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SPURIOUS EFFECTS OF ELECTRON EMISSION FROM THE GRIDS OF A RETARDING FIELD ANALYSER ON SECONDARY ELECTRON EMISSION MEASUREMENTS. RESULTS ON A (111) COPPER SINGLE CRYSTAL

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Résumé. — On étudie les effets parasites d'un système analyseur 4 grilles à champ retardateur dans les mesures d'émission d'électrons secondaires de faible énergie.

Leur comportement est étudié et les deux pics qui apparaissent dans le spectre énergétique sont interprétés comme résultant d'une émission tertiaire par les grilles. On montre qu'il est nécessaire de séparer le pic électronique secondaire vrai de ces pics parasites.

On donne le spectre et les rendements σ et η pour un cristal (111) de cuivre de propreté contrôlée par spectroscopie Auger.

Abstract. — Spurious effects of a four grid retarding field analyser were studied for low energy secondary electron measurements. Their behaviour was investigated and two peaks in the energy spectrum were interpreted as resulting from tertiary electrons from the grids. It was shown that the true secondary electron peak has to be separated from these spurious peaks. The spectrum and the yields σ and η obtained for a Cu (111) crystal after a surface cleanliness control by Auger spectroscopy are given.

1. Introduction. — Secondary electrons emitted with an energy of a few electron volts come from the surface atomic layers. The emission is thus very sensitive to the nature of the surface. The vacuum quality and surface preparation as controlled by Auger spectroscopy are then decisive tests of measurement quality. The progress in vacuum techniques and the development of investigation methods for surfaces provide more precise values for secondary electron emission. However there are still differences in the energy distribution of the true secondary electrons as given by different authors. Also theoretical studies [1] prove that we must consider, simultaneously all the *observables* of the phenomenon. With a view of reducing these differences we measured the energy distribution of the secondary electron emission of a copper single crystal (111). These studies, which were carried out with a retarding field Auger spectroscope showed that, in the low energy region of the spectrum, it was imperative to separate the spurious effects from the true phenomenon. If this is not done, the spectrum may be greatly distorted.

2. Apparatus and experimental procedures. — The measurement device is a standard 4 grid Vacuum Generators Auger spectroscope (Fig. 1). Baking at 300 °C gave a 10^{-10} torr vacuum so introducing negligible surface contamination by residual gases.

FIG. 1. — Four grid device.

The target is bombarded at normal incidence. The electron gun produces a primary electron beam accelerated with a variable voltage with energies from a few eV to 2 500 eV and with an intensity smaller than

2 μ A. During the measurements, the variation of the primary current intensity is less than 1 %. The analysis system grids are made of stainless steel wires 0,04 mm in diameter (31 meshes per cm), each of them having 75 % transparency.

Usually the G_4 grid is earthed and in this case, ensures the existence of a free-field space around the target. A potential V_R is applied to the G_3 grid allowing the passage of only those electrons with an energy E_s higher than eV_R . In fact, in order to improve the apparatus resolution (up to 1.4 % for a primary energy of 100 eV) the G_3 grid is split into G_{3A} and G_{3B} . The G_2 grid is earthed eliminating tertiary electron emission by the collector. It also reduces the capacity coupling between G_3 and the collector. The collector accelerates the electrons that have passed through the grid system. The energy distribution $N(E)$

and its derivative are obtained by modulating the G_3 grid.

The target (6 mm in diameter and 1.5 mm thick) was sliced out by electro-erosion from single crystal bar of copper (5 N) oriented by the Laué method. The analysed surface was mechanically polished with a 1 μ diamond paste then electrolytically polished in a 66 % orthophosphoric acid solution. After rinsing in distilled water and drying, the target was immediately placed in vacuum. The target undergoes an in-situ cleaning consisting of a series of argon ion sputtering (accelerating voltage : 430 eV, current density : 2 μ A/cm²) and annealing (400 °C) cycles. The surface cleanness is controlled by Auger spectroscopy. The spectrum shown (Fig. 2A) was obtained after three cycles. On the same figure we also presented the complete energy spectrum for $E_p = 200$ eV (Fig. 2B).

FIG. 2A. — Auger spectra of Cu (111). ($E_p = 1\,500$ eV ; V_{pp} : (a) : 2 V ; (b) : 10 V.)

