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Kinetic electron emission from Al, Cu, and Au surfaces exposed to oxygen

O. Benka and J. Pürstinger
Institut für Experimentalphysik, University Linz, A-4040 Linz, Austria

A. Koyama
The Institute of Physical and Chemical Research, Wako, Saitama 351-01, Japan
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Kinetic electron emission is studied for the impact of 3-keV electrons and of 4-MeV He^{2+} ions on polycrystalline Al, Cu, and Au targets, as a function of oxygen exposure. For electron-induced emission, the yield and the change of the work function is measured, whereas for ions the distribution of the number of emitted electrons is measured. The distributions found are very well fitted by Pólya distributions, giving the ion-induced emission yield and the Pólya parameter b. For Au targets, no influence of oxygen exposure upon electron emission was found. For Al targets, exposure up to 50 L gives a reduction of the emission yield; further exposure results in oxidation of aluminum and increases the yield. The work function is found to decrease with increasing oxygen exposure. The parameters b of the Pólya distributions are almost independent of oxygen exposure. For Cu targets, oxygen exposure gives a decreasing emission yield, and a slightly increasing work function. The results are discussed in terms of existing models of oxygen adsorption. [S1050-2947(97)07510-0]

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I. INTRODUCTION

In recent years, kinetic electron emission has been investigated in detail for the impact of ions on metals [1–5], whereas electron emission induced by the impact of electrons (secondary electron emission) was studied much less. Kinetic electron emission is sensitive to the work function of the target. Therefore, not contaminated, clean target surfaces are required for reliable results. These can be obtained using ultrahigh vacuum (UHV) and target cleaning and target controlling devices (sputter gun and Auger spectrometer). Unfortunately many of the older investigations do not fulfill these requirements [6].

The influence of adsorbates on kinetic ion-induced electron emission (IEE) and electron-induced electron emission (EEE) has been rarely investigated so far. Some authors [1,7] claim that the electron emission yield of clean metal surfaces is always lower than the yield of contaminated surfaces. On the other hand we found for Cu targets that a clean surface gives a higher yield compared to a contaminated surface [3]. Oxide layers on metals or semiconductors were found to increase the yield [8,9], in agreement with expectations, because insulators have in general a higher electron emission (EE) yield than metals [6]. For aluminum a large increase of IEE emitted at an angle of 48° to the surface normal was found when Na was deposited on the surface [10]. It is proposed that this increase of IEE is due a to change of the work function.

The effect of gas adsorption on clean C foils on the IEE yield was studied by Arrale *et al.* [11] for the impact of 2-MeV F^{3+} ions. Oxygen and nitrogen increased the yield, whereas water vapor decreased it slightly. Ferron *et al.* [12] investigated the effect of oxygen exposure of clean Al and Mo surfaces on the IEE yield induced by 2–60-keV Ar^{+} ions. For Al they found an increase of the yield for increasing oxygen exposure, and they concluded that this increase is not due to a change of the work function. Tucek and Champion [13] studied also the effect of oxygen exposure of aluminum on IEE for impact of Na^{+} ions with energies below 500 eV. They found also an increasing yield for increasing oxygen exposure, but the increase is much smaller than the increase found by Ferron *et al.* [12]. They proposed that the enhanced electron emission is due to excitation of adsorbed oxygen atoms.

The purpose of this study is to investigate in detail the influence of oxygen exposure and beginning oxidation of metals on the kinetic EE. The projectiles used are 3-keV electrons and 4-MeV He^{2+} ions, both of which excite target electrons mainly by direct Coulomb interaction. Therefore similar effects are expected for both projectiles. The EE yield is measured for clean polycrystalline metal surfaces that are gradually exposed to oxygen. In addition, the distribution of the number of emitted electrons, which can provide an estimate of the contribution of cascade electrons to the total EE [14], is studied for ion impact . The results of the EE yield measurements are discussed with respect to the results obtained by other surface characterization methods [15–17].

