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The surface chemistry of vapor phase lubricants: tricresylphosphate on Ni(1 0 0)

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Abstract

Tricresylphosphate (TCP) is known to serve as an excellent high temperature vapor phase lubricant with some metals such as Fe but not with others such as Ni. The surface chemistry of *m*-TCP has been studied on clean and phosphorous covered Ni(1 0 0) surfaces in order to understand the differences between its reactivity on Fe and Ni. Our results show that upon heating to 800 K *m*-TCP decomposes on the clean Ni(1 0 0) surface to deposit carbon and phosphorous with the evolution of H_2, CO, benzene, and toluene into the gas phase. During further heating to 1000 K, all the carbon on the surface dissolves into the Ni bulk leaving only phosphorous. The adsorption and heating of *m*-TCP on the phosphorous-modified Ni surface does not result in significant decomposition. The clean Ni substrate is able to activate TCP decomposition in much the same way that it decomposes on the clean Fe surface. The lack of significant differences in the chemistry of TCP on the clean metal surfaces cannot explain the fact that TCP vapor will form thick lubricating films on Fe but not on Ni.

Keywords: Vapour phase lubrication; Tricresylphosphate; Ni surface; Tribology

1. Introduction

Vapor phase lubrication (VPL) is a method proposed for use in high temperature applications where traditional liquid lubrication is not possible [1–3]. In VPL, the lubricant is delivered through the vapor phase to the hot surfaces of mechanical components where it reacts to deposit a solid lubricating film. Organophosphates such as tertbutylphenylphosphate (TBPP, $(C_4H_9C_6H_4O)_3P{=}O$) and tricresylphosphate (TCP, $(CH_3C_6H_4O)_3P{=}O$) have been studied and tested extensively as vapor phase lubricants. VPL using TBPP as the lubricant was successfully demonstrated in recent tests with a gas turbine engine [4,5].

VPL has been studied using many materials as substrates. VPL works readily with many metallic substrates, because most are active catalysts for the thermal decomposition of organophosphate lubricants. This results in the deposition of thick (~1 μm) films of a solid lubricant that are thought to consist of graphitic carbon bound in a matrix of polyphosphate glass [6]. Interestingly, it has been reported that at substrate temperatures of 970 K TCP will form lubricious films on Fe and Cu but not on Ni [7]. On the Ni surface, the film deposition rate was significantly lower than on either the Cu or the Fe surfaces. The explanation for this has been that Fe and Cu play catalytic roles in decomposing TCP to form lubricating films while Ni does not. On the other hand, it is widely known that unsaturated hydrocarbon compounds decompose quite readily on Ni surfaces. Ni surfaces show reactivity towards adsorbed aromatic hydrocarbons such as benzene, phenol and toluene in the sense that during heating they completely decompose to deposit carbon on the surface [8–10]. Ni will also induce the thermal decomposition of adsorbed olefins and alkynes such as C_2H_2 and C_2H_4 [11,12]. In previous studies using trimethylphosphite (TMP, $(CH_3O)_3P$) as a model for TCP, it was found that the surface chemistry of trimethylphosphite was quite similar on both Cu and Ni [13–15]. This leaves the question of the origin of the differences in VPL of Fe, Cu and Ni surfaces unanswered. The goal of this paper is to examine the surface

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chemistry of TCP on Ni(1 0 0) to try to address this question.

In our previous related work, we have studied the surface chemistry of *m*-TCP on the clean surface of an Fe foil. During heating to 800 K, *m*-TCP completely decomposed depositing carbon and phosphorous on the Fe surface and releasing H_2, CO, toluene, and *m*-cresol into the gas phase. Fe is clearly a reactive surface for TCP decomposition [16]. Additional work showed that the three isomers of TCP (*ortho*-, *meta*-, *para*-TCP) all have similar surface chemistry on Fe [17].

In this work, we studied the thermal reaction of *m*-TCP on Ni(1 0 0) and phosphorous modified Ni(1 0 0). The results show that the surface chemistry of *m*-TCP on clean Ni is not significantly different from that on Fe and thus that clean Ni is active for *m*-TCP decomposition. On the modified Ni surface, adsorbed phosphorous atoms inhibit the dissociation of adsorbed hydrocarbons, resulting in the molecular desorption of a greater fraction of the adsorbed *m*-TCP than was observed on the clean Ni(1 0 0) surface.

