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N_2O decomposition and formation of NO_x species on Fe–ferrierite. Effect of NO and CO addition on the decomposition and the role of surface species

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The decomposition of a mixture of $^{15}N_2O$ and $^{14}N_2O$ does not lead to a formation of $^{14}N^{15}N$ on Fe–ferrierite, so that the N–N bond is not dissociated during this reaction. On the other hand, mono-nitro NO_x surface species are created during the N_2O decomposition, and are desorbed as NO_2 and NO into the gas phase above 300 °C. The reaction of N_2O with active oxygen is suggested to explain their formation. NO_2 and NO are also found after the addition of NO to N_2O which substantially enhances the nitrous oxide decomposition. The addition of CO instead of NO accelerates the N_2O decomposition and suppresses the formation of surface NO_x species. The dependence of the decomposition of nitrous oxide on the added CO and/or NO is different. Operation of both agents is discussed together with the temperature of the Fe–ferrierite pretreatment.

KEY WORDS: decomposition of N_2O; Fe–ferrierite; surface species desorbed; effect of NO and/or CO, low- and high-temperature pretreated samples; isotope labeling.

1. Introduction

Enhancing role of several organic compounds on the decomposition of nitrous oxide over Fe-zeolites has been reported in several papers (e.g., refs. [1,2]); NO and CO belong to the important inorganic agents [3,5–7]. The role of the latter compounds has been assumed to consist in the acceleration of oxygen desorption which is assumed to be the slowest step in the N_2O decomposition [3]. However, this assumption has been criticized in a recent paper by Heyden *et al.* [4]. Pérez-Ramírez *et al.* [5–7] found a different role of isolated and oligonuclear Fe species in the operation of NO as well as in the simultaneous function of NO + CO. Sang *et al.* [8] suggest that NO_x species formed during the NO-assisted decomposition of nitrous oxide play a role of oxygen storage and can change the pure oxide-oxo redox mechanism without NO in redox cycles between nitro–nitrite or nitrite–nitrate species.

Formation of surface NO_x species during the decomposition of N_2O alone has also been reported [9–12]. The present study aims to contribute to the role of the NO_x intermediates comparing the decomposition of N_2O, $^{15}N_2^{18}O$, $^{14}N_2O + {}^{15}N_2O$, $N_2O + NO$ and $N_2O + {}^{13}CO$ at various temperatures, as well as the composition of desorbed surface species during the temperature programmed desorption (TPD). The effect of the dehydroxylation by temperature pretreatment of Fe–ferrierite is discussed and compared with the NO effect on the N_2O decomposition. ^{18}O labelled nitrous oxide is employed to check the mechanism of dioxygen formation from decomposing N_2O.

2. Experimental

Fe–ferrierite (further Fe/FER) was prepared by the reaction of H–ferrierite (Si/Al = 8.5) with $FeCl_3$ solution in acetylacetone [13]. The sample contains 0.55 wt% of iron (Fe/Al = 0.6) located predominantly in cationic positions. Prior to measurements, Fe/FER (50–150 mg) was pretreated *in situ* in vacuum for 4 h at 450 °C (LTP – low temperature pretreatment) or at 650 °C (HTP – high temperature pretreatment). Then the sample was held in 2 kPa of oxygen and evacuated during the cooling to the reaction temperature of 250, 280 and 300 °C. Nitrous oxide is decomposed at 250 °C to N_2 while the majority of oxygen remains captured on the zeolite, at 280 °C the released dioxygen predominates over that which remains captured, and at 300 °C the stoiocheometric ratio of N_2 and $1/2O_2$ in the gas phase appears.

About 500 Pa of $^{14}N_2O$ (99.99) either alone or in mixture with $^{15}N_2O$ (99 at.%, Iconisotopes) or NO (99.9, MG Co) or ^{13}CO (Aldrich Chem. Co., 99 at.%, both CO and NO in various ratios to N_2O) was introduced onto the sample in a quartz reactor (163 mL volume), and a negligible amount of the gas phase was led by a needle valve into a Balzers QMG 420 quadrupole mass spectrometer. ^{13}CO was employed to distinguish between molecular ions of N_2O, CO_2, N_2 and CO. The participation of zeolite oxygen atoms in the decomposition of nitrous oxide over LTP and HTP

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samples was checked using ^{18}O labelled N_2O ($^{15}N_2^{18}O$, 99 at.%, Medical Isotopes, Inc., USA). Details concerning the latter experiments are given in ref. [14]. After the reactions (monitored for 60–90 min) the gas phase was shortly evacuated (below 5 Pa), temperature decreased to 180 °C in the closed reactor (the pressure increased to ca 7–9 Pa) and temperature programmed desorption (TPD) with heating rate of 5 °C/min was started after the evacuation below 10^{-4} Pa. The gases released were led by a direct inlet into the mass spectrometer.

3. Results

3.1. Mono-nitro surface species from N_2O decomposition

The decomposition of $^{14}N_2O$ at 250 °C over Fe/FER is shown in figure 1a, the following TPD in figure 1b. The same is displayed in figure 1c and d for the almost equimolecular mixture of $^{14}N_2O$ with $^{15}N_2O$. Nitrous oxide decomposes at this temperature releasing dinitrogen, while the majority of corresponding oxygen remains captured in the zeolite. Only $^{14}N_2$ and $^{15}N_2$ appear during the decomposition of the mixture of ^{14}N and ^{15}N nitrous oxide; $^{14}N^{15}N$ is not formed (in measurable amounts), so that the N–N bond is not broken during the decomposition. However, this has to occur at the surface as follows from the TPD curves (figure 1b and d): small amounts of NO_2 and NO are released together with the evolved dioxygen. These NO_x species were not present as impurities in the parent nitrous oxide.