II. EXPERIMENT

The measurements were performed in an UHV chamber with a base pressure of 3×10^{-10} mbar. The targets were produced by evaporation on silicon or on polished stainless steel backings. After preparation the targets were moved to a manipulator in the UHV chamber without breaking the vacuum. The thickness of the evaporated layers was about 100 μg/cm^2, which is much larger than the mean escape depth of electrons. Before measurements all targets were sputter cleaned using 3-keV Ar ions until no carbon or oxygen contamination was visible in the Auger electron spectrum (AES). Other contaminations were not found. This gives an upper limit for contaminations of about 0.02 ML. After beginning the measurements, the oxygen partial pressure was increased in the UHV chamber by a leak valve to

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the range of 10^{-9} mbar. Subsequently measurements were performed for different oxygen exposures. The composition of the surface layer as a function of oxygen exposure was investigated by Auger spectroscopy.

The electron gun of the Auger electron spectrometer was used to measure changes of the target work function. The target potential was set to 36 V, and the low-energy peak of the emitted electrons was measured. The lowest electron energy measured yields the difference of the work functions of the target and of the spectrometer. For evaluation the energy was taken at half intensity of the peak on the low-energy side (E_W). A change of E_W following exposure to oxygen gives the change of work function, if the work function of the spectrometer is not influenced by oxygen exposure. For Au targets no change of E_W was observed for increased oxygen pressure; therefore the influence of oxygen exposure on the work function of the spectrometer was neglected. By this method, we can determine changes of work function to an accuracy of about 0.05 eV. Here local work functions at the place of electron emission are measured, not the average values found by the frequently used more accurate Kelvin method [15,18].

3-keV electrons from the electron gun of the Auger spectrometer were used to measure the EEE yield. The angle between the electron beam and the surface normal was 30°. At a constant primary electron beam current, the target current was measured for a target potential of both +36 V (I_+) and −36 V (I_-). The EEE yield was then given by

$$\gamma = 1 - \frac{I_-}{I_+}.$$

The change of the electron-impact energy due to the additional target potential (±36 V) was considered negligible. Since the positive voltage of 36 V might be too low to suppress emission of all electrons from the target and since reflected primary electrons could also contribute I_+, the accuracy of the absolute value of the yield γ was estimated to be about 25%. The reproducibility of yield measurements was about 0.5%, and the relative yield (with respect to the yield of the clean metal surface) had an accuracy of about 0.5%.

The IEE was measured using the emission statistics method. The experimental setup and evaluation of the measured data are described in detail in Ref. [19]. 4-MeV He^{2+} projectiles were obtained from the 1.6-MV tandem accelerator of Linz University. Here, the target potential was −19 kV, so that the emitted electrons were accelerated and focused to a solid-state detector (PIPS type). From the measured electron spectra, emission amplitudes C_n were determined giving the number of events for emission of $n = 1,2 \ldots$ electrons. All measured distributions of C_n could be fitted very well using Pólya distributions. From the fitted Pólya distributions, the mean number of emitted electrons, the emission yield γ, and the Pólya parameter b were obtained. The Pólya parameter b describes the deviation from a Poisson distribution (for $b=0$ the Pólya distribution becomes a Poisson distribution).

An advantage of the emission statistics method for investigating EE is that almost every ion impinging on the target is detected by the emitted electrons; therefore a very small ion beam current is sufficient. A counting rate of 10^3 events per second would correspond to a beam current of about 10^{-16} A. Such a small ion current will hardly influence the surface conditions and the adsorbed gas layers.

III. RESULTS

A. Aluminum

Aluminum targets were first sputter cleaned until C and O peaks were no longer visible in the Auger spectrum. Then the partial pressure of oxygen was increased to about 1×10^{-8} mbar and kept constant while Auger spectra were measured. In the differentiated energy spectra the peak heights (difference maximum to minimum) were evaluated for O (*KLL*) and for Al (*LVV*) transitions. For Al, two contributions could be separated, a line at 66 eV corresponding to a transition in metallic Al and a line at 53 eV, which appeared after longer oxygen exposure, due to Al *LVV* transitions of aluminum oxide. In Fig. 1 the dependence of the

