WHAT CAN BE DONE WITH HIGH VOLTAGE ELECTRON MICROSCOPY ?

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Introduction

High voltage electron microscopy has been developed about twenty years ago when Dupouy and colleagues constructed the first microscope working at 1.200 kV (1). Ten years later the 3 MV microscope was built in Toulouse (2). This last microscope can work slightly above 3 MV. Now, through the world, about 52 H.V.E.M. work.

It is, in fact, difficult to define exactly what is H.V.E.M. compared to conventional electron microscopy. Most of the time it is considered that 300 kV and above concern H.V.E.M. because the relativistic effects begin and also because the technique for the high voltage generator demands new solutions.

As every method, H.V.E.M. is not a general panacea. It has advantages but also can be at the origin of difficulties. These problems are principally related to the size of the apparatus for a part but also to the fact that some energy is given to the sample by the incident electrons. Sometimes this transfer of energy can be the origin of some artefacts. We shall discuss below about advantages and disadvantages of H.V.E.M. For some more complete discussion we refer to (3).

1) - Theoretical resolution limit.

The first advantage of H.V.E.M. is the theoretical limit of resolution. The objective of the microscope has a transfer function which is depending on the spherical aberration and on the defocusing (the position of the plane which is taken as object for the lens is important). This transfer function can be written as a function of the frequency which is to be resolved within the sample. If $\vec{\nu}$ is the spatial frequency, $A(\vec{\nu})$ is the

$$T(\vec{\nu}) = \frac{1}{M} A(\vec{\nu}) \exp -i\frac{2\pi}{\lambda}\left(\frac{1}{4} C_s \lambda^4 \nu^4 - \frac{\Delta z}{2} \lambda^2 \nu^2\right) = \frac{1}{M} A(\vec{\nu}) \exp i\gamma(\vec{\nu})$$

aperture function equal to zero out of the aperture and 1 inside the aperture. $\gamma(\vec{\nu})$ which represents the phase shift due to the objective can be optimized by using the right defocusing. The behaviour of this transfer function is quite favourable to H.V.E.M. which can give information at the level of 1 Å or slightly less. More precisely from this point of view the best voltage can be depending on the level of information which is demanded. Some detailed calculations have been recently done in the laboratory for instance and confirm this point (4). This interest is related to the wavelength associated to the electron (see table 1), and to the spherical aberration ($\sim$ 1 mm at 100 keV, 4.2 mm at 1.2 MeV and 6.6 mm at 3 MeV in our laboratory) which is not varying too rapidly when increasing the energy of the incident electron.

On the other hand, some calculations by Zeitler and Thomson (5) have shown that for sin-

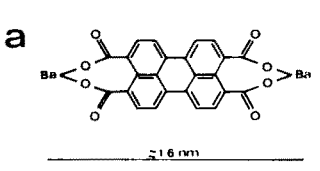
gle atoms the gain in contrast is about 3 in bright field and 6 in dark field for carbon atoms and respectively 2 and 7 for gold atoms (without any substrate).

V	10 kV	40 kV	100 kV	500 kV	1 MV	2 MV	3 MV	6 MV
$\beta = v/c$	0.1950	0.3741	0.5482	0.8629	0.9411	0.9791	0.989	0.9969
λ (Å)	0.1220	0.0602	0.0370	0.0142	0.0087	0.0050	0.0036	0.0019
λ^{-1}(Å^{-1})	8.194	16.62	27.02	70.36	114.7	198.3	277.8	523.5
m/m_o	1.01957	1.0783	1.1957	1.9785	2.9569	4.9138	6.870	12.742

Table 1. Values of various parameters in electron microscopy, as a function of voltage.

Moreover the interpretation of contrast is simpler in H.V.E.M. because a simpler theoretical treatment can be used (weak phase approximation). More precisely at high voltage this approximation can be used for thicker samples than at lower voltages. The origin of that is due to the writting of the scattered wave, which can be expressed as : $\Phi(t) = \exp i\chi(t)$ where t is practically the normal to the sample with $\chi = \frac{-e}{hv}\int_o^t U(t)dt$ where v is the velocity of the incident electron and U is related to the potential V by $U = \frac{2m}{\hbar^2} V$. The change in phase of the incident wave is governed by the projected potential written above.

