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Spatially resolved EELS of GaAs/GaAlAs heterostructures

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Abstract. — Using typical beam parameters of the STEM and a parallel detector for the EEL spectra it is possible to detect principally the Al-L and Al-K edge in a $GaAs/Ga_{(1-x)}Al_xAs$ heterostructure with a spatial resolution better than 2 nm, if the relative Al concentration is higher than $x = 0.2$. However, quantification of the Al edges is difficult due to minor As edges in front of the Al edges and a weak signal to noise ratio of the Al edges. Thus, for quantitative microanalysis the Ga-L edge is much more suitable. After background subtraction it is possible to determine the Ga concentration with an accuracy of $x \leq 0.05$ in $(1 - x)$. Therefrom the Al concentration x can be calculated. The spectra taken with different probe currents demonstrate that very small probe currents (5 pA) and probe diameters down to the limit of 0.2 nm can be used to perform chemical analysis of GaAs/GaAlAs heterostructures.

1. Introduction.

Lattice matched GaAs/GaAlAs interfaces play an important role in heterojunction devices. Chemically abrupt interfaces are essential for high device performance such as of heterostructure field-effect transistors [1]. The Al-concentration x determines the bandgap and the refractive index of $Ga_{(1-x)}Al_xAs$ layers important for applications in optoelectronic devices [2]. Typical layer thicknesses are in the nm range and less (down to one monolayer [3]. Therefore spatially resolved characterisation techniques are required for such structures. The spatial resolution of electron energy loss spectroscopy (EELS) of thin specimens is governed by the lateral dimensions of the electron probe and should be under that point of view superior to x-ray microanalysis, where spatial resolution is limited by elastic scattering [4].

On the other hand the electron probe should contain as much current as possible in order to get sufficient signal to noise ratios in microanalytical measurements. Only a scanning transmission electron microscope (STEM) equipped with a cold field-emission source provides beam parameters which allow microanalytical work to be performed with optimum spatial resolution. Typical beam parameters at 100 keV are 1 nm probe diameter and 1 nA probe current. Using a special pole piece in a STEM probe diameters in the range of 0.2 nm but with small probe current (5 pA) can be achieved [5]. Therefore EELS in a field-emission STEM should offer a subnanometer resolution.

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Using serial detectors for EELS long exposure times may cause both beam damage to the specimen and decreased spatial resolution due to system instabilities. By means of a parallel detector EEL-spectra can be recorded in much shorter time with therefore better spatial resolution and with smaller radiation dose to the specimen [6, 7]. The combination of a field-emission STEM equipped with a high resolution pole piece and a parallel detector for the EEL spectra is therefore the most promising technique for high spatial resolution microanalysis presently [8].

The spatially resolved determination of x in the $Ga_{(1-x)}Al_xAs$ layers can be done using either the Ga or the Al edge. Unfortunately the detection and quantification of the Al edges in GaAlAs is very difficult in EELS. It was reported that up to $x = 0.25$ Al in $Ga_{(1-x)}Al_xAs$ does not lead to an obvious Al-K edge in serial EELS [9]. Additionally the minor As-L_1 edge close to the Al-K edge causes trouble in background estimation of the Al-K edge.

It is the intention of this paper to report on the application of spatially resolved EELS with parallel detection to GaAs/GaAlAs heterostructures in a field-emission STEM using small electron probes and therefore small probe currents. By doing so comparisons are made for using the Ga and the Al edges for the determination of the parameter x.

2. Experimental procedure.

All spectra and micrographs were recorded in a field-emission STEM (VG microscopes: HB 501) operated at 100 keV. The micrographs were obtained using high angle annular dark field and (200) dark field imaging techniques [10, 11]. All spectra were recorded by additional use of a high resolution pole piece. The parallel detection system for EELS (see Fig. 1) consists of an 80° magnetic sector field corrected to second order aberrations, three magnetic quadrupole lenses [12], and a thin single-crystal scintillator (YAG) lens-optically coupled to an intensified photodiode array (EG&G PAR 1422).

