Iodine adsorption on clean and cesiated W(110) studied with UPS and metastable impact electron spectroscopy (MIES)

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Abstract

Photoemission spectra (UPS with HeI) and metastable impact electron spectroscopy (MIES with $He^*(1s2s)$) are reported for the adsorption of iodine alone, the coadsorption of iodine and cesium, and the adsorption of CsI on W(110) at room temperature. It is demonstrated that the I(5p) ionization is sensitively dependent on the chemical environment of the iodine adsorbate. For iodine in a strong external field, such as for adsorption on clean metals, the photoemission spectra reflect the Stark splitting of the (5p) orbital while the MIES spectra are due to the Auger capture process. When adsorbed near to cesium iodine feels a comparatively weak external field and the emission spectra both for UPS and MIES (due to the Auger deexcitation process) reflect the I(5p) finestructure splitting in the final state after the emission process.

1. Introduction

The adsorption of halogen atoms, in particular in combination with alkali atoms on metallic surfaces, has received considerable interest. Data for the adsorption of halogen atoms on clean metals have been published [1–3]. They are of great interest in the field of catalysis (the epoxidation of ethylene in the presence of halogenes may serve as an example [4]). Furthermore, data for the coadsorption of halogen atoms with molecular species, such as water and/or ammonia, at high temperatures are essential to understand the kinetics and dynamics of high-temperature corrosion processes. The adsorption of alkali halides and alkali earth halides, i.e. halogen adsorption in the presence of alkali atoms, has received

attention due to the possible stimulated desorption processes [5–8]. Alkali halides are of fundamental interest for the study of lateral interactions in chemisorption systems.

It has been shown, that the features from iodine ionization are strongly dependent on the chemical environment of the adsorbed I atom [7]. There are two limiting situations for the iodine adsorption:

(1) The (5p) electrons feel a rather weak field:
   The photoemission peak displays the finestructure (FS) splitting of the free iodine atom (produced in the photoemission of I^-). Examples for this situation are:
   ● Photoemission of I^- in CsI molecules (either in the free molecule [9,10] or adsorbed molecularly [7]). The attraction of the 5p electrons towards the Cs^+ is largely counterbalanced by the electron affinity of the iodine (atom) [9];

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● Photoemission of iodine is an isotropic environment, such as found in bulk CsI crystals [11] or in certain large molecules at liquid surfaces [12].

(2) The I 5p-electrons feel a strong external field. A Stark field type splitting of the 5p orbital into σ and π components according to their projection with respect to the surface normal is observed [9,10]. One example for this situation is the photoemission from iodine chemisorbed on many metals (see Refs. [1–3] and references therein). In particular, the adsorption of I on Fe(110) and Ag(100) represents well-studied systems.

The photoemission of iodine in molecules containing halogen represents an intermediate case: the finestructure splitting of the free (iodine) atom $\Delta E_{FS} = E(^2P_{1/2} - {}^2P_{3/2}) = 1.5\xi$ is not observed, but values of about ξ (= 0.63 eV). Examples are the photoemission features found for alkylhalides or halogenhydrides (with ξ-values between 0.55 and 0.62 eV) [13]. The reduced finestructure splitting is due to the axial electric field felt by the iodine atom.

In order to exploit the sensitive dependence of the iodine photoemission features on the chemical environment, we have studied the photoemission (UPS with HeI) and the metastable induced electron emission (metastable impact electron spectroscopy (MIES) with He*(2^3S)) spectra from iodine adsorbed on clean and partially cesiated W(110) as well as CsI adsorption on W(110). MIES appears to be uniquely suited to separate contributions to the iodine ionization originating from the outermost edge of the adlayer clearly from those due to iodine located deeper in the adlayer.

2. Experimental

The apparatus has been described in detail previously [14–16]. Briefly it is equipped with a cold-cathode gas discharge source for the production of metastable He*(2^3S/2^1S) ($E^* = 19.8/20.6$ eV) atoms and HeI photons ($E^* = 21.2$ eV). The triplet:singlet ratio is in the range of 7:1. Due to the fact that He*(2^1S) atoms are converted into He(2^3S) atoms in front of the surface very efficiently [17,18] only deexcitation from He(2^3S) is observable with

the exception of He(2^1S) induced feature beyond the He(2^3S) Fermi level. The He(2^3S):HeI excitation ratio is about 1:50. HeI excited electrons are therefore not visible in the MIES spectra with the exception of small contributions beyond the Fermi level. To perform UPS measurements the metastable atoms are scattered out of the beam by He–He* collisions in a buffer chamber. The collection of a single MIES spectrum requires less than one minute; an UPS spectrum requires about 4 min. MIES and UPS measurements are performed using an hemispherical analyzer (VSW HAC5000) with an energy resolution of 250 meV. MIES and UPS spectra are shown as a function of kinetic energy, the Fermi levels are found at $E_{kin} = 21.2$ eV (UPS) and $E_{kin} = 19.8$ eV (MIES), respectively. All electron spectra are taken under an incident angle of 45° and an emission angle of 0° to the surface normal, respectively. The low energy cut-off of the UPS spectra then directly reflects the work function of the surface. The substrate is continuously exposed to the adsorbate (I, Cs, and CsI) while the spectra are collected. The coverages given with the spectra are related to the first complete adlayer (denoted by 1 ML) of the respective adsorbate on W(110) at room temperature. All spectra are corrected in order to compensate the energy dependence of the hemispherical analyzer. Furthermore the apparatus is equipped with the standard tools LEED and AES.

