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X-RAY DIFFRACTION IN SEMICONDUCTOR INDUSTRY AND RESEARCH

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ABSTRACT

The paper gives a short overview on application of X-ray diffractometry (XRD) and reflectometry (XRF) in semiconductor industry and research. The first part is devoted to the construction of X-ray diffractometer, the second one to the most important measurements in three steps of semiconductor device construction: i) growth and preparation of substrates, ii) growth of epitaxial layers, iii) processing. The following crystallographic properties of substrates are measured: dislocation density, radius of bowing and concentration of point defects. For epitaxial layers and their structures, XRD gives composition of each layer, strains and interfacial roughness. Finally, in processing, the metal and insulator polycrystalline layers are used. The task of XRF and XRD is to examine the chemical composition of these layers, density, strains, as well as interfacial and surface roughness.

1. INTRODUCTION

First experiment of X-ray diffraction from a crystal was done by Max von Laue in 1912. This observation not only gave an evidence of a crystallographic order of atoms but also started a big branch of science- crystallography based on X-ray diffraction. Within the next 30 years, the structure of more than a thousand substances was established. In parallel, Bragg brothers and others developed X-ray diffraction theory for perfect crystals. Ironically, almost half a century had passed when we gained a real profit of this theory, when the crystals got good enough and the measurement accuracy got high enough to observe phenomena predicted by those famous scientists.

First X-ray diffractometer was constructed in 1948 by laboratory of Philips Analytical. The instrument used a counter instead of a photo plate and had a mechanical rotations of the sample and the counter.

Modern diffractometers possess a series of hardware inventions (beam monochromators, position sensitive counters, etc.), but the most spectacular progress has been achieved due to personal computers used for automatic data collection and interpretation of results. As an example can be given time for a typical phase analysis. Twenty years ago it took almost a week, whereas now it is less than 5 minutes.

X-ray diffractometer has become the most basic analytical tool for any lab that manufacturers any material: single crystals, epi layers or any polycrystalline ones.

The aim of this paper is to show the state-of-the-art of X-ray diffractometry and reflectometry used for examining crystallographic properties of materials in semiconductor industry. In the first part, the most modern diffractometer design will be discussed that should help in choice of diffractometer configuration. In the second part, I will give examples what and how should be measured.

2. X-RAY DIFFRACTOMETER

Every diffractometer consists of three parts: i) primary beam path, ii) goniometer to mount the sample, iii) diffracted (reflected) beam path.

2.1. Primary beam path

For your particular measurement you should decide on the parameters of the primary beam: its divergence, size and brightness.

X-ray beam is produced in tubes with copper anodes giving wavelength of about 1.54 Å (other wavelengths are rarely used in semiconductor research). The most intense light can be obtained from rotating-anode units, however, because of their high price and complexity, they are not frequently used. The ordinary X-ray tubes produce typically a very divergent radiation from a line of 1 mm x 10 mm. If the light is extracted in direction perpendicular to that line, we have line focus, if in parallel, we have point focus of 1mm x 1 mm. There are as well microfocus tubes giving very bright beam of size less than 0.1 mm. Such tubes can be used, for example, for examining single-chip devices.

In order not to loose too much of divergent radiation, multilayer bent mirrors (Goebel) are used. The mirror collects tube radiation of divergence more than 10 degrees, collimating it to less than 1 degree, increasing the beam brightness by about ten times. Further collimation is done using a set of slits down to a divergence of a few arc minutes.

For the most precise measurements, it is necessary to decrease the primary beam divergence down to a few seconds. It is done by multiple reflections from U-shaped germanium crystals (Bartels monochromator). Figure 1 shows the idea of such monochromator. It should be mentioned, that in the direction perpendicular to the plane shown, the beam divergence is of about 3 arc deg, what creates a big problem for data interpretation in case of bad-quality crystals.

2.2. Goniometer head

There are two options for the goniometer configurations: 1) vertical (so called theta-theta) and 2) horizontal.

