

Metallic Nanostructured Materials and Thin Films

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1. Introduction and Scope

The field of metallic nanostructured materials and thin films has witnessed rapid growth over the past decades, driven by the continuous demand for miniaturization, efficiency, and multifunctionality in science and technology [1–3]. Metallic nanomaterials, whether in the form of nanoparticles, nanowires, or patterned architectures, exhibit remarkable optical, electrical, magnetic, and catalytic properties that can be finely tuned through structural control at the nanoscale [3–5]. At the nanoscale, metallic systems exhibit emergent physical and chemical phenomena that enable unprecedented control over optical, electronic, and catalytic responses [5–13], phenomena inaccessible in bulk metals. Thin film technologies, in turn, have long been indispensable for electronics, coatings, sensors, energy devices, and biomedical interfaces, and their integration with nanoscale metallic systems has created unprecedented opportunities for innovation [14–19]. When organized into thin films and multilayers, metallic nanostructures serve as versatile platforms for functional integration, bridging fundamental discoveries with device-oriented applications [19–21].

A particular emphasis is placed today on metallic nanoparticles, which, depending on their size, shape, and composition, offer extraordinary potential in catalysis, sensing, imaging, and energy conversion. Noble metal nanoparticles (Au, Ag, Pt) are especially valued for their plasmonic and catalytic properties, while transition metal nanoparticles (Fe, Co, Ni, Cu) underpin advances in magnetism, spintronics, and sustainable catalysis [21–28]. Thin films and multilayers bring complementary advantages: precise thickness control, tailored interfaces, and the possibility of combining dissimilar materials to achieve synergistic effects, such as enhanced hardness, diffusion resistance, or quantum well states. By stacking metallic layers at the nanometer scale, researchers can engineer artificial materials whose properties far exceed those of their constituents [29,30].

The progress of this field has been made possible by the continuous evolution of processing techniques, ranging from physical and chemical vapour deposition methods (magnetron sputtering, pulsed laser deposition, atomic layer deposition) to bottom-up strategies such as sol–gel synthesis, chemical reduction, and colloidal assembly [31–35]. Additive nanomanufacturing, self-assembly, and laser-based methods further enrich the toolbox, enabling both large-area production and fine patterning with sub-micrometer resolution [36–40]. Recent advances in operando and ultrafast characterization—electron microscopy, synchrotron radiation spectroscopy, scanning probe methods—allow researchers to directly monitor structural evolution and dynamics, providing invaluable feedback for tailoring performance. Increasingly, artificial intelligence and multiscale simulations are integrated into this workflow, accelerating discovery and optimization of metallic nanostructures [41–43].

Beyond academic laboratories, metallic nanostructured materials and thin films are making their way into industrial applications. In the energy sector, nanostructured metallic



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catalysts are key to hydrogen evolution, CO₂ reduction, and fuel cell efficiency. Thin films enhance the performance of batteries, supercapacitors, and thermoelectrics by controlling charge transport and scattering processes. In information technologies, metallic multilayers underpin magnetic storage, spintronic devices, and emerging neuromorphic architectures. Biomedical applications are equally promising: antibacterial coatings for implants, plasmonic nanostructures for diagnostics and therapy, and multifunctional metallic films for bioelectronics and wearable sensors. These examples highlight the strong connection between fundamental research, scalable fabrication, and technological deployment [44–49].

In summary, metallic nanoparticles, thin films, and multilayers represent a vibrant frontier where nanoscale engineering converges with real-world applications. They exemplify how controlled synthesis, advanced characterization, and computational design are reshaping our capacity to innovate in materials science [50]. The following section presents the diverse contributions gathered in this Special Issue, reflecting the richness of current research and pointing toward the challenges and opportunities that will define the next stage of development.

2. Contributions

In this editorial, we seek to capture these cutting-edge directions by bringing together contributions from leading experts across disciplines. The collected works not only chart the current state of the art but also highlight the challenges, opportunities, and cross-disciplinary synergies that will define the field in the coming years. By placing emphasis on innovation and application-oriented perspectives, this volume is intended to serve both as a reference for specialists and as a catalyst for novel ideas and collaborations at the intersection of physics, chemistry, engineering, and nanotechnology. Next, we'll present in brief the 9 papers that are included in this reprint.

