Design, construction, and performance of a low-cost deposition system for thin film multilayers

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The design, development, and performance of a low-cost deposition system for thin film multilayers are presented. The aim for the construction of the apparatus was to prepare multilayer films of variable composition with a single layer thickness ranging from a few to hundreds of angstroms. To ensure a high flexibility of the film stoichiometries up to five different elements can be deposited independently onto the substrate via electron beam sources or Knudsen cells. The design of the transfer system has been kept very simple although it has a secure load–unload system to provide reliable transport from the antechamber to the deposition chamber and vice versa. The films can be deposited simultaneously onto four substrate platelets. The deposition process is controlled by a computer and all process-parameters are monitored. The shutters of the evaporation cells are activated by a computer providing reproducible deposition processes. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1406918]

Many materials with interesting optical, electronic, and magnetic properties needed for various technological applications are synthesized by classical high temperature bulk methods. There are some disadvantages of these methods such as the high temperatures necessary to enhance the diffusion of the reactants, the long reaction times required to obtain homogeneous products, and the lack of control of the reaction. In several contributions it was claimed that solid state chemistry at such high temperatures is still an art and not science.[1,2]

However, additional properties can be tailored by reducing the initial diffusion length of the reactants by depositing them in alternating thin films on a nanometer scale. This reduces massively the time scale of complete intermixing or allows diffusion at lower temperatures.

In some cases this low temperature avoids a premature nucleation of ''bulk-type'' thermodynamic stable compounds and enables the formation of new kinetically stabilized compounds. During the last few years the group of D.C. Johnson investigated several thin film couples and their work revealed some highly interesting aspects of the reactivity and properties of such multifilms. Some selected examples of the large number of reports are given in Refs. 3–7.

The aim of our efforts was the design and implementation of a small and economical deposition chamber that allows one to deposit thin multilayer films of variable composition onto different substrates. The preparation and characterization of thin film compounds with different magnetic properties are the center of our interests. It is well documented that chromium chalcogenides show a wide variety of structural, electrical, and magnetic properties. The structures of chromium tellurides in the composition range CrTe to Cr_5Te_8 are strongly related and they are often metals undergoing a transition into the ferromagnetic state at relatively high temperatures.[8–15] Hence we started our experiments with Cr–Te multilayers to study the physical properties of such thin films. In the present contribution we describe the low-cost deposition system that is used to prepare thin film multilayers.

For the design and construction of a deposition apparatus several requirements were formulated.

(1) Several small or one large substrate should be applicable for the deposition process to get either identical samples for different, partly destructive measurements or to obtain one big sample to maximize film mass for differential scanning calorimetry (DSC) measurements.
(2) A substrate rotation and suitable evaporator cells should provide homogeneous layer quality during the deposition process.
(3) The substrate temperature should be variable to enable the investigation of the influence of deposition temperature onto layer growth.
(4) The deposition should be performed at a vacuum better than 10^{-7} mbar to minimize the level of contamination.
(5) It should be possible to deposit different materials simultaneously (codeposition) or in variable sequences (multilayer).
(6) The deposition rates have to be monitored during the deposition process.
(7) An antechamber is required to save evacuation time after a sample replacement, as the antechamber is much smaller than the deposition chamber. Such an arrangement protects the evaporation materials against contamination by the permanent vacuum in the deposition chamber.
(8) The antechamber should be able to be equipped with several sample holders to make an evacuation necessary only after a combined exchange of sample holders.
(9) Spare flanges should be accessible for the attachment of further equipment.
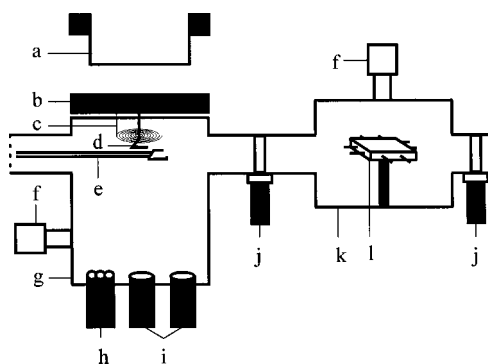
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FIG. 1. Sketch of the deposition system. a: cooling flange (optional), b: heating flange, c: filament heating, d: rotatable hook, e: manipulator, f: turbomolecular pump, g: deposition chamber, h: electron beam source, i: Knudsen cell, j: gate valve, k: antechamber, and l: carousel.

(10) To enable characterization of the films with surface sensitive methods the antechamber should be attachable to a glove box or a movable vacuum chamber.

