxygen or (NO, NO_2) is adsorbed forming basic surface oxygen species active for soot oxidation.

1. Introduction

Diesel engines emit large quantities of particulate matter (PM) and nitrogen oxides, both precursors of photochemical smog; PM leads to respiratory problems and increase risk of cancer [1]. The most promising technique of soot removal is after-treatment consisting in particulate capture and subsequent catalytic oxidation [2]. In the absence of catalyst, PM spontaneously burns in air at about 600–650 °C. This temperature range is not regularly achieved in the typical diesel operations. If an excess of soot is collected on the filter, the exhaust gas temperature increases due to the increased back-pressure, and leads to a sudden burn off, which might occasionally cause the filter temperature to raise above the melting point of the filter itself. Controlled regeneration of the filter can be performed either by heating of the exhaust gas and/or the filter to the PM ignition temperature, or by lowering of this temperature by use of oxidation catalyst [3]. Several catalytic systems are mentioned in the references for PM combustion – platinum based ones exhibit a high activity, however they easily deactivate by sintering as well as are sensitive to sulfur poisoning and expensive [4]. Among other systems lowering the ignition temperature of diesel soot, oxides and chlorides of transition metals, alkali-based materials or combination of mentioned compounds have also been proposed [5–7]. Attempts have been made to elaborate catalyst for PM combustion on the basis of either simple metal oxides or mixed oxides [8–10].

Titanates of Sr (Ba, Mg) represent a group of perovskites, which have excellent dielectric, ferroelectric properties and are commonly used for electroceramics and electronic or optoelectronics devices [11]. Transition metals based mixed oxides with perovskite-like structure exhibit catalytic properties for combustion processes [12]. Although catalytic properties of titanates with perovskite-like structure are less known, it can be expected that these materials posses high thermal stability. Therefore it is interesting to determine their catalytic properties for diesel soot combustion. $SrTiO_3$ was proposed for methane flameless combustion [13,14]. $SrTi_{1-x}Mg_xO_3$ and $CaTiO_3$ were studied for oxidative coupling of methane [15,16]. Calcium titanates were investigated as catalysts for partial oxidation of light hydrocarbons [17]. It is commonly accepted that alkali-based materials improves catalyst-soot contact by increasing surface mobility and the ability to form carbonate species to release carbon dioxide. High activity of potassium containing compounds in some wet-deposition routes and associated with dissolution of potassium salt in the solvent, leading to tight contact between the soot and catalyst surface, has been reported [18]. Jimenez et al. discussed positive effects of potassium addition in terms of K electron donor properties, which can increase oxygen reactivity of the M=O bond [19]. In a previous paper [20], we reported that $SrTiO_3$-based perovskites prepared by ceramic method and either partially substituted by K (Cs, Li) or impregnated with the same amount of alkali metals, show high activity for

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Table 1
Results of SSA determination and CHOL decomposition measurements.

Sample	S_{BET}, m^2/g	CHOL decomposition			
		CHOL conversion, %	Selectivity to CHEN, %	Selectivity to CHON, %	CHON/CHEN
$SrTiO_3$	8.9	20	37	61	1.6
$Sr_{0.9}K_{0.1}TiO_3$	3.5	24	31	69	2.2
$Sr_{0.8}K_{0.2}TiO_3$	1.8	28	26	74	2.9
$Sr_{0.6}K_{0.4}TiO_3$	2.0	25	27	73	2.7
$Sr_{0.5}K_{0.5}TiO_3$	3.9	25	27	73	2.7

soot combustion. Lately we have found, that incorporation of K into the perovskite-like structure by sol–gel method increases stability of K-substituted perovskite catalyst [21]. Additionally, a copper-substituted perovskite catalyst – prepared also by sol–gel method – [22] showed an interesting performance for soot combustion in the presence of NOx.

The aim of this work was to determine effect of the partial substitution of Sr in $SrTiO_3$ by potassium on the catalytic activity of this material in soot combustion in the presence of NO (NO_2) and O_2. Titanates with different substitution degrees ($Sr_{1-x}K_xTiO_3$, $x = 0.1$–0.5) were prepared according to sol–gel procedure.

2. Experimental

$Sr_{1-x}K_xTiO_3$ ($x = 0.1$–0.5) mixed oxides with perovskite-like structure were prepared by sol–gel method using H_2O_2, citric acid, titanium(IV) isopropylate and aqueous solutions of Sr and K nitrate. The solvent was evaporated and remaining viscous syrup was then dried and solid product of decomposition was calcined in air at 850 °C for 6 h.

