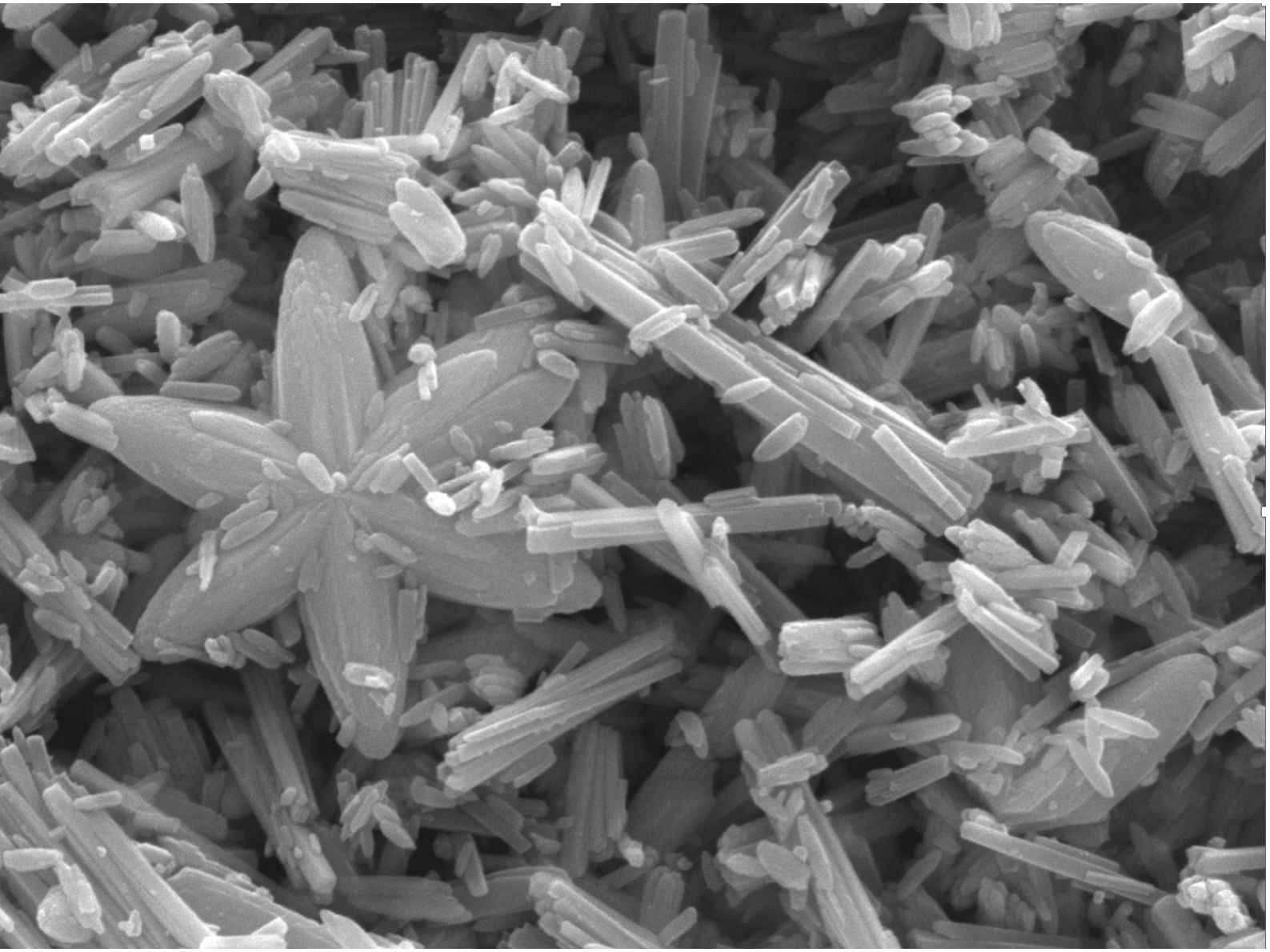
CISUP ESEM-FEG Laboratory
2024 Activity Report

