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Hydrogen production by steam reforming (SR) of DME over Cu catalysts and de-NOx performance of a combined system of SR + LNT

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ABSTRACT

This study was performed to develop a DME SR catalyst with high H_2 selectivity using a heat source and water in the exhaust emissions of vehicles. The final goal of the research is to develop a combined system of SR + LNT for improving the de-NOx performance of the catalyst system in a DME engine. The scope of this research encompasses the optimization of the Cu concentration for the SR catalyst, evaluation of the NOx conversion efficiency of the LNT catalyst according to the kind of reductant (H_2, CO, and DME), and evaluation of the de-NOx performance of the LNT catalyst and a combined system of SR + LNT. As a result of experiments concerning DME SR catalysts manufactured through the sol–gel method, the Cu20%/γ-Al_2O_3 catalyst showed the highest DME conversion and H_2 selectivity. The combined system of SR + LNT using a Cu20%/γ-Al_2O_3 SR catalyst increased the NOx conversion by about 20% compared to the LNT catalyst alone at temperatures over 300 °C. In the combined system of SR + LNT, a supply of 1% of DME results in a higher NOx conversion than a supply of 0.75%, but results in NH_3 slip and higher fuel consumption; therefore, supplying 0.75% of DME is favorable.

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1. Introduction

DME has the appearance of an excellent and efficient alternative fuel for use in a diesel engine. It promises smoke-free combustion because of a high oxygen content of 34.8% and cetane number of 55 [1]. A DME engine has slightly lower NOx (nitrogen oxide) emissions than a diesel engine. It can reduce NOx emissions further through EGR (Exhaust Gas Recirculation). However, excessive use of EGR results in output reduction due to the lower combustion enthalpy; therefore, the EGR rate is limited. Thus, the reduction of NOx is an important challenge when DME is applied to diesel engines [2,3]. Various NOx reduction methods such as LNT (Lean NOx Trap), Urea-SCR (Selective Catalytic Reduction) [4,5], and HC-SCR are well known as after-treatment systems. Urea-SCR is the most effective among these techniques, and is employed mainly to reduce NOx in the case of heavy-duty diesel engines [4,5]. On the other hand, LNT can effectively remove the adsorbed NOx using reductants (H_2, CO and HC) that can be obtained through the post-injection of fuel without a separate device after the adsorption of NOx in a lean condition [6]. However, LNT also uses fuel as a reductant that worsens its fuel consumption. Thus, it is necessary to supply an optimal reductant with a high NOx conversion efficiency. H_2 is an optimal reductant for LNT with fast reactivity; therefore, it has excellent activity at low temperature [7]. Several researchers have investigated a DME SR (Steam Reforming) reaction to generate H_2 by reacting DME with H_2O followed by a partial oxidation reaction to produce H_2 and CO by reacting DME with O_2 [7–10]. Currently, research is underway on the generation of H_2 using low-temperature plasma and diesel fuel cracking catalysts to enhance the selectivity of H_2 [11]. Copper (Cu) is a relatively cheap transition metal for the DME SR catalyst and precious metals are also being developed for the DME SR catalyst [12]. The DME SR reaction has high H_2 selectivity but it is an endothermic reaction; therefore an external heat source and H_2O have to be supplied for the reaction. If DME is directly injected into the exhaust pipe in front of the DME SR catalyst, it can efficiently use the heat source and water vapor from the exhaust gas, which are necessary for the SR reaction [13–15]. However, these studies have not considered the heat source and water vapor in the exhaust emission for the DME SR reaction. Especially, there is no research on the improvement of the NOx conversion of the LNT catalyst using the H_2 or CO that is generated by the DME SR catalyst as a reductant. This study was conducted to develop a DME SR catalyst with high H_2 selectivity using the heat source and water in the exhaust emission of vehicles. The final goal of the research is to develop a combined system of SR + LNT to improve the performance of the de-NOx catalyst system in a DME engine. As of now, the SR catalyst produces H_2 and CO for use as reductants for the LNT catalyst.

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Table 1
Specifications of the catalyst.