Alkali halide layers are produced by thermal evaporation of single crystal chips [6,7]. Cs is evaporated using SAES getters sources [14,15]. I is evaporated from an electrochemical cell by heating AgI. The cell originally designed by Wagner [19] has been adapted for operation under ultrahigh vacuum conditions [20].

The base pressure of the UHV system is 7×10^{-11} Torr. During the evaporation of the adsorbates the pressure is about 2×10^{-10} Torr for CsI, 3×10^{-10} Torr for Cs and 5×10^{-10} Torr for I. MIES measurements are performed in a He atmosphere of about 1×10^{-9} Torr, UPS is performed in a He pressure of about 8×10^{-9} Torr.

All measurements were performed at room temperature. The coverages were determined utilising UPS measurements for the determination of the work function change upon exposure and their comparison with previous measurements [21]. Cs and I grow on

W(110) at room temperature with a constant sticking coefficient below 1 ML. Both I and Cs form only one complete adlayer on W(110) at room temperature [21]; however, CsI is capable of forming thick multilayers. The determination of the CsI coverage has been performed according to the results of Ref. [21]: up to the point where the work function upon CsI exposure reaches its final value the Auger signals of Cs and I increase linearly. After the work function saturation they increase linearly too with a smaller slope. It has therefore been concluded, that the first adlayer has been completed at the mentioned point [21]. According to this result the coverages have been indicated in the spectra.

3. Experimental results

The figures shown below present the variation of the electron spectra with exposure to Cs, I and CsI. All spectra are plotted using identical scales and are separated by a constant offset.

3.1. Cs / W(110)

In order to simplify the discussion of the Cs induced feature in the case of I and Cs coadsorption the results for the adsorption of Cs on W(110) were also studied; similar results have been published previously [22–24]. Figs. 1a and 1b show the MIES and UPS spectra for partially cesiated W(110) as a function of Cs coverage.

The peaks arising from the $Cs(5p_{1/2}, 5_{3/2})$ orbitals are found at binding energies of about 12.0 and 13.5 eV (with respect to the Fermi energy) for MIES and UPS [14,22,25,26]. A peak denoted by Cs(6s) in Fig. 1a appears in the MIES spectra at coverages higher than $\theta_{Cs} \approx 0.4$ ML (in units of the first complete adlayer on W(110) at room temperature) at a binding energy below 1 eV, which has been discussed previously [23]. It is mostly due to autodetachment (AU) of $He^{-*}(1s2s^2)$ formed by the resonant transfer (RT) of a surface electron to the He^* [24,27]. The low-energy onset of the spectra shifts towards lower binding energies due to the decreasing work function. The smooth bottom spectra of Fig. 1a arises from a two electron Auger process (Auger capture (AC)), which occurs on high work function
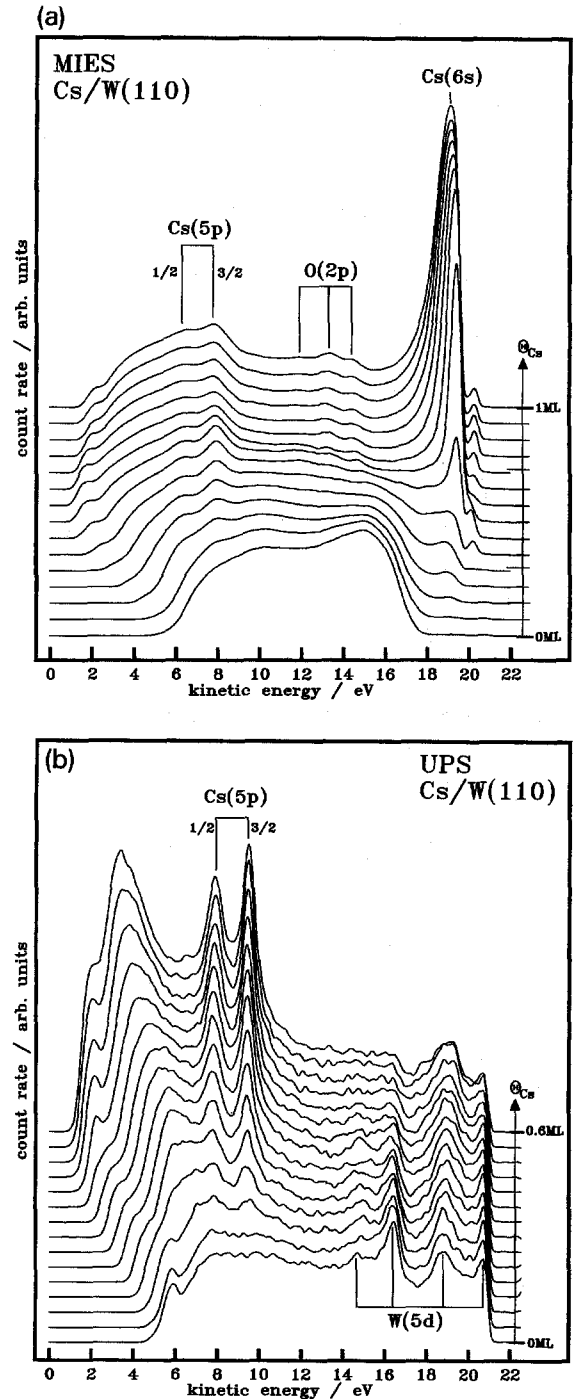

