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$Ru_3(CO)_{12}$ and $Mo(CO)_6$ Adsorbed on Ru(001) and Au/Ru: An Infrared Reflection-Absorption Study

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Abstract

We obtained IRAS and TDS data for $Ru_3(CO)_{12}$/Ru(001) and $Mo(CO)_6$/Au/Ru systems for metal carbonyl coverages between submonolayer and approximately 20 monolayers. We characterized the C-O stretching mode of both systems (4 cm^{-1} FWHM) and a deformation mode of $Mo(CO)_6$ at 608 cm^{-1} (1 cm^{-1} FWHM). Both IRAS and TDS data suggest adsorption and desorption of metal carbonyls as molecular species with a preferential orientation in the overlayers. The IR intensity of the C-O stretch per a C-O bond projected onto the surface normal is approximately twice (five times) larger for $Ru_3(CO)_{12}$ ($Mo(CO)_6$) at submonolayer coverages than for CO/Ru(001) at θ_{CO}=0.68.

1. INTRODUCTION

Transition metal carbonyls have been studied with a variety of experimental techniques [1] including vibrational (infrared, Raman) spectroscopies [2]. The close relationship between these compounds and CO adsorbed on various transition metal surfaces, by far the most studied systems in surface science, is obvious. Metal carbonyls are relevant for both fundamental understanding of substrate-adsorbate systems and for applications in catalysis and thin film technology. So, for example, experimentally determined vibrational and electronic parameters for $Rh_6(CO)_{16}$ [3] and $Ni(CO)_4$ [4] have been used in a theoretical treatment of CO adsorption on single crystal metal surfaces. As far as the applications are concerned, the use of transition metal carbonyls as precursors of heterogeneous catalysts (both mono- and bi-metallic) has been suggested [5] and later explored experimentally [6,7]. Also, the possibilities of thin metal film deposition on semiconductor and insulator substrates from adsorbed metal carbonyls have been studied [8,9].

Vibrational properties of CO overlayers adsorbed on single crystal metal surfaces have been extensively investigated by infrared reflection-absorption (IRAS) [10] and electron energy loss spectroscopies (EELS) [11]. The spectra have been analyzed using the concepts of chemical (static) and dynamic dipole-dipole coupling effects, the latter being very important for C-O vibrations due to a high value of the dynamic dipole moment of this mode [12]. Vibrational spectra of bulk molecular crystals (including solid state metal carbonyls) have been interpreted with the use of

static field and correlation field (dynamic) effects [13-15]. Although the approaches by solid state and surface spectroscopy groups are clearly closely related, there has not been much interaction between them. A vibrational study dealing with metal carbonyls of different symmetries interacting with well defined metal surfaces can bring these two groups closer together as well as answer at least some of the following questions:
- Are the metal carbonyls adsorbing as intact molecules?
- Do they form ordered overlayers?
- Is a CO-covered surface different from a clean surface for metal carbonyl adsorption?
- How important is the symmetry of the adsorbate?
- How do the metal carbonyl overlayers interact with photons and electrons of various energies?
- What is the relation between s- and p-polarized IR spectra for metal carbonyl multilayers?
- How do IRAS results compare with solid state IR and Raman spectra of these compounds?
- Do the metal carbonyls desorb as intact molecules?

We studied the interaction of $Ru_3(CO)_{12}$ [16] (D_{3h} point group) and $Mo(CO)_6$ (O_h point group) with Ru(001) and with Ru(001) covered with 2-3 monolayers of Au (Au/Ru). We obtained IRAS results for metal carbonyls at coverages between ≈0.2 and ≈20 monolayers at these surfaces as well as on CO-saturated Ru(001). In addition to IR absorption bands due mainly to C-O stretching motion observed for both studied metal carbonyls, we detected a deformation mode of $Mo(CO)_6$ at 608 cm^{-1}. Our IR results imply molecular adsorption of both carbonyls with a preferential orientation in the adsorbed overlayers. Thermal desorption spectra imply molecular desorption of both species. Evidence for ordering comes both from analysis of IR spectra based on the symmetry of vibrational modes observed and from narrow full widths of the absorption bands. So, for instance, absence of bands attributable to C-O stretches of equatorial CO's of adsorbed $Ru_3(CO)_{12}$ implies orientation of the Ru_3-plane of $Ru_3(CO)_{12}$ parallel to the surface [16]. The widths (FWHM) of the absorption bands were only ≈1 cm^{-1} for the deformation mode of $Mo(CO)_6$ and ≈4 cm^{-1} for the C-O stretching modes of both $Ru_3(CO)_{12}$ and $Mo(CO)_6$.

