

SCANNING ELECTRON MICROSCOPY WITH LOW ENERGY ELECTRONS

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ABSTRACT

A method of scanning electron microscopy (SEM) of nonconductive specimens, based on measurement and utilisation of the critical energy of electron impact, is described in detail together with examples of its application. The critical energy, at which the total electron yield curve crosses the unit level, is estimated on the base of measurement of the time development in the image signal from beginning of irradiation. The method is programmed and implemented as a module to the controlling software of the microscope type VEGA, where it secures fast search for the critical energy value.

1 SPECIMEN CHARGING IN THE SCANNING ELECTRON MICROSCOPE

When observing non-conductive specimens in SEM, one of the crucial obstacles is the specimen charging, which is caused by difference between the numbers of impacting and emitted electrons. In a nonconductive specimen the dissipated charge does not flow away from the illuminated point fast enough. Then the electric field of the localized charge influences the trajectories of both the primary and signal electrons moving towards the detector. So the geometry as well as the intensity scale of the image is damaged. The surface charge and its potential spontaneously rise as long as the charge equilibrium is achieved, which usually corresponds to local potential differences in the keV range. If we work above so-called critical energy, the surface is charged negatively (see Fig. 1) and negative charge retards the primary electrons. Below the critical energy the surface is positively charged and some of the emitted electrons are attracted back to the specimen.

The threshold between the positive and negative charging is an important point. Here the amount of electrons, impinging on the specimen and emitted from it, is identical. In this case no charge accumulates on the specimen surface. Because at tens and units of eV the electron yield again falls below 1.0, two critical energies are available for nonconductive targets. Of those, the upper one is suitable for the experiment, namely for two reasons. First, this energy of impact is spontaneously established because changes in the impact energy owing to charging, when proceeding from both higher and lower side, are always directed toward this point. Second, a higher energy of electron impact is generally more advantageous for taking high quality micrographs.

2 ELIMINATION OF THE SPECIMEN CHARGING

The classic approach to minimizing the charging is to increase the specimen surface conductivity. It can be reached by coating it with a thin metal or other conductive layer. An alternative way, suitable for biomedical specimens, consists in staining them with salts containing heavy metal atoms, which are also able to increase the conductivity. Both these methods are generally successful with exceptions of some fibrous structures. However, the recently used environmental SEM with the specimen placed in the gas pressure above 20 Pa eliminates the problem via compensation of the surface charge by gas ions.

The optimum electron energy for observation can be found by means of comparison of a pre-irradiated area with its surroundings immediately after lowering the magnification. This enables one to reveal whether after the first irradiation the emission changes, which should happen at all energies except the critical energy. The practical knowledge of this trick contributed to the proposal of a new method published in 1994 by Frank and Müllerová [2]. For determination of the critical energy the authors suggested to utilise the measurement of the time dependence of the total electron emission signal. The method is called the non-charging electron microscopy.

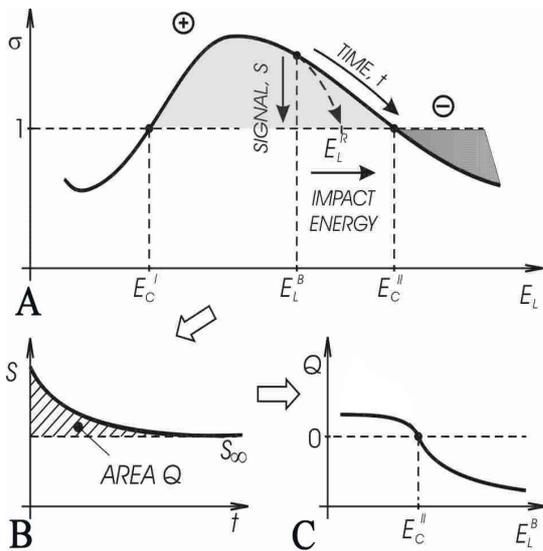


Fig. 1: The typical dependence of the total electron yield on the electron landing energy (A). The time dependence of the image signal (B). The dependence of area Q on the electron landing energy (C).

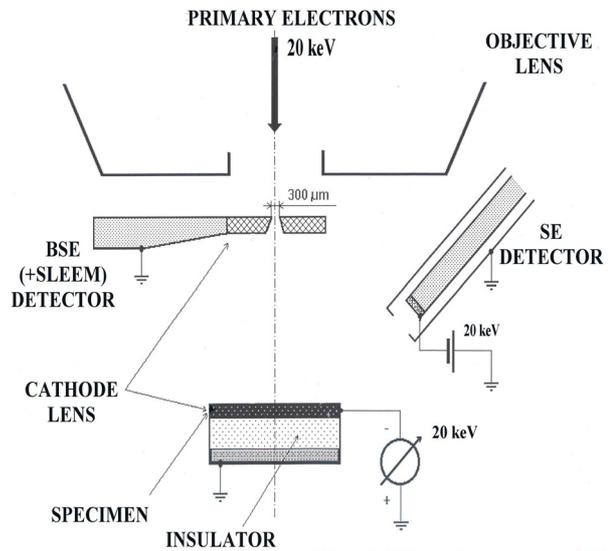


Fig. 2: Scheme of the specimen chamber configuration with the cathode lens.

