interpreted as an evidence of the progressive formation of larger clusters of vacancies which are poor electron traps. It should be kept in mind too that each electron trap which is not an F-center must have at least two vacancies (the F'-center is not stable at room temperature). Ueta and Känzig also observed the formation of the M and R bands during plastic deformation and the growth of the R band up to 48 hours after plastic deformation. Thus if one considers that there are some free positive-ion vacancies, the ratio of about four vacancies to one trap is not out of line.

6. The annealing experiments indicate that the presence of vacancies has no measurable effect on hardness: The vacancies anneal out completely at 350°C, while hardness remains unaltered up to about 750°C annealing temperature. In addition there was no correlation between the change of density and amount of work done in deforming the crystal, which probably reflects the fact that only a small amount of energy of deformation goes into creation of vacancies.[26] It is thus quite likely that hardness is associated with a disturbance of the dislocation pattern, especially of the Frank-Read sources, during the process of deformation and not with the presence of vacancies as slip obstacles. This is in agreement with some of the experiments discussed in the introduction. It should be remembered, of course, that one has to be careful in drawing conclusions from hardness, an essentially surface property, about internal phenomena.

7. Formation of deformation bands was analyzed in terms of the operating slip systems and the structure and orientation of the deformation band boundary.

ACKNOWLEDGMENT

The authors wish to thank Professor F. Seitz for his interest and valuable discussions during the progress of this work.

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Effects of Contamination on the Characteristic Loss Spectrum of Tungsten*

C. J. Powell,† J. L. Robins,‡ and J. B. Swan
Department of Physics, University of Western Australia, Nedlands, Western Australia

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Two types of contamination, the adsorption of a monolayer of atmospheric gas and the deposition of carbonaceous material under electron bombardment, were found to affect the characteristic electron energy-loss spectrum of tungsten. Spectra were obtained by analyzing with a 127° electrostatic spectrometer the energy distribution of 850-volt electrons scattered by the target through 90°. The deposition of the carbonaceous material was associated with the growth of the carbon Auger peak. Each type of contamination could be removed by heating the target in vacuum to a suitable temperature.

INTRODUCTION

THE results of electron scattering experiments at low energies are markedly affected by the occurrence of two common types of surface contamination of the specimen. One type arises from the adsorption of a monolayer of residual atmospheric gas, and many workers[1] in this field have recognized the need for thorough outgassing of the specimen. Some direct effects of this contamination have been reported by Harrower.[2] The other type of contamination, which has also been observed in many systems,[3–5] is the deposition of carbonaceous material on those portions of the specimen irradiated by the electron beam.

The purpose of this paper is to show how these two types of contamination influence the characteristic energy-loss spectrum[6] of tungsten.

APPARATUS

The specimen used in these experiments, a tungsten wire 0.01 in. in diameter, was the target for primary electrons from a Pierce-type electron gun.[7] Scattered

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† I.C.I.A.N.Z. Research Fellow, University of Western Australia.

‡ General Motors-Holden Postgraduate Research Fellow, University of Western Australia.

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[2] G. A. Harrower, Phys. Rev. **102**, 1288 (1956).

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[5] R. Castaing and J. Descamps, Compt. rend. **238**, 1506 (1954).

[6] L. Marton, Revs. Modern Phys. **28**, 172 (1956).

[7] J. R. Pierce, *Theory and Design of Electron Beams* (D. Van Nostrand Company, New York, 1954), second edition, Chap. 10.

