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Structural and functional nanocrystalline materials: A Review

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Abstract

Structural and functional nanocrystalline materials have garnered significant attention in materials science due to their unique properties and potential applications. Nanocrystalline materials are characterized by grain sizes on the order of nanometers, leading to enhanced mechanical, electrical, optical, and magnetic properties compared to their bulk counterparts. This review explores the synthesis methods, structural characterization techniques, and functional properties of nanocrystalline materials. Various synthesis routes, including mechanical alloying, sol-gel methods, and chemical vapor deposition, are discussed, along with their effects on grain size, composition, and phase stability. Structural characterization techniques such as transmission electron microscopy, X-ray diffraction, and atomic force microscopy are crucial for understanding the grain structure, defects, and phase transformations in nanocrystalline materials. Furthermore, the functional properties, including mechanical strength, electrical conductivity, optical transparency, and magnetic behavior, are elucidated with a focus on the relationship between grain size, microstructure, and performance. Applications of nanocrystalline materials in diverse fields such as catalysis, sensors, energy storage, and biomedical devices are highlighted, showcasing their potential for technological advancements. Finally, current challenges and future perspectives in the synthesis, characterization, and utilization of structural and functional nanocrystalline materials are discussed, emphasizing the need for interdisciplinary research efforts to unlock their full potential in various applications.

Keywords: nanocrystalline materials, nano structuring, synthesis techniques, mechanical properties, Hall-Petch connection,

Full length article

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Introduction

In materials science, nanocrystalline materials are an exciting new field with an excess of creative applications potential for many different fields [1]. These materials differ from their conventional counterparts due to their distinct structural and functional features, which are characterized by their crystalline grains that are nanoscale in size. In this review, we dig into the complex realm of nanocrystalline materials, examining their various functional capabilities and their structural features [2]. In terms of structure, NC materials have a great concentration of grain boundaries that greatly impact their mechanical, thermal, and electrical properties [3-6]. These interfaces prevent dislocation motion, which results in extraordinary strength, hardness, and wear resistance [7]. As a result, nanocrystalline materials are excellent choices for advanced structural applications in the biomedical, aerospace, and automotive industries [8]. Moreover, the finely tuned grain structure improves diffusion kinetics, enabling quick solid-state reactions and new processing avenues for the synthesis of complex materials with customized properties [9]. Because of their distinct nanoscale dimensions and interfaces, nanocrystalline materials have a wide range of functional characteristics in addition to their structural characteristics [10]. These materials show exceptional optical transparency, magnetic behavior, electronic transport capabilities, and catalytic activity, creating opportunities for a variety of uses in information technology, environmental remediation, energy conversion, and sensing [11]. Additionally, the ability to precisely manipulate grain size, composition, and doping to tune these attributes presents never-before-seen possibilities for the development of cutting-edge materials with improved functionality and performance [12].

The aim of this review to give a comprehensive summary of the recent advancements in the synthesis, characterization, and applications of structural and functional nanocrystalline materials [13]. By elucidating the underlying principles governing their behavior at the nanoscale, we seek to uncover the potential avenues for harnessing their unique properties towards addressing pressing societal challenges and driving technological innovation. Through a systematic examination of the key developments in this field, we endeavor to inspire further exploration and interdisciplinary collaboration aimed at unlocking the full potential of nanocrystalline materials in shaping the future of materials science and engineering [14].

2. Classification

Nanostructured materials divided into four groups based on how many dimensions they had: 3D: Equiaxed bulk solids; 0D: nanoclusters; 1D: multilayers; 2D: nanograined layers given below in the table 1 [15]. Throughout this review, we will mostly concentrate on 3D equiaxed bulk solids. Nanocrystalline coatings are not something we will use. On the other hand, one-dimensional nanostructures called nanowires have significant electrical characteristics [16].Grain size is another factor that can be used to classify materials: materials with ultrafine grain sizes have grain sizes over 500 nm (often in the sub-micrometer range) while materials with nanograined sizes have grain sizes below 500 nm, typically in the range of 100–200 nm. Nanomaterials are further categorized as NM's crystallized from amorphous solid or nanomaterials created from other ways where the initial material is usually crystalline, depending on the starting material from which they are made [17].

3. Synthesis of NC materials

One can create nanocrystalline materials by either dissolving the polycrystalline majority material into crystal-like components the size of nanometers or by consolidating tiny clusters. These techniques are distributed into bottom up and top down categories. The nanostructure must be arranged layer by layer and atom by atom using the bottom-up method. By breaking down the microstructure into a nanostructure, we begin with the bulk material in the top-down method [18]. The following are the main synthesis techniques given below in the figure 1.