3 PRINCIPLE OF THE METHOD OF IMAGING AT CRITICAL ENERGY

The magnitude of the surface potential depends on the total electron emission, which is defined as the number of emitted particles per one impacting electron. The total electron

emission is then $\sigma = \delta + \eta$, where δ and η are the yields of the secondary (SE) and (BSE) electrons emission, respectively. In Fig. 1A we see the typical dependence of the total electron emission on electron energy. Between E_C^I and E_C^{II} more electrons are outgoing than incoming. This results in positive charging of the specimen surface. The inverse situation, when the specimen surface is negatively charged, is marked as a dark area.

It has been proved that the specimen charging projects itself into changes in the detected image signal as indicated in Fig. 1A,B. The integral change, i.e. the area Q under the $S(t)$ curve, can be taken as a measure of the charging rate. If a set of measurements at different impact energies is made, we can determine the energy for which the area was smallest or even zero (see Fig. 1C); this is the critical energy. The signal has to be collected from an area not illuminated before, in order to secure identical initial conditions for each measurement.

4 EQUIPMENT

Figure 2 shows configuration of the microscope adapted, by inserting the cathode lens, for the method of non-charging electron microscopy. Primary beam passes the whole column with energy at least 10 keV and it is retarded by the electric field in front of the specimen surface. The specimen is insulated and negatively biased. This adaptation is suitable for any standard SEM; we use the microscopes VEGA TS 5130 MM (Tescan Brno) and Tesla BS 343.

5 SOFTWARE FOR DETERMINATION OF THE CRITICAL ENERGY

A program module has been created as a part of the control software of the VEGA microscope. The module enables easy and fast determination of the critical energy. It is written in the Microsoft C++ programming language and it runs under operating system Windows 95 or higher. For determination of the critical energy it is necessary to examine a range of the landing energies, where the critical energy probably appears (see [3]). The module adjusts the first landing energy and records the time response of the detected signal in different points of specimen (see Fig. 3A,B). The resulting time dependences of signal are processed with the algorithm described in [3], which determines the mean dependence and its integral (i.e. the area under the curve, see Fig. 3C). This value is stored in a buffer. Then the module sets another landing energy from the interval and the measurement continues. The result is a dependence of the integral on the landing energy of electrons (Fig. 3D). The critical energy for the examined specimen corresponds to the point where $Q(E)$ crosses the zero level (see Fig. 1C). The specimen bias is then set so that the impact energy of electrons is just the critical one and the final image of the specimen is recorded.

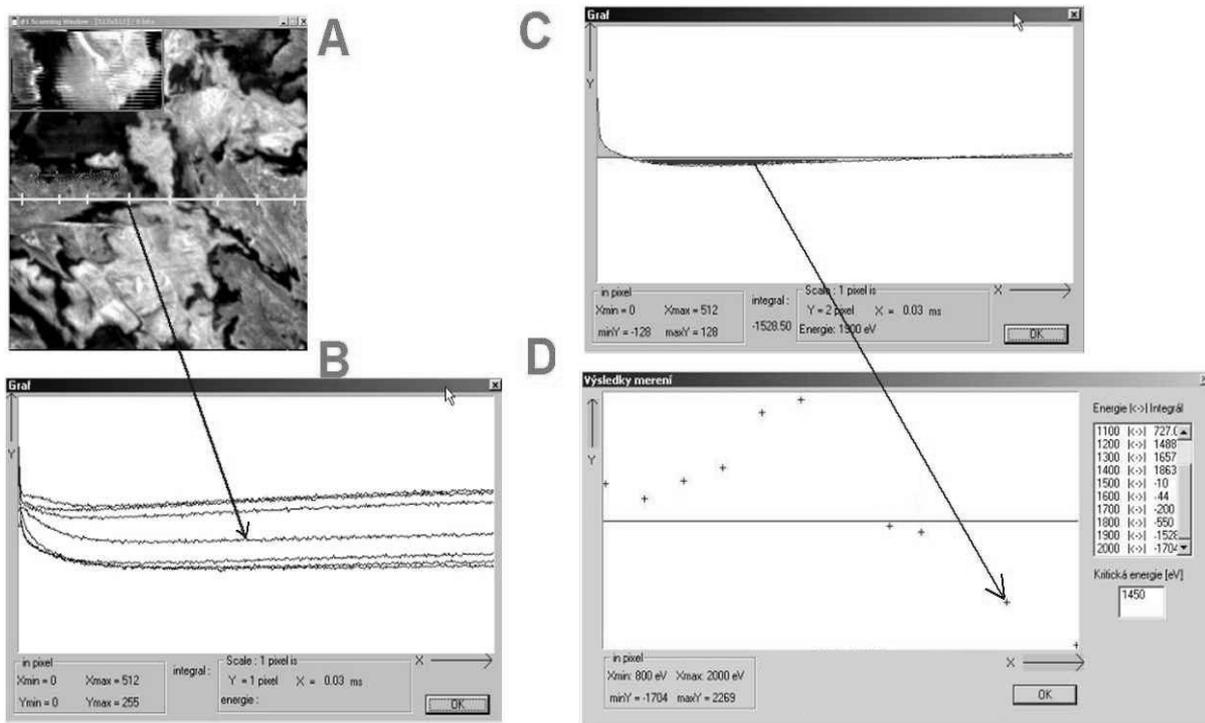
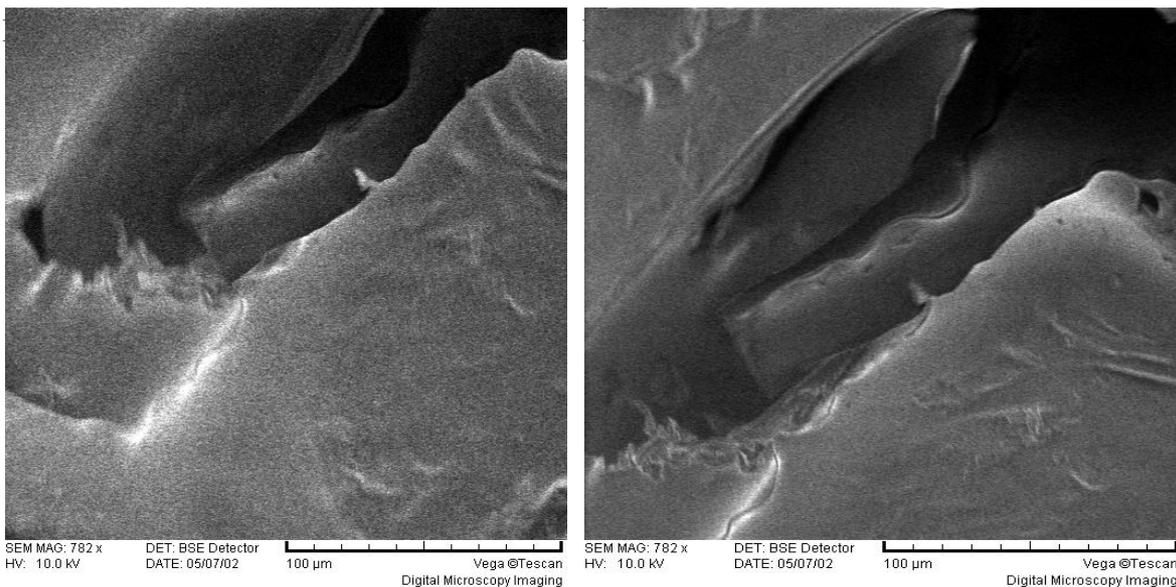


Fig. 3: *The procedure of finding the critical energy.*

6 VERIFICATION OF THE METHOD

The method has been verified on a set of specimens. One of them was the soft elastic plastic foam (other examples see [3]). It was not made conductive before testing. Under



standard conditions the specimen is charged, as shown in Figure 4. We can compare the

image with that taken at the critical energy 1450 eV (see Figure 5). Obviously the traces of charging are substantially suppressed and well negligible at 1450 eV.

7 CONCLUSION

The software for determination of the critical energy is fully operational and can be used for observation of non-conductors. These need not be coated with conductive layers so that the specimen preparation is simplified.

The measurement time is a few minutes and depends on values of input parameters. After the optimum electron energy is found, it can usually be utilized also for other fields of view on the same specimen.

The method is not suitable for observation of all nonconductive specimens. The main obstacles are the requirements on the topology of a specimen surface, which has to be smooth with a roughness not exceeding tens of μm . If the specimen consists of several different areas of a greater size then they have to have similar values of the critical energy.

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REFERENCES

- [1] Frank, L. - Zdražil, M. – Müllerová, L: Scanning Electron Microscopy of Non-Conductive Specimens at Critical Energies in a Cathode Lens System. *Scanning* 23 (2001) 36 – 50.
- [2] Frank, L. – Müllerová, I.: Zero-charging electron microscopy in a cathode lens equipped SEM. *Proc. ICEM 13, Paris 1994, vol.1, 139 – 140.*
- [3] Mika, F.: Diplomová práce. FEKT VUT Brno, 2002.