2. EXPERIMENTAL

The experiments were performed in a multilevel UHV chamber (base pressure $2x10^{-10}$ torr) equipped with a top-level 2 inch diameter IR cell, and CMA (cylindrical mirror analyzer) for AES (Auger electron spectroscopy), LEED (low energy electron diffraction), QMS (quadrupole mass spectrometer), ion sputter gun, metal carbonyl sources, and a Au source at lower levels.

The Ru(001) crystal (suppplied and polished on both sides by the Materials Science Center of Cornell University), a 1 mm thick disk of 12 mm diameter, was attached to a XYZ manipulator by spotwelding to two W wires of 0.5 mm diameter. The crystal can be cooled to ≈80 K by liquid nitrogen and resistively heated to 1560 K. The temperature is measured by a W5%Re/W26%Re thermocouple spotwelded to the top edge of the crystal. The crystal was cleaned in a standard way [17] by Ar-ion sputtering and oxygen adsorption-desorption cycling. The cleanliness and order of the surface were checked by AES and LEED, respectively. The Au source consisted of a ribbon

(≈1x5x0.2 mm) of ultrapure gold heated resistively through a Ta spiral. The Au deposition rate was 1 monolayer in ≈5 min.

$Ru_3(CO)_{12}$ (Strem Chemicals) was placed in an independently pumped glass vial separated from the main chamber by a gate valve. The vial can be resistively heated; it was kept at 322±1 K during $Ru_3(CO)_{12}$ dosing. Deposition rate of $Ru_3(CO)_{12}$ was 1 monolayer in ≈200 s; dosing was accomplished by opening the gate valve and allowing the metal carbonyl to travel through a Cu drift tube (20 cm long, 1.5 cm inner diameter) facing the Ru crystal.

$Mo(CO)_6$ (K & K Laboratories) was placed in an independently pumped glass test tube and kept at 273 K (an ice-water bath) during deposition. Dosing was performed through a glass capillary array doser facing the crystal. The deposition rate was dependent on the $Mo(CO)_6$ flow rate through the leak valve behind the doser; it varied between ≈15 s and ≈60 s per monolayer.

During the collection of vibrational data, the photon beam from an FTIR instrument (Mattson Cygnus 25) is focused on the Ru crystal in the IR cell with an off-axis parabolic mirror (8 inch focal length) through a KBr window. The average angle of incidence of the IR beam on the crystal is 85° from the surface normal. After reflecting off the crystal, the photon beam exits the IR cell through another KBr window and is focused by a KBr lens on a broad-band HgCdTe detector. The optical path outside UHV is purged with dry, CO_2-free air. Unpolarized IR radiation was used in the experiments discussed in this paper.

The TDS (thermal desorption spectroscopy) data were obtained with a differentially pumped QMS (UTI 100C) equipped with a stainless steel skimmer with a ≈3 mm diameter opening facing the crystal. The skimmer was biased at -70 V to avoid interaction of electrons originating in the QMS ionizer with the metal carbonyl overlayers.

The determination of $Ru_3(CO)_{12}$ coverage (θ_{RC}) and $Mo(CO)_6$ coverage (θ_{MC}) was based on the following assumptions: a) both metal carbonyls adsorb as intact molecules; b) one adsorbed molecule occupies ≈90 $Å^2$ and ≈50 $Å^2$ in case of $Ru_3(CO)_{12}$ and $Mo(CO)_6$, respectively; c) the total amount of CO originating from the adsorbed metal carbonyls is well represented by the (m/e=28) signal. The saturation coverage of CO/Ru(001) (θ_{CO}=0.68) [18] served as a reference.

