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Report:

Soda-lime glasses containing Ag particles were investigated by X-ray absorption spectroscopy. Ag was introduced into the surface of the glass matrix or was deposited on the glass surface as doped silica film, respectively. The purpose of the present work was to characterize the atomic structure of crystalline particles as well as of the glass-particle interface in order to describe the relations to the macroscopic properties. The behaviour of crystalline precipitation should strongly be influenced by the procedure of particle formation and with that by the thermal history. Therefore, different procedures of particle formation were examined.

Soda-lime glasses containing SiO_2 and Na_2O as main components were used either as base glass for ion exchange and Ag implantation experiments or as substrat for sol-gel layers. Small amounts of Sb or Fe act as reducing agents to initiate the formation of crystalline particles. Ag K-edge spectra (25 5 14 eV) were recorded at the ESRF on the beamline BM08/GILDA between liquid nitrogen temperature and room temperature using a Si (3 11) crystal monochromator. The incident X-ray intensity as well as the transmitted photons were measured with an ionization chamber, whereas the fluorescence intensity was collected by a plastic scintillator. A 500 μm Al foil was used to absorb the low energy radiation. Harmonic rejection was achieved by detuning the monochromator crystals at the mid point of the reflectivity curve. Silver foils and Ag_2O were used as crystalline standards for data analysis.

Soda-lime glasses containing small amounts of silver within a thin surface layer, which were produced by an short ion exchange of Sb doped glasses, could be investigated

by the transmission mode. The silver precipitations were formed during an annealing at 635°C. The Fourier transforms of the experimental data indicate three coordination shells (see Fig. 1). The structural parameters derived from EXAFS data by curve fitting with theoretical amplitude and phase functions using FEFF 5 show two Ag-O coordination spheres, the first one at 0.213 nm with a coordination number of 1.9 and the second at 0.279 nm. This represents a typical behaviour of Ag/Na ion-exchanged soda-lime glasses. The third shell can be identified as Ag-Ag correlations caused by the existence of nanoscale silver particles. The Ag-Ag distance of 0.289 nm determined at 81 K shows a similar value as the bulk material. Thus, the usually expected lattice contractions for small isolated particles did not occur for silver in Sb doped glasses.

Ag particles in a silica layer of 1 µm thickness were detected by the fluorescence mode. The Fourier transforms demonstrate that nearly all of the silver ions were reduced to neutral atoms followed by the formation of particles (Fig. 2). Here, the average Ag-Ag separation of 0.291 nm indicates a small dilatation of the lattice. The coordination number of 9 reflects the average particle size of 6 nm which was determined by electron microscopy. The remaining Ag-O correlation at approximately 0.215 nm should reflect the interaction of silver atoms at the particle surface with the oxygens of the surrounding glass matrix. The ion implanted Ag particles could also be observed by the fluorescence detection. However, because of the small thickness of the silver containing layer of 200 nm the signal-to-noise ratio was insufficient to evaluate the structural data. Therefore, the number of scans has to be increased for each EXAFS spectrum.

The discussion of the preparation methods and the interpretation of experimental data with respect to Debye-Waller factors and Ag-Ag bond lengths as a function of temperature are in progress.