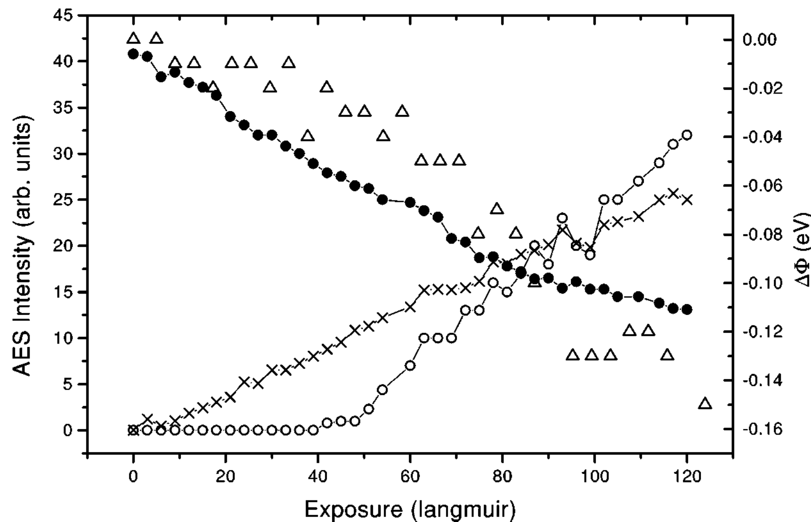

FIG. 1. Auger line intensities (difference maximum to minimum in differentiated energy spectrum) for metallic Al (–●–) (*LVV* at 66 eV, the values are divided by 10), for oxidized Al (–○–) (*LVV* at 53 eV), for O (–×–), and the change of work function ΔΦ (△) vs O exposure.

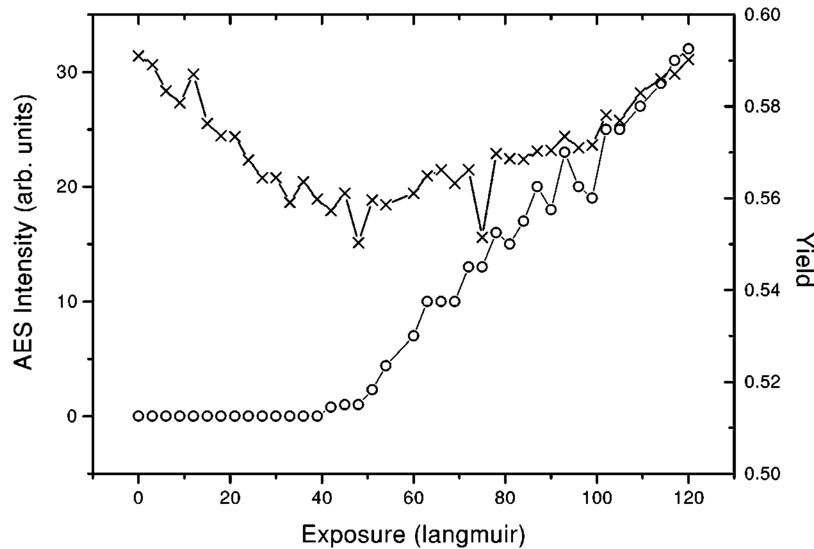

FIG. 2. Electron emission yield (–×–) and Auger line intensity of oxidized Al (–○–) for the impact of 3-keV electrons on aluminum vs O exposure.

individual peak heights on the oxygen exposure is shown. It can be seen that the oxygen peak increases almost linearly with time, but the aluminum oxide line becomes visible only after an exposure of about 50 L. On the other hand the metallic Al line decreases from the very beginning. Evidently, there are two phases of oxygen adsorption: phase (1) when oxygen is adsorbed without oxidizing Al and phase (2) when aluminum is oxidized.

Figure 1 shows also the change of work function $\Delta\Phi$, relative to the work function of clean aluminum, as a function of O exposure. A decreasing work function was found for increasing oxygen exposure. In phase (1) (under 40 L) the rate of decrease ($d\Delta\Phi/dL$) is slightly smaller than in phase (2). For oxygen exposures larger than shown in Fig. 1 the work function is found to decrease slightly further and $\Delta\Phi$ approaches a value of about -0.35 eV.