FIG. 2B. — Energy spectrum of Cu (111) ($E_p = 200$ eV).

3. **Spurious grid effects.** — The presence of parasite peaks in the energy distribution $N(E)$ was first reported by Morisson and Lander [2]. More recently, Wei *et al.* [3], Cross [4], Shimizu [5] reported similar observations. Considering the importance of these phenomena we measured the influence of the peaks on the energy distribution of the true secondary electrons. The measurements were made on a (111) crystal copper target before cleaning with a 300 eV primary energy in a 2×10^{-10} torr vacuum with a 30 mV modulation voltage amplitude.

3.1 **SHIFTS WITH THE VARIATION OF THE GRID VOLTAGES.** — The normal spectrum $N(E)$ (Fig. 3) obtained for negative values of V_{G_3} ($V_{G_3} = -E$) is completed by the signal obtained for positive values of this potential. The curve (1) was obtained for $V_{G_4} = V_{G_2} = 0$ V and has a *positive* peak at $V_{G_3} = 0.3$ V and a *negative* peak at $V_{G_3} = 1.4$ V. On the same curve we observe that the negative peak is shifted when the G_2 voltage changes from 0 V to 15 V (spectra (2), (3), (4)), its extremum corresponding to $V_{G_3} = V_{G_2} + 1.4$ V. The energy distribution of the

FIG. 3. — Shifts of the negative peak with G_2 grid voltages.

FIG. 4. — Shifts of the positive peak with G_4 grid voltages.

true secondary electrons is considerably modified when V_{G_2} varies from 0 to 5 V. On the other hand, the extremum and the half height width do not change when V_{G_2} varies from 5 to 15 V. The positive peak position also depends on V_{G_4} as shown on figure 4 obtained for $V_{G_2} = 20$ V. Then the peak position is given by $V_{G_3} = V_{G_4} + 0.3$ V. The energy distribution is greatly modified only when V_{G_3} varies from 0 to 5 V (Curves (1) and (2)). The intensity of the distribution-maxima increases with V_{G_4} , probably related to an increase of the collected current.

3.2 INTERPRETATION. — For positive values of V_{G_3} , the positive and negative peaks express respectively an increase or a decrease of the collector current. Cross [4], using a 3 grid analyser, obtained only a positive peak and attributed these variations to tertiary emission of the grids. As for the presence and the shift of the two peaks, our observations confirm those of Wei *et al.* who attributed them to field penetration i. e. to a transparency variation of the filtering grid. On figure 5, we reproduce the spectrum 3 from figure 4 and the corresponding variations with V_{G_3} of the grid currents i_{G_4} and i_{G_2} . For $V_{G_3} > 0$, there is a good agreement between the decrease in i_{G_4} , the increase in i_{G_2} , and the peak position. That is to say a more important number of electrons going through G_3 are able to reach G_2 and the collector, increasing i_{G_2} and I_c . From these measurements we were able to calculate $-di_{G_4}/dV_{G_3}$ and dI_c/dV_{G_3} and verify the agreement between these two quantities. In fact dI_c/dV_{G_3} in the energy distribution and in this range corresponds to the positive peak.

Origin of the i_{G_4} variation. — Initially rejected by

Wei *et al.* on the ground of the relative importance of the parasite peaks compared with the true peak, the tertiary emission hypothesis was developed by Cross who compared the principal peak intensity $N_1(E)$ and the positive parasite peak intensity $N_2(E)$ and attributed the latter to the emission of grids G_3 and G_4 . In fact, as we have pointed out, the emission of grid G_3 generates the negative peak and does not contribute to the positive peak. Figure 6 describes the mechanisms of creation and shift of the different peaks for a four grid analyser. Potentials barriers $-eV_G$ are referenced to the target level. The creation of positive and negative peaks is described for positive values of V_{G_4} and V_{G_2} with $V_{G_2} > V_{G_4}$. On the schematic diagram the abscissa and the variable potential barrier $-eV_{G_3}$ were put at the same level for three intermediate characteristic positions 1, 2, 3 corresponding respectively to the recording of secondary, positive and negative peaks. The energy distribution of the secondary electron is obtained when $-eV_{G_3}$ is slightly higher than the target level (position 1). Filtering of tertiary electrons from grid G_4 by grid G_3 occurs when the variable barrier $-eV_{G_3}$ is slightly higher than the constant barrier $-eV_{G_4}$ (position 2). We can then explain the positive peak recording whose position is localized for $V_{G_3} = V_{G_4}$. A variation of this voltage gives an equal shift of the parasite peak. The filtering of tertiary electrons from G_{3A} and G_{3B} by grid G_2 occurs when $-eV_{G_3}$ becomes lower than $-eV_{G_2}$ (position 3). Consequently the collector current decreases. The result is that the origin of the negative peak is determined by $V_{G_3} = V_{G_2}$.

