

EFFECT OF LENGTH ON THE PERFORMANCE OF LEAN NO<sub>x</sub> TRAPSVitaly Y. Prikhodko<sup>(1)</sup>, Ke Nguyen<sup>(1)</sup>, Jae-Soon Choi<sup>(2)</sup> and Stuart Daw<sup>(2)</sup>

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**ABSTRACT:** *The effect of monolith length on the NO<sub>x</sub> performance of Lean NO<sub>x</sub> Traps (LNTs) has been investigated using a bench flow reactor (BFR). The composition of the catalyst samples consists of Pt/K supported on  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> washcoat. Samples of 2.22-cm in diameter and three different lengths of 2.54, 5.08 and 7.62 cm were evaluated at a fixed gas hourly space velocity (GHSV) using long and short-cycle experiments with only H<sub>2</sub> as the reductant in the rich phase, and the NO<sub>x</sub> performance was compared between samples of different lengths. No significant difference in performance was observed in long and short-cycle experiments with full regeneration. On the other hand, significant difference in performance was observed in short-cycle experiments with partial regeneration: the longer the sample the better the performance. The intra-catalyst concentration of H<sub>2</sub> measured by a Spatially Resolved Capillary Inlet Mass Spectrometer (SpaciMS) at different axial locations in short-cycle experiments with partial regeneration indicated that higher H<sub>2</sub> consumption in shorter samples is responsible for the reduction of NO<sub>x</sub> performance.*

**Keywords:** *Exhaust Gas Emissions, Exhaust Gas Aftertreatment, NsOx solution.*

## 1. INTRODUCTION

Lean NO<sub>x</sub> Trap (LNT) offers higher NO<sub>x</sub> conversion efficiency and wider operating temperature window than Selective Catalytic Reduction (SCR) and Lean NO<sub>x</sub> Catalyst (LNC), making LNT an attractive candidate for NO<sub>x</sub> abatement in lean-burn engine exhaust. In LNT technology, NO<sub>x</sub> reduction is achieved by storing NO<sub>x</sub> during normal lean exhaust conditions and reducing the stored NO<sub>x</sub> during periodic rich excursions of short duration.

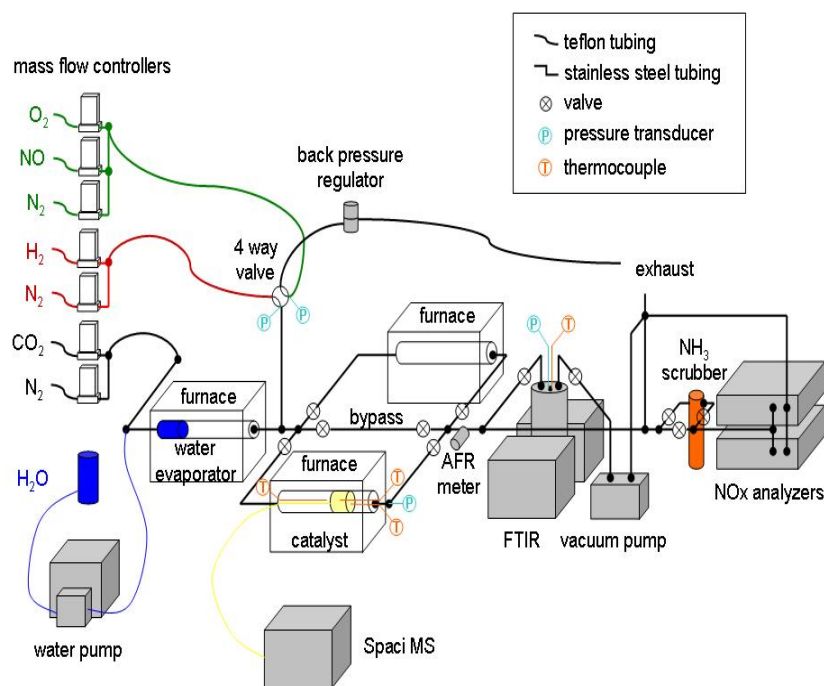
A typical LNT catalyst consists of a honeycomb-like ceramic monolith coated with three primary components: precious metals such as Pt, Pd and Rh, alkali or alkaline earth metals such as Ba and K supported on high surface  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> washcoat. During normal lean exhaust conditions, NO is oxidized to NO<sub>2</sub> over the precious metal and stored on alkali/alkaline earth metals in the form of nitrites or nitrates. During periodic rich excursions, NO<sub>x</sub> is released from the storage sites and (ideally) reduced to N<sub>2</sub> over the precious metals [1-13].

