

# Advanced Processing Technologies for Innovative Materials

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## 1. Introduction and Scope

There is a need for further, in-depth research that explores the synthesis of newly developed materials created using advanced technologies. Their potential uses in various applications, as well as their ability to operate under increased thermal and mechanical loads and in the presence of moisture or other contaminants, are of particular interest [1–3]. The latest progressive technology approaches to industrial realities must be adapted for the sixth technological paradigm [4–6]. Many questions have arisen with regard to the quality of products' surfaces post-production [7–9] and repair, particularly with regard to additive manufacturing, 3D printing, or synthesis [10–13], which lend such products exceptional operational properties [14–16]. As such, it is necessary not only to conduct research under various conditions but also to develop new functional coating, surface cleaning, and processing methods [17–19]. Fundamental issues related to the synthesis and processing of multicomponent objects, structures, and composites, the creation of new classes of materials and nanocrystalline alloys, advanced surface treatments and multilayer coatings, and the development of new approaches and technological solutions using the latest achievements in the information sphere are of particular interest to the industry [20–25]. In particular, process monitoring and productivity improvements in the scale of real serial and mass production of newly developed methods and technologies deserve special attention [26–30].

For the last few decades, cutting-edge advances in processing, synthesis, and research methods and technologies relating to developed materials, objects, and coatings have featured in outstanding scientific publications, attracting widespread attention from the research community. In addition, progressive approaches have received multiple awards at the most prestigious scientific conferences, industrial exhibitions, and other events around the world.

This Special Issue is devoted to the latest achievements in the technologies of production and synthesis of innovative 0D–2D, nano-, and functionally gradient objects, nanocomposites, nanoporous structures (aerogels) and coatings, and new processing methods and technologies based on plasma and laser treatment, electrophysical and chemical processing and synthesis, as well as research on their exploitation properties and the development of digital models and machine learning approaches for the needs of the industry.

## 2. Contributions

Ten scientific articles, three communications, and a review were published in the presented Special Issue, each addressing a critical topic relating to the processing and synthesis of innovative materials. These topics include surface treatment, coating, and film deposition technologies; the synthesis of multicomponent objects and their behavior under various exploitation conditions (cooling, increased thermal and mechanical loads, etc.); and their mechanical and physical properties, including optical and electrical properties, for use in the aviation and tool industry and in different sensory and electronic devices [31–39]. These topics are described in greater detail below.



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- Technologies for improving the surface quality of metallic parts produced by laser additive manufacturing and developing of the technology of ion polishing those parts in gas-discharge plasma and subsequent coatings for use in the aviation industry are reviewed. Other finishing techniques, such as mechanical machining, chemical etching, surface plastic deformation, ultrasonic cavitation abrasive finishing, and laser ablation, are also considered [31].
- Comparative research is conducted on the mechanical machining technologies that are used to improve the surface layer condition of the ceramic tools, such as diamond grinding and diamond grinding–lapping–polishing. The development of advanced double-layer (CrAlSi)N/DLC coating deposition technology on SiAlON ceramics used as cutting tools for turning nickel-based Inconel 718-type superalloys is also explored [32].
- Research is conducted on the machinability of insulating material, such as Al<sub>2</sub>O<sub>3</sub> oxide ceramics, using the developed mono- and multilayer assistive coatings based on a Cu-tape and Cu-Ag “sandwich” for advanced electrical discharge machining in a ZnO powder–water medium [33].
- One study explores the influence of shallow (at −20 °C) and deep (at −196 °C) cryogenic treatment of a magnesium nanocomposite Mg/2 wt.%CeO<sub>2</sub> produced via the disintegrated melt deposition method followed by hot extrusion, and then investigate its influence on the porosity, grain size, dislocation density, ignition temperature, microhardness, lattice strain, 0.2% compressive yield strength, ultimate strength, and fracture strain of this material when it is operated in sub-zero conditions and strength-based constructions [34].
- Two innovative nanocrystalline soft magnetic Fe-based nanoperm-type alloys are created by mechanical alloying. Their alloys are initially composed of Fe<sub>85</sub>Zr<sub>6</sub>B<sub>8</sub>Cu<sub>1</sub> (at.%) and Fe<sub>80</sub>Zr<sub>5</sub>B<sub>13</sub>Cu<sub>1</sub> (at.%). Their operating temperature range (thermal stability and Curie point) and magnetic properties depending on the Fe/B ratio were evaluated. Additionally, high-saturation magnetic flux density is determined to be of interest for the development of low-dimensional systems, such as 0D-2D objects [35].
- Metal–organic chemical vapor deposition selective area epitaxy of Al<sub>z</sub>Ga<sub>1−z</sub>As (0 ≤ z ≤ 0.3) bulk layers is developed using a passivating mask with ultrawide windows—in other words, a SiO<sub>2</sub>-mask/window of alternating stripes of 100 μm—on a GaAs substrate to produce strained quantum wells of AlGaAs/GaAs solid solutions (0D objects) in light-emitting structures for optoelectronic applications [36].
- Researchers explore the physical and optical properties of colloidal nanoparticles, particularly AgInS<sub>2</sub> quantum dots (0D objects), produced by a newly developed scalable manufacturing method of additive 3D-printing microfluidic chips for microfluidic synthesis. An increased product yield of 60% is observed [37].
- Research is conducted to evaluate the frequency peaks of epitaxial 2D objects. Graphene is chemically grown on 6H-SiC substrates using a Raman spectrometer and cooled in a range from room temperature to −180 °C to ensure that the graphene is properly insulated against moisture [38];
- Thermal synthesizing technology for carbide ceramic (β-SiC) film deposition on a mono-silicon substrate is developed, in which porous silicon is used as an intermediate layer for adhesion in the double-layer structure of β-SiC/por-Si/mono-Si composition. This allows the developed carbide ceramic film to meet the needs of electronics and proto-electronics in light of the indicated quantum size effects (0D–2D objects or, in other words, nano-objects) [39].

