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In situ observation of the nanocrystallisation process in vanadate glasses in the XAS synchrotron experiment

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Introduction

Nowadays, more and more energy is generated from renewable power sources. However, because these sources are highly sensitive to weather changes, there is a need for energy storage systems stabilisating the grid.

Currently one of the biggest limitations in improving battery properties is poor cathodic conductivity. Various strategies have been proposed to overcome this problem. An approach, developed by our group, involves the synthesis of glassy analogs of cathode materials for lithium- and sodium-ion batteries, followed by heat treatment to induce nanocrystallisation. This process results in the formation of glass-ceramic composites, where nanosized crystallites are embedded within a glassy matrix, leading to significantly enhanced electrical conductivity.

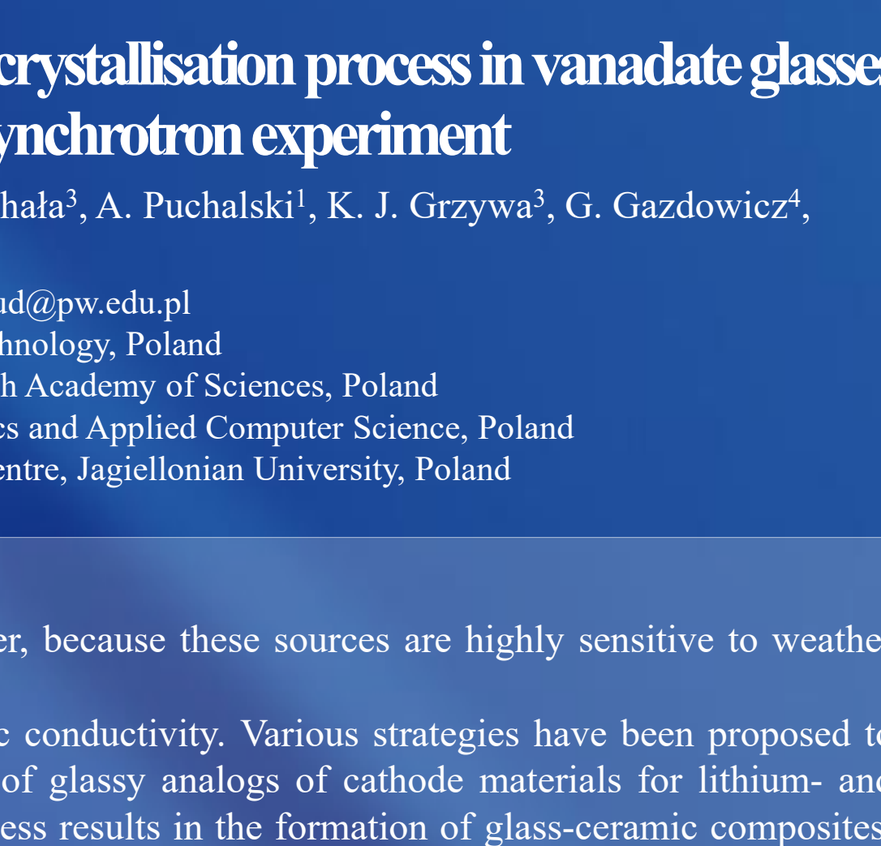
Aim of the study

The goal of this work is to understand what changes in our material during crystallisation using DC current, DTA, and HT-XAS measurements.

Synthesis

In this work, we present the results of $95V_2O_5 \cdot 5P_2O_5$ glasses synthesised at 900°C for 15 min.

Homogenise proper amount of substrates → **Melt it for a specific time and temperature** → **Cool it down on copper plates**