2. Experimental

All experiments were conducted in an ultra-high vacuum (UHV) chamber with a base pressure of 3×10^{-10} Torr. The chamber was equipped with an Ar^+ ion sputter gun, a cylindrical mirror analyzer for Auger electron spectroscopy (AES), a quadrupole mass spectrometer (QMS) for temperature programmed reaction spectroscopy (TPRS), a low energy electron diffraction (LEED) optics for surface structure determination, and a high molecular weight doser used to introduce the *m*-TCP vapor into the chamber. The Ni(1 0 0) surface was cleaned by several cycles of Ar^+ ion sputtering and annealing to 800 K. This procedure produced an atomically clean Ni(1 0 0) surface, as verified by AES and LEED. The Ni(1 0 0) sample could be cooled to 100 K and heated resistively to 1200 K.

During TPRS experiments, the Ni(1 0 0) sample was initially cooled to 180 K and positioned in front of the gas dosing tubes prior to adsorption of the molecule of interest. Leak valves were used to introduce high vapor pressure compounds such as 3-iodotoluene and *m*-cresol into the chamber. In order to adsorb *m*-TCP, which has an extremely low vapor pressure, a high molecular weight doser was used. This doser has a heated glass sample vial at the end of a long collimating tube. The *m*-TCP was heated to 410 K in the glass vial of the doser to raise its vapor pressure to the point that its flux from the end of the dosing tube was sufficient to adsorb a monolayer during exposure times of a few minutes. The dosing tube was moved into the chamber to position the end of the tube approximately 5 mm from the Ni(1 0 0) surface. Under these conditions, *m*-TCP vapor is adsorbed onto the Fe surface without causing undue contamination of the UHV chamber. During exposure of the surface to vapors, the chamber pressure was kept at below 10^{-8} Torr. After the Ni(1 0 0) surface was exposed to *m*-TCP for a given period of time, it was positioned in front of the QMS. Once the chamber pressure dropped back to 3×10^{-10} Torr, the sample was heated at a rate of 2 K/s to perform TPRS. During heating, the QMS monitored the signals at several m/q ratios simultaneously in order to detect molecules of interest desorbing from the surface. During the TPRS experiment, the chamber pressure did not exceed 10^{-8} Torr.

The Ni(1 0 0) sample was obtained from Monocrystals Co. The *m*-TCP ($(CH_3C_6H_4O)_3P{=}O$, 97%) was purchased from Fisher Scientific. The purity of the *m*-TCP was verified by its mass spectrum.

3. Results

3.1. Surface chemistry of m-TCP on clean Ni(1 0 0)

3.1.1. m-TCP decomposition on clean Ni(1 0 0)

TPRS has been used to study the decomposition kinetics of *m*-TCP on the Ni(1 0 0) surface and to identify reaction products appearing in the gas phase. Fig. 1 shows the TPR spectra of *m*-TCP obtained from clean Ni(1 0 0). The *m*-TCP was adsorbed on the Ni(1 0 0) surface at 180 K by exposing it to *m*-TCP vapor from the doser for 1 min. This was sufficient to produce a multilayer film of adsorbed *m*-TCP. During heating at 2 K/s, a number of desorption features were observed for each of the m/q ratios monitored. The peak at 220 K is assigned to multilayer desorption of *m*-TCP because its intensity grows indefinitely with increasing *m*-TCP exposures. In addition, this assignment of the peak at 220 K is supported by the observation of the fragment $m/q = 95$ (PO_4^+) at 220 K. At higher temperatures, H_2, CO, benzene, and toluene are observed desorbing from the surface. H_2 desorbs over a broad temperature range of 260–700 K with peaks at 300, 380, and 550 K. The first two peaks appear to be rate-limited by the intrinsic desorption kinetics of H_2 since it is known to desorb from Ni(1 0 0) at 330 K [18]. H_2 desorption at 550 K is rate-limited by the cleavage of C–H bonds in C_xH_y fragments that are generated during the decomposition of *m*-TCP. This spectrum is similar to that observed during *m*-TCP decomposition on Fe foil. The CO desorption peaks are located at 340 and 630 K. The latter can be attributed to CO production by recombination of adsorbed carbon and oxygen. The recombination of carbon and oxygen on Ni surfaces is known to occur over a wide temperature range around 600 K depending on the sur-