The shape of the low-energy EEE spectra was not significantly changed by oxygen exposure. Within the uncertainty of measurement (5%), no change of the width [full width at half maximum (FWHM)] of the low-energy peak was found for low oxygen exposure (up to 50 L). For large oxygen exposure a decrease of about 10% of the width was observed. This is in agreement with the known fact that the width of the low-energy peak of insulators is smaller than that of metals [6].

The measured electron emission yield induced by 3-keV electrons is given in Fig. 2, together with the oxidized Al (53 eV) Auger line, as a function of O exposure. It can be seen that the yield decreases at first slightly to a minimum value at an O exposure close to the onset of phase (2), then the yield increases. Even at an exposure as large as 10 000 L the yield still increases with increasing O exposure and does not approach a constant value. At these large O exposures the Auger peaks of O and Al (53 eV) converge to a constant value. Long series of measurements of Auger spectra or EEE yield caused problems, because enhanced aluminum oxidation was found at intensely bombarded target areas. Therefore the impact position of the electron beam on the target was often changed during long measurements.

EEE yield as a function of O exposure was also measured at different partial pressures. The exposure that gives mini-

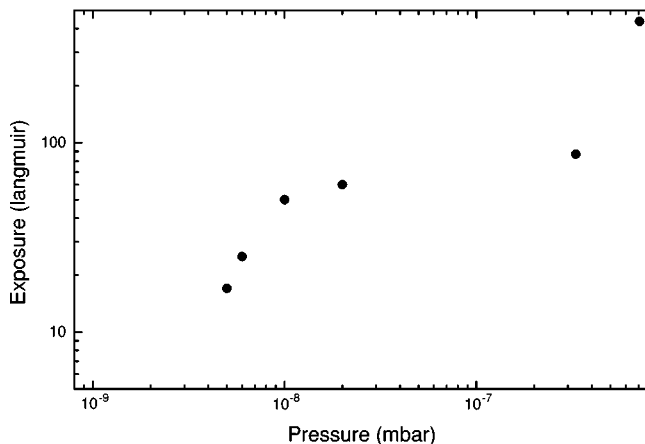

FIG. 3. The O exposure giving minimum EE yield, vs O pressure.

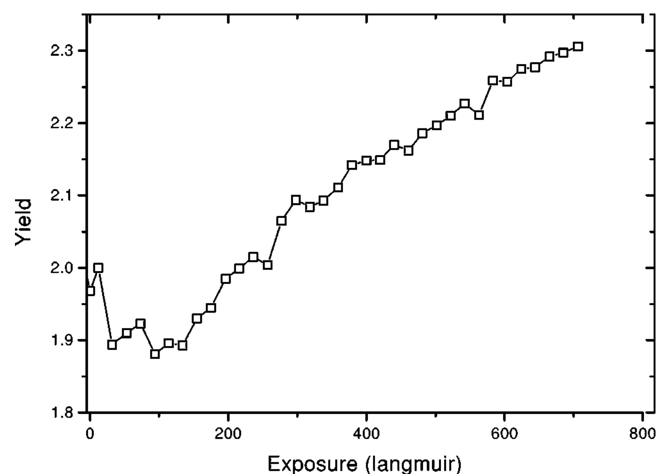

FIG. 4. Electron emission yield of Al for impact of 4-MeV He^{2+} ions, vs O exposure.

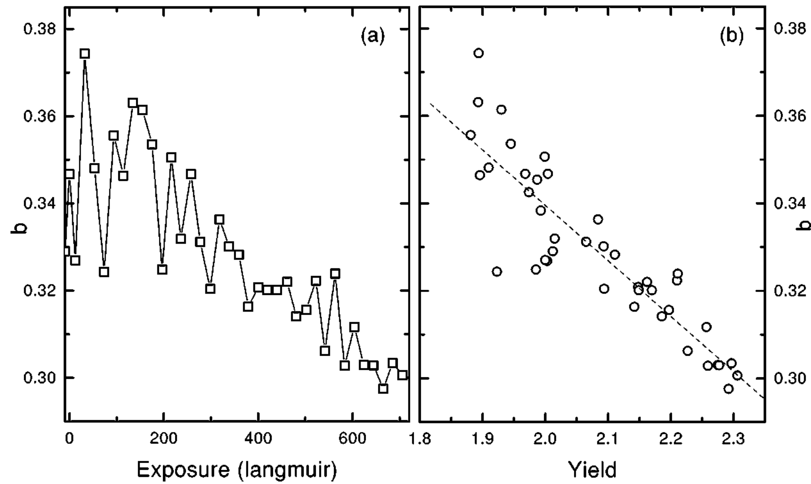

FIG. 5. Pólya parameter b of the distribution of the number of emitted electrons for the impact of 4-MeV He^{2+} ions on Al (a) vs O exposure; (b) vs emission yield. The dashed line gives a linear fit.

mum yield ($L_{\min}$) was about 50 L at about 10^{-8} mbar, but at higher pressures (10^{-7} mbar) the minimum yield occurred at higher exposures. Figure 3 gives $L_{\min}$ as a function of O pressure.

IEE yield versus O exposure is shown in Fig. 4 for impact of 4-MeV He^{2+} ions. It can be seen that the IEE yield is similar to that of the EEE yield. The IEE yield was obtained from emission statistics discussed above. The parameter b of the fitted Pólya distributions is shown versus O exposure in Fig. 5. The error of b is estimated at about 10%. The variation of b is small, and opposite to the variation of the yield. Figure 5 shows the b values versus yields, corresponding to different O exposures. It can be seen that there is roughly a linear relation with $\Delta b/\Delta\gamma = -0.13$.

B. Copper and gold

For Cu the height of the O *KLL* Auger peak and the change of the work function for 3-keV electrons is shown versus O exposure in Fig. 6. Here, both the O peak height and the work function increase with increasing O exposure. This is contrary to the behavior of Al. The shape of the 60-eV *MVV* line of Cu did not change with increasing O exposure in contrast to aluminum, where the Auger transitions involving valence electrons indicated an oxide state.

The EEE yield is given in Fig. 7 for impact of 3-keV electrons. It can be seen that the yield decreases slightly with increasing O exposure. A continued increase of the yield at larger O exposures, as found for aluminum, was not observed.

For Au targets, the Auger spectrum was found to be unchanged with increasing O exposure. An O Auger peak was not found. The EEE and the IEE yields were also found to be constant, independent of O exposure.

IV. DISCUSSION

A. Aluminum

The adsorption of oxygen on aluminum and the subsequent oxidation were studied in recent years for single-crystal surfaces [17,18,20] and recently also for polycrystalline aluminum [16]. Arranz and Palacio [16] investigated in detail Auger spectra for aluminum exposed to oxygen. For evaluation they used a factor analysis method. Their results are in good agreement with the results of Auger measure-

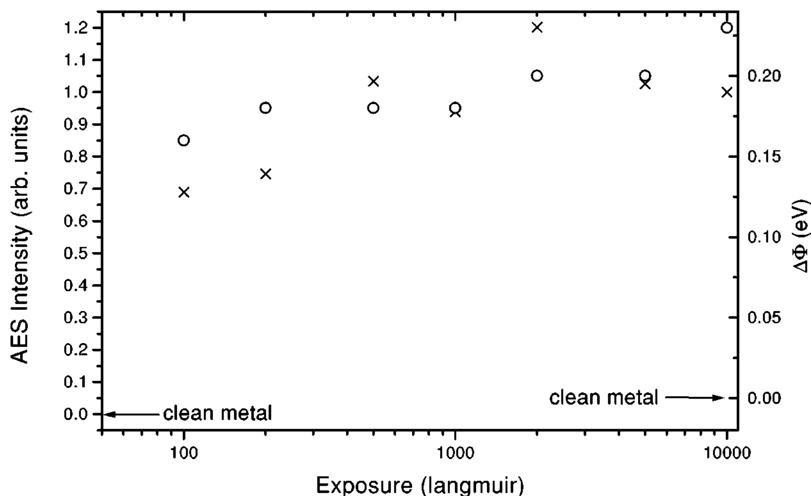

FIG. 6. Auger line intensity of oxygen (×) and change of work function $\Delta\Phi$ (○) for impact of 3-keV electrons on Cu vs O exposure.

