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Surface reactivity studies of bimetallic complexes, $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(C{\equiv}C)_n(Ph_3P)(ON)Re(\eta^5\text{-}C_5Me_5)$ ($n = 2,4,6$): candidates for molecular wires

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Abstract

Conjugated polyalkynyl carbon chains, candidates for molecular wires, $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(C{\equiv}C)_n(Ph_3P)(ON)Re(\eta^5\text{-}C_5Me_5)$ ($n = 2$ ($Re^*C_4Re^*$), 4 ($Re^*C_8Re^*$), 6 ($Re^*C_{12}Re^*$)), have been deposited as thin films on air passivated copper surfaces and examined by X-ray photoelectron spectroscopy before and after exposure to air for various periods of time. The reactivity patterns of these thin films have been determined by examination of the oxidation state of Re and the chemical states of carbon, oxygen and phosphorus. Rhenium, which is zero valent in the starting compounds ($Re^*C_4Re^*$), ($Re^*C_8Re^*$) and ($Re^*C_{12}Re^*$) reacts to form Re^{3+}, Re^{4+} and Re^{5+}. Their thin film reactivities will be discussed and compared to their solution electrochemistry. We have found $Re^*C_4Re^*$ and $Re^*C_8Re^*$ to be more reactive than $Re^*C_{12}Re^*$ in agreement with the cyclic voltammetry results. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: X-ray photoelectron spectroscopy (XPS); Rhenium; Oxidation; Molecular wires; Bimetallic complexes

1. Introduction

The miniaturization of electronic components is a major challenge for the semiconductor industry, and it has brought together various different fields of science. The usual approach is to miniaturize existing larger components. However this cannot continue indefinitely as ultra miniaturization will reach a physical limit. The very large-scale integration (VLSI) method is already very near to that limit.

Today chemistry is pioneering other approaches by building electrical devices out of small molecules and therefore starting on the smallest possible size scale. One of the simplest electrical components are wires. To build molecular wires by organic chemistry it is necessary to make large and sufficiently stable molecules with a conjugated carbon chain, that can transport charge carriers, due to the delocalized π orbitals [1]. A variety of several different compounds have been reported in recent years that could be used as molecular wires (e.g. [2–17]). A review on the current developments in molecular wires has been published by Ward [1].

Numerous bimetallic complexes have been prepared in which metals are linked by unsaturated ligands [18]. Among many attributes, the bridging moiety can mediate several types of charge transfer phenomena [19]. Some, such as photoinduced electron transfer [20,21] or hyperpolarizability [22], are of potential practical importance. Others, such as electron delocalization in mixed valence compounds [19,23,24], have considerable theoretical importance. Much effort has been directed at the "tuning" or optimization of these properties, which have received particular attention and speculation from the standpoints of non-linear optics [22] and molecular devices such as wires, amplifiers and switches [1,17–19,25,26] and digital circuits [27,28].

Compounds which have the general formula $L_nMC_xM'L'_{n'}$ (**I**), where the metals are linked by the most

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basic and fundamental class of unsaturated organic ligand — an sp carbon chain [29], have lagged somewhat behind those with other types of unsaturated bridges [18,23,24]. However, they are now under intense study in numerous laboratories, and relatively common for $x = 2$ [30] and $x = 4$ [31–34].

A homologous series of complexes with the general formula (**I**) with both short and long carbon chains was synthesized, and key structural, electronic, and chemical properties as functions of redox states and chain lengths were characterized [7,8,35,36]. These efforts have emphasized the sterically congested and strongly π donating chiral rhenium endgroup (η^5-C_5Me_5)Re(NO)(PPh_3), which carries 16 valence electrons as a neutral species. Interestingly, recent data for sp carbon chains that bear organic endgroups suggest that there may be no practical upper limit on length [29] — an attractive scenario for possible device-like applications.

Important complementary efforts with C_6–C_8 complexes of other metal fragments have appeared from several laboratories [9,37–39]. This work has been spearheaded by Lapinte, who has focused on the achiral and more electron releasing iron endgroup (η^5-C_5Me_5)Fe(dppe) [26].