Catalyst	Composition (wt%)	BET (cm^2/g)	Pore volume (cm^3/g)
SR	Cu20/γ-Al_2O_3	32.05	0.077
LNT	1Pt/0.03Rh/0.02Pd	39.88	0.125

2. Experimental details

2.1. Preparation of catalysts

The DME SR catalysts were directly manufactured through the sol–gel method [7,16], and a commercialized LNT catalyst (Ordeg Co.) was used. The loaded quantities of Cu in the SR catalysts manufactured by the sol–gel method were 10, 20, and 30 (Cu metal, wt%) of the washcoat. Aluminum isopropoxide ($Al[OCH(CH_3)_2]$, Sigma–Aldrich, 99.9%) was dissolved in 100 ml of H_2O at 70 °C for 20 min. A proper amount of Cu precursor ($[Cu(NO_3)_2 2.5H_2O]$, Sigma–Aldrich) was put in; then, 10 ml of ethylene glycol was injected. The mixture was stirred for an hour, followed by the addition of HNO_3 to adjust the pH to two. The solution was washed, and then dried for 24 h at 130 °C. The washcoat was milled for 2 h, and then calcined for 5 h at 500 °C. A calcined Cu-washcoat was mixed with H_2O for preparing the slurry, stirred for 2 h, and then repeatedly dipped in a substrate. All the Cu catalysts were calcined at 500 °C for 2 h; then, they were reduced with 10% H_2/N_2 at 300 °C for 4 h. The detailed specifications for the DME SR catalysts and the LNT catalyst are shown in Table 1.

2.2. Experimental apparatus

In order to evaluate the performance of the DME SR catalysts and the NOx conversion of a combined system of SR + LNT, a laboratory-scale normal-pressure fixed-bed model gas catalyst reactor was set up, as shown in Fig. 1 [7]. A cylindrical quartz tube of 19 mm inner diameter and 350 mm length was used as the catalytic reactor, and the catalysts were fixed to the center of the reaction tube. The experiment used a catalyst of 18 mm diameter and 30 mm length (600 cpsi, Cordierite, Cu/γ-Al_2O_3) for the DME SR catalyst and 18 mm diameter and 15 mm length (400 cpsi, Cordierite) for the LNT catalyst. The hydrocarbons and DME were measured by a GC (Gas Chromatograph, HP 6890) with an FID (Flame Ionization Detector) and a TCD (Thermal Conductivity Detector). A PLOT U column and a Molecular Sieve 5A column, respectively, were used to measure the DME, H_2, CH_4 and CO with Ar carrier gas. A Horiba 554JK was used for CO_2 analysis. The combined system of SR + LNT had the DME SR catalyst at the front and the LNT catalyst at the rear, and the interval between the catalysts was 5 mm. The catalyst temperature of the front of the SR catalyst (T1 point), between the rear of the DME SR catalyst and front of the LNT catalyst (T2 point), and the rear of the LNT catalyst (T3 point) were measured using a 1.6 mm K-sheath thermocouple. FTIR (Fourier Transform Infrared Spectroscopy, MIDAC) was used for the measurement of NOx. A heat exchanger was installed to prevent the DME gas temperature from decreasing due to the latent heat of evaporation. The H_2O necessary for reactions was supplied while the water temperature was kept constant via a saturated steam pressure and N_2 was passed through water, whereby the water vapor was separated from GC before measurement.

2.3. Experimental method

The experimental conditions to evaluate the H_2 selectivity of the DME SR catalysts and the NOx conversion using the combined system of SR + LNT are shown in Table 2. The evaluation of the H_2 selectivity of the DME SR catalysts used 14,000 h^{-1} as the SV (space velocity). The SR catalyst temperature was T1 and varied in 50 °C intervals from 200 °C to 500 °C in steady conditions. Before reaction, all catalysts were reduced with 10% H_2 at each

