FIB-SEM/microtoming prepared Cross Section of a Stone Wool Fiber enabling (S)TEM investigation

Phd Student Krestine Hofstedt Rasmussen[1,2], Ph.D Denis V. Okhrimenko[2], Ph.D Louise J. Belmonte[2], Ph.D Dorthe B. Ravnsbæk[3], Ph.D Espen D. Bøjesen[1]
[1]Interdisciplinary Nanoscience Center, Aarhus University, Aarhus, Denmark, [2]ROCKWOOL A/S, Hedehusene, Denmark, [3]Department of Chemistry, Aarhus University, Aarhus, Denmark

Stone Wool Fibers (SWFs) are aluminosilicate fibers widely used for building and housing insulation. Rocks are melted in a high-temperature furnace (~1500°C) and spun into elongated and cylindrical amorphous fibers approximately 5 µm in diameter and 2 mm in length [1]. ROCKWOOL is a world leading producer of mineral wool fibers used for building and housing insulation. ROCKWOOL continuously transitions to a greener production. This transition potentially influences the redox chemistry and thus the atomic scale network, which consequently could influence the fire resilient properties of SWFs. Our goal is to create a thin (approx. 40-140 nm) cross section of a single stone wool fiber to be able to obtain sub-nanometer scale structural and compositional information using (scanning) transmission electron microscopy ((S)TEM) based techniques. Only by exploring and understanding the structure of SWFs on all length scales (micro, nano, and atomic), will we fully be able to understand the mechanisms behind the fire protective properties of SWFs.

Current strategies used by ROCKWOOL to prepare cross sectional samples include mechanical polishing of SWFs embedded in an epoxy matrix. However, this technique is not suitable for STEM-based investigations due to the large sample thickness and the high likelihood of surface contamination caused by polishing. Thus, a new approach for thin sectioning optimal for (S)TEM analysis is needed. A major obstacle to obtaining the sought-after cross-section cross section is the non-conductive nature of the SWFs. Here, we present the current state of our exploration using focused ion beam scanning electron microscopy (FIB-SEM) and testing of using ultra-microtoming of fibers cast into an epoxy matrix for the preparation of cross sections of SWFs for further (S)TEM investigations. While bombarding the fiber with ions in the milling process, high precision is obtained by continuously monitoring and guiding the process by scanning the electron beam across the area of interest [2,3]. The non-conductive nature of the fibers will lead to build up of charge on the fiber surface and may cause deflection of the beam that otherwise enables the high-resolution imaging, which ensures a precise milling process. The cross-sectional lamella is prepared using a FEI VERSA 3D FIB-SEM system equipped with a Gallium ion source or a Leica EM UC7 ultra-microtome with a diamond knife. In our approach, the fibers for FIB-SEM are carefully placed in/on carbon paste that is added to a silicon wafer. Hereafter, the fibers are coated with 600 nm gold (Au) to shield the fiber throughout the milling process. Preliminary results have shown that optimal conditions for the milling include a low I-beam current of a maximum of 5 nA to avoid damage to the fiber upon sputtering.

Furthermore, the tilt angle used to properly clean and mill the thin section of a fiber is further under improvement. Currently, tilt angles of 50.5° and 53.5° are used as the FIB column is located at 52°. In general, the aim is to estimate the right trade-off between acquiring optimal imaging and milling without too much charge build up on the specimen [4]. A test of using ultra-microtoming for thin sectioning is investigated simultaneously to acquire the best possible cross section. This technique also ensures that no damage to the structure of the fiber is created as with FIB-SEM might lead to. A thin section of approx. ~40 nm was acquired with microtoming and initially investigated in STEM mode using a TESCAN Clara scanning electron microscope. Both techniques show promising initial results on our way to creating a thin cross section of fiber. However, both techniques also show some obstacles to overcome in the process. For FIB, damage upon sputtering should preferably be avoided and strategies to minimize charging need to be optimized. For microtoming, a major current obstacle is to optimize the mechanical properties of the epoxy to match those of the fibres to optimize the cutting process. The process of acquiring this cross section for TEM investigations is still in the primary stages, focusing on practicing the lift-out process of the cross section and mapping out the charging effects.

Keywords:

Stone Wool, aluminosilicates, FIB-SEM, TEM

Reference:

[1] J. E. Shelby, Roy. Soc. Chem., 2021, 3rd ed.;
[2] R. M. Langford, Microsc. Res. Tech., 2006, 69(7), 538-549; [3] L. A. Giannuzzi and F. A. Stevie, Micron, 1999, 30(3), 197-204;
[4] D. C. Joy and C. S. Joy, Micron, 1996, 27(3-4), 247-263