Fig. 1. MIES spectra (a) and UPS spectra (b) during Cs adsorption on W(110). The coverage is indicated in the spectra. The bottom spectrum corresponds to clean W(110).

surfaces [28]. With decreasing work function the one electron process (Auger deexcitation (AD)) comes up with an energy balance analogous to UPS, but carrying an excitation energy of 19.8 eV instead of 21.2 eV (HeI). The position and the relative distance of the Cs(5p) derived peaks remain constant during the adsorption. The structures denoted by O(2p) indicates an oxygen contamination below 1×10^{-2} L O_2 which is visible with MIES due to the extremely high surface sensitivity of this technique. The small peak appearing beyond a Cs coverage of 0.4 ML on the right side of the Cs(6s) feature is due to AU of $He^{-*}(1s2s^2)$ formed via $He(2^1S)$ [24] carried in the He^* beam, which has not been converted into $He(2^3S)$ prior to deexcitation. In the UPS spectra emission involving Cs(6s) electrons is not observable. The excitation cross section of low energy photons and s-orbitals almost vanishes [9]. Possible contributions are masked by the strong substrate emission.

3.2. I / W(110)

Figs. 2a and 2b show the MIES and UPS spectra during increasing I exposure on W(110). The bottom spectrum shows a reduced intensity below E_F as compared to the spectra of Fig. 1a and Fig. 3a. This is caused by the gas pressure produced by the heated I evaporation cell. This pressure is higher than for Cs or CsI (see Section 2). The difference is visible due to the extremely high surface sensitivity of MIES. The work function remains large during the iodine adsorption. Therefore, Auger deexcitation is not possible; the MIES spectra (arising from AC) are unstructured. Due to the I adsorption, the surface density of states (SDOS) is strongly reduced which causes the weakening of intensity just below E_F. An analogous behaviour is found adsorbing oxygen on transition metals [29]. The SDOS at E_F is strongly reduced by charge transfer to the I adsorbate filling up the I(5p) shell.

Fig. 2b shows the UPS spectra of the same system. With increasing coverage a broad structure (labelled A) appears at a binding energy $E_B = 5.7$ eV. This structure has been associated to I chemisorption on tungsten [1] and appears at similar binding energy on Ag(100) [3] and Cu(110) [2]. The contributions from the $I(p_z)$ and $I(p_{x,y})$ ionization
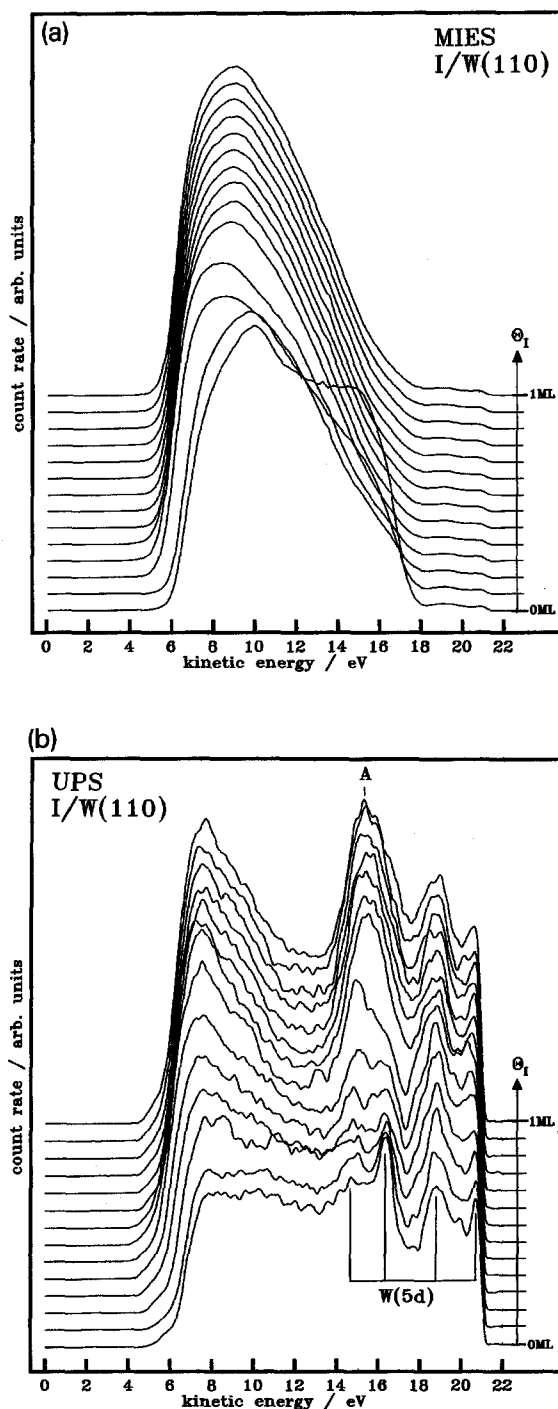