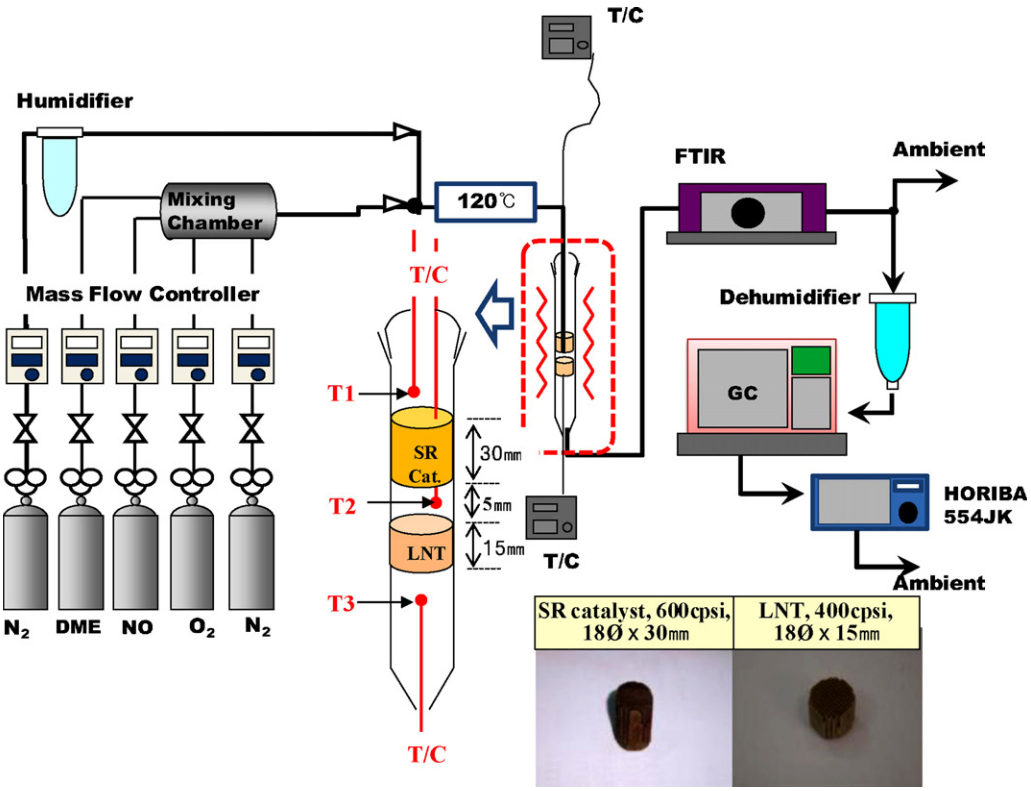

Fig. 1. Experimental apparatus and catalysts.

Table 2
Experimental conditions.

Parameter	DME steam reforming	Combined system reforming of SR + LNT	
		Lean	Rich
Total flow (L/min)	2	2	
SV (h^{-1})	14,000	28,000	
DME (%)	1	–	1
H_2O (%)	5	5	
Balance gas	N_2	N_2	
NO (ppm)	–	500	–
O_2 (%)	–	10	–
Lean/rich time (s)	–	55	5

temperature in order to measure the H_2 selectivity under same conditions. The DME conversion of the catalyst was obtained from the conversion (%), viz., (([DME_{in}] – [DME_{out}])/[DME_{in}]) × 100, by measuring the gas concentration at the inlet and outlet of the catalyst reaction part. The selectivity was obtained from the reactant gas productivity (%), viz., ((yield ratio of the reactant gases/yield ratio of ($H_2 + CO + CH_4 + CO_2$)) × 100(%)) [12]. In the combined system of SR + LNT, Cu20%/γ-Al_2O_3 was used as the DME SR catalyst. The lean and rich conditions were maintained for 55 and 5 s, respectively. SV was maintained at 28,000 h^{-1} for the LNT catalyst because of the varying volume of the catalyst. A combined system of SR + LNT accompanies both endothermic and exothermic reactions. DME performs endothermic reaction with the SR catalyst, but the LNT catalyst performs exothermic reaction. Therefore the temperature varies depending on the position of the catalytic system. Thus the T2 setting of the temperature is usually called the "temperature" that was used for evaluating the DME and NOx conversions, selectivity. The experiment was performed in both steady and transient conditions. The measurement of the specific surface area of a catalyst used a physical adsorption method (the BET (Brunauer–Emmett–Teller) method) for measuring the N_2 gas adsorption quantity pursuant to the pressure change at liquid nitrogen temperature (77 K) after pretreatment in a 1×10^{-6} Torr vacuum condition at 300 °C; it involved taking a part of the sample catalysts. The particle size of the DME SR catalyst was analyzed by TEM (Transmission Electron Microscopy, × 200k, JEM-2000FX2, Shimadzu).

3. Results and discussion

3.1. Performance regarding H_2 and CO generation in DME SR catalysts

Fig. 2(a) shows the DME conversion of the Cu10, 20, and 30 wt%/γ-Al_2O_3 DME SR catalysts and Fig. 2(b) shows the reaction product selectivity of the Cu20%/γ-Al_2O_3 catalyst. Of the DME SR catalysts, the Cu20%/γ-Al_2O_3 catalyst yield the highest DME conversion and H_2 selectivity. The DME conversion of the Cu20%/γ-Al_2O_3 catalyst at 350 °C was 80%; at this time, the H_2 selectivity was 71%. The H_2 selectivity was 73% and the CO selectivity was 5% at 400 °C. Over 450 °C, the H_2 selectivity decreased, but the CO selectivity increased as the DME SR reaction became increasingly incomplete. Finally, at 500 °C, the selectivity of H_2 and CO_2 gradually decreased due to the reverse water shift reaction [17]. Methanol was not observed as an intermediate product because of the fast reaction over the Cu in the SR catalyst. A very small CH_4 selectivity was detected in the result (not shown in the figure). The generation of CO is undesirable for the purpose of supplying pure H_2 to fuel cells, but the production of a small amount of CO besides H_2 is also useful as a reductant for the LNT catalyst.