The substrate holders are suited for carrying up to four substrates with sizes ranging from 10×10 to $25 \times 25 \, mm^2$ to get identical samples during the same deposition run. The deposition chamber is pumped by a 220 l/s Pfeiffer turbomolecular pump which provides a base pressure in the low 10^{-9} mbar range. The independent pumped antechamber (50 l/s Edwards turbomolecular pump) ensures that the vacuum within the deposition system is preserved when the samples are removed from the deposition chamber. Additionally, the deposition chamber is equipped with a 27 cm linear and
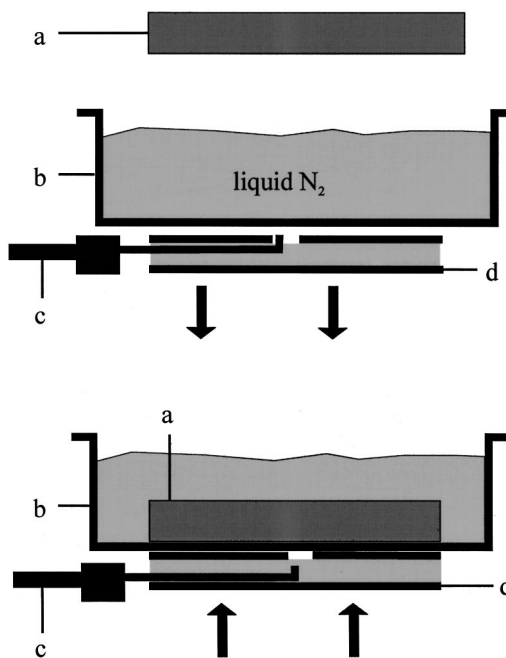

(a)

(b)

FIG. 2. The manipulators load–unload mechanism at the heating flange (a) left: side view and right: top view of the substrate holder locked at the manipulator; (b) left: side view and right: top view of the substrate holder locked at the hook; a: manipulator, b: manipulator finger, c: hook, and d: hollow tube.

FIG. 3. The load–unload procedure at the cooling flange. Top: side view of the substrate holder locked at the manipulator; bottom: side view of the substrate holder locked at the cooling flange; a: magnet, b: cooling flange, c: manipulator, and d: substrate holder.

rotary motion actuator based on a magnetic coupling system designed by the group of Kipp *et al.* (Institute for Experimentalphysik, University of Kiel).

Two different flanges carrying the substrate holder during the evaporation were manufactured. The first flange that is denoted as "heating flange" enables rotating and simultaneous heating of the substrate in the range from 300 to 700 K during deposition. The second flange which is the "cooling flange" allows one to cool down the substrate to 100 K during deposition (see Fig. 1).

In Fig. 2 the load and unload procedure of the heating flange is displayed. The substrate holder is equipped with two small tubes into which the two fingers attached to the manipulator fit. In the middle of the right tube a small notch is located. At the end of the right finger of the manipulator a mounted bulge hooks into the notch after the manipulator is rotated anticlockwise. After this operation the substrate holder is attached to the manipulator and can now be transported to the hook of the heating flange by pulling back the manipulator. A clockwise rotation of the manipulator detaches the substrate holder. To allow a free rotation of the substrate holder the manipulator must be completely pulled out so the substrate is in the correct position for the deposition of the films.