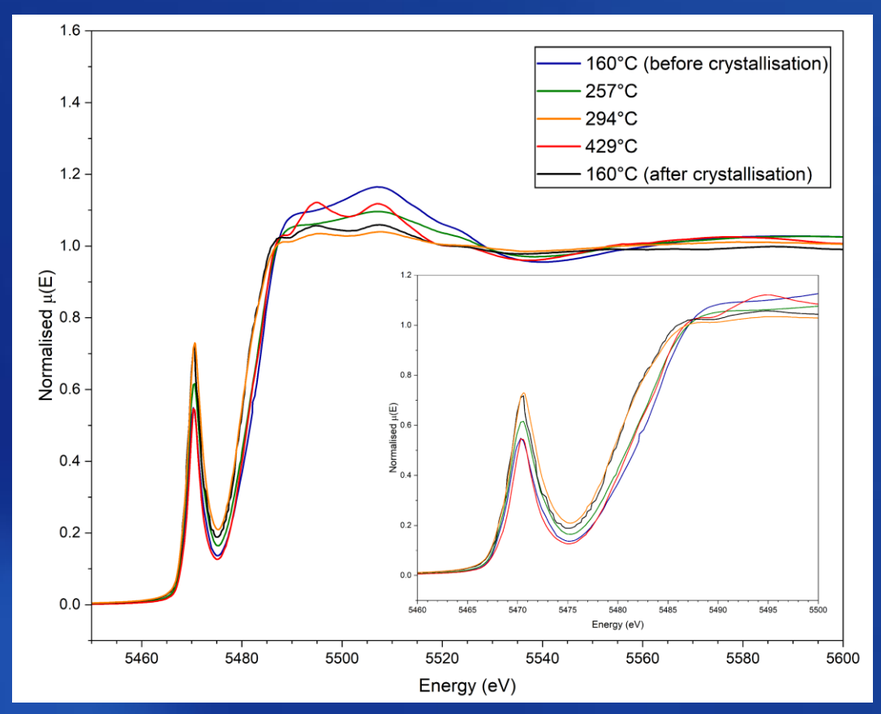


Results

Fig. 1 (top) presents the Arrhenius plots of the electrical conductivity for the glass samples heated up to 280°C, complemented by the DTA curve (inset). At crystallisation temperature (230 °C), dramatic increase of conductivity is observed.

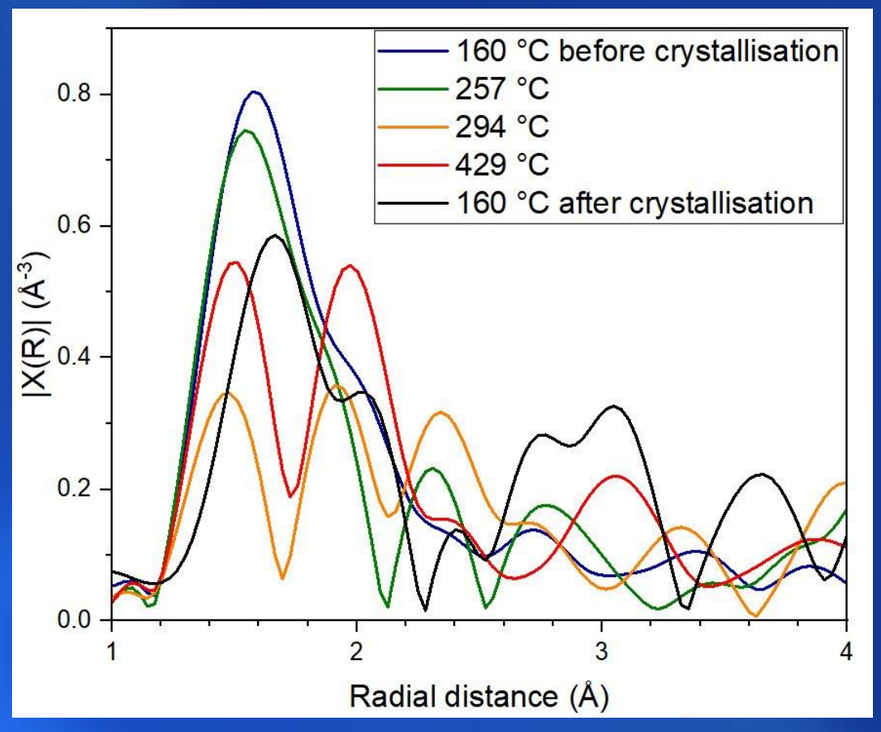
In Fig 2 (middle) the temperature-dependent evolution of the vanadium K-edge is illustrated. Interestingly, absorption energy shifts toward lower values by approximately 2–3 eV (during the crystallisation process). After around 300 °C, the trend is reversed. In contrast, the pre-peak position shifts only by around 0.3 eV. These observations suggest a subtle modification in the V–O bonding and the local electronic structure, such as changes in covalency or network connectivity, rather than a change in the vanadium oxidation state.

In Fig 3 (bottom) there are the RDF functions calculated using Athena software. It shows that the RDF function varies significantly with temperature. While the glass structure remains stable before the crystallisation, it undergoes a reorganisation above the crystallisation temperature.



Conclusions

The 95V2O5·5P2O5 glass was investigated using multiple experimental techniques. The material demonstrates noteworthy characteristics in both XAS and DC measurements. While the most significant transitions in the DC and DSC data are concentrated near the crystallization temperature, the absorption energy undergoes a continuous evolution throughout the entire thermal range, reaching a minimum at approximately 300 °C. Furthermore RDF changes mostly above crystallisation temperature.



Acknowledgements

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