Fig. 3 shows TEM images of the catalyst size and dispersion of Cu particles manufactured in the sol–gel method. The Cu10%/γ-Al_2O_3 particles were homogeneously dispersed; however, the Cu30%/γ-Al_2O_3 particles with the highest loading of Cu grew through the coherence of Cu particles. As a result of Fig. 2, Cu20%/γ-Al_2O_3 appears to yield better H_2 selectivity; further, the particle size is smaller and the dispersion more homogeneous. Cu might change into CuO in the lean condition of the exhaust gas. However, CuO had to perform an oxidation reaction with a part of DME, and was reduced again to Cu. Because DME injected at a rich condition reacted with the oxygen that was intercepted over the combined system of SR + LNT, the remaining DME would react again over the Cu of the SR catalyst and form hydrogen [17,18].

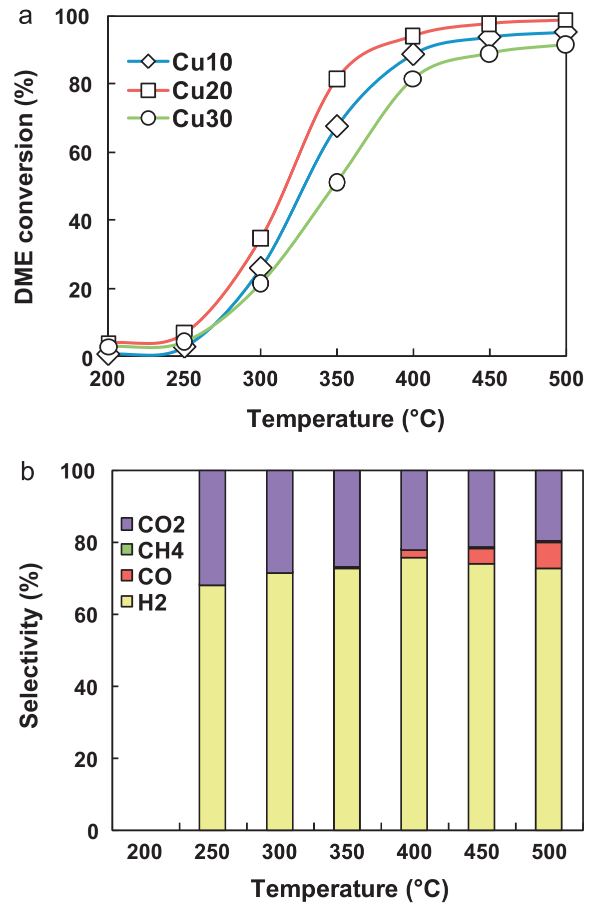

Fig. 2. (a) DME conversion. (b) Selectivity of products under the Cu20%/γ-Al_2O_3 catalyst.

3.2. NOx conversion of a combined system of SR + LNT

Fig. 4 shows the NOx conversion of the LNT catalyst according to the kinds of reductant. The final objective of this research is to improve the NOx conversion via a combined system of SR + LNT. Therefore, at first, the H_2 and CO generated from the DME SR reaction were simulated using reductants of the LNT catalyst. In the DME SR reaction, 6 mol% of H_2 was generated when DME of 1 mol% reacted as per the reaction. This result shows the NOx conversion at 200 °C, 350 °C, and 450 °C at the front of the LNT catalyst under a temperature of T2 in a steady-state condition. The NOx conversion was higher as the supply quantity of the reductant is greater; the LNT catalyst showed the highest NOx conversion at 350 °C. Especially, H_2 is a more effective reductant regarding the NOx conversion compared with other reductants, such as CO and DME, at 200 °C. Supplying H_2 rather than CO or DME as a reductant yields a better NOx conversion for the LNT catalyst at all tempera-