The tertiary electrons emitted by the grid G_2 , under usual experimental conditions ($V_c > V_{G_2}$), always

FIG. 5. — Variation of the currents on the grids G_4 and G_2 .

FIG. 6. — Filtering mechanism.

reach the collector and give an extra nearly constant direct contribution to the collector current. On figure 7, we show how positive and negative peaks interfere in the case where $-eV_{G_2} > -eV_{G_4}$. The filtering of the tertiary electrons from G_3 is the same as above. However, the tertiary electrons emitted by the grid G_4 whose energy is less than $-e(V_{G_2} - V_{G_4})$ do not reach the collector and the corresponding part of the positive peak disappears in agreement with reported experimental results (Fig. 8). In fact, on the grids there may be not only a tertiary emission but also different physical mechanisms such as elastical deflexion reducing the normal momentum of the secondary electrons. The quantitative importance of these effects is difficult to estimate. However, we can roughly determine the importance of the parasite peaks from the values $(i_{G_4})_1$ and $(i_{G_4})_2$ of the current i_{G_4} on each side of the positive peak (Fig. 5). Let I_s be the secondary current emitted by the target and α the transparency of grid G_4 . The grid G_4 is reached by a current $(1 - \alpha) I_s$. For $V_{G_3} - V_{G_4} \sim -8$ V, only back scattered tertiary electrons have sufficient energy to leave the grid G_4 . The corresponding current is $i_{t_4}^b$ and $(i_{G_4})_1 = (1 - \alpha) I_s - i_{t_4}^b$. If $V_{G_3} - V_{G_4}$ is slightly

positive, with the hypothesis that all tertiary electrons emitted by G_4 are accelerated by G_3 towards the collector, the current emitted is i_{t_4} and $(i_{G_4})_2 = (1 - \alpha) I_s - i_{t_4}$. In our experimental conditions for copper, $\sigma \simeq 1.2$ and $\eta \simeq 0.3$ [5]. One quarter of the secondary current I_s emitted by the target comes from back scattered electrons with energies between 50 and 300 eV and three quarters comes from true secondary electrons ($E_s < 50$ eV). If $V_{G_4} = 10$ V, these electrons are accelerated towards the grid G_4 and reach it with mean energies of about 200 and 20 eV, respectively. G_4 is made of steel ($\sigma = 0.3$ at 20 eV and 1.2 at 200 eV [6]), also $\eta = 0.3$ at 200 eV [7]. The total tertiary current emitted by the steel grid is :

$$i_{t_4} = \frac{3}{4} (1 - \alpha) I_s \cdot 0.3 + \frac{1}{4} (1 - \alpha) I_s \cdot 1.2$$

the backscattered tertiary current is :

$$i_{t_4}^r = \frac{1}{4} (1 - \alpha) I_s \cdot 0.3$$

and

$$\frac{i_{G_4}(1)}{i_{G_4}(2)} \sim 1.8.$$

Experimentally (Fig. 5) $i_{G_4}(1) = 1.06 \mu A$ and

FIG. 7. — Mechanism of disappearance of the positive peak for $V_{G4} > V_{G2}$.

FIG. 8. — Results showing the disappearance of the positive peak for $V_{G4} > V_{G2}$.

$i_{G4}(2) = 0.5 \mu A$ giving nearly the same value for the ratio.