3.2. Effect of added NO and CO on the decomposition of N_2O

The decomposition of nitrous oxide at 250 °C is affected differently by the amount of added NO and CO. This is exemplified in figure 2a, in the bottom part for *the addition of NO* onto LTP (open circles) and HTP

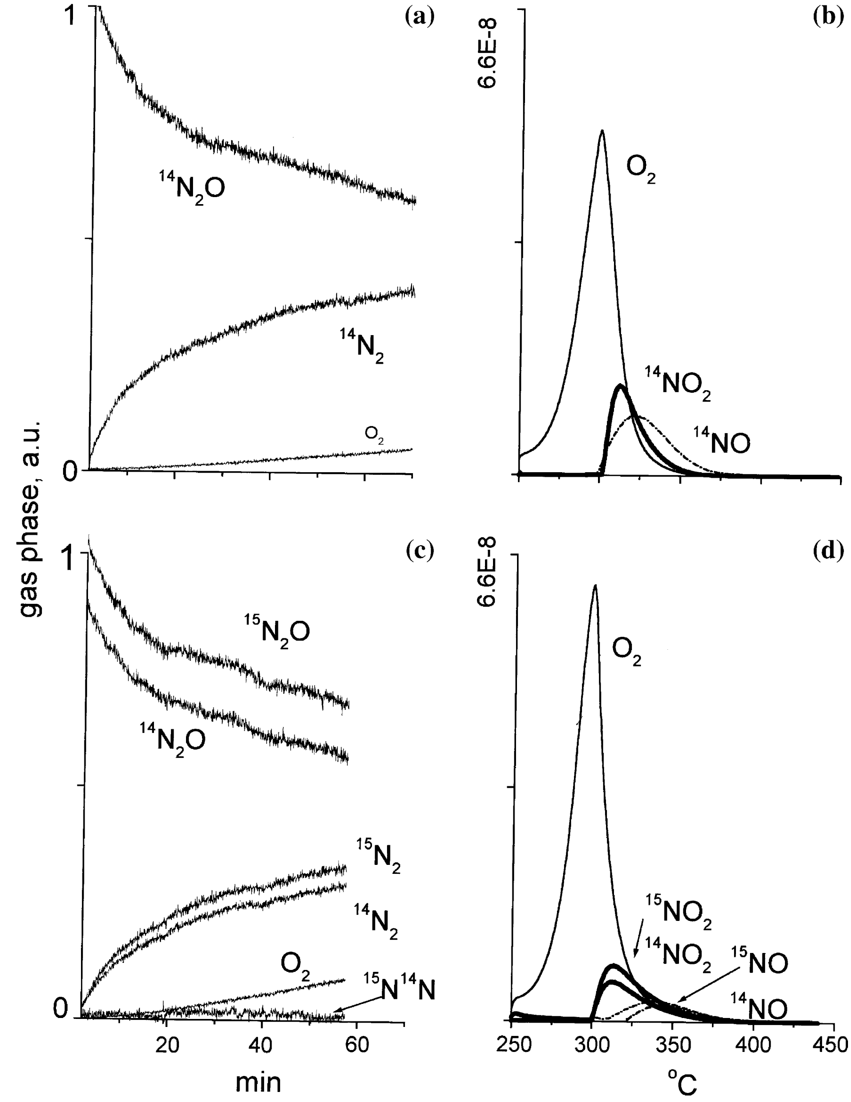

Figure 1. Decomposition of $^{14}N_2O$ and $^{14}N_2O + {}^{15}N_2O$. Following TPD 147 mg of Fe/FER, 250 °C, 33 mmol of nitrous oxide: (a) time dependence of $^{14}N_2O$ decomposition, (b) following TPD, (c) time dependence of 1:1.1 mixture of $^{14}N_2O$ and $^{15}N_2O$, (d) following TPD.

(solid circles) samples. The 100% decomposition of N_2O under the experimental conditions employed equals to 33 μmol. It can be seen that the HTP treatment almost doubles the N_2O decomposition without the addition of nitric oxide (the increase marked by a bold arrow). Very similar decomposition increase (1.9× vs. 1.8×) occurs over the LTP sample after the addition of small amounts of NO (NO/N_2O ratio 0.1 – dotted arrow). Higher amount of added NO (NO/N_2O ratio 0.5–1) does not furthermore substantially affect the decomposition of nitrous oxide. Addition of NO over the HTP sample increases less the N_2O decomposition than over the LTP sample (1.2×, dashed arrow). Again, higher amount of added NO does not further contribute to the decomposition of nitrous oxide.

Addition of CO affects the N_2O decomposition differently. The CO/N_2O ratio 0.1 has virtually no effect, while higher CO amounts proportionally increase the N_2O decomposition as can be seen in the top part of figure 2a. The HTP treatment of Fe/FER does not markedly contribute to the effect of carbon monoxide – the extent of the N_2O decomposition with added CO increases in the same way over LTP as well as over HTP Fe/FER samples.

