Preliminary experiments for the production of basalt ceramic fibers

D. Pico, T. Go, G. Seide, T. Gries

Institut fuer Textiltechnik der RWTH Aachen University

Otto Blumenthal Strasse 1, 52072 Aachen, Germany

Aim of this work is to produce nano-crystallized fibers from commercial basalt fibers in order to improve their thermal stability. Inorganic materials present two different atomic structures, amorphous or crystalline. Usually crystalline is defined as a solid state of aggregation where the atoms or the molecules are arranged in a regular, periodic manner. Amorphous or non-crystalline solid is, on the contrary, a solid that lacks the long-range order characteristic of crystal arrangements. Due to their production process, glass and basalt fibers present an amorphous structure and they have a glass transition point T_g, if they are heated up to liquid state. In Figure 1 is shown the process of crystallization in function to the specific volume. The amorphous solid present a glass transition point (T_g) lower that the melting point (T_m). Under particular condition, the material would start to crystallize, shifting the T_g at higher temperatures until T_m by a complete crystallization. Therefore, the structure modification from amorphous to crystalline would higher the operating temperature of the fibers ($T_m > T_g$).

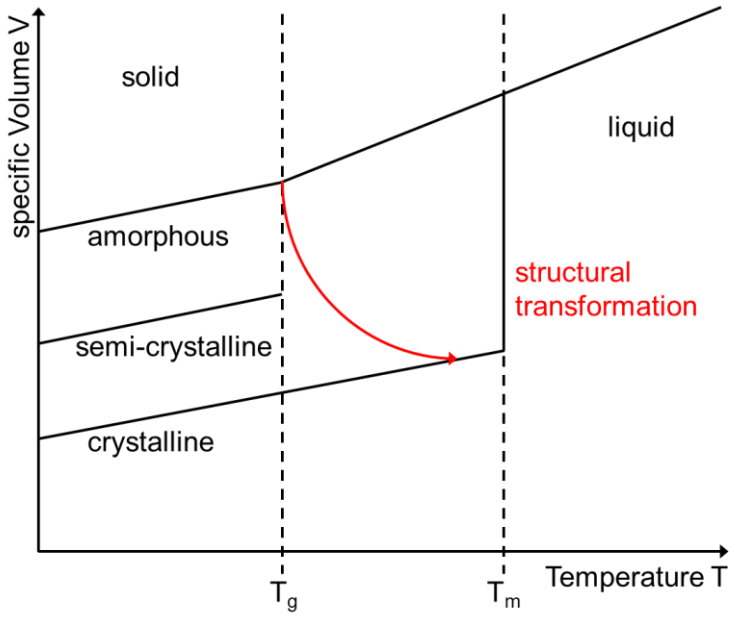

Figure 1: Structural transformation from amorphous to crystalline solid structure and consequent shift of the T_g to T_m.

Due to their chemical composition, only basalt fibers have been taken in consideration. Basalt fibers present a quite high predisposition to crystallize. Basalt

fibers, as reported in table 1 contain more than 5 % Fe_xO_y and a high quantity of nucleating agents like TiO_2.

%	Basalt
SiO_2	48,8~51,0
Al_2O_3	14,0~15,6
$FeO+Fe_2O_3$	7,3~13,3
CaO	10
MgO	6,2~16
$Na_2O + K_2O$	1,9~2,2
TiO_2	0,9~1,6
MnO	0,1~0,16

Table 1: Basalt fibers - chemical composition range.

The conversion from amorphous to crystalline takes place during a heat treatment with a suitable temperature profile. During this research project inorganic amorphous basalt fibers are converted into basalt ceramic fibers by modulating the temperature profile between a temperature range with high nucleation rate and a temperature range with high crystal growth. Between nucleation temperature and crystal growth temperature, it should be possible to influence the structures without changing the fiber integrity due to partial melting of the fiber surface. The production of basalt fibers with high thermal stability will allow reducing the costs of fibrous material for thermal application up to 1000 °C.

The preliminary experiments allowed reaching around 66 % crystalline fraction in basalt fibers. The solid state was analyzed by using wide-angle X-ray diffraction (WAXD) technique. In figure 2 are shown the structures of basalt fiber before and after treatment. The crystallinity rate was measured over 66 %.

