SPUTTER DEPTH PROFILING OF OPTICAL WAVEGUIDES USING SECONDARY ION MASS SPECTROMETRY*

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Abstract

The technique of sputter depth profiling by means of secondary ion mass spectrometry of samples with high resistivity is reviewed. As examples we discuss optical waveguides made in lithium niobate by titanium indiffusion and implantation and also yttrium iron garnet waveguides grown by liquid phase epitaxy on gadolinium gallium garnet. Depth profiling of these waveguide structures has been performed and the necessary precautions to prevent charging by the primary ion beam are discussed. In some cases, coating with a metallic layer is adequate, but a more universal method is charge neutralization by an additional electron beam.

1. Introduction

Sputter depth profiling by means of secondary ion mass spectrometry (SIMS) is an established technique to measure concentrations of dopants as a function of depth, since this method has the advantage of high sensitivity and high depth resolution.

Most of the experimental work has been done with semiconductors and in the case of implanted dopants absolute concentration values can be determined from the total implanted dose. Several excellent reviews of relevant measurements exist (Hofmann 1980, Zinner 1980, Magee, Honig and Evans 1982, Wittmaack 1982).

In SIMS, a beam of energetic primary ions impinges on the sample and initiates a collision cascade. A flux of sputtered target atoms and ions leaves the sample, the ions are then focussed onto a mass spectrometer and mass analyzed. In many instruments, energy analysis by electrostatic fields is also possible.

In depth profiling, the mass peaks of interest are recorded as the primary beam erodes the sample, thus giving information on the elemental concentration as a function of time. The time scale can be converted into a depth

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* Dedicated to Prof. J. Giber on his 60th birthday

scale as long as there is a linear relationship between depth and erosion time. A change in the ion yield with depth will distort the profile, the chemical environment should therefore not change to a degree that the ion yield is altered significantly.

In depth profiling by sputter erosion, a crater is formed and the influence of sputtering from the crater edges has to be eliminated. With a stationary primary ion beam, there will always be a large contribution of atoms sputtered from the crater walls, which means from smaller depth, and this will distort the profile seriously. Rastering of the ion beam, thus producing a wide crater with flat bottom in combination with synchronous gating of the signal, detecting only ions from the central flat part of the crater, is the usual way of solving this problem (Wittmaack 1977). Even then, redeposition of atoms sputtered from the crater walls into the bottom has to be avoided since electronic gating cannot discriminate against this effect.

In the case of insulators, the bombardment of the sample by the primary ion beam leads to electrostatic charging and a shift in potential. The change in energy of the sputtered secondary ions leads to a complete loss of signal or at least to a severe decrease in mass resolution, especially when using quadrupole mass filters. Charging can also lead to enhanced migration of dopants (Magee and Harrington 1978).

Several methods have been used to overcome the problem of charging, they include metallic coating (Bremer et al. 1989), the use of diaphragms in combination with negative primary ions (Morgan et al. 1977) charge compensation by electron bombardment (Wittmaack 1979) and neutral primary beam bombardment (Van den Berg 1986). The most universal method requiring minimum sample preparation is electron beam bombardment, and in this paper we discuss the technique of depth profile measurements of optical waveguide structures using both coating and electron beam neutralization methods.

2. Instrumentation

The experimental set up is shown in Fig. 1 (Kischkoweit et al. 1987). It is based on a ultrahigh vacuum system with a based pressure of $2 \cdot 10^{-10}$ Torr. The primary ion beam system consists of an Atomika plasma ion source. Wien filter and fine focus lens the cylindrical deflector on the low pressure side of the differentially pumped system serves to eliminate neutral beam components. For depth profile measurements, the beam can be rastered by deflection plates incorporated in the fine focus lens over an are of up to 1.5×1.5 mm^2, the gated area can be chosen from 10% to 100% of the total area. The secondary ions are focussed onto the entrance slit of the analyzer by an extraction lens (Magee et al. 1978a). Mass spectra are recorded using