The majority of oxygen from decomposing nitrous oxide remains captured in the zeolite during the decomposition of nitrous oxide at 250 °C. Both NO as well as CO are oxidized by this captured oxygen to NO_2 and CO_2, respectively. However, under the experimental conditions employed, NO_2 remains adsorbed on the zeolite (only a very small part of NO_2 accompanies the release of dinitrogen during the decomposition), while the majority of carbon dioxide is released during the reaction. Besides the oxidation by captured oxygen, a minor fraction of dioxygen which was evolved into the gas phase together with dinitrogen during the decomposition of nitrous oxide is consumed by these reactants. Thus the O_2/N_2 ratio in the gas phase during the decomposition of nitrous oxide alone is ca 5 times lower than the theoretical value of 0.5 (for HTP sample ca twice lower) and further decreases in the presence of CO over both LTP and HTP Fe/FER. The addition of NO decreases the O_2/N_2 value in a less pronounced way than that of CO (see figure 2b).

The integrated values of TPD curves for O_2, NO and NO_2 are displayed in figure 2c after the decomposition of N_2O alone and after $NO + N_2O$ (in 0.1, 0.5 and 1 NO/N_2O ratios). It follows from figure 2c that the addition of NO decreases the release of dioxygen and increases that of nitric oxide and nitrogen dioxide. With increasing amount of added nitric oxide its fraction in the TPD products increases and that of nitrogen dioxide keeps almost constant. The effect of the HTP treatment is manifested in a slightly higher fraction of desorbed NO and especially in the shift of the NO desorption temperature to a higher value. This is even more pronounced after the N_2O decomposition at higher temperature (280 °C) as is shown below in figure 6. Practically no surface species besides a very small amount of CO_2 were formed during the interaction of $CO + N_2O$. For that reason, the TPD products are not shown here.

During the N_2O decomposition at 300 °C the oxygen from decomposing N_2O does not almost remain captured on Fe/FER and the O_2/N_2 ratio is near to the theoretical value of 0.5 (figure 3). The effect of addition of NO or CO on the decomposition rate of nitrous oxide is negligible (see the insert in figure 3), however, both NO and CO are oxidized by the produced oxygen, so that the O_2/N_2 ratio in the gas phase during the decomposition of nitrous oxide decreases. Figure 3 displays the NO and CO effect over LTP Fe/FER; the HTP sample behaves almost in the same way, i.e., it does not exhibit any increased N_2O decomposition after the addition of NO or of CO, and NO_2 and CO_2 appear in the gas phase instead of O_2.

3.3. *TPD After the interaction of NO and N_2O alone and after the addition of NO in 0.05 and 0.1 NO/N_2O ratio*

The TPD curves after reactions at 250 °C are displayed in figure 4A: for NO alone (a), N_2O alone (b), $NO + N_2O$ (NO/N_2O ratio 0.05) – (c), and $NO + N_2O$ (NO/N_2O ratio 0.1) – (d). It can be seen that the NO_x surface species left after the interaction with NO alone appear in a lesser amount than when the sample was exposed to N_2O alone. Also the maximum of released dioxygen lays at higher temperature after the interaction of NO than of N_2O. The lowest fraction of added NO increases the amount of NO_2 and partially shifts the maximum of dioxygen release towards that of NO_2 (cf. sections b and c). The addition of NO (in 0.1 ratio) decreases the dioxygen release and shifts its maximum completely to that of NO_2. The integrated values of TPD curves for dioxygen, nitrogen dioxide and nitrogen monoxide are shown in figure 4B. It follows that the amount of desorbed NO_2 considerably increases with the addition of NO to N_2O.

3.4. *Effect of surface species left after the interaction at 250 °C of NO or N_2O on the following interaction of N_2O and NO, respectively*

Figure 5 illustrates the decomposition of N_2O (a), followed by the interaction with NO after short evacuation (b), and following TPD (c). The opposite order is shown in figure 5d–f. It can be seen that reactions of NO and N_2O after the samples with preceding N_2O and NO interactions are accelerated compared to the reactions of N_2O or NO alone, and that TPD (c) is similar to that after $NO + N_2O$ reaction (cf. figure 4A, (d)), but with higher fraction of desorbed NO, while TPD (f) corresponds rather with that in figure 4A, (c), but with higher fraction of NO_2.