3. RESULTS AND DISCUSSION

We studied the interaction of $Ru_3(CO)_{12}$ with clean Ru(001), Ru(001) saturated with CO, and Ru(001) covered by 2-3 monolayers of Au (Au/Ru) [16,19], and the interaction of $Mo(CO)_6$ with the same surfaces [19]. Here, we concentrate on the IRAS results of $Ru_3(CO)_{12}$/Ru(001) and $Mo(CO)_6$/Au/Ru and only briefly deal with the other systems. We also present TDS results that give complementary information to the IRAS data.

Figure 1 shows IR spectra of the C-O stretch region of $Ru_3(CO)_{12}$/Ru(001) for θ_{RC}≈0.2-7.7. The spectra shown are considerably simpler (in particular, no absorption bands below 2020 cm^{-1}) and the bands are at least 3x narrower than IR spectra of $Ru_3(CO)_{12}$ on different supports [20-22], in solution [23], and in the solid state [23,24]. Our interpretation of the IRAS results is based on a normal mode analysis of $Ru_3(CO)_{12}$ [23] and IR reflection spectra of monocrystalline $Ru_3(CO)_{12}$ [24].

The bands in the 2030 and 2080 cm^{-1} regions, ν_{35} (A_2'' symmetry for D_{3h}) and ν_{16} (E')(following notation of [24]), respectively, are dominated by

stretches of axial CO's of $Ru_3(CO)_{12}$. The weak broad band around 2060 cm^{-1} represents CO from the background adsorbed directly on the Ru(001) surface. The absorption feature at 2124 cm^{-1} seen at higher θ_{RC} is assigned to the ν_1 (A_1') mode of axial CO's. ν_1, IR forbidden for an isolated molecule, was also observed in IR spectra of $Ru_3(CO)_{12}$ in CsI disks [24]; its infrared activity can in both cases be explained by site symmetry and correlation field effects [13].