Even though changing gas hourly space velocity affects NO<sub>x</sub> conversion in LNTs, i.e., NO<sub>x</sub> conversion increases with decreasing GHSV due to longer residence time [14, 15], NO<sub>x</sub> conversion should not be affected by changing the sample length as long as the space velocity or the residence time remains constant. However, significantly different results are often obtained from LNT evaluation experiments performed under the same conditions, i.e., same catalyst formulation, temperature, space velocity, and gas mixture composition [16]. As different sample sizes are often used in LNT evaluation as imposed on by reactor size, sample length is suspected as the culprit of LNT performance disparity.

The main objective of the present investigation is to determine if and how monolith length affects LNT performance. Samples of same diameter of three different lengths were evaluated at a fixed GHSV using a bench-flow reactor. To maintain constant GHSV, irrespective of the length of the sample, the volumetric flow rate or the linear gas velocity in the sample has to be increased with increasing sample length.

## 2. EXPERIMENTAL APPARATUS AND PROCEDURE

A bench flow reactor illustrated schematically in Figure 1, was used to investigate the effect of monolith length on LNT performance. Individual components of simulated exhaust gases are introduced into the reactor via mass flow controllers. For lean/rich cycling a solenoid-actuated four-way valve with fast switching time (0.2 s) was used to introduce the lean or rich pulse into the reactor. A peristaltic pump was used to deliver liquid water to the water evaporator in which liquid water absorbed onto quartz wool evaporated and was swept by inert carrier gases. Directly downstream of the water evaporator, inert gases and water vapor were combined with lean or rich gases and introduced to the reactor.



**Figure 1.** Schematic of the bench flow reactor

LNT catalyst sample of 2.22 cm in diameter was wrapped in Zetex ceramic fiber strips to prevent gas bypass and then inserted in a 2.54-cm OD quartz tube, which in turn is placed inside a tubular furnace. The catalyst sample was typically positioned 25.4 cm from the quartz tube inlet. The section upstream of the catalyst sample was filled with glass beads to promote mixing and uniform heating of the incoming gases. Intra-channel gas phase speciation was performed by Spatially Resolved Capillary Inlet Mass Spectrometer (SpaciMS), which was developed at the Oak Ridge National Laboratory (ORNL) for application to the BFR and other types of emissions and engine measurements where extractive sampling is possible [17]. The SpaciMS used 185  $\mu\text{m}$  OD capillary probes to transport gas mixtures from the reactor to the mass spectrometer. The sampling ends of the capillary probes were inserted through the front end fitting of the reactor and positioned in the radial center at different locations along the

catalyst length. The other ends of the probes were connected to a multiple-port valve leading to spectrometer's ionization chamber

Exiting gases were analyzed with a MIDAC Fourier Transform Infrared Spectrometer (FTIR) and two California Analytical Instruments NO<sub>x</sub> chemiluminescence analyzers. The FTIR equipped with 375 mL and 3-m path length gas absorption cell was used for measuring the concentration of NO<sub>2</sub>, N<sub>2</sub>O, NH<sub>3</sub>, CO, CO<sub>2</sub> and H<sub>2</sub>O. Upstream of the NO<sub>x</sub> analyzers, an NH<sub>3</sub> scrubber was used to prevent NH<sub>3</sub> from interfering with NO<sub>x</sub> measurements.

A full-size Pt/K/γ-Al<sub>2</sub>O<sub>3</sub> LNT monolith of cordierite substrate with a cell density of 31 cells per square centimeter supplied by EmeraChem under the trade name of SCONO<sub>x</sub> was used in the present study. Samples of 2.22-cm in diameter and length of 2.54, 5.08 and 7.62-cm were cored from full-size monolith and evaluated with two sets of cycling experiments: long and short-cycle experiments. Short-cycle experiments were intended to approximate the catalyst operation in engine exhaust applications while long cycling were performed to explore processes which are not, otherwise, observed in short-cycle experiments. Both sets of cycling experiments were performed at three different temperatures of 200, 300 and 400°C and a fixed GHSV of 30,000 h<sup>-1</sup>. In the present investigation H<sub>2</sub> was used as the sole reductant in all of the experiments. Two different H<sub>2</sub> concentrations were used in short-cycle experiments: one to simulate partial regeneration and the other full regeneration. The H<sub>2</sub> concentration for partial regeneration experiments represents the concentration of the reductant that is below the theoretical amount needed to reduce the integral amount of NO<sub>x</sub> that enters during the trapping part of the cycle. Full regeneration uses significantly higher reductant concentration than theoretical one to ensure that regeneration of the catalyst is not limited by the amount of reductant. The theoretical amount of reductant is calculated based on the assumption that all of the NO<sub>x</sub> entering the reactor is stored as KNO<sub>3</sub> during the lean phase, and reduced to N<sub>2</sub> during the rich phase. Typical operating conditions for long and short-cycle experiments are given in Tables 1 and 2, respectively.