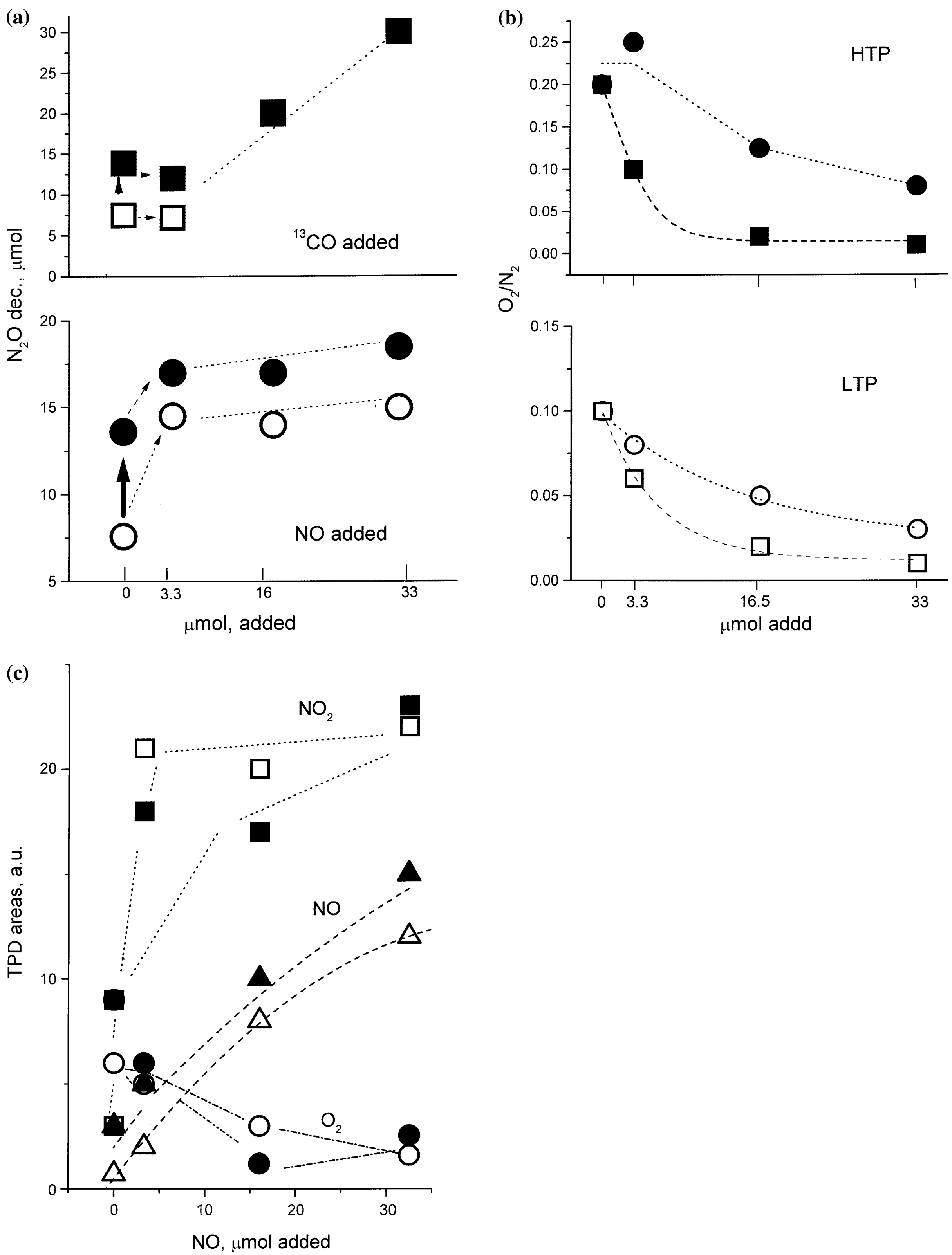

Figure 2. Effect of NO and ^{13}CO on the decomposition of N_2O at 250 °C over LTP and HTP Fe/FER, 70 mg of Fe/FER, decomposition after 80 min (a) decomposition of 33 μmol of N_2O. Bottom: $N_2O + NO$ (0–33 μmol), open circles – over LTP Fe/FER, solid circles – over HTP Fe/FER. Top: the same for $N_2O + {}^{13}CO$, open and solid squares for LTP and HTP Fe/FER, respectively. (b) O_2/N_2 ratios during the N_2O and $N_2O + NO$ or CO decomposition. Bottom: open circles and squares for $N_2O + NO$ and $N_2O + CO$, respectively, over LTP Fe/FER. Top: the same over HTP Fe/FER, solid symbols. (c) TPD areas for NO_2, NO and O_2 after the decomposition of N_2O and $N_2O + NO$, open and solid symbols for LTP and HTP Fe/FER, respectively.

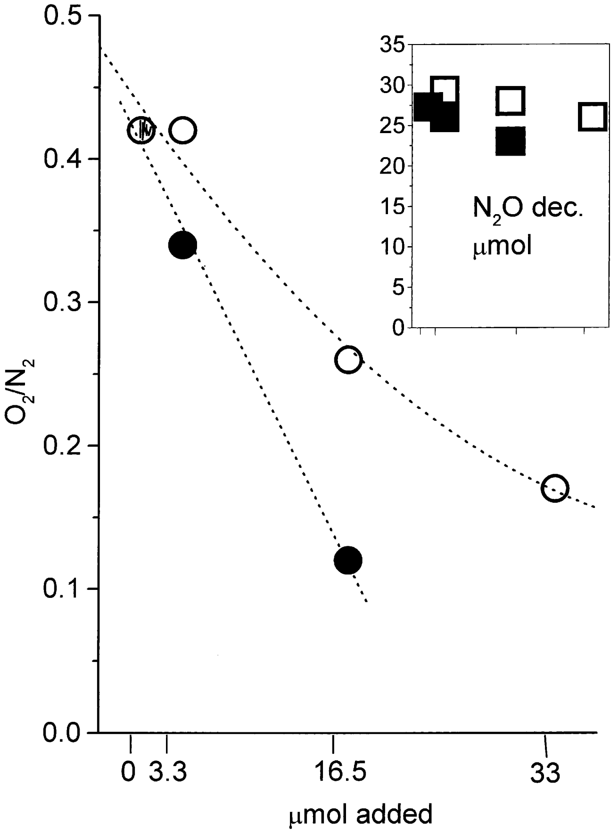

Figure 3. Effect of CO and NO addition on the decomposition of N_2O at 300 °C, 70 mg of Fe/FER, decomposition of 33 μmol N_2O after 50 min O_2/N_2 ratios: N_2O and $N_2O + NO$ (open circles) and $N_2O + CO$ (solid circles), insert: N_2O decomposed, open and solid squares for added NO and CO, respectively.