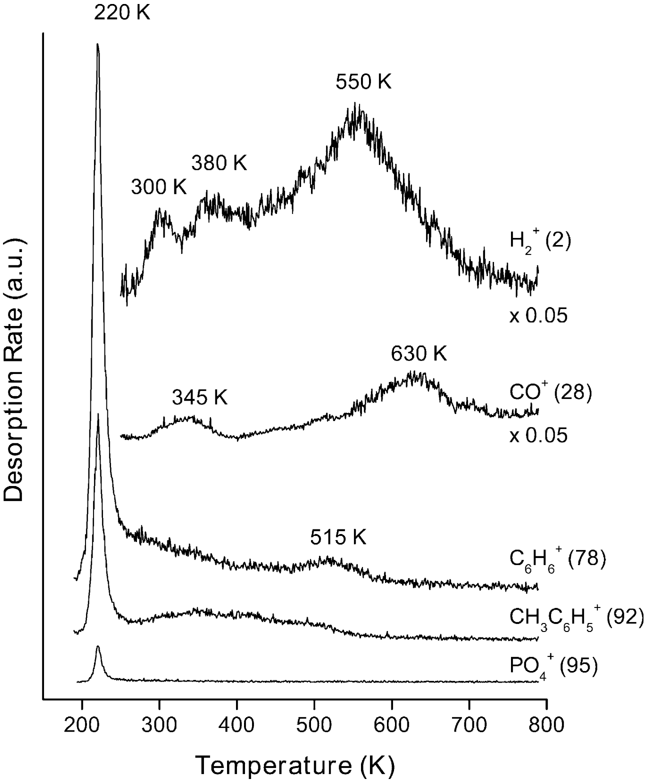

Fig. 1. TPR spectra of *m*-TCP adsorbed onto Ni(1 0 0) at 180 K. The spectra were generated by monitoring the fragments with $m/q = 2$ (H_2^+), 28 (CO^+), 78 ($C_6H_6^+$), 92 ($CH_3C_6H_5^+$) and 95 (PO_4^+). The heating rate was $\beta = 2$ K/s.

face conditions [9,19,20]. During trimethylphosphite decomposition on Ni(1 1 1), carbon and oxygen recombination to form CO has been observed at 630 K [13]. The CO desorption temperature of 340 K is lower than that of 450 K observed for molecular desorption from the clean Ni(1 1 1) surface [21], suggesting that the process is desorption rate-limited but that the desorption temperature is lowered by the presence of other species on the surface. Dickens and Stair showed that methyl radical reaction with the oxidized Ni(1 0 0) surface results in CO desorption at ~330 and ~630 K, and attributed the low temperature CO desorption peak to the decomposition of a surface alkoxy species [22]. CO desorbs initially at 420 K from phenol decomposition on Ni(1 1 0) [9]. As will be seen later, CO desorbs at 395 K during the TPRS of *m*-cresol on Ni(1 0 0). These CO desorption temperatures from phenol and *m*-cresol decomposition are close to the CO desorption temperature of 450 K from clean Ni(1 0 0). Given that the major difference between *m*-TCP and *m*-cresol or phenol is that *m*-TCP contains phosphorous, it is possible that adsorbed phosphorous lowers the temperature of CO desorption. Benzene desorbs over a broad temperature range around 300 K and in a peak at 520 K. This pattern is similar to that of benzene desorption at ~225 and 445 K observed during iodobenzene decomposition on Ni(1 0 0) [10]. Toluene desorbs in the temperature range from 260 to 580 K. These temperatures are similar to those observed for toluene desorption during *m*-TCP decomposition on the Fe surface [16,23]. Unlike the results obtained on the Fe surface, little *m*-cresol desorption was observed.

3.1.2. *Ni(1 0 0) surface composition after m-TCP decomposition*

The thermal decomposition of *m*-TCP on the Ni(1 0 0) surface results in desorption of reaction products and in the deposition of carbon and phosphorous onto the surface. Fig. 2 shows Auger spectra of the clean Ni(1 0 0) surface and the same surface after adsorption of a multilayer of *m*-TCP and subsequent heating to 800 K. The Auger spectrum of the clean Ni(1 0 0) surface exhibits only Ni peaks at 65, 107 eV and three peaks in the energy range 650–870 eV. The Auger spectrum of the surface following *m*-TCP decomposition reveals the presence of adsorbed phosphorous at 120 eV and carbon at 273 eV in addition to the peaks attributable to Ni. No adsorbed oxygen is observed in the Auger spectrum. Oxygen is removed

