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In-situ beam driven experiments by Electron time-correlation microscopy of amorphous structures

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Background & Aims

Amorphous materials are widely present and not straight-forward to study. However, similar to X-ray photon correlation spectroscopy (XPCS) the method of electron correlation microscopy (ECM) was introduced [1]. This approach using the time correlation of diffracted or dark-field intensities was applied at elevated temperatures. However, it was shown that this way of data analysis gives also proper materials parameters at room temperature (RT) [2]. Besides that, also pure materials made amorphous by ion implantation were recently studied [3]. To understand the influence of the beam and the measured signals, results from different materials are compared and discussed. In this work, the imaging and analysis conditions are examined to understand the arising quantitative signals, and to be able to compare ECM data obtained at elevated temperatures to that of electron time-correlation microscopy (EtCM) obtained at any temperature, even at RT where the thermal activation is small.

Methods

The analyzed materials are on the one hand metallic glasses as FeNiP or PdNiP, and on the other hand pure elements as Si or Ge. The metallic glasses were processed by rapid quenching techniques, and the pure elements were synthesized by ion implantation. EtCM is used to probe the dynamics and relaxation times from RT up to the glass transition temperature. The samples for the heating experiments were prepared using a focused-ion beam (FIB) and the lamellae were mounted onto a in-situ heating chip. Additionally, all samples were also structurally characterized using diffraction including fluctuation electron microscopy (FEM). Besides a basically disordered structure, also a preferred length-scale for ordering within the amorphous phase can be given. This number should typically not change during the EtCM experiments.

Results

The analysis of time series of dark-field images enables to measure the intensity variation over time, related to a structural relaxation time. The function of the intensity correlation can be given for every image pixel, thus also a relaxation time constant and a stretching exponent can be deduced. The measurement needs a certain amount of time and dose to achieve reliable numerical values. Different materials show different numbers, however, all materials show that the electron beam and its dose-rate or cumulative-dose are relevant for the achieved numerical values. The dose-rate can be even too high, to induce crystallization at RT. Thus, the dose-rate is measured and given for all experiments.

Conclusion

The imaging conditions during EtCM matter for the numerical values and further treatment of the result. Best results are obtained using low-dose settings and long measurement times. However, it must be noted that amorphous structures show relaxation times on basically all time scales, from atomic jumps on the pico-second scale up to bulk relaxation in terms of (millions of) years. Experimentally, the maximum measurable time scale is less than half of the overall experimental time, and the minimal measurable time scale depends on the frame time for two frames. It can be concluded, that EtCM experiments at RT are beam driven relaxation studies, helpful to understand the intrinsic time scale for relaxation in any amorphous matter.

Keywords:

amorphous, metallic glass, in-situ, time-correlations

Reference:

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