**Table 1.** Long cycling operating conditions

Mode	Time	Gas Composition
Lean	15 min	300 ppm NO, 10% O <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>
Rich	10 min	0.2% H <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>
Lean	15 min	300 ppm NO, 10% O <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>
Rich	10 min	0.5% H <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>

**Table 2.** Short cycling operating conditions

Mode	Time	Gas Composition
Lean	56 s	300 ppm NO, 10% O <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>
Rich	4 s	1% H <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>
Lean	56 s	300 ppm NO, 10% O <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>
Rich	4 s	2% H <sub>2</sub> , 5% H <sub>2</sub> O, 5% CO <sub>2</sub> , balance N <sub>2</sub>

Before each cycling experiment, the catalyst was heated to 500°C and subjected to the regeneration condition consisting of 1% H<sub>2</sub>, 5% H<sub>2</sub>O, 5% CO<sub>2</sub> and a balance of N<sub>2</sub> for 15 minutes for cleansing off adsorbed NO<sub>x</sub> from catalyst surface. The catalyst was then cooled down to the desired operating temperature.

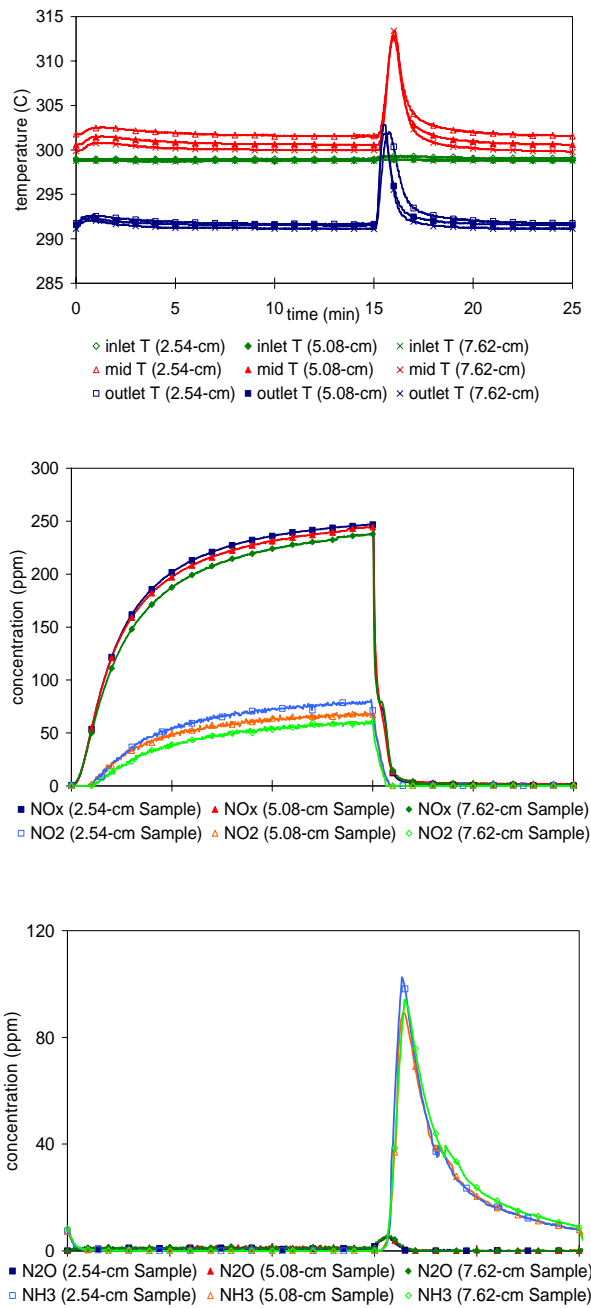
To investigate the mechanism for reductant consumption due to the catalytic reaction between H<sub>2</sub> and O<sub>2</sub> at the interface between the lean and rich phases; the extent of which depends on the degree of mixing at the lean/rich interface, a N<sub>2</sub> purge of 10 s between lean and rich pulse – referred to as back-mixing experiments – was introduced into the BFR. With a N<sub>2</sub> purge an inert environment between the lean and rich front was created, which should eliminate H<sub>2</sub> consumption due to lean/rich front back-mixing.