Luigi Folco, Randa Ishak, Gabriele Paoli

Laboratory web-page: https://cisup.unipi.it/labs/FEI-Quanta-450-ESEM-FEG



Cover image. Metal hydroxide: ellipsoidal, and flower-like structures. Secondary electron image (field of view: 7 µm).
Courtesy of Maria-Beatrice Coltelli and Randa Anis Ishak Nakhla (DICI-UniPi

CISUP ESEM-FEG laboratory: 2024 activity report

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Abstract – This report serves to inform the UniPi community on the activities carried out by the CISUP ESEM-FEG FEI QUANTA 450 laboratory in 2024. Activities include: i) laboratory implementation; ii) hours worked; iv) research products; vi) education; vii) issues and objectives. During the year 2024, the laboratory worked for over 714 hours for testing, research and education, providing support to many national and international research projects (n = 19), documented by many scientific papers published in international journal (n = 37). Remarkable is the number of MSc, PhD students and trainees who benefited of the lab support for their thesis (n= 42), as well as of short courses conducted withing BSc, MSc and School of Doctorate programmes (n = 11).

1. Laboratory implementation

We have acquired the C-thread and sputter coater Leica EM ACE 200. It produces homogenous and conductive metal or carbon coatings for SEM, EPMA and TEM analysis, operating at 10^{-3} mbar (Figure 1). It is installed in the Microprep lab at DST-UniPi and serves three CISUP laboratories: ZEISS Crossbeam 550 FIB-SEM, the High-Resolution Field Emission Gun Transmission Electron Microscope (HR FEG-TEM) 200kV, and the ESEM-FEG FEI Quanta 450 laboratories.

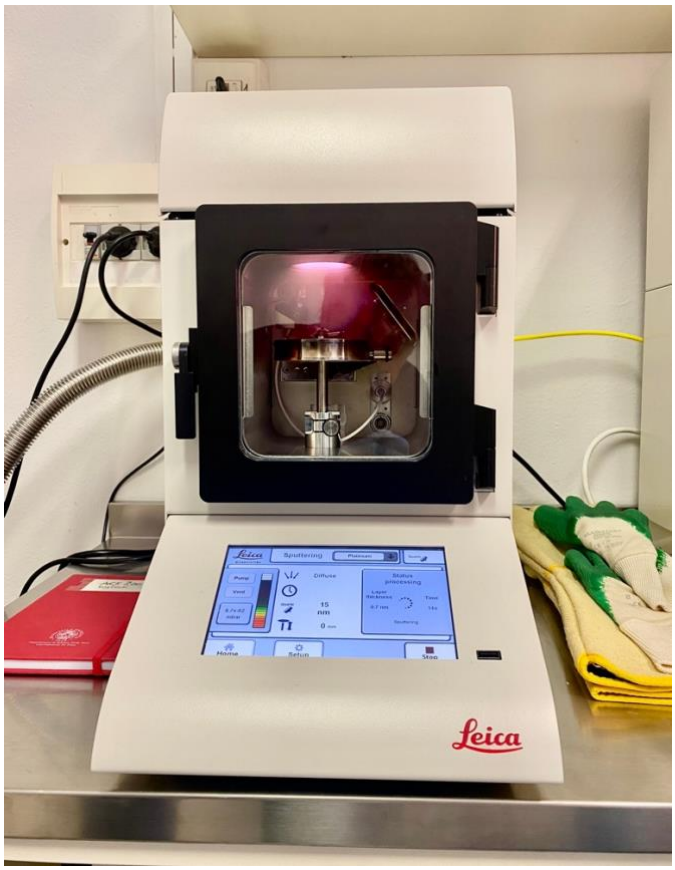

Figure 1. The C-thread and sputter coater Leica EM ACE 200 operating at the Microprep laboratory, DST-UniPi

2. Hours worked

The number of hours worked by the ESEM-FEG FEI QUANTA 450 laboratory in 2024 is 714. The machine has been used mainly for service (512 hrs), testing (174 hrs) and teaching activities (28 hrs).

