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Dynamic Volume Change Measurement in Environmental Scanning Electron Microscope

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Moisture transport is one of the most important phenomena in organic materials in a variety of fields. As the result of this process in the fields of biology, polymer, food, and agriculture, volume change like swelling and shrinkage have received a lot of theoretical and experimental attention. However, many questions remain about the nature and behavior of moisture hydration and dehydration because of the limitations of the experimental techniques to study it. Although many methods are available to measure the static volume change both directly and indirectly when volume reaches its equilibrium, for example tilting sample stage in ESEM to make stereoscopic images, it is difficult to determine *in situ* the volume evolution during relative humidity (RH) changing, especially at the beginning of RH change due to the time limitation to tilt the stage without introducing additional errors. We describe here a procedure that provides a rapid way of measuring volume change as the function of relative humidity and time.

The object to be studied (raw rice powder) is put inside a FEI XL-30 ESEM under low vacuum mode, followed by the optimum pump down procedure in order to minimize the volume artifacts at water vapor flushing step. [1] Sample is held 30 minutes at each RH environment before image capturing at equilibrium state. Meanwhile video images are recorded from the beginning of relative humidity change by the pre-set frame capture interval. The projected area is then measured by ImageJ software. Figure 1 shows the principle of stereoscopic images and the reconstructed 3D view at different relative humidity by MeX software, from which the desired volume value can be calculated. [2] If the volume change is isotropic, the dynamic behavior of volume change can be indirectly monitored by the change of projected area, hence the arbitrary volume value at any time will be interpolated from the equilibrium volume values at both initial and stable states.

Figure 1 also shows that the surface roughness of object reduces with increasing volume when RH rises from 66% to 99%. Figure 2 shows the sequences of the normalized projected area change in different RH environments as the function of time. This experiment demonstrates how fast the specimen volume change with the variation of RH. The projected area change, or the volume change, in zone **II** (92% to 99%RH) is much faster than that of in zone **I** (67% to 92%RH) in hydration process though the RH gradient in zone **I** is much higher. Comparatively, zone **V** (87% to 50%RH) has much less volume change than that of in zone **III** (99% to 97%RH). Most of the volume changes (both swelling and shrinkage) happen around the saturation condition (100%RH). All the phenomena are additionally verified in figure 3, which shows the comparison of the normalized volume change by stereoscopic images and the normalized projected area change. Therefore the combination of stereoscopy technique and ESEM is efficient in studying both static and dynamic volume change behaviors of organic materials in various relative humidity environments. [3]

References

[1] R.E. Cameron and A.M. Donald, *J. Materials Sci*. 173 (1994) 297.
[2] J. Russ, The Image Processing Handbook, 4th Edition, CRC Press LLC, (2002) 64.

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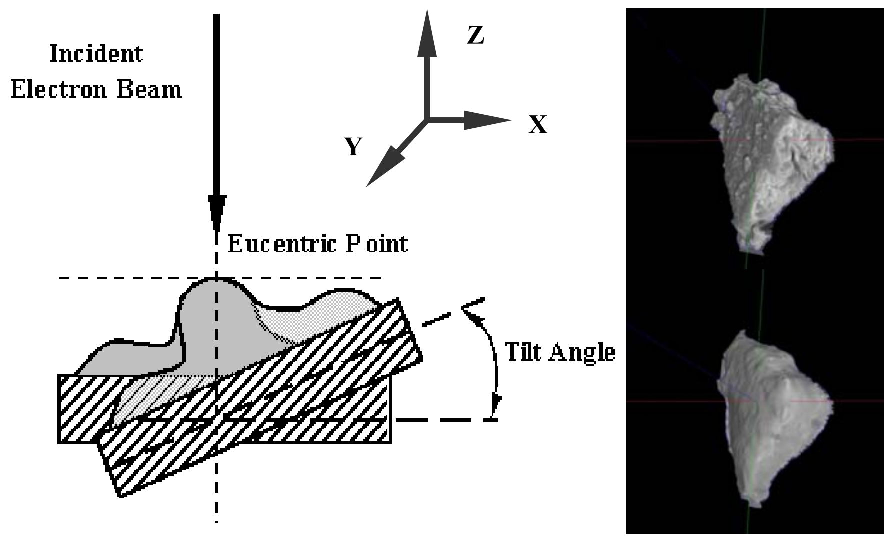

FIG. 1. Principle of stereoscopy in ESEM and 3-D view of rice powder in 66%RH (above) and 99%RH (below).

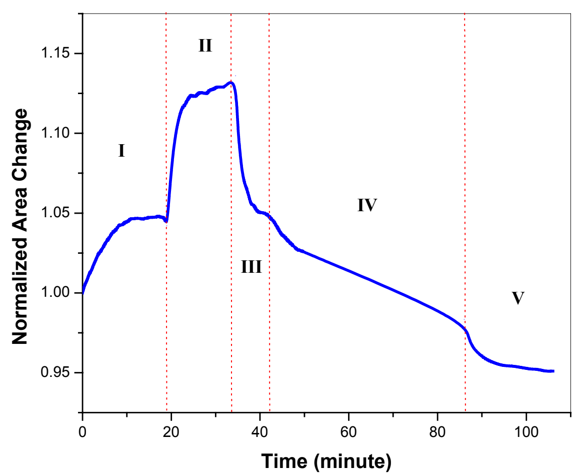

FIG. 2. Time sequences of the projected area change in different RH environments: zone I (67% to 92%RH), zone II (92% to 99%RH), zone III (99% to 97%RH), zone IV (97% to 87%), and zone V (87% to 50%RH).

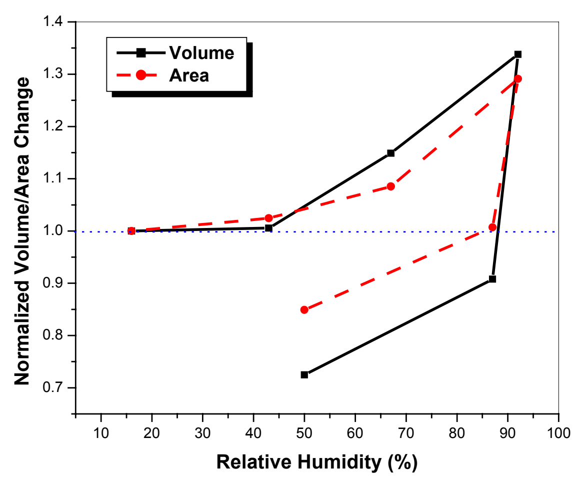

FIG. 3. Comparison of volume change (by stereoscopic images) and projected area change (by frame recording) as the function of relative humidity.

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