Identification and Quantification of Crystalline Phases in Thermally Treated Stone Wool

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Stone wool is a fibrous, glassy insulation material which is produced by melting and subsequent quenching and spinning of primary and secondary silicate resources like basaltic rocks or recycling slags. In this work the crystallization behaviour of Rockwool® HT fibres and its connection with the high temperature stability are investigated.
Systematic two-step heat treatments of stone wool fibres are conducted. The pre-heat treatments are performed at 0.8 • T_g, 0.9 • T_g, 1.0 • T_g, 1.1 • T_g and 1.2 • T_g where T_g = 957 K. The heat treatments are conducted both in air and in a reducing atmosphere (90 % N_2 + 10 % H_2) at 1.2 • T_g, 1.25 • T_g and 1.3 • T_g. Both treatments are executed for different durations (10, 30 and 60 min). Thermogravimetry (TG) of pre-heated and untreated fibres shows that between 500 and 700 °C a mass increase takes place which is associated with the oxidation of iron and the formation of surface crystals which is in agreement with the results of Kirkegaard et al. (2005). It is observed by scanning electron microscopy (SEM) analyses of pre-heated fibres that the surface crystallization begins below T_g and that the size of the crystals accounts for between 20 and 50 nm. By differential scanning calorimetry (DSC) of untreated and pre-heated fibres it is found that between 850 and 1050 °C bulk crystallization occurs.
In contrast to previous work (Kirkegaard et al., 2005) by means of X-ray diffraction (XRD) albite ($Na[AlSi_3O_8]$) and gehlenite ($Ca_2Al[AlSiO_7]$) can be identified in most samples after the second heat treatment besides augite ($(Ca,Mg,Fe,Ti,Al)[(Si,Al)_2O_6]$). The presence or absence of a certain crystalline phase does not correlate with the shape stability at high temperatures. Quantification by the Rietveld program AutoQuan using ZnO as an external standard shows that both the pre-heat and the heat treatment influence the mineralogical composition. The amorphous percentage decreases with increasing duration of both thermal treatments and with decreasing FeO content of the fibres. Short thermal treatments and an oxidizing atmosphere lead to the preferred formation of augite, long thermal treatments and a high temperature during the second heat treatment increases the amount of albite whereas gehlenite forms especially in a reducing atmosphere. The role of the pre-heat treatment as the process of nucleation and the function of the heat treatment as the process of crystal growth are discussed.
References
Kirkegaard L, Korsgaard M, Yue Y, Mørup S (2005) Redox behaviour of iron bearing glass fibres during heat treatment under atmospheric conditions. Glass Sci. Technol. 78: 1-6

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