3. Users

In 2024, the users of the ESEM-FEG FEI QUANTA 450 laboratory include internal (UniPi) researchers, PhD and MSc students from the DII, DF, DST, DRT-NTMC, DICI, DCCI, INFN-Pisa, but also researchers from other Universities and Research institutes, e.g., Open University (UK), Museo Nazionale dell'Antartide, Università degli Studi di Milano-Bicocca, California Institute of Technology (USA), Université de Lille (France), Hassan II Université, Casablanca (Morocco). We record collaborations with external parties, including KMe Italy Spa, Linari Nanotech. Few nice images, courtesy of material and life science researchers, are shown below (Figures 1, 2, and 3).

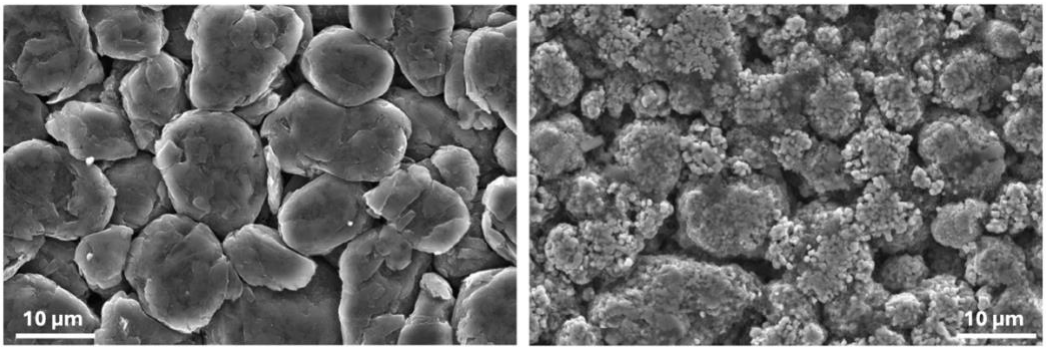

Figure 1. Secondary electron images of commercial lithium-battery electrodes. Left panel: Negative electrode with micrometric graphite particles. Right panel: Positive electrode with secondary particles of active material connected by an electro-conductive binder. Courtesy of Monica Puccini, Federica Barontini and Antonio Bertei (DICI-UniPi).

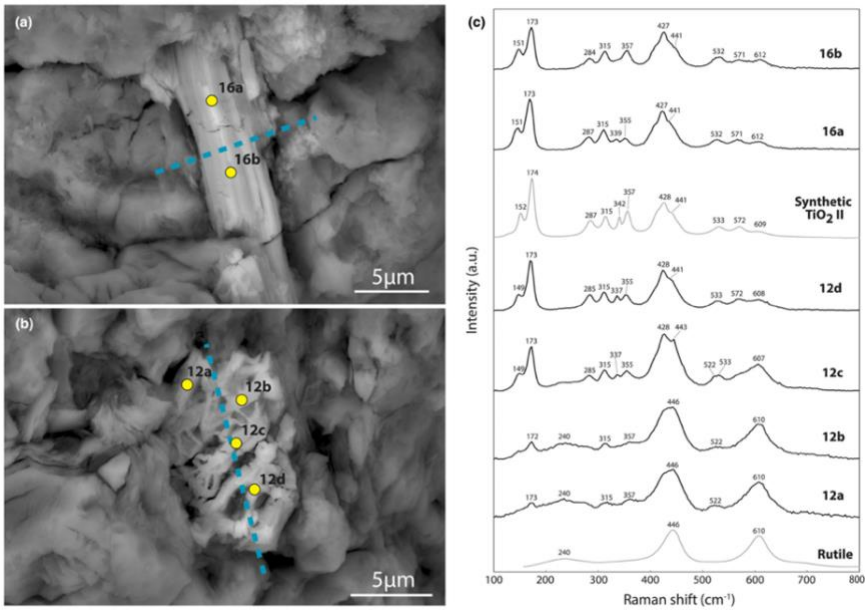

Figure 2. Copy of Figure 1 of the paper by Campanale et al. (2024), currently at Università degli Studi di Milano-Bicocca. The left panels are BSE images of shocked Ti-oxided grains in microscopic impact ejecta from the Australasian microtektite layer which were taken at the CISUP ESEM-FEG FEI QUANTA 450 labopratory. These images were instrumental for devising strategies for subsequent Raman analysis. The work provide new clues in understanding the high pressure (12 – 15 GPa) rutile to TiO2 II polymorphism (Campanale et al. 2024, MAPS, https://doi.org/10.1111/maps.14137. Courtesy of Fabrizio Campanale (formerly @DST-UniPi).