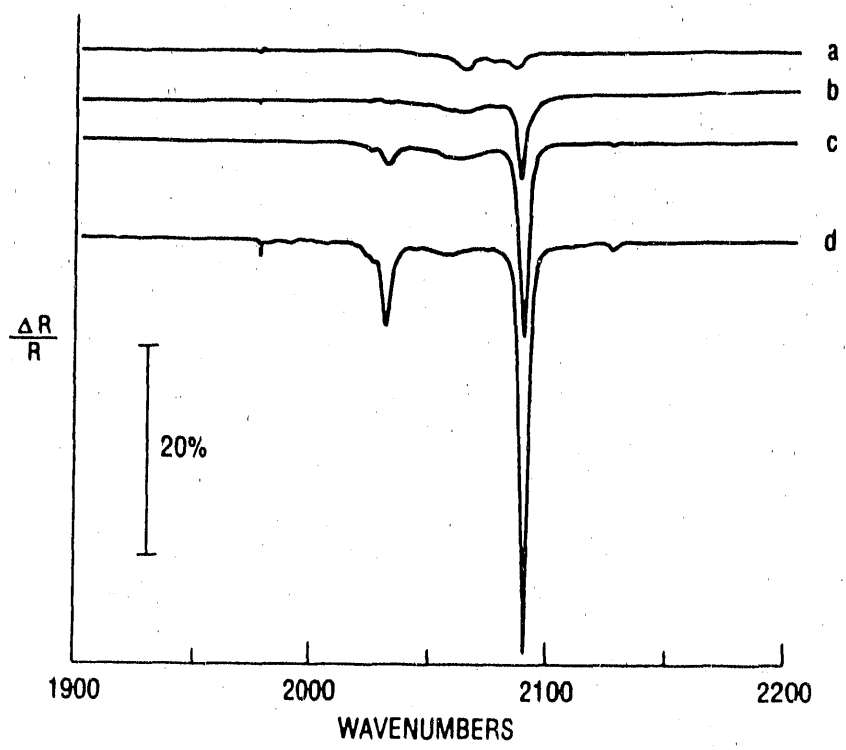

FIGURE 1: IRAS spectra of $Ru_3(CO)_{12}$/Ru(001). T=85 K (after a 230 K anneal), res. 1 cm^{-1}, 4 min. signal averaging. 2028 (ν_{35}, 4 cm^{-1} FWHM), 2087 (ν_{16}, 4 cm^{-1} FWHM), and 2124 (ν_1) cm^{-1} bands are due to C-O stretches of axial CO's of $Ru_3(CO)_{12}$; 2058 cm^{-1} band originates from CO adsorbed from the background directly on Ru(001). a) $\theta_{RC}\approx0.2$; b) $\theta_{RC}\approx1.0$; c) $\theta_{RC}\approx1.6$; d) $\theta_{RC}\approx7.7$. The spike at 1975 cm^{-1} in spectrum d is caused by an irreproducible feature in both single beam and background spectra.

The excitations of IR allowed modes due mainly to the C-O stretch of equatorial CO's have all frequencies below 2020 cm^{-1} [23,24]. The IR surface selection rule for metals allows only excitation of vibrational modes with a component of the dynamic dipole moment perpendicular to the surface [10]. The absence of any modes dominated by equatorial modes in IRAS (no bands below 2020 cm^{-1}) implies orientation of adsorbed $Ru_3(CO)_{12}$ with the Ru_3-triangle parallel to the surface. The narrow FWHM of the 2087 cm^{-1}

band - 4 cm^{-1} at all coverages shown (1 cm^{-1} less than ν_{C-O} of the ordered $(\sqrt{3}x\sqrt{3})R\ 30°$ structure of CO/Ru(001) [25]) - is a strong evidence of locally ordered adsorbate overlayers. We note that due to the strong dynamic dipole moment of the C-O stretch, dipole-dipole coupling might contribute to vibrational mode narrowing as demonstrated in a study of thin $KReO_4$ films [26].

CO TDS spectra of the $Ru_3(CO)_{12}$ overlayers shown in Fig. 1 are presented in the lower panel of Figure 2. They show a single zeroth-order desorption peak in addition to desorption features from CO/Ru(001) at 390 and 450 K. We attribute this peak to molecular $Ru_3(CO)_{12}$ since we simultaneously detected Ru_3^+ (m/e=306), Ru_2C^+ (m/e=216), and Ru_2^+ (m/e=204) fragments characteristic of molecular $Ru_3(CO)_{12}$ [27]. These are shown in the upper panel of Fig. 2.

The combined IRAS and TDS results thus suggest formation of locally well-ordered $Ru_3(CO)_{12}$ overlayers at all coverages studied (θ_{RC}≈0.2-10). There is no conclusive evidence for adsorbate ordering from LEED since $Ru_3(CO)_{12}$ overlayers are sensitive to electron-induced damage [19]; the only LEED pattern observed was a diffuse (1x1).

Figure 3 displays the IRAS spectra of $Mo(CO)_6$ for θ_{MC}≈0.3-5.6. The interpretation is based on a normal mode analysis [28] and on single crystal IR reflection spectra [29] of $Mo(CO)_6$. $Mo(CO)_6$ (O_h point group) has four triply degenerate (T_{1u}) IR allowed normal modes. Two of them (ν_8 and ν_9, following the notation of [28]) have frequencies below 500 cm^{-1} and therefore outside of our spectral range in the current configuration. The other two T_{1u} modes are shown in Fig. 3: ν_6 - mainly C-O stretch, and ν_7 - a complex motion involving Mo-C-O and C-Mo-C bending as well as some Mo-C stretching. The most striking feature is the narrowness of ν_7: 1 cm^{-1} FWHM. On the other hand, both peaks attributed to ν_6 have FWHM of 4 cm^{-1}, same as the C-O stretch in the $Ru_3(CO)_{12}$/Ru(001) system.

