2021-2022 Activity Report

KE500EI Kenosistec electron-beam evaporator and Amira-MBraun solutions glove box with vacuum deposition system

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Laboratory web page: https://cisup.unipi.it/labs/kenosistec-e-beam-evaporator-ke500ei/

Introduction

The Deposition and Microfab Facility of CISUP is an integrated laboratory built around two major pieces of equipment for the deposition of thin films made of different classes of materials (oxides, metals, and organic compounds) on whatever type of substrate. The current technologies for thin-film deposition allow a surface or a sample to be covered by one or more layers of a desired material, with typical thickness values ranging from a few nanometers to about 100 micrometers. Crucial in semiconductor, photovoltaic, and optoelectronic industries, being capable of depositing a material with high degree of control on its thickness and stoichiometry is also important for many other scientific fields of modern science. The CISUP Facility comprises both thermal and electron/ion beam assisted deposition methods, both belonging to the class of physical vapor deposition (PVD) technologies. Briefly, a substrate is covered by the material delivered by a target within a high-vacuum chamber. Methods developed in the laboratory include both electron-beam and thermal deposition technologies.

This report aims to inform the UniPi community about the activities carried out by the integrated CISUP Deposition Facility during 2021 and 2022 (up to Oct. 2022). Sections will briefly describe the performed work that included: i) huge effort made to set the laboratory and launch its implementation; this also includes access modes, technical services, safety development, etc.; ii) worked hours/user statistics; a very large part of worked hs was addressed to educational purposes, with other slots being employed to set-up processes, initial research tasks, and joint collaboration agreements with public bodies; iii) products as related to the three 'missions' of the University: (a) research, (b) education, and (c) outreach, including technology transfer. Significant issues during the lab launch stage included the Covid-19 pandemic, that delayed initial R&D tasks, as well as the preliminary instrumental set-up. The here presented activity, indeed, is not meant as a single-instrument effort, but as an integrated facility which led to the development of an entirely new, specific laboratory environment. Laboratory utilities, including nitrogen, oxygen, argon, as well as compressed air, had to be set and optimized (a few improvements on gas distribution lines are still in progress at the time of the present report, with specific funds achieved by the Department of Physics); the operation of the integrated environment had to be started; entirely new evaporation processes had to be implemented on the equipment (as general guideline, each new material generally takes several weeks of preliminary experiments in order to set all the different process parameters). Overall, the impact of the Facility went beyond UniPi, activating several collaborations and paid contracts in the framework of scientific collaborations (especially with CNR and INFN). It also allowed to submit research projects for developing new materials and devices (especially photonic devices) based on the facility. Some of these proposals are under evaluation by funding agencies at the time of preparation of the present report.

In agreement with the CISUP guidelines, a complete price list was published on the CISUP website with the instructions for quote requests. However, we would like to point out also a different model for the development of complex and relevant instrumental facilities where processes, characterization techniques, and manufacturing methods are not immediately accessed by most of users (since these process and techniques are state-of-art, very complex, and all required specific optimization steps for each single material). This model involves custom-based, highly personalized approaches to the specific requirements of users, scientific partners, and companies, that can be much better achieved in the form of **-externally funded- scientific collaborations** in which the second partner pays in order to sustain part or all the costs of a given R&D activity. Advantages of this model include sharing intellectual property and setting an effective, iterative synergy with other partners finally leading to achieve desired materials and devices in a joint fashion.

Key performance indicators

Overall, the laboratory during its first two years of operation:

- mobilized the work of **21 people (researchers, technicians, students)**;
- led to enhanced research capacity, favoring the preparation and submission of at least **5** research proposals (EU Pathfinder calls, MUR PRIN projects, PNRR projects);
- was used in the work of **2 MS and 3 PhD thesis**;
- led to enhanced interaction with **two main public Research Bodies** (with **external funds** delivered to the lab)
- attracted a significant **private donation**;
- secured a gross income of about **55 kEUR**, plus a **13,2 k€** contribution for running costs from Dep.t of Physics users;

- worked for a total estimated of **3500 hs;**
- led to **1 main scientific paper** on photonics (currently in preparation, Tredicucci group); enhanced research collaborations in this framework included the Universidad Autónoma de Madrid and the University of Milan Bicocca.

