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Combinatorial synthesis and characterization of a ternary epitaxial film of Co and Mn doped Ge (0 0 1)

F. Tsui [a,*], B.A. Collins [a], L. He [a], A. Mellnik [a], Y. Zhong [b], S. Vogt [b], Y.S. Chu [b]

[a] Department of Physics and Astronomy, University of North Carolina, Chapel Hill, NC 27599, USA
[b] Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

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Abstract

We report combinatorial molecular beam epitaxy synthesis and properties of a ternary epitaxial film of Co and Mn co-doped Ge grown on Ge (0 0 1) substrate. Structural effects were examined in situ by reflection high-energy electron diffraction and ex situ by microbeam X-ray diffraction techniques, and magnetic properties were probed by using magnetooptic Kerr effect. Ternary epitaxial phase diagrams have been studied for total doping concentrations up to 30 at.%, where regions of coherent epitaxy and rough disordered growth and those of near room temperature ferromagnetic ordering have been identified.
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Keywords: Epitaxial film; Magnetic semiconductor; Group IV semiconductor; Transition metal doping; Combinatorial thin film

1. Introduction

Advances in the synthesis of doped magnetic semiconductors have continued to enable a wide range of activities in the science and technology of spintronics. Recent publications [1,2] show that complementary doping using two transition metal dopants into Ge (0 0 1) lattice during molecular beam epitaxial (MBE) growth can stabilize epitaxial growth at significantly higher doping concentrations (as high as 15 at.%) than those from using a single dopant. It appears that using a dopant with a larger atomic radius than that of the Ge host in conjunction with a dopant with smaller atomic radius, at an appropriate atomic ratio between the two, can compensate for the internal stress caused by the individual dopants in the host lattice. Comprehensive studies of the ternary phase diagrams are needed, in order to explore interactions between the transition metal dopants beyond the effect of strain and, thus, enhance our ability to control synthesis and properties of this type of materials systems. The use of combinatorial approach is necessary for carrying out such studies of complex multi-component materials systems. Here, we present combinatorial MBE synthesis and characterization of a ternary epitaxial system that contains transition metals, Co and Mn, co-doped into Ge (0 0 1) in the range of doping concentrations between 0 and 30 at.%.

2. Experiment

The ternary $Co_xMn_yGe_{1-x-y}$ sample was grown on a semi-insulating Ge (0 0 1) substrate using combinatorial MBE techniques. The substrate was first cleaned using the standard RCA method, and then degassed at 600 °C under UHV conditions. A growth anneal cycle was employed to prepare a Ge (0 0 1) buffer layer of 380 Å thick, preceding the growth of the ternary combinatorial film. In each cycle, a thin Ge layer (typically 30–100 Å) was deposited at 250 °C and then annealed at 600 °C. The resulting surface is atomically smooth across the entire substrate with 2×1 surface reconstructions, as determined by real-time scanning reflection high-energy electron diffraction (RHEED) measurements.

The ternary film was subsequently grown by sequential multilayer deposition of Ge, Co and Mn trilayers. Each trilayer consists of three submonolayer wedges, a Ge wedge with thicknesses from 1.6 to 2 Å, a Co wedge with thicknesses from 0 to 0.4 Å and a Mn wedge with thicknesses from 0 to 0.7 Å. Each element (wedge) is spread linearly over

* Corresponding author. Tel.: +1 919 962 0305; fax: +1 919 962 0480.
E-mail address: ftsui@physics.unc.edu (F. Tsui).

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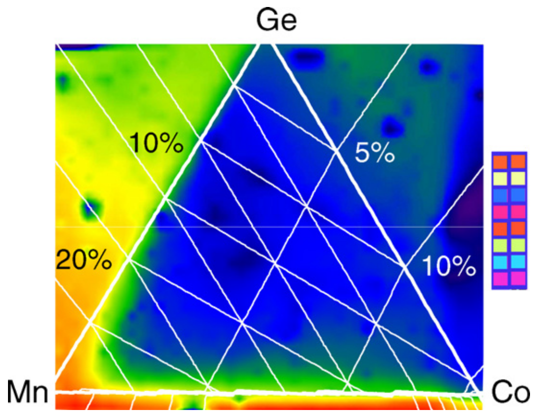

Fig. 1. (Color online) Specular reflectance at 658 nm of the $Co_xMn_yGe_{1-x-y}$ (0 0 1) combinatorial film with a nominal thickness of 500 Å. It was taken over a 2D mesh with spacing of 100 μm × 100 μm using a diode laser focused to a FWHM of 100 μm. The composition and the corresponding grid are determined by microbeam XRF measurements. Warm colors correspond to high reflectance and cold colors correspond to low reflectance. The range of reflectance shown is between 100% (red) and 80% (dark blue). The height of the image is 5 mm. The substrate is much larger than the region shown, and the sample is broken near the top of the image (not shown).

a lateral distance of 5 mm along a direction that is 120° rotated from the other two, creating a triangular region of the sample containing all three elements. The thicknesses expressed here are in Ge equivalent values, i.e. converted to the same atomic density as Ge matrix rather than the native metallic matrices for the two transition metals. The resulting concentration range for Co (x) is between 0 and 17 at.%, and the corresponding values for Mn (y) are 0 and 29 at.%. A triangular shaped shadow mask [3] was used in combination with pneumatic source shutters and real-time atomic absorption spectroscopy flux monitors to control the thickness profiles of the three elements and, thus, the composition profile across the substrate. To illustrate the physical extent of the ternary sample, a specular reflectance image is shown in Fig. 1. Ge and Co were evaporated from electron beam hearths and Mn flux was from an effusion cell. The growth rates were maintained at ~0.1 Å/s, at a substrate temperature of 250 °C and a base pressure of $\sim 10^{-10}$ Torr. The sample was terminated with a Ge layer (25 Å) and followed by a post-growth anneal at 450 °C. The nominal film thickness is 500 Å.

Magnetic properties were examined ex situ using magnetooptic Kerr effect (MOKE). A diode laser at a wavelength of 658 nm was used to perform longitudinal MOKE at near normal incidence (incident angle about 15° from normal). The full width at half maximum (FWHM) of the laser spot was focused to about 100 μm in diameter at the sample, which corresponds to a compositional variation of <0.5 at.% within the laser spot. A precision translation stage was used to scan the sample with respect to the laser. The MOKE signal was modulated using a photoelastic modulator, and the Kerr rotation and dichroism signal were detected separately via lock-in amplifiers. The DC specular reflectance signal was also collected, as shown in Fig. 1. A Joule–Thomson refrigerator mounted on the translation stage between the pole pieces of an electromagnet was used to vary sample temperature between 78 and 590 K.

Composition and structural properties were studied using X-ray microbeam techniques at the 2-BM beamline of the Advanced Photon Source (APS) at Argonne National Laboratory. Both microbeam X-ray diffraction (XRD) and X-ray fluorescence spectroscopy (XRF) techniques were used. A micron-sized X-ray beam (~10 μm in lateral dimensions) was scanned across the sample, using a precision sample-stage mounted at the center of a Huber 4-circle diffractometer. The spot size of the X-ray beam was about 10^3 times smaller than the ternary sample, which corresponds to a compositional variation of <0.05 at.% within the beam. The latter value is adequately small even with a modest spreading of the beam on the sample owing to different diffraction conditions, i.e. different incident angles. Both out-of-plane XRD measurements along [0 0 L] and 2D reciprocal space mapping in the (HHL) plane were carried out through various reflections, including (0 0 4) and (1 1 3).

In addition to a point detector for high resolution XRD experiments, an area detector (CCD with 2048 pixels × 3072 pixels) was also used for powder XRD experiments. The area detector enables simultaneous detection of scattering intensities within a relatively large region of reciprocal space, thus, enhancing sensitivity to intensities from certain types of disorders. The CCD was first aligned to the substrate using the Bragg reflections and then positioned to cut through the features in reciprocal space, such as the twinned {0 2 2} reflections resulting from stacking faults along ⟨1 1 1⟩. The 2θ values for the CCD images were calibrated using a CeO_2 NIST powder diffraction standard. An energy-dispersive florescence detector was used for the XRF measurements to quantify the amount of Co and Mn in the sample (composition grid in Fig. 1) using calibrated references, and it was also used to determine film composition during XRD experiments.

Both 2D mesh scans, typically a 100 μm × 100 μm grid over the entire sample or regions of interest, and line scans were used to probe the composition dependence, including XRD, XRF and MOKE experiments.

3. Results and discussion

The specular reflectance for the ternary region of the $Co_xMn_yGe_{1-x-y}$ (0 0 1) epitaxial film is in general lower than that of the binary regions of the film, as shown in Fig. 1, and is comparable to that of the bare substrate. In particular, the binary Mn_yGe_{1-y} portion of the film exhibits high specular reflectance indicative of metallic alloys, such as the Co_xMn_y binary alloys (shown at the bottom of Fig. 1). The amount of diffusive scattering at this wavelength is low and nearly uniform across the sample, excluding various extrinsic macroscopic flaws (e.g. pits) on the sample (the dark spots in Fig. 1). The qualitatively low specular reflectance of the ternary region with respect to the binary counterparts provides not only a convenient means for positioning the sample, but also a strong evidence that a large of portion of the ternary film is semiconducting.

The crystalline properties of the film exhibit distinctive composition dependent regions, as determined by XRD

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