The process of purifying water using regenerating iron-based adsorptive media and plasma-activated water generation as biocidal agents for growing plants, and the technology of production and properties of porous polymer materials that are used in filters and drug-loading tablets [40–42], are also discussed in depth, as follows:

- An innovative regenerating iron-based adsorptive media and a relevant device for removing arsenic (As) from drinking water are developed [40].

- Research is conducted to determine the effects of barrier and bubble-pulsed discharges on the properties—particularly the ionic speciation and concentrations, pH index, and electrical conductivity—of plasma-activated water produced from distilled and groundwater [41].
- A carbon dioxide-assisted polymer compression method is developed for the creation of porous polymer products with laminated fiber sheets. This method can be used to design filters and drug-loading tablets [42].

Analytical models, software, and interactive databases of production technologies of nanoporous aerogels and metallic parts are also developed [43,44].

- The researchers developed software for digital models of porous and nanoporous structures such as aerogels to create new materials based on SiO<sub>2</sub>, silica–resorcinol–formaldehyde, polyamides, carbon, polysaccharides, proteins, etc., to predict their properties (thermal and electrical conductivity, mechanical properties, sorption, and solubility) and pore size distribution using the lattice Boltzmann method and the cellular automaton particle dissolution model [43].
- A machine-learning-based approach and the database of basic and critical geometrical features for laser-based additive manufacturing from stainless steel for feature identification and part segmentation are developed to improve manufacturability, which can be extended to other technological processes and materials [44].

The authors of [31] identify the defects in additively manufactured machinery products made of stainless steel and various metallic alloys, such as cobalt, nickel, aluminum, and titanium alloys, which are earmarked for application in the airspace industry [45,46]. Mechanical machining, chemical etching, surface plastic deformation, ultrasonic cavitation abrasive finishing, laser ablation, and ion polishing in gas-discharge plasma are observed [47]. The method of gas-discharge plasma processing for finishing laser additive manufactured parts and subsequent wear-resistant coating is proposed for the first time. All the existing technologies are classified into three groups based on the possibility of volumetric processing and the nature of the exposure, which may be thermal, chemical, mechanical, or combined. The observed post-processing methods possess several disadvantages related to the nature of surface destruction, which influences the intensity of the wear on the working surfaces as a part of a unit or mechanism, along with the positive effect of plastic deformation and recrystallization of near-surface layers, erosion processes lead to stress states of the surfaces [48].

Innovative post-processing approaches, such as ion polishing in gas-discharge plasma, have no analogs in modern industry and may accelerate the transition to the next technological paradigm [49–51] by increasing the reliability and availability of additive manufacturing parts. Such approaches encompass the following stages:

- Granules of 40–100 μm in size are removed from the product's surface to achieve a roughness parameter R<sub>a</sub> (Arithmetic Mean Deviation) of 30 μm. Negative-voltage microsecond pulses of up to 30 kV are applied to the product during its immersion in the plasma.
- The product is polished with concentrated ions or fast argon atoms under an angle greater than 60°.
- A coating is deposited by evaporating liquid metal magnetron targets.

The condition of the surface layer and advanced double-layer (CrAlSi)N/DLC coating deposition [32] significantly influence the operational life of SiAlON ceramic cutting tools in machining Inconel 718-type chrome–nickel alloy. This type of alloy is often used to produce sophisticated aircraft turbine engine parts [52] and is machined by the most wear- and heat-resistant cutting tools that work under increased mechanical and thermal loads composed of oxide, nitride, and oxide-nitride cutting ceramics [53]. SiAlON is known for its excellent properties but is also very brittle, which can lead to machining difficulties. Industrially produced inserts feature numerous defects on their working surfaces; these serve as stress concentrators and provoke the destruction of the cutting edge by tearing the conglomerates

of the material. This shortens the operational life of the cutting tool. Additional diamond grinding, lapping, and polishing and complex double-layered coating (consisted of an additional DLC coating and trinitride underlayer for better adhesion) deposition exhibit improved the properties of the cutting insert surface, resulting in a microhardness of  $28 \pm 2$  GPa and an increase in the average friction coefficient at  $800^\circ\text{C}$ , which was  $\sim 0.4$ . The average operational life of the insert under the increased loads improved, reaching 12.5 min, which was 1.8 and 1.25 times greater than that of the industrial cutting insert and double layer-coated insert without additional diamond grinding, lapping, and polishing.

The authors of [33] aim to resolve the issues with the electrical properties of the insulators that, under normal conditions, cannot be processed by electrophysical technologies [54]. This is especially relevant for insulating materials such as oxide and nitride ceramics [55], which are needed to produce cutting tools for machining titanium and nickel-based alloys used in the aircraft industry as was mentioned above, especially during the production of gas turbine engines [52,53]. During the cutting process, these alloys create intensive thermal and mechanical loads in the contact zone of the tools with a temperature up to  $800\text{--}1200^\circ\text{C}$  and also tense state of up to  $1.3 \times 10^8\text{--}8.7 \times 10^9$  N/m<sup>2</sup>. The chips tend to stick to the tool's cutting edge due to high adhesion, increasing the loads and promoting the snatching of conglomerates and faster tool wear. One of the most promising (though difficult-to-machine) cutting ceramics is Al<sub>2</sub>O<sub>3</sub> (melting point of  $2045\text{--}2345^\circ\text{C}$ , coefficient of linear thermal expansion of  $7.0\text{--}8.0 \times 10^6$  K<sup>-1</sup> (at  $20\text{--}1000^\circ\text{C}$ ), which possesses thermal conductivity of  $20\text{--}25$  W/(m·K) (at  $20\text{--}100^\circ\text{C}$ ), Mohs hardness of 9) in addition to its excellent mechanical and physical properties. The authors of this paper proposed an advanced electrical discharge machining method using assistive means such as conductive coatings and powder-mixed water-based medium. Hydrocarbon-based media, such as oils and kerosene, should be avoided when machining Al-based materials due to the risk of the formation of explosive Al<sub>3</sub>C<sub>4</sub> products formed during its interactions with water, H<sub>2</sub>↑ and O<sub>2</sub>↑ gasses, which can be highly damaging to the machine and its filtration system. ZnO powder was chosen since it is a direct-gap semiconductor with a wide band gap E<sub>g</sub> of  $3.30\text{--}3.36$  eV. ZnO powder-mixed deionized water-based medium with a ZnO concentration of  $7\text{--}100$  g/L and Cu-Ag and Cu mono- and multi-layer coatings, which were composed of  $40\mu\text{m}$ -thickness copper tape and Ag adhesive, were used as assistive means. The total thickness of the assistive coatings was between  $40$  and  $120\mu\text{m}$ . A material removal rate of  $0.0032\text{--}0.0053$  mm<sup>3</sup>/s was achieved at a concentration of  $14$  g/L, a discharge pulse frequency of  $2\text{--}7$  kHz, and a pulse duration of  $1\mu\text{s}$ . The obtained data expand the scope of use of this sought-after material in the tool industry by highlighting new possibilities for its application.