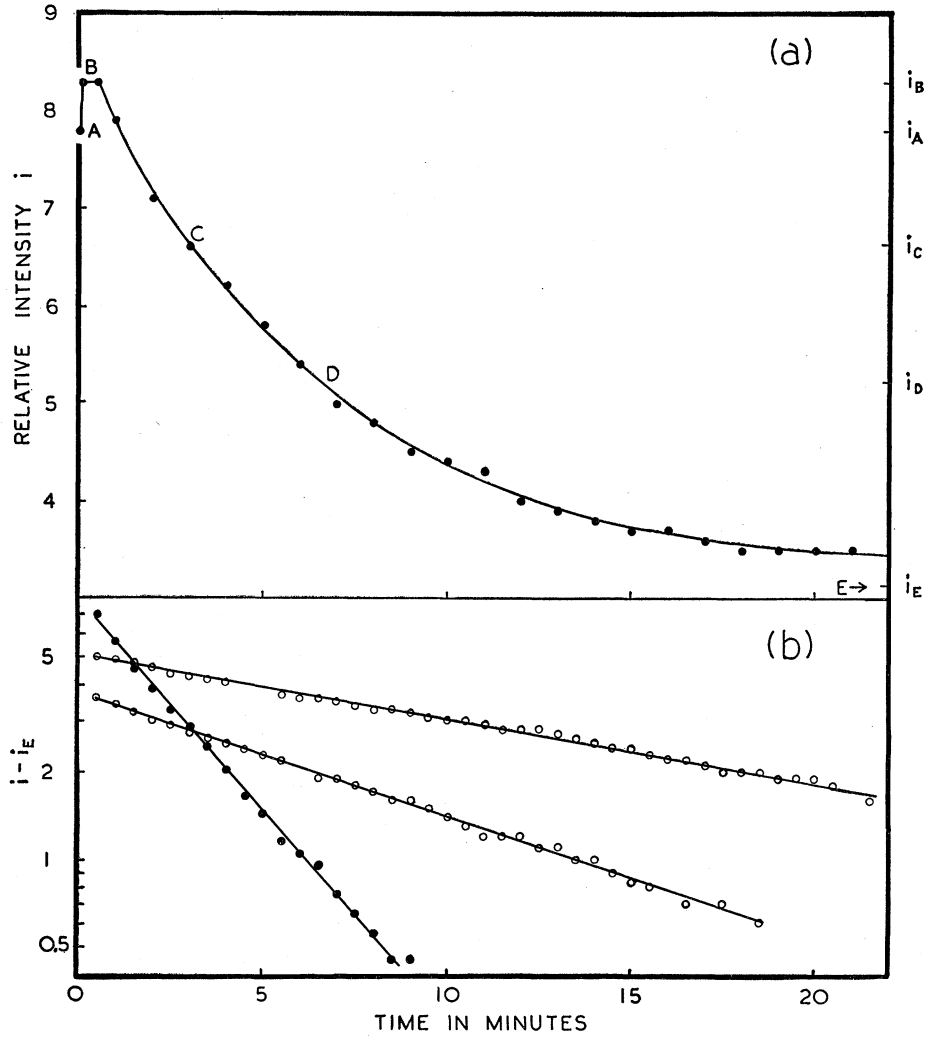

FIG. 1. (a) Relative intensity i of elastically reflected primary electrons as a function of time. Point A corresponds to a clean tungsten target and B, C, D and E to increasing degrees of contamination. (b) $i-i_E$ for three different rates of contamination plotted on a logarithmic scale as a function of time.

and secondary electrons which left this target in a direction at right angles to the primary beam passed through the entrance slit of the 127° electrostatic spectrometer[8] into the space between the curved deflector plates. These plates, made of aluminium, were 3 in. wide and of radii 9.25 in. and 10.75 in., respectively, and were clamped between two Perspex sheets. The plates were maintained symmetrically above and below earth potential and the Perspex was coated with a thin layer of graphite in order to prevent accumulation of a surface charge. The adjustable aluminium entrance and exit slits, each 0.5 in. high, were grounded and the target also was maintained at earth potential. The temperature of the target could be raised by passing a heating current through it.

The detector, a 17-stage beryllium-copper electron multiplier, was placed immediately behind the exit slit of the spectrometer and all electrons which were focussed on this slit passed directly onto the first dynode, the resulting output pulses being amplified and counted. The potential difference between the deflector plates, which was measured with a Tinsley Vernier Potentiometer, was varied by fixed intervals and spectra were scanned by observing the counting rates at the corresponding electron energies.

The spectrometer was constructed entirely of nonmagnetic materials and the pumps were located at some distance from the deflector plates. In addition, the earth's magnetic field was reduced to less than 0.1% of its normal intensity over the region between the deflector plates using a two-coil system.[9] The axis of the coils coincided with the axis of the deflector plates, and by tilting the apparatus this common axis was aligned with the earth's field.

With a target-entrance slit distance of $1\frac{3}{4}$ in., slit widths of 2.5×10^{-3} in., and the target bombarded by electrons accelerated through 850 volts, the width at

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half maximum intensity of the peak of elastically reflected electrons was 1.4 ev.

RESULTS

All measured counting rates were normalized in order to present undistorted intensity-energy spectra and the numerical results quoted are the best values of a large number of determinations.