In the vertical configuration, the sample is placed horizontally and only slightly adjusted (if necessary) on the goniometer head. The biggest advantage of this configuration is that the sample is not glued nor mechanically clipped. Its disadvantage is rather low angular precision (a few arc seconds), not sufficient for examinations of nearly perfect epi layers and crystals.

The most flexible goniometer head is so-called “open Eurlarian cradle” that enables to put the sample in almost every position with respect to the primary beam.

Almost all measurements are done in reflection mode (Bragg case). However, in some special cases, measurements can be done in transmission mode.

2.3. Reflected beam

The reflected beam is analyzed either by a set of slits or U-shape germanium crystals and then recorded by a counter. The 2D detectors are more and more frequently used as they speed significantly the measurements up (but not in all cases).

3. EXAMPLES OF MEASUREMENTS

3. 1. Rocking curves

Figure 1 shows the diffractometer configuration used for crystals or epi layers of a high crystallographic perfection. The measurement is done with a primary beam well monochromatized and a detector wide open. After somehow troublesome adjustment of the sample, the dependence of reflected beam intensity vs. angular position of the sample is recorded (rocking curve).

The experimental data is then compared with simulations done based on a dynamical X-ray diffraction. Fig. 2 presents an example of the rocking curve for a high-perfection multiple quantum well (MQW) of InGaN/GaN. Comparison with a simulation gave an exact information on InGaN composition and thickness of quantum wells and barriers.

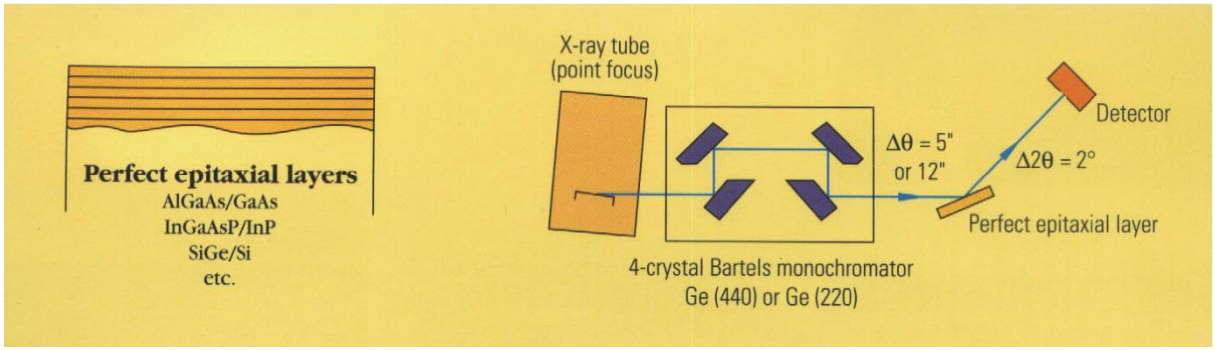

Fig. 1. Double axis configuration (other names: double axis, rocking curve configurations).

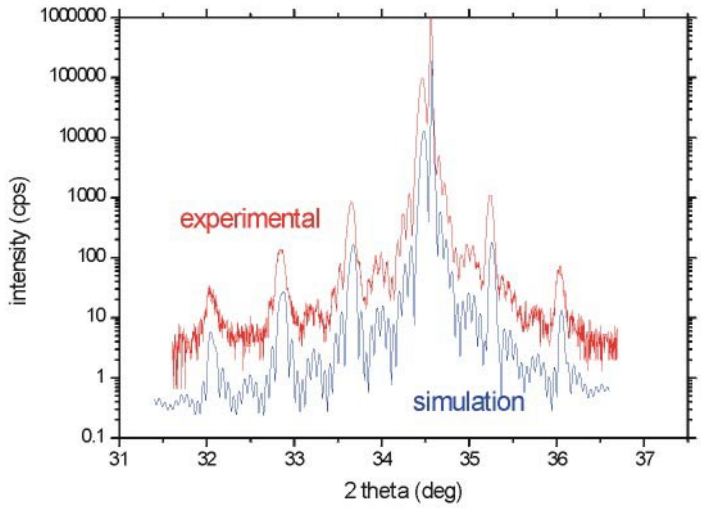

Fig. 2. Rocking curve from multiple quantum well of $In_xGa_{1-x}N/GaN$. The results of simulations: x=3.2%, d(well)=3.2 nm, d(barrier)=7.1 nm.

3. 2. Triple-axis measurements

In the case of worse quality materials, or when we want to measure lattice parameters of the crystal. triple-axis configuration is used, when an analyzer crystal is put in front of the detector (Fig. 3). It enables to measure precisely (accuracy Da/a of about 10 ppm) the lattice parameters in various crystallographic directions.

Fig. 4 shows an example of the curve obtained in triple-axis mode for GaN layer on sapphire implanted with Mg atoms. The left-hand side shoulder is caused by lattice expansion (implantation damage) removed by annealing at high pressure.

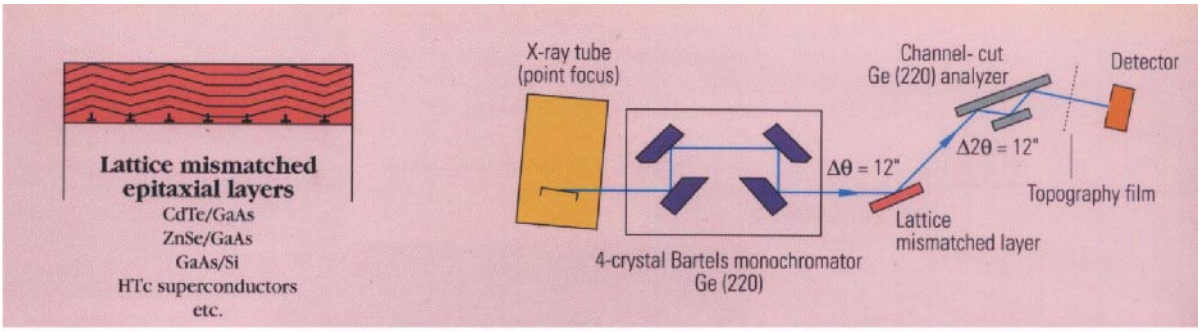

Fig. 3. Triple axis (triple crystal) configuration

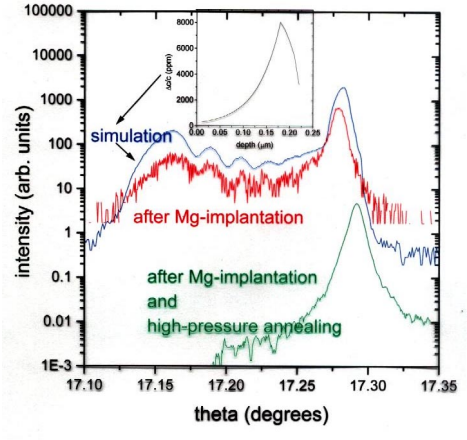

Fig. 4. Triple-axis curves for GaN/sapphire layer after Mg implantation and annealing. The inset shows the depth dependence of strain extracted from the experimental curve.

The most common application of the triple axis configuration is reciprocal lattice mapping used for evaluation if the epitaxial layer is strained or relaxed to the substrate. Fig. 5 shows the reciprocal lattice maps taken for AlN layers on GaN substrates. One can see that the thin layer is almost fully strained to GaN (the same lattice parameters parallel to the surface), whereas the thick one is relaxed.

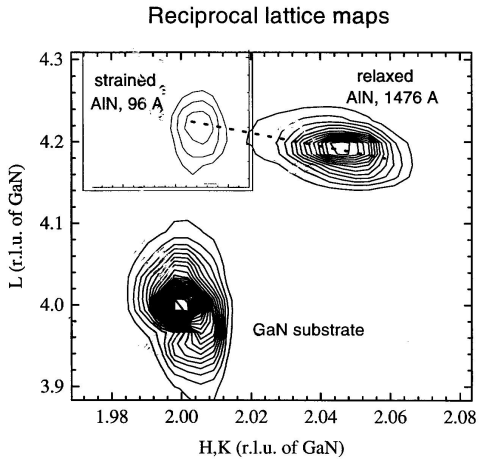

Fig. 5. Reciprocal lattice maps (in hkl units) for an asymmetrical reflection 22.4.

3. 3. Thin layer analysis

Fig. 6 shows a configuration for analysis of thin (up to about 200 nm) layers of any kind-crystalline, polycrystalline or amorphous.

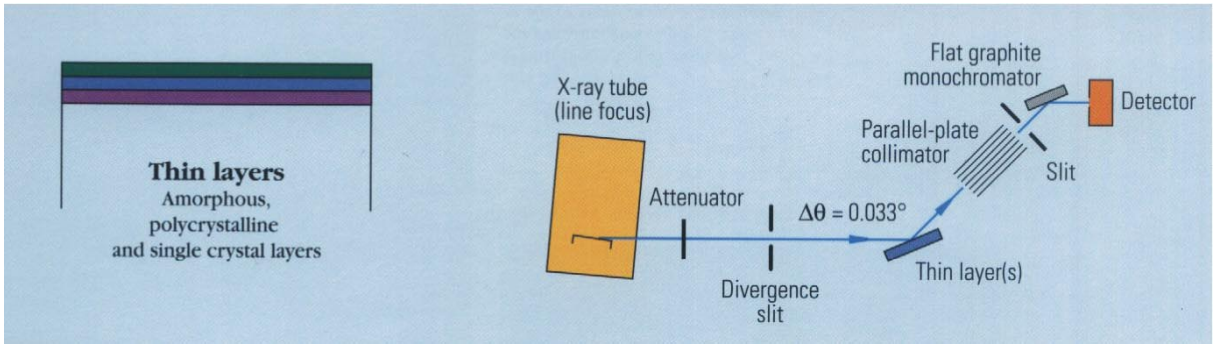

Fig. 6. Thin-film configuration.

When using very narrow slits to collimate the primary and reflected beam and when the sample is big (more than 10 mm) and flat, it is possible to examine the reflection (not diffraction) of the X-ray beam.

Fig. 7 shows an example of the reflection curve for Ni layer on Si. From the curve it is possible to estimate layer density (from critical angle), layer roughness (from the inclination of the curve and the oscillation amplitude) and layer thickness (from oscillation period). The RMS roughness and thickness can be estimated with accuracy of 0.2-0.4 nm.

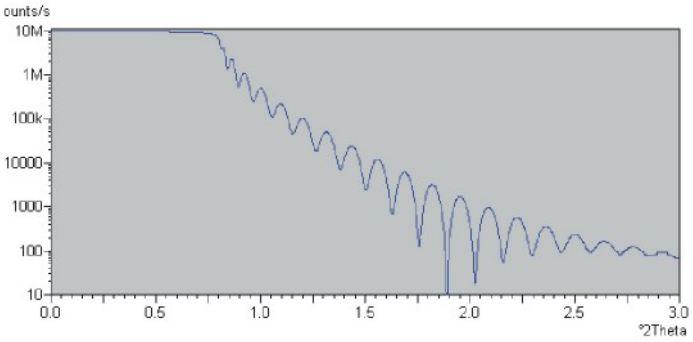

Fig. 7. Reflectivity curve for 60 nm Ni on Si. The surface roughness is of 1.5 nm.

3. 4. Bragg-Brentano configuration

For the phase analysis of a thin polycrystalline layer, in front of the detector the parallel plate collimator is placed. Having an incidence angle fixed at a low value (below 1 deg), this collimator defines a reflection angle and it is possible to obtain a diffractogram even for very thin layer. By changing the incidence angle we can also vary the X-ray-beam penetration depth and examine the phases at different depth. If the polycrystalline material is thicker than about 100 nm, the standard Bragg-Brentano configuration (Fig. 8) gives higher intensity and resolution.