Fig. 2. MIES spectra (a) and UPS spectra (b) during I adsorption on W(110). The coverage is indicated in the spectra. The bottom spectrum corresponds to clean W(110).
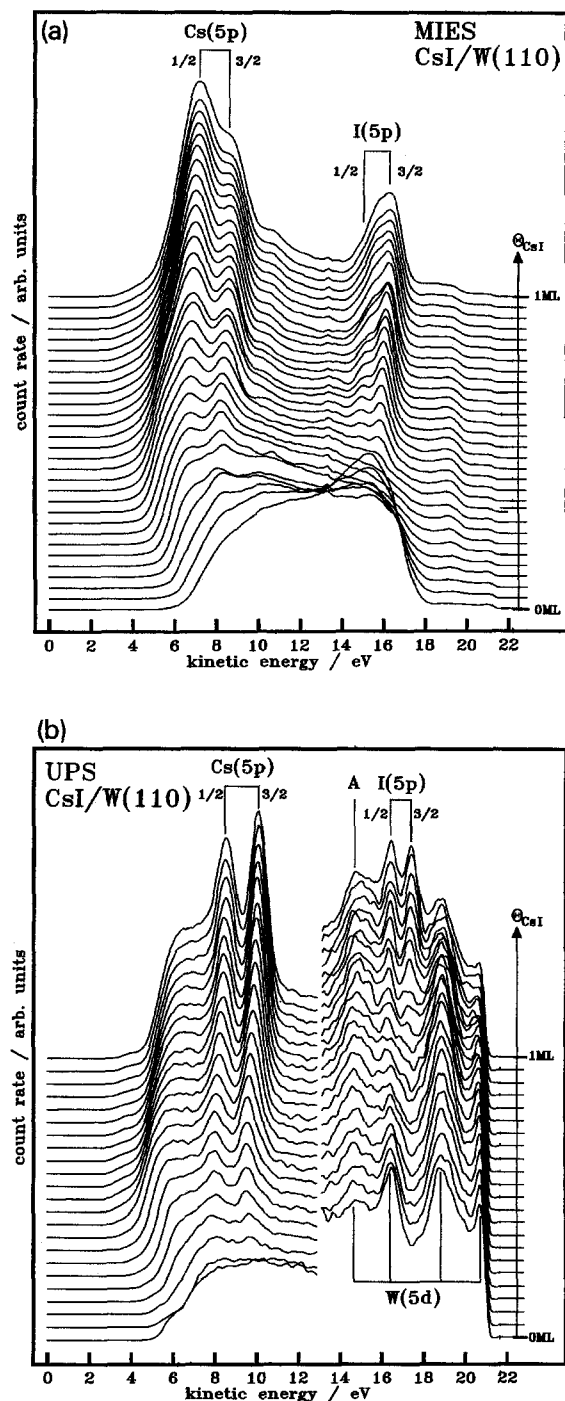
Fig. 3. MIES spectra (a) and UPS spectra (b) during CsI adsorption on W(110). The coverage is indicated in the spectra. The bottom spectrum corresponds to clean W(110).

are not clearly resolved. The shoulder around 17 eV ($E_B = 4.5$ eV) must most likely be attributed to W(5d).

3.3. CsI / W(110)

The adsorption of CsI on W(110) was studied by us in some detail previously [7]. Figs. 3a and 3b present the MIES and UPS spectra obtained during the adsorption of CsI but at evaporation rates smaller than those of Ref. [7]. The top curve of the figures corresponds to a coverage of about 1 ML CsI on W(110) [21].