In addition it seems it has been shown for crystalline materials that both experimentally and theroretically (6) the contrast is better and the number of informations more important in H.V.E.M.. In our laboratory single atoms fixed on organic molecules deposited on very thin boron substrats ($\sim$ 10-15 Å) have been observed (7,8) (fig.1). Some nice experiments have been also recently done by Uyeda at 500 keV (9), on molecules taking advantage of the less important energy losses as an overall for ionization



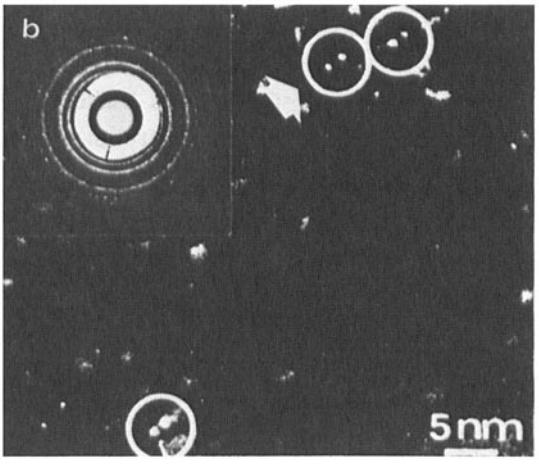


Fig.1. Single atoms of Ba observed at 3 MeV on an amorphous substrate (10-15 Å thick).

a)- localization of Ba atoms (16 Å apart).

b)-Conical illumination. Over-impression of the diffraction pattern of gold.

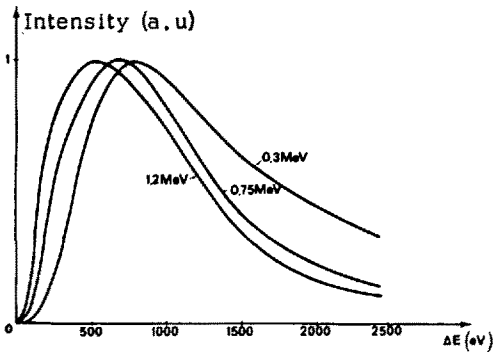


Fig.2. Electron energy loss spectra through 3 μm aluminium foils for 0.3, 0.75 and 1.2 MeV electrons (thick foils).

when the energy is increased (fig.2).

The resolution which was discussed here is only possible for thin materials even if for the reasons given above, it is possible to use thicker samples in H.V.E.M. for this purpose.

2) - Available thickness

Two types of limit of thickness can be defined : the first one is related to the range of the electrons in the sample (total penetration). It defines the thickness which gives the loss of all the kinetic energy of the incident electrons. The second one is the available thickness which gives a possibility to get informations on the sample through an image in electron microscopy.

The first penetration, the total penetration, has been studied in detail for instance by Arnal (10). An order of magnitude is in aluminum 2.1 mm, 8.1 mm respectively for electrons of 1 MeV and 3 MeV and 0.6 mm in copper at 1 MeV.

The available thickness has been studied by Humphreys (11) and Rocher et al. (12) and Thomas and Lacaze (13). The gain in penetration is an important point in favour of H.V.E.M..

The models to explain the available thickness are based on electron-phonon interactions which give a behaviour of the absorption coefficient proportional to Z^2/v^2 (scattering) where Z is the atomic number. The penetration is therefore more important in the case of light materials. However the behaviour as a function of the energy is rather pessimistic, specially for light materials, compared to the experimental results. At the present time this discrepancy is not fully understood. It could be due to the fact that the limitation in penetration is not only due to this effect but is related to the so-called top-bottom effect (14). The contrast is smoothed because of the scattering of electrons which is observed through the top-bottom effect.

The chromatic aberration is not obviously the most important factor for the penetration as it can be seen from the expression given for instance in (15, 16).