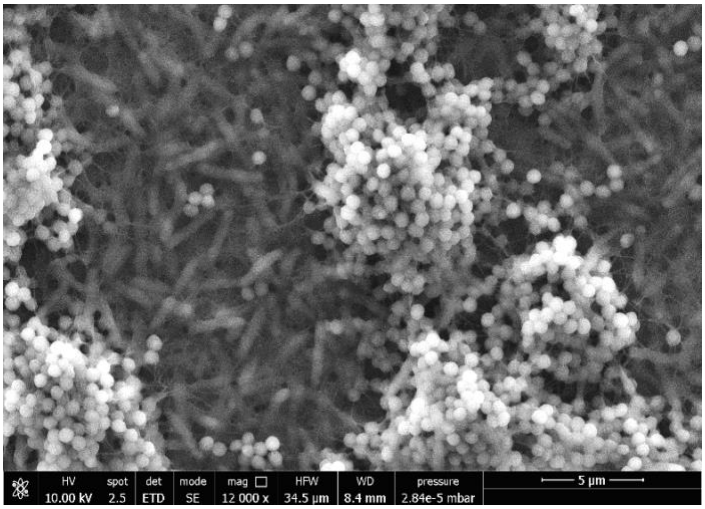

Figure 3. Secondary electron image of a mixed species biofilm (*Pseudomonas aeruginosa* + *Staphylococcus aureus*) formed on collagen scaffold, mimicking an infected wound. Kaya et al., Int J Mol Sci. 2024 25(4):2087. doi: 10.3390/ijms25042087. Courtesy of Giovanna Batoni (DRT-NTMC-UniPi)

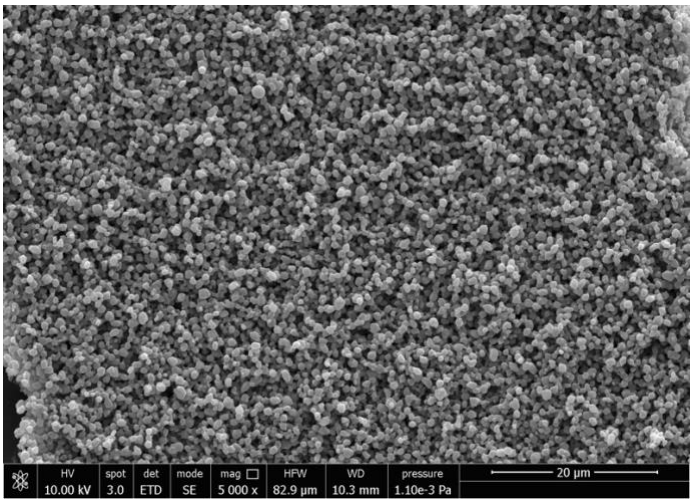

Figure 4. Secondary electron image of Poly(lactic-co-glycolic) acid (PLGA) nanoparticles produced through electrospray. Courtesy of Bahareh Azimi and Serena Danti, DICI.

4. Research products

In 2024, the ESEM-FEG FEI QUANTA 450 laboratory has provided analytical support to several research groups, PhD and MSc students involved in material analyses and nanofabrication. Many papers acknowledging the lab have been published, including one with a dedicated journal cover (Figure 5); several (not listed) are under preparation. Those that have been published and are available on-line are:

Albano, G., Portus, L., Martinelli, E., Pescitelli, G., Di Bari, L., Impact of Temperature on the Chiroptical Properties of Thin Films of Chiral Thiophene-based Oligomers, ChemPlusChem, 2024, 89(6),e202300667 (https://chemistry-europe.onlinelibrary.wiley.com/doi/10.1002/cplu.202300667)

Azimi B., Rasti A., Fusco A., Macchi T., Ricci C., Hosseinifard A., Guazzelli L., Donnarumma G., Bagherzadeh R., Latifi M., Roy I., Danti S., Lazzeri A. (2024) Bacterial cellulose electrospun fiber mesh coated with chitin nanofibrils for eardrum repair. Tissue Engineering - Part A. 30(7-8), 340–356. DOI: 10.1089/ten.TEA.2023.0242