At low CsI exposures the presence of I on the substrate does only lead to a decrease of emission intensity at high kinetic energies for MIES, similar to the findings of Fig. 2. The absence of any sharp feature suggests that He* is deexcited via the AC process when interacting with iodine atoms. At CsI exposures larger than those leading to the work function minimum, the structure I(5p) develops, displaying a finestructure splitting of about 1.0 eV. We conclude that only for these exposures I atoms in a weak external field do exist; certainly these I atoms cannot be in direct contact with the W substrate. A relatively well resolved (at least for small exposures) finestructure doublet from AD of He* involving Cs(5p) electrons is visible in the spectra even at the lowest CsI exposures. This implies that the AD process occurs whenever He* interacts with Cs atoms.

In UPS the structure A is discernible although it partly overlaps with the substrate emission. When the work function minimum is passed, the structure I(5p) (seen with MIES) emerges and displays the finestructure splitting (at least for small CsI exposures) of the free I atom (0.95 eV). Both structures, A and I(5p), coexist although I(5p) grows faster upon further exposure. A well-resolved finestructure doublet from the Cs(5p) ionization is found. The double peak, denoted by I(5p), consists of two components separated by about 0.95 eV. The identification of the feature can be made on the basis of photoelectron spectroscopy results from gaseous CsI [9,10] and UPS data from CsI bulk material [7,11]: the two peaks at $E_B = 3.9$; 4.8 eV are due to formation of the two finestructure states ($^2P_{1/2,3/2}$) of iodine (p^5, ^2P) (after removal of a (5p) electron from

$I^-(5p^6)$). Adding the work function to the binding energies (E_B) we obtain the ionization energies 6.9 and 7.8 eV with respect to the vacuum level; these values have to be compared with the ionization energies 7.2 and 8.1 eV of the free CsI molecules [9,10].

Additional measurements (not presented here) have been performed coadsorbing Cs and I simultaneously. The results are very similar to those presented in Fig. 3 if the stoichiometry of Cs and I is in the range of unity. Otherwise the spectra are rather complex.

3.4. I / (W(110) + Cs)

Figs. 4a and 4b show a set of spectra for the adsorption of I on Cs covered W(110) surface. The surface is precovered with about 1 ML Cs.

The bottom spectrum of Cs/W(110) has been discussed in Section 3.1. During the exposure to iodine the Cs(6s) electrons are transferred to the iodine coadsorbate. As a consequence the formation of He^{-*} becomes inhibited: the peak at E_F disappears. The same behaviour is observed during the exposure of a Cs saturated W(110) surface to oxygen [22]. The structure A is practically absent in MIES. The structure I(5p) is most clearly discernible at the point where the Cs(6s) electrons are consumed by the coadsorbing I atoms. With increasing I coverage the I(5p) peaks are shifting to lower binding energies by about 1.6 eV. Simultaneously the splitting is reduced from 0.95 to 0.65 eV. The centre of the I(5p) feature is finally located at the position, where UPS detects structure A (Fig. 4b). The Cs(5p) peaks are not shifted. The shift of the I(5p) peaks follows the change of the work function which is manifested in the low energy onset of the spectra. Therefore, it follows that additional I atoms are located in positions where no coupling to the substrate, but coupling to the vacuum level takes place.