When electrons pass through a sample they have, if it is thick enough, an output most probable angle θ_p which varies as $\sqrt{t}$, where t is the thickness. The angular distribution (gaussian distribution) centered on θ_p is characterized by $\theta_G(\theta_G^2 = 2\theta_p^2)$ and the intensity collected through the aperture (see fig. 3) is given by :

$$I(\alpha) = I_o \frac{\alpha^2}{\theta_G^2} = I_o \frac{0.86\pi^3 a_o^2 (1-\beta^2)}{NZ^2\lambda^4 e^{2a/b_t}}$$ where the detail of the expression is obtained

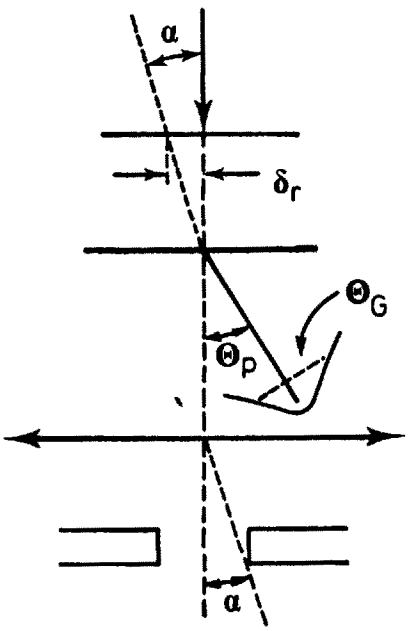


Fig.3. Drawing corresponding to the top-bottom effect.

from ref. 17. I_o is the incident intensity, α the angle of collection, a_o the Bohr radius for hydrogen, β the ratio of v with the velocity of light c, N the number of atoms by cm^3, $e^{2a/b}$ is a constant which depends on the screening of the atom. If we give for the penetration a given value of $I(\alpha)/I_o$, say 10^{-3}, we get for two voltages 1 and 2 :

$\frac{t_1}{t_2} = \left(\frac{\alpha_1}{\alpha_2}\right)^2 \frac{(1-\beta^2)_1}{(1-\beta^2)_2} \left(\frac{\lambda_2}{\lambda_1}\right)^4$. This ratio is independant of Z which means it is more available for low Z when phonon problem is not important. Moreover that model would be more useful for biological samples. It means the absorption coefficient varies with t.

3) - Processus of interaction electron - sample.

a)- elastic interaction. The basic model is well-known (18). It is based on the coulomb interaction of charged particles, with the nucleus and electrons of atoms. By using a coulomb screened potential, it is found the elastic differential cross section :

$\sigma_e(\alpha) = 4\pi\left(\frac{\lambda}{a_o}\right)^2 \frac{Z^2}{k^4} \frac{1}{(\alpha^2+\alpha_o^2)^2}$ with α the scattering angle and α_o the screening angle (α_o)= 1/ka, a screening radius and $k = \frac{2\pi}{\lambda}$. The total cross section is easily found and varies as $1/\beta^2$.

b) - inelastic interactions. These interactions are principally at small angle.

α). global treatment. The global inelastic interactions are characterized by a cross section which can be written between α_1 and π. $\sigma_{in}(\alpha_1,\pi) = \frac{\sigma_e(o,\pi)}{Z}\left(2Ln(1+\frac{\alpha_o^2}{\alpha^2}) - \left(\frac{1}{1+\alpha^2/\alpha_o^2}\right)\right)$

The ratio of the total inelastic and elastic cross section is given by :

$\frac{\sigma_i(o,\pi)}{\sigma_e(o,\pi)} = \frac{4}{Z} Ln\left(\frac{\alpha}{\alpha_E}\right)$ where $\alpha = \frac{\gamma\Delta E}{E(\gamma^2-1)m_o c^2}$ with the ionization energy :

$\Delta E = 9.732 + 58.8\ Z^{-0.19}$ which is available for Z above 13 about. The ratio is nearly constant with the energy of electrons.

The inelastic cross section includes different processus. Principally we can notice the excitation of electrons of the inner shells and also of the continuum.

β). inner shell excitations (19, 20, 21). The expression of the cross sections can be written :

$\sigma_K = \frac{2\pi e^4}{m_o c^2} \frac{f_{K,L...}}{E_{K,L...}\beta^2} \log \frac{\alpha^2 + \alpha_E^2}{\alpha_e^2}$ where e is the charge of the electron, f_K the oscillator strength ("proportion" of electrons participating per atom to the excitation K, L...). E_K, the energy of the edge, increases with Z, for a same type of excitation. $\alpha_E \cong \frac{E_{K,L...}}{E(E+2)}(E + 1)$ where E is the incident energy. The behaviour as a function of energy is shown on fig. 4.

