

† Electronic Supplementary Information (ESI) available: [SEM and EXAFS characterisation]

Scanning Electron Microscopy

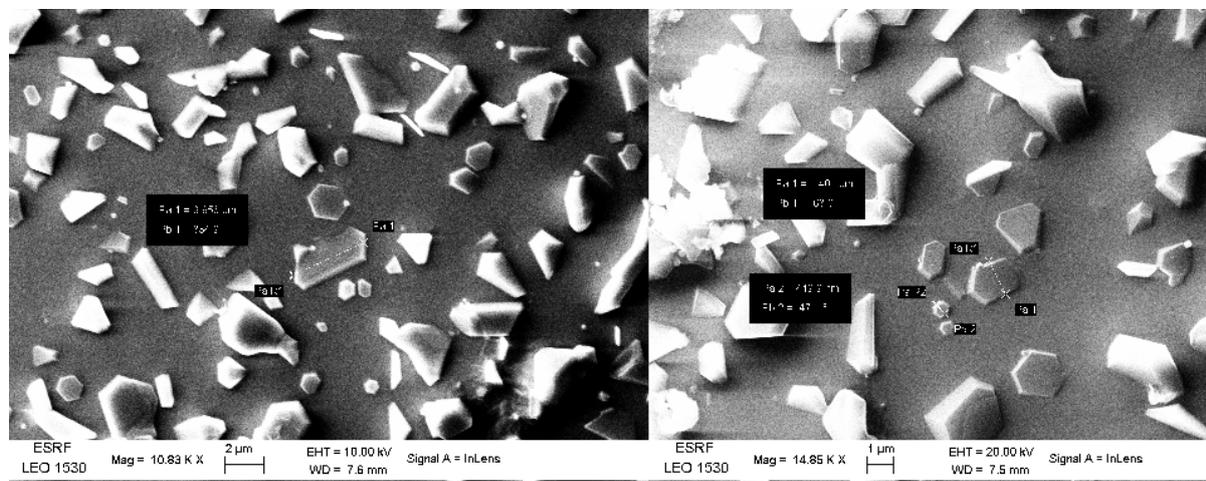


Figure S1 SEM images of the surface of a sample pre-annealed ex-situ followed by in-situ anneal at 650°C for 2 hours 10 minutes coated with 200Å of tungsten.

EXAFS characterisation

Pb L-III edge (13.035 KeV) Extended X-ray Absorption Fine Structure (EXAFS) spectra of our glass samples together with Pb foil, PbS and PbO pellets (diluted with Boron Nitride) were collected at the Dutch-Belgian Beamline (DUBBLE) at the European Synchrotron Radiation Facility (ESRF). The energy of the X-ray beam was tuned by a double-crystal monochromator operating in fixed-exit mode using a Si(111) crystal pair. The samples were measured in a closed-cycle He-cryostat (Oxford Instruments) at 80 K to minimize the noise induced by thermal Debye-Waller factor. EXAFS spectra of the glass samples were collected in fluorescence mode using a 9-element Ge detector, whereas reference spectra of the metallic Pb foil, PbO and PbS pellets were collected in transmission mode using Ar/He-filled ionization chambers. Due to the low concentration of Pb present in the glass samples, 25 scans were collected in fluorescence mode per sample in order to improve the signal/noise ratio.

The EXAFS spectra, were energy-calibrated, averaged and further analyzed using the program Viper¹

The isolated EXAFS oscillations were k^3 – weighted and Fourier transformed over the k -range from 3 to 12 Å⁻¹ using a Bessel window function. The data were fitted using Viper code which involved the theoretical phase and amplitude functions calculated by *ab initio* code FEFF 8.2. The global fit parameters that were allowed to vary during the fitting procedure were the coordination number (N), distance R (Å), Debye-Waller factor (σ^2) and the energy shift ($\Delta E_{k=0}$). The threshold energy $E_{k=0}$ was defined at 13035 eV. The results are shown in Figure S2 below

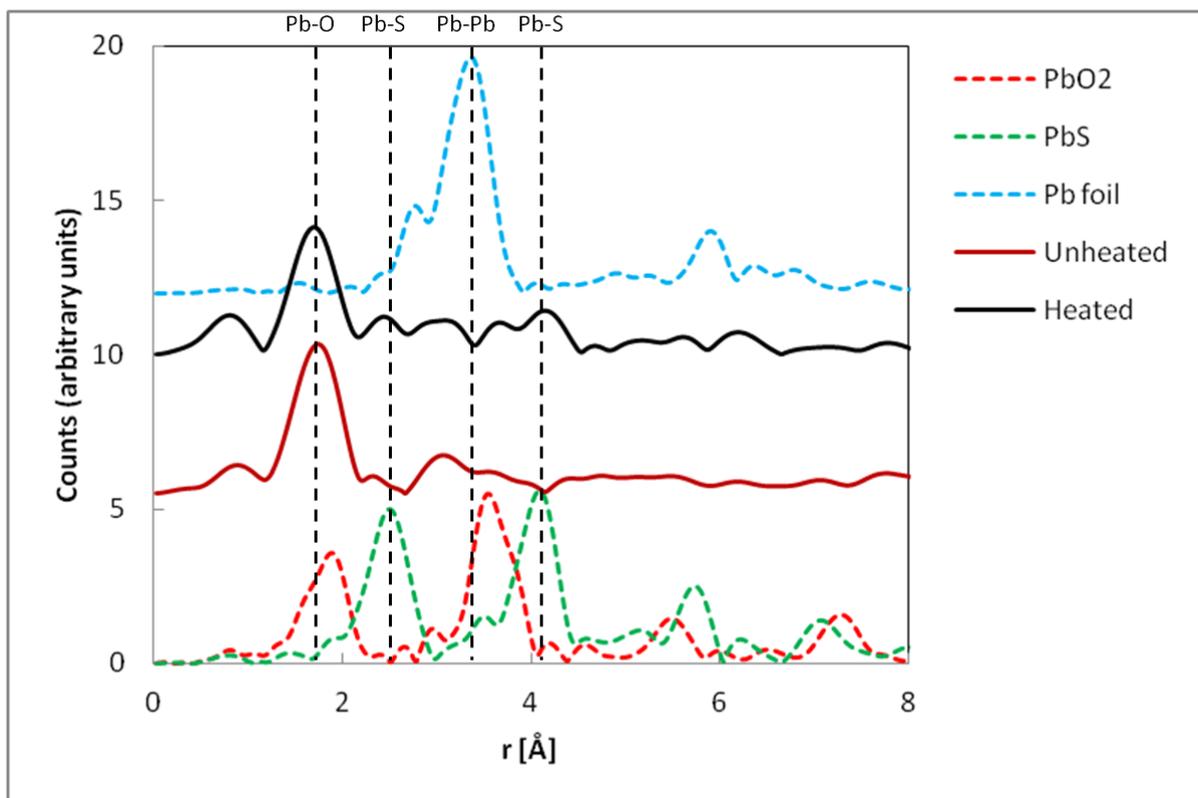


Figure S2 Fourier transform EXAFS plots, uncorrected for phase shifts, for both unheated and annealed glass samples (solid lines) compared to three reference materials (Pb foil, PbO₂, PbS) (dashed lines). The vertical dashed reference lines indicate the Pb-O, Pb-S and Pb-Pb contributions to the spectra. No evidence for Pb-Pb interaction is found in the glass samples. The unheated glass only shows Pb-O contributions whereas the heated sample shows possible Pb-S contributions.

1. K. K.V., J. Phys. D: Appl. Phys. 34, 209-17, 2001.