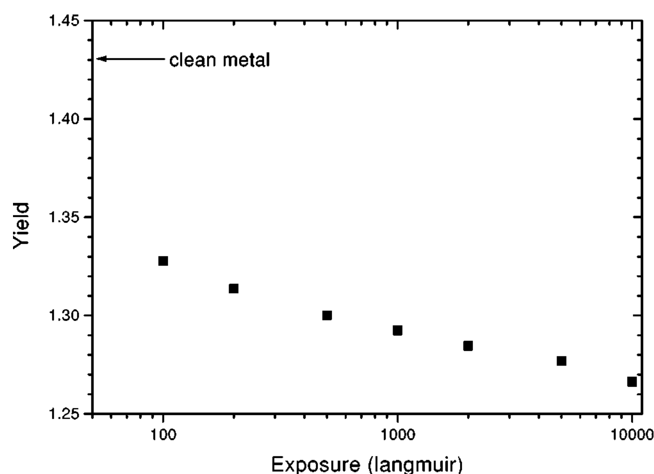

FIG. 7. Electron emission yield for impact of 3-keV electrons on Cu vs O exposure.

ments of this investigation. Exposure of oxygen until about 50 L [phase (1)] leads mainly to adsorption in a chemisorbed state. The chemisorbed oxygen gives rise to an oxygen Auger line and decreases the intensity of the metallic Auger line. At 50-L exposures only less than 1 ML O is adsorbed at the surface because of small sticking coefficient (about 0.01 [17]) for oxygen on aluminum. With further oxygen exposure oxidation of aluminum starts [phase (2)] and the aluminum *LVV* line of aluminum oxide appears in the Auger spectrum at lower energy compared to the line of metallic aluminum.

Chemisorbed oxygen is expected to increase the work function, because of transfer of negative charge to adsorbed O atoms [17]. But for polycrystalline Al [22] and recently for single-crystal Al surfaces [18,21], the work function was always found to decrease with increasing oxygen exposure. For single crystals the amount of decrease depends on the type of crystal surface. The largest decrease is found for the (100) surface of more than 1.2 eV. The change of work function for polycrystalline aluminum found in our work is in agreement with results of Michel *et al.* [18] for the (111) surface, where chemisorbed O gives only a small decrease of work function, while for aluminum oxide a decrease of about 0.3 eV is found. The question is still open why chemisorbed oxygen [phase (1)] does not increase the work function [17].

To understand kinetic electron emission, one can divide the process into three steps [6]: (1) production of primary electrons by ionization of target atoms by projectiles and by decay of plasmons excited by projectiles; (2) production of secondary electrons by fast primary and secondary electrons (cascade electrons), transport of electrons to the target surface; (3) escape of electrons with enough energy from the target surface.

The well-known fact that the EE yield of metal oxides is higher then that of pure metals [6,23] can be qualitatively understood on this basis: In step (1), mainly atomic processes at high energies are important, which are insensitive to changes of binding energies of the order of 10 eV. The difference is attributed to step (2), where the absorption length of low-energy electrons is a dominating factor. Low-energy (<10 eV) electrons have a much larger mean free path in metal oxides, because of the large band gap, than in metals. Therefore also electrons excited at a large depth can contribute to electron emission in metal oxides. Step (3) is influenced by the work function of the target. Decreasing work functions would enhance the probability for escape of electrons from the target. As the mean energy of electrons emitted from metals is about 5–10 eV [6], changes of work function of the order of 1 eV have only a minor influence on the EE yield. Therefore the large increase of the EE yield for oxides compared to metal is in the main not due to a change of work function, but it could contribute.