The trap above the oil diffusion pump was left unfilled and the pressure in the vacuum system was normally about 3×10^{-5} mm of mercury. The net electron current to the target was maintained at 40 μa corresponding to a current density at the target of 1.5 ma/cm^2, and the accelerating voltage was usually 850 volts, though some observations were made at 600 volts. The tungsten target was initially prepared for use by heating it to white heat to remove volatile impurities.

At the commencement of each series of contamination measurements the target was heated to about 1500°C and the spectrometer controls were adjusted to focus elastically-reflected primary electrons (the elastic peak). Following rapid cooling of the target, the elastic peak intensity, i, was observed to change with time as shown in Fig. 1(a). As the monolayer adsorption time[10] is less than one second at the normal operating pressure, it was concluded that the initial rapid change in counting rate resulted from contamination of the tungsten surface by adsorbed atmospheric gas. Since it was always possible to remove this contamination by heating the target to about 1500°C, it was assumed that the intensity at A, i_A, corresponded to reflection from a clean tungsten surface. For 850-ev electrons it was found that when the surface was thus contaminated, the observed intensity, i_B, was greater than i_A, whereas for the same energy Harrower's results[2] indicate that i_B is less than i_A. However, the large scatter of Harrower's experimental points at 500, 800, and 1000 volts may make his interpolation unreliable. The ratio $(i_B-i_A)/i_A$ was found to be 0.06 ± 0.03.[11] For a primary energy of 600 ev, the measured value of this ratio was -0.17 ± 0.03, the sign being the same as that found by Harrower.

It was found that the target surface could be kept in the condition represented by point B for an indefinite length of time by heating the target to about 400°C. The characteristic loss spectra of the target in the states corresponding to points A and B were recorded and typical curves are shown in Fig. 2(a) and (b), respectively. The characteristic energy loss values and their probable errors[11] are given in Table I. The spectrum obtained from the clean tungsten differs from that obtained by Harrower[12] in both shape and position of the peaks, possibly because of the different scattering angles in the two experiments.

TABLE I. Characteristic energy loss values for the target in the states corresponding to A, B, and E of Fig. 1(a).

Designation	Characteristic energy losses (electron volts)				
A	···	10.6±0.4	24.3±0.2	43.3±0.5	52.8±0.4
B	···	11.7±0.4	25.0±0.2	44.3±0.5	53.5±0.4
E	5.8±0.6	···	24.8±0.8	···	···

[10] H. D. Hagstrum, Rev. Sci. Instr. **24**, 1122 (1953).
[11] The probable errors quoted here were determined from the differences between the observed values and their mean.

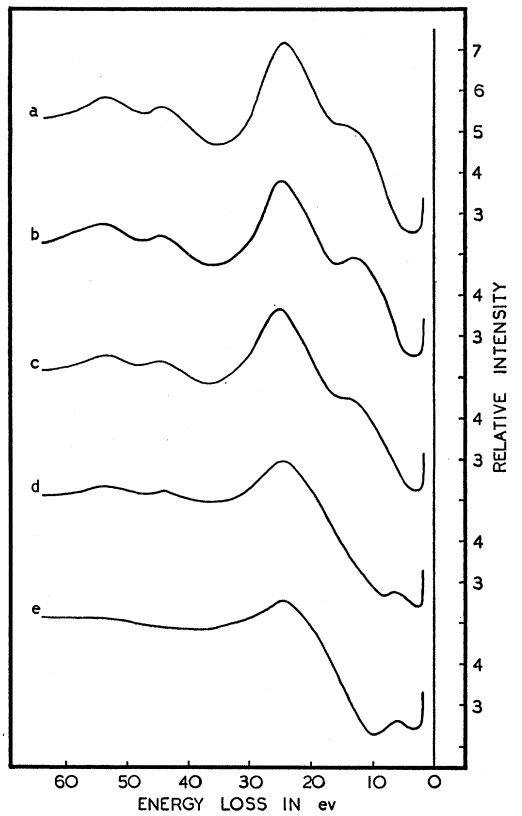

FIG. 2. Typical characteristic energy loss spectra obtained at stages of contamination corresponding to A, B, C, D, and E of Fig. 1(a).

