Microcapsules (MC) are used in many research areas such as the development of self-healing materials [1], drug delivery [2] and other areas where controlled release of an active material, protected from its environment by encapsulation, is desired. In this work, we compared the surface morphology and shell thickness of melamine-formaldehyde microcapsules using a scanning electron microscope (SEM) equipped with a field emission gun (FEG). Cyclohexane was encapsulated by a melamine-formaldehyde wall via polycondensation in an oil-in-water emulsion. Capsules with different amounts of core content were prepared. A later removal of the core by soxhlet extraction provided the hollow microcapsules used for characterization. The capsules were embedded in an epoxy matrix and samples were prepared using microtomy to study the shell thickness. These capsules, designed to encapsulate self-healing agents, are spherical in shape with an approximate diameter between 5 and 30 µm. SEM images show that the shell thickness varies from 650 to 700 nm for MC-VI (F/M ratio = 3.25, pH = 5.0, core content ~ 85%) whereas in the case of MC-VII (F/M ratio = 3.25, pH = 5.0, core content ~ 60%), the variation is from 1.10 to 1.25 µm. In order to measure the shell thickness without the epoxy matrix, a thin strip was removed from the surface of an individual microcapsule using Focused Ion Beam (FIB). In this case, the measured thickness values are nearly half of the values when the capsule is embedded in an epoxy matrix, which may be due to the diffusion of the epoxy into the microcapsule during the embedding and curing stages of the latter preparation. Although the surface of the capsule appears smooth, higher magnification shows a granular type surface composed of grains having sizes in the range 100-160 nm, which indeed allows for some porosity. The difference in shell thickness between the two types of capsules as observed in the SEM images can further imply that these microcapsules are having different mechanical strengths.

References:

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Fig. 1: SEM image of a melamine-formaldehyde microcapsule MC-VII.

Fig. 2: SEM image of a single MC-VII microcapsule (inset) and a magnified image prepared for thickness measurement using FIB.

Fig. 3: SEM of a cross-section of MC-VII microcapsules in an epoxy matrix.

Fig. 4: Magnified SEM image of the surface of a MC-VII microcapsule showing the granularity.