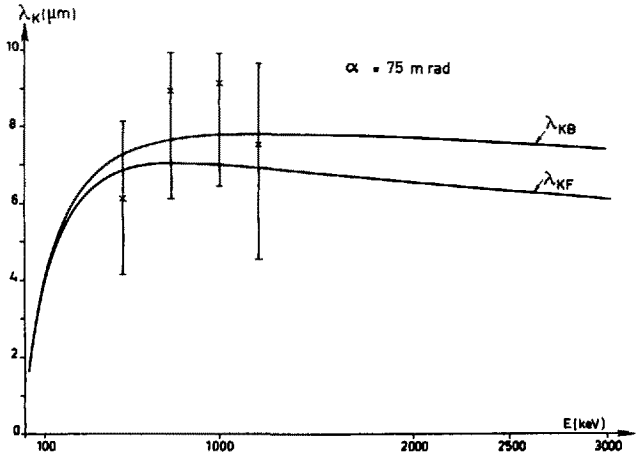


Fig.4. Inner shell excitation free path (carbon).
λ_{KB} corresponds to the Bethe Model,
λ_{KF} corresponds to the Fano treatment.
Experimental points are marked with bars of error.

All the excitations have a cross section which is roughly varying as $1/\beta^2$ (mean free path as β^2). If we look on fig.5, the cross section as a function of the angle ($\sigma(o \to \alpha)$) is more concentrated on the forward direction when the incident energy is increased. In this direction the background due to excitations of the continuum is minimum. Therefore it is easier to detect electrons at high voltage than at lower voltages. This is favorised by the smallness of optical aberrations at high voltage. These points explain why it has been possible to detect losses up to 12.000 eV about 1.2 MeV, instead of 2.000 eV about at 60 keV.

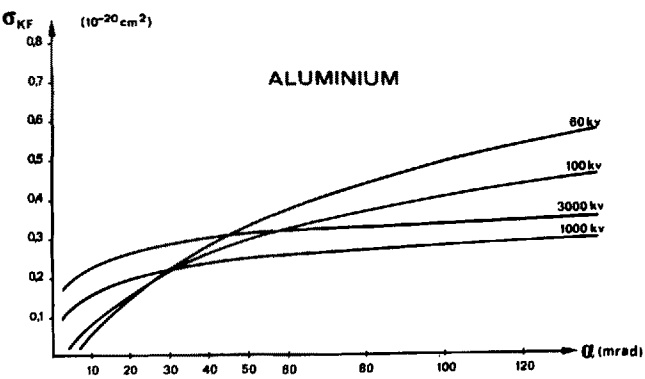


Fig. 5. Cross sections (integrated) as a function of the scattering angle for different energies in the case of aluminium.

Amongst other excitations, plasmons can play an important role. They are quite easy to observe in aluminium or carbon. Table 2 gives different order of magnitude of cross sections determined for high and low energy.

4) - Dynamical theory or classical treatment.

a)- Classical treatment. The condition for using classical treatment is $\lambda \ll a$ which has to be related to the uncertainty relations.

Details about the problem of semiclassical treatment, as use of WKB methods, have been

Al	σ_e	σ_i	σ_i/σ_e	σ_K	$\sigma_{plas.}$	$\sigma_{cont.}$	σ direct knock-on
100 keV	$1.288\ 10^{-18}$ cm^2	$1.327\ 10^{-18}$	1.03	$2.76\ 10^{-21}$	$5.528\ 10^{-20}$	2 to 10 K	0
1 MeV	$4.37\ 10^{-19}$	$5.23\ 10^{-19}$	1.2	$8.73\ 10^{-22}$	$1.675\ 10^{-20}$	2 to 10 K	$50\ 10^{-24}$

Table 2.

given by Berry and Ozorio de Almeida (22).