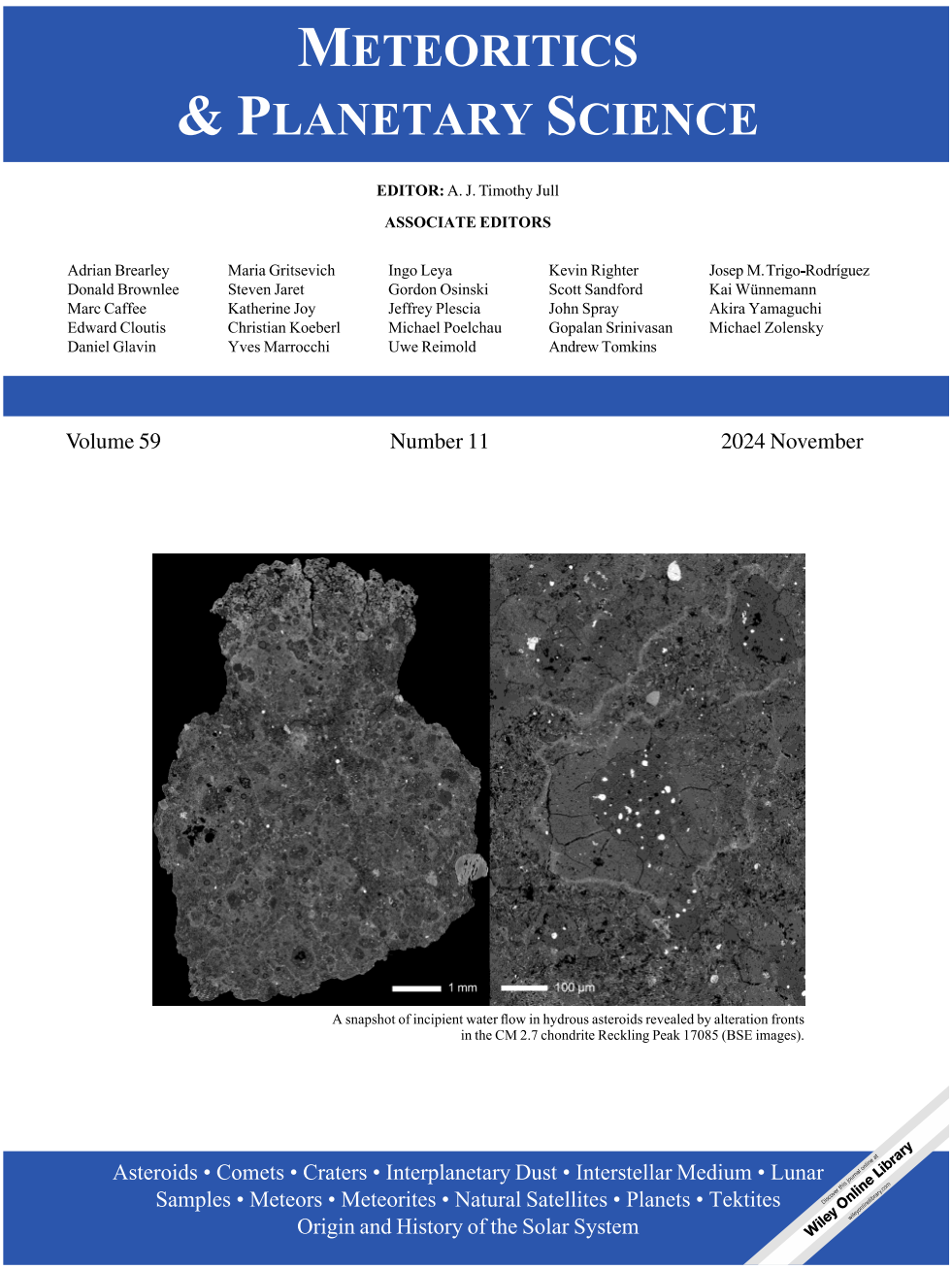

Figure 5. The cover of the November 2024 issue of Meteoritics and Planetary Science features a large area BSE image map (left panel) obtained at the CISUP ESEM-FEG FEI QUANTA 450 laboratory, by one of our former MSc Student Anna Musolino (currently at CEREGE, France). Musolino et al. (2024) Meteoritics and Planetary Science https://doi.org/10.1111/maps.14261

Azimi B., Labardi M., Sorayani Bafqi M.S., Macchi T., Ricci C., Carnicelli V., Scarpelli L., Hussain I., Matino F., Scaglione M., Pisignano D., Lazzeri A., Uddin M.J., Persano L, Danti S. (2024) Remnant polarization and structural arrangement in P(VDF-TrFE) electrospun fiber meshes affect osteogenic differentiation of human mesenchymal stromal cells. Materials and Design. 241, 112973. https://doi.org/10.1016/j.matdes.2024.112973

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Bresciani G., Guelfi M., Dosa M., Guiotto V., Crocellà V., Lessi M., Taddei M. (2024) Cu(II)-Based Coordination Polymers Containing 1,4-Bis(1*H*-imidazol-1-yl)benzene and Monovalent Fluorinated Anions. *Cryst. Growth Des.* 24, 8999-9010. https://doi.org/10.1021/acs.cgd.4c01038

Campanale F., Mugnaioli E., Folco L., Parlanti P., Gemmi M. (2024) TiO_2 II: The high-pressure Zr-free srilankite endmember in impact rocks. *Meteoritics and Planetary Science* 59, 529 – 543. https://doi.org/10.1111/maps.14137

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Folco L., Masotta M., Rochette P., Del Rio M., Di Vincenzo G. (2024) Australasian microtektites: early target-projectile interaction in large impacts on Earth. *Geochemical Perspectives Letters* 31, 22 – 26. https://doi.org/10.7185/geochemlet.2427

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Pecorini G., Braccini S., Simoni S., Corti A., Parrini G., Puppi D. (2024). Additive Manufacturing of Wet-Spun Poly(3-hydroxybutyrate-co-3-hydroxyvalerate)-Based Scaffolds Loaded with Hydroxyapatite. Macromolecular Bioscience, 24(6), 2300538. https://doi.org/10.1002/mabi.202300538

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Shisseh T., Aoudjehane H.C., Hewins R., Folco L., Zanda B., Agee C.B., Jacquet E., Zennouri L., Leili M.H., Pont S. (2024) On the origin of metallic iron in eucrite breccias: Effects of impact shock and mixing on the surface of (4) Vesta. *Icarus* 412, 115981. https://doi.org/10.1016/j.icarus.2024.115981

Spagnuolo L., Beneventi D., Dufresne A., Operamolla A. (2024) High Yield Synthesis of Cellulose Nanocrystals From Avicel by Mechano-Enzymatic Approach, *ChemistrySelect*, 9, e202401511. https://doi.org/10.1002/slct.202401511

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Van de Perre, D., Serbruyns, L., Coltelli, M.-B., Gigante, V., Aliotta, L., Lazzeri, A., Geerinck, R., Verstichel, S. (2024) Tuning Biodegradation of Poly (lactic acid) (PLA) at Mild Temperature by Blending with Poly (butylene succinate-co-adipate) (PBSA) or Polycaprolactone (PCL). *Materials 17*, 5436. https://doi.org/10.3390/ma17225436

Remarkable is also the number of research projects that are benefitting of the service produced by the ESEM FEG FEI QUANTA 450 laboratory. They are:

- PRIN 2022 *Piezoelectric cochlear implant: the new paradigm for sensorineural hearing loss (PROMISE).* Stefano Berrettini (Dip. Patol. - UniPi)
- MUR progetto PON 2014–2020 (D.M. 1061/2021, CUP I59 J21017690008). Laura Spagnuolo (DCCI)
- MAECI Progetto di grande Rilevanza NANOFUN - Grant Number PGR01179, CUP I53C22003350001
- Programma di Ricerche in Artico (PRA) 2021 *Integrated Reconstruction of Ice sheet DYnamics during late quaternary Arctic climatic transitions (IRIDYA)*. Caterina Morigi (DST-UniPI)
- Programma Nazionale delle Ricerche in Antartide - PNRA 2022 Volcanoes for Cryosphere and Atmosphere (VolCA). P.I. Sergio Rocchi (DST-UniPi)
- PRIN 2022 *Integrated procedures for FAST ChARging with online state-of-health evaluation of lithium-ion batteries (FASTCAR).* Antonio Bertei, Monica Puccini, Federica Barontini (DICI-UniPi), Giovanni Lutzemberger (DESTEC-UniPi), Maria Paola Carpanese (DICCA-UniGe), Alessandro Ruvio (DIAEE-UniRM1).
- PRIN 2022*: HEATED - PHrEatic erupTions at Active volcanoEs: processes, source parameters and physical models of eruption Dynamics.* Raffaello Cioni (DST-UniFi), Marco Pistolesi (DST-UniPi).
- PRIN 2022 *Cosmic Dust II: Cosmochemistry and Space Tweezers Technologies for Solar System Science and Exploration.* Luigi Folco (DST-UniPi).

- PRIN 2022 *Nano-focus on metamorphic garnet*. Bernardo Cesare (DG-UniPd), Enrico Mugnaioli (DST-UniPi).
- PRIN2022 "PROVES: an integrated PetRO Volcanological monitoring approach applied to Mt. Etna and Stromboli" (PRIN 2022N4FBAA) (PI M. Masotta, DST UniPi).
- ASI-MUR exPE15 *SpaceItUP!*. Luigi Folco (DST-UniPi).
- JAXA 2022 *First Ryugu sample announcement of opportunity*. Marco Ferrari (IAPS-INAF-Roma), Luigi Folco, Enrico Mugnaioli, Matteo Masotta (DST-UniPi).
- PNRA, Antarctic Meteorites classification. Sonia Sandroni and Alfonso Fiorelli (Museo Nazionale dell'Antartide).
- STOP-VIRUS (IBF-CNR/AOUP). Giorgio Soldani (IFC-CNR), Mario D'Acunto (IBF-CNR).
- 859752-HEL4CHIROLED-H2020-MSCAITN-2019, Lorenzo Di Bari (DCCI).
- (PNRR) THE—Tuscany Health Ecosystem, Spoke 7—Innovating Translational Medicine—Sub-project 5—Innovative models for management of infections caused by antibiotic-resistant bacteria (Project code: ECS00000017; CUP I53C22000780001).
- PRIN 2022 "RePowder: recycled powder for sustainable metal additive manufacturing". ID# P2022MEFCJ (DICI-UniPi)
- PRA_2022_65. Determinazione di tensioni residue combinando il metodo del foro e digital image correlation, DIC. (DICI-UniPi)
- "La suscettività all'idrogeno degli acciai al carbonio per pipeline e tubing. Valutazione di nuovi metodi di caratterizzazione", Contract JRA 5210001883- Eni S.p.A, (DICI-UniPi)
- PRIN 2020 *Sustainable Preservation Strategies for Street Art* (SUPERSTAR). Francesca Modugno (DICI - UniPi)

6. Education and Outreach

In 2024, the laboratory has provided analytical support to the following PhD, MSc, BSc students, trainees, and early research scientists:

- Aaron Kuriakose Prakash Ciampi (MSc, DICI, UniPi)
- Alberto Pio Grassi (MSc, DST, UniPi)
- Alfonso Fiorelli (AR, MNA, UniSi)
- Alice Tomassini (PhD, DST-UniPi/UniVe)
- Ambra Canovai (MSc, DCCI, UniPi)
- Andrea Taddeucci (PhD, DCCI, UniPI and Diamond Light Source, UK)
- Andrea Valzano, Corso di Laurea Magistrale in Chimica DCCI, UniPi
- Annalisa Galletti (MEng, DICI, UniPi)
- Beatrice Cecchini (Under-graduate student, MSc, DRT-NTMC -UniPi)
- Caterina Campinoti (PhD, DCCI, UniPi)
- Cinzia Guglielmi (MSc, DCCI, UniPi)
- Costanza Paccagnini, Corso di Laurea Triennale in Chimica DCCI, UniPi
- Debora Lazzerini (MSc, DST, UniPi)
- Elena Ciampi (MSc, DICI, UniPi)
- Elena Kouramanis, Erasmus+ visiting student, Paesi Bassi, presso DCCI UniPi
- Eleonora Pace (trainee, DST, UniPi)
- Esingül Kaya (AR, DRT-NTMC -UniPi)
- Fabio Colle (PhD, UniParma)
- Federico Paolino, PhDsc, DSCM-DCCI, UNIPI
- Francesca Campioni (MSc, DST, UniPi)
- Greta Biale (PhD, DCCI, UniPi)
- Karina Semonova (MSc, DST, UniPi)
- Laura Spagnuolo, PhDsc, DSCM-DCCI, UNIPI
- Leonardo Barlucchi (PhD, DCCI, UniPi)
- Marco Masi (PhD, DST, UniPi)
- Marlene Palyart, Erasmus+ visiting student, Francia, presso DCCI UniPi
- Marta Bianchi (PhD student, PhD Course in Clinical and Translational Science, UniPi)
- Martina Bertella (BSc, DCCI, UniPi)
- Matteo Cei (MSc, DCCI, UniPi)
- Matteo Salvadori (PhD, DST, UniPi)
- Miriam Gizzi (BSc, DST, UniPi)
- Niccolò Cappellini (BSc, DST-UniPi)

- Niccolò Magnani (MSc, DST, UniPi)
- Niccolo Medica, Corso di Laurea Magistrale in Chimica (DCCI, UniPi)
- Nicola Lomuscio, Corso di Laurea Magistrale in Chimica DCCI, UniPi
- Roberta Rovelli (Under-graduate student, MSc, DRT-NTMC -UniPi)
- Roberto Borriello (PhD, DST, UniPi-UniVe)
- Samuele Ottaviani (MSc, DST, UniPi)
- Saverio Caporalini (PhD student, PhD Course in Clinical and Translational Science, UniPi)
- Simone Pagni, Corso di Laurea Magistrale in Chimica DCCI, UniPi
- Sofia Lorenzon (PhD, DST, UniPi)
- Viviana Lo Giudice (Under-graduate student, MSc, DRT-NTMC -UniPi)

In 2024, the laboratory has been used for the following UniPi courses:

- Electron Microscopy of Nanomaterials (laurea magistrale in Materials and Nanotechnology)
- Cosmochemistry – laboratory activities (laurea magistrale in Scienze e Tecnologie Geologiche)
- Planetary Geology – laboratory activities (laurea Triennale in Scienze Geologiche)
- Petrologia Sperimentale (laurea Magistrale in Scienze e Tecnologie Geologiche, WGE-LM)
- Microscopia e diffrazione elettronica per materiali cristallini – (laurea magistrale in Scienze e Tecnologie Geologiche)
- Scanning electron microscopy of in vitro infection models (PhD Course in Clinical and Translational Science, UniPi; Master degree in Biology applied to Biomedicine, UniPi)
- Nuclear Materials, course (laurea magistrale in Ingegneria Nucleare)
- Materials, module (Laurea Professionalizzante: Tecniche per la meccanica e la produzione - UniPi)
- Antropologia Scheletrica, Forense e Paleopatologia, (Master Course, UniPi-UniBo-UniMi)
- Analytical Chemistry Laboratory (laurea magistrale in Chimica industriale)
- Laboratorio di Chimica Analitica 5 – (laurea magistrale in Chimica, curriculum analitico)

6. Issues and objectives

Performance **-** We would like to increase the performance of the ESEM-FEG FEI QUANTA 450 laboratory through the implementation of the EBSD analyses.

Cite our lab in your work. The papers which benefited of the work carried out in our FEI QUANTA 450 ESEM-FEG laboratory listed above do acknowledge our lab correctly in their methodology chapters (and acknowledgements) ad we are most grateful to that. We strongly encourage to carry on with this far practice because beneficial to the entire CISUP community. Please check our laboratory web page, to correctly cite our lab/instrument in your papers using – for instance - a sentence like the following one:

"Scanning electron microscopy analyses were performed using a FEI QUANTA 450 ESEM-FEG at the Centro per la Integrazione della Strumentazione della Università di Pisa (CISUP)"

Thanks!