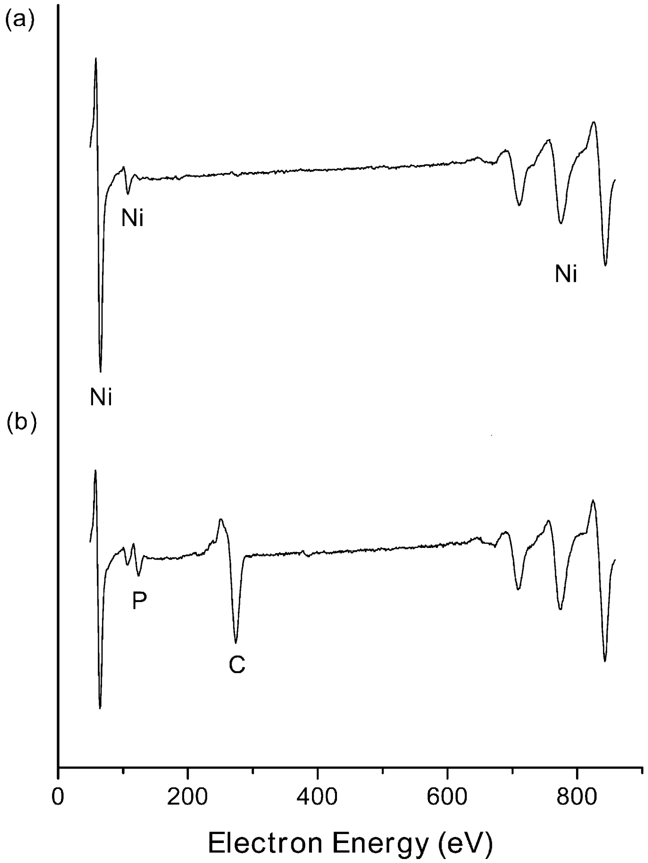

Fig. 2. Auger spectra of (a) clean Ni(1 0 0); and (b) following adsorption and thermal decomposition of *m*-TCP by heating to 800 K. The decomposition of *m*-TCP results in the deposition of phosphorous (120 eV) and carbon (273 eV). Oxygen is not observed since it desorbs as CO via recombination with adsorbed carbon.

from the surface by recombination with carbon at 630 K to form CO which desorbs as shown in Fig. 1. The shape of the lower energy region of the carbon peak suggests the formation of a layer of graphitic carbon on the surface [24]. It must be noted that the adsorbed carbon starts to diffuse into the Ni bulk after heating past ~800 K and has completely disappeared from the surface after heating to 1000 K. Previous studies have shown that carbon deposited on the Ni(1 1 0) surface by decomposition of phenol remains on the surface to temperatures of 700 K but then dissolves into the bulk during heating to 900 K [9,25,26]. The important point to be gleaned from the results shown in Figs. 1 and 2 is that *m*-TCP decomposes on the Ni(1 0 0) surface to deposit graphitic carbon and phosphorous.

3.1.3. Thermal decomposition of model compounds on Ni(1 0 0)

In order to understand the reaction mechanism of *m*-TCP, TPRS experiments have been performed using 3-iodotoluene and *m*-cresol on the Ni(1 0 0) surface. The most likely initial steps in the decomposition of *m*-TCP are the cleavage of the P–O bond to produce an adsorbed *m*-cresoxy intermediate or the cleavage of a C–O bond to produce an adsorbed tolyl group. The initial steps in the decomposition of *m*-cresol and 3-iodotoluene are expected to be cleavage of the O–H and C–I bonds, respectively, to produce adsorbed *m*-cresoxy and adsorbed tolyl, respectively. The production of these decomposition intermediates is believed to occur on the basis of the results of numerous previous studies showing that iodobenzene generates phenyl groups on Pd(1 1 1) [27], Pt(1 1 1) [28], Ag(1 1 1) [29], and Cu(1 1 1) [30] and phenol generates phenoxy groups on Ni(1 1 0) [9] and Ni(1 1 1) [31]. By creating *m*-tolyl and *m*-cresol groups on the Ni(1 0 0) surface and studying their subsequent thermal decomposition we can probe the mechanism of *m*-TCP decomposition.