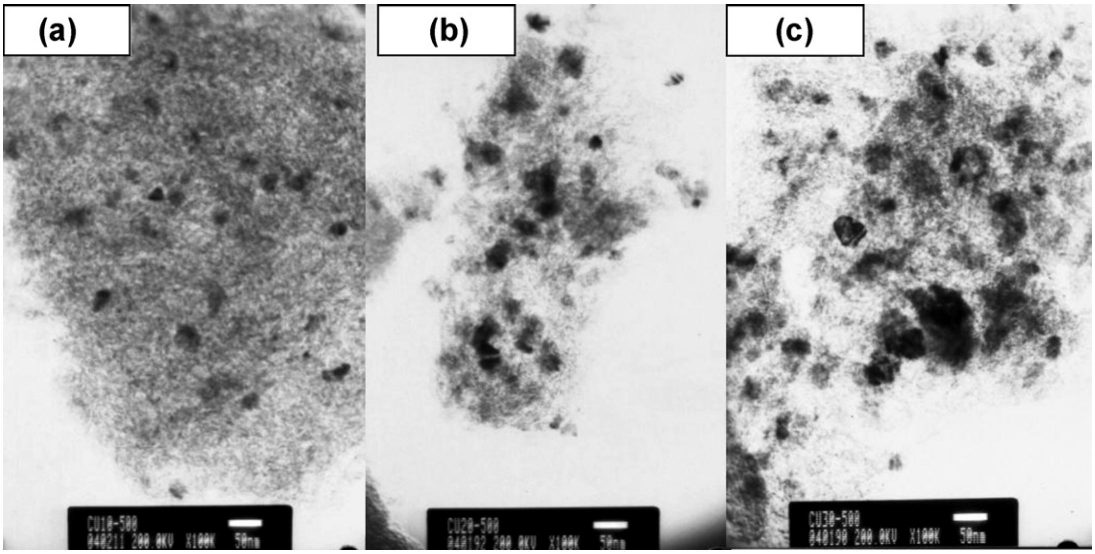

Fig. 3. TEM images of Cu catalysts manufactured by the sol–gel method ((a) Cu10%, (b) Cu20%, and (c) Cu30%, × 100k).

ture. This is why this research will generate H_2 using the DME SR catalyst.

Fig. 5 represents the NOx conversion of the combined system of the LNT catalyst and the SR (Cu20%/γ-Al_2O_3) catalyst. The NOx conversion of the combined system of SR + LNT was higher than that of the LNT catalyst with DME of 1%. This is because H_2 is generated over the DME SR catalyst and used as a reductant for the LNT catalyst. However, the variation in the NOx conversion with the reductant at 200 °C was small between the LNT catalyst and the combined system of SR + LNT, which was because the SR catalyst could not reach the activation temperature. The NOx conversion at a high temperature of 450 °C was similar or lower compared to that at 350 °C. Thus, it is found that a combined system of SR + LNT is necessary to improve the de-NOx performance of the LNT catalyst for DME-fueled engines.

Fig. 6 shows the characteristics of the temperature profiles at the LNT catalyst and the combined system of SR + LNT. Fig. 6(a) shows the T2 temperature at the front of the LNT catalyst and the T3 temperature at the rear of the LNT catalyst. T2 was the temperature setting of the furnace. The T3 temperature was increased due to the DME oxidation reaction with the O_2 that was adsorbed on the catalyst in lean and low temperature conditions. The T2 temperature also was increased over the temperature setting because of the thermal radiation heat transfer of the LNT catalyst over 350 °C.

Fig. 6(b) shows the temperature profiles when the temperature setting was T1. The T2 temperature was higher than the T1 temperature. T1 dropped due to the endothermic reaction of DME [19,20]. Therefore, T1 required a large heat source to rise up to the set temperature. Further T2 and T3 temperature rose over this time because of DME oxidation on the LNT catalyst. T2 was affected by both the endothermic reaction of the SR catalyst and the exothermic reaction of the LNT catalyst.

Fig. 6(c) shows the temperature profiles when the temperature setting was T2. T1 dropped due to the endothermic reaction of DME, and T2 did not require a large heat source compared to that of the T1 temperature setting. T2 gradually decreased due to the active endothermic reaction at the DME SR catalyst over 350 °C. This phenomenon also serves to suppress the aging of catalysts by decreasing the temperature of the LNT catalyst [21]. T3 increased compared to the T2 temperature setting because of the exothermic reaction of the LNT catalyst. Here, the T2 point has been determined as the catalyst temperature for evaluating the de-NOx performance of the catalysts.