The particles become more and more "classical" when increasing the energy. It means it becomes more correct to speak about trajectories.

b) - Dynamical interactions. Using the secular expression for dynamical theory (23, 24), it is possible to show that for two crystals and two different energies, the condition to have the same Bloch wave coefficients can be written (systematic reflexions) :

$$E_2 = \frac{g_2^2}{g_1^2}\frac{U_{g_1}}{U_{g_2}}(m_oc^2 + E_1) - m_oc^2 \qquad U_g = \frac{2m}{\hbar^2}V_g \ . \quad \text{If } g_1 = g_2 \text{ and knowing that } U_{g_1} \simeq 1.6 \text{ MV}$$

If $g_1 = g_2$ and knowing that $U_{g_1} \sim 3.5\ U_{g_2}$ with reference to gold and aluminium respectively, and doing $E_1 = 0.1$ MV, we shall get the same type of contrast with $E_2 \simeq 1.6$ MV for aluminium. That has been experimentally checked by Rocher (24), Roucau and Ayroles (25) in more details taking into account the influence of temperature (Debye Waller correction on U_g).

We note the parameter g_2^2 / U_{g_2} which is also important in other considerations.

In a review, Humphreys and Whelan (26) discussed the condition $\frac{g_2^2}{U_{g_2}} < 1$, which is necessary to have many beam effects. Because $U_{g_2} = U_{g_{2.o}}(1 + \frac{E}{m_oc^2})$ where $U_{g_{2.o}}$ is the term non corrected for relativity, we see that increasing the energy increases the dynamical interactions. This condition is also available for channeling.

c)- Critical voltage. It has been shown that for the second order bragg condition it is possible, in many cases, to find an accelerating voltage for which the intensity of this bragg spot becomes practically zero (see 3 for a review). This effect can be easily understood in a three beams treatment. The interest of this effect is due to his important sensisivity to the orientation of the sample, it means to the excitation of accidental reflexions and to the temperature also. The variation with temperature is, in the case of copper (440), about 1 kV per degree. This effect gives the possibility to test rather easily the Debye correction for instance. On the figure (6) we observe that the best Debye temperature is 325 K and also the Debye model is not correct above 600 K about (27).

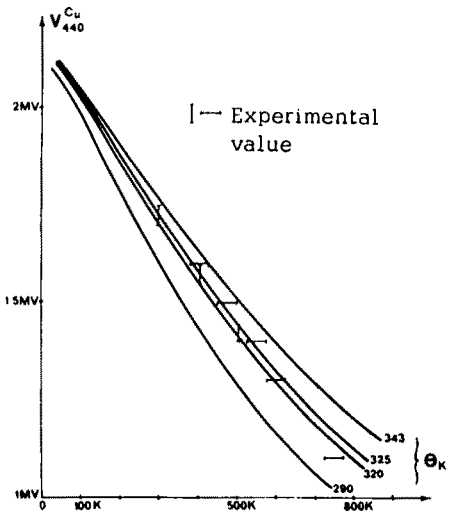


Fig.6. Critical voltage effect for the 440 reflexion in copper (27) :
- horizontal bars of determination of critical voltage by variation of temperature.
- vertical bars the temperature of the sample is constant.
Fitting is tried with different values of the Debye temperature.

This effect has been also used to obtain some information on the ordering of alloys (28).

So it is interesting to use this effect to test the structure factor or more precisely the potential which is used to deduce the scattering atomic factor curve. This point is the first interest of this relativistic effect. The determination of the atomic factor variation as a function of the scattering angle needs the determination of different points to be more precise. That is one of the interest of very high voltages ($\sim$ 3 MeV) which give the possibility to get different points on the curve (27). However this test is limited at the angles given by the lowest reflexion which is used. ($\sim 0.2 \frac{\sin \alpha}{\lambda}$ in copper). So it is quite interesting to compare the results obtained by the critical voltage and the ones given by direct scattering experiments which can give results quite close to the forward direction.

5) - Applications in metallurgy and biology.

a)- in situ experiments. The interest of H.V.E.M. in "in situ" experiments is essentially due to the available thickness compared to conventionnal electron microscopy (fig.7) (12). The spatial resolution of the method is good in comparison with the other methods which are used at the present time : X rays, Soft X rays, neutron topography (see ref. 29 for a review). The large gap between the pole pieces enables to fabricate a very "microlab" in the microscope itself.