The physical and mechanical properties of a Mg/2 wt.%CeO<sub>2</sub> nanocomposite at two cryogenic temperatures of  $-20^\circ\text{C}$  (253 K) and  $-196^\circ\text{C}$  (77 K) were researched to investigate this material's ability to operate in sub-zero conditions [34]. The nanocomposite was produced by the disintegrated melt deposition method followed by hot extrusion [56]. The shallow cryogen treatment at  $-20^\circ\text{C}$  reduced the porosity by 10.4%, and the deep cryogen treatment in liquid nitrogen (LN<sub>2</sub>) [57] at  $-196^\circ\text{C}$  (boiling point) by 43.3%. The ignition temperature was reduced by  $1^\circ\text{C}$  and amounted to  $635^\circ\text{C}$  for the samples after the cold treatment, whereas it increased by  $38^\circ\text{C}$  and amounted to  $674^\circ\text{C}$  for deep cryogen-treated samples. The grain size of the treated samples was  $2.9 \pm 1.0\mu\text{m}$  and  $2.8 \pm 0.6\mu\text{m}$ , exceeding the sizes of the initial samples ( $2 \pm 0.6\mu\text{m}$ ). Both samples had superior microhardness compared to the initial samples:  $89 \pm 5$  HV and  $92 \pm 4$  HV (correspondingly, 20% and 24% more than the initial samples). This can be related to their ability to increase the dislocation density, reduce porosity, and strain the lattice. The 0.2% compressive yield strength of the treated samples ( $186 \pm 17$  MPa for shallow cryogenic treatment,  $203 \pm 5$  MPa for deep cryogenic treatment) was up to 14% greater than for untreated samples ( $178 \pm 19$  MPa), indicating that the sub-zero treatments improved the Mg nanocomposites' performance in strength-based constructions. The 0.2% compressive yield strength of the samples after deep cryogenic treatment was  $\sim 9\%$  higher than that of the samples after the cold treat-

ment. After shallow cryogenic treatment ( $-20\text{ }^{\circ}\text{C}$ ), the ultimate strength of the samples ( $441 \pm 12\text{ MPa}$ ) was reduced by 2.5% compared to that of the samples after deep cryogenic treatment ( $452 \pm 15\text{ MPa}$ ), and was 7% lower than for untreated samples ( $473 \pm 16\text{ MPa}$ ). The fracture strain was  $16.5 \pm 0.7\%$  for untreated samples,  $29.1 \pm 1.0\%$  for the samples after shallow cryogenic treatment, and  $29.7 \pm 1.2\%$  for the samples after deep cryogenic treatment. The fracture surfaces of the samples showed no visible difference ( $45^{\circ}$  shear fracture). Both types of treated samples demonstrated higher roughness than the initial ones. Overall, after treatment at  $-196\text{ }^{\circ}\text{C}$ , the samples demonstrated an increase in the considered properties of the Mg nanocomposite, while the samples subjected to treatment at  $-20\text{ }^{\circ}\text{C}$  exhibited similar values at lower costs.

Two innovative nanocrystalline soft magnetic nanoperm-type Fe-based alloys of the Fe-Zr-B-Cu system with the initial chemical composition of  $\text{Fe}_{85}\text{Zr}_6\text{B}_8\text{Cu}_1$  (at.%) and  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$  (at.%) were obtained by mechanical alloying [35]. Those alloys are sought after for applications such as magnetic sensors and actuators [58]. Those types of nanocrystalline soft magnetic alloys have recently been developed as a replacement for ferrites. Their key properties include the thermal stability of the magnetic phase and soft-hard behavior; they are also characterized by their low magnetic coercive force, high saturation magnetic flux density, and high magnetic permeability (response to the magnetic field). Reduced magnetic coercive force and increased magnetic permeability reduce core losses in devices under alternate-current magnetic fields and controlling those characteristics reduces energy consumption [59,60]. The high saturation magnetic flux density is favored for developing low-dimensional systems [61] such as those described below.

- For 0D systems, particles are confined to a single point (e.g., quantum dots) [36,37].
- For 1D systems, particles are confined to a line (e.g., carbon nanotubes).
- For 2D systems, interactions are confined to a plane (e.g., graphene) [38,39].

In other words, magnetic flux density is used to create consolidated systems composed of many particles. One of the critical characteristics of these alloys is the Curie point—the temperature above which alloys lose their permanent magnetic properties, which determines their range of operating temperatures. The magnetic properties also change depending on the alloy's phase (amorphous, crystalline), which is determined by powder production technology. Two main technologies are used to produce alloy powders: gas atomization and mechanical alloying. Both methods are implemented during powder metallurgy as the step before powder sintering and consolidation. Gas atomization allows us to obtain soft magnetic spherical particles of the mainly amorphous phase with a superior magnetic response, while mechanical alloying primarily produces smooth particles that are more specific and irregular. In gas atomization, with a high dispersion of particle sizes and a particle size growth, the particle's phase can also be both amorphous and nanocrystalline. Such powder production technology also prevents contamination of the cutting tools that happened during mechanical alloying. Mechanical alloying is mostly used to produce Fe-based nanocrystalline alloy powders. It should be noted that as the crystalline size increases, the alloy loses its soft behavior. The thermal stability of these alloys is also determined by crystalline growth, which depends on the apparent activation energy of crystallization. In this context, mechanical alloying is preferable for creating metastable alloys of nanocrystalline (supersaturated solid solutions or high-entropy alloys) and amorphous phases. The soft magnetic response can be improved by optimizing the milling modes in mechanical alloying and adding other elements. The main difference between the two developed alloys is in the Fe/B elements ratio of 85/8 and 80/13, where Fe determines the amount of magnetism. Correspondingly, the magnetization of  $\text{Fe}_{85}\text{Zr}_6\text{B}_8\text{Cu}_1$  alloy is expected to exceed that of  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$ . At the same time, B is responsible for reducing the crystalline size, which increases the magnetic response by decreasing the coercive force. In such alloys, B usually determines the formation of the amorphous phase or more refined nanocrystalline phase, often leading to a loss of saturation magnetization. Zr and Cu were chosen equally in both alloys. Zr hinders crystalline growth due to the relatively large atom size, whereas Cu provokes a high density of nanocrystallization



and hinders the growth of larger crystals. Heating these alloys can also influence the crystalline growth and reduce soft magnetic behavior. The thermal stability of the alloys was researched via thermal analysis of the apparent activation energy of the crystalline growth and the Curie point. Exothermic processes occurred in the 450–650 K temperature range and were associated with tension relaxation. The apparent activation energy of the main crystallization process was determined by Kissinger and isoconversion methods (differential calorimetry) and was compared with the previously obtained data. The lowest value of apparent activation energy (288 kJ/mol) was obtained for the  $\text{Fe}_{85}\text{Zr}_6\text{B}_8\text{Cu}_1$  alloy. The transition temperatures, such as the peak crystallization and Curie points, were higher in the  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$  alloy. The magnetic response was similar when the saturation magnetization was 5% higher in the  $\text{Fe}_{85}\text{Zr}_6\text{B}_8\text{Cu}_1$  alloy than in the  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$  alloy. The high-saturation magnetization and low coercive force are necessary for the feasibility of the applications in devices based on a soft magnetic behavior. Although the saturation magnetization is slightly lower in the  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$  alloy, the decrease in the coercive force of  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$  alloy is relatively significant due to the smaller size of the nanocrystals. The magnetic behavior, combined with a high thermal stability front crystalline growth associated with loss of soft magnetic behavior in  $\text{Fe}_{80}\text{Zr}_5\text{B}_{13}\text{Cu}_1$  alloy, indicates that the reduction in Fe content does not significantly decrease its operating ability under the same conditions.

$\text{Al}_z\text{Ga}_{1-z}\text{As}$  epitaxial layers ( $0 \leq z \leq 0.3$ ) were produced by metal–organic chemical vapor deposition on a n-GaAs (100) substrate 2 inches in diameter with a  $\text{SiO}_2$  mask of stripes of 100  $\mu\text{m}$  in wide (100  $\mu\text{m}$  wide  $\text{SiO}_2$  mask/100  $\mu\text{m}$  wide window) [36]. The monolithic integration of electro-optical elements is one of the issues of modern photonics that can be resolved by selective area epitaxy [62]. This technology facilitates the implementation of control and generation of optical radiation and electrical signals for the needs of optoelectronic devices, and other relevant operations in the production of single-mode lasers with mono-integrated modulators and couplers, multiwavelength single-mode laser systems, monolithic semiconductor sources of femtosecond laser pulses, and tunable semiconductor lasers with ultra-wide tuning ranges. The growth of nano-objects such as quantum dots [37] and nanowires is also induced by metal–organic chemical vapor deposition and molecular or chemical beam epitaxy. Epitaxial growth is produced using a passivating mask, which forms areas that suppress growth deposited on the substrate. Then, the epitaxial growth is produced in mask windows. The geometric dimensions of the mask and windows depend on the composition and properties of the grown epitaxial layers due to mass conservation during the growth process. For the first time, experimental and theoretical studies have been carried to evaluate the growth of layers of AlGaAs/GaAs solid solutions obtained using selective area epitaxy in ultrawide windows. The operating pressure in the reactor of the setup for epitaxial growth was 77 Torr. The samples were grown for 10 min on an n-GaAs (100) of 2 inches in diameter at a temperature of 750 °C and a rotation speed of 1000 rpm in hydrogen ( $\text{H}_2$ ) (carrier gas). Trimethylgallium and trimethylaluminum were the atoms sources. Two sample types were grown epitaxially.

- One was grown without a mask (standard epitaxial growth).
- The second was grown with a mask of  $\text{SiO}_2$  stripes of 1000 Å in thickness and 100  $\mu\text{m}$  in width (selective area epitaxial growth).

Four  $\text{Al}_{z_0}\text{Ga}_{1-z_0}\text{As}$  samples of each type were produced, with the following compositions ( $z_0$ ) and growth rates:

- $z_0 = 0$ , growth rate of 200 Å/min.
- $z_0 = 0.11$ , growth rate of 225 Å/min.
- $z_0 = 0.19$ , growth rate of 247 Å/min.
- $z_0 = 0.3$ , growth rate of 286 Å/min.

During the initial production stage of the second type of samples, alternating stripes of the mask were produced by ion-plasma sputtering. The pattern was oriented in a [011] direction and produced by lithography and etching in a buffered oxide solution (buffered

oxide etch 5:1). The patterned substrate was annealed at 750 °C for 20 min in the arsine flow ( $\text{AsH}_3$ ) prior to the AlGaAs layer growth. The deposition of polycrystals with linear dimensions that did not exceed 100 nm was observed. The density of polycrystals slightly decreased towards the mask/window interface. A negligible number of polycrystals were detected in the mask area within 1  $\mu\text{m}$  of the mask/window interface. A certain threshold concentration on the mask face, above which heterogeneous nucleation occurred, was observed for each reactant species. The threshold was higher for mask areas with improved roughness than those with high roughness. Polycrystals precipitate when one of the threshold concentrations is exceeded. The areas of the mask/window interface where nucleation did not occur were observed. The absence of polycrystals was observed on the surface of one of the samples. The microphotoluminescence spectra for samples of the second type were studied with a spatial resolution in the window region. The experimental data were compared with the simulation results obtained by the developed vapor-phase diffusion model. The mass diffusion constant and a surface reaction rate constant ratios were 85  $\mu\text{m}$  for Ga and 50  $\mu\text{m}$  for Al, which suggested that the proposed model could be used to predict the properties of the layers, such as the growth rate, layer thickness, and the layer composition distribution in the development of heterogeneous multilayer structures and optoelectronic devices.

Colloidal nanoparticles, such as  $\text{AgInS}_2$  quantum dots [37], are a new material that improves the functionality of many sensory and electronic devices. The most relevant issue associated with the development of colloidal nanoparticle synthesis is adapting the developed technology to real manufacturing conditions. The new method proposed in this study is an additive printing of chips for microfluidic synthesis [63,64]. This method reduces costs, and it can be scaled and automated to increase productivity by up to 60% and improve optical properties, such as the position, shape, and width of the photoluminescent band and the photoluminescent quantum yield of quantum dots. An increase in the synthesis temperature of the  $\text{AgInS}_2$  quantum dots led to a linear increase in photoluminescent quantum yield. The photoluminescent quantum yields of samples synthesized in the microfluidic chip at 40, 60, and 90 °C were 0.9, 1.8, and 3.6%. Since the samples produced by microfluidic synthesis of the  $\text{AgInS}_2$  quantum dots at 90 °C exhibited the most significant photoluminescent quantum yield, they were synthesized at this temperature for 18 and 180 s to evaluate their optical properties and yield. The new method of flow hydrothermal synthesis for three-component  $\text{AgInS}_2$  quantum dots by additive manufacturing, resulting in the formation of microfluidic chips, demonstrated the applicability of photopolymer resin-based chips without noticeable defects of the crystalline lattice and the degradation of mechanical properties that can negatively influence microfluidic chip channels. The microfluidic chip had a significantly greater mass and heat transfer coefficient than the conventional flask reactor. The photoluminescence quantum yield of samples synthesized by the developed technological method for 18 and 180 s was about 2.5 times higher than that of samples synthesized in laboratory conditions (in a flask).

Two-dimensional materials, particularly graphene [38], have recently become relevant in light of their specific carrier mobility in electronic device applications. Graphene layers are produced physically [65] and chemically (CVD and epitaxial) [66] on a metal, semiconducting, and insulating material basis. The chemical-based growing methods are considered to be the most promising for providing high-quality graphene to meet the high demands of the market. The most technologically promising graphene-growth method for a SiC substrate was achieved on Si- and C-faces. The type of face influences the properties of the graphene. The growth of the graphene on the Si-face leads to the formation of a C buffer underlayer between the graphene and Si-face. Charge transfer and substrate interaction of graphene grown on the Si-face affect its electronic properties compared to the C-face, which usually exhibits increased carrier mobility that depends on the layer's quantity. The most critical issue associated with graphene growth on the C-face is the thick layers and the inability to accurately monitor their number. Changes in the surrounding temperature can influence the properties of atomically thin graphene, making

it more attractive for the sensory industry. A change in wetting properties is noted during surface functionalization, but the results of many studies on this topic are controversial. The primary sources of inconsistent graphene properties are surface charge, defects, and absorption of species (adsorbates). The adsorption of ambient molecular species on graphene surfaces leads to doping and changes in graphene's electrical and optical characteristics. However, when this process not properly controlled, it can lead to degradation, affecting the stability and reliability of the graphene-based device. The single-atom-thick graphene surface can absorb bulk aerosol and moisture contamination in the air. When exposed to moisture, graphene adsorbs contaminants on open surfaces at a temperature below the room's ambient temperature. It is important to keep such surfaces clear in order to maintain the properties of highly sensitive graphene. A change in the Raman spectrum of epitaxial graphene upon cooling due to moisture condensation can influence the evaluation of the material's quality. Raman spectroscopy is one of the most accurate research methods for detecting grown graphene contamination. A semi-insulating 6H-SiC substrate doped with vanadium (V) with a thickness of 369  $\mu\text{m}$  was studied. The substrates were polished on both Si and C faces. The experimental graphene was produced using a furnace at a temperature of 1550  $^{\circ}\text{C}$  on the atomically flat 6H-SiC substrate for 25 min in a  $<10^{-10}$  Torr vacuum. Si evaporates from the substrate at a high temperature when carbon atoms form graphene. Optical images of the graphene surface grown on the carbon face of the 6H-SiC substrate were obtained via an optical microscope fixed to a Raman spectrometer ranging from room temperature to  $-180^{\circ}\text{C}$ . A Raman spectrometer featuring a 532 nm excitation laser with a spot of 2  $\mu\text{m}$  was utilized for the temperature-dependent spectra of the graphene. The moisture on the epitaxial graphene on the 6H-SiC substrate was evaluated by comparing Raman peaks with ice. Peaks in the 500–750  $\text{cm}^{-1}$  frequency range and at  $\sim 1327\text{ cm}^{-1}$  were considered to be indicators of airborne contaminants. At the same time, a wide peak at  $\sim 1327\text{ cm}^{-1}$  was observed at room temperature due to water spots on the sample. This peak is of key importance, since it can be mistakenly considered as a D band of graphene when it is observed at lower than room temperature and is associated with graphene defects. The study emphasizes the importance of using Raman spectrometer to investigate graphene below room temperature and its moisture insulation.

The authors of [39] focus on the innovative technology of  $\beta$ -SiC film synthesis on a mono-silicon substrate by integrating porous silicon (por-Si) as an intermediate layer that increases adhesion between the film and substrate ( $\beta$ -SiC/por-Si/mono-Si nano-objects). SiC films deposited on the insulating ceramic substrate can also be used in graphene synthesis [67]. The results are relevant to the development of the semiconductor industry. The morphology showed that the produced SiC film possessed agglomerates of 2–6  $\mu\text{m}$ , with 70–80 nm pores observed on those agglomerates. The synthesized  $\beta$ -SiC/por-Si/mono-Si heterostructure had crystallographic orientations (hkl) of (111) and (220) for Si and SiC corresponding to crystalline structures. X-ray diffraction analysis showed a shift toward lower angles (the peak at  $2\theta = 35.6^{\circ}$ ), indicating quantum size effects corresponding to the nano-objects. The peak at an angular position of  $2\theta = 35.6^{\circ}$  corresponds to  $\beta$ -SiC in a zinc-blend-type lattice, confirming the structural integrity and crystallinity of the SiC layer. A synthesized heterostructure is required for photodetectors, light-emitting diodes, and sensing technologies. The thermal conductivity of SiC and the insulating properties of por-Si can be used in advanced electronics. Due to the effects of quantum size, the proposed film-synthesizing approach can also be used in quantum computing. The proposed methodology substantially improves the lattice mismatch and adhesion that hinders conventional synthesis on the Si-substrates. Future development of the approach lies in the field of optimal porosity of the intermediate layer, mechanical properties in thermal and electrical conditions, and application of heterostructure in electronics and optoelectronics.

Another study included in this Special Issue proposes a new setup that can be used to remove toxic elements, such as arsenic (As), from water [40]. Many technologies have been developed to remove arsenic from drinking water [68,69], including oxidation (photochemical, photocatalytic, biological etc.) techniques, membrane-based technologies (micro-, ultra-



and nanofiltration, or reverse osmosis), coagulation/flocculation, ion exchange, adsorption onto solid media (activated  $\text{Al}_2\text{O}_3$ , Fe-based sorbents, zerovalent iron ( $\text{Fe}^0$ )), indigenous filters, miscellaneous sorbents, and metal–organic frameworks. One technique involves adsorption onto iron media, with regular replacement of the adsorbents. The formation of iron oxide/hydroxide ( $\text{FeO}(\text{OH})$ ) consists of a chemical reaction, a dehydration phase, and a grinding/granulating phase. Iron oxide/hydroxide can be obtained by combining a  $\text{Fe}^{+3}$  ion with an  $\text{OH}^-$  ion. The necessary amount of time is allotted for the  $\text{Fe}(\text{OH})_2$  to flocculate based on the reaction of  $\text{FeCl}_3$  or  $\text{Fe}_2(\text{SO}_4)_3$  reagents, and then it is passed through a filter press. The resulting sludge is dehydrated according to the following techniques: freezing and non-mechanical separation with translucent and granular sludge comprising 50% water, followed by thermal drying in a rotary drum or belt dryer until only 20% of the water remains. The next phase involves grinding/granulating the sludge, such as  $\text{Fe}(\text{OH})_2$ . The resulting media is used to remove arsenic from water. The principle of adsorption on  $\text{Fe}(\text{OH})_2$  involves a reversible chemical exchange, which proves its effectiveness in removing arsenic and other substances from water; this is supported by many studies and has been accepted on an international basis. However, this method of replacement is quite costly. Adsorptive media technology requires replaceable Fe-based media that can only be used once. The replacement cost comprises approximately 80% of the overall service cost. Thus, a new portable setup based on the principles of iron media regenerating was developed and tested in Central Italy. In 2019–2023, the proposed system was used to regenerate iron media to restore the system's ability to adsorb arsenic in water. The legal threshold of As-content in water is  $10 \mu\text{g}/\text{L}$ . When the level of arsenic concentration in water exceeds this threshold, iron media regeneration occurs, and the arsenic concentration in water is minimized. A system that can regenerate the media to make it more economically profitable is highly sought after by the industry. The advantages of this newly developed approach are the renewing of the filter bed with the restoration of its adsorption capacity, the absence of solid waste, the absence of disposal costs, the positive impact on the environment, the reduced service time, the fact that no equipment needs to be replaced, elimination of media production, and related material and transport costs.

Distilled water and groundwater were subjected to low-temperature plasma produced by barrier and bubble discharges [41]. Research on the effect of non-equilibrium low-temperature plasma of electric discharges in the air on water and aqueous solutions is of interest to the industry, particularly with regard to the development of installations for plasma-activated water (PAW) production [70,71]. PAW results from plasma action in water or aqueous solutions (e.g., phosphate-buffered saline, etc.) in the presence of oxygen  $\text{O}_2$  or a mixture of  $\text{O}_2+\text{N}_2$  at atmospheric pressure. PAW is used in biofilm removal, wound healing, bacterial inactivation, and to increase seed germination rates and subsequently accelerate plants' growth, inactivate pathogens, rescue fungus infections, and preserve crops due to the reactive oxygen and nitrogen species (RONS), relatively short-lived radicals ( $\bullet\text{OH}$ ,  $\text{NO}\bullet$ ), superoxide ( $\text{O}_2^-$ ), peroxyxynitrate ( $\text{ONOO}_2^-$ ), and peroxyxynitrite ( $\text{ONOO}^-$ ). The stated effects on plant growth are attributed to the activity of water nitrates, nitrites, ammonium ions ( $\text{NH}_4^+$  or  $[\text{NH}_4]^+$ ), and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). PAW is considered to be a sustainable and promising solution for biotechnological applications due to the transient nature of its biochemical activity and the potential economic and environmental benefits of using ambient air rather than rare or expensive chemicals. This approach can potentially reduce the technological costs of growing plants when used in tandem with other biotechnologies to improve germination and further plant growth [72,73]. There are many methods of discharge treatment of water solutions to obtain plasma-activated water. The spread approach in which the barrier discharge is used consists of the pulsed arc and corona discharge that occurs directly in a water solution when the high-voltage electrode is covered with a polyethylene or ceramic dielectric (barrier) layer (the heterophase method). The overloading leads to the decay of the useful chemicals in water solution when the formation of active oxygen and nitrogen species is limited by the low concentration of  $\text{N}_2$  and  $\text{O}_2$  dissolved in water. Thus, water should be constantly saturated with the indi-

cated gasses or air to produce plasma-activated water in hydrodynamic setups. Another promising production method is based on the discharge that occurs directly in the volume and on the surface of air bubbles. Bubble discharges make initiating a discharge easier than electrohydraulic setups of heterophase methods. The pulse voltage is applied, and the type of plasma-forming gas determines the type of particles and their concentration. Devices that discharge treatment of water droplets or a thin water film to produce PAW have low productivity (measured in liters/hour). The types of electric discharges (barrier and in bubbles) considered in the study were chosen due to their prevalence in applied research. The discharges are constructively implemented and facilitate the development and production of technological installations. During the study, the ionic composition of two types (distilled and ground) of water treated with a low-temperature plasma formed by two pulsed discharges was revealed in atmospheric pressure air. After the exposure, the properties of both types of water, such as their magnesium and calcium ion concentrations, pH index, and electrical conductivity, were compared. The bubble discharge in groundwater showed maximum productivity for the  $\text{NO}_3^-$  anions. The barrier discharge in air, followed by water saturation with plasma products, is the most suitable for distilled water. The maximum energy input (thermalizing) into the stock solution is ensured in both treatments. From the point of view of energy consumption, both types of discharge treatment are suitable for obtaining approximately equal amounts of  $\text{NO}_3^-$  anions. This is a reasonably simple way to convert calcium carbonates ( $\text{CaCO}_3$ ) from insoluble to soluble calcium nitrates ( $\text{Ca}(\text{NO}_3)_2(\text{H}_2\text{O})_x$ ). Insoluble carbonates pass into soluble nitrates when interacting with  $\text{NO}_3^-$  anions. Treatment with discharges did not significantly affect their concentration of potassium and sodium cations ( $\text{K}^+$ ,  $\text{Na}^+$ ) in water; the content of potassium and sodium cations did not change during 10 min of exposure and amounted to 1.065 and 9.395 mg/L. Carbonates  $\text{K}_2\text{CO}_3$  and  $\text{Na}_2\text{CO}_3$  are soluble salts with electrical conductivity of 280  $\mu\text{S}/\text{cm}$  in water. As a result of the action of the discharge, additional  $\text{NO}_3^-$  and  $\text{NO}_2^-$  anions appear, leading to the formation of potassium and sodium nitrates ( $\text{KNO}_3$ ,  $\text{NaNO}_3$ ), which are also soluble salts. The complex compounds that affect the hardness of water, particularly  $\text{Ca}^{++}$  ions, are released into the solution. These features of the water treatment process using pulsed discharges should be considered when designing setups for industrial plasma-activated water production of groundwater for hydroponic plant growing technologies, in which a water solution enriched with  $\text{NO}_3^-$  anions is required.

The carbon dioxide-assisted polymer compression method is one of the methods used to produce porous polymer products with laminated sheets made of poly(ethylene terephthalate) fiber with a diameter of 8  $\mu\text{m}$  [42]. Polymer fibers are placed in a specific direction along the sheet, and the intersections of the fibers are crimped in the presence of  $\text{CO}_2$ , forming a porous structure [74]. This orientation in a porous material is anisotropic [75]. The anisotropy of the permeation behavior in carbon dioxide-assisted polymer compression porous materials is of interest to the production of the drug-loaded tablet [76] and was assessed based on the aspect ratio of the dye solution permeation of the fiber-spread direction via the fabric-lamination direction. Quantitative evaluation of the anisotropy of permeation was performed, and the phenomenon was understood by linking it to the structure of the sample. Experiments were conducted using limited conditions of dye solution permeation with a slow injection rate to emphasize and examine the anisotropy of the structure. For the actual design of the component, the permeation rate and the amount of permeation are essential considerations. A dye solution was syringed into the 80-ply and 160-ply laminated porous polymer products. The aspect ratio decreased steadily with a decrease in porosity (0.63 for the 80-ply laminated product and 0.25 for the 160-ply laminated product) and was evaluated as 2.73 and 2.33, respectively. A 3D structural analysis showed that as the compression ratio increases, the fiber-to-fiber connection also increases due to an increased quantity of adhesion points, resulting in a decrease in the anisotropy of permeation. The hypothesis that an increase in the number of oriented fibers per unit volume could increase the anisotropy was disproved. Cross-sections of the obtained porous polymer with high porosity, which were subjected to X-ray computed

tomography, showed less fiber-to-fiber bonding, and the number of fiber bonding points increased as the porosity decreased. Since permeation of the dye solution occurs along the fiber surface, more bonded fibers promote permeation between the upper and lower fiber surfaces, resulting in less anisotropy of permeation. Functional components, such as filters and tablets, are important industrial components. Therefore, structural anisotropy is essential when designing filters and drug-loading tablets using carbon dioxide-assisted polymer compression porous materials.

Information-analytical software has been developed in C# on the .NET framework by Mendeleev University of Chemical Technology of Russia (version 2.0) and is aimed to create digital models of structures of porous materials such as aerogels and new nanoporous materials to predict a set of properties (thermal and electrical conductivity, mechanical properties, sorption, and solubility) and pore size distribution [43]. Models facilitate the description of hydrodynamics of multicomponent systems, heat and mass transfer processes, dissolution, sorption, and desorption in processes in porous and nanoporous structures [77,78]. Digital models for different types of aerogels can be created. The pore size distribution was chosen as a criterion to compare the results obtained for each model with the experimental findings. The deviation of the resulting curves did not exceed 15%, showing a correlation between the digital and experimental results. The software includes both the existing and newly developed models. The existing models were used to model porous structures when the original models were developed for aerogels of silicon dioxide SiO<sub>2</sub>, silica–resorcinol–formaldehyde, polyamide, carbon, polysaccharides (chitosan, cellulose), and protein and related processes (the dissolution of active pharmaceutical ingredients and mass transportation in porous media). The developed models have a wide range of input parameters for each type of aerogel, considering the features of the current sample. The software allows for modeling processes such as hydrodynamics inside digital porous structures using the lattice Boltzmann method and the cellular automaton particle dissolution model. The lattice Boltzmann method can be combined with cellular automata models, which calculate sorption, mass transfer, and dissolution inside porous structures. Software modules can be expanded with new cellular automata and other discrete models. Aerogels of silicon dioxide, silica–resorcinol–formaldehyde, polyamide, carbon, chitosan, cellulose, and protein were developed with the suggested original information-analytical software. Their thermal and electrical conductivity, mechanical properties, sorption, and solubility were predicted. These models establish a connection between structure geometry and properties, allowing for the development of materials with the required properties, such as new nanoporous materials. They also facilitate cellular automata models (original developments and independent implementation of existing models) with wide possibilities for varying their parameters and adding new modules. The software can potentially reduce the required number of full-scale experiments and, consequently, the costs of developing new porous materials.