Prior to the catalyst evaluation, the catalyst samples were “degreened” to achieve stable and reproducible levels of their activity, by performing short storage-reduction cycles for 8 hours at 550°C, which is 150°C higher than the highest operating temperature used in the present study.

### 3. RESULTS AND DISCUSSION

Long-cycle experiments were performed on 2.54-cm, 5.08-cm and 7.62-cm-long samples at temperatures of 200, 300 and 400°C using two different reductant concentrations of 0.2 and 0.5% in the rich phase. Figure 2 shows typical catalyst outlet gas concentrations and temperature profiles for 2.54, 5.08 and 7.62 cm-long samples with 0.2% H<sub>2</sub> in the rich phase of the cycle at 300°C. Three cycles were run and the results are shown only for the third cycle. No significant difference in the catalyst outlet gas concentrations and temperatures is observed between 2.54, 5.08 and 7.62-cm-long samples with 0.2% H<sub>2</sub> as the reductant. Similar results were also obtained at 200 and 400°C. The cycle average NO<sub>x</sub> conversion efficiencies for 2.54, 5.08 and 7.62-cm-long samples closely match each other at any given temperature. Table 3 lists the cycle average NO<sub>x</sub> conversion efficiencies for experiments performed at 300°C. The differences in NO<sub>x</sub>/NO<sub>2</sub> profiles and NO<sub>x</sub> conversion efficiencies between samples of three different lengths seem to indicate that the longer the sample the better the performance. However, the differences in NO<sub>x</sub> conversion efficiencies fall within the experimental uncertainty, which makes it difficult to conclude whether these differences result from length variation or experimental limitations.

A second set of long-cycle experiments was performed with 0.5% H<sub>2</sub> concentration in the rich phase (results are not shown). Except for one major difference – NH<sub>3</sub> and N<sub>2</sub>O are produced in larger quantities – the trends for these cycles are similar to those with 0.2% H<sub>2</sub>. As in the case of long-cycle experiments with 0.2% H<sub>2</sub> in the rich phase, no significant difference is observed between 2.54, 5.08 and 7.62-cm-long samples with temperature. Thus, in long cycle experiments the LNT's performance is not affected by sample length at a given temperature and reductant concentration.

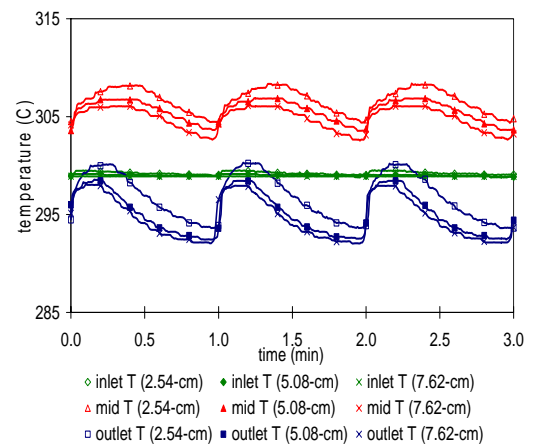
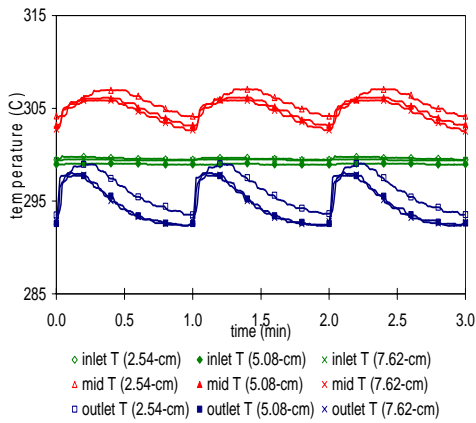
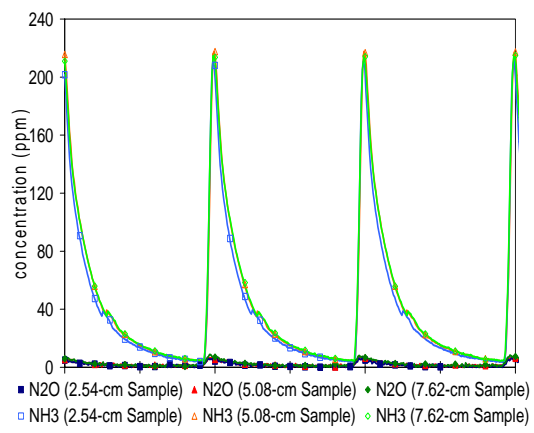
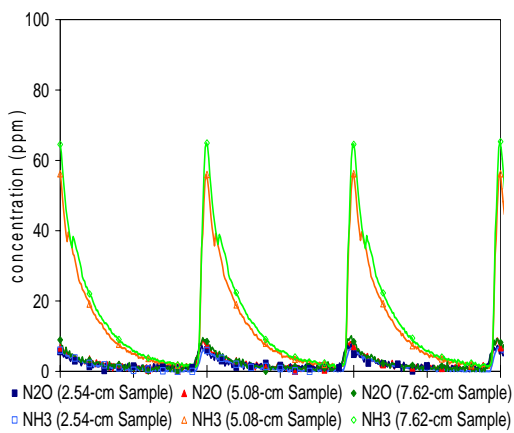
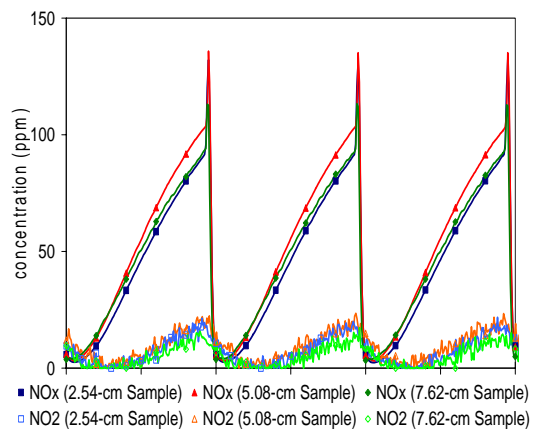
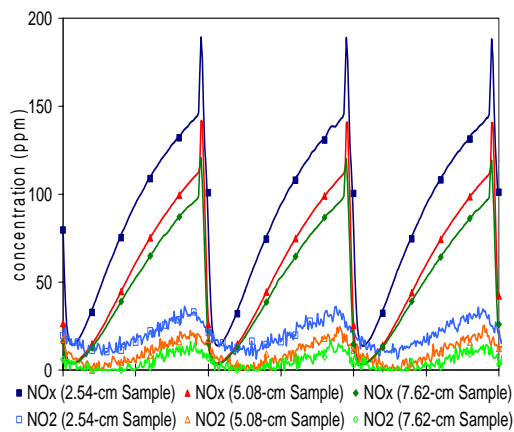


