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Measurement of Elastic Cross-Sections for Gases

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In the ESEM and VPSEM the incident electrons travel through low pressure gas to reach the specimen. The beam is therefore scattered, resulting in a loss of contrast and beam current. This is of particular important in microanalysis because the X-ray signal will come from the entire width of the scattered beam. To account for such effects it is necessary to be able to predict the magnitude of scattering expected from a gas interacting with an electron of given energy. This can be done using Monte Carlo simulations [1], but to apply these techniques it is necessary to know the cross-section of the gas. Even for a simple gas this cross-section may be difficult to predict unless the atomicity of gas (i.e. the effective atom cluster size of the molecule) is known.

We have therefore measured the cross-section experimentally using the technique of Gauvin [2]. In this method, a stationary beam of electros is focused on to a small object. The X-ray signal from the object is then measured as a function of gas pressure. As the pressure increases electrons are scattered into the skirt and so miss the object and the X-ray signal falls. The total elastic cross-section of the gas can then be calculated from Eq. (1) [3]:

$$\ln(I_p) = -\frac{D\sigma_{el}^{t}}{RT}P + \ln(I_0) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

where I_p is the measured X-ray signal at a pressure P, D is the gas path length, R is the gas constant and T is the temperature. σ can thus be deduced from the slope of the $\ln(I_p)$ vs P curve.

The experiments were performed in a Hitachi S-3500N VPSEM with beam energies of 10, 15, 20, 25 and 30keV. For each energy level, the gas path length was kept as 12mm and the magnification was x18K. The X-ray intensity was obtained by collecting the X-ray signals emitted by the surface of a fine aluminum wire of 25 μm diameter inlaid in epoxy resin. To avoid charging build up resulted from the nonconductive resin, the specimen was coated with gold. The selected fluorescent X-ray peak in this experiment was Al kα whose characteristic energy is 1.5 keV and the counting time was 300 seconds. Once the EDS starts to collect signals, the only variable was the gas pressure, which was varied from 1Pa up to 270 Pa and then back from 270 Pa to 1 Pa. The tough task for us was the introduction of the gases other than air into the system. In our experiments, a small gas container with a gas regulator was connected directly to the intake nozzle of the system by a very short gas pipe system.

A linear relationship between the gas pressure and ln (I) are affirmed by our experimental data as shown in Fig.1. Based on the slope in Fig.1, the total elastic cross-section for gases could be calculated by the derived equation above. The calculated cross-sections of Air and He are listed in Table 1. There appears a deviation about the order of 10 between the experimental cross-sections data and the theoretical total cross-section computed from Mott scattering theory [4], which may result from several possible factors: the determination of the gas path length (GPL), the alteration of the gas temperatures inside the specimen chamber, the stability of gas pressure and the introduction of gases. In our experiments, the working distance is considered as the gas path length while the actual GPL may be longer than the working distance, because some gas could enter the upper column through the pressure limiting aperture; secondly the actual temperature of the gas inside the chamber may significantly different from its temperature outside the chamber since the expansion of the gas into the low vacuum of the SEM causes cooling. Consequently several tests were conducted to confirm the GPL and temperature variation and our experimental results indicate that it is reasonable to choose the working distance as the gas path length and the temperature variation is small enough to be ignored.

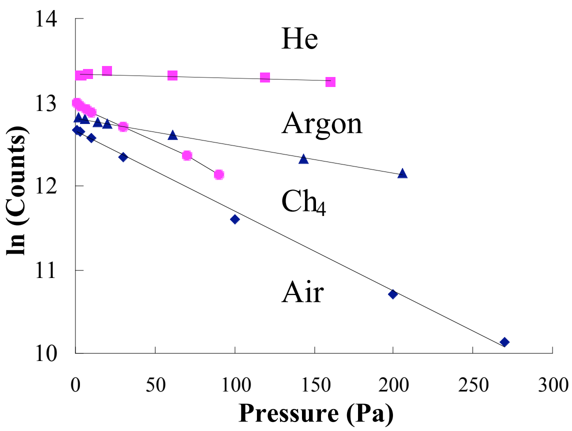

FIG.1 Linear Relationship between Gas Pressures and X-ray Intensities

TABLE 1 Elastic Cross-section of Air and He

Beam Energy (KeV)	Elastic Cross-section (cm^2/atom)			
	He		Air	
	Measured	Mott	Measured	Mott
10	3.11E-18	4.39E-19	5.25E-17	6.79E-18
15	2.76E-18	2.96E-19	4.49E-17	4.61E-18
20	1.73E-18	2.22E-19	3.28E-17	3.49E-18
25	1.04E-18	1.81E-19	2.73E-17	2.82E-18
30	1.49E-18	1.53E-19	1.52E-17	2.37E-18

The problem which causes most concern is the gas pressure control system. In the VPSEMs, the pressure is controlled by a computer operated leak valve and a suitable feedback circuit [3]. Typically this leads to a condition in which the pressure cycles slowly varies with time about the nominal value as the valve opens and closes, and resulting the difficulty of the accurate determination of the pressure and a misunderstanding of the gas condition inside the chamber. These problems could directly affect the accuracy of our cross-section calculation.

In conclusion, the linear relationship between the gas pressure and the ln (I) are confirmed and the theoretical basis of the calculation of the total elastic cross-sections (□) has been validated. The data shown clearly illustrates how different gases scatter electrons by different amounts. Although the problems identified might contribute some error we do not believe that any of these is significant enough to explain the difference between the measured and theoretical values. This suggests that in this pressure regime most gas molecules are aggregated in clusters. Further work is in progress to test this hypothesis. Also in our future work, a capacitance manometer will be used for more precise gas pressure control to produce a more accurate measurement of the elastic cross-section of gases.

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