The splitting of the ν_6 mode can be explained using the CO (m/e=28) TDS results shown in Figure 4. The IR peak at 2036 cm^{-1} and the TDS peak at 245 K can be associated with the first $Mo(CO)_6$ layer. These features show saturation in both IRAS and TDS. The IR peak at 2043 cm^{-1} and the TDS feature with the ≈200 K onset show no saturation limit and we attribute them to additional $Mo(CO)_6$ layers. The differences between the first and additional layers are probably caused by the interaction of the Au/Ru surface with the first layer while already the second layer exhibits bulk-like behavior. The upper panel of Figure 4 shows TDS traces corresponding to Mo^+ (m/e=98) and $Mo(CO)_6^+$ (m/e=266). Our TDS data are in agreement with CO TDS traces from $Mo(CO)_6$/Rh(100) where a zeroth-order peak with the onset at ≈200 K was observed together with a 280 K feature assigned to first-layer $Mo(CO)_6$ or its fragments [30].

The evidence presented in Figures 3 and 4 thus strongly suggests molecular adsorption of $Mo(CO)_6$ on Au/Ru even in the first layer and formation of locally ordered overlayers, a behavior similar to the $Ru_3(CO)_{12}$/Ru(001) system.

$Ru_3(CO)_{12}$ and $Mo(CO)_6$ adsorbed on CO-saturated Ru(001), $Ru_3(CO)_{12}$/Au/Ru, and $Mo(CO)_6$/Ru(001) show a similar behavior as the systems discussed here in more detail. One difference involves a narrowing (≈15 to 7 cm^{-1}) of ν_{CO} of CO/Ru(001) when Ru(001) is saturated by CO prior to metal carbonyl adsorption. We attribute this observation to a higher degree of ordering in the CO-overlayer compared to CO coadsorbed from the