3.5. $^{15}N_2{}^{18}O$ Decomposition over LTP and HTP Fe/FER

The decomposition of ^{18}O labelled nitrous oxide was carried out at 280 °C, when only a smaller part of oxygen remains captured in the zeolite. This is displayed in figure 6, in the top part over LTP Fe/FER, in the bottom part over the HTP sample. Figure 6a shows the decomposition of $^{15}N_2^{18}O$ and ^{18}O concentration in the evolved dioxygen, figure 6b gives the isotopic dioxygen species released during the decomposition of the labelled nitrous oxide, figure 6c – the TPD curves of evolved dioxygen isotope species, and figure 6d – the TPD of NO_x compounds. It can be seen that the N_2O decomposition is much more rapid over the HTP Fe/FER, but in spite of it the ^{18}O concentration in released dioxygen is higher over the latter sample than over the LTP sample. In line with this, the fraction of molecular $^{18}O_2$ is higher over the HTP sample than over the LTP Fe/FER. The amount of zeolite framework oxygens participating in the decomposition of nitrous oxide is after 80 min almost the same over both samples (Ozeol/Fe≅1.4, for details see the preceding paper [14]); this means that in the same time interval a lesser number of zeolite oxygens participates in the reaction over the HTP sample. The ^{18}O content in the TPD products is lower than in dioxygen formed directly during the decomposition of $^{15}N_2^{18}O$ due to the additional ^{18}O exchange of surface species at higher temperatures. However, there is a striking difference in the ratio of released NO and NO_2 between both pretreatments (right-hand side of figure 6): the HTP sample releases much more NO than NO_2 compared to the LTP sample and the NO desorption occurs at higher temperature. Simultaneously, the release of dioxygen isotope species is also shifted to higher temperature.

4. Discussion

4.1. Mono-nitro surface species formed during the decomposition of nitrous oxide alone

NO_2 and NO were evidenced in the gas products during TPD after decomposition of nitrous oxide (figures 1 and 4A(b)). It is well known that N_2O yields over Fe-zeolites very active oxygens (α oxygen according to Panov *et al.* e.g. [15]). Therefore, the active oxygens can be assumed to react with nitrous oxide:

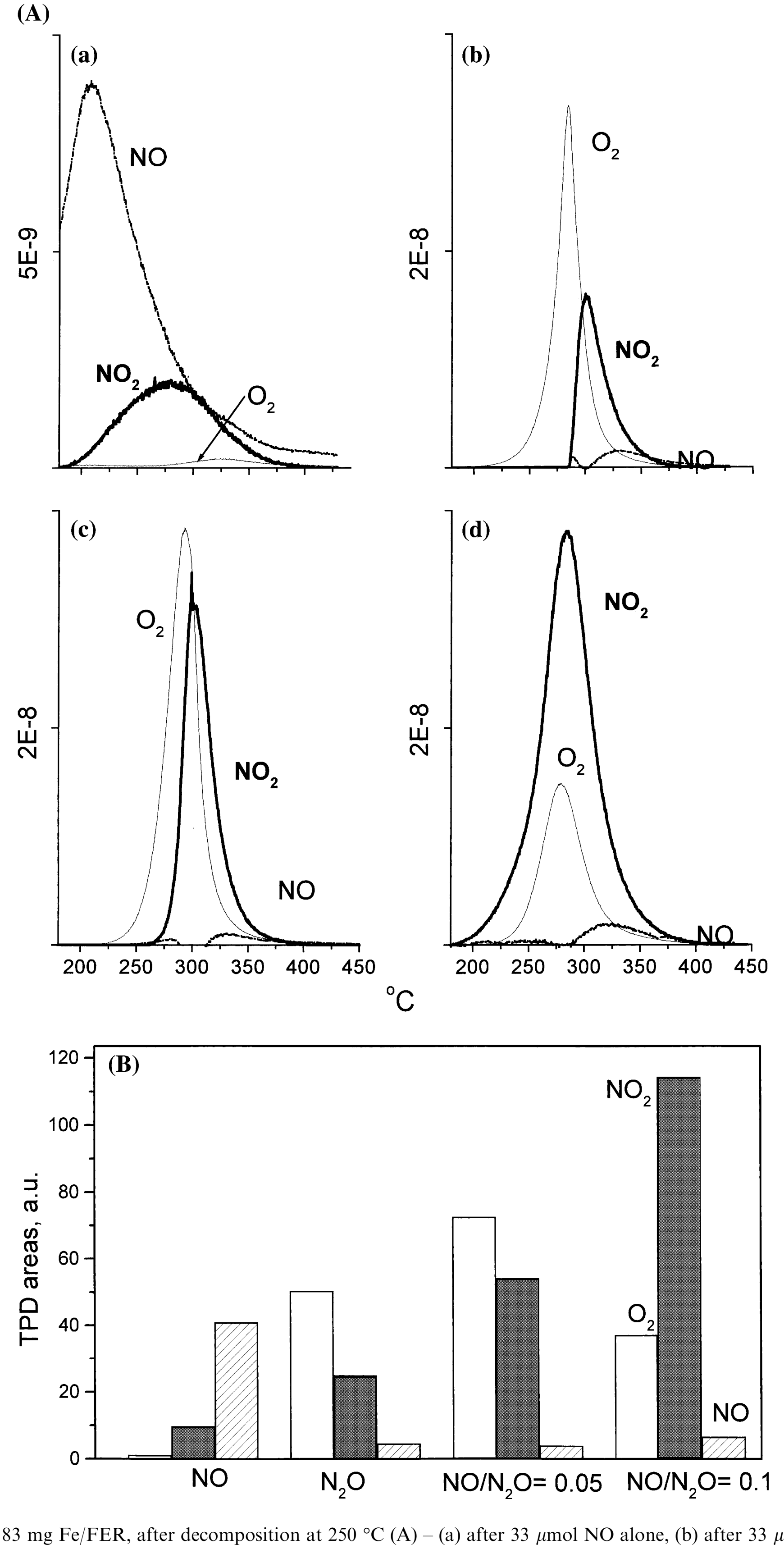

Figure 4. TPD curves, 83 mg Fe/FER, after decomposition at 250 °C (A) – (a) after 33 μmol NO alone, (b) after 33 μmol N_2O alone, (c) after NO/N_2O ratio 0.05, (d) after NO/N_2O ratio 0.1. (B) TPD areas, open columns for O_2, solid for NO_2 and striped for NO.

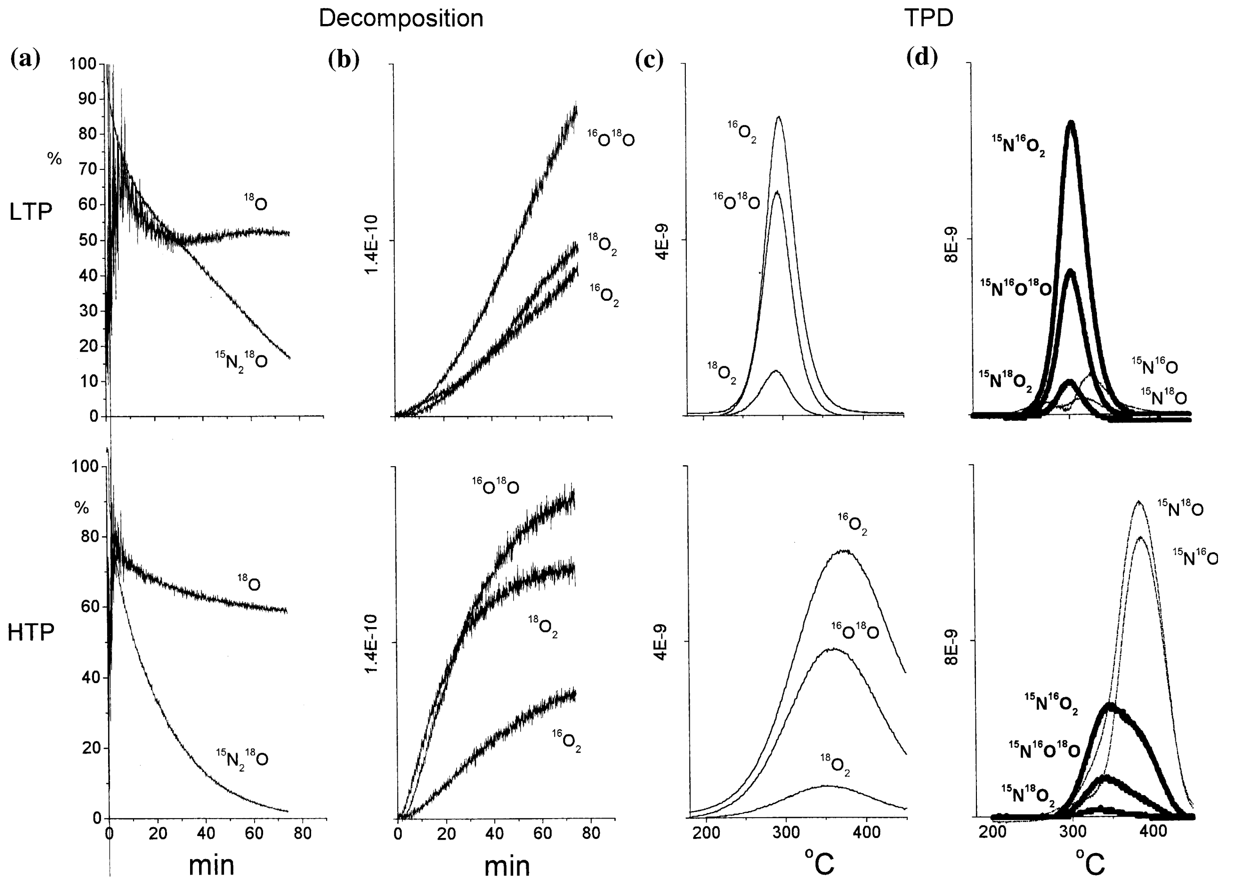

Figure 5. Effect of surface species left by NO or N_2O interaction on N_2O and NO reaction, and following TPD 147 mg of Fe/FER, 250 °C, NO or N_2O 33 μmol (a) N_2O decomposition, (b) following NO reaction, (c) final TPD, (d) NO interaction, (e) following N_2O decomposition, (f) final TPD.

$$N_2O + O \rightarrow 2NO, \qquad (1)$$

$$NO + O \rightarrow NO_2. \qquad (2)$$

The oxidation of N_2O to NO by oxygen is known to proceed in the stratosphere or at high temperature [16]. Recently, Turek [12] has also reported a side reaction forming nitric oxide from nitrous oxide over Cu–ZSM-5: $2N_2O \rightarrow 2NO + N_2$; simultaneously, he evoked a similar Musgrave's scheme for the homogeneous N_2O decomposition.

