HUMIDITY ADJUSTMENT IN ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY

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Abstract

Possibilities of humidity adjustment and measurement in the specimen chamber of the environmental scanning electron microscope (ESEM) at low pressure are described in this work. Optimal pumpdown sequence and filling-in of water vapors to the specimen chamber problems are solved simultaneously. Suggested cycle serves for obtaining of suitable environment in the vicinity of the examined specimen and can be exploited for observation of battery mass structures in the process of their utilization.

Introduction

A well known advantage of ESEM is the possibility of observation of specimens in the environment of different gases or water vapors. Gaseous environment enables observation of insulation specimens without influence of the charging effect. Presence of gases or vapors enables also observation of wet or water containing specimens, phase interfaces, etc. Regarding to the effectiveness of ionization of water vapors by electrons, the usage of environment of water vapors brings also considerable higher signal amplification in comparison with the environment of other gasses when ionization detector is used for signal detection. ESEM however has also its limitations. The pumpdown process, where ambient air is replaced by water vapors, used when hydrated systems are imaged, can cause significant specimen changing or damaging due to uncontrolled water evaporation and boiling. In this case, it is necessary to have information about humidity of the environment in the specimen chamber.

Problem Formulations

For optimization of the pumping procedure, we need to obtain information about the amount of waters vapor in the vicinity of the specimen. The most common method used for the measurement of the environment humidity is the method based on absorption of water into polymer materials [1]. Impedance sensors, capacity sensors or sensors based on measurement of changes of mechanical properties are used for this purpose.
Impedance sensors use a polymer structure to which hydrophilic ions are bound. Electric charge of these ions is compensated by opposite ions. When humidity is increased,

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hydrophilic particles absorb water. A typical representative of such a material is Nafion as a cation exchanger with firmly bound sulpho groups, while hydrogen ions are the contraions. The impedance of the sensor is measured by an alternating current. The changes of the detector impedance can be up to several orders. The impedance dependence on the environment humidity is usually non-linear, and such sensors show certain hysteresis.
Capacity sensors use a hydrophobic polymer as a dielectric material of the capacitor (material with small permittivity is chosen). Even a small amount of water absorbed on the surface of the detector, with regard to the high permittivity of water ($\varepsilon_r = 80$), significantly changes the permittivity of the dielectric material and, consequently, the capacity of the device. The dependence of the capacity of the sensor on the amount of water is linear. Polyamides or derivates of cellulose are used most frequently as dielectric material.
The sensors based on measurements of changes of mechanical properties contain a polymer, e.g. polyvinyl alcohol and finely dispersed carbon particles. Polymer volume changes due to moistening or drying, result in a change of ohmic resistance among conductive grains of carbon *(swelling-type sensors).* This resistance is then measured. Mechanical changes caused by the change of humidity are also used in piezo-resistor sensors.
It is possible to use microwave sensor for measurement of humidity [2], but this method for our purpose is too complicated.
Two criteria exist for optimization of maximum humidity adjustment at specimen chamber filing sequence. A steady state must be achieved, when in the vicinity of specimen the environment of saturated water vapors exists and the whole pumping as well as fillings process must cause the least possible evaporation of water from the specimen. Otherwise, boiling of water inside the specimen occurs and, consequently, the specimen can be damaged.

Problem Solutions

Humidity measurement

For our purposes we used a capacity microprocessor controlled sensor [3] equipped with an element for temperature measurement. The output signal in our measurements was voltage.
We placed the sensor into the specimen chamber in a distance approx. 8 mm from the specimen surface. The used sensor is specified and calibrated, for the humidity measurement at the atmospheric pressure. Therefore, we had to carry out a new calibration. As the “humidity value = 0” level we chose the voltage value measured at a long-term exposition of the sensor to vacuum at pressure of 10^{-3} Pa. The voltage was $V_h = 4.12$ V. As the comparative “humidity value = 100” level we chose a voltage of $V_h = 6.55$ V. This voltage was measured at the pressure of 1100 Pa, when the environment of saturated water vapors at room temperature was created in the specimen chamber.
Following repeated experiments at pressure reducing under 1100 Pa allowed to state the dependence of “humidity level” - eq. 1:

$$\textit{humidity level} = 8.97 \times 10^{-3}\, p + 1.82 \qquad (1)$$

The obtained dependences are presented in Fig 1.