Laboratory implementation: main CISUP installed equipment and set utilities:

1. Kenosistem KE500EI

This is a complex deposition system with twin electron source and ion source, suitable for co-deposition processes of, among others:

(a) dielectrics: silica, titania, alumina, hafnia, tantala, zirconia, etc.;

(b) semiconductors: silicon, germanium;

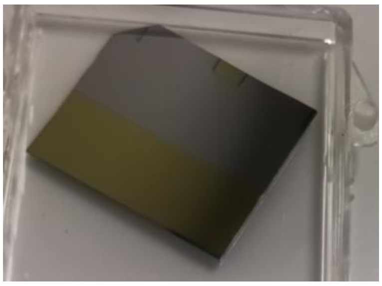
(c) metals: aluminum, nickel, molybdenum, platinum, gold, titanium, silver.

The KE500EI equipment is a single batch system mainly designed for R&D applications that require versatility and facility of use. The process chamber is built with AISI304 stainless steel, and has a height larger than 0.9 m, which is at the base of highly uniform sample coverage. The chamber is provided with an internal shield, easily removable for effective cleaning. The chamber mounts several spare flanges for additional services or for possible future implementations, and it is provided with a water cooling line. The substrate holder is designed to carry up 1 wafer of 8”. Smaller substrates or irregular pieces of substrate can be placed on the substrate holder, enabling high versatility in terms of usable samples and targeted applications. The system carries one twin-electron beam gun source with 10 kW and 6 kW emitters, respectively, with six crucibles, direct water cooled brazed version with 20 cc capacity, and an extra ion source for sample cleaning and assisted-deposition. Quartz lamps are used to support deposition processes up to 800°C. A powerful primary pump and turbo pump allow one to reach quickly the required high degree of vacuum, and a load lock provides better cleanliness of the process chamber, hence cleaner and better-controlled deposition and faster cycle time. The whole system is under control of PLC and a PC is used for the management of human-system communication.

Materials and devices deposited by electron-beam evaporation, with multi-process optimization during the first two years of operation were:

- **Silica**
- **Titania**
- **Titanium**
- **Silica-titania multilayers for Distributed Bragg Reflectors (to be used in optical microcavities)**
- **Tantala (currently in progress, see figure below)**

This constitutes the starting point of a **portfolio of e-beam deposited materials**, that once optimized can be available to all the UniPi community.


Sample of tantala deposited on silicon.

2. MBraun modular glove-box with integrated thermal deposition system

This equipment is used for processes under controlled, nitrogen atmosphere. Two glove-boxes are equipped with 4+3 overall gloves, polycarbonate windows (10 mm thickness), leak rate below 0.05 vol%/h, feedthrough connections (for fiber optics, electrical connections etc.), decontamination chambers, system for gas purification and removal of H2O and O2 down to < 1 ppm. Sensors are present to monitor residual oxygen and humidity, and a thermal evaporator is embedded in the system with a proprietary vacuum system. The system is suitable for the deposition of both metals and organic molecules (under low temperature conditions to preserve molecular function). The precision in terms of control of the deposition rate is of 0.01 Angstrom/second. Our outlook is to set-up an integrated platform where both deposition of molecules and optical/electrical characterization may be performed under controlled atmosphere and in a continuous way, i.e. with no exposure to ambient conditions.

Materials and devices deposited by thermal evaporation, with multi-process optimization during the first two years of operation were:

- **Gold**
- **Silver**
- **Chromium**

3. Installed Utilities and Services for the lab

3a. Technical gases with specific distribution lines have been installed. Operating gases include nitrogen, oxygen, and argon. A safety box has also been installed to contain gas containers, with lines connecting the internal and outer regions of the box, and specialized equipment for pressure control. Uninterrupted power supplies (UPS) were installed in the lab, to guarantee continuous and stable powering, especially for continuously running equipment (glove-boxes).

3b. Fume hoods with internal filtration. This was an effective solution to give the laboratory a proper point for chemical manipulation without need for complex construction works.

