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ScienceDirect

Thin Solid Films 516 (2008) 5137 – 5143

thin solid films

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Study of iridium/germanium interaction in a lateral diffusion couple

A. Habanyama [a,b], C.M. Comrie [a,*]

[a] Department of Physics, University of Cape Town, Rondebosch 7700, South Africa
[b] Department of Physics, University of Zambia, P.O.Box 32379, Lusaka 10101, Zambia

Received 18 April 2006; received in revised form 14 December 2007; accepted 4 January 2008
Available online 11 January 2008

Abstract

Ion beam analysis using micro-Rutherford backscattering spectrometry has been used to investigate the interaction between germanium and iridium in a lateral diffusion couple. Optical microscopy, scanning electron microscopy and atomic force microscopy have also been employed. When samples of germanium islands on iridium films are annealed within a range of temperatures between 600 to 800 °C, substantial lateral diffusion is observed, resulting in a number of reaction regions. Micro-Rutherford backscattering analysis indicates that the phase Ir_3Ge_7 stretches across the original island interface at all temperatures, with the phase Ir_4Ge_5 forming in the reaction region with unreacted iridium. The phase $IrGe_4$ is observed to nucleate in the middle of the island at temperatures above 800 °C. Depth information is readily obtainable from micro-Rutherford backscattering spectrometry which is used in conjunction with atomic force microscopy data to estimate the densities of the phases formed. The results demonstrate the complementary nature of the techniques used for studying lateral diffusion couples.

Keywords: Iridium; Germanium; Diffusion couples; Microbeam; Rutherford Backscattering Spectrometry (RBS)

1. Introduction

The samples used for studying lateral diffusion reactions are composed of a thick island of one material on top of a much thinner film of another material. Upon annealing the island material would react with the underlying film, going through a sequence of phases until the most island-material rich phase is formed. Since no further reaction with the underlying film is possible, the most island-material rich phase may then grow laterally until it attains a critical width, after which other phases appear and grow simultaneously. This is a case of multiple phase formation as would be found in bulk diffusion couples. Lateral diffusion couples thus provide the transition between thin film and bulk behavior.

Lateral diffusion couples have been used to study the interaction between a metal and a semiconductor [1–7] or between two metals [7–13]. With respect to the former, the bulk of the work was done with silicon. Various procedures have been developed to study these couples. Initially, sample structures were grown on Al_2O_3 substrates or oxide coated silicon wafers; the oxide (SiO_2) provided an insulating buffer layer to prevent reaction with the underlying silicon. For quantitative analysis, the diffusion length was measured with scanning electron microscopy (SEM) and the composition determined by energy dispersive spectroscopy (EDS) [1–3].

To improve lateral resolution a procedure developed by Chen et al. [4] has been used by a number of workers [4,8–13]. This procedure involves the deposition of lateral diffusion sample structures on a single-crystal sodium chloride substrate. The diffusion couples are then floated off on de-ionized water which dissolves the sodium chloride, thereby leaving self-supporting sample structures which are then annealed. The reaction kinetics are analyzed using transmission electron microscopy, the reacted phases having been identified from their crystallographic structure by selected area diffraction [4,8–13]. Here too the chemical composition is determined by EDS. More recently, micro-Rutherford backscattering spectrometry (μRBS) has been used to study lateral diffusion couples [5–7]. The major advantage of this technique is its ability to give depth information. Ding et al. [5] used this technique to study lateral diffusion couples of the Ni-Si system, de Waal [7] used it to

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* Corresponding author. Tel.: +27 21 650 3326; fax: +27 21 650 3343.
E-mail address: comrie@science.uct.ac.za (C.M. Comrie).

0040-6090/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.tsf.2008.01.005

study the Cr–Si and Ru–Al systems while Nemutudi et al. [6] used the technique to study the Pt–Ge system.

2. Experimental details

Thermally oxidized single-crystal silicon wafers were used as substrates. Electron beam vacuum deposition was carried out at pressures in the low 10^{-5} Pa range. A study of the phase formation sequence was carried out prior to the lateral diffusion study. This study was carried out with conventional thin film couples using RBS and X-ray diffraction (XRD) for phase identification. Sample structures with 550 nm of germanium and 90 nm of iridium deposited onto an oxidized silicon wafer were used. A thin layer of titanium (2 nm) was deposited onto the SiO_2 prior to the deposition of the Ge; this Ti layer reacted with SiO_2 forming a 'glue' without which the structure did not adhere.

In a further preliminary investigation the marker technique was used to monitor atomic mobility during phase formation. The term marker refers to a material deposited in the sample as a reference plane in monitoring the direction of flow of atoms. A thin layer of Ti acted as an inert marker interposed between coupling layers of Ir and Ge; the Ge layer being at the surface of the sample. Upon annealing of this structure both Ge and Ir atoms could have diffused across the marker at different rates causing it to shift in position towards the dominant diffusing species. The marker Ti signal was monitored by RBS for different annealing times. The dominant diffusing species during phase formation was determined by observing the relative shift of the marker.

In the preparation of lateral diffusion couples, a thin film of one material was first deposited. A silicon mask with an array of 390×780 µm^2 windows, was then brought into contact with the substrate without breaking vacuum. Island material was then deposited through the mask to form structures consisting of iridium islands on germanium films, and germanium islands on iridium films. The samples were then diced into nine or twelve identical samples, each with two or three islands. Furnace annealing was used to activate solid state interaction after which the samples were analyzed.

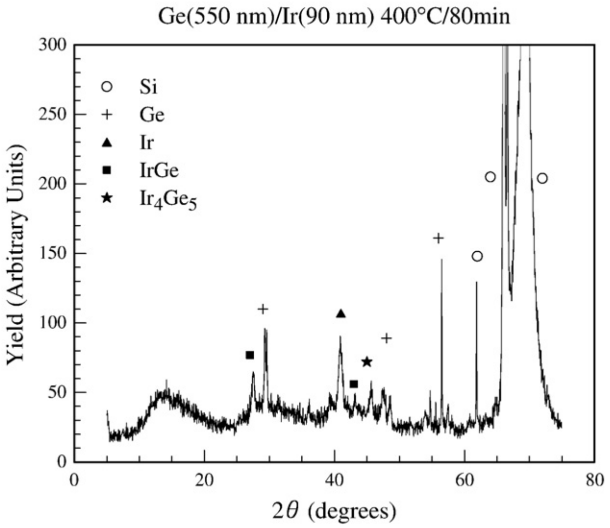

Fig. 1. X-ray diffraction spectrum, of a sample of composition SiO_2/Ge (550 nm)/Ir (90 nm) after annealing at 400 °C for 80 min.

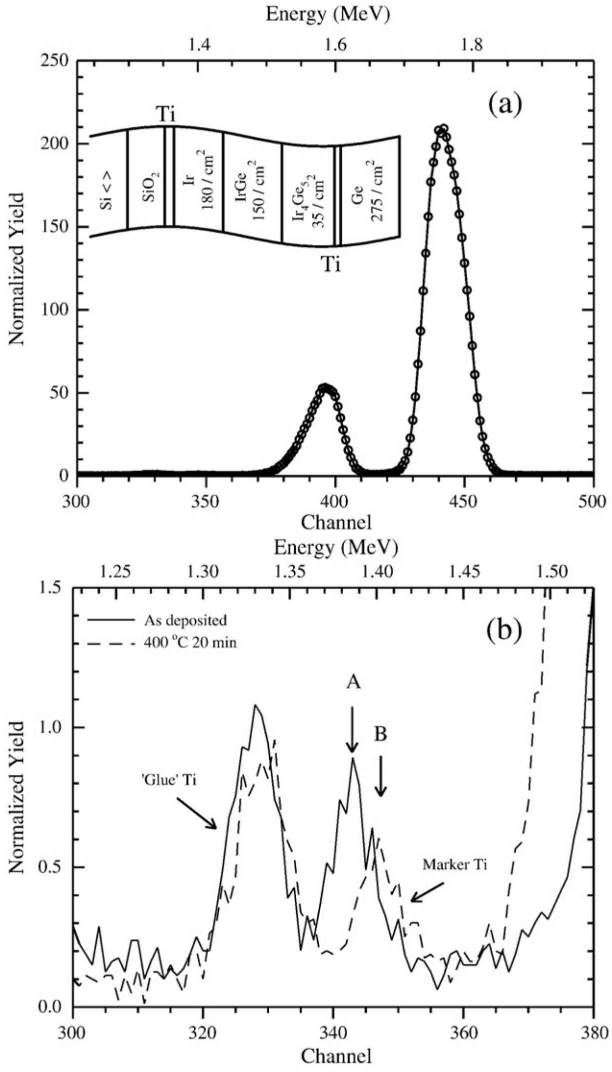

Fig. 2. (a) A diagram showing the results obtained from a RUMP simulation on an RBS spectrum of a sample annealed at 400 °C for 20 min. (b). Ti 'glue' and Ti marker signals before and after annealing. The marker signal shifted by 0.02 MeV to higher energies (A to B), while the Ti 'glue' signal did not shift.

The samples were examined by optical microscopy, and representative samples selected for further analysis by µRBS. A 2 MeV α-beam focused down to a diameter of ~ 2 µm was scanned across a well defined area of the samples. This area, typically of 400×400 µm^2, was chosen to include all reaction regions observed in the optical micrographs. Sample orientation was adjusted in such a way that the interfaces of the regions of interest lay horizontally in line with the original island edge so that the microprobe beam scanned parallel to the original island interface. RBS spectra were recorded along with position information. Typically, 128×128 spectra were generated in each run and once it had been established that no variation in composition was observed parallel to the interface, spectra were