SYNCHROTRON BASED X-RAY ABSORPTION SPECTROSCOPY FOR STRUCTURAL ANALYSIS OF BASALT FIBERS

Henning Lichtenberg[1], Boris Mahltig[2], Wantana Klysubun[3], Alexander Prange[1], Dmitry Doronkin[4] and Josef Hormes[5]

[1] Hochschule Niederrhein University of Applied Sciences, Reinarzstr. 49, D-47805 Krefeld, Germany, Henning.Lichtenberg@hs-niederrhein.de
[2] Faculty of Textile and Clothing Technology, University of Applied Sciences Niederrhein, Webschulstr. 31, D-41065 Mönchengladbach, Germany, Boris.Mahltig@hs-niederrhein.de
[3] Synchrotron Light Research Institute, 111 University Ave. Muang District, Nakhon Ratchasima 30000, Thailand, wantana@slri.or.th
[4] Institute of Catalysis Research and Technology, Karlsruhe Institute of Technology, Hermann-von-Helmholtz-Platz 1, D-76344 Eggenstein-Leopoldshafen, Germany, Dmitry.Doronkin@kit.edu
[5] Institute of Physics, Rheinische Friedrich-Wilhelm-University, Nussallee 12, D-53115 Bonn, Germany, hormes@physik.uni-bonn.de

Abstract: X-ray absorption spectroscopy (XAS) at synchrotron light sources is a powerful non-destructive characterization technique providing detailed information about the atomic environment of selected chemical elements in both crystalline and X-ray amorphous materials without elaborate sample preparation. The penetration depth of X-rays enables bulk analyses and *in situ* characterization of functional materials under 'working conditions' (e.g. high temperature).
Basalt fibers are amorphous materials produced from molten volcanic rocks. Compared to common natural or synthetic fibers, they exhibit higher thermal stability, probably due to their high iron content [1]. At ~ 500 °C a drastic decrease in mechanical strength was observed, along with decomposition of sizing agents on the surface. At higher temperature, changes in bulk structure (crystallization) and further damage are expected [2].
For this project two types of basalt fibers (basalt roving and uncoated chopped fibers) were heated in a muffle furnace (600°C - 900°C). XAS spectra at the K absorption edges of silicon, calcium, iron and titanium were recorded at the XAS beamlines BL8 (Synchrotron Light Research Institute, Thailand [3]), ASTRA (SOLARIS, Poland [4]) and P65 (PETRA III, DESY) using X-ray monochromators along with ionization chambers and semiconductor detectors for measuring incoming, transmitted and fluorescence intensities. The results show a significant influence of temperature on the iron K-edge absorption spectra above 600 °C: With increasing temperature the absorption edge shifts to higher energies, indicating progressing oxidation of the iron atoms in the fibers, while the intensity of the absorption maximum increases. Titanium K-edge spectra are significantly influenced by heating as well. These experiments demonstrate the potential of X-ray absorption spectroscopy as an analytical tool to investigate structural changes in basalt fibers upon heating, in the future also *in situ*, and to correlate them with the observed changes in mechanical properties.

Keywords: *basalt fiber, X-ray spectroscopy, synchrotron*

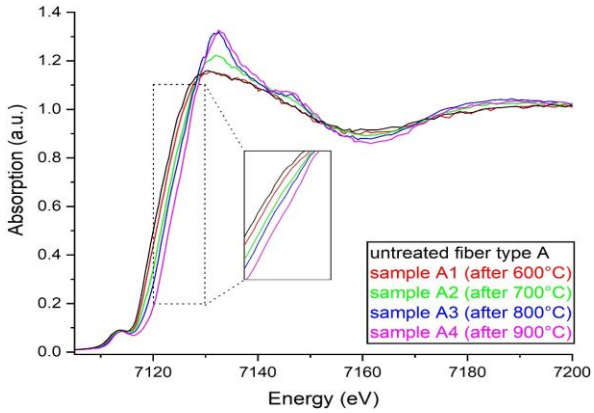

Figure 1 Iron K-edge X-ray absorption spectra of basalt fibers heated at different temperature

ACKNOWLEDGEMENT*: This project was partly supported within the grant „Innovative Hochschule – Leuchtturm NR - Aus der Höhe in die Breite" (03-IHS-084) by the Federal Ministry of Education and Research (Germany) and the EU Horizon2020 program (952148-Sylinda), and by the Polish Ministry and Higher Education project "Support for research and development with the use of research infrastructure of the National Synchrotron Radiation Centre SOLARIS" under contract nr 1/SOL/2021/2.*

REFERENCES

[1] Gutnikov, S.I.: Effect of the reduction treatment on the basalt continuous fiber crystallization properties. J. Non-Cryst. Solids 368, 45, 2013
[2] Overkamp, T., Mahltig, B., Kyosev, Y.: Strength of basalt fibers influenced by thermal and chemical treatments. J. Industrial Textiles 47 (5), 815, 2018
[3] Klysubun, W.: Performance and status of beamline BL8 at SLRI for X-ray absorption spectroscopy. J. Synchrotron Rad. 19, 930, 2012
[4] Hormes, J.: A new SOLARIS beamline optimized for X-ray spectroscopy in the tender energy range. Nucl. Instrum. Methods Phys. Res. B 489, 76, 2021