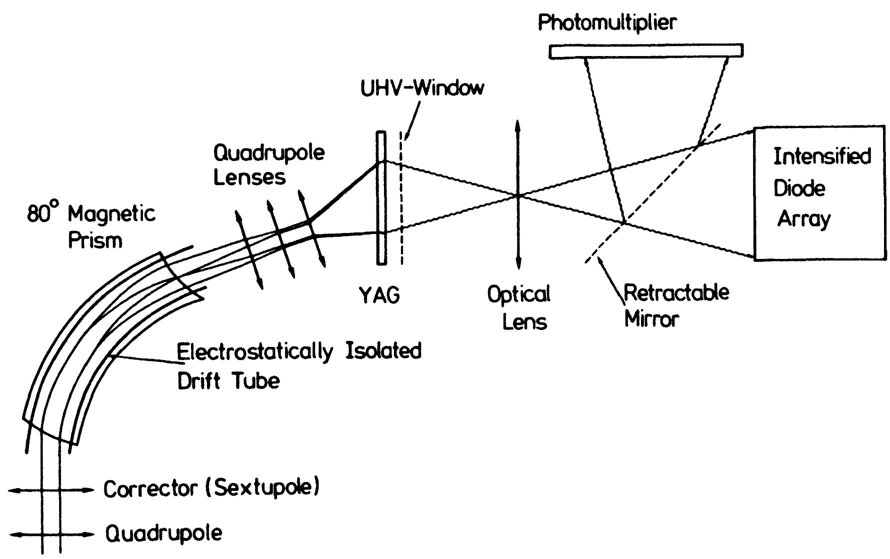

Fig. 1. — The parallel detector system for EELS.

The photodiode array is controlled by an optical multichannel analyser (EG&G PAR 1460). A known voltage applied to the electrostatically isolated drift tube allows the calibration of the energy losses. Bright field imaging is still possible by means of a mirror and a photomultiplier (Fig. 1). The parallel detector is described in detail in [8]. Background subtraction in the spectra was performed according to Trebbia [13]. All spectra were corrected for dark counts and for inhomogenous response of the photodiodes. The photodiodes were operated at a temperature of −30 °C for all measurements.

3. Results.

A high angle annular dark field image (atomic number contrast) of a MOVPE grown GaAs/GaAlAs heterostructure is shown in figure 2. Using a probe current of 1 nA and a probe diameter of 1 nm EEL spectra within the thick GaAs, GaAlAs, and AlAs layers were recorded with the parallel detector within 20 seconds. The spectra are shown in figure 3. The background under the Ga edge is subtracted in all spectra. The Ga-$L_{2,3}$ edge starting at 1115 eV is obvious for the GaAs and the GaAlAs spectrum. All spectra show the As-$L_{2,3}$ edge at 1323 eV. The As-L_1 edge at 1520 eV is very weak. The Al-K edge at 1560 eV is clearly visible in the GaAlAs and AlAs spectrum. The energy resolution obtained is better than 2 eV.

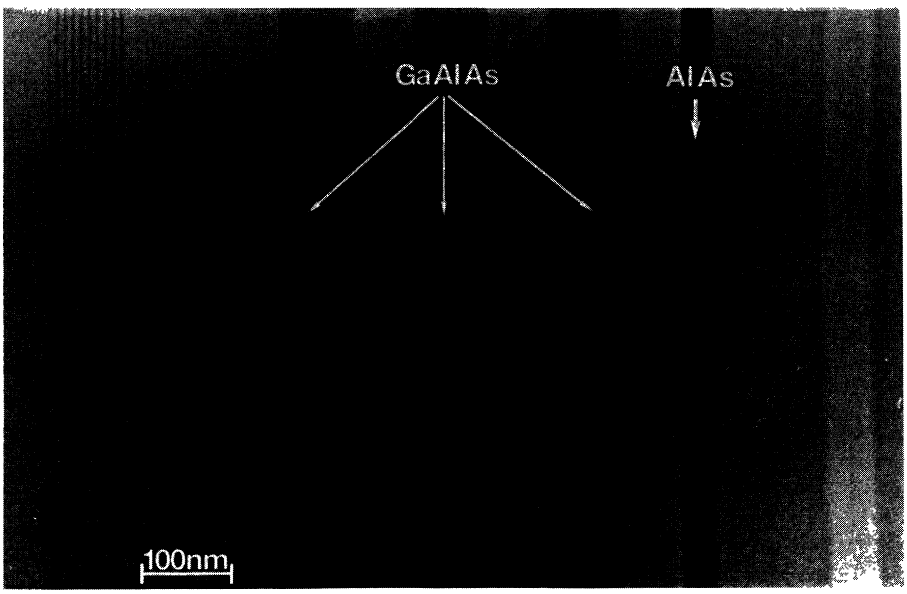

Fig. 2. — High angle annular dark field image (atomic number contrast) of a MOVPE grown GaAs/GaAlAs heterostructure.

