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A compact molecular-beam epitaxy apparatus for *in situ* soft X-ray magnetic circular dichroism experiments

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An economical and easily movable molecular-beam epitaxy (MBE) apparatus which prepares magnetic ultrathin films and superlattices with atomically well controlled interfaces has been designed and constructed. Cleaning and characterization of substrates, sample deposition in a layer-by-layer fashion, and characterization of samples both during and after growth can be carried out in a single ultrahigh vacuum (UHV) chamber. This MBE apparatus is combined with UHV high-field magneto-optical instruments for *in situ* soft X-ray magnetic circular dichroism experiments on two-dimensional magnetic systems.

Keywords: molecular-beam epitaxy; magnetic circular dichroism.

1. Introduction

In the past decade, the magnetism of ultrathin films and superlattices has attracted much interest both experimentally and theoretically (Farrow *et al.*, 1993; Bland & Heinrich, 1994) because these two-dimensional systems exhibit a variety of novel properties, such as enhanced magnetic moments, metastable phases, perpendicular magnetic anisotropy (Carcia *et al.*, 1985), giant magnetoresistance (Baibich *et al.*, 1988) and oscillatory interlayer exchange coupling between ferromagnetic metal layers across non-magnetic spacer layers (Parkin *et al.*, 1990). From a technological point of view as well, these two-dimensional systems are becoming increasingly important as candidates for new devices such as next-generation magneto-optical recording media. It is essential to prepare samples with atomically well defined interfaces for studies of the origins of these phenomena, since recent investigations have suggested that the interface characteristics have an important role in determining the magnetic properties of the two-dimensional systems. As molecular-beam epitaxy (MBE) is the most reliable method for growing ultrathin films and superlattices in a layer-by-layer mode (Herman & Sitter, 1989), it is highly desirable to study the properties of two-dimensional systems *in situ* using samples produced by MBE.

Soft X-ray magnetic circular dichroism (SXMCD) in core-level absorption (Chen *et al.*, 1990; Koide *et al.*, 1991) and in photoemission (Baumgarten *et al.*, 1990) using circularly polarized synchrotron radiation (CPSR) has proven to be a powerful technique for studying the electronic and magnetic states of magnetic substances. This new technique, combined with the recently discovered MCD sum rules (Thole *et al.*, 1992; Carra *et al.*, 1993), allows an element-specific and separate determination of the spin and orbital magnetic moments. This remarkable advantage of SXMCD makes it especially suited for studies of multicomponent magnetic systems. Many SXMCD investigations have been performed recently on ultrathin films and superlattices (or multilayers), giving a great deal of information about the electronic and magnetic states in these two-dimensional systems (Stöhr, 1995). However, few SXMCD experiments have been reported so far which used samples with an atomically well controlled morphology and structure. Much controversy still exists concerning magnetism–structure correlation. Hence, there are increasing demands for *in situ* SXMCD experiments on samples epitaxically grown by MBE.

MBE machines are currently commercially available. However, most of them are very big, complicated and expensive. For use on experimental stations in synchrotron radiation facilities, an MBE instrument should be easy to move, install and remove. To perform *in situ* SXMCD experiments in combination with ultrahigh vacuum (UHV) high-field magneto-optical instruments or a photoemission spectroscopy (PES) chamber, we have designed and constructed a compact MBE apparatus which enables both the growth and characterization of magnetic ultrathin films and superlattices with well controlled interfaces down to the monolayer or even sub-monolayer level.

2. Design and construction

Only limited space is available on two vacuum ultraviolet–soft X-ray CPSR helical undulator beamlines (BL-28A and AR-NE1B) at the Photon Factory (Kagoshima *et al.*, 1992, 1995). This requires that cleaning and characterization of the substrate surfaces,

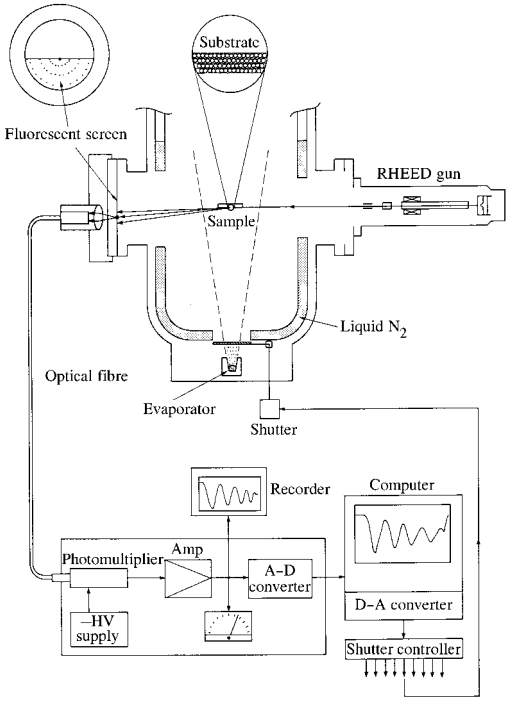

Figure 1
Sample growth is monitored in a layer-by-layer fashion using RHEED intensity oscillations. The shutters of the evaporators are controlled by a phase-locked epitaxy technique using a computer.

deposition of samples including buffers, and characterization of the samples produced should be carried out in a single UHV main chamber. Special care must be taken in designing MBE equipment to fulfil this requirement.

We first imposed the standard specifications of MBE machines:

(i) a UHV chamber with a base pressure of less than 5×10^{-11} torr and a pressure of $\sim5\times10^{-10}$ torr during evaporation is indispensable;

(ii) stable and deposition-rate controllable MBE sources are mandatory;

(iii) substrates for samples have to be exchanged without breaking the vacuum of the MBE chamber.

To satisfy requirement (i), the vacuum chamber incorporates a liquid nitrogen cryo-shroud and is evacuated by a $500\,l\,s^{-1}$ sputter ion pump, a $450\,l\,s^{-1}$ magnetically levitated turbo-molecular pump (TMP) and a titanium sublimation pump (TSP). Specification (ii) has led to the use of electron-beam guns and Knudsen cells with shutters as MBE sources. Specification (iii) requires an additional small substrate-loading chamber separated from the MBE chamber by a gate valve.

We further imposed the following specifications:

(iv) final cleaning of the substrates should be carried out in the MBE chamber;

(v) sample growth must be monitored and controlled in a layer-by-layer mode;

(vi) the absolute sample thickness must be determined during deposition;

(vii) it should be possible to grow wedge-shaped samples;

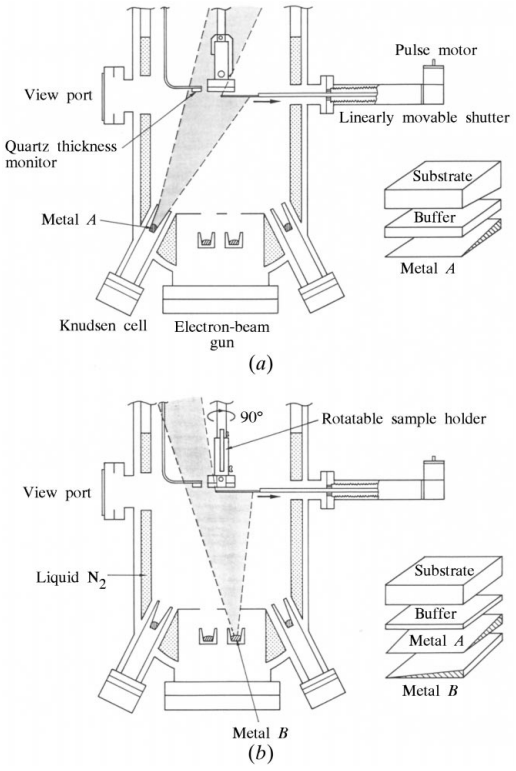

Figure 2
Expected processes for producing wedge-shaped samples using a linearly movable shutter and a quartz thickness monitor. (*a*) A wedge-shaped layer of metal *A* is produced. (*b*) The sample holder is rotated by 90° around a vertical axis. A layer of metal *B* wedge-shaped in the direction perpendicular to the wedge of the metal *A* layer is produced.

(viii) samples and substrates must be characterized *in situ* in the MBE chamber.

To fulfil requirement (iv), an Ar ion gun was attached to the MBE chamber and a sample holder was designed to allow heating of substrates up to ~1000 K. Specification (v) has led us to employ a phase-locked epitaxy technique using intensity oscillations of reflection high-energy electron diffraction (RHEED) patterns (Neave *et al.*, 1983), as shown in Fig. 1. Fluorescence emitted from RHEED spots on a fluorescent screen is collimated by a lens and then transmitted through an optical fibre to a photomutiplier. The output signal is amplified to drive an *x*–*y* recorder and is also displayed on a computer. The shutters of the evaporators are computer controlled *via* a shutter controller. As shown in Fig. 2, requirements (vi) and (vii) are satisfied by the combined use of a quartz-crystal thickness monitor and a linearly movable shutter driven by a pulse motor. Furthermore, a sample holder that can be rotated by 360° about a vertical axis makes it possible to grow layers of different metals (*A* and *B*) which are wedge-shaped in perpendicular directions. Specification (viii) has resulted in a design that allows Auger electron spectroscopy (AES) and low-energy electron diffraction (LEED) as well as surface magneto-optical Kerr-effect (SMOKE) measurements.

An MBE apparatus with side ports at three different heights was fabricated. Ports at the highest level are used for AES and LEED optics, an Ar ion gun, and view ports. Shutters protect these instruments from impinging molecular beams during evaporation. Ports at the intermediate level are equipped with a RHEED gun, a fluorescent screen for RHEED and view ports for *in situ* SMOKE experiments using an He–Ne laser. At the lowest level there are a pumping port and ports for a substrate-loading chamber and for sample transfer. The loading chamber has an O-ring-sealed window to allow rapid exchange of substrates and is

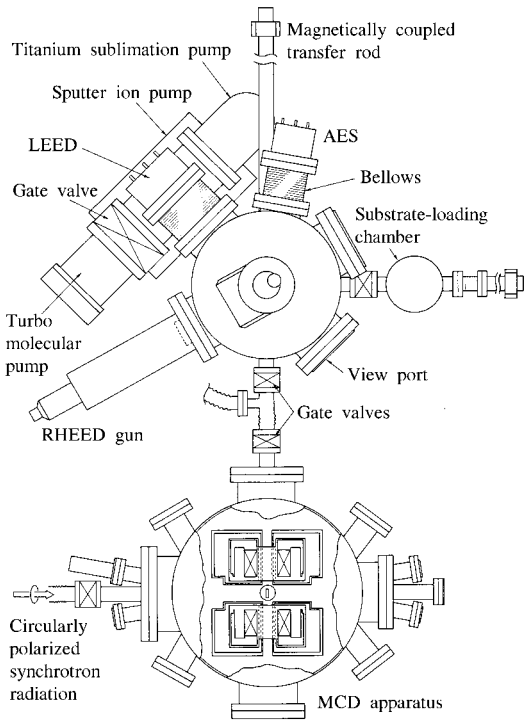

Figure 3
Plan view of an anticipated combination of the present MBE apparatus and a magneto-optical instrument for *in situ* SXMCD measurements requiring high magnetic fields.

pumped by a TMP. The sample manipulator, attached at the top, comprises a two-axis rotary-motion feedthrough, a tilt mechanism, an x–y translator (in the horizontal plane) and a z translator, allowing all the movements necessary for RHEED, AES, LEED and SMOKE measurements.

3. Performance and applications

The MBE vacuum chamber satisfied the specification of UHV. The beam spot size of the RHEED gun was found to be less than 100 μm in diameter when operated at 30 kV. This is small enough to make a RHEED observation. The sample substrate could be heated to temperatures of ~1000 K with an accuracy better than ±1 K using a heater and a temperature controller.

When low magnetic fields are sufficient for sample magnetization, the present MBE apparatus allows *in situ* SMOKE and SXMCD experiments by incorporating a small electromagnet. Fig. 3 shows a plan view of an example of anticipated *in situ* absorption SXMCD experiments requiring high magnetic fields. The MBE apparatus will be connected to a UHV superconducting magnet (Koide *et al.*, 1992) *via* gate valves. The samples prepared in the MBE chamber will be transferred under UHV to a sample holder within the magneto-optical instrument using a magnetically coupled transfer rod. This MBE equipment is intended to be combined with a UHV magnetic field modulation instrument (Shidara *et al.*, 1992) for measuring very small SXMCD. It is also possible to perform an SXMCD experiment in PES by combining the MBE apparatus with a PES chamber incorporating a pulse magnet.

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References

Baibich, M. N., Broto, J. M., Fert, A., Nguyen Van Dau, F. & Petroff, F. (1988). *Phys. Rev. Lett.* **61**, 2472–2475.
Baumgarten, L., Schneider, C. M., Petersen, H., Schafers, F. & Kirschner, J. (1990). *Phys. Rev. Lett.* **65**, 492–495.
Bland, J. A. C. & Heinrich, B. (1994). Editors. *Ultrathin Magnetic Structures I & II.* Berlin: Springer.
Carcia, P. F., Meinhaldt, A. D. & Suna, A. (1985). *Appl. Phys. Lett.* **47**, 178–180.
Carra, P., Thole, B. T., Altarelli, M. & Wang, X. (1993). *Phys. Rev. Lett.* **70**, 694–697.
Chen, C. T., Sette, F., Ma,Y. & Modesti, S. (1990). *Phys. Rev. B*, **42**, 7262–7265.
Farrow, R. F. C., Dieny, B., Donath, M., Fert, A. & Hermsmeier, B. D. (1993). Editors. *Magnetism and Structure in Systems of Reduced Dimension.* New York: Plenum Press.
Herman, M. A. & Sitter, H. (1989). *Molecular Beam Epitaxy.* Berlin: Springer.
Kagoshima, Y., Miyahara, T., Yamamoto, S., Kitamura, H., Muto, S., Park, S.-Y. & Wang, J.-D. (1995). *Rev. Sci. Instrum.* **66**, 1696–1698.
Kagoshima, Y., Muto, S., Miyahara, T., Koide, T., Yamamoto, S. & Kitamura, H. (1992). *Rev. Sci. Instrum.* **63**, 1289–1292.
Koide, T., Shidara, T. & Fukutani, H. (1992). *Rev. Sci. Instrum.* **63**, 1462–1465.
Koide, T., Shidara, T., Fukutani, H., Yamaguchi, K., Fujimori, A. & Kimura, S. (1991). *Phys. Rev. B*, **44**, 4697–4700.
Neave, J. H., Joyce, B. A., Dobson, P. J. & Norton, N. (1983). *Appl. Phys. A*, **31**, 1–8.
Parkin, S. S. P., More, N. & Roche, K. P. (1990). *Phys. Rev. Lett.* **64**, 2304–2307.
Shidara, T., Koide, T. & Fukutani, H. (1992). *Rev. Sci. Instrum.* **63**, 1501–1504.
Stöhr, J. (1995). *J. Electron Spectrosc. Relat. Phenom.* **75**, 253–272, and references therein.
Thole, B. T., Carra, P., Sette, F. & van der Laan, G. (1992). *Phys. Rev. Lett.* **68**, 1943–1946.