Thus an increase of EE yield for electron and ion excitation found in phase (2) beyond about 50-L oxygen exposure compared to metals can be explained by the large band gap of aluminum oxide, which increases the escape depth for electrons and, to a lesser extent, by the decrease of work function.

The decreasing EE yield for increasing O exposure in the chemisorbed phase (1) would be in agreement with an expected increasing work function [17]. But the measured slight decrease of work function for polycrystalline Al is in contradiction to expectations. Therefore the decreasing yield cannot be explained by a change of work function [step (3)]. But a chemisorbed O atom (O_{ad}) will have an influence on both steps (1) and (2). It is known [17] that oxygen molecules are not adsorbed as such, but after dissociation as individual O atoms accumulating negative charge. In step (1) the O_{ad} atoms can be ionized by the projectile and contribute to primary electron production. The IEE yield is known to be roughly proportional to the energy deposited by the projectiles in a surface layer [6]. The mean energy deposited per atom is described by the stopping cross section which gives for 4-MeV He ion impact on O a value of 23 $\mathrm{eV\,cm^2/10^{15}}$ atoms and for impact on Al 31 $\mathrm{eV\,cm^2/10^{15}}$ atoms [24]. The larger stopping cross section for Al compared to O is roughly due to the larger number of bound electrons. Therefore it can be concluded that, in step (1), an O atom will contribute less to electron emission than an Al atom.

In step (2) O_{ad} will contribute to scattering and energy loss of the low-energy secondary electrons. Because low-energy electrons can transfer energy by scattering mainly to valence electrons, the electronic state of target or adsorbed atoms is important for step (2). It is assumed [17,13] that O_{ad} accumulates negative charge and therefore its electronic state will be close to that of an O^- ion. Because the electron affinity for O^- ions is only 1.4 eV, O^- ions can take even small amounts of energy from secondary electrons and contribute considerably to energy loss.

If we assume that the contribution of an O_{ad} atom to scattering and energy loss to low-energy electrons [step (2)] is roughly equal to that of an Al atom, but the contribution to electron production is less than an Al atom [step (1)], a decrease of the EE yield can be expected for increasing O exposure, as found experimentally for the adsorption phase (1).

According to this model an O atom in aluminum oxide will also contribute less to electron production than an Al atom [step (1)], but because of the large band gap of aluminum oxide it will also contribute only little to the energy loss of secondary electrons [step (2)], which gives a larger EE yield for aluminum oxide than for metallic aluminum.

Fast light ions and electrons (velocity larger than Bohr velocity) excite target electrons mainly by direct Coulomb interaction [25] [step (1)]. Production of cascade electrons, transport, and escape of electrons from the target surface [steps (2) and (3)] should not depend on the projectile. Therefore it is expected that the effect of oxygen exposure on the yield is the same for electron and He^{2+} ion impact. The experimental results confirm this model.

Slow heavy ions with bound electrons excite target electrons mainly by other mechanisms (e.g., by level crossing). This fact may explain why in previous investigations using slow heavy-ion impact [12,13] no decrease of the EE yield for low oxygen exposure [phase (1)] was observed.

In our model for the emission statistics of IEE [14] we proposed that the b parameter of the Pólya distributions describing emission statistics is a measure of the contribution of secondary (cascade) electrons to total electron emission (primary electrons excited by the projectile and secondary electrons). If it is now assumed that the difference of energy loss for low-energy electrons [step (2)] for different oxygen chemical states (chemisorbed and oxide) is the same for primary and secondary low-energy electrons and that the change of electron emission after oxygen exposure is mainly due to change of energy loss for low-energy electrons, then the b parameter of Pólya distribution should not depend on the O exposure.

The change of IEE yield $\Delta\gamma$ and of the b parameter Δb due to O exposure can be compared to the changes due to a variation of projectile energy. Projectiles with higher energies will have a smaller probability for target ionization, thus producing a smaller amount of primary electrons and a smaller emission yield. Step (2), when cascade electrons are produced, and step (3) will not be influenced by a change of projectile energy. The contribution of primary electrons is therefore smaller for higher projectile energies, giving a larger value of the parameter b [14].