The specific surface area (SSA) of prepared materials was determined by the BET method from N_2 adsorption data obtained at 77 K by a standard volumetric method using an Autosorb-1 Quantochrome Instruments apparatus.

X-ray powder diffraction patterns were recorded on a DRON-3 operating at 40 kV and 30 mA, using CuK_α radiation, range 20–80°, step 0.5°/min.

Acid–base properties of prepared materials were determined by decomposition of cyclohexanol (CHOL). Acidic sites of the surface are believed to be involved in dehydration of CHOL to cyclohexene (CHEN), while dehydrogenation to cyclohexanone (CHON) proceeds over both acidic and basic sites [23]. Experiments were carried out in a continuous-flow fixed bed reactor. The catalyst (0.5 g) was held on quartz wool, total gas flow 20 dm^3/h (dry N_2 99.999%) saturated with CHOL (0.17 vol.%); temperature 300 °C. Products of CHOL decomposition were analyzed by GC-FID.

Catalytic activity in soot combustion was tested in quartz reactor placed in the furnace. Before the test soot (Printex U from Degussa) and catalyst were mixed in proportion 1:5 in loose contact. The mixture was heated up to 550 °C in flow of 20 dm^3/h either of 10% O_2 in Ar or (1500 ppm of NO + 10% O_2 in Ar) or (1500 ppm of NO_2 + 10% O_2 in Ar). Composition of gaseous products was determined using Multor 610 (CO) and Ultramat 6 (CO_2). Temperature of soot ignition was fixed as the point of CO concentration in the exhaust gas higher than 0.05 vol.%. The test repeatability was determined by carrying out three experiments of soot combustion on $SrTiO_3$ – differences between ignition temperatures were no bigger than 5 K.

The isothermal rate of soot oxidation to CO ($IRSO_{CO}$) and CO_2 ($IRSO_{CO_2}$) was determined at temperature of 30 (40 and 50 °C) below the point of soot ignition. For that, the catalyst/soot mixture was heated in N_2 flow up to temperature of soot ignition, then temperature was lowered and 10 vol.% of O_2 into nitrogen or (1500 ppm NO_2 + 10% O_2) was added. Concentration of CO_2 (CO) in effluent was measured after 30 min of test lasting.

3. Results and discussion

XRD patterns of studied materials (not shown here) reveal intense peaks corresponding to the crystalline perovskite phases and also some weak signals of TiO_2 and $SrCO_3$ and Sr_2TiO_4 are detected.

The SSA data (shown in Table 1) indicate that $SrTiO_3$ possess the highest SSA (8.9 m^2/g) and that the partial substitution of Sr by K lowers the SSA values. Results of CHOL decomposition are presented also in Table 1. Selectivity to CHEN is a measure of acidic strength of active sites of studied materials, while the basic properties may be expressed as a ratio of (selectivity to CHON)/(selectivity to CHEN). Substitution of Sr by K up to $x = 0.2$ slightly increases conversion of CHOL and lowers selectivity to CHEN. All K-substituted titanates exhibit higher value of CHON/CHEN ratio than bare $SrTiO_3$.

Oxygen mobility can be related with metal–oxygen bond at the perovskite surface and thus to the number and to the strength of the basic sites. In this sense, Martin and Duprez [23] found a tentative correlation between the oxygen mobility and the surface basicity: the rate of oxygen surface diffusion increases when the oxide surface basicity increases, but when the strength of the metal–oxygen bond decreases The increase of the basic character of K-substituted strontium titanates, observed in Table 1, can be interpreted considering that in these structures the oxide anions have only two coordinations with strontium cations, while they are polarized by the small cations Ti^{4+}. When Sr^{2+} ($r = 0.144$ nm) is substituted by bigger K^+ ($r = 0.164$) electron density on surface oxygen ions increases.

Maximum of CHON/CHEN ratio is observed for material described by $x = 0.2$. Materials containing higher amount of K ($x > 0.2$) show only somewhat lower CHOL conversion and CHON/CHEN ratio. The partial substitution of Sr by K increases electron density on the $SrTiO_3$ because oxygen vacancies are formed to keep the lattice charge balance. Thus, it can be concluded that the partial substitution of Sr^{2+} cation by potassium leads to the presence of more electrophilic oxygen species on the surface and, consequently, the surface exhibits more basic character.