Additive manufacturing technologies allow the production of products of complex shapes made from various types of materials [44]. Many of those technologies based on using laser source, namely laser powder bed fusion, are limited by the physical properties of the used materials, such as the thermal conductivity of the surrounding medium, the internal stresses, and the warpage or product weight [79]. One study aimed to solve the problem of creating machine learning algorithms for the needs of additive manufacturing [80] to identify the product's hard-to-manufacture geometrical features. Four features were considered:

- An overhanging surface with an angle in the range of 10–70° and a length of the overhanging plane in the range of 10–25 mm (critical of over 45°).
- Fine walls and slits with a thickness of 0.1–15 mm (critical of 0.1–5 mm).
- Horizontal and vertical holes with a diameter of 2–15 mm (critical of 6–15 mm).
- Helix tube (critical in the whole range of sizes and shapes).

The segmentation of these features permits the application of different manufacturing strategies to improve production ability. The algorithm is trained based on laser bed fusion

of stainless steel. It identifies simple geometrical features which are hard to produce. The developed approach allows the treatment for the new product to be manufactured by laser powder bed fusion with 88% efficiency. A database containing basic and hard-to-manufacture geometrical features was generated during the study. Every identified feature received its production limitations. Convolutional neural network architecture was trained to identify critical geometrical features and was introduced into the developed database. During testing, the developed algorithm produced segmentation of the feature and recognized untrained complex shapes, such as helix tubes. The approach confirmed its efficiency in complex geometry segmentation. This approach can be improved by the topology indexation and the definition of the algorithm input space. The output data of the developed classification are a collection of 3D geometries representing the uncritical basic volume part and the critical additive manufacturing features. The databases can be expanded and retrained for other technological processes and materials by defining of a new set of features. The study proposes the use of a new file format for additive manufacturing technologies that can be enriched with the necessary 3D feature data, such as .3mf. The open-source XML-based file format can include the features in a file which will be automatically processed by a build processor. Further development of build processors is necessary to adapt to the newly proposed 3D part processing.

### 3. Conclusions and Outlook

This Special Issue investigates various types of technologies, devices, and approaches used in the creation and manufacturing of innovative and progressive materials (including low-dimensional systems such as 0D–2D objects—quantum dots, quantum wells, graphene, etc.). The following critical aspects of the development of the new and improved industrial productions are also addressed.

- The surface quality of additive manufacturing parts of stainless steels and a wide range of alloys, their explosive ablation, ion polishing in gas-discharge plasma, and coating deposition [31].
- The surface quality of SiAlON after diamond grinding and diamond grinding–lapping–polishing and prior to double-layer trinitride and DLC coating deposition, and their effects on the durability of the cutting insert in turning nickel superalloy [32].
- Advanced electrical discharge machining of alumina, which is achieved using assistive coating and powder suspension [33].
- Shallow (at  $-20\text{ }^{\circ}\text{C}$ ) and deep (at  $-196\text{ }^{\circ}\text{C}$ ) cryogenic treatment of magnesium nanocomposite produced through disintegrated melt deposition followed by hot extrusion [34].
- Mechanical alloying of two nanocrystalline soft magnetic Fe-based nanoperm-type alloys using Fe-Zr-B-Cu composition to produce low-dimensional systems (0D–2D objects) [35].
- The use of metal–organic chemical vapor deposition selective area epitaxy using an  $\text{SiO}_2$  mask with ultrawide () windows ( $100\text{ }\mu\text{m}$ ) on a GaAs substrate to produce strained quantum wells (0D objects) [36].
- A new manufacturing method for synthesizing  $\text{AgInS}_2$  quantum dots (0D objects) in a 3D-printed microfluidic chip [37].
- Insulation of 2D material (a 2D object), such as epitaxial graphene chemically grown on 6H-SiC substrates, from moisture that adsorbs contaminants under cooling conditions (from  $20\text{ }^{\circ}\text{C}$  to  $-180\text{ }^{\circ}\text{C}$ ) and the influence of the moisture on its properties [38].
- Thermal synthesis of carbide ceramic ( $\beta\text{-SiC}$ ) film on a silicon substrate using porous silicon as an intermediate layer for creating nano-objects [39].
- Water purification technology which removes arsenic via regenerating iron-based adsorptive media [40].
- Plasma-activated water generation of distilled water and groundwater by barrier and bubble-pulsed discharges for growing plants [41].

- Anisotropy of the permeation behavior in carbon dioxide-assisted polymer compressive porous materials [42].
- Analytical software for digital models of porous and nanoporous structures such as aerogels to create new materials based on SiO<sub>2</sub> [43].
- Machine learning algorithms that are used to identify the critical geometrical features produced by laser powder bed fusion of stainless steel [44].

Most of the proposals related to the creation of innovative materials and technologies for processing and production have huge industrial potential and are scalable or, in some cases, are already suitable for serial implementation. This was one of the key considerations of the Guest Editors when selecting articles for publication in this Special Issue. The developed technologies and approaches are expected to be introduced into modern production to accelerate the transition to the sixth technological paradigm.

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