If at all, the structure I(5p) (clearly visible with MIES) is seen weakly in UPS and appears partly masked by the contribution from the tungsten substrate. At intermediate exposures (marked by I(5p) in Fig. 4a) in MIES the iodine emission appears to be dominated by a species which shows a finestructure splitting of about 0.65 eV. It is pointed out that the structure I(5p) apparently follows the work function
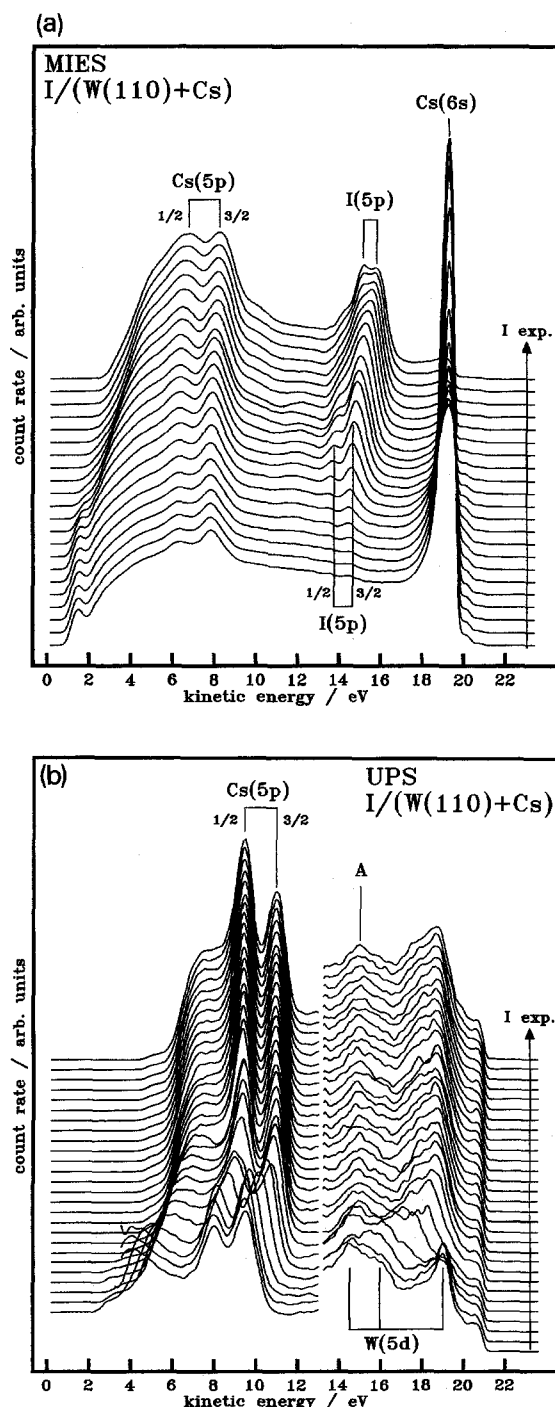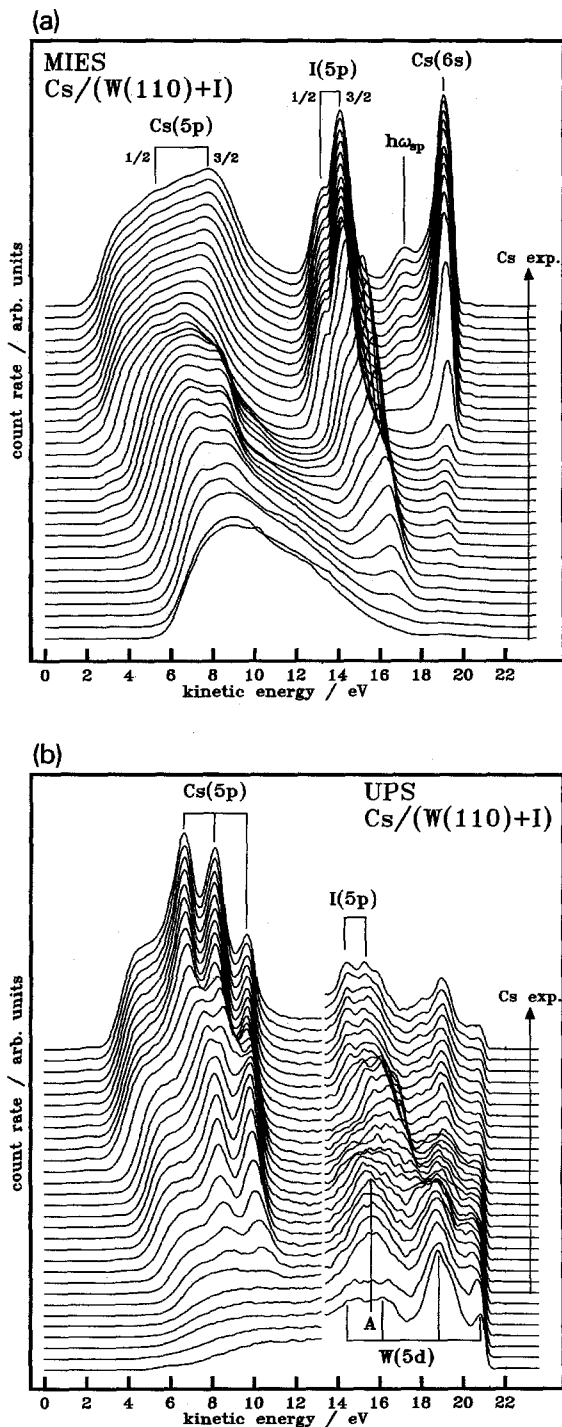

Fig. 4. MIES spectra (a) and UPS spectra (b) during I codeposition on the W(110) surface saturated with 1 ML Cs. The bottom spectrum corresponds to the Cs saturated W(110) surface.

change upon iodine exposure in contrast to structure A which does not move with exposure. This is characteristic for a species which is only weakly coupled to the substrate. The work function of the cesiated surface (1.4 eV at a coverage of 0.6 ML) rises by about 3.6 eV upon iodine exposure. It saturates at about the exposure where the Cs(6s) feature has disappeared.

3.5. Cs / (W(110) + I)

Figs. 5a and 5b show the MIES and UPS results for the adsorption of Cs on an I saturated W(110) surface.