Depending on the velocity of the phenomenon the images can be recorded on plates ($\lesssim$ 1-2 s.) or magnetic video tape recorders (exposure time $\sim$ 1/25 s. - 1/65 s.).

α) plastic deformation. The interest of these studies is due to the possibility of deformating a sample at low ($\sim$ 20 K), or high ($\sim$ 1100 K) temperature in the microscope itself with velocities as low as 10^{-7} up to 10^{-3}/s about. So it is possible to follow at the microscale of dislocations the behaviour of a material during the deformation.

The use of this method gives the possibility of testing some models, to understand if a mechanism is active. Some interesting results have been obtained 5 - 10 years ago by Furubayashi (30), Vesely (31), and Imura (32) for instance. The subject was concentrated on BCC materials, principally on molybdenum. The screw dislocations become quite straight at low temperature ($\lesssim$ 110 K in Nb, room temperature in Mo) because of their

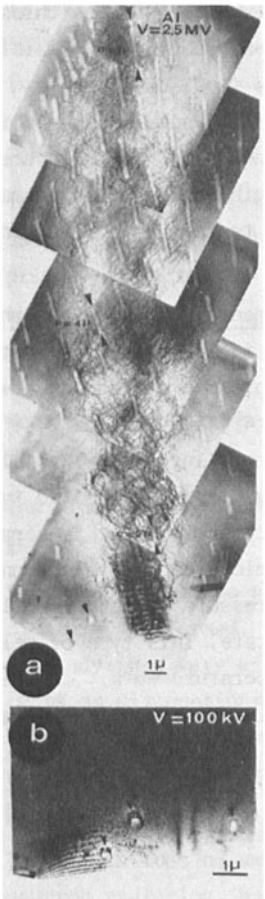


Fig. 7. Some area taken at 100 kV and 2.5 MV. The gain in penetration is about 10. Be fibres in the matrix of aluminium give an easy way to determine the penetration (ref. 12).

core structure . It was thought that these screw dislocations are responsible of stage I while stage O is due to the movement of edge dislocations. This point has been checked and Vesely was able to describe mechanisms of dislocations sources. It has shown two types of sources acting respectively at low and high stress (31).

On the other hand, Imura and coworkers (32) have studied the mobility of edge and screw dislocations in Fe-Si. They determined the velocity of edge dislocations in a Lüders band ($\sim 2.10^{-2}$cm/s).

So, with the work of Louchet et al. (33, 34) concentrated on BCC materials (fig. 8), these studies, which have been done in detail, have given some quantitative informations and the behaviour of BCC materials related to the double kink mechanism (the velocity of a screw dislocation is proportional to its free length) is understood in a much better way than before. The waiting time in a Peierls valley has been determined in some cases. Some studies on softening have been also developed (35).

In FCC materials Henderson et al. (36) have studied the behaviour of dislocations. In an Al 1 % Mg alloy they have shown that the velocity of dislocations is included between 0.01 μm/s. and 1 μm/s.for stresses between 5 and 15 MN/m^2. An interesting result was also that with the same limits the density of mobile dislocations varies between 10 and 35 %.

Caillard and Martin, in aluminium, have observed different non usual gliding planes (37). Some interaction mechanisms (attractive or repulsive trees) between dislocations have been studied and creep studies are in progress (38) .

We can in abstract say that the in situ experiments can be useful to study :
- geometrical informations on interaction mechanisms between dislocations on clusters and dislocations sources ; - collective behaviour ; - quantitative informations.

These two last types of informations demand a lot of experiments. Quantitative informations are obviously easier when the geometry is simple (low temperature in BCC materials).