As a next step narrower GaAs/GaAlAs layers were investigated. Figure 4 shows a high angle annular dark field image (atomic number contrast) of a GaAs/GaAlAs superlattice buffer layer. The GaAlAs layers are 1.8 nm thick. Figure 5 shows the GaAlAs spectrum recorded in the superlattice compared to a spectrum of GaAs recorded under identical conditions. Both spectra are background subtracted. Whereas a comparison of the Ga-intensities is possible indicating a

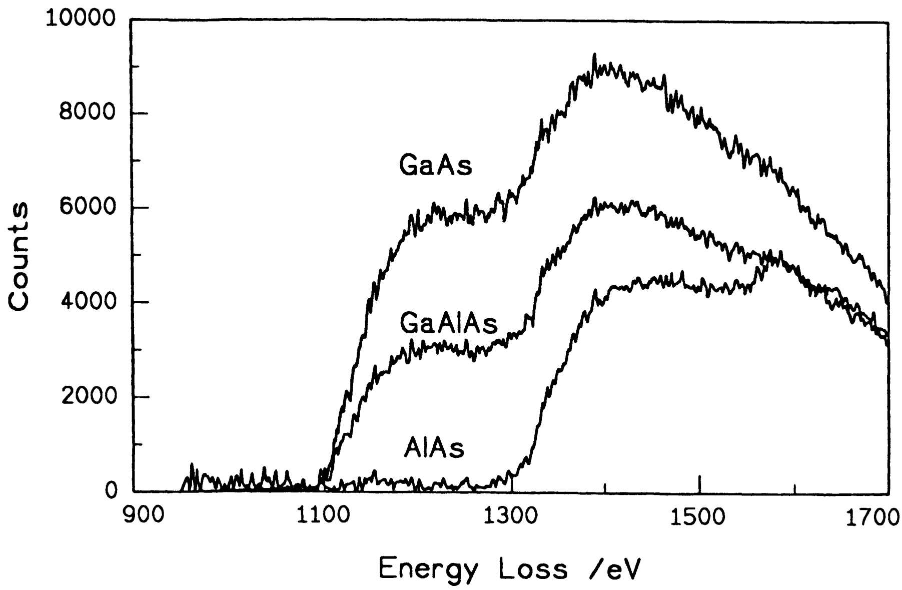

Fig. 3. — EEL spectra of GaAs, $Ga_{0.56}Al_{0.44}As$, and AlAs (probe current 1 nA, probe diameter 1 nm, 20 s recording time).