3c. Advanced commissioning of installed equipment was also carried out, mainly during 2021. Extensive efforts and collaborative interactions with involved companies were put in place, leading to full equipment operation. Consumables for commissioning, educational purposes, and all the initial tests on the equipment were provided by users from the Department of Physics (13,2 k€).

3d. Chemical boxes were installed for storage of chemical products in safety conditions.

3e. One unit of technical personnel of the Department of Physics (Nicola Puccini) was trained for autonomous operation on the equipment. Overall, seven technicians were involved in the lab development: Gabriele Paoli (CISUP), Nicola Puccini, Enrico Andreoni, Francesco Francesconi, Paolo Marsili, Marco Bianucci, Enrico Maccioni (Department of Physics). Ilaria Grassini (Dept. of Physics) was also involved as in charge of chemical databases in the Department of Physics.

3f. The laboratory was equipped with a precise routine for the access of new users, for recording rules and changes related to safety management, and for training procedures. **All these materials and documents** are available and **can be shared with other CISUP labs if needed**, in view of setting common, highly qualified CISUP practices and rules to manage complex instrumental facilities.

All the points involving safety were developed in tight collaboration with the Safety&Environment Offices of UniPi (Sabrina Arras), and with support of the UniPi technical offices (Guido Morucci, Simona Burchi, Filippo Scalsini, Maria Luisa Cialdella). These utilities and processes were mainly implemented in early 2021. Training activities were extended in 2022. Autonomous users included Andrea Camposeo (glove-box) and Luana Persano (glove-box; e-beam evaporation), both research directors at CNR-Istituto Nanoscienze. Further improvements (mounting of additional gas lines) are currently in progress.

Education, outreach and technology transfer

The laboratory was used and is being intensively used for the following thesis work:

- Alessio Cargioli, MS in Physics: Optical Microcavities Based on Organic Photoswitchable Systems in the Strong Coupling Regime
- Stefano Casimirri, MS in Physics (in progress). On complex microcavities for photonics and energy.
- Annachiara Albanese, PhD student (in progress, cycle 36). On organic photonics.
- Lorenzo Lavista, PhD student (in progress, cycle 37). On complex microcavities and three-dimensional intelligent systems.
- Giorgia Silvestrelli, PON PhD student (in progress, cycle 37). On new deposited materials and deposition processes for sustainability.

Outreach: on-site lab visits were organized in the framework of several institutional collaborations. Visitors included the directors of local CNR Institutes (INO, IPCF), the director of the Chemical Sciences department of CNR, and the director of the European Gravitational Observer (EGO), as well as representatives of various companies. The laboratory was also involved in outreach initiatives for the 2nd-year students in Physics (October 7, 2022).

Technology transfer: technology transfer and scientific collaboration agreements were achieved with CNR and INFN.

Statistics

Due to the large machine time devoted to initial process optimization and relevant maintenance time needed in the launching stage, it was not possible to establish a fully functional recording method. Indeed, differently from hs related to external users, not all UniPi hs were productive yet, namely not all of them led to material deposition/optimization. Below we report estimated working hs and related statistics. A new recording method with individual instrument time-sheets will be implemented in the forthcoming stage of the laboratory development, when a fully operating regime is expected to be reached. An electronic time-sheet platform shared with autonomous users is currently being designed.

Glove boxes and the here embedded evaporator are continuously running during the year; this leads to an estimated number of hours of 1400/years for glove boxes (2450 hs for 21 months). These were largely employed for students (under-nitrogen processes and sample storage, and thermal deposition processes of metals). About 60 hs (corresponding to 10k€) were employed for a R&D agreement with CNR. The electron-beam deposition is on average active for 600 hs/year (1050 hs for 21 months. Note: maintenance time is largely out of this amount). About 200 hs were used for R&D with the Department of Physics and INFN.

User type	**hs (MBraun)**	**hs (Kenosistec)**	**overall %**
UNIPI (research)	-	400	11
UNIPI education (MS and PhD students)	2390	450	81
External/joint R&D agreements	60	200	8
Total	2450	1050	100