Transparency variation. — Wei *et al.* have interpreted the i_{G4} current variations as due to the transparency variation of G_4 when the field behind this grid goes from an accelerating to a retarding state. To explain the measured variations of G_4 this transparency must vary from 63 % to 87 %. The field penetration and the corresponding change in the trajectories of low energy electrons may be the cause of this transparency variation, but this hypothesis hardly accounts for the dissymmetric shape of the parasite

peaks, similar in fact, to the shape of the secondary electron energy distributions. In the same way, the field penetration cannot explain the attenuation and the suppression of the positive parasite peak when V_{G4} becomes higher than V_{G2} (Fig. 8), whereas in our interpretation, this suppression is an immediate consequence of the filtering of tertiary electrons from grid G_4 by grid G_2 . Finally, when the primary energy E_p varies from 50 eV to 1 500 eV, we observed that the parasite peak amplitude was maximum for $E_p = 300 - 400$ eV where the secondary emission yield of steel is maximum [6].

3.3 CHOICE OF G_4 AND G_2 VOLTAGES. — When V_{G4} or $V_{G2} = 0$ we observed an important dependance of the maximum position and half height width of the energy distribution in relation to the nearness of positive (or negative) peak. To separate the parasite peaks from the principal peak during the recording, we choose to apply a sufficient positive voltage to grids G_2 and G_4 . But this voltage must be sufficiently low so as not to modify greatly the secondary electron trajectories. Also the current collected by grid G_4 increases quickly, doubling when V_{G2} increases from 0 to 20 V. This increase is due to a growth of the true

collection angle for secondary electrons which are accelerated towards grid G_4 . It is not a major drawback in itself but simultaneously an increasing number of low energy secondary electrons are stopped by grid G_4 , developing a risk of increased disturbance in the measurements. So, on figure 9, we show that the sec-

FIG. 9. — Shifts as a function of voltage on the grids G_2G_4

dary peak shifts towards higher energies, when V_{G_4} increases. Thus, it is important to keep the V_{G_2,G_4} voltage as low as possible. To make the best choice we drew several energy spectra for $V_{G_2,G_4} = 0; 3; 5; 10$ and 15 V. Simultaneously the positive and negative peaks are shifted and to measure the influence of this shift we give in table I the values of the maximum position $E_{\max}$ and the half height width $E_{1/2}$ for the secondary peak.

TABLE I

V_{G_2/G_4} in volts	0	3	5	10	15
$E_{\max}$ in eV	1.4	0.9	0.8	1	1.2
$E_{1/2}$ in eV	5.7	4.1	3.8	3.6	3.6

We finally chose a voltage of 10 V because the characteristic values of the secondary peak do not vary strongly when V_{G_2,G_4} varies from 5 to 15 V.

4. Results on a (111) single-crystal copper target. —

We measured energy distributions and σ , η , δ yields for a (111) copper target after Auger control of the surface cleanness.

4.1 ENERGY DISTRIBUTION (Fig. 10). — The results presented were obtained with $V_{G_2,G_4} = 10$ V, for a primary current $i_p \sim 2 \mu A$ and a primary energy of 300 eV. The maximum of the true secondary electron distribution occurs at $V_{G_3} = -2$ V. The zero value is obtained for $V_{G_3} = -0.3$ V and corresponds to the difference between the target and the grid G_3 work functions. Our results are compared (Fig. 11) with different other results [8, 9, 10] for a normalized value of maximum amplitude. Except for Scheibner and Tharp all these curves are narrow. This difference may be attributed to the spurious effect of the grids

FIG. 10. — True secondary spectrum of Cu (111) ($E_p = 300$ eV).

FIG. 11. — True secondary spectra obtained by other authors for copper.

because their apparatus was similar to ours with $V_{G_2,G_4} = 0$. We observed that the curve width increased by 50 % when V_{G_2,G_4} goes from 10 V to 0 V and gives the energy distribution of Scheibner [2] as reported by Amelio. The results obtained by Bronshtein and Seah on polycrystalline copper targets have a comparable half height width but rather different maximum positions. However Seah used a 70° oblique incidence and collected the secondary electrons in a very narrow solid angle (12 milliradians). For $E > E_{\max}$ our peak has a shape similar to Seah's and may be described by a power law

$$N(E) \propto \frac{1}{(E + 4.5)^{1.6}} \frac{1}{(E + 0.35)}$$

but differs for $E < E_{\max}$ with a more rapid increase and then a shift of the maximum position. However, Seah noticed that for higher angles of emergence, the secondary peak moves to higher energies (for silver he reported a 50 % variation when the angle of emergence varies from 0 to 60°).