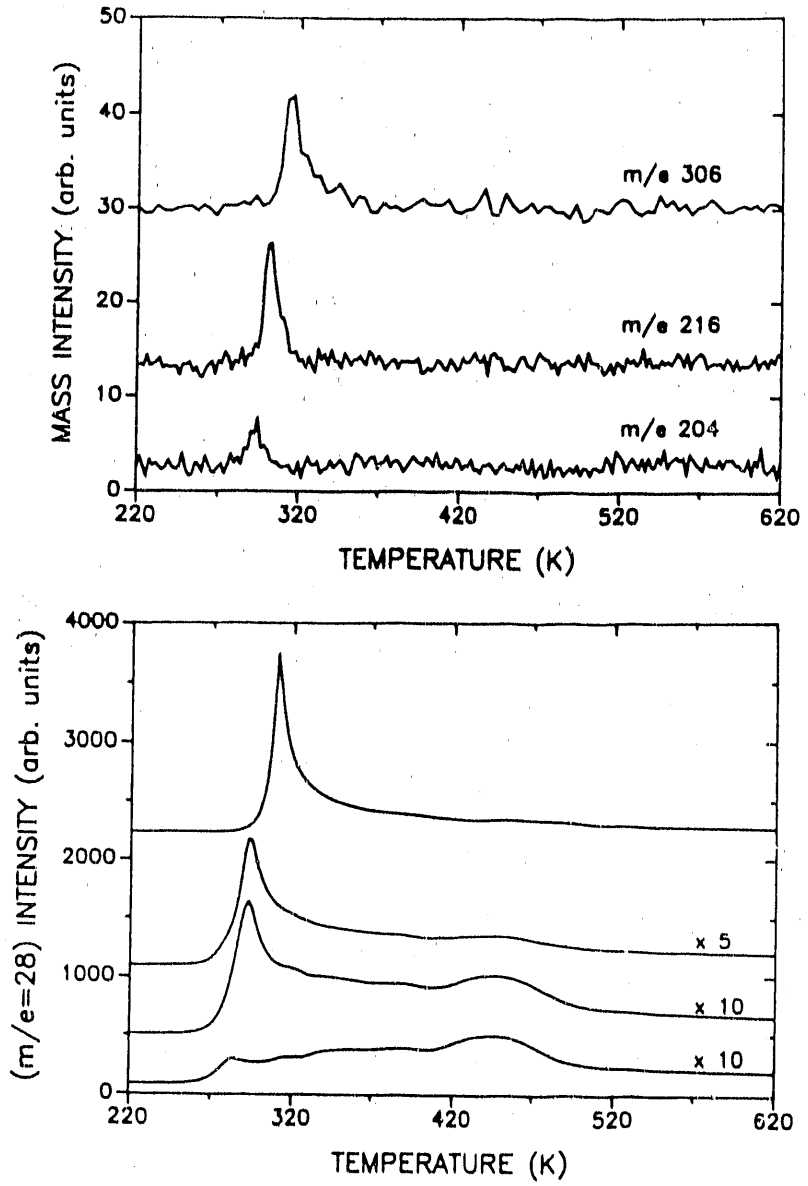

FIGURE 2: TDS spectra (2 K/s) from $Ru_3(CO)_{12}$/Ru(001) overlayers. Traces in the bottom panel were obtained after recording IRAS spectra a-d of Fig. 1. From bottom to top:
CO^+(x10), $\theta_{RC}\approx0.2$; CO^+(x10), $\theta_{RC}\approx1.0$; CO^+(x5), $\theta_{RC}\approx1.6$; CO^+(x1), $\theta_{RC}\approx7.7$; Ru_2^+ (m/e=204), $\theta_{RC}\approx3.0$; Ru_2C^+ (m/e=216), $\theta_{RC}\approx9.0$; Ru_3^+ (m/e=306), $\theta_{RC}\approx20.0$.

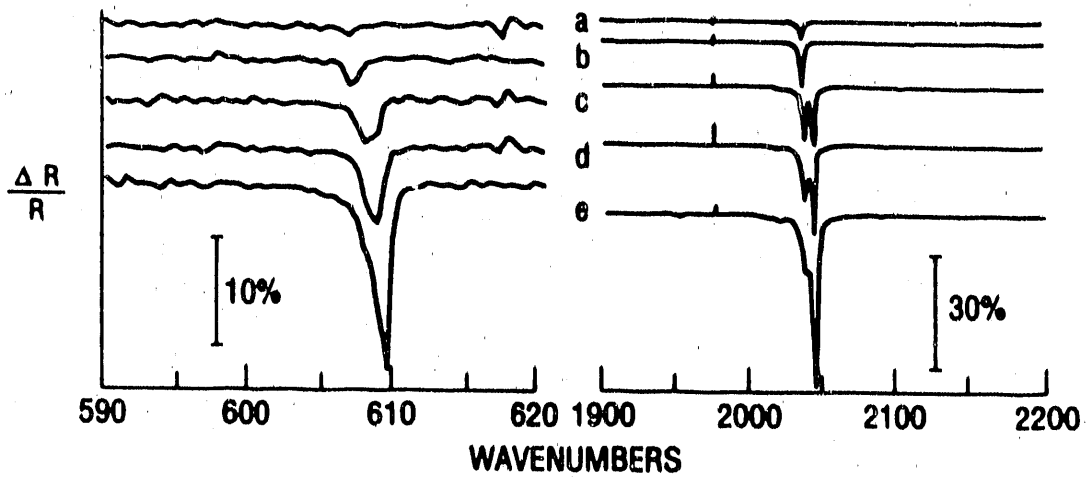

FIGURE 3: IRAS spectra of $Mo(CO)_6$/Au/Ru. T=85 K (after a 180 K anneal), res. 0.5 cm^{-1}, 10 min. signal averaging. 608 cm^{-1} peak: ν_7, 1 cm^{-1} FWHM; 2036, 2043 cm^{-1} peaks: ν_6, 4 cm^{-1} FWHM.
a) $\theta_{MC}\approx 0.2$; b)$\theta_{MC}\approx 0.6$; c) $\theta_{MC}\approx 1.7$; d) $\theta_{MC}\approx 2.5$; e) $\theta_{MC}\approx 5.6$.
The spikes at 1975 cm^{-1} are caused by an irreproducible feature in both single beam and background spectra.

