2 Experimental Techniques and Instrumentation

Methods and instruments used in this work are described in this chapter. Regarding TEM there are numerous techniques in addition to the basic functionality of imaging. The analytical techniques EDXS and EELS were used in combination with STEM to acquire interdiffusion profiles for evaluation. The two techniques are described in detail in the subsequent sections especially considering quantification of the experimental data. A basic overview of TEM is, for example, given in the books of Williams & Carter [133] and Reimer & Kohl [134]. SEM was used during the preparation process of the samples. The textbook by Reimer [135] explains SEM in detail.

2.1 Electron Microscopical Instrumentation

Microstructural characterization and HRTEM were conducted using two different transmission electron microscopes. The Philips CM200 FEG/ST microscope (now: FEI company, Hillsboro, Oregon, United States of America (USA)) was operated at 200 kV and is equipped with a Field Emission Gun (FEG) and a 4k×4k TemCam-F416 CMOS camera from TVIPS (Tietz Viedeo and Imaging Processing systems, Munich, Germany). The FEI Titan³ 80-300 microscope (FEI company) was operated at 300 kV and is equipped with an aberration corrector, for short $C_s$-corrector (CEOS – Corrected Electron Optical Systems GmbH, Heidelberg, Germany), in the imaging lens system. Single energy-dispersive X-ray spectra were collected using the NORAN Vantage system (Noran Instruments Inc., now: Thermo Fisher Scientific, Waltham, Massachusetts, USA) of the Philips CM200 FEG/ST microscope with a Ge X-ray detector and a probe diameter of about 2 nm. EDXS line scans were acquired utilizing the 30 mm EDAX Si(Li) X-ray detector with an ultra-thin window and an energy resolution of 136 eV (EDAX Inc., Mahwah, New Jersey, USA) in the FEI Titan³ 80-300 microscope. EELS was performed with the post-column Tridiem 865 HR Gatan Imaging Filter (GIF) (Gatan Inc., Pleasanton, California, USA) of the FEI Titan³ 80-300 microscope. EELS spectra can be with an energy resolution of 0.7 eV and a total channel count of 2048. The GIF was
operated at a dispersion of 0.5 eV/channel for elemental quantification. SAED was performed on the Philips CM 200 FEG/ST microscope and the diffraction patterns were recorded on imaging plates from DITABIS (Digital Biomedical Imaging Systems AG, Pforzheim, Germany).

A LEO 1530 microscope (LEO Electron Microscopy Inc., now: Carl Zeiss NTS GmbH, Oberkochen, Germany) equipped with a FEG and a GEMINI© column was used for SEM.

Diffraction patterns were simulated with the software package Java Electron Microscopy Simulations (JEMS) version 3.3826U2009 by Stadelmann [136].

2.2 Transmission Electron Microscopy (TEM)

In TEM the sample is illuminated by a defocused electron beam which is transmitted through the sample and then used for imaging. The first transmission electron microscope of this type was built by Knoll and Ruska in 1932 [137]. It uses condensor lenses to produce the illuminating beam. A lens system below the sample is used to form the image with at least three lenses, i.e., an objective lens, an intermediate lens, and a projector lens. This arrangement can be used in two different modes – the imaging or the diffraction mode. They are selected by the excitation of the intermediate lens. It is either used to magnify the first intermediate image (imaging mode) or the diffraction pattern (diffraction mode) formed by the objective lens. The first intermediate image or the diffraction pattern is then further magnified by the projector lens onto an electron-sensitive scintillator and Charge-Coupled Device (CCD) camera which is used for detection.

The contrast in the final TEM image arises because of the scattering of the incident electrons by the specimen. Amplitude and phase of the electron wave can both be changed during its transit through the specimen and, thus, both can contribute to the image contrast. Hence, the fundamental distinction in TEM is between amplitude contrast and phase contrast. Both contrast types are normally involved in the image formation process. Usually, the imaging conditions are selected in a way that one contrast type dominates to be able to interpret the image accordingly.

Three different techniques used in this work are described in more detail. BFTEM and HRTEM are imaging techniques, whereas the diffraction mode is used for SAED. In-depth descriptions of these techniques are given in textbooks for TEM, e.g. [133, 134].