Advanced approaches for the analysis of micro- to nano-quartz particles using SEM, ESEM, FIB-SEM, SBF-SEM

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Background incl. aims
Quartz micro- to nano-particles are used in a wide variety of industries, especially in the semiconductor industry, optics, as a filler for epoxy resins, etc.. During the preparation of these particles, it is essential to know and control one of the crucial parameters - the particle size distribution (PSD). The production process itself significantly affects the PSD as well as other morphological characteristics. The shape, texture, and size distribution of the particles have a significant impact on the material's mechanical strength, density, electrical properties, and thermal properties. Various methods are used to evaluate PSD. This paper discusses laser diffraction (LD), and microscopy methods: Scanning Electron Microscopy (SEM), Environmental Scanning Electron Microscopy (ESEM), Focused Ion Beam Scanning Electron Microscopy (FIB-SEM) and Serial Block-Face Scanning Electron Microscopy (SBF-SEM). The applications, sample preparation requirements, advantages and accuracy of PSD analysis for the above methods are compared and discussed. The study focuses on ESEM as an interesting, useful and cost-effective technique with many advantages for the study of quartz micro- to nano-particles.

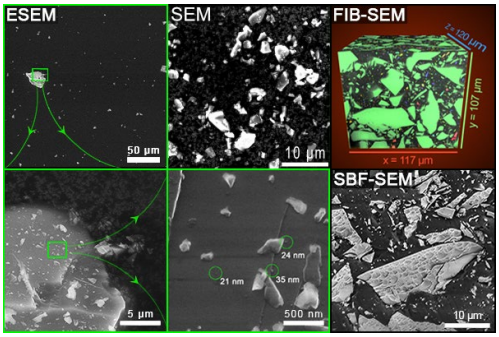
Methods
Quartz powder was first measured using LD. For SEM and ESEM imaging, a non-commercial ESEM AQUASEM II was used. The samples were observed in vacuum and in water vapor at 200 Pa, at 2 magnifications to cover all particle sizes, and using three different types of detectors to detect backscattered and secondary electrons. The particles embedded in the resin were observed using high-resolution ESEM QUANTA 650 FEG at a water vapor pressure of 200 Pa equipped with a Gaseous Secondary Electron Detector. Sub-nanometer particles were observed with high magnification and under pressure of 200 Pa. A procedure has been developed for particle analysis using the Mountains SEM 9 Expert software. Next, the particles embedded in the resin were analyzed by FIB-SEM Tescan Amber. From the resin cube, layers with a thickness of 100 nm were successively sputtered with a Ga+ beam. The last method was SBF-SEM, where layers were sequentially cut from a 1x1x1 mm resin cube using a ConnectomX ultramicrotome in a Tescan Clara UHR SEM microscope.

Results
The result of particle analysis by LD is a plot of the cumulative distribution function of particle sizes. The size parameter is the equivalent spherical diameter. Comparisons were made between SEM/ESEM analyses of the particles, as well as between the powders themselves and their embedding in resin. Our results confirm that the ESEM provides accurate shape and size characterization of quartz particles at vapor pressures in the order of hundreds of Pa and working distances in the low mm range, regardless of the type of detector used. These conclusions were confirmed by the analysis of smaller particles (0.5 - 2 µm). ESEM has also enabled the identification and analysis of particles tens of nm in size. Images acquired with FIB-SEM were processed with 2 software. ORS Dragonfly provided an advanced view of the incorporation of particles embedded in resin. The second, IP-SDK, allowed precise distribution and annotation of individual particles, which allowed accurate statistical evaluation of their distribution, sphericity, volume, aspect ratio, etc. Images obtained using the SBF-SEM method showed that during the cutting of each layer, the particles were disintegrating, breaking off, and being pulled out, preventing proper particle analysis.

Conclusions

LD is very fast, efficient and widespread method of particle analysis over a large size range. Its disadvantage is the impossibility to know the real morphology of the particles. SEM and ESEM are able to image not only the powder particles but also the particles embedded in the resin, making it possible to fully control the production and use of these particles in other products, which is their great advantage. The ability of SEM and ESEM to image the real morphology and shape of the particles also allows quantification of other morphological parameters, and provide information on the exact shape of the particles. ESEM is a full-fledged and innovative particle analysis tool, and brings a number of advantages, such as a significant reduction in sample charging, thus avoiding the formation of image artifacts that distort the resulting statistics. The potential risk of microscope damage due to contamination of released particles is also significantly reduced. It allows imaging of non-conducting particles without prior modification, and another important advantage is the ability to reveal structural details of even sub-micrometer sized particles. FIB-SEM is one of the most modern, and very advanced tools that allows the measurement of 3D morphological properties of particles, in particular their spatial distribution. However, it is crucial to choose the correct particle labelling procedures, as the accuracy of the resulting distribution depends on this. ORS Dragonfly is a great imaging tool, while IP-SDK provides better statistical evaluation capabilities. SBF- SEM proved to be unsuitable for this type of sample due to the high hardness of the resin.

Graphic:



Keywords:

particle-size distribution, ESEM, SBF-SEM, FIB-SEM

Reference:

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