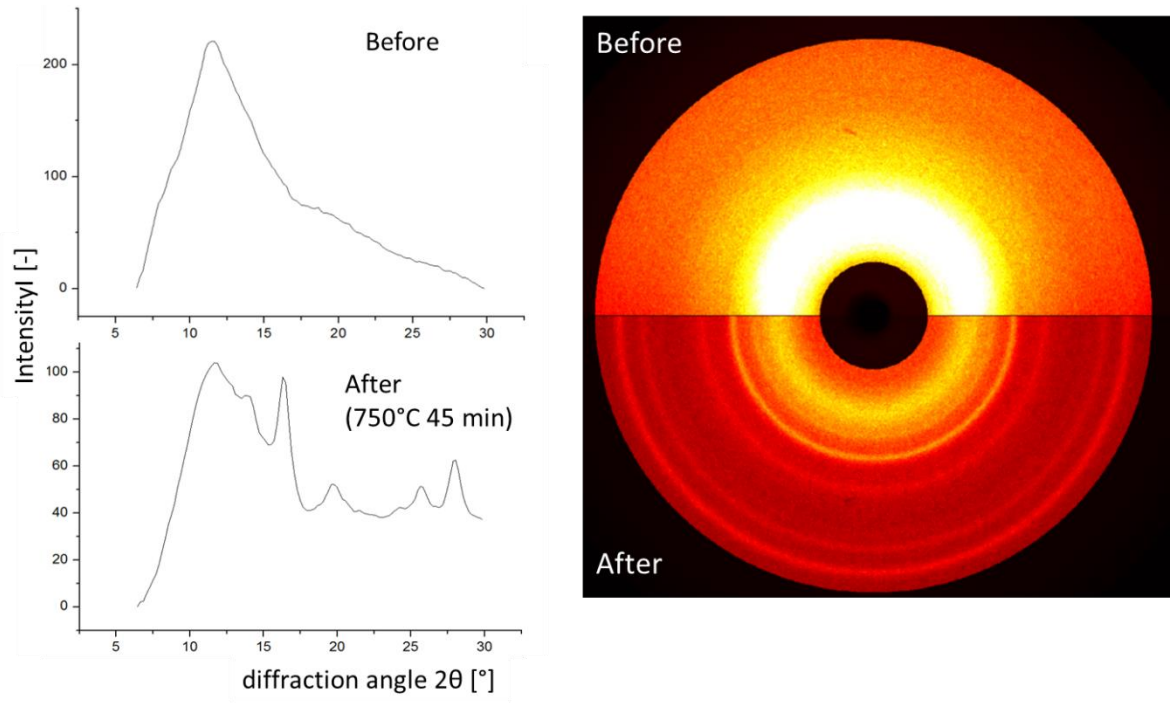

Figure 2: wide-angle X-ray scattering (WAXS) of basalt fibers before and after the thermal treatment.

Economic aspects of this project are related to the strong reduction of the production cost compared to existing ceramic fibers by manufacturing ceramic fibers from basalt precursor fibers, shown in Figure 3.