Fig. 1 summarizes results of soot combustion. In the 10% O_2 in N_2 atmosphere, soot mixed with inert material (SiC) ignites at ca 635 °C. For bare $SrTiO_3$ soot ignition temperature is lowered to 495 °C and, for $Sr_{0.8}K_{0.2}TiO_3$, it decreases to 380 °C after incorporation of K into strontium titanate structure. Incorporation of more $x = 0.2$ of K only slightly affects on the soot ignition temperature.

In the presence of 1500 ppm NO in the gas phase, a decrease in the soot ignition temperature to 570 °C (in the case of inert material – SiC) is observed. When using $SrTiO_3$, the soot starts combust at 440 °C (to see Fig. 1) and, for the K-substituted catalysts, the temperature of soot ignition continuously lowers with increasing the amount of incorporated K up to 315 °C in case of $Sr_{0.5}K_{0.5}TiO_3$. Again, the most pronounced effect of K on soot ignition temperature is observed for $x = 0.2$. The introduction of more than 20 mol% of

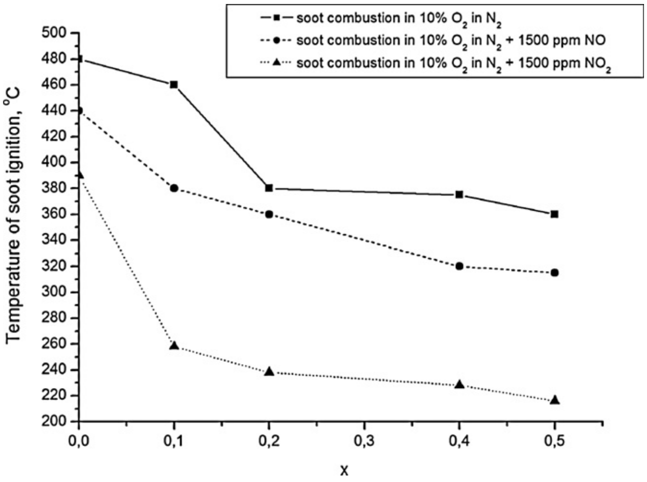

Fig. 1. Results of catalytic activity tests: soot ignition temperature vs. Sr substitution degree.

potassium increases concentration of oxygen vacancies, but excess of anionic vacant position reduces oxygen ions concentration [24]. This is probably due to interaction between the oxygen vacancies and lattice stress, Merino et al. [25] showed that in the case of partial substitution of lanthanum by calcium in $LaCoO_3$, the highest concentration of $O_2{}^{2-}$ and O^- ions occur when a 20 mol%. ($La_{0.8}Ca_{0.2}CoO_3$) calcium is introduced and, after exceeding this value, the concentration of oxygen ions on the surface of perovskite decreases.

In the presence of NO temperature of soot ignition is ca. 30–50 °C lower than in the absence of this gas. This effect on catalyst activity can be explained by reduction of NO on the oxygen vacancy which are formed due to partial Sr^{2+} substitution by K^+ in $SrTiO_3$ structure [26]:

$$2e + \text{oxygenvacancy} + NO \rightarrow O^{2-} + N_2 \tag{1}$$

and generated oxygen ions can oxidize the soot surface.

The presence of 1500 ppm of NO_2 in gas phase (10% O_2 in N_2) greatly affects the temperature of soot ignition: for inert material it is 400 °C, on the $SrTiO_3$ lowers to 390 °C while on K-containing samples decreases to 220 °C for $Sr_{0.5}K_{0.5}TiO_3$. The positive effect of NO_2 on soot combustion is commonly known and reaction of NO_2 with soot can be described by following scheme [27]:

$$-C* + NO_2 \rightarrow -C(O) + NO \tag{2}$$

$$-C(O) + NO_2 \rightarrow -C(ONO_2) \tag{3}$$

$$-C(ONO_2) \rightarrow CO_2 \text{ or } CO + NO + -C* \tag{4}$$

where C*: carbon active site (coordinatively unsaturated carbon atoms formed by the decomposition of oxygen containing surface complexes); –C(O): surface oxygen complexes (SOCs); $-C(ONO_2)$: peroxynitrate complexes.