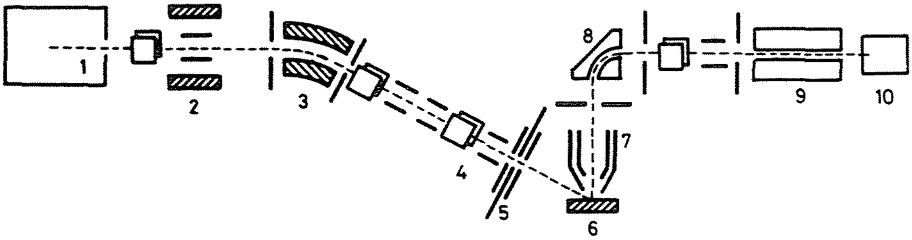

Fig. 1. Experimental set-up, schematic

1 Ion source
2 Wien filter
3 Cylindrical capacitor
4 Beam scan electrodes
5 Einzel lens
6 target
7 secondary ion lens
8 energy analyzer
9 quadrupole
10 multiplier

a 90°-spherical energy analyzer (Grundner et al. 1974) and an Extranuclear quadrupole mass spectrometer. The energy analyzer serves to eliminate high energy ions that would degrade mass resolution, it can also be used for ion scattering spectrometry (ISS). Data processing is performed with a Hewlett-Packard computer taking care of mass selection, multiplexing, graphical display and data storage.

The angle of incidence of the ion beams is 62° to the surface normal. During rastering, there is a small change of this angle of about 0.25°, the change of erosion rate across the crater is less than 1.5%, which can be neglected for our purposes.

A more serious problem could be sputtering of material from the crater edges into the bottom, material which is then measured. This effect will degrade the depth resolution when depth profiling from high concentration to low concentration, the falling edge of the profile will be broadened. The raster width in both perpendicular directions should be at least five times larger than the beam diameter (Wittmaack 1977).

3. Measurements

3.1 $LiNbO_3$ waveguides

Optical waveguides, the basic elements for optical signal circuits, can be produced in electrooptical materials by various methods like titanium indiffusion (Schmidt and Kaminov 1974), proton exchange (Jackel and Rice 1981), ion implantation (Wei et al. 1974) and combination of these techniques.

As a first example, we investigated a waveguide in $LiNbO_3$ produced by titanium indiffusion (Bremer et al. 1989). A titanium layer, 70 nm thick, was evaporated onto the $LiNbO_3$ substrate and diffused for 16 h at 1000°C. After diffusion, the sample was evaporated with a layer of gold approximately 1 μm thick. This proved adequate to prevent charging.

The depth profile of titanium was measured with 2 μA Ar^+ ion beam of 10 keV, 300 μm diameter, which was rastered over an area of 1.5×1.5 mm^2. The etch rate was about 1 μm/h. A gated area of 10% of the rastered area was selected.

The measured depth profile for titanium is shown in Fig. 2. It closely follows a gaussian distribution (solid line), as expected from diffusion theory. Not shown in Fig. 2 is the Nb^+ matrix reference signal, which is constant, indicating a constant etch rate and ion yield. The depth scale was determined by a laser stylus measurement of the final crater depth. Control measurements of crater depth as a function of sputtering time confirmed the linear relationship between depth and time.

Deviations of the measured profile at large depth is probably due by redeposition effects from the crater edges caused by the tail of the ion beam profile. Focussing of the beam to a smaller diameter should improve this situation.

The concentration scale was calibrated by the total amount of incorporated titanium, given by the layer thickness. The diffusion constant calculated from the depth profile is $D(1000°C) = 5.25 \times 10^{-17}$ m^2/s.

An alternative method of producing waveguides is to use ion beam mixing (Bremer et al. 1989). The sample was coated with some tens of nm of titanium and subsequently irradiated by a 3 MeV Ti^+ beam, with a total

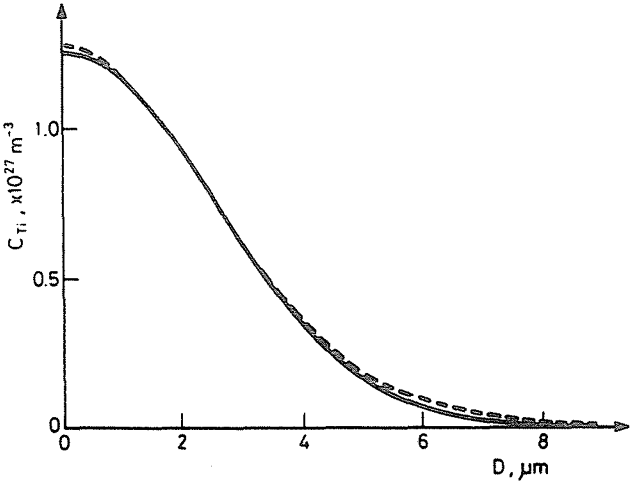

Fig. 2. Titanium depth profile of diffused sample measured by SIMS. 70 nm of titanium were diffused into $LiNbO_3$ for 16 h at 1000°C. The broken line represents the measured distribution, the solid line represents the Gaussian fit

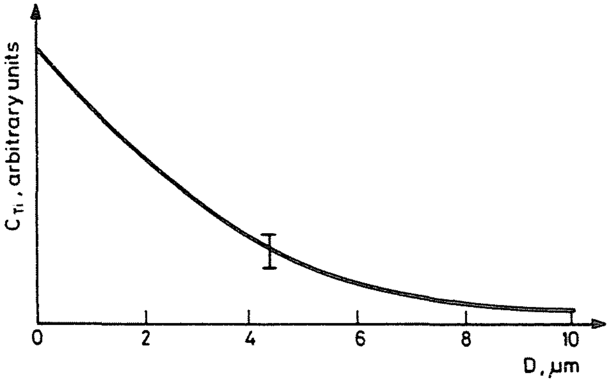

Fig. 3. Titanium depth profile of sample produced by ion beam mixing of a 40 nm titanium layer (beam energy 3 MeV, total dose $10^{16}/cm^2$) and epitaxial regrowth for 7 h at 1000°C. The concentration scale is not scaled

dose of $1 \times 10^{16}/cm^2$. The titanium layer was only partly incorporated. Radiation damage by the high energy ion beam was removed by epitaxial regrowth at 1000°C for 7 h. The depth profile of titanium is shown in Fig. 3. The concentration cannot be scaled because the total amount of incorporated titanium is unknown.

There is a considerable difference in the shape of the mixing profile from that made by thermal diffusion. An even larger variety of profiles can be generated by direct implantation since the range of the implanted species is easily controlled by the beam energy (Bremer et al. 1988).

3.2 *Magnetic garnets*

Magnetic garnets like yttrium-iron garnet (YIG) are the basic materials for magnetooptic devices. Sputtering depth profile measurements suffer from charging of the sample and electron beam irradiation has been used for charge neutralization (Wittmaack 1979). In our experiments the electron gun was operated between 100—300 eV with currents of up to 10 μA, which is about 10 to 20 times larger than the primary ion current. This method proved very efficient in eliminating the charging and can be applied to almost all insulators.

In the case of very high resistivity materials coating will not be sufficient, since charge will build up in the crater as soon as the beam pierces the conducting layer. Also, in many cases coating of the sample may not be wanted since the surface could be altered by segregation effects and ion beam mixing by the primary beam.

The samples consisted of yttrium-iron garnet layers 2—8 μm thick grown by liquid phase epitaxy on gadolinium-gallium (GGG) substrates (Brockmeyer

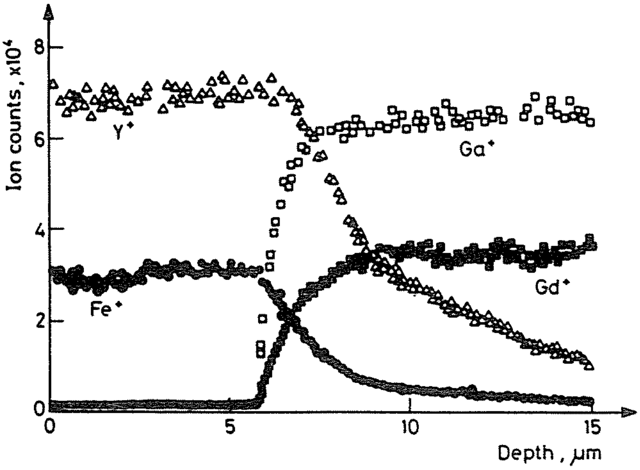

Fig. 4. Depth profile of an yttrium iron garnet waveguide grown by liquid phase epitaxy on gadolinium gallium garnet substrate. The ion count rate for Y, Fe, Gd, Ga is shown as a function of depth. For depth scale calibration see text

1989). The YIG layer acts as the waveguide, since its refractive index is higher than that of the substrate.

The depth profile of a YIG film grown on a (111) GGG substrate is shown in Fig. 4 (Priggemeyer 1989). The sample was sputtered with a 10 keV Ar^+ beam of 1 μA, rastered by 1.5×1.5 mm^2. Surface charging was compensated by electron bombardment at 300 eV and 10 μA. The total depth of the crater was measured with a laser stylus, the thickness of the YIG film was known from interferometric measurements (Brockmeyer 1989). The two values were used to transform the time scale into a depth scale. Within the error limits of these measurements, the etch rate remains constant to within ±20% from film to substrate.

The flat profiles of the elements Y and Fe show that the YIG film grown by liquid phase epitaxy is very homogeneous, indicating that the growth conditions were indeed well controlled.

4. Conclusions

We haved shown in this paper that the technique of sputter etch profiling in insulators has now reached a standard comparable to that achieved in metals and semiconductors, provided appropriate precautions are taken against charging effects. Among these methods, electron beam neutralization is the most universal method, since it can be applied to almost all samples.

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