Some discussions are around the validity of in situ plastic deformations in H.V.E.M. because of the production of point defects above the threshold. See for this point the discussion of Kubin and Martin (39) and Jouffrey (29). The best way would be to work below

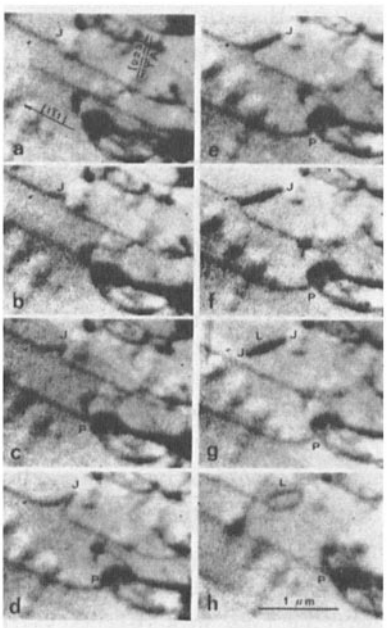


Fig. 8.Deformation of Nb at 110 K. Formation of a closed loop from a jog (F.Louchet).

the accelerating voltage corresponding to the threshold what is not always very easy but often possible. The influence of defects seems troublesome at low temperature. The diminution of the accelerating voltage limits the available thickness and the influence of surfaces can become not negligible. Therefore in many cases the experimentalist has to find an optimum for his experiments.

β)-Experiments within environmental cells. Since the first proposal of Marton (40) in 1935, many attempts have been done to observe samples in particular atmospheres. We refer to Swann (41) and Parsons (42) for a review respectively in metallurgy and in biology. These cells can be roughly classified in two types : - window type; the object environment is separated from the column of the microscope by electron transparent windows. So the pressure is rather well defined but their use is delicate. This type of cell can be preferable for low temperature work. - differentially pumped cells; in this attachment there is a chamber where the vacuum is intermediate between the one of the microscope and the pressure in the cell. There are four apertures to align in this case.

The applications of these cells can be :

- Low temperature application : solidified gazes (43)or liquid materials at room temperature. Radiation induced corrosions as in the case of silicon which has been reported by Swann (41).
- Room and high temperatures : it is possible to study the influence of different gazes as nitrogen, oxygen .... Swann has studied for instance the reduction of hematite to magnetite at 650° C in a 5 % H_2/Ar mixture at 30 torr (41). Other studies on water reaction with concrete have been tried.

γ)- Radiation damage. During the observation in the microscope, the sample can suffer different types of damage which can be troublesome in some cases. At the opposite, H.V. E.M. can be a unique tool for researchers who are interested in.

The deposited energy gives an increase in temperature which is, in most of the cases, not important at all ($\sim$ 1 degree). It depends on the sample and its connection to the specimen holder. In defavourable cases the heating can be however very important.

The two principal effects are : formation of point defects and ionization.

- Formation of point defects. The order of magnitude of the cross section corresponding

to the displacement of one atom out of its natural site is about 50 barns, what is quite small. However this processus is cumulative and so can give quite strong effects. The cross section is given by the Mac-Kinley and Feshbach (44) expression which underestimates the values for heavy elements. So it is preferable in these cases to use Oen's results (45)..

The number of pairs created by second and by atom is given by $C = \emptyset \sigma_d N_d$ with $N_d = E/2E_d$ as a first approximation when E_d is the energy of displacement and E the energy of the primary electrons. $\emptyset$ is the flux of electrons ($\sim 10^{16-20} e/cm^2/s$).

The energy transferred in a knock-on is given by : $T = \frac{2E}{M_{at}c^2}(E + 2m_o c^2) \sin^2 \frac{\alpha}{2}$ where α is the scattering angle of the incident electron through this processus.

We see that the number of pairs can be quite high after a few minutes ($C \sim 5.10^{-5}/s$. for 1 MeV).

The order of magnitude of the displacement energy is 166 keV for Al and 1.2 MeV for gold.

So it is possible to study the kinetics of formation of clusters. First, interstitial clusters are formed. Vacancy clusters can be observed later on. It is also possible to study in a very efficient way the interaction of point defects and pre-existing defects as dislocations, planar defects and defects in volume. The influence on precipitation, ordering, formation of new phases, creep, recrystallisation, the formation of voids is also interesting. In this last case many attempts have been done as was reported by Makin (46).

The simulations of swelling which can happen in nuclear reactor cores because of the neutron radiation, have been extensively studied. The conclusion is that there are some differences between the neutron and H.V.E.M. experiments but this last technique enables to get very rapid useful informations on the role of impurities, of defects, grain boundaries on the swelling rate.

See also on these problems on radiation damage the review of Urban (47).