3.1 Mechanical alloying

Through extreme plastic deformation and structural fragmentation of coarse-grained arrangement, ball milling creates nanostructured materials. Powder particles are repeatedly distorted till the preferred arrangement is attained in a dry state. This process is known as mechanical alloying. Throughout this technique, mixtures of fundamental or pre-alloyed powders are crushed in apparatus with high-energy compressive effect pressures, like erosion grinders, shaker grinders, and ball grinders, in a protected environment [19].The setup for the ball milling operation is shown in Fig. 2. It has been demonstrated that, given enough grinding time, practically any material may yield grains the size of nanometers. The size of the grains is inversely proportional to melting temperature. By increasing the melting period the size of the grains also increases [20].

3.2 Inert gas condensation

The process of IGC involves the evaporation of a metal through various approaches like resistive warming, radiofrequency warming, spluttering, electron beam warming, laser/plasma warming, or ion spluttering [21]. Condensed fine particles are transported to the collector device by convection currents, which are produced when the inert gas is heated by the vaporization source and chilled by the liquefied nitrogen-filled collection device (cold finger) [22]. Scraped off deposit is placed in a compaction mechanism. Two steps are involved in the process of compaction: (a) low pressure pellet compacting and (b) high pressure space compaction. In order to limit the amount of trapped gases and maintain the cleanliness of the particle surfaces, the scraping and compaction procedures are conducted under ultrahigh vacuum settings. Equiaxed (3D) crystallites are created by the inert gas condensation process [23]. The powder typically has a few nanometer sized crystal and a restricted size distribution. The crystal size is dependent on the noble gas pressure, the degree of evaporation, and the gas composition [24]. It is possible to make very small particles by using light inert gases (like Xe) instead of heavy ones and by dropping the air density in the space or the evaporation rate [25]. Many of the early studies on the mechanical properties of NC materials used the IGC process as a key instrument. One drawback is the potential for porosity and powder contamination as a result of inadequate association [26]. Since most early work employed cold consolidation, there is also a chance of poor particle bonding. However, the outcomes acquired with specimens processed in this manner laid the groundwork for our comprehension [27]. Another method for creating nanocrystalline alloys is to evaporate the various metals from multiple evaporation sources. A better vapor mixing can be achieved by rotating the cold finger. The metals can be made into oxides, nitrides, carbides, etc. by keeping the environment carbonaceous or by adding oxygen or nitrogen gases to the chamber. Additionally, metastable phases are also formed at sufficiently small particle sizes [28].This makes it possible to synthesize a wide range of nanocrystalline materials using this approach. The measured peak densities of the metal samples in their as-compacted state are approximately 98.5% of their bulk densities. On the other hand, it has been demonstrated that porosity significantly affects mechanical strength, particularly in tension [28]. The compressive yield stresses of the IGC-synthesized nanocrystalline Pd and Cu samples are summarized in the table 2. When relating the strength of the NC Cu and Pd samples to their coarse-grained equivalents, Weertman et al. discovered a significant density dependence. They were also more likely to fail. Suryanarayana discovered that their strongest micro Cu sample had a compressive yield strength of roughly 500 MPa. The Vickers hardness values are shown in the table as Hv divided by 3, which generally translates to the yield strength in the case of little work-hardening. Unlike the ductile yield strength scenario, the compressive values of σy scale well with Hv/3. Weertman reported that a notable rise in toughness was observed in the NC Cu and Pd samples that were created [29].

3.3 Electrodeposition

The electrodeposition approach produces a wide range of nanograin materials, including pure metals, mixtures, and intricate system with particles as small as 20nm, as compared to other methods. It also has the advantage of being inexpensive, producing great manufacture proportions, having few size and shape restrictions, and having a high likelihood of being transferred to the coating and electroforming industries already in place [30]. Erb et al. have been researching the synthesis, arrangement and characteristics of (NC) Ni produced using pulse electrochemical deposition over the last few years. They showed that, in comparison to traditional polycrystalline nickel, distinctive and frequently better features arise from particle improvement of electroplated Ni into the nanoscale range [31]. By carefully controlling the agitation and electrical conditions, it is possible to produce multilayered (1D) metal electrodeposition using one electrolyte, which is far more convenient than utilizing two separate electrolytes. This technique can also be used to create 3D nanostructure crystallites by leveraging the interface between one ion and the other's deposition [32].

It has been revealed that electrochemical deposition produces particle sizes in the nanoscale range when the variables are selected so that the crystallization of fresh particles is preferred over the progress of current particles [33]. Greater accumulation amounts, the creation of acceptable composites in the immersion, the addition of appropriate surface-active elements to lessen ad-atom external dispersion, and other techniques were used to achieve this [34]. Porosity-free final goods that don't need further consolidation processing can be produced with this method. In addition, this technique offers high production rