Experimental report HC-1750

In preparation of and during the experiment HC-1750, we have developed an optical setup to collect the optical emission from a GaAs membrane under external mechanical stress. In order to collect light from a large solid angle, we have used a concept based on mirror optics, as shown in Fig. 1. Light from the center of rotation of the ID01 diffractometer is collected by a parabolic mirror and coupled into an optical fiber using a second mirror of the same type. The fiber is coupled to a spectrometer (Shamrock 193i) and the spectra are analyzed using a PCO CCD camera.

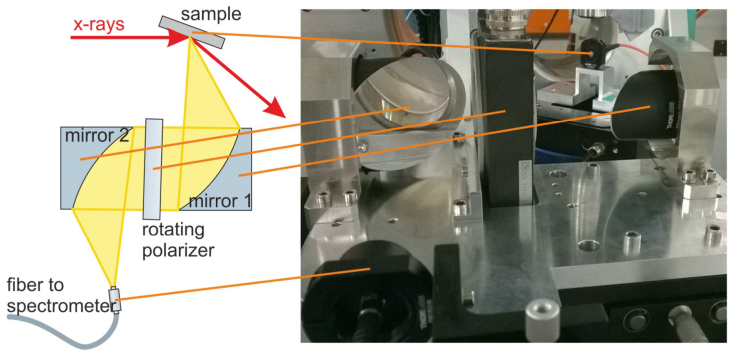

Figure 1: sketch and image of the mirror optics setup to collect GaAs band gap luminescence

The excitation is performed directly using the incident x-ray beam, in this case at an energy of 10.375 keV. Several difficulties had to be addressed during the development of the setup:

- x-ray excited optical luminescence (XEOL) is not yet well understood in the hard x-ray regime, so no reference data on intensities to be expected exist, making alignment and optimization of the setup difficult.
- due to delivery problems with the mirrors, the original design had to be slightly adapted, and only Al-covered mirrors were available at the time of the experiment, with rather bad reflectivity values for the wavelength of interest (GaAs band gap luminescence around 870 nm wavelength)
- the mirror setup was pre-aligned prior to the experiment, so no motorized stages for the mirrors had been foreseen. It turned out, however, that mutual alignment of the two mirrors is more critical than estimated, in order not to lose too much intensity when coupling the light into the fiber. Realignment is, however, rather time consuming.

Nevertheless, it was possible to record XEOL spectra from the GaAs membrane, as shown in Fig. 2. Beside the main GaAs luminescence, several other peaks are also visible; they are related to the piezo material ($[Pb(Mg_{1/3}Nb_{2/3})O_3]_{0.72}$–$[PbTiO_3]_{0.28}$; PMN–PT), the exact origin still needs to be determined.

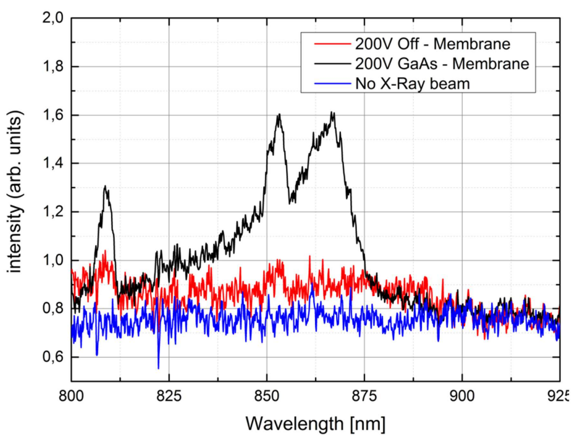

Figure 2: XEOL signal w/o beam (blue), with the beam on the piezo off the GaAs (red) and on the GaAs membrane (black).

To determine the line shift of the bandgap luminescence as a function of strain, we have used two different sample layouts. The first sample was a GaAs membrane Au-bonded onto a monolithic piezo substrate. Figure 3(a) shows the XRD rocking scan around the (004) Bragg reflection without piezo voltage and with 200 V applied to the piezo, Figure 3(b) the corresponding luminescence spectra. With the very limited signal to noise ratio of the luminescence signal available at that stage of the experiment, the data quality is not sufficient to evaluate a line shift. One limiting factor here, compared to conventional PL experiments in the lab is the fact that measurements are carried out at room temperature, leading to very broad emission lines; expected lineshifts are in the order of 2 nm. Another experimental issue is that when a voltage is applied to the piezo, its strain state changes quickly within a very short time (seconds; the rate of voltage change), but then ’’creeps’’ over a longer period of time, and even after about 1 hour is not completely settled.

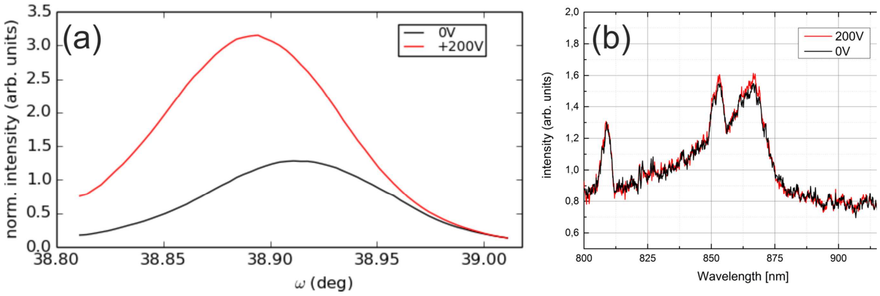

Figure 3: (a) XRD and (b) XEOL signals of the GaAs on the monolithic piezo device with and w/o applied voltage.

To circumvent those shortcomings, we switched to a second device, where the piezo is structured into a pattern of six “legs” as shown in Fig. 4(a). Here, different voltages can be applied pairwise on opposite legs, and the membrane, which in this case is bonded using SU8 photoresist, exhibits much larger strain variations as has been confirmed prior to the beamtime using PL measurements in the optical lab in Linz. Hence small drifts should be less problematic, and larger line shifts due to the larger strain values should make the data evaluation easier. Unfortunately, a very unexpected problem arose with this sample, as shown in Fig. 4(b), depicting the XRD rocking scans of the GaAs membrane on top of one of the piezo legs: obviously, repeating the same scan under applied voltage to the piezo not only

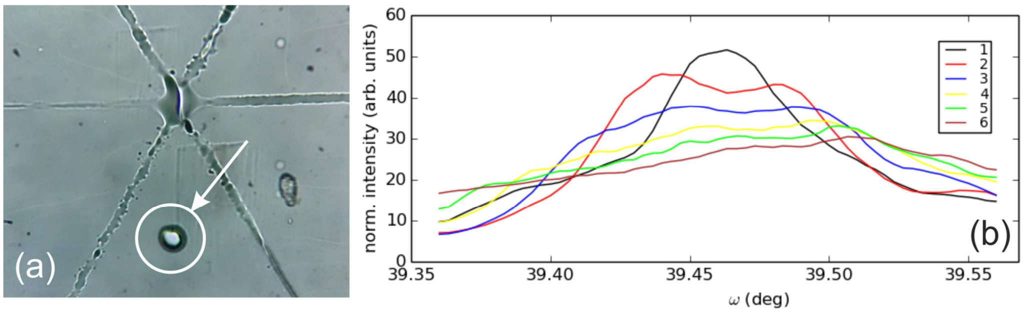

Figure 4. (a) microscope image of the six-leg piezo device, the area where the SU8 degraded due to x-ray exposure is marked (b) XRD rocking scan series around the (004) peak.

shows a finite drift due to Piezo saturation, but a complete change of the rocking scan shape, with a dramatically increasing width. This indicates that the membrane is bending considerably. Inspection of the sample after a total of about 30 min of exposure to the x-ray beam in an optical microscope revealed that actually the SU8 was degrading and irreparable damage to the bonding occurred, which led to a local lift-off of the membrane, resulting in very broad rocking scans. Since the strain measurements in several azimuths take much longer, it was finally not possible to extract valid strain data and hence establish the relation between strain and PL line shift.