LNT catalyst with sulfur poisoning is usually required beyond 600 °C for desulfurization [22], and this high temperature can accelerate the thermal deactivation of the LNT catalyst. However, the

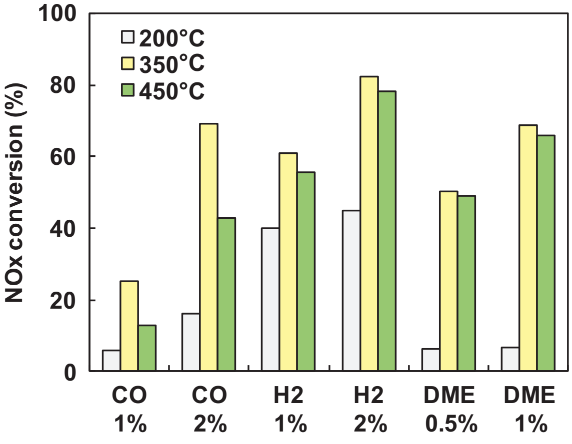

Fig. 4. NOx conversion according to the kind and supply quantity of reductant.

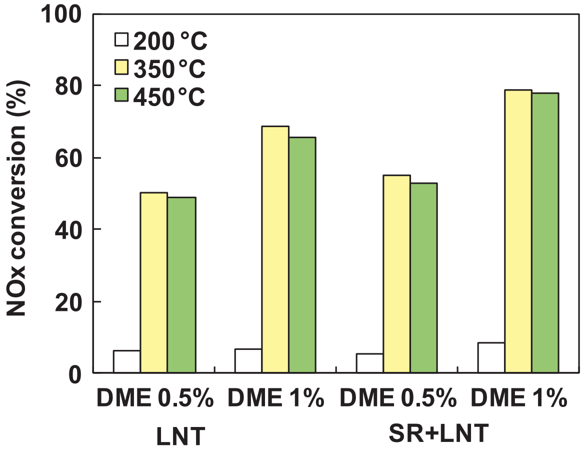

Fig. 5. NOx conversion for LNT and a combined system of SR + LNT according to the quantity of DME.

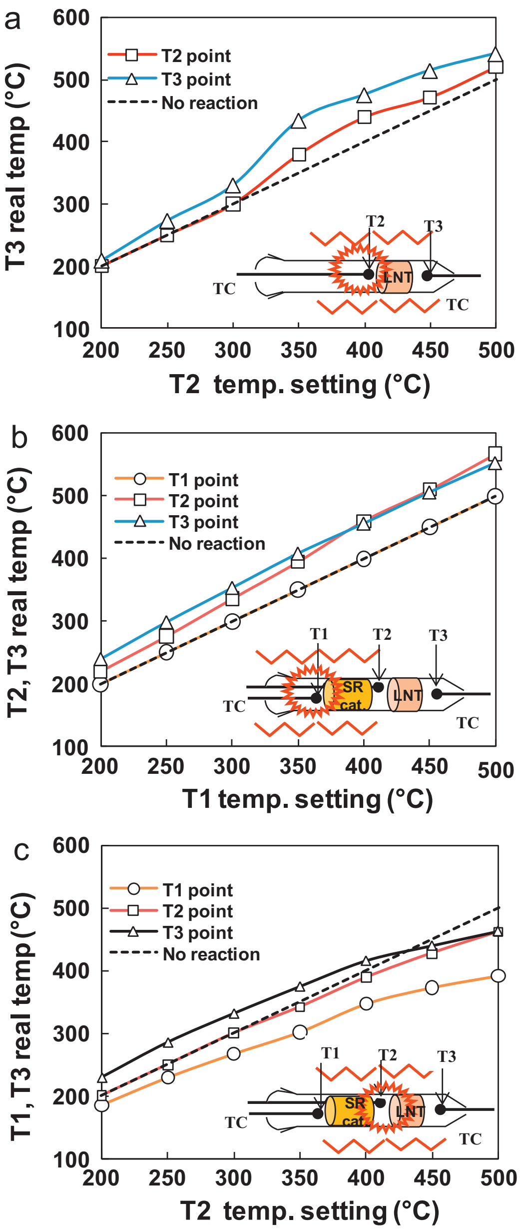

Fig. 6. Catalyst temperature in the LNT and a combined system of SR + LNT. ((a) LNT, temp. S. = T2, (b) SR + LNT, temp. S. = T1, (c) SR + LNT, temp. S. = T2).

combined system of SR + LNT does not need a high temperature for desulfurization because DME is sulfur-free fuel.