The bottom MIES spectrum shows the W(110) surface saturated with iodine. Upon moderate exposure to Cs a feature due to AD involving iodine atoms ($E_B \approx 4.3$ eV with a shoulder at $E_B \approx 5.2$ eV) develops. The feature shows neither a clear finestructure splitting nor is its shape and energetic position identical with structure A. Its considerable shift with the work function indicates that the responsible species cannot strongly be coupled to the substrate. When the surface starts to show metallic properties, the feature shows indications of finestructure splitting (of about 0.95 eV). Upon cesiation emission near E_F appears which initially is due to AD involving the Cs(6s) electrons. Upon further exposure to Cs the peak Cs(6s) appears due to AU of He^{-*} ($1s2s^2$) (see Fig. 1a). In the top spectra of Fig. 5a a peak in the shoulder of the Cs(6s) peak (denoted by $\hbar\omega_{sp}$) appears with a distance of 2.05 eV. This peak was attributed to the generation of a surface plasmon with an energy loss of 2.1 eV [30]. This gives strong evidence that a Cs multilayer grows up. At least at this point the surface has clearly developed metallic properties. The emission below about 10 eV kinetic energy is mostly due to AD involving the Cs(5p)

Fig. 5. MIES spectra (a) and UPS spectra (b) during Cs codeposition on the W(110) surface saturated with 1 ML I. The bottom MIES spectrum corresponds to the a I saturated W(110) surface. In the UPS spectra I adsorption is completed with the sixth spectrum. Cs coadsorption starts with the seventh (from bottom) UPS spectrum.

electrons. Comparison with the UPS results suggests that contributions from Cs atoms bound to the substrate and to iodine must be present (see below). As for the coadsorption of I on Cs/W(110) a remarkable shift of the I(5p) peaks with the work function takes place; the Cs(5p) peaks are also shifting. The conclusions are the same as above (Section 3.4): the atoms are not coupled to the Fermi level of the substrate, but to the vacuum level.

The UP spectra of the I covered W(110) surface as a function of Cs exposure are rather complex. In order to be able to distinguish between contribution from W(5d) and I the first six spectra show the growth up of the I adlayer from zero to saturation coverage. The I adlayer is complete at the point where the line above A ends. The UPS spectra initially show structure A (the seventh spectrum) well known from Fig. 2. As the MIES spectra show, the surface has developed metallic properties, when the peak Cs(6s) from AU of He^{-*} and the surface plasmon loss peak appear. Under these conditions structure A is replaced (or is at least superimposed) by the structure I(5p). A considerable shift of the features occurs in the region where the surface metallizes correlating with a drastic work function decrease. The spectra display three peaks due to the Cs(5p) ionization. Obviously there exists two sets of $Cs(^2P_{1/2,3/2})$ components. One set of peaks is due to Cs adsorbed on the substrate. The other set – strongly shifting with the work function – is obviously due to Cs which has only weak contact with the surface (such as located on-top of I atoms).

4. Discussion

The adsorption of halogen and oxygen atoms – both being strongly electronegative species – and their coadsorption with alkali atoms, here Cs, on metals can be expected to show close similarities. In the following section the situations will be compared. We can expect to benefit from the oxygen case (which is well studied [22,25,26,32]) as far as the structure of the adlayer and its growth is concerned. However, in the oxygen case there may be complications due to the possibility of molecular adsorption in the presence of alkali atoms.

4.1. Oxygen and halogen on clean W(110)

At room temperature oxygen is well known to adsorb atomically on W(110). UPS displays a peak with a width of about 2 eV centred at $E_B \approx 6$ eV. Normally, the $O(2p_z)$ and $O(2p_{x,y})$ splitting is not resolved [31]. MIES displays a pure AC spectrum [22,29] whose shape is indicative for a drastic reduction of the SDOS near E_F due to oxygenation [29]. The UPS and MIES spectra obtained for I/W(110) show essentially the same behaviour:

● The UPS spectrum displays a peak at $E_B = 5.7$ eV of a width of 1.5 eV;
● The MIES spectrum is a pure AC spectrum which points out again a strong decrease of the SDOS near E_F due to iodine adsorption;
● The deconvolution of I/W(110) data and O/W(110) data show the same drastic decrease of the SDOS just below E_F.

The appearance of AC spectra requires that the He 2s electron can be transferred resonantly into the substrate. This implies that – even for tungsten saturated with iodine – there are empty states present in the substrate into which the transfer can take place. This suggests that the iodine bonds covalently to the surface, e.g. undergoes hybridization with tungsten states.