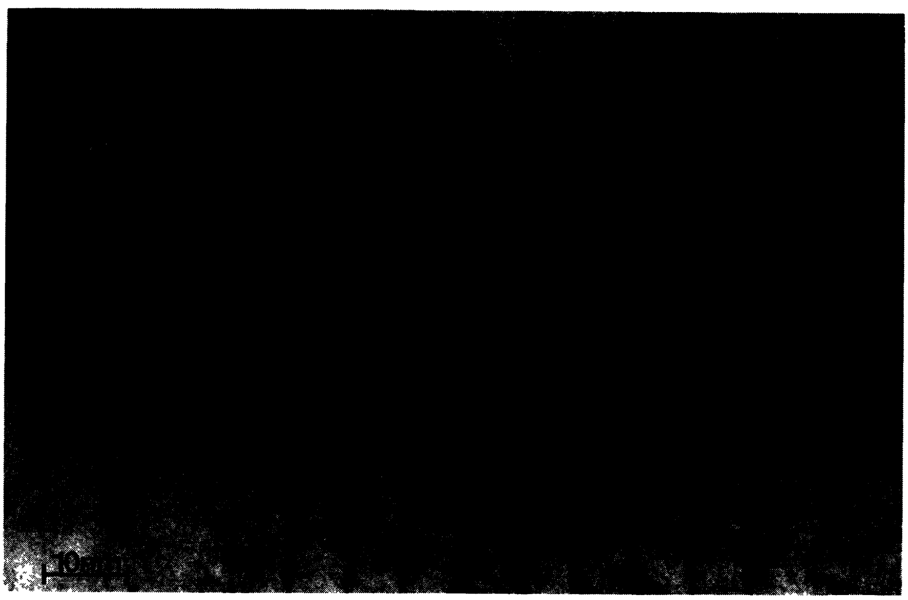

Fig. 4. — High angle annular dark field image (atomic number contrast) of a GaAs/GaAlAs superlattice buffer layer.

composition of $x = 0.5$ for the $Ga_{(1-x)}Al_xAs$ superlattice layers, a detection of the Al-K-edge cannot be done due to low signal to noise conditions.

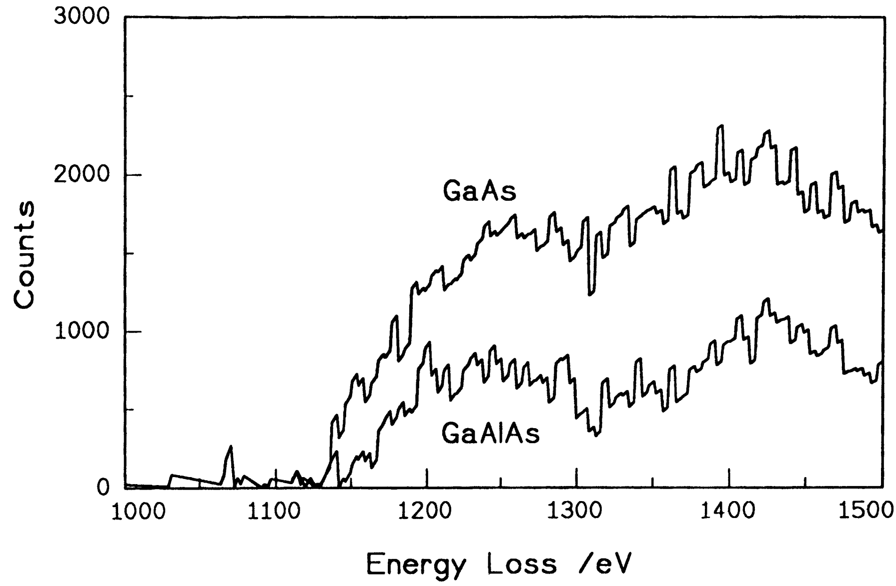

Fig. 5. — EEL spectra of GaAs and of $Ga_{0.5}Al_{0.5}As$ recorded in the superlattice after background subtraction. (probe current 100 pA, probe diameter 0.5 nm, 20 s recordind time).

In a further step the probe current was reduced from 1 nA to 5 pA in four steps with ideally corresponding probe diameters from 1 nm to 0.2 nm (according to [5]). The EELS results for GaAs, GaAlAs, and AlAs are shown in figure 6. The four sets of spectra were recorded under identical conditions except probe current. The recording time for one spectrum was 20 s. For the smallest probe current only the GaAs and AlAs spectra are plotted in order to allow distinguishing the spectra. It is obious that even for the smallest probe current and therefore for the smallest probe diameter the Ga edge and the As edge can give analytical information. The Al-K-edge is only visible for probe currents of 1 nA (and more).

Facing the fact that the Al-K edge is difficult to detect the Al-L edge at 72 eV was investigated. Figure 7 shows a (200) dark field image of the MBE grown GaAs/GaAlAs heterostructure as used for the EELS measurements of the Al-L edge. The arrows indicate the regions where spectra were taken. The results are shown in figure 8. Both spectra were recorded with a probe current of 1 nA. The difference between GaAs and GaAlAs is obvious. Background subtraction was not performed due to problems of background estimation in the low loss region. Using smaller probe currents than 1 nA the Al-L edge was not clearly detectable.