Fig. 7 shows the NOx conversion depending on the temperature when DME of 1% is supplied as the reductant in the LNT catalyst and in the combined system of SR + LNT. As mentioned above, SR + LNT (T1) shows the result of the temperature setting T1, and SR + LNT (T2) shows that of the temperature setting T2. Although both are combined systems of SR + LNT, the NOx conversions of the combined systems of SR + LNT (T1) and SR + LNT (T2) are very different because the temperature of the SR and LNT catalyst was different. The NOx conversion of the combined system of SR + LNT (T1) was 76% at 250 °C, but rapidly decreased over 350 °C. On the other hand, the NOx conversion of the combined system of SR + LNT (T2) showed a slight difference from that of the LNT catalyst at 250 °C. However, the NOx conversion was about 20% higher compared to that of the LNT catalyst at higher temperature. This is because the H_2 and some CO generated from the DME SR catalyst were used as reductants for the LNT catalyst. It is necessary to develop a DME SR catalyst with higher H_2 selectivity at low temperature in order to improve the NOx conversion of SR + LNT at low temperature of 200–250 °C. Beyond 450 °C, NOx may not be adsorbed onto the LNT catalyst, and is immediately desorbed; therefore, the NOx conversion decreases slowly [23].

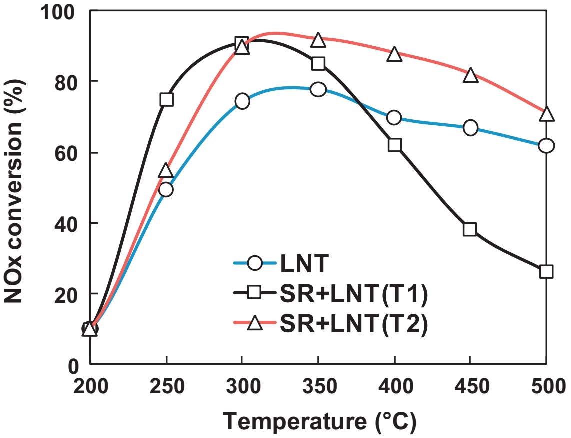

Fig. 7. NOx conversion according to the temperature setting.

Fig. 8 illustrates the NOx conversion according to the supply concentration of DME in the LNT catalyst and the combined system of SR + LNT supplied with 0.75% and 1% of DME, respectively. Supplying 0.75% and 1% of DME to the combined system of SR + LNT yield a higher NOx conversion than supplying 1% of DME to the LNT catalyst. The NOx conversion was similar to those of LNT and the combined system of SR + LNT although the H_2 selectivity of the SR catalyst was very low at 250 °C. This is the performance regarding NOx conversion with only the LNT catalyst. However, the highest NOx conversion in the combined system of SR + LNT was about 90% at 350 °C. Especially, the supply of only 0.75% of DME to the combined system of SR + LNT resulted in a 10% improvement in the NOx conversion beyond 300 °C. Supplying 0.75% of DME can diminish the penalty in terms of fuel consumption, while the NOx conversion is maintained at about 85%.

Fig. 9 shows the concentrations of the reaction gases according to the supply quantities (0.75% and 1%) of DME for the combined system of SR + LNT in a transient condition. Supplying 1% of DME to a combined system of SR + LNT yields a higher NOx conversion (as seen in Fig. 8), but NH_3 of 100 ppm is emitted. NH_3, which is an intermediate product, is generated in line with the H_2 concentra-

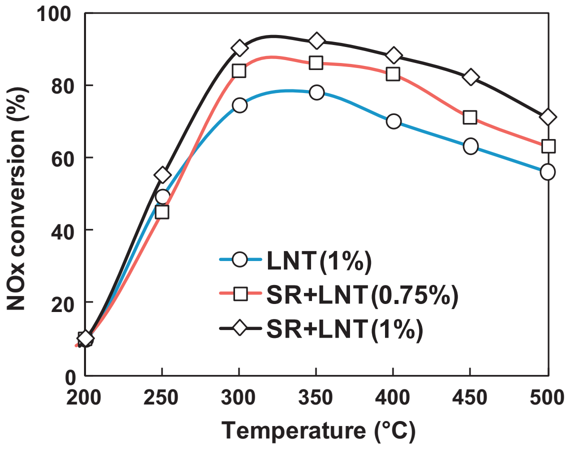

Fig. 8. NOx conversion according to the supply concentration of DME in the de-NOx system.