**Figure 2.** Outlet gas concentrations and temperature profiles for 2.54, 5.08 and 7.62-cm-long samples in long-cycle experiments with 0.2% H<sub>2</sub> in rich phase at 300°C

**Table 3.** NO<sub>x</sub> conversion efficiencies in long-cycle experiments with 0.2% H<sub>2</sub> in rich phase

<b>Length \ Temp.</b>	300°C
2.54-cm	25.4%
5.08-cm	26.4%
7.62-cm	29.1%

Figure 3 shows the catalyst outlet gas concentrations and temperature profile for short-cycle experiments with 1% H<sub>2</sub> in the regeneration period, whereas Table 4 lists the cycle average NO<sub>x</sub> conversion efficiencies. Results for 2% H<sub>2</sub> at 300°C are shown in Figure 4 and Table 5.



**Figure 3.** Outlet gas concentrations and temperature profiles for 2.54, 5.08 and 7.62-cm-long samples in short-cycle experiments with 1.0% H<sub>2</sub> in rich phase at 300°C

**Figure 4.** Outlet gas concentrations and temperature profiles for 2.54, 5.08 and 7.62-cm-long samples in short-cycle experiments with 2.0% H<sub>2</sub> in rich phase at 300°C

A total of 120 cycles were performed but only 3 representative cycles acquired toward the end of the 120-cycle run are shown in the figures. The general trends for the short-cycle experiments are similar to those obtained in the long-cycle experiments. No significant difference is observed between 2.54, 5.08 and 7.62-cm-long samples in short-cycle experiments with 2% H<sub>2</sub> in regeneration phase, and a difference of 15% is observed between 2.54 and 7.62-cm-long samples using 1% H<sub>2</sub> (partial regeneration). On the other hand a difference of only 3.4% is observed between 5.08 and 7.62-cm-long samples with 1% H<sub>2</sub>, which is within the experimental uncertainty; and thus it is inconclusive to attribute this difference to variation in length. Amount of NH<sub>3</sub> produced in 5.08 and 7.62-cm-long samples is very similar and significantly larger than in 2.54-cm-long sample in experiments with 1% H<sub>2</sub>. Results of short-cycle experiments show that catalyst's performance is affected by sample length during short - cycle experiments when regeneration is limited by the amount of reductant available. On the other hand, increasing the catalyst's length from 5.08 to 7.62 cm appears not to cause significant change in performance regardless of reductant concentration.