In their review entitled “Materials Design and Development of Photocatalytic NO_x Removal Technology”, Bari et al. [contribution 1] advancements in photocatalytic NO_x removal, with a focus on materials design strategies, intrinsic properties, fundamental developmental aspects, and performance validation. This work also presents research gaps, emphasizing the need to understand the comprehensive mechanistic photocatalytic process, favoured conditions for generating desired reactive species, the role of water concentration, temperature effects, inhibiting strategies for photocatalyst-deactivating species, and the formation of toxic NO₂.

Next, Kim et al. highlight results on “Microgrid-Patterned Ni Foams as Current Collectors for Ultrafast Energy Storage Devices” [contribution 2]. The authors fabricated Ni foam current collectors with a tunable surface geometry by using scalable *steel use stainless* (SUS) meshes as rigid stamps during the roll pressing process. The Ni foams presented differently sized 3D microgrid-patterns on their surfaces, which varied based on the mesh scales used for their processing. More importantly, the microgrid-patterned Ni foams exhibited different surface geometries, i.e., width/depth of the microgrid patterns, and specific surface roughness parameters. The surface modification of the Ni foam enhanced its charge transport kinetics, resulting in a robust electric double layer, even under ultrafast charge/discharge conditions (>15 A/g). Their study findings suggest that modifying the surface geometry of a Ni current collector via the stamp micro-patterning method can be a novel strategy for improving the ultrafast-rate capability of EDLC electrodes without requiring specific optimization of the active materials.

Following, the paper entitled “New Method to Recover Activation Energy: Application to Copper Oxidation” by Barchiesi et al. [contribution 3] brings a new input on the calculation of the activation energy that helps to understand and to identify the underlying phenomenon of oxidation. The authors propose a new method without any a priori

hypothesis on the oxidation law to retrieve the activation energy of partially and totally oxidized samples subject to successive annealing. The method handles the uncertainties in the measurement of metal and oxide thicknesses at the beginning and at the end of the annealing process. The possible change in oxidation law during annealing is included in the model. By using an adapted Particle Swarm Optimization method to solve the inverse problem, the authors also calculate the time of final oxidation during the last annealing. The authors apply the method to successive annealings of three samples with initial nanometric layers of copper, at ambient pressure, in the open air.

The work of Kobierska et al. provides insight into “How Cracks Induced by Straining Influence the Tribological Properties of Mo Films Deposited on Polyimide Substrates” [contribution 4]. Thin film materials used in flexible electronics that are usually deposited on polymer substrates must withstand a variety of static and dynamic mechanical loading conditions to ensure adequate reliability of the device. Tribological loads are also among these loading conditions, and suitable characterization methods and strategies are required for analysing friction and wear for a variety of tribological contact situations. The Authors provide results on Mo films deposited on polyimide substrates by high-power impulse magnetron sputtering and then pre-conditioned by straining to several strain levels, including crack onset strain and strains within the crack saturation regime.

Next, Zhou and Wang report on “Mechanical Properties of Thermally Annealed Cu/Ni and Cu/Al Multilayer Thin Films: Solid Solution vs. Intermetallic Strengthening” [contribution 5]. This original paper provides the readers with an accurate insight on Cu/Ni and Cu/Al multilayers, with individual layer thickness varying from 25 nm to 200 nm, and co-sputtered Cu-Ni and Cu-Al single-layer films that were deposited at room temperature via magnetron sputtering and further annealed from 100 °C to 300 °C. Both multilayer systems exhibit an increase in hardness with increasing annealing temperature. However, the Cu/Ni system shows a gradual and moderate hardness increase (up to 30%) from room temperature to 300 °C, while the Cu/Al system displays a sharp hardness surge (~150%) between 125 °C and 200 °C. The co-sputtered Cu-Ni and Cu-Al samples consistently demonstrate higher hardness than their multi-layered counterparts, albeit with distinctly different temperature dependence—the hardness of Cu-Ni increases with annealing temperature while Cu-Al maintains a constant high hardness throughout the entire temperature range.

Another hot topic is concerning the “Elimination of Low-Angle Grain Boundary Networks in FeCrAl Alloys with the Electron Wind Force at a Low Temperature”, as presented by Rahman et al. [contribution 6]. This study presents an electron wind force-based annealing approach to recover cold-rolling-induced residual stress in FeCrAl alloy below 100 °C in 1 min. This is significantly lower than conventional thermal annealing, which typically requires temperatures around 750 °C for about 1.5 h. A key feature of their approach is the athermal electron wind force effect, which promotes dislocation movement and stress relief at significantly lower temperatures. The electron backscattered diffraction (EBSD) analysis reveals that the concentration of low-angle grain boundaries (LAGBs) is reduced from 82.4% in the cold-rolled state to a mere 47.5% following electropulsing. This level of defect recovery even surpasses the pristine material’s initial state, which exhibited 54.8% LAGBs.

Microwave dewetting of thin gold films is an efficient method for fabricating gold nanoislands with controlled morphology. When a continuous gold film is exposed to microwave irradiation inside a kiln, rapid and localized heating occurs due to the interaction of the metal with the electromagnetic field. This induces surface diffusion and Rayleigh-like instabilities in the thin film, breaking it up into discrete nanoparticles or nanoislands. The process is faster and often more energy-efficient than conventional thermal annealing, while

also enabling tunability of the resulting nanostructures through variation in film thickness, substrate properties, and microwave power. The resulting gold nanoislands exhibit unique plasmonic properties, making them valuable for applications in sensing, catalysis, and photonic devices. In their work, entitled “Controlled formation of nanoislands during microwave annealing of Au thin films”, Al-Rubaye et al. [contribution 7] demonstrate gold nanoisland fabrication by starting from gold thin films of 4, 5, 6, 8, and 10 nm thickness, respectively. These films are deposited directly onto cost-efficient BK7 glass substrates by using a thermal evaporator, and subsequently, microwave irradiation is used. This procedure leads to the controlled formation of gold nanoislands when the thin-film samples are placed in a furnace/kiln. The heat generated by the microwave radiation is focused inside the furnace due to its heat-resistant graphite coating and reaches over 550 °C, the required annealing temperature. Such a cost-effective method enables the fabrication of high-specification gold nanoislands, saving time and energy, on various substrates.

Concerning complex and hybrid materials, Kresnik et al. present in their work entitled “Hybrid Nanostructures of Fe₃O₄ and Au Prepared via Coprecipitation and Ultrasonic Spray Pyrolysis” some very interesting results [contribution 8]. The coupled processes of coprecipitation and ultrasonic spray pyrolysis were used to synthesize Fe₃O₄-Au hybrid nanostructures. The first coprecipitation method enabled the synthesis of Fe₃O₄ nanoparticles by mixing iron salt ions (Fe²⁺ and Fe³⁺) and ammonia as the base, and ultrasonic spray pyrolysis was used as the coating process of the Fe₃O₄ nanoparticles with Au. The formatted hybrid nanostructures consist of Fe₃O₄ nanoparticles that have Au on their surface in the form of gold nanoparticles (AuNPs). AuNPs have a crystalline structure and range in size from 10 to 200 nm. It was found that ultrasonic spray pyrolysis produces separate AuNPs too, and not just on the Fe₃O₄ surface, suggesting a bimodal formation of AuNPs. The zeta potential of the Fe₃O₄ nanoparticles showed poor stability (−15 mV), indicating a high tendency to aggregate, and the zeta potential of the hybrid nanostructures was also very low ($\cong 0$), which, comparatively, means even worse stability. The saturation magnetization of the Fe₃O₄ nanoparticles was 35 emu/g, which is relatively lower than that of bulk Fe₃O₄, while the saturation magnetization of the hybrid nanostructures was significantly lower (0.1 emu/g) compared to the Fe₃O₄ nanoparticles.

Soft magnetic composites are a new type of soft magnetic material made by insulating and coating the surface of metal in soft magnetic powder with a high saturation magnetic induction intensity using high-resistivity organic or inorganic compounds and pressing the coated powder using powder metallurgy technology. Widely used in transformers, such material relay electronic devices and measuring instruments due to their high saturation magnetization, high electrical resistivity, low core loss, and three-dimensional isotropic magnetic properties. In their work, Li et al. propose the “Preparation and Characterization of MnFe₂O₄/Fe Soft Magnetic Composites by Surface Oxidation” [contribution 9]. MnFe₂O₄/Fe soft magnetic composites (SMCs) are designed by the surface oxidation method, and the MnFe₂O₄ layer is utilized as the insulation coating. The effect of annealing temperature on the insulation layer is investigated, and its relationship with the magnetic properties of the MnFe₂O₄/Fe SMCs is explored. The best overall performance is obtained at 50 mT and 100 kHz with saturation magnetization $M_s = 205$ emu/g, amplitude permeability $\mu_a = 100$, and a core loss of 234.9 W/kg. Therefore, their work provides a method to develop a novel insulating coating to reduce core loss, which is of great significance to the investigation of other Fe-based soft magnetic composites for applications in high-frequency magnetic fields.

To conclude, the contributions gathered in this volume underscore the vitality and breadth of research in metallic nanostructured materials and thin films. From fundamental investigations of nanoscale phenomena to application-driven advances in energy, catalysis,

electronics, and biomedicine, the works presented here reflect both the maturity of the field and its continuing capacity for innovation. The integration of novel fabrication methods, operando characterization, and data-driven design approaches is accelerating discovery and enabling functionalities once considered beyond reach. As this collection demonstrates, metallic nanostructures and thin films are not only central to current technologies but also poised to play a transformative role in addressing global challenges—ranging from sustainable energy production to next-generation information systems and healthcare solutions.

3. Conclusions and Perspectives

Despite remarkable progress, major challenges remain in achieving scalable fabrication, long-term stability, reproducibility across platforms, and sustainable processing routes. Bridging laboratory-scale demonstrations with industrial deployment continues to represent a critical bottleneck, particularly for multifunctional metallic nanostructures operating under real working conditions. This Special Issue, “Metallic Nanostructured Materials and Thin Films”, demonstrates the vitality and diversity of research at the intersection of nanoscale design, advanced fabrication, and application-driven innovation. The contributions gathered here highlight how metallic systems, engineered at the scale of atoms to microns, are opening new opportunities in energy conversion, catalysis, information technologies, flexible electronics, and biomedicine. Taken together, they reflect both the maturity of the field and its continuing capacity for surprise and transformation.

Beyond academia, the insights and innovations highlighted here hold direct relevance for industry, policy, and society at large. The development of sustainable catalytic processes, energy-efficient coatings, advanced sensing platforms, and bio-integrated devices reflects how metallic nanostructures can contribute to the green transition, digital transformation, and improved public health. By fostering stronger connections between research, innovation ecosystems, and industrial applications, this collection aims not only to document the state of the art but also to inspire solutions that extend well beyond the laboratory.

At LP3 (UMR 7341/CNRS—Aix-Marseille Université), we are strongly engaged in international networks such as RIANA (Research Infrastructure Access in Nanoscience & Advanced Materials, <https://riana-project.eu/>, accessed on 1 January 2025), Lasers4EU (<https://www.lasers4.eu/>, accessed on 1 January 2025), and Laserlab-Europe AISBL (<https://laserlab-europe.eu/>, accessed on 1 January 2025), which promote open access to advanced infrastructures and strengthen collaborations across Europe. These frameworks provide unique opportunities for researchers to share expertise, exchange young scientists, and build new partnerships around laser–materials interactions, thin film engineering, and nanostructured coatings. A concrete example is the visit of Dr. Alaa Alasadi, supported by a mobility fellowship via Campus France (<https://www.campusfrance.org/>, accessed on 1 January 2025), whose six-month collaboration at LP3 led to fruitful exchanges and new joint projects. We warmly invite colleagues to make use of these networks, engage in joint initiatives, and expand the collaborative spirit that drives innovation in our field.

We also wish to warmly thank all contributing authors for their high-quality work, the reviewers for their thoughtful evaluations, and the editorial team of Metals for their professional support throughout this process. Their combined efforts have ensured the success of this volume.

Encouraged by the breadth and impact of the studies presented here, we are pleased to announce that a second volume of this Special Issue will be launched in the near future. We look forward to receiving new contributions, to fostering cross-disciplinary dialogue, and to inspiring collaborations that will carry this vibrant field even further. We warmly encourage researchers worldwide to join this effort by contributing their most recent results to the next edition.

Conflicts of Interest: The authors declare no conflicts of interest.

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