4.2. *Effect of added NO and CO on the N_2O decomposition and on the surface species*

The same compounds as after the decomposition of N_2O alone (i.e., NO and NO_2) appear after the reaction of $N_2O + NO$ at 250 °C in the TPD products (figures 2c and 4A and B). As the addition of NO substantially increases the extent of nitrous oxide decomposition while the desorbed surface species are qualitatively the same as after the decomposition of N_2O alone, the effect of the amount of added NO was checked and compared with the addition of CO which does not form any NO_x surface species. It follows from figure 4A, (c) that added NO in 0.05 ratio to N_2O (converges the maxima of dioxygen and nitrogen dioxide, and that 0.1 NO/N_2O ratio results in the same temperature of both maxima with NO_2 predominating over O_2. The latter NO/N_2O ratio substantially increases the extent of N_2O decomposition, (dotted arrow in the bottom of figure 2a) while the same amount of added CO does not almost affect the decomposition of nitrous oxide (top part of figure 2a). It can be therefore assumed that the role of both reactants is basically different: no surface NO_x species are created in the presence of CO; carbon monoxide only removes the captured oxygens and this removal is not sufficient for low CO amount. On the other hand, the same fraction of NO results in the substantial increase of N_2O decomposition which supports the Sang's assumption [8] of the change of the mechanism – presumably to a nitrite–nitrate one, or to a co-catalytic function of NO assumed by Kögel *et al.* [17]. However, this should concern only a relatively small part of NO_x species, as the increasing amount of NO increases the N_2O decomposition only slightly (figure 2a, bottom). The occupation of active sites by a larger amount of NO_x species can explain this behaviour under our experimental conditions, but a low dependency of N_2O

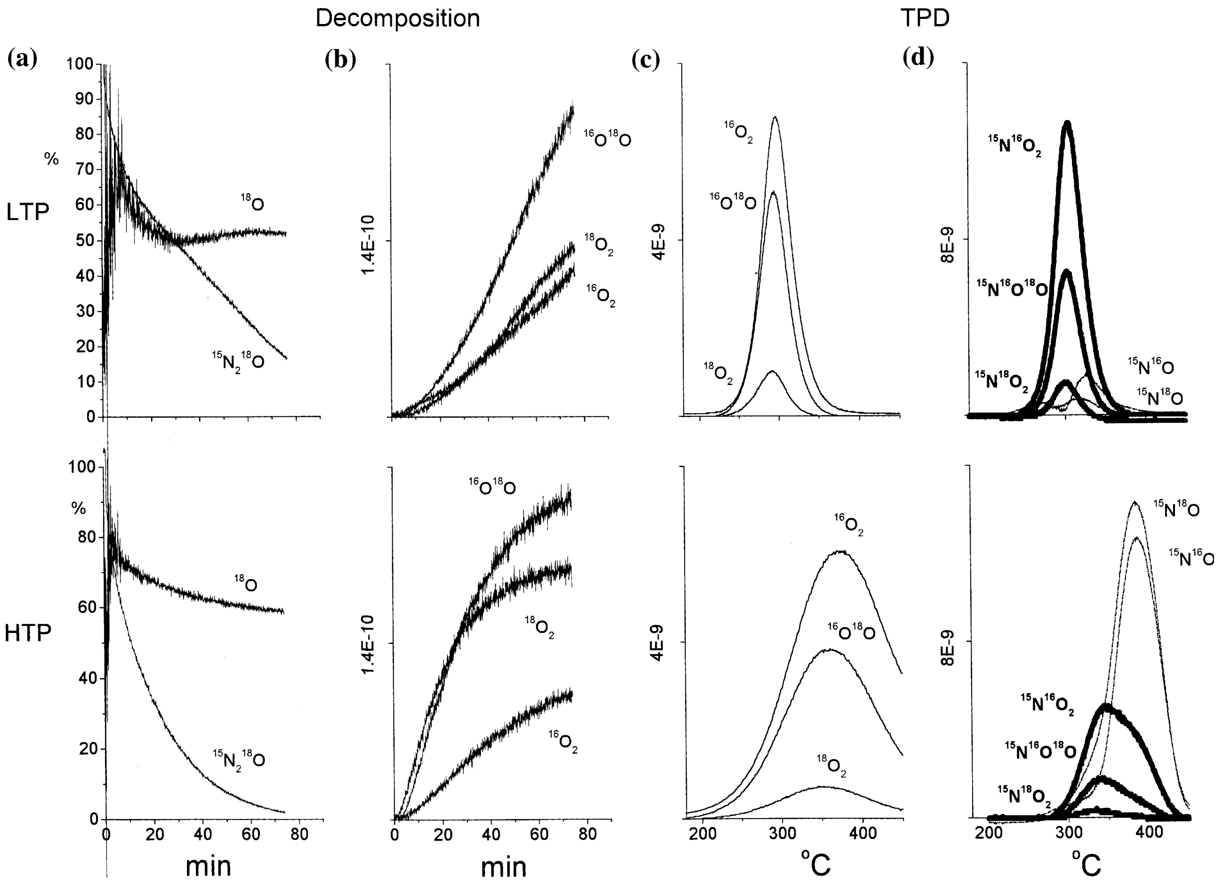

Figure 6. Comparison of $^{15}N_2{}^{18}O$ decomposition at 280 °C over LTP and HTP treated Fe/FER, 91.6 mg Fe/FER, decomposition of 16 μmol $^{15}N_2{}^{18}O$, bottom for HTP Fe/FR, top for LTP Fe/FER, (a) decomposition of $^{15}N_2{}^{18}O$ and ^{18}O concentration in evolved dioxygen (%), (b) individual isotopic dioxygen species released during the decomposition, (c) TPD – dioxygen isotopic species, (d) TPD – NO_2 and NO isotopic species.