4. Discussion.

Using typical beam parameters of the STEM (1 nA probe current, 1 nm probe diameter) combined with parallel detection of the EEL spectra it is possible to detect the Al-L and Al-K edge in a GaAs/GaAlAs heterostructure with a spatial resolution better than 2 nm, if the Al concentration x is more than 0.2 in the $Ga_{(1-x)}Al_xAs$ layers. For recording times less than 20 s and smaller probe currents than 1 nA (and therefore better spatial resolution) the decreasing signal to noise ratio of the as recorded spectra does not allow the detection of Al edges. To overcome this probem

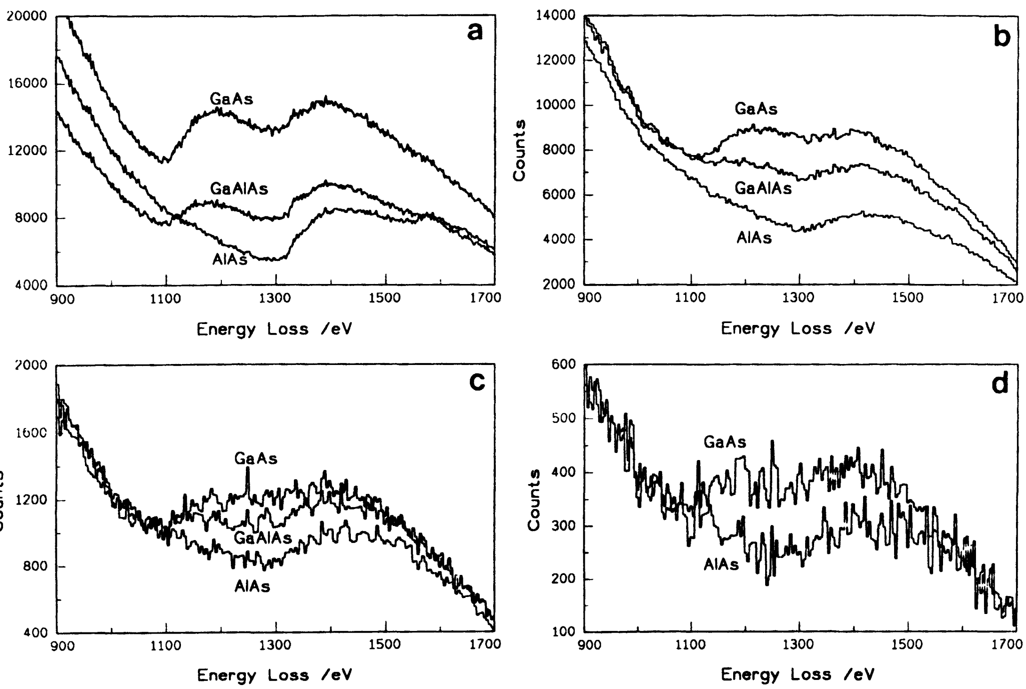

Fig. 6. — EEL spectra of GaAs, GaAlAs, and AlAs recorded under identical conditions as for figure 5 except probe current. The probe current was 1 nA in a), 100 pA in b), 15 pA in c), and 5 pA in d). The obtainable corresponding probe diameters vary from 1 nm to 0.2 nm.

a special processing technique like second difference spectra acquisition [14] has to be used in future.

A further problem to get direct control of the Al concentration in GaAlAs via EELS is correct background estimation for the Al-L and Al-K edges. In both cases minor As edges (As-$M_{4,5}$ at 42 eV and As-L_1 at 1520 eV) in front of the Al edges do not allow to apply routine background subtraction methods for the quantification of the Al edges.

For quantitative microanalysis of GaAs/GaAlAs heterostructures the Ga-L edge is much more suitable. After background subtraction it is possible to determine the Ga concentration with an accuracy of $x \leq 0.05$ in $(1 - x)$. For that purpose the Ga intensity recorded in a GaAs layer is used as an internal standard and compared to the Ga intensity recorded in a GaAlAs layer. From such data the Ga concentration can be determined and the Al concentration can be calculated.

The spectra taken with different probe currents demonstrate that very small probe currents (5 pA) and probe diameters down to the range of 0.2 nm can be used to perfom chemical analysis of GaAs/GaAlAs heterostructures.