**Table 4** NO<sub>x</sub> conversion efficiencies in short-cycle experiments with 1.0% H<sub>2</sub> in rich phase

Length \ Temp.	300°C
2.54-cm	65.7%
5.08-cm	77.1%
7.62-cm	80.5%

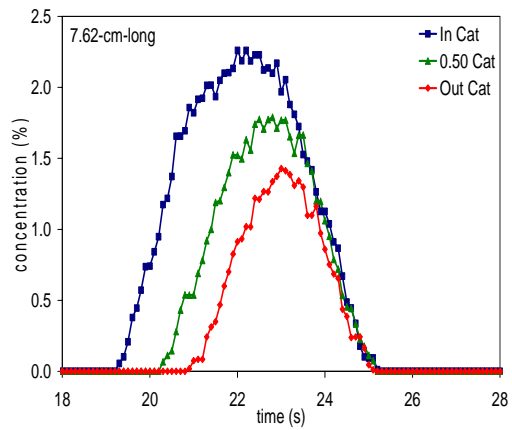
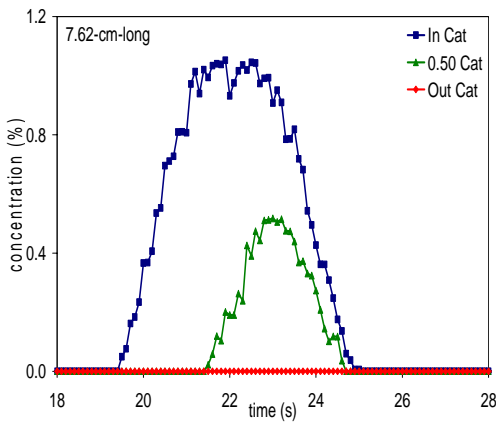
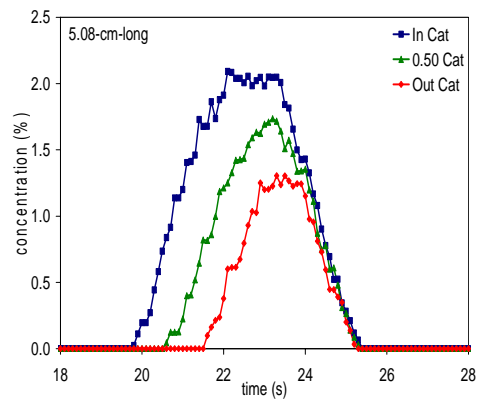
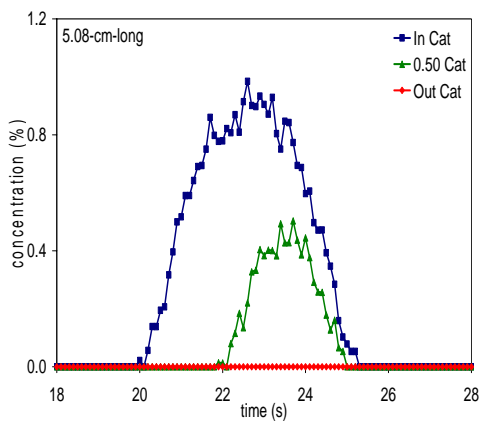
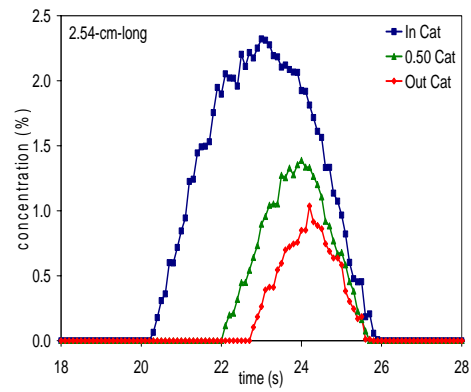
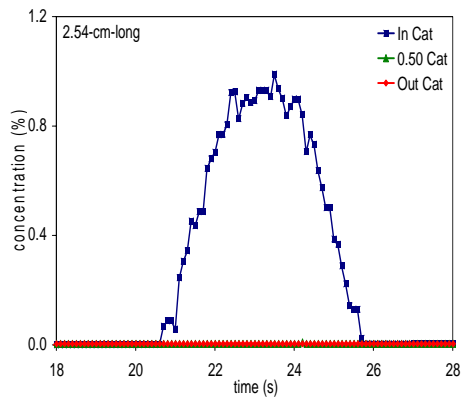
The intra-catalyst concentration of H<sub>2</sub> was measured at three different axial locations – catalyst inlet, mid-section and exit – with SpaciMS, and the H<sub>2</sub> concentration profiles for short-cycle experiments at 300°C are shown in Figures 5 and 6. From these profiles, information on the consumption of H<sub>2</sub> along the length of 2.54, 5.08 and 7.62-cm-long samples can be extracted. As seen in Figure 5, in short-cycle experiments with 1% H<sub>2</sub> in the rich phase, H<sub>2</sub> is completely consumed in the front half of the 2.54-cm-long sample; whereas about 25% of H<sub>2</sub> remains in the 5.08 and 7.62-cm long samples. With 2% H<sub>2</sub> in the rich phase of the short-cycle experiments, 80% is consumed in 2.54-cm-long sample, whereas approximately 60% of H<sub>2</sub> is consumed in the entire length of 5.08 and 7.62-cm-long samples (see Figure 6).