TPR spectra obtained during heating of 3-iodotoluene adsorbed on the Ni(1 0 0) surface are shown in Fig. 3a. Molecular 3-iodotoluene desorbs from the multilayer film at 200 K. H_2 desorbs at 315 and 480 K. Previous studies have shown that adsorbed toluene multilayers desorb molecularly from the Ni(1 0 0) surface at 180 K and are accompanied by H_2 at 340 and 490 K [10]. Our TPR spectra of adsorbed 3-iodotoluene are quite similar to those obtained using toluene in the sense that they reveal the same products desorbing at the same temperatures.

TPR spectra obtained during heating of *m*-cresol adsorbed on the Ni(1 0 0) surface are shown in Fig. 3b. The *m*-cresol multilayer desorbs at 209 K which is close to the desorption temperature of 200 K observed for phenol multilayers on the Ni(1 1 0) surface [9]. H_2 evolves over the temperature range from 250–580 K with the peaks at 300, 385, 490, and 595 K. CO desorbs at 395, 505, and 595 K. These H_2 and CO desorption features are very similar to those observed during phenol decomposition on the Ni(1 1 0) surface [9]. Phenol decomposition yields H_2 desorption at 288, 405, 480, and ~570 K, and CO desorption at 420, 480, ~570, and 680 K.

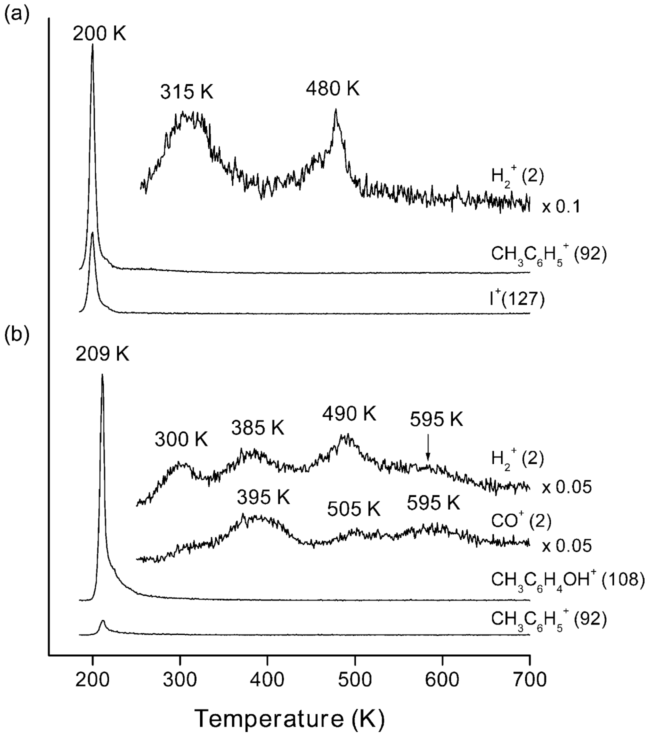

Fig. 3. TPR spectra of (a) 3-iodotoluene; and (b) *m*-cresol adsorbed onto Ni(1 0 0) at 180 K. The H_2 desorption profile from *m*-TCP is closer to that from *m*-cresol than that from 3-iodotoluene, suggesting that P–O bond scission is the initial step in the decomposition mechanism. The heating rate was $\beta = 2$ K/s.

The TPR spectra obtained from toluene and *m*-cresol on the Ni(1 0 0) surface are not identical to those of *m*-TCP. This is not surprising given that the decomposition of the *m*-TCP also results in the deposition of phosphorous onto the surface and that this may influence the subsequent decomposition mechanism. Nonetheless, comparison of the H_2 desorption spectra obtained during decomposition of 3-iodotoluene and *m*-cresol and *m*-TCP, suggests that the *m*-TCP chemistry resembles that of *m*-cresol more than that of 3-iodotoluene. This suggests that it is likely that *m*-TCP decomposes by initial cleavage of its P–O bonds to produce *m*-cresoxy surface intermediates.