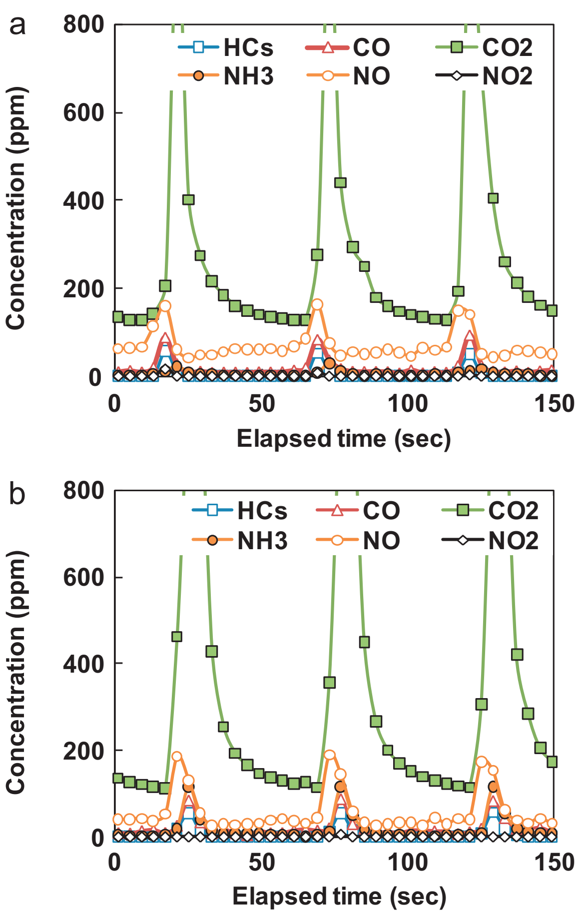

Fig. 9. Concentrations of the reaction gases according to the quantity of DME in a combined system of SR + LNT (SV 28,000 h^{-1}, DME 1%, NO 500 ppm, O_2 10%, N_2 balance, H_2O 5%, and temperature 350 °C) (a) DME 0.75%, (b) DME 1%.

tion in rich conditions, as described in Eq. (1) [24].

$$Ba(NO_3)_2 + 8H_2 \rightarrow 2NH_3 + BaO + 5H_2O \quad (1)$$

H_2 is formed via the water–gas shift reaction; therefore, the quantity is dependent on the CO concentration in the exhaust gas. A loss in the catalyst function in the LNT catalyst may result in the partial oxidation of HC to CO rather than CO_2, thereby increasing the exhaust H_2 concentration and subsequent NH_3 generation [24,25]. Many studies are ongoing to improve the NOx conversion by attaching NH_3-SCR catalysts to the rear of the LNT catalyst to meet the NH_3 slip regulation of 10 ppm in the exhaust emission of a vehicle; the regulation takes effect in 2014 [25–27]. On the other hand, supplying 0.75% of DME is poorer in terms of the NOx conversion than a supplying 1% of DME (as seen in Fig. 8), but NH_3 is emitted a slightly. It has been found that supplying 0.75% of DME is more favorable than supplying 1% as a reductant when considering the fuel penalty of the after-treatment system for DME-fueled vehicles.

4. Conclusions

This study was performed to develop a DME SR catalyst with high H_2 selectivity using the heat source and water in the exhaust emission of vehicles and the combined system of SR + LNT to improve the de-NOx performance of the after-treatment system in DME engines. The following conclusions were obtained as a result of the research. Of the DME SR catalysts manufactured in the sol–gel method, the Cu20%/γ-Al_2O_3 catalyst showed the highest DME conversion and H_2 selectivity. Beyond 450 °C, the H_2 selectivity decreased, but the CO selectivity increased with increasing incompleteness of the DME SR reaction. The combined system of SR + LNT using the Cu20%/γ-Al_2O_3 catalyst yielded a NOx conversion that was about 20% higher than that under the LNT catalyst alone beyond 300 °C. This is because the H_2 and some CO generated from the DME SR catalyst were used as reductants for the LNT catalyst. It is necessary to develop a DME SR catalyst with higher H_2 selectivity at low temperature in order to improve the NOx conversion of SR + LNT at low temperature of 200–250 °C. In the combined system of SR + LNT, supplying 1% of DME results in a higher NOx conversion than 0.75%, but also results in NH_3 slip and higher fuel consumption; therefore, a supplying 0.75% of DME is favorable. In the combined system of SR + LNT, supplying 0.75% and 1% of DME yielded higher NOx conversion than supplying 1% of DME in the case of LNT catalyst. Especially, supplying only 0.75% of DME in the combined system of SR + LNT resulted in a 10% improvement in the NOx conversion beyond 300 °C. It can diminish the penalty regarding fuel consumption, while the NOx conversion is maintained at about 85%. It was found from the transient test that supplying 0.75% of DME as a reductant is more favorable than supplying 1% when considering the fuel penalty of the after-treatment system for DME-fueled vehicles.

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