background with the metal carbonyls [16]. Also, we saw splitting of ν_{16} in the $Ru_3(CO)_{12}$/Au/Ru system for $\theta_{RC}\leq\approx 3$ together with 3 additional (m/e=28) features in TDS; these indicate disorder and a possible fragmentation in the $Ru_3(CO)_{12}$ overlayer at low coverages [16].

The last feature of the IR spectra we want to briefly address in this paper are the intensities of the absorption bands due to C-O stretches of both metal carbonyls studied. For both $Ru_3(CO)_{12}$/Ru(001) and $Mo(CO)_6$/Au/Ru at submonolayer coverages the absorption per C-O bond projected onto the surface normal is larger than for $\theta_{CO}=0.68$ of CO/Ru(001). In case of $Ru_3(CO)_{12}$, using ν_{16} at $\theta_{RC}\approx 1.0$ (Fig. 1b), 90 $Å^2$ and 6 absorbing CO's per $Ru_3(CO)_{12}$, we arrive at a roughly twofold enhancement per CO compared to CO/Ru(001), $Int(CO)_{RC}/Int(CO)_{ads}\approx 2$. For $Mo(CO)_6$/Au/Ru, using ν_6 at $\theta_{MC}\approx 0.6$ (Fig. 3b), 50 $Å^2$ and 3.5 absorbing CO's (corresponds to a geometry with a maximum projection of C-O bonds onto the surface normal with three oxygens interacting equivalently with the substrate), we get $Int(CO)_{MC}/Int(CO)_{ads}\approx 5$. Since unusually large specular inelastic cross-sections were reported in an EELS study of $Ni(CO)_4$/Ni(111) [31], it seems that the dynamic dipole moment of the C-O stretch of metal carbonyls on metal surfaces is generally enhanced compared to CO adsorbed directly on