We can conclude that our results and Seah's are similar and that the differences are due to different experimental conditions.

4.2 YIELDS. — For one primary electron, the target emits σ secondary electrons with δ electrons having an energy smaller than 50 eV and η electrons having an energy greater than 50 eV. To evaluate σ we must measure the total secondary current i_s and the primary current i_p . In fact, we measure the target current i_c and i_s ,

$$\sigma = \frac{i_s}{i_p} = \frac{i_s}{i_s + i_c}.$$

To attract all electrons towards the collector suppressing the charge of the fluorescent layer and the tertiary emission of the collector, we applied the same potential to the 4 grids and to the collector and measured the total secondary current in the collector.

We applied a +200 V potential, because for this value, the measured current i_s was more stable for a ± 50 V variation around this value. Palmberg [11] with a 3 grid system suggested using a 300 V post acceleration. Under these conditions, the total yield curve $\sigma(E_p)$ is to within experimental uncertainty, in good agreement with Bronshtein's results [6] (Fig. 12). To determine η we measure the current i'_s reaching the

FIG. 12. — Secondary yields σ and η of copper. Bronshtein : —
our results : Δ : σ
 $\odot$: η

collector after passing through the filtration system, for a retarding potential of 50 V. The i_p current is determined as above and if τ is the analyser transparency

$$\eta = \frac{1}{\tau} \frac{i'_s}{i_s + i_c}.$$

To evaluate τ we used Richard's method [12] which is applied with a 2 grid analyser to give each measurement the transparency value of the grids. The identification with our system was obtained by connecting G_2 to the collector and by applying the retarding potential to grid G_3 . The values obtained for η are compared on figure 12 with Bronshtein's results [13]. Our results correspond to a collecting angle of 100° and it is not surprising that our values are smaller than those of Bronshtein. For a cosinusoidal distribution of secondary electrons, the corrective factor should be approximatively 1.65 whereas the experimental

corrective factor is 1.5 for a 500 eV primary energy. We must remark that the measured current includes tertiary electrons emitted by grid G_3 as well as secondary electrons filtered by the same grid. Also the determination of the transparency of grids G_4 and G_3 is only approximate and does not take into account the secondary electron energies or an eventual transparency variation when grid G_3 goes from 0 to -50 V. These first measurements have enabled a check to be made of the values of Bronshtein. The values of δ are obtained by subtracting η from σ .

Results for the energy spectra obtained with a cylindrical mirror analyser (CMA) [14] and correlated with image contrasts in a scanning electron microscope (SEM) are quite different from our results.

The origin of the disagreement is still unknown but some remarks may be made :

1) SEM image contrast seems to be related more to variations of the emission intensity than to the emission intensity itself.

Then an image without contrast will be an image in which each point of the surface has an equal emission intensity whatever the energy spectrum of the target.

The presence of impurities on the surface, resulting, for instance, from diffusion by local heating or others processes creates surface heterogeneities leading to contrasted images in SEM.

2) The 4 grid analysis system collects all the electrons emitted from bombardement of the surface whereas the CMA collects only in a finite solid angle. It is thus possible that the two collected distributions differ without either being in error.

3) In the same way CMA with its subsequent electron multiplier has a gain equal to $E.K(E)$ where $K(E)$ is the multiplier gain. This gain is higher for high energies than for low energies. The situation is different for a 4 grid analyser which gives $N(E)$ directly.

5. Conclusion. — The object of this work was to adapt a 4 grid retarding field analysis system to secondary electron emission studies. Such a device enables a control to be made of the cleanness of the analysed surface by Auger spectroscopy and hence provides precise information concerning the nature of the target. But if all necessary precautions are not taken, this system produces spurious effects which we have studied in detail. We showed that the origin of these perturbations is related to grid emission due either to an elastic reflection of secondary electrons on the grids in which they lose normal momentum or to a true tertiary electron emission from the grids under secondary bombardment. We proposed a means of separating the true and the spurious peaks. We thus obtained the energy distribution for secondary electrons emitted by a (111) copper single-crystal. The discrepancy in the experimental results may be due to the spurious effects on the grids.

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