The differences between (a) and (b) of Fig. 2 could be due to other effects dependent on the target temperature. However, the fact that the target had to be heated to 1500°C to obtain spectra of the type (a) is consistent with measurements of sorption of nitrogen and oxygen on tungsten at various temperatures,[10,13,14] and suggests that the spectrum change is due to target contamination by atmospheric gas, probably nitrogen.[10,15]

The second type of contamination is the deposition of carbonaceous material on the target under electron bombardment. König[16] has shown that the deposit consists principally of carbon, while Ennos[4] has concluded that the contamination arises from the inter-

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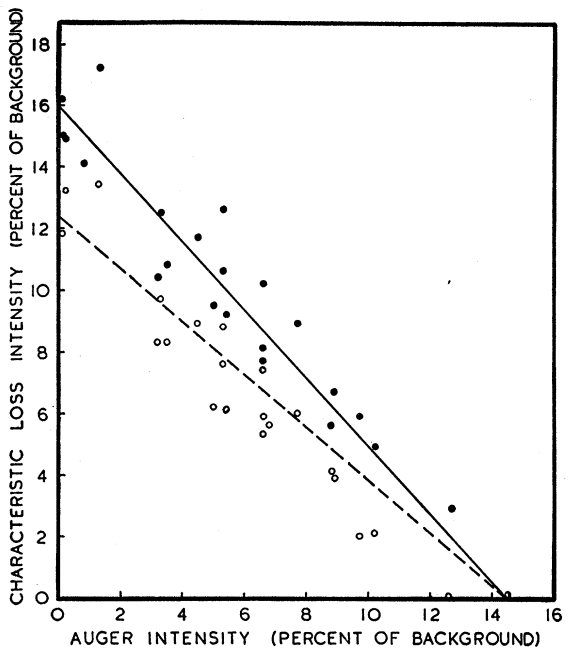

FIG. 3. The percentage height above background for the 53.5-ev (closed circles and full line) and the 44.3-ev (open circles and dashed line) losses plotted against the percentage height above background of the carbon Auger peak. The percentage heights are obtained from [(height of peak—background)/background]×100.

action of the electron beam with hydrocarbon molecules (originating from pump oils, vacuum greases, etc.) adsorbed directly onto the target surface from the vapor phase. The results of Castaing and Descamps,[5] however, indicate that the processes involved may be more complicated.

Figure 1(a) shows how the gradual deposition of this contamination affects the elastic peak intensity, which is seen to decrease exponentially with time from i_B towards a steady value i_E. Different contamination rates, due possibly to different partial pressures of organic vapors in the target chamber, were observed under the same conditions of bombarding current and voltage and in Fig. 1(b) three such series of values of $i-i_E$ are plotted on a logarithmic scale as a function of time.

At any stage, formation of further contamination could be prevented by heating the target to about 400°C. It was thus possible to record characteristic loss spectra for various degrees of target contamination. In Fig. 1(a), C and D are arbitrarily chosen points on the contamination curve and Figs. 2(c) and (d) are the corresponding characteristic loss spectra. The characteristic loss spectrum of the target in the final stage of contamination corresponding to E in Fig. 1(a) is shown in Fig. 2(e), and the energy loss values are given in Table I. Curves (b) to (e) of Fig. 2 therefore show the changes in the characteristic loss spectrum of the target as the contamination proceeds. It is seen that the tungsten loss peaks gradually disappear and are replaced by those of the contamination layer. Both the spectrum and the energy-loss values of this contamination agree with other results obtained for carbon.[17]

As soon as a characteristic loss spectrum had been recorded, the secondary electron spectrum in the region of the carbon Auger peak[18] was scanned. This peak was observed to grow as the contamination proceeded; its appearance is corroborative evidence that the contaminant is principally carbon. It was found that this Auger peak grew and the tungsten characteristic-loss peaks decreased exponentially with time at the same rate as the elastic peak intensity decreased. In Fig. 3, the percentage heights above background of the 44.3- and 53.5-ev loss peaks are plotted against the percentage height above background of the carbon Auger peak. From a least-squares analysis, the intercepts on the abscissa axis were found to coincide within the limits of probable error, and both lines are drawn to pass through the mean value. The intercepts on the ordinate axis are the percentage heights above background of the appropriate tungsten loss peaks when the target is in a condition represented by B in Fig. 1(a). The scatter of the experimental points in Fig. 3 is mainly due to the difficulty in locating the continuous background of primary electrons that have suffered various energy losses by inelastic collisions.

It was found that the carbonaceous contamination could be removed by heating the target to red-heat in vacuum, thereby returning to point B in Fig. 1(a). As it was possible to remove both types of contamination by heating the target to an appropriate temperature, the same target was used in all measurements.

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