NO_2 reacts with carbon active site producing surface oxygen complexes (SOCs) –C(O) (2). SOCs react with NO_2 from gas phase and form $C(ONO_2)$ (3), which can decompose above 200 °C to CO_2 or CO and NO or NO_2.

Potassium can also promote combustion of soot according to mechanism proposed by Aneggi et al. [28]. The oxidized form C–O–AM (AM – alkali metals) reacts with carbon from soot forming CO and reduced carbon–oxygen complexes (C–AM) is formed. In the next step the C–AM is reoxidized by molecular oxygen:

$$C{-}O{-}AM + C \rightarrow C{-}AM + CO \tag{5}$$

$$C{-}O{-}AM + CO \rightarrow C{-}AM + CO_2 \tag{6}$$

$$2C{-}AM + O_2 \rightarrow 2C{-}O{-}AM \tag{7}$$

However this mechanism needs direct contact between potassium and soot surface and therefore is less probable.

Additionally, the partial substitution of divalent Sr^{2+} by monovalent K^+ in $SrTiO_3$ changes electron density distribution in the structure because oxygen vacancies are formed to keep the lattice charge balance. Formation of oxygen ions on vacancies cannot be excluded and Oliva et al. [13] suggest that on the $SrTiO_3$ surface O^- ions could be formed through following elementary steps:

$$\text{solid} \rightarrow \text{solid} + + e-$$

$$O_{2(g)} + 2e^- \rightarrow 2O \tag{8}$$

$$O^- + O_{2(g)} \leftrightarrow O_3 \tag{9}$$

$$O^- + \text{solid}+ \rightarrow [O^- - \text{solid}^+] \tag{10}$$

where solid+ is either Sr^{2+} or Ti^{4+} and the activation of oxygen through the first two (thermodynamically not favored) reactions occurs through its adsorption and incorporation into the oxide crystal lattice.

Merino et al. [27] also proposed that oxygen-deficient vacancies can create sites for oxygen activation.

According to both mechanisms vacancies are not necessary to produce ionic oxygen species. Nevertheless, it was also reported [29] that adsorbed $O_2{}^{2-}$ are formed by interaction between oxygen molecule and oxygen vacancy:

$$Vo^{\bullet\bullet} + O_2 \rightarrow O_2{}^{2-} + 2h+ \tag{11}$$

(Kroger–Vink notation: $Vo^{\bullet\bullet}$ – oxygen vacancy, h+ – positive hole). Thus, oxygen from gas phase is adsorbed and activated on these vacancies, what increases concentration of "surface oxygen" species possessing basic character. Then the active surface oxygen species are transported through the surface oxide layer to the catalyst-soot interface, where the soot particles are oxidized to carbon oxides. In a previous work [30], we found that because temperature of potassium desorption from K-substituted $SrTiO_3$ is ca 120 °C higher than temperature of soot ignition on this material, a direct effect of potassium on the catalytic activity (e.g. vaporization and direct contact with soot) can be excluded. Thus, activity of $Sr_{1-x}K_xTiO_3$ can be explained by creation of vacancies and increasing concentration of basic surface oxygen species on perovskite A similar effect was previously observed when copper partially substituted titanium on perovskite structure [22]. Consequently, the studied material can be considered as an oxygen carrier transferring oxygen from the gas phase to soot surface. It is generally accepted that the promotion by potassium can facilitate the dissociative adsorption of molecules by lowering the surface work function [31]. In the case of molecular oxygen, the electron transfer from the catalyst surface to the antibonding orbital of oxygen molecule leads to the formation of strongly reactive surface oxygen species such as O^- and $O_2{}^-$ which are known as efficient soot oxidation agents [32]. It is worth to notice that proposed model is fairly close to both Mars–Van Krevelen mechanism and the presented by Haber electronic approach of catalytic oxidation [34]. Gaseous oxygen is adsorbed and activated on the catalyst surface. Resulting reactive oxygen species (electrophilic) attack then the reactive sites on soot surface C* (rich in electron density) to give an oxygen containing intermediates. It is clear that close contact between soot and catalyst is essential requirement in such model.

On the other hand, the incorporation of K into structure of studied materials strongly increases the value of IRSO in spite of lowering of the temperature of measurements (Table 2). The IRSO on K-substituted catalysts is ca one order higher than on the $SrTiO_3$ and the r_{CO_2}/r_{CO} ratio is sensitive on the degree of substitution x. Once again, the most visible effect of potassium is for $x = 0.2$.