- Ionization. These problems have ben reviewed in the inelastic events.

b) - Chemical analysis. It is possible to use H.V.E.M. in an efficient way to perform chemical analysis by using inner shell excitations. This method is quite useful principally for light elements but is also available for heavier materials by using L, M, .. excitations. The available thickness is superior to 2000 Å instead of 300 Å about 80 kV.

Two methods can be used (20, 21) :

α)- an area is selected by one aperture and the spectrum is obtained. This is shown on figure 9.

β)- an image is obtained by using only the electrons which have lost the energy corresponding to the inner shell excitation. So it is possible to localize the areas where is

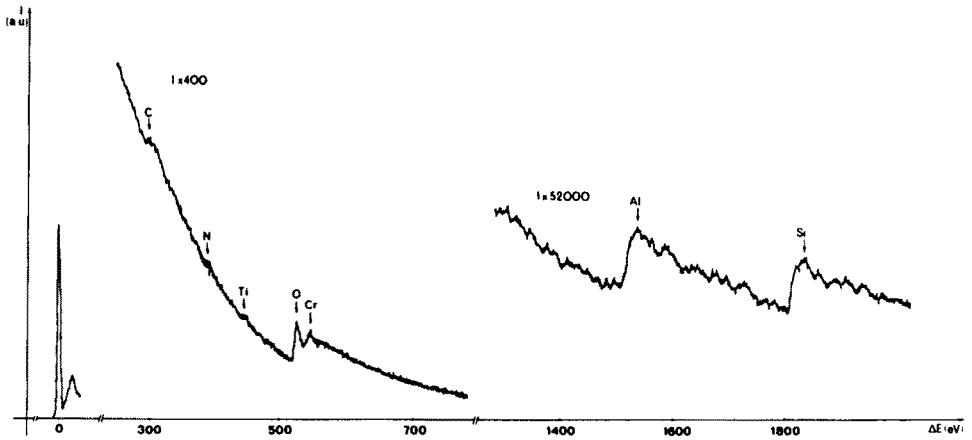


Fig. 9 - Spectrum of a thin section of a pathological lung (21).

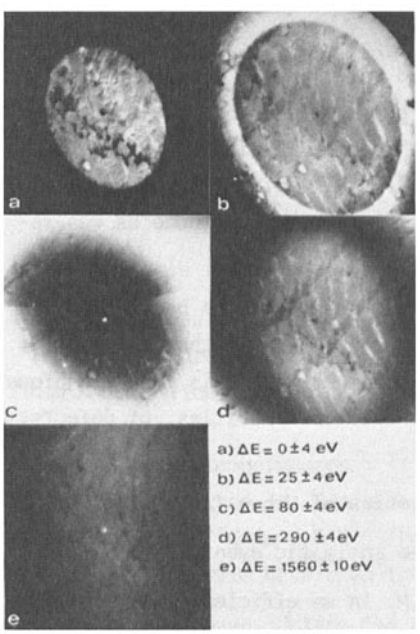


Fig. 10. Filtered images obtained by selecting a given kind of electrons. For instance, in d the electrons which have been used for the image had an energy E = 1 MeV-1560 eV ± 5 eV. In c and e the small cluster (∿ 450 Å in diameter) appear white (the corresponding losses are characteristic of the L and K excitations in Al.

present a given element. The figure 10 shows such application.

c) - Miscellaneous applications. In this part we can note : three dimensionnal reconstruction by use of quantitative stereography and also integrated stereo technique (48) concerning in the use of a system of parallel cylindrical lenses which contains as many as ninety pictures of the same area (e.g. ± 45°) ; magnetic materials (49) ; melting aspects and solidification (50) ; semiconductors (51) ; recrystallisation in aluminium (52, 53,54), in copper (55), in titanium (56) and some other materials, phase transitions in vanadium sesquioxyde (57) ; composites materials (58,59), cavitation (60) ; ceramics and minerals (61, 62, 63) ; polymers (64,65) ; ionic crystals (66) ; amino acids (67) ; superplasticity (68).

6)- Conclusion

In most of these studies the interest is related to the important available thickness which gives an aspect of the sample closer to the bulk material. For instance in precipitations it is often possible to observe big precipitates or tangles of dislocations. Sensitive materials are also interesting to be studied because of the β^2 law (see ionization).

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