3.2. Surface chemistry of m-TCP on P modified Ni(1 0 0)

To assess the effect of the presence of adsorbed phosphorous on the reactivity of the Ni(1 0 0) surface, TPRS of *m*-TCP was performed using a phosphorous modified Ni(1 0 0) surface. The phosphorous modified Ni(1 0 0) surface was produced by *m*-TCP adsorption followed by annealing to 1000 K. By 800 K, adsorbed oxygen was removed via carbon and oxygen atom recombination followed by CO desorption. At 800 K, the only surface species remaining were carbon and phosphorous. Upon further heating to 1000 K, most of the carbon atoms disappear from the surface by diffusion into the Ni bulk. We did not observe the desorption of any carbon containing products. The high temperature dissolution of carbon into Ni has been observed on the Ni(1 1 0) surface. Russell and coworkers have shown that the carbon left on the Ni(1 1 0) surface following decomposition of phenol dissolved into the bulk at temperatures above 700 K [9]. After heating *m*-TCP adsorbed on the Ni(1 0 0) surface to 1000 K, only phosphorous atoms are left on the surface.

The TPR spectra of *m*-TCP on phosphorous modified Ni(1 0 0) are presented in Fig. 4. As on the clean surface, the *m*-TCP multilayer desorption temperature is 220 K. The most distinct differences between the TPR spectra obtained on the clean and the phosphorous covered surfaces are seen in desorption peak shifts and peak area changes. The principal H_2 and CO desorption peaks shift from 550 and 630 K on the clean surface to 570 and 680 K, respectively, on the phosphorous modified surface. Furthermore, the areas under the H_2 and CO peaks decreased with respect to those obtained on the clean Ni(1 0 0) surface. This suggests that C–H bond scission in hydrocarbon fragments on the surface is inhibited by phosphorous. The amounts of desorbing benzene and toluene increased by comparison with those observed on the clean surface. This indicates that the adsorbed phosphorous atoms suppress the decomposition of aromatic ring fragments, resulting in increasing benzene and toluene desorption. Similar results were observed during trimethylphosphite decomposition on an Fe(1 1 0) surface modified by 0.18 ML of adsorbed phosphorous. The presence of phosphorous atoms on the Fe(1 1 0) surface inhibited the decomposition of trimethylphosphite, resulting in greatly increased amounts of formaldehyde production and a decrease in the amount of CO desorption [32].

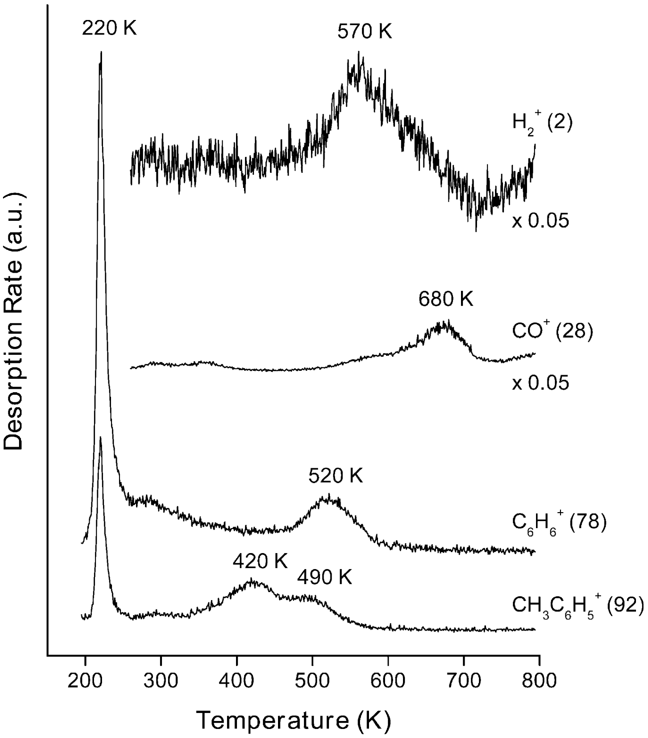

Fig. 4. TPR spectra of *m*-TCP on phosphorous covered Ni(1 0 0). Compared to Fig. 1, it is clear that H_2 and CO production decreased while benzene and toluene production increased. This indicates that adsorbed phosphorous inhibits the dissociation of the aryl rings on the surface.

The decrease in the amount of *m*-TCP decomposition on the Ni(1 0 0) surface as a result of the presence of adsorbed phosphorous is also revealed by Auger spectra taken before and after TPRS experiments on the phosphorous modified surface (Fig. 5). The spectrum obtained after the adsorption and decomposition of the *m*-TCP on the phosphorous covered surface reveals only a small increase in the amplitudes of the C and P peak. The amount of carbon deposition is negligible when compared to the amount deposited on the clean surface (Fig. 2). On the phosphorous covered surface, the majority of the adsorbed *m*-TCP desorbs rather than decomposing during heating, indicating that Ni loses its reactivity as a result of phosphorous poisoning. The implication of this result for VPL will be discussed in more detail in the next section.