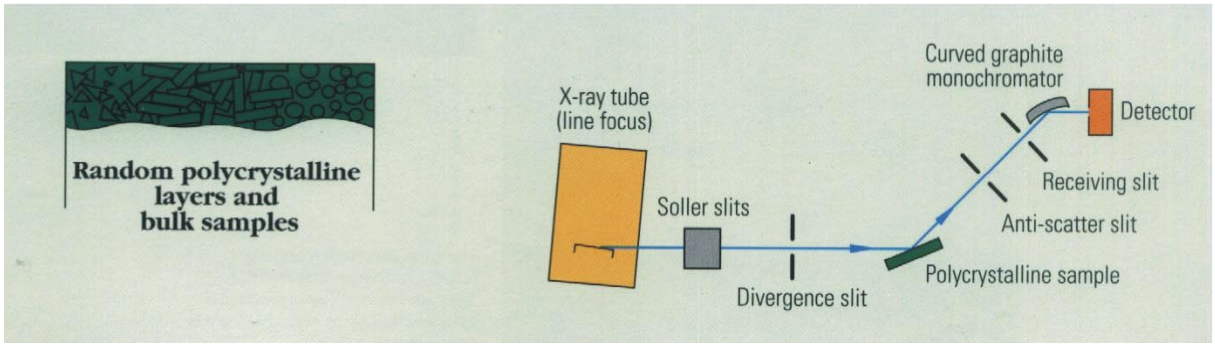

Fig. 8. Bragg-Brentano configuration for analysis of polycrystalline samples.

The idea of this configuration is to use a divergent primary beam that illuminates a large area of the sample. The reflected beam is convergent and focused at the receiving slit for the highest possible accuracy. A typical X-ray diffractogram for a polycrystalline sample is shown in Fig. 9.

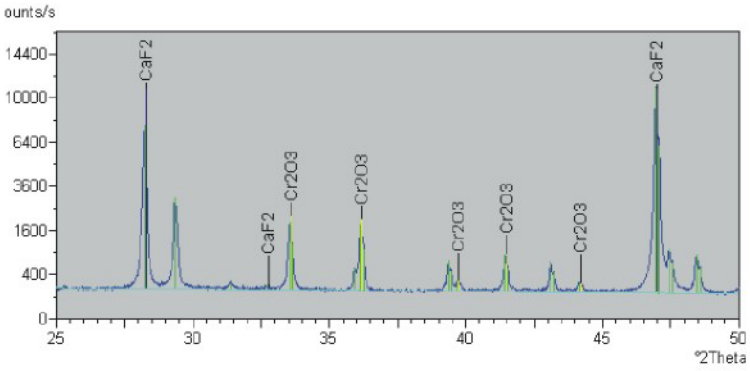

Fig. 9. Diffractogram for a mixtrure of $CaCO_3$ (peaks not labelled), Cr_2O_3 and CaF_2.

4. CONCLUDING REMARKS

X-ray diffraction and reflectivity is the most common technique in every lab producing materials for electronics. However, it is always a trade-off between time of analysis and amount of information to be extracted. Sometimes, only the basic info is needed, for example, the composition of AlGaAs layer on GaAs. Such info can be obtained within a few minutes. However, if one needs density of defects in that layer, the analysis may last for a few months, what is usually not acceptable by technology. Even worse situation is with less perfect crystals. In that case, there is no good theory that could be quickly applied.

In recent years, there has been a rapid development of diffractometer hardware. In the nearest future we expect that the 2D detectors of a very high resolution will be widely introduced enabling even quicker analysis without missing a lot of information that is lost with the conventional or even position-sensitive detectors.

Interesting applications of X-ray diffractometers are their mounting on-line in the production systems, for example, in the epi-growth machines. However, the technical problems (for example, vibrations) are too difficult to be easily overcome.