



Aalborg Universitet

AALBORG UNIVERSITY
DENMARK

The boron speciation quantification in alkali borosilicate glasses by electron energy loss spectroscopy

Yang, G.; Cheng, S.; Zhao, Y.; Peng, M.Y.; Skibsted, J.; Yue, Yuanzheng

Published in:

The 24th International Congress on Glass - Abstracts

Publication date:

2016

Document Version

Publisher's PDF, also known as Version of record

[Link to publication from Aalborg University](#)

Citation for published version (APA):

Yang, G., Cheng, S., Zhao, Y., Peng, M. Y., Skibsted, J., & Yue, Y. (2016). The boron speciation quantification in alkali borosilicate glasses by electron energy loss spectroscopy. In The 24th International Congress on Glass - Abstracts (pp. 183). International Commission on Glass (ICG).

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- ? Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- ? You may not further distribute the material or use it for any profit-making activity or commercial gain
- ? You may freely distribute the URL identifying the publication in the public portal ?

Take down policy

If you believe that this document breaches copyright please contact us at vbn@aub.aau.dk providing details, and we will remove access to the work immediately and investigate your claim.

The boron speciation quantification in alkali borosilicate glasses by electron energy loss spectroscopy

G. Yang¹, S. Cheng¹, Y. Zhao², M. Peng², J. Skibsted³, Y. Yue⁴

¹ Electronic Materials Research Laboratory, Key Laboratory of the Ministry of Education & International Center for Dielectric Research, Xi'an Jiaotong University, Xi'an, 710049, China

² State Key Laboratory of Luminescent Materials and Devices, South China University of Technology, Guangzhou, 510640, China The author affiliation and full address should be located here in 8 point, centred and in italics

³ Department of Chemistry and Interdisciplinary Nanoscience Center (iNANO), Langelandsgade 140, DK-8000 Aarhus C, Denmark

⁴ Section of Chemistry, Aalborg University, DK-9000 Aalborg, Denmark

g.yang@mail.xjtu.edu.cn

Transmission electron microscopy and related analytical techniques have been widely used to study the microstructure of different materials. However, few research works have been performed in the field of glasses, possibly due to the electron-beam irradiation damage⁽¹⁾. In this study, based on our previous research, we have developed a method based on electron energy loss spectroscopy (EELS) data acquisition and analysis, which enables determination of the boron speciation in a series of ternary alkali borosilicate glasses with constant molar ratios. A script for the fast acquisition of EELS has been designed and a new fitting criterion was developed, from which the fraction of BO₄ tetrahedra can be obtained by fitting the experimental data with linear combinations of reference spectra. Figure 1 illustrates the boron K-edge spectra (strictly speaking energy loss near edge structure, “ELNES”) of three glasses (LBS, NBS and KBS are lithium-, sodium- and potassium borosilicate glass) with different fitting methods⁽²⁾. The peak A was solely from trigonal BO₃ and peak B was mainly attributed by tetrahedral BO₄, while peak C contains signals from both configurations. The measured BO₄ fractions (N₄) obtained by the new fitting method are consistent with those from ¹¹B MAS NMR data, suggesting that EELS be an alternative and convenient way to determine the N₄ fraction in glasses.

In addition, in order to investigate the effect of cerium oxide on the glass microstructure, 5 mol% CeO₂ was added to the KBS glass (Ce-KBS). The BO₄ fraction in the Ce-KBS glass was found to be lower than that in the KBS glass and the Ce oxidation state was quantified to be close to 4⁺. The boron time-resolved EELS spectra were recorded to quantify the electron beam irradiation effect to the glass network. The results clearly demonstrate that the transformation from BO₄ to BO₃ units can be efficiently suppressed by adding CeO₂ to the borosilicate glasses. Meanwhile, Ce oxidation state was found to decrease during the electron beam irradiation, which indicates that Ce ions act as a buffer to retard the damage of the glass network during TEM observation. More details about the EELS analysis of the borosilicate glasses will be discussed.

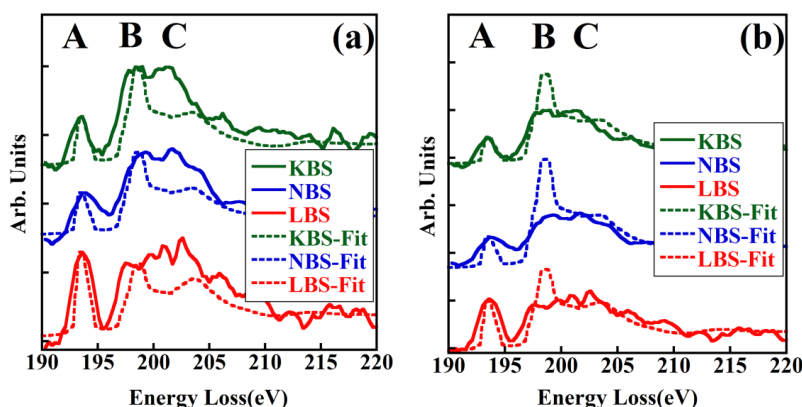


Figure 1: Experimental boron K-edge EELS spectra of the lithium-, sodium- and potassium borosilicate glasses (solid lines) along with the fitted spectra (dashed lines) by (a) the previously reported method and (b) the fitting method developed in this work

Acknowledgements

This work was financially supported by the National Natural Science Foundation of China (No. 51202180) and the authors thank the Danish Strategic Research Council for financial support to the LowE-CEM project.

1) H. Sauer, R. Brydson, P. N. Rowley, W. Engel, and J. M. Thomas, Determination of coordinations and coordination-specific site occupancies by electron energy-loss spectroscopy: an investigation of boron-oxygen compounds, *Ultramicroscopy*, **49** (1993)198

2) S. D. Cheng, G. Yang, Y. Q. Zhao, M. Y. Peng, J. Skibsted and Y. Z. Yue: “Quantification of the boron speciation in alkali borosilicate glasses by electron energy loss spectroscopy”, *Scientific Reports* 5 (2015) 17526.