In order to check the limits of spatial resolution with EELS it will be neccessary to record line scans at high energy losses (1 keV – 2 keV) across heterostructures. The combination of a field-emission STEM and parallel detection of EELS as described in this paper should allow to record such linescans within reasonable recording times (some minutes).

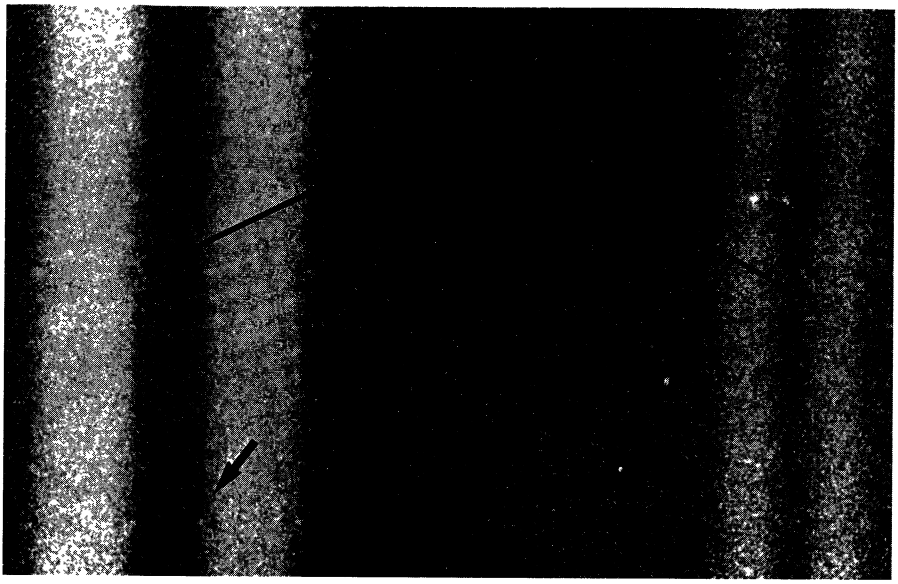

Fig. 7. — (200) dark field image of a MBE grown GaAs/GaAlAs heterostructure.

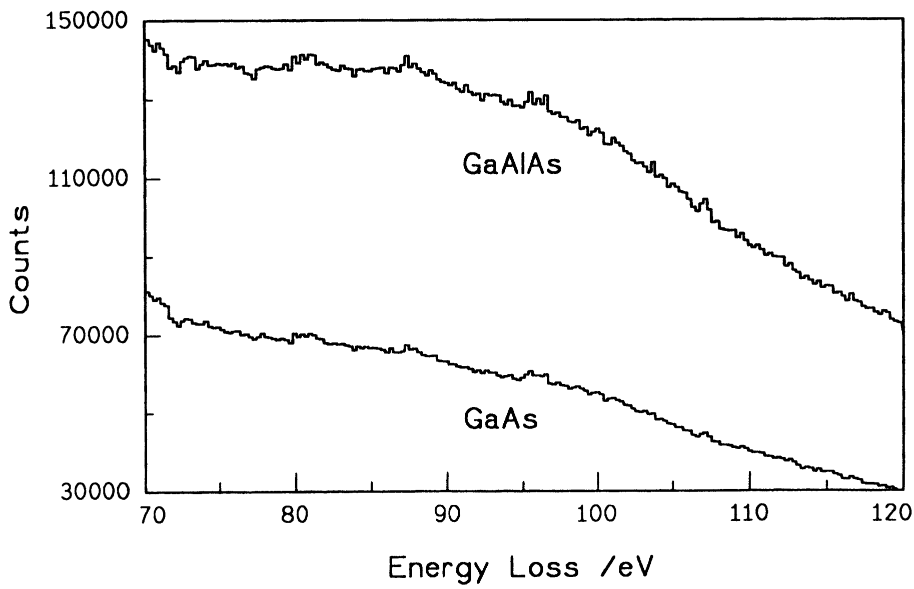

Fig. 8. — EEL spectra of GaAs and GaAlAs. The Al-L edge in the GaAlAs spectrum is visible (probe current 1 nA, probe diameter 1 nm, 2 s recording time).

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