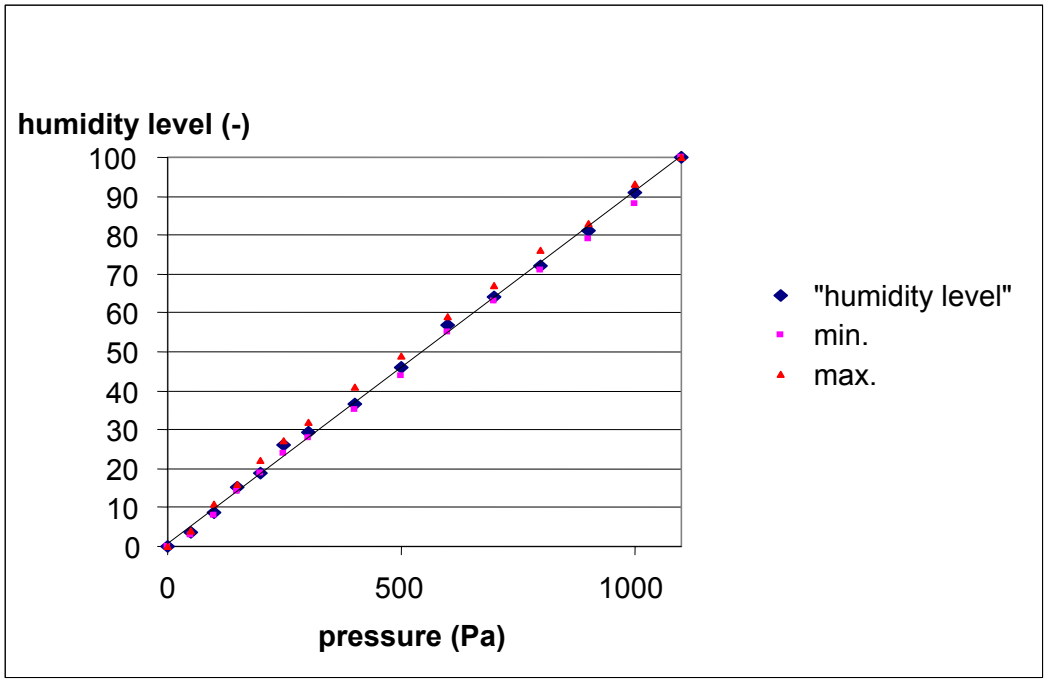

Fig. 1 *Dependence of the "humidity level" on the pressure for a saturated state.*

Humidity adjustment

Several variables influence the process of pumping and filling-in water vapors. The most important ones are the initiating [4] humidity in the specimen chamber, the temperature of water in the water vapors generator, range of pressures at the pumping process and the number of cycle repetitions.
Next experiments were therefore oriented to the solving of these problems. As a result of these experiments the optimum pumping procedure was suggested. This procedure consists of following steps:

1. Injection of a small amount of water (0.5 ml) before the specimen chamber pumping.
2. Start of pumping procedure. Evaporation of water begins and continues until the original humidity level in the specimen chamber is achieved. When water droplet is evaporated the pressure begins to decrease. The pumping proceeds to the moment, when pressure decreases to 700 Pa.
3. At pressure of 700 Pa the irrigation valve is opened and water vapors fill-in process from the generator begins. Pressure increases; the process is finished at the pressure of 1100 Pa.
4. Repeating of steps 2 and 3 at least four times. It would be adequate for achieving of sufficient humidity.

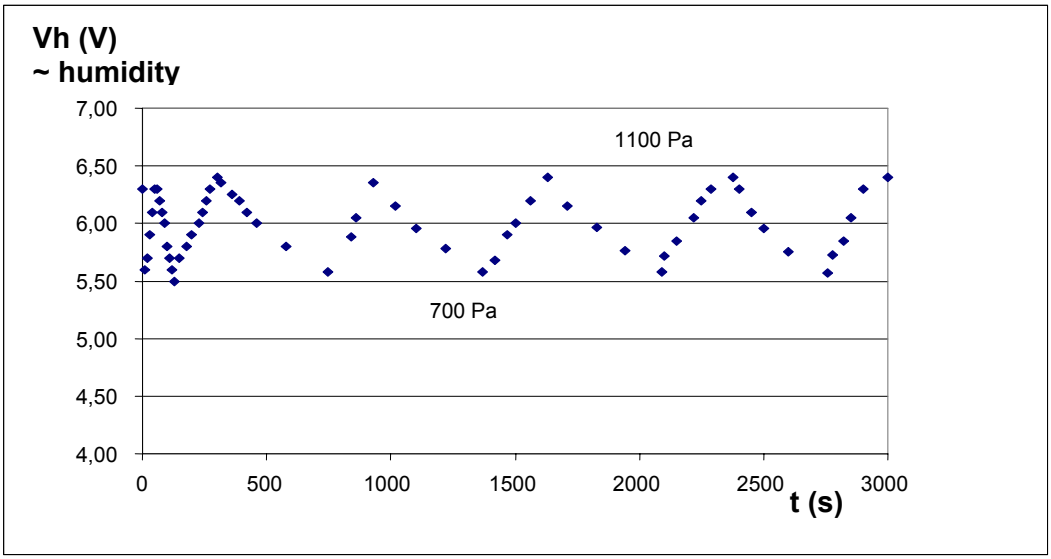

Fig. 2 *Pumping cycle.*

Conclusion

The process creation of saturated water vapors environment in the specimen chamber of the ESEM is very important for optimization conditions for wet specimen structure observation. The environment and the whole pumping process enable to observe water containing specimens as well as battery masses with the content of electrolyte. Many pictures of structure of battery masses were made under saturated water vapors conditions. The pictures will be shown at oral presentation.

Acknowledgements

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