4. Discussion

Our current understanding of the deposition of lubricant films from vapor phase arylphosphates such as TCP is that the deposition rates depend on parameters including: lubricant surface chemistry, the interdiffusion of substrate material and the lubricant film, and the effect of surface oxides on lubricant decomposition. In this work, we have probed the thermal chemistry of *m*-TCP on well-defined Ni(1 0 0) surfaces in order to understand why it is that TCP does not serve as a good vapor phase lubricant with Ni, whereas it does lubricate Fe and Cu surfaces. Our TPRS results show that the H_2 desorption profiles from *m*-TCP decomposition on the Ni(1 0 0) surface bear more resemblance to those observed during decomposition of *m*-cresol than those observed during decomposition of 3-iodotoluene. At this point, it is worth noting that our previous study of *m*-TCP thermal decomposition on an Fe foil resulted in TPR spectra that were almost identical to those obtained using *m*-cresol [16,17]. This suggests that the initial step in the TCP decomposition mechanism on both surfaces is cleavage of the P–O bond to produce an adsorbed cresoxy intermediate. In this work on the Ni(1 0 0) surface, the case for the formation of the cresoxy intermediate during the *m*-TCP reaction mechanism is not as clear as on the Fe surface.

m-TCP decomposes on the Ni(1 0 0) surface to deposit carbon and phosphorous which can serve as lubricating films at temperatures up to 800 K. At temperatures above 800 K, the carbon begins to diffuse into the Ni and by 1000 K only phosphorous remains on the Ni(1 0 0) surface. This observation may be related to the role of diffusion in VPL technology. If the presence of carbon on the surface is important for VPL, then diffusion into the bulk will limit the performance of VPL to temperatures below 1000 K. On the other hand, if Ni bulk atoms can diffuse to the surface, then diffusion is advantageous because it keeps exposing reactive sites to the TCP vapor. While interdiffusion of metal substrate and lubricant films does occur during VPL, it is not clear that this is the origin of the differences between the ability of TCP to vapor lubricate different metals. A recent study showed that interdiffusion of Fe and the lubricant film and interdiffusion of Cr and the lubricant film both occur during VPL on those metals. However, it has also been observed that TCP lubricates Fe but not Cr [33].

TPRS experiments of *m*-TCP on phosphorous covered Ni(1 0 0) showed that phosphorous does influence the decomposition of TCP. Fig. 6 presents the relative yields of *m*-TCP decomposition products as a function of phosphorous coverage. For H_2 and CO, the yields are plotted relative to the yields from the clean Ni(1 0 0) surface. For benzene and toluene, the yield was defined relative to the yields from surface with 70% phosphorous coverage. The phosphorous coverage was defined based on the Auger peak-to-peak intensity of the P peak at 120 eV and the Ni peak at 848 eV both scaled by their Auger elemental sensitivities (0.27 for P and 0.54 for Ni at a primary beam voltage of 3 keV [34]). It is clear that the H_2 and CO yields decrease while the benzene and toluene yields increase as the phosphorous coverage is increased. Adsorbed phosphorous deactivates the Ni(1 0 0) surface for *m*-TCP decomposition.

One thing that should be pointed out is that under the UHV conditions of this study, the thermal