**Table 5** NO<sub>x</sub> conversion efficiencies in short-cycle experiments with 2.0% H<sub>2</sub> in rich phase

Length \ Temp.	300°C
2.54-cm	82.1%
5.08-cm	79.5%
7.62-cm	81.7%

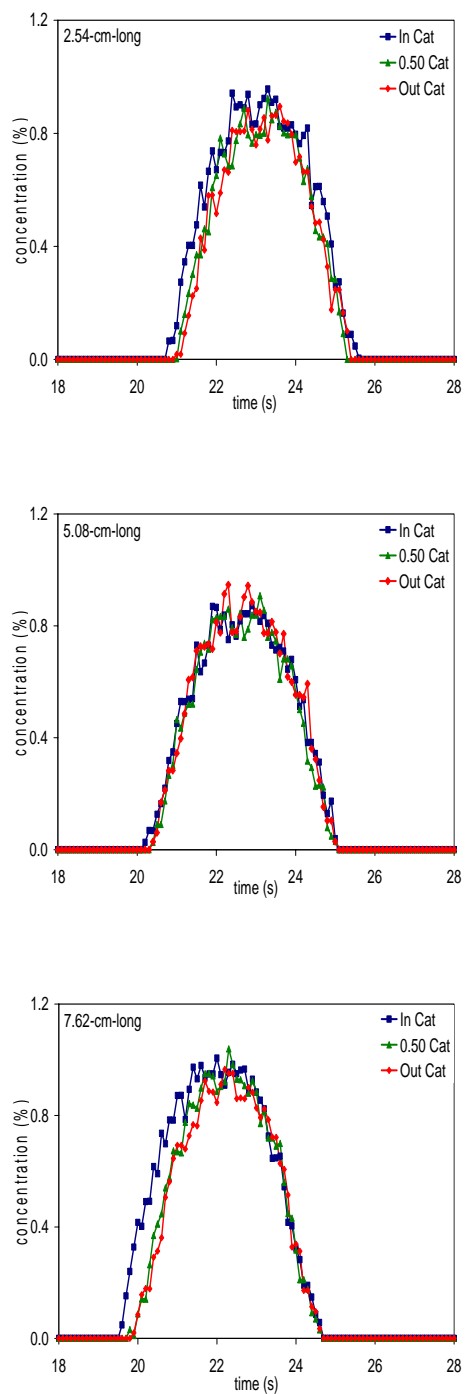
Higher consumption of H<sub>2</sub> in 2.54-cm-long sample should result in higher NO<sub>x</sub> conversion, however the opposite is observed: 2.54-cm-long sample has lower NO<sub>x</sub> conversion efficiency than longer samples in short-cycle experiments with partial regeneration, and no difference is observed with full regeneration.

Different H<sub>2</sub> consumption in 2.54, 5.08 and 7.62-cm-long samples seems to indicate that the consumption of H<sub>2</sub> might possibly occur via the catalytic reaction between H<sub>2</sub> and O<sub>2</sub> at the interface between the lean and rich phases. The extent of H<sub>2</sub> consumption depends on the degree of mixing at the lean/rich interface. Higher degree of back-mixing would result in higher H<sub>2</sub> consumption and lesser amount of H<sub>2</sub> available for reducing stored NO<sub>x</sub>, thereby lowering catalyst's NO<sub>x</sub> conversion. The extent of lean/rich front back-mixing can be seen in Figure 7, in which a N<sub>2</sub> purge of 10s is introduced between the lean and rich front, seems to eliminate H<sub>2</sub> consumption due to mixing at the lean/rich interface. As seen in the figure, N<sub>2</sub> purge decreases H<sub>2</sub> consumption in the front half of the 2.54-cm-long sample by 35% in experiment with 1% H<sub>2</sub> in rich phase and by about 10% in both 5.08 and 7.62-cm-long samples. Similar results are obtained in back-mixing experiments with 2% H<sub>2</sub> (not shown).