When the evaporation is finished the following manipulations guarantee a secure removal of the substrate holder. To facilitate the attachment of the fingers of the manipulator to the tubes the right finger is a little bit longer than the left finger. First the longer finger is moved a small distance into the tube to prevent rotation of the substrate holder. Afterwards the clockwise rotation, the hook of the heating flange fixes the substrate holder thus preventing the holder from being pushed off when the manipulator fingers are moved to
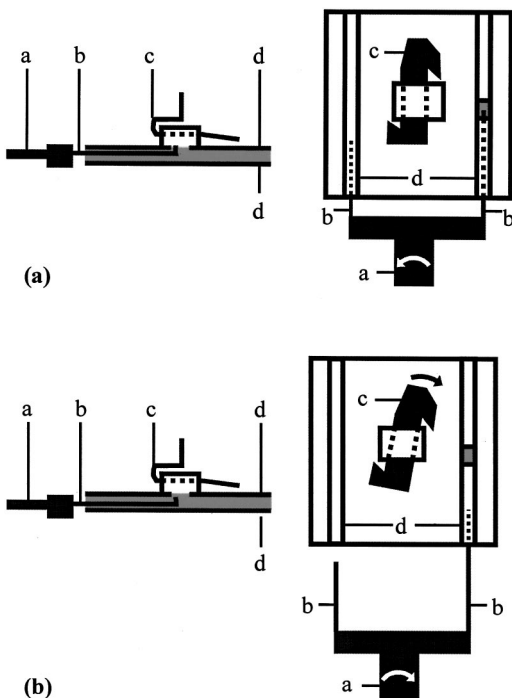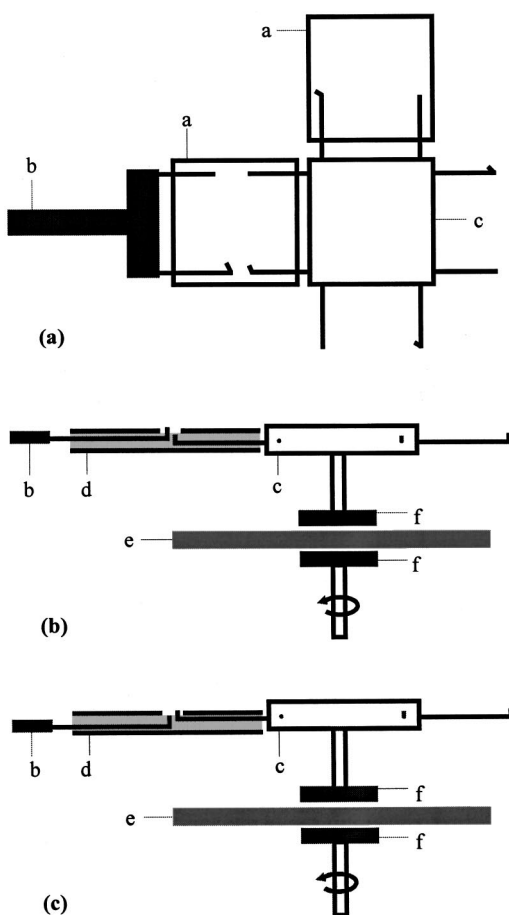
FIG. 4. Sample carousel of the antechamber: (a) top view; (b) sample holder locked at the manipulator; and (c) sample holder locked at the carousel; a: substrate holder, b: manipulator, c: carousel, d: hollow tube of substrate holder, e: bottom flange of the antechamber, and f: magnet.

the final position. After this procedure an anticlockwise rotation of the hook releases the substrate holder which can now be transported into the antechamber with the manipulator.

In Fig. 3 the load and unload procedure is shown when the cooling flange is attached to the deposition chamber. The substrate holder being attached to the manipulator is positioned below the cooling flange. A magnet is put into the cooling flange that is filled with, e.g., liquid nitrogen. The magnet adheres the substrate holder at the bottom of the cooling flange and guarantees a very good cooling of the substrate. After the deposition process is finished the magnet is removed thus detaching the substrate holder from the cooling flange.

Figure 4 shows the sample carousel that can carry four substrate holders within the antechamber. After the deposition the substrate holder is transferred to the carousel by rotating an empty position on the carousel towards the manipulator. The manipulator is pushed forward until the fingers of the carousel snap into the tubes of the substrate holder. Mechanical inexactness occurring during this procedure can be balanced by a slight twisting movement of the manipulator.

After the substrate holder is attached to the fingers of the carousel the substrate holder is unfixed from the manipulator

by a clockwise rotation of the manipulator. A rotation of the carousel allows that another substrate holder can be picked up with the manipulator. An anticlockwise rotation detaches the substrate holder from the carousel and can be now transferred to the position for film deposition. The whole transfer system was constructed using commercial materials. All rotational movements are realized via magnetic coupling mechanisms. A few hundred operations have demonstrated that the system is highly reliable.

The preparation of multilayers requires the opening and closing of the shutters of the evaporation cells to produce the required layer thickness. Chronological precise deposition sequences with the recording of all available process parameters are essential for a constant layer quality. Exclusive manual control of this process tends to become a time-consuming job with erratic performance. This consideration led to the decision to automate the deposition by a computer program.

The program is written completely under Delphi 4.0 (Borland) and requires a personal computer with at least 133 MHz, 8 MB RAM, a display resolution of 640×480 pixels, and Microsoft Windows 95. Recording and graphic display of process parameters with tuneable time resolution is possible. Any deposition data can be entered via simple commands like "open chromium," "close chromium," and so on. The deposition duration can be either limited by time or by the quantity of deposited material. Additionally, the usage of deposition sequence loops for the preparation of periodical multilayers is also supported.

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