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Original

Sequential microcontroller-based control for a chemical vapor deposition process

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Abstract

A cost-effective direct liquid injection system is developed for a chemical vapor deposition process using a microcontroller. The precursor gas phase is controlled by the precise sequential injection of a liquid precursor solution to a vaporizing chamber prior deposition. The electronic control system allows the human–machine interface through a LCD display and a keypad matrix. The core of the electronic system is based on an electro mechanical injector operated in time and frequency as a sequential control system by a popular PIC16F877A chip. The software has been developed in the BASIC language and it can be easily modified through an ICSP programmer for different sequential automatized routines. The injection calibration test has proven the linearity of the injection control system for different operation parameters. The results reported the sequential injection MOCVD deposition of alumina thin film.

Keywords: Microcontroller; Chemical vapor deposition; Direct liquid injection; Thin films

1. Introduction

Metal organic chemical vapor deposition (MOCVD) is an important technique for the synthesis of materials in the form of coatings and thin films for the electronics and semiconductor industry. This process, allows depositing solid materials in the form of thin films and powder from a metal-organic precursor by mean of a chemical reaction on a thermally activated surface.

Usually, metal organic precursors do not have enough vapor pressure for being effectively transported to a thermally activated surface; when the precursor reaches the sample, it decomposes to produce a desired material in the form of thin film. In order to increase the vapor pressure, precursors must be heated to increase their volatility and transported by means of a carrier gas. The heating and cooling process of the precursor in sublimation and bubbler technologies have some drawbacks. Precursors can partially decompose after thermal cycles, reducing the reproducibility of the thin film deposition process (Sovar Samélor, Gleizes, & Vahlas, 2007). The gas phase concentration is variable in time due to its difficult control, and this is because temperature and mass flow variables are coupled in a same device. Moreover, the evaporation rate also fluctuates in time; it has a strong dependence on the remaining amount of precursor in the container.

Direct liquid injection arises as an important technique which can reduce intrinsic drawbacks of conventional precursor delivery methods. In this sense, if a constant vapor precursor concentration can be reached, conformal smooth thin films can be produced with this method; the process can be transferred to academic and research environments to satisfy industrial applications. As the precise control of the main variables for the MOCVD process is a key factor in order to obtain high experimental reproducibility, precise control of the precursor

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flow becomes one of the more relevant issues which must be treated efficiently; in this way, direct liquid injection MOCVD has proved to solve this problem showing good results to deposit some oxides (Mungkalasiri et al., 2009; Na, Kim, Yong, & Rhee, 2002).

The direct liquid injection technique allows reducing the drawbacks related with the bubbler and sublimation-based technologies. With this technology, a micro amount of the precursor solution is atomized and heated in a vaporizing chamber in order to be immediately released to the reactive zone where the deposition takes place. The precursor solution is pressurized under an inert atmosphere in order to avoid contact with the air and moisture which could lead to a premature reaction and aging, moreover the precursor solution is maintained all the time at room temperature just an instant before being atomized and released. In this manner, there is not enough time for the precursor to present chemical changes. The micro amount is controlled electronically by the opening time and frequency of the injector, by this way is possible to maintain a more stable and constant precursor vapor flow into the reactor chamber, with these characteristics, is possible to increase the reproducibility of the experiments and operate the system in a safer and convenient way.

The atomic layer deposition processes has also already taken an advantage of this delivery precursor method producing high quality thin films (Leskelä & Ritala, 2002). A precise amount of precursor is injected sequentially for each cycle; the growth is constant and complex structures can be obtained (George, 2010). In this context, the microcontrollers are useful electronic devices which can be adapted for chemical laboratory facilities for monitoring and control of dedicated processes. The low cost and high availability of such devices allows the development of electro mechanical systems for analytical instrumentation in academic and research environments (Kubínová & Šlégr, 2015; Rajendran & Neelamegam, 2004; Ramana & Malakondaiah, 2007; Urban, 2014, 2015).

The present work is dedicated to the development of a low cost sequential microcontroller-based direct liquid injection control system based on a PIC16F877a microcontroller. This system can be used in a direct liquid injection MOCVD reactor to produce coatings, thin films and nanostructures. The microcontroller and the electro mechanical injector are coupled through a power electronic stage. The microcontroller control contributes to the improvement in the repeatability accuracy of the thin film deposition process; moreover, the microcontroller interface via a matrix keyboard and LCD allows changing the injection parameters in real time through automated sequential routines.

2. Design scheme

The core of the injection control system is a Pic16F877a microcontroller. The design scheme is shown in Figure 1. This microcontroller has been selected because of its low cost, high availability and friendly user programming tools. This CMOS FLASH-based 8-bit microcontroller has 5 ports with embedded peripherals such as ADC, timers, USART and longtime data retention around 40 years. These functionalities make of this microcontroller an attractive device for this application.

Block B contains the user interface to the data input to the controller by means of a keypad which allows selecting the time and frequency desired to the operational parameters of the injector. The selection of an operation routine can be modified at any time during the processing if desired. If it is desired to obtain a constant gas phase feeding to the reactor, the routine must be kept constant, which makes it possible to obtain conformal smooth thin films. If it is desired to modify the reaction ration rate through a gas phase modification, the routine can be modified, in fact automated routines can be programmed to obtain different growing rates in a reproducible way. Several injectors can be combined with a wide spectrum of injection parameters to obtain new complex materials with unique electrical, chemical, and mechanical functionalities. For this reason, the time-on and frequency of operation parameters are crucial for this process. In this sense, with this control system the gas phase can be precisely controlled and each combination can modify the gas phase through the reaction chamber and modify velocity and concentration profiles to finally modify the reaction rate to deposit from single to complex coatings and thin films. The block D contains a power electronic stage which transfers the control signal processed from the microcontroller to the injector. The core of this stage is an electro mechanical injector. For the correct atomization of solutions, the injector must be kept at 40 psi. The control signal is transferred to the injector through

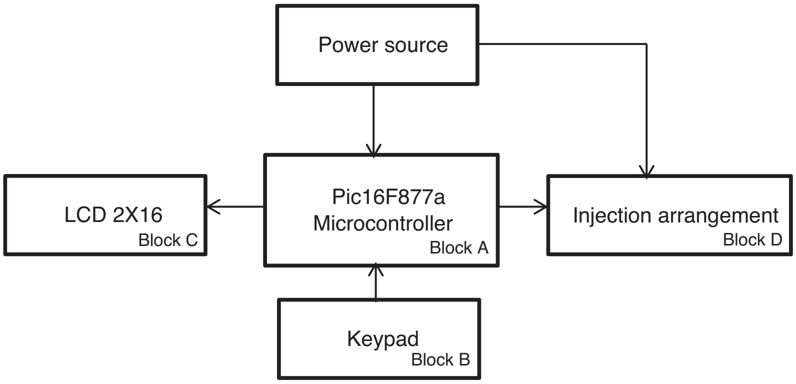

Figure 1. Block diagram of microcontroller based injection control.