According to the proposed model [14], a change of the yield due to a projectile energy change should also change b, but a change of yield due to O exposure should not change b. For 4 MeV He^{2+} impact a slope $\Delta b/\Delta\gamma = -0.32$ is obtained for changing projectile energies [14], whereas a slope $\Delta b/\Delta\gamma = -0.13$ is found for O exposure in this work (Fig. 5). This difference is in agreement with the proposed model. A more detailed model than the proposed one [14] for emission statistics of EE would be necessary for understanding the small dependence of b on the O exposure.

In previous studies of O exposure on aluminum [17,18,20] the dependence of the onset of aluminum oxidation on the oxygen pressure was not investigated. As discussed above, the minimum of EE yield is an indication that at this point oxidation of aluminum is becoming important. Therefore dependence of L_{min} on O pressure found (Fig. 3) is an indication that the onset of aluminum oxidation depends on the O pressure. If the onset of aluminum oxidation does not only depend on the amount of adsorbed O but also on the time of exposure, a dependence of L_{min} on the pressure is expected. A larger O pressure will give the same exposure L_{min} in a shorter time. But if this shorter exposure time is not large enough to start oxidation, then oxidation will commence at a later time, resulting in a larger value L_{min}. Therefore the dependence of L_{min} on the pressure found may be an indication that the onset of aluminum oxidation depends not only on the amount of O exposure, but also on time.

B. Gold and copper

Gold was used as a reference target. As expected, no change of work function and no change of yield due to O exposure was found. This proves that our results for other target materials are indeed target properties, and do not have systematic errors depending on O pressure.

For Cu the decreasing EE yield and increasing work function for increasing O exposure is an indication that O is only chemisorbed on Cu and copper is not oxidized, corresponding to phase (1) of Al. The increase of work function found is in agreement with published data for single-crystal surfaces [15,26]. It is also found for Cu single-crystal surfaces that low pressure oxygen exposure as high as 10^5 L does not produce an oxide layer [15]. For an explanation of the decreasing EE yield the same model as proposed for Al can be applied.

Because of the increased work function it can be assumed that the adsorbed O is accumulating negative charge, forming a state similar to an O^- ion. In step (1), the O^- ion will lead to a decreased electron production compared to that of a Cu atom, because the number of bound electrons is smaller for O^- ions than for Cu atoms giving also a smaller stopping cross section. The energy loss of low-energy electrons [step (2)] can hardly be estimated for O^- ions compared to metallic Cu atoms. Both have the possibility to take a small amount of energy from secondary electrons and contribute to energy loss. The increased work function will decrease the escape probability for EE [step (3)] resulting in a lower EE yield. Therefore the adsorbed O^- ions may only contribute in step (2) to an increased yield, but taking also steps (1) and (3) into account a slightly decreasing EE yield for increasing O exposure may be expected, as experimentally observed.

V. CONCLUSION

The influence of oxygen exposure upon kinetic electron emission was measured for metal surfaces. For Au the EE yield remained constant, whereas for Al a nonlinear dependence of the EE yield upon O exposure was found. For small O exposures the yield decreased and a minimum yield was obtained, when aluminum oxidation began. Further aluminum oxidation increased the yield. Incipient aluminum oxidation was determined by Auger spectroscopy. It is proposed that the different effects of oxygen on the EE yield for adsorbed O and for O in aluminum oxide are mainly due to differences in energy-loss contributions to low-energy (<10 eV) electrons, because of different valence electron energies. Therefore EE yield measurements can provide information on the electronical (chemical) state of adsorbed atoms on surfaces. For Cu the decreasing yield for increasing O exposure indicates that O is adsorbed not in the state of copper oxide, but in a state with low excitation levels like that of O^- ions.

Measurements of EEE yield can be performed in a short time, therefore this technique may be useful to study fast adsorption processes on surfaces. First measurements of yield change for O exposure at higher O partial pressure gave

indications of a time-dependent oxidation process. Improvements of the experimental setup are in progress that should make faster measurements of EE yield possible. Further investigations of the pressure and temperature dependence of O adsorption on metals by electron emission are planned.

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