Table 2
Isothermal rate of soot oxidation in (10% $O_2 + N_2$).

Catalyst	$r_{CO_2} \times 10^7$ $\mathrm{mol_{CO_2}/m^2_{cat.}\ s}$	$r_{CO} \times 10^7$ $\mathrm{mol_{CO}/m^2_{cat.}\ s}$	r_{CO_2}/r_{CO}	$r \times 10^7$ $\mathrm{mol_{soot}/m^2_{cat.}\ s}$
$SrTiO_3$ (T_m = 450 °C)	1.6	1.2	1.3	2.8
$Sr_{0.9}K_{0.1}TiO_3$ (T_m = 430 °C)	4.4	2.2	2.0	6.6
$Sr_{0.8}K_{0.2}TiO_3$ (T_m = 350 °C)	8.7	2.7	3.2	11.4
$Sr_{0.6}K_{0.4}TiO_3$ (T_m = 345 °C)	8.3	2.1	4.0	10.4
$Sr_{0.5}K_{0.5}TiO_3$ (T_m = 330 °C)	4.5	1.1	4.1	5.6

T_m – temperature at which IRSO was determined.

Table 3
Isothermal rate of soot oxidation in (1500 ppm NO_2 + 10% $O_2 + N_2$).

Catalyst	$r_{CO_2} \times 10^6$ $\mathrm{mol_{CO_2}/m^2_{cat.}\ s}$	$r_{CO} \times 10^6$ $\mathrm{mol_{CO}/m^2_{cat.}\ s}$	r_{CO_2}/r_{CO}	$r \times 10^6$ $\mathrm{mol_{soot}/m^2_{cat.}\ s}$
$SrTiO_3$ (T_m = 370 °C)	1.4	1.1	1.3	2.5
$Sr_{0.9}K_{0.1}TiO_3$ (T_m = 240 °C)	4.2	1.9	2.2	6.1
$Sr_{0.8}K_{0.2}TiO_3$ (T_m = 210 °C)	10.7	3.2	3.3	13.9
$Sr_{0.6}K_{0.4}TiO_3$ (T_m = 200 °C)	9.9	2.5	3.9	12.4
$Sr_{0.5}K_{0.5}TiO_3$ (T_m = 190 °C)	5.4	1.3	4.2	6.7

T_m – temperature at which IRSO was determined.

In the presence of 1500 ppm NO_2 in gas phase, the IRSO values are again notably increased even considering the decrease in the measurement temperature. Note that, in the presence of NO_2 the effect of potassium substitution, even positive, is less pronounced than for O2 atmosphere. Finally, the presence of NO_2 does not affect on the r_{CO_2}/r_{CO} ratio in comparison to NO (Table 1) for which the value of this ratio depends on the degree of strontium substitution by potassium (Table 3).

The results presented in this work can be explained considering that on the perovskites surface various basic oxygen species differing in electrophilic character O_2^-, O_2^{2-} or O^- can exist [27,33]. Partial substitution of Sr by potassium in $SrTiO_3$ increases the amount of basic oxygen species on the surface in comparison with the undoped one (Table 1) and it can expect that materials exhibiting higher CHON/CHEN ratio will be more active for oxidation as it was confirmed by the soot ignition temperature and IRSO values (as well as CO_2/CO ratio). Thus, the incorporation of K into $SrTiO_3$ creates oxygen vacancies in the surface and the most effective situation is when the degree of substitution is 0.2. O_2 (NO or NO_2) from gas phase can be adsorbed and activated on these vacancies increasing the share of "active surface oxygen species" that are transported through the surface oxide layer to the catalyst-soot interface, where the soot particles are oxidized to carbon oxides (CO/CO_2).

4. Conclusions

Partial substitution of strontium by K in $SrTiO_3$ creates oxygen vacancies where molecular oxygen or (NO, NO_2) is adsorbed forming basic surface oxygen species active for soot oxidation. The most efficient substitution degree is $x = 0.2$. Over $Sr_{0.8}K_{0.2}TiO_3$, the soot ignition temperature is as low as 380 °C in a (10% O_2 in Ar) and 240 °C in a (1500 ppm NO_2 + 10% O_2 in Ar) while over inert material corresponding temperatures are 635 °C and 570 °C respectively.

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