decomposition on the addition of higher NO amount was also found in flow experiments [18]. The increasing fractions of CO (figure 2a, top part) almost linearly increase the extent of nitrous oxide decomposition, clearly by removing oxygen atoms from the active sites.

Under the experimental conditions employed, the enhancing (though presumably different) effect of both reactants on the decomposition of N_2O is pronounced at the decomposition temperature during which some of the oxygen produced remain captured in the zeolite. At the temperature at and above 280 °C the removal of oxygen atoms by "self-combination" predominates over their removal with the aid of CO or NO, so that the acceleration of the N_2O decomposition by NO or by CO is not expressed (see the insert in figure 3).[1] However, even in this case the active oxygen participates in the oxidation of CO, as the $CO+O_2$ reaction does not occur in the absence of nitrous oxide at this temperature over Fe/FER.

4.3. $^{15}N_2{}^{18}O$ *Alone at 280 °C – effect of the temperature pretreament of Fe/FER*

The employment of ^{18}O labelled nitrous oxide enables to recognize the participation of the zeolite oxygens in the decomposition route of N_2O [14]. The relationship to the above experiments can be find if comparing the isotopic composition of the released dioxygen: figure 6 shows in line with preceding experiments that the HTP sample exhibits considerably higher decomposition activity than the LTP Fe/FER, but from the very beginning of the decomposition the fraction of $^{18}O_2$ is much higher over HTP Fe/FER than over the LTP sample (HTP versus LTP treatments are reported in more detail in ref. [19]). This can be explained either by a lesser mobility of zeolite oxygens after the HTP treatment, similarly as has been suggested by Roy *et al.* [20] for the decomposition of N_2O over Fe/MFI, or, which seems to us as more probable, by an easier desorption of dioxygen from the sample pretreated at high temperature. This can be directly related to defects created by the dehydroxylation which may promote the association of oxygens from decomposing nitrous oxide. The other alternative can be a higher capacity of the HTP sample for the adsorbed NO species produced by the

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[1] Note: The decrease of the beneficial effect of NO with increasing temperature occurs at much higher temperature under flow conditions [18] when the "self-combination of oxygen atoms" is slower than their reaction with NO.

decomposition of nitrous oxide. The enhancing role of NO and/or CO on the decomposition of nitrous oxide is therefore less pronounced. The higher fraction of NO from the HTP Fe/FER in the desorbed products can be due to a lower concentration of active oxygens, so that the reaction sub (1) predominates over that sub (2). Nevertheless, the evolution of dioxygen at higher temperature (close to peakening nitric oxides) from HTP Fe/FER than from the LTP sample points probably to more strongly held NO_2 species decomposing to NO and O_2. The effect of HTP pretreatment on the composition of TPD products is much less pronounced after the decomposition of nitrous oxide at temperatures below 280 °C.

5. Conclusions

N–N bond in nitrous oxide during its decomposition to nitrogen and oxygen does not dissociate; however, "mono-nitro" NO_x surface species are formed presumably by the reaction of N_2O with active oxygen atoms captured on the zeolite.

The role of NO and CO in the acceleration of the decomposition of nitrous oxide is basically different. N_2O below 280 °C is decomposed to dinitrogen while the majority of oxygen atoms remains on the zeolite, occupies the active sites active and limits further decomposition of N_2O. Carbon monoxide removes the captured oxygens forming CO_2 which is released into the gas phase and regenerates the active sites. The increasing CO/N_2O ratio proportionally increases the nitrous oxide decomposition. On the other hand, NO in low NO/N_2O ratios reacts with captured oxygens to active NO_x species through which the enhanced N_2O decomposition proceeds; the amount of these new active sites is limited and higher NO/N_2O ratios do no longer contribute to the acceleration of the N_2O decomposition.

Function of these two accelerating reactants strongly depends on temperature of the N_2O decomposition. The captured oxygens can recombinate and desorb at higher temperatures in the absence of NO or CO and therefore the potential of these two reactants for the N_2O decomposition decreases. Under these conditions, both NO and CO react with active oxygen to NO_2 and CO_2, but the amount of N_2O decomposed does not increase.

The dehydroxylation of Fe/FER by HTP treatment leads to formation of defects which accelerate the decomposition of nitrous oxide: either (i) the recombination of two surface oxygens originating from N_2O is facilitated by these defects, or (ii) higher concentration of NO formed and kept at the surface species catalyzes further the decomposition. The enhancing effect of CO and/or NO is therefore less pronounced on the HTP samples.

It should be stressed that the above conclusions concern zeolites with low content of iron located predominantly in cationic positions.

Acknowledgments

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