**Figure 5.** H<sub>2</sub> consumption trends in 2.54, 5.08 and 7.62-cm-long samples in short-cycle experiments with 1.0% H<sub>2</sub> in rich phase at 300°C

**Figure 6.** H<sub>2</sub> consumption trends in 2.54, 5.08 and 7.62-cm-long samples in short-cycle experiments with 2.0% H<sub>2</sub> in rich phase at 300°C



**Figure 7.** H<sub>2</sub> consumption trends in 2.54, 5.08 and 7.62-cm-long samples in back-mixing short-cycle experiments with 10s N<sub>2</sub> purge and 1.0% H<sub>2</sub> in rich phase at 300°C

Thus, higher consumption of H<sub>2</sub> in 2.54-cm-long sample than in 5.08 and 7.62-cm samples can be attributed to higher degree of back-mixing in the shorter sample. For example, in the 2.54-cm-long sample as much as 35% of H<sub>2</sub> is consumed from the catalytic oxidation at the lean/rich interface – compared to only 10% in the longer sample – resulting in a reduction of 15% in NO<sub>x</sub> conversion in short-cycle experiments with 1% H<sub>2</sub>. The higher consumption of H<sub>2</sub> in shorter sample is further confirm by smaller quantity of NH<sub>3</sub> formed in 2.54-cm-long sample, since NH<sub>3</sub> production decreases with decreasing H<sub>2</sub> concentration [18].

#### 4. CONCLUSIONS

The effect of monolith length on the NO<sub>x</sub> performance of Lean NO<sub>x</sub> Traps was investigated in the present study. Samples of three different lengths were evaluated with long and short - cycle experiments at a fixed GHSV.

No significant difference in performance was observed in long and short-cycle experiments with full regeneration. On the other hand, significant difference is observed in short-cycle experiments with partial regeneration: the longer the sample the better the performance. A difference as large as 20% in NO<sub>x</sub> conversion efficiency was observed between 2.54 and 7.62-cm-long samples with lower performance in shorter sample.

H<sub>2</sub> consumption trends during short-cycle experiments indicate different degrees of lean and rich front back-mixing for samples of different lengths. Higher back-mixing results in a higher H<sub>2</sub> loss via its oxidation by O<sub>2</sub> and lesser H<sub>2</sub> availability for reducing stored NO<sub>x</sub>, which in turn affects catalyst's performance when regeneration is limited by amount of H<sub>2</sub> available. In 2.54-cm-long sample as much as 35% of H<sub>2</sub> is consumed from the catalytic oxidation at the lean/rich interface resulting in a reduction of 15% in NO<sub>x</sub> conversion in short-cycle experiments with partial regeneration.

### CƠ CHẾ MẤT TÁC DỤNG CỦA BỘ XÚC TÁC NO<sub>x</sub> ĐÃ BỊ LÃO HÓA NHIỆT

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**TÓM TẮT:** Bộ xúc tác làm sạch NO<sub>x</sub> (LNTs) có chứa các chất Pt, Pd, Rh, Ba, Ce, Zr, và các chất phụ gia khác trên lớp nền của hợp kim  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> được đưa vào khảo sát trong trạng thái lão hóa nhiệt ở các nhiệt độ 750, 880, 930, và 1070°C. Dùng các phương pháp XRD, STEM/EDS, BET phân tích khả năng lưu giữ NO<sub>x</sub>, oxy hóa NO, chuyển biến NO<sub>x</sub> và phân tích DRIFTS để xác định các cơ chế mất tác dụng của bộ xúc tác ở nhiệt độ lão hóa cao. Khi sự phân tán PGM giảm, sự oxy hóa theo mol bề mặt PGM giảm ở các nhiệt độ đánh giá 200, 300, và 400°C. Sự lưu giữ NO<sub>x</sub> và nhiệt độ tách hấp thu theo chương trình (TPD) thí nghiệm thực hiện với DRIFTS ở 200, 300, và 400°C chỉ ra rằng một lượng đáng kể NO<sub>x</sub> được lưu giữ trên nền  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> dưới dạng các nitrates ở 200 và 300°C trước lão hóa. Tuy nhiên, hầu như không có chất nitrate nào trên nền alumina sau khi lão hóa ở 900 và 1000°C, kết quả là có sự giảm đáng kể khả năng lưu giữ NO<sub>x</sub>. Không có sự tạo thành các nitrates.

**Từ khóa:** Độ phát thải, Xử lý khí thải, Giải pháp Nox.

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