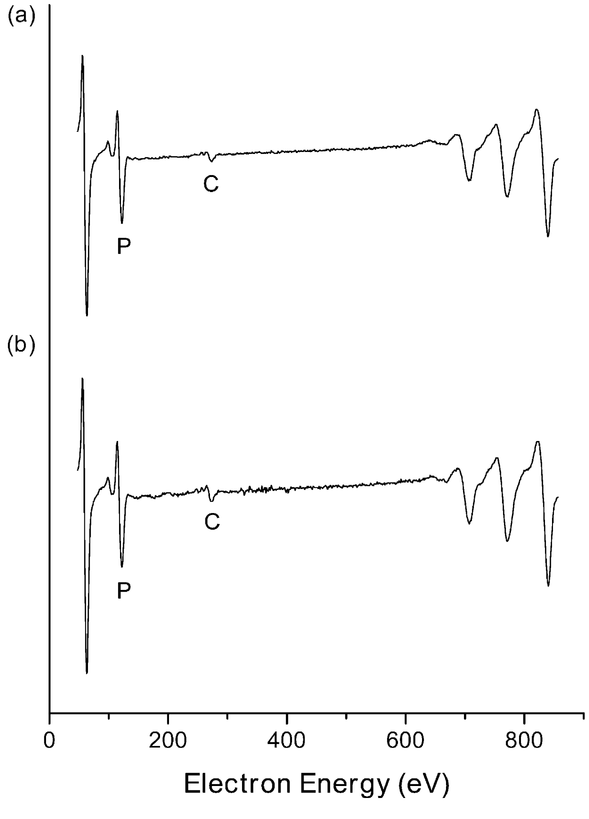

Fig. 5. Auger spectra of (a) P modified Ni(1 0 0); and (b) the surface after *m*-TCP decomposition by heating to 800 K. The phosphorous modified surface was produced by *m*-TCP adsorption at 180 K followed by annealing the surface to 1000 K.

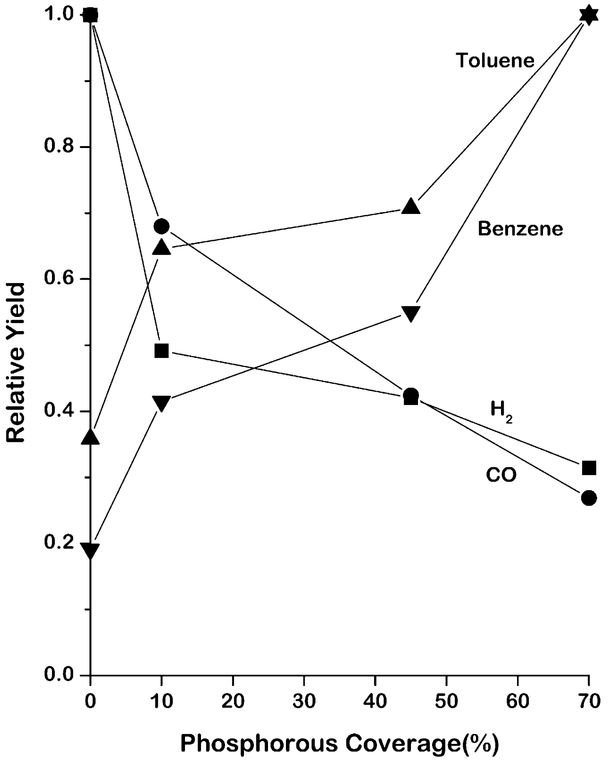

Fig. 6. Relative yields of *m*-TCP decomposition products as a function of phosphorous coverage on the Ni(1 0 0) surface. Phosphorous was deposited by adsorbing *m*-TCP and annealing to 1000 K prior to adsorbing *m*-TCP for the TDP measurement. The reactivity of Ni for *m*-TCP decomposition decreases with its phosphorous coverage.

decomposition of *m*-TCP on the Ni(1 0 0) surface does not deposit oxygen onto the Ni(1 0 0) surface. A recent study of VPL has shown that the lubricious films include oxygen that is present in the form of a polyphosphate glass which serves as a binder for the graphitic carbon [35]. This suggests that the overall reaction mechanism needed to produce the vapor lubrication films cannot rely solely on the oxygen present in the *m*-TCP and may require the presence of ambient oxygen. The fact that our observations have been made in the absence of oxygen must be considered in trying to understand their relevance to the surface chemistry that occurs under the condition of VPL.

5. Conclusions

m-TCP thermally decomposes on clean Ni(1 0 0) to deposit carbon and phosphorous onto the surface with the desorption of products such as H_2, CO, benzene and toluene. This indicates that Ni is reactive for TCP decomposition. The most likely path for *m*-TCP decomposition on the Ni(1 0 0) surface involves initial scission of P–O bonds to produce a *m*-cresoxy intermediate. This result suggests that the surface chemistry of *m*-TCP on the clean Ni surface is not directly related to its performance as a vapor phase lubricant with Ni.

The reactivity of Ni(1 0 0) for *m*-TCP decomposition decreases with phosphorous coverage. Substantially increased amounts of organic desorption products were observed on phosphorous modified Ni surface. Adsorbed phosphorous passivates the Ni surface by inhibiting the complete decomposition of *m*-TCP resulting in decreased carbon deposition.

Acknowledgements

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