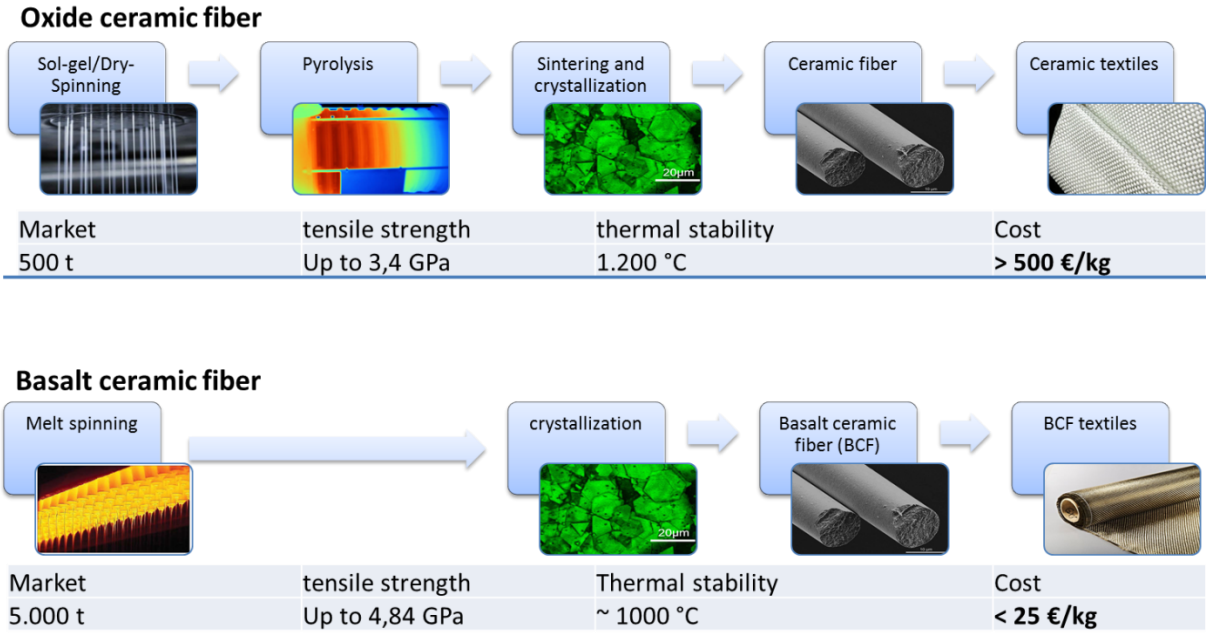

Figure 3: Comparison of the manufacturing processes and the cost of ceramic fibers and basalt ceramic fibers

If considered the chemical composition of basalt fibers (table 1) it is evident that several crystal textures are possible. Some example of these structures, but not exclusively are:

- Diopside ($MgCaSi_2O_6$) with melting point 1391 °C
- Wollastonite ($CaSiO_3$) with melting point 1540 °C
- Kirschsteinite ($CaFeSiO_4$) with melting point 1503 °C
- Anorthite ($CaAl_2Si_2O_8$) with melting point 1400 °C
- Forsterite (Mg_2SiO_4) with melting point 1890 °C

Each of them has a thermal stability by far over 1200 °C.

Considering the mechanical properties of these new fibers, it is evident that the fibers under thermal treatment will firstly lower their mechanical properties. In figure 4 is shown the expected behavior of the mechanical properties along the process.

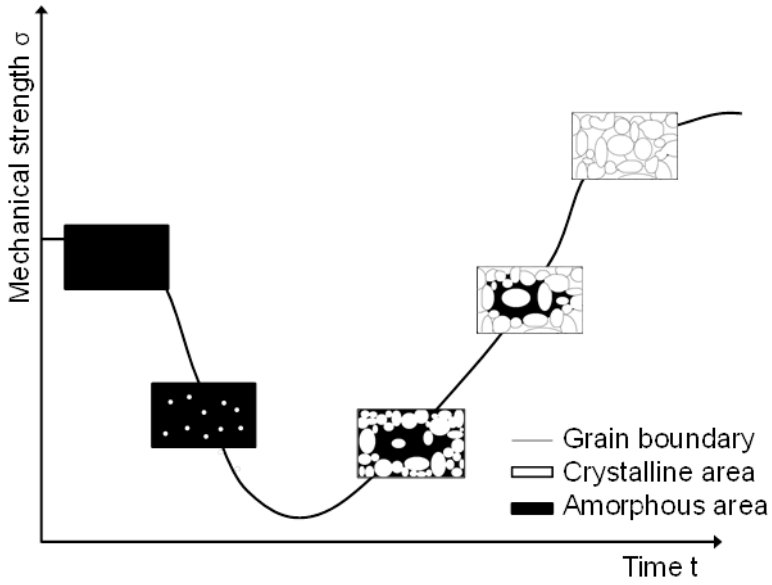

Figure 4: Behavior of the basalt fibers under thermal treatment depending on the crystalline rate.

Several two steps fiberization processes, the precursor fiber present quite different mechanical, physical and chemical properties to the final product. Carbon fibers and ceramic fibers are obtained from a precursor whose characteristics are different and worst if compared to their output. Also in this process, by completing the conversion to a crystalline lattice, the fibers will reach better mechanical and thermal properties that the input material.

Principle of this development is that crystalline fibers can be obtained by amorphous basalt fibers (glass structure). This principle is confirmed, for example, by the production of monolithic glass ceramics (e.g. cooking hobs from Ceran®). In this research project, inorganic, amorphous fibers (basalt fibers) are subjected to deglass

process. The crystal lattice is converted to a crystalline structure, and the thermal stability of the fiber is thereby raised from 800 °C up to 1000 °C.