4.2. Oxygen and iodine coadsorbed with cesium atoms

We do not discuss the features obtained during the early stage of the oxygen exposure (below 1 L) on a cesiated surface. Under these conditions spectra are complicated by the presence of molecular species, such as O_2^-, in the selvedge of the surface [22,32]. Simpler spectra are obtained for exposures beyond 1.5 L. They consist (both with UPS and MIES) of two, only partially resolved peaks, O_a and O_s with $E_B = 6.0$ eV and 8.0 eV, respectively [22]. It appears to be common consensus, that these features (also seen with XPS [32]) are due to photon and metastable induced ionization of atomic oxygen located close to the adsorbate (O_a) and on the substrate (O_s) [22,32]. At the exposure under consideration the strong features near E_F (at $E_{kin} = 19.8$ eV) (see Figs. 1 and 4) denoted by Cs(6s) (also seen during oxygenation) has disappeared completely indicating that the Cs

valence electrons have been transferred into the oxygen 2p-orbital. The MIES spectra essentially suggest that an insulating adlayer has been formed at this stage with a bandgap of the order of 8 eV. Again a similar pattern is observed during the iodine exposure:

● For iodine adsorption on the substrate the feature A is found (in the UPS spectra) (see in particular Figs. 1 and 5b) in which the spin–orbit coupling of the iodine atom is broken up by the strong interaction with the substrate;

● For iodine in close contact with Cs, the (doublet) feature I(5p) is observed (both with UPS and MIES) (see Figs. 3 and 5; late stage of exposure). The iodine atom I($5p^5\,^2P$) produced in the emission process feels only a weak external field when close to a Cs core; consequently the atomic FS-coupling is preserved giving rise to the I(5p) FS-doublet whose components are separated by 0.95 eV.

Thus, we conclude that structure A corresponds to O_s in the oxygen case and I(5p) to O_a. The results suggest that structure A is always seen when the iodine has the possibility for direct contact with the substrate, and forming a covalent bond (as discussed in Section 4.1). This appears also to be the case in the early stage of the exposure to CsI (see Fig. 4 up to the work function minimum), and, of course, in the early stage of Cs adsorption on the I precovered W(110) surface.

On the contrary, structure I(5p) (and, in particular, a well-developed FS-splitting) is seen most clearly when iodine atoms are embedded within an alkali layer (Fig. 5, late stage of exposure to Cs) or adsorbed on top of an alkali layer (Fig. 4a), or when CsI-molecules adsorb undissociated as it is possibly the case in the late stage (beyond the work function minimum in Fig. 3) of CsI exposure.

At present it remains unclear why the species giving rise to structure A is not directly seen with MIES (while MIES efficiently detects the species which produces O_s). The following explanation appears to be plausible:

The iodine species which produces feature A (in UPS) is forming a covalent bond with the substrate (see Section 4.1). The approaching He* is, in a first step, resonantly ionized when interacting with the covalently bound iodine atoms. The electron transfer

is a local process, i.e. it occurs between the He* projectile and the iodine adsorbate. Therefore, it is implied that there are empty states above E_F at the adsorbate which give the possibility for a local tunnel process of the He 2s electron into the substrate.

In contrast, the oxygen adsorbate (at least when coadsorbed with alkali atoms) captures two surface electrons thus completing its 2p-shell. The resulting negative ion's bond with the surface is highly ionic. Therefore no empty states allowing resonant tunnelling of the He 2s electrons are available. Thus, He* becomes deexcited via Auger deexcitation.

Support for this scenario comes from results for the neutralization of slow D^+ ions interacting with various adsorbates on W(110) and Si(100) [33,34]. It was found that the probability for the resonant transfer of surface electrons into the 1s-hole of D^+ is strongly reduced when the adsorbate bond has predominantly ionic character such as in KI [33] and CsCl [34]. On the other hand, the resonant transfer of electrons to the projectile was found to be efficient as long as the adsorbate forms a covalent bond with the substrate. As discussed above, the ionically bound adsorbate shields the projectile ion from being neutralized by resonant transfer of surface electrons. In quoting the neutralization behaviour of D^+ ions at this point we suppose that the same argumentation can be used when the electron transfer takes place into the opposite direction, and, moreover, that there exists no essential difference between the D 1s and the He 2s orbitals as far as the electron transfer is concerned.

5. Summary

The electron emission from W(110) exposed to pure iodine and to cesium and iodine simultaneously at room temperature was studied with photoelectron spectroscopy (UPS with HeI) and metastable impact electron spectroscopy (MIES with He*(2^3S)). The iodine induced emission does sensitively depend on the chemical environment of the iodine adsorbate: in the UPS spectra iodine atoms interacting with the tungsten substrate give rise to emission which reflects the Stark splitting of the I(5p) orbital in the strong external field produced by the I–W interaction. The contribution of such iodine atoms to the

MIES spectra is due to the Auger Capture process after resonance ionization of the He* probe atoms. Iodine atoms adsorbed near to cesium feel a comparatively weak external field: the UPS – as well as the Auger Deexcitation MIES spectra – display a double peak reflecting the I(5p) fine structure splitting (0.95 eV) in the iodine final state.

The spectra are compared to those obtained during the oxygenation of clean and partially cesiated W(110) and show close similarities: in both situations the spectra are caused by the electron emission involving iodine (oxygen) atomically adsorbed on the substrate or close to the cesium atoms.

Acknowledgement

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