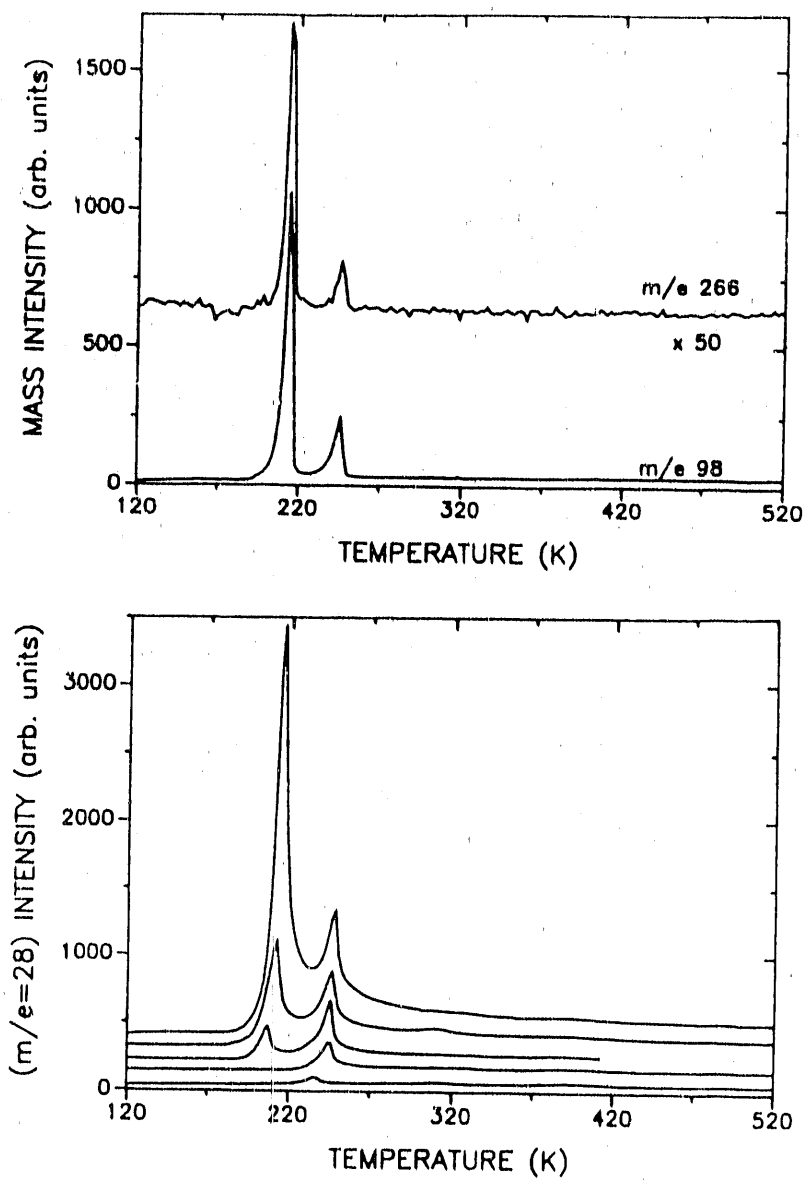

FIGURE 4: TDS spectra (2 K/s) of $Mo(CO)_6$/Au/Ru obtained after recording IRAS spectra a-e of Fig. 3. From bottom to top:
CO^+, $\theta_{MC} \approx 0.2$; CO^+, $\theta_{MC} \approx 0.6$; CO^+, $\theta_{MC} \approx 1.7$; CO^+, $\theta_{MC} \approx 2.5$; CO^+, $\theta_{MC} \approx 5.6$; Mo^+ (m/e=98), $\theta_{MC} \approx 5.6$; $Mo(CO)_6^+$ (m/e=266), $\theta_{MC} \approx 5.6$.

the surface. We do not fully understand the reason for this enhancement but find the explanation offered in [31] feasible: it was suggested there that the movement of the center of mass of adsorbed metal carbonyls with respect to the surface might increase the dynamic dipole moment of the adsorbate vibrational modes. Another possibility is an intensity transfer caused by dipole-dipole coupling from modes of lower frequencies (e.g., ν_{16} might be gaining some intensity from ν_{35} in case of $Ru_3(CO)_{12}$). However, it is clear that more work, both experimental and theoretical, needs to be done on these and similar systems to arrive at a reasonable level of understanding of this phenomenon.

4. SUMMARY AND OUTLOOK

We have presented IRAS and TDS results for $Ru_3(CO)_{12}$/Ru(001) and $Mo(CO)_6$/Au/Ru for metal carbonyl coverages ≈0.2-20. We characterized the C-O stretching modes of both systems (4 cm^{-1} FWHM) and a deformation mode of $Mo(CO)_6$/Au/Ru at 608 cm^{-1} (1 cm^{-1} FWHM). The interpretetion of both IR and thermal desorption spectra clearly suggests molecular adsorption and molecular desorption of the studied metal carbonyls with a preferential orientation and local order in the adsorbed overlayers.

Another interesting aspect of the metal carbonyls/metal systems that should be further studied is the intensity enhancement of vibrational transitions. We observed a two- and five-fold enhancement of the C-O stretch fundamental [relative to that of CO adsorbed directly on Ru(001)] of $Ru_3(CO)_{12}$/Ru(001) and $Mo(CO)_6$/Au/Ru, respectively.

We are currently investigating the interaction of adsorbed metal carbonyls with photons and electrons of various energies and the differences in absorption of p- and s-polarized IR radiation of thicker metal carbonyl overlayers. These studies will establish the potential of metal carbonyls as precursors of heterogeneous catalysts and for their use in thin film technology, and serve for better understanding of the relationship between bulk molecular crystals and their thin films.

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