For any device like application of such compounds, they have to be connected to surfaces and at interfaces. Therefore, the interaction of molecular wires with metal surfaces and thin oxide films is of interest. Since electronic devices are usually used in air, the stability of molecular wires against oxidation in air is another important property. The aim of this paper is to show that such compounds can be studied on surfaces using surface science methods. In the present work, we show results from a preliminary study of the surface chemical reactivity of thin films of molecular wires with the general formula (**I**) on an air passivated metal surface in air and in vacuum by means of X-ray photoelectron spectroscopy (XPS). The results are expected to provide insight into the thin film stability resistance to oxidation and vacuum stability of these interesting molecular entities. A more thorough investigation of this system is currently under way.

2. Experimental

The structure of the molecular wire candidates used in this study ((η^5-C_5Me_5)Re(NO)(PPh_3)(C≡C)$_n$(Ph_3P)(ON)Re(η^5-C_5Me_5) ($n = 2, 4, 6$)), where n stands for the number of carbon–carbon triple bond units in the oligoacetylene chain, is shown in Fig. 1. We will use the following notation for the different molecules analyzed in this study: $n = 2$ (Re*C_4Re*), $n = 4$ (Re*C_8Re*), $n = 6$ (Re*C_{12}Re*).

The samples were dissolved in toluene and the solution was spread on a fresh polished polycrystalline copper surface. It has been shown previously that a clean copper surface passivated at room temperature in air consists of Cu_2O [40]. The sample was inserted in the vacuum chamber and the toluene was evaporated at a background pressure of 5×10^{-6} mbar. The sample was then inserted into the spectrometer UHV chamber. All the thin films of Re*C_xRe* on the copper surfaces studied proved to be stable under vacuum conditions for extended periods of time. No changes in the XPS signals could be detected after several days in vacuum.

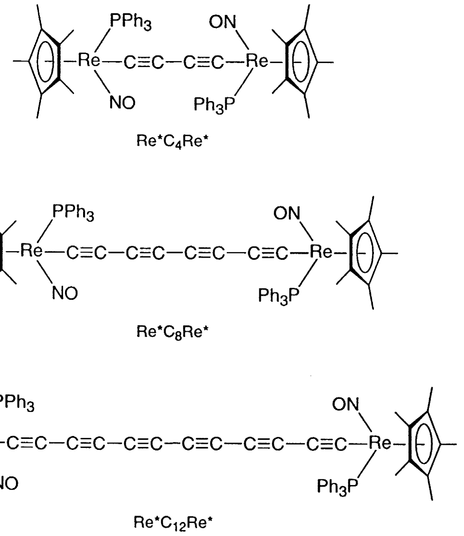

Fig. 1. Structures of Re*C_4Re*, Re*C_8Re* and Re*C_{12}Re*.

The thin films were examined using a Kratos XSAM 800 X-ray photoelectron spectrometer at a background pressure of 1×10^{-9} mbar. A magnesium anode was used and the Cu $2p_{3/2}$ signal (932.2 eV for Cu_2O [41]) was used as an internal reference to calibrate the binding energy. After initial XPS analysis the thin films were oxidized in air for 24 h followed by XPS analysis and additional oxidation in air for 24 h, followed by a final analysis with XPS.

3. Results and discussion

The results of the XPS investigation are shown in Figs. 2–6. The XPS peak identification and binding energy information are given in Table 1. A typical general scan of the 0–1000 eV binding energy range is shown in Fig. 2. The main peaks are carbon and oxygen. It is clear that the deposited samples effectively cover the passivated Cu substrate. The compounds investigated are clean according to NMR with a detection limit of 1% after preparation. On the copper surface, XPS shows silicon 2p and 2s signals (Fig. 2). These signals are probably due to accumulation of silicon impurities (less than 1% according to NMR) on the surface. The most likely

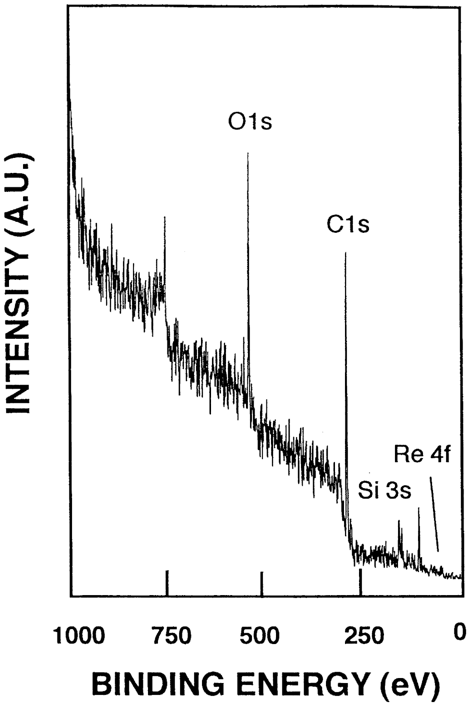

Fig. 2. XPS spectra of a Re*C_{12}Re* film. General scan before oxidation.

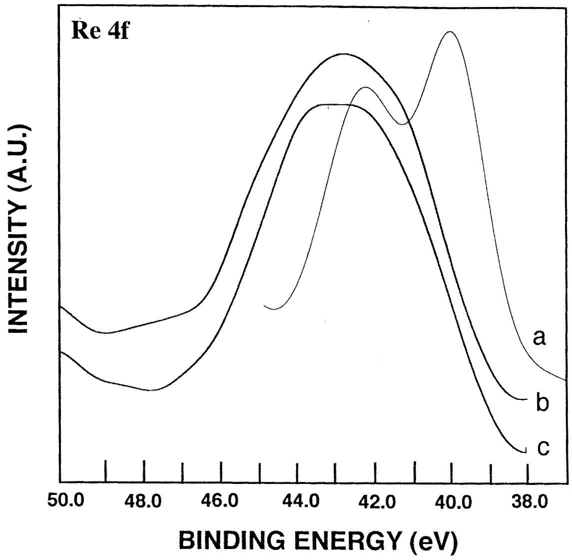

Fig. 3. Re 4f XPS spectra of Re*C_4Re* (a) before oxidation, (b) after 24 h in air and (c) after 48 h in air.

source of the silicon is the chromatography purification step over silica gel. In Table 1 the binding energies of the chemical states of Re, C, O, N and P are documented for the films before and after oxidation.

Although the compounds have an NO ligand on the rhenium (Fig. 1) we could only detect a N1s signal on the oxidized Re*C_4Re* compound (Table 1). This might be due to the low atomic sensitivity factor of nitrogen, the low atomic percentage and to the fact that this group is probably hidden under the relative large triphenylphosphine and pentamethylcyclopentadienyl groups. The binding energy for the N1s signal is about 407 eV which is in the range of organic NO_2 groups and nitrates [41].

On the Re*C_4Re* and Re*C_8Re* compounds we detected a small P 1s signal with a binding energy of 130 eV, which is in the range of triphenylphosphine [41]. The phosphorus peaks shift to higher binding energy after

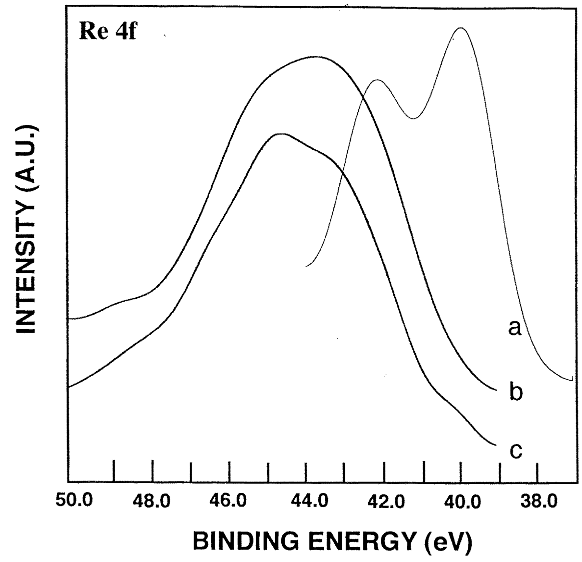

Fig. 4. Re 4f XPS spectra of Re*C_8Re* (a) before oxidation, (b) after 24 h in air and (c) after 48 h in air.

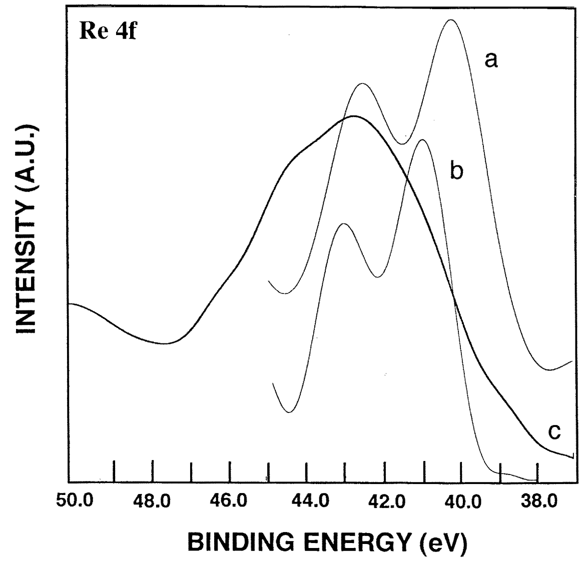

Fig. 5. Re 4f XPS spectra of Re*C_8Re* (a) before oxidation, (b) after 24 h in air and (c) after 48 h in air.

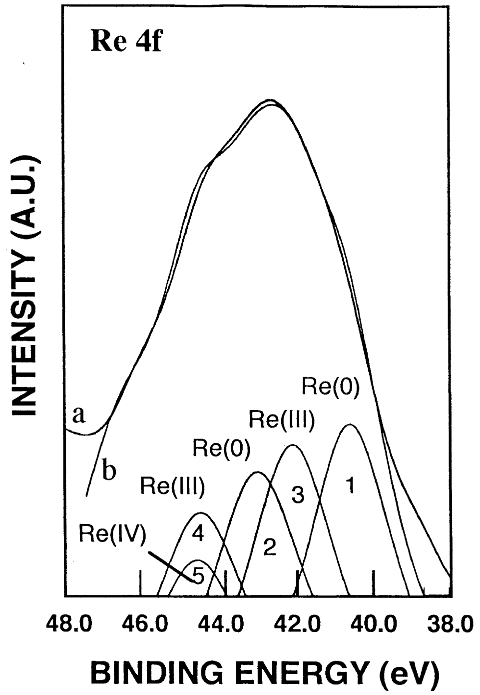

Fig. 6. Re 4f XPS spectra of Re*C_{12}Re* after 48 h in air: (a) experimental data, (b) sum of deconvoluted peaks 1–7 (see text for details). Peaks 1 and 2 correspond to Re (0), peaks 3 and 4 correspond to Re(III) and peak 5 corresponds to Re(IV).

oxidation. With about 132 eV the binding energy of the P 1 s signal is in the range of Ph_3P–O and Ph_3–M species [41], where M stands for a transition metal. This indicates that during oxidation of these compounds the ligands are oxidized.

The carbon 1 s signal is in a binding energy range between 284 eV before oxidation and 285 eV after oxidation (Table 1) which is well within the hydrocarbon region [41]. The carbon 1 s signal for hydrocarbons with CO species is above 286 eV. After oxidation the peaks are about 1 eV broader at half-maximum than before oxidation, retaining the symmetric overall shape. The broadening of the C 1s peaks might indicate the appearance of some CO and CRe^{n+} species.

The oxygen 1 s signals are in a binding energy range between 532 eV before and 533 eV after oxidation. These values of the binding energies are well above transition metal oxides [41]. The oxygen signals probably consist of a part due to adsorbed oxygen and a part due to the NO ligand of the compound. The oxidation leads to a broadening of the O 1 s peaks. The full-width at half-maximum is about 0.4 eV broader than in the pristine samples. This might be due to the formation of CO and ReO species.

In the following we discuss the rhenium 4f signal of the three compounds in more detail. Figs. 3–5 show the Re 4f signals of the pristine, 24 and 48 h oxidized Re*C_4Re*, Re*C_8Re* and Re*C_{12}Re* compounds, respectively. During oxidation the peak envelope maxima shift to higher binding energy. This clearly indicates that oxidation of the rhenium atoms occurs.

Fig. 3a shows the Re 4f signal for the pristine Re*C_4Re* compound. The binding energy for the Re $4f_{7/2}$ is 40.0 eV, corresponding to zero valent rhenium [41]. The distance to the Re $4f_{5/2}$ peak is about 2.4 eV as is to be expected for zero valent Re [41]. After oxidation for 24 h (Fig. 3b) the peak envelope of the Re 4f signal has its maximum at 42.6 eV. The $4f_{7/2}$ and the $4f_{5/2}$ peaks are not resolved. After 48 h of oxidation (Fig. 3c) the peak maximum shifts only slightly to 42.8 eV with not much change in the overall peak shape.

The Re*C_8Re* compound shows a similar behavior during oxidation in air (Fig. 4). The rhenium 4f signal in the original compound (Fig. 4a) is at a binding energy of 39.9 eV for the $4f_{7/2}$ peak. The distance to the $4f_{5/2}$ peak is about 2.4 eV, again corresponding to zero valent rhenium. Oxidation of the Re*C_8Re* sample for 24 h in air (Fig. 4b) leads to a 3.2 eV shift in the envelope of the rhenium 4f signal to higher binding energies. The peak maximum is at 43.1 eV and a shoulder is visible around 44.5 eV. The $4f_{7/2}$ and the $4f_{5/2}$ peaks are not resolved. Additional oxidation for 24 h in air (Fig. 4c) leads to a binding energy at the peak maximum of 44.2 eV. A shoulder at around 43.0 eV is clearly visible.

Table 1
XPS data for Re*C_4Re*, Re*C_8Re* and Re*C_{12}Re*

Sample	Re 4f	C 1s	O 1s	N 1s	P 1s
Re*C_4Re* pristine	40.0 eV	284.1 eV	532.0 eV	No signal	130.0 eV
Re*C_4Re* 24 h air	42.6 eV	284.8 eV	532.6 eV	407.3 eV	131.5 eV
Re*C_4Re* 48 h air	42.8 eV	284.6 eV	532.5 eV	406.8 eV	131.9 eV
Re*C_8Re* pristine	39.9 eV	283.7 eV	531.3 eV	No signal	130.0 eV
Re*C_8Re* 24 h air	43.1 eV	284.8 eV	532.6 eV	No signal	131.7 eV
Re*C_8Re* 48 h air	44.2 eV	285.3 eV	533.1 eV	No signal	132.4 eV
Re*C_{12}Re* pristine	40.2 eV	284.2 eV	531.9 eV	No signal	No signal
Re*C_{12}Re* 24 h air	41.4 eV	285.0 eV	532.9 eV	No signal	No signal
Re*C_{12}Re* 48 h air	43.7 eV	284.9 eV	532.8 eV	No signal	No signal

The spectra of the Re*C_{12}Re* sample show a slightly different behavior (Fig. 5). The Re 4f signal of the pristine Re*C_{12}Re* (Fig. 5a) shows a binding energy for the Re $4f_{7/2}$ of 40.2 eV. The distance to the Re $4f_{5/2}$ is about 2.4 eV. The distance between the two 4f peaks and the shape strongly resemble those of zero valent rhenium metal again [41]. After oxidation in air for 24 h (Fig. 5b) the Re 4f signal starts at about 1.0 eV higher binding energy as before the oxidation, leading to a peakmaximum for the Re $4f_{7/2}$ of 41.4 eV. This corresponds to Re^{3+} in a complex with three Cl and three organophosphor ligands [41]. This indicates that some oxidation of the rhenium has occurred. But in contrast to the Re*C_4Re* and Re*C_8Re* compounds the Re $4f_{7/2}$ and $4f_{5/2}$ signals are still resolved and the shape is the same as for zero valent rhenium. That might indicate that only a small portion of the rhenium atoms is oxidized after 24 h. Additional oxidation in air for 24 h (Fig. 5c) leads to a similar rhenium signal as after 24 h of oxidation of the Re*C_4Re* and Re*C_8Re* compounds. The binding energy of the peak envelope of the Re*C_{12}Re* compound shifts up to 43.7 eV corresponding to a 2.3 eV shift. A shoulder on the high binding energy edge at about 45.3 eV can clearly be seen.

In order to get more information about the rhenium oxidation states that lead to these Re 4f XPS signals, we made a decomposition of these peaks. Fig. 6 shows the peaks that we used to model the peak shape of the Re*C_{12}Re* compound after 48 h of oxidation in air. We were able to get a good fit of the peak shape using the following peaks: Peak 1 at 40.6 eV and peak 2 at 43.0 eV which correspond to zero valent rhenium [41]. Peak 3 at 42.1 eV and peak 4 at 44.5 eV which approximately correspond to Re^{3+} according to [41]. Peak 5 at 44.6 eV and Peak 6 (not visible in Fig. 6 due to the small intensity) at 47.0 eV correspond to Re^{4+} [41]. Peak 7 (not visible in Fig. 6 due to the small intensity) at 46.4 eV corresponds to a rhenium oxidation state higher than IV. The pairs correspond to the $4f_{7/2}$ and $4f_{5/2}$ doublets.

Using these peaks with the binding energies, the full-widths at half-maximum and the ratio of the peak heights of the doublets held constant we could fit the overall shapes of all the rhenium 4f peaks by changing the peak heights only. These results suggest that in all samples we have contributions of four different rhenium oxidation states. It is difficult to distinguish between the five stable oxidation states of rhenium (III, IV, V, VI and VII) [42]. Different ligands have been shown to influence the binding energy of Re oxidation states [42]. The binding energy for the $4f_{7/2}$ signal for Re^{3+} and Re^{2+} was reported as 44.8 and 44.5 eV, respectively [43]. In a study of oxidation of rhenium films at atmospheric pressures, Tysoe, et al. [44] decomposed their Re 4f signals using Re^0, Re^{4+}, Re^{6+} and Re^{7+}. As Re(I) has the same binding energy for the $4f_{7/2}$ signal as zero valent rhenium [40], we cannot rule out that there is no zero valent rhenium left after the oxidation. As Re(VII) is volatile and only a small amount of Re(VII) was found in the oxidation of rhenium films [44] we think that the peak at the highest binding energy of our decomposition corresponds to Re(V) or Re(VI). The peak at 44.6 eV corresponds to Re(IV) in agreement with [41]. The peak at 42.1 eV corresponds to Re(III) [41]. A definitive assignment of binding energies to the different Re oxidation states is not possible with the resolution of the XPS results.

The shifts seen in the carbon, oxygen and phosphorous signals together with the changes in the Re 4f signal suggest that oxidation takes place on the whole molecular wire after initial oxidation of the central rhenium atoms.

The different behavior of Re*C_{12}Re* compared to Re*C_8Re* and Re*C_4Re* can be connected to the reactivity of the compounds. Comparing the Re 4f peaks after 24 h of oxidation Re*C_{12}Re* shows a lower reactivity towards oxidation than Re*C_8Re* and Re*C_4Re*. In order to get a clearer picture of the different reactivities we compared the XPS results after oxidation in air to the cyclic voltammetry results in solution for the same compounds [7,8]. Compounds where two metal centers are linked with a conducting bridge may undergo several distinct oxidation–reduction steps. This applies to the complexes of type (**I**). During cyclic voltammetry of solutions of type (**I**) complexes with ruthenium, iron and rhenium the sequentially and (semi)reversible loss of electrons leads to the corresponding cationic species

Table 2
Electrochemical and thermal reactivities of molecular wires films

Name	CV results E^o [V] first & sec. ox. [7,8], see text	Vacuum (% Re oxidized)	24 h in air (%Re oxidized)	48 h in air (%Re oxidized)
Re*C_4Re*	0.01 0.54	0	60	60
Re*C_8Re*	0.24 0.52	0	80	80
Re*C_{12}Re*	0.46 0.65	0	$\leqslant 10$	60

[7,8,31,45,46]. Oxidation of thin films of these compounds in air provides irreversible oxidation to higher oxidation states. A comparison of the electrochemical and thermal reactivities of molecular wires is shown in Table 2. The E° values from [7,8] have been reported relative to a ferrocene $E^{\circ\prime}$ value of 0.56 V (An authoritative review [47] has proposed protocols for standardizing E° values from a variety of solvent/electrolyte combinations to a common SCE reference. These employ ferrocene $E^{\circ\prime}$ values of 0.46 V in $CH_2Cl_2/n\text{-}Bu_4N^+PF_6^-$. In order to facilitate comparisons of the E° values with others in the literature, this convention was adopted and utilized for Table 2). The reactivities for oxidation in air were calculated as the ratio of the oxidized rhenium 4f peak area to the total rhenium 4f peak area, which were calculated from the decomposition of the peaks. In case of the $Re^*C_{12}Re^*$ after 24 h of oxidation in air we propose only a small amount of oxidized Re atoms, as the peak shape for the Re 4f signal obviously changes markedly with oxidation (see Figs. 3–5). The cyclic voltammetry data (Table 2) show a reactivity order (lower E° means higher reactivity) of $Re^*C_4Re^* > Re^*C_8Re^* > Re^*C_{12}Re^*$ for the first oxidation. Second oxidation shows an order of $Re^*C_4Re^* \approx Re^*C_8Re^* > Re^*C_{12}Re^*$ (Table 2). Comparing the reactivity calculated from the XPS results for oxidation of the thin films in air after 24 h and after 48 h shows an order of reactivity of $Re^*C_4Re^* \approx Re^*C_8Re^* > Re^*C_{12}Re^*$ (Table 2), which corresponds to the order in the second oxidation step in cyclic voltammetry in solution. In this comparison one has to bear in mind that oxidation in air leads to higher oxidation states (up to 5 +) than the cyclic voltammetry data, because oxidation in air cannot be limited to the first and second oxidation which has been done in the cyclic voltammetry study.

4. Conclusions

Thin films of $Re^*C_{12}Re^*$, $Re^*C_4Re^*$ can be successfully deposited on air passivated metals such as copper. These films have been shown to be uniformly covering the surface enough to suppress the substrate visibility with XPS. They have been shown to be stable in vacuum for extended periods of time that allow examination with XPS before and after exposure to air for 24 and 48 h.

The chemical attack of the molecular wire thin films by air has been shown to cause oxidation of the Re metal centers. The Re atoms in the compounds which were initially zero valent were oxidized in a stepwise fashion to Re(III) and then to Re(IV) and possibly higher oxidation states. The dominant oxidation state of the rhenium atoms after oxidation in air is Re(IV) with both Re(III) and higher oxidation states than Re(IV) also being simultaneously present.

According to the XPS results on the copper surface the $Re^*C_{12}Re^*$ compound is more stable in air than the $Re^*C_8Re^*$ and the $Re^*C_4Re^*$ compounds, which is in agreement with cyclic voltammetry data.

The changes in XPS spectra of C, O, P and N during oxidation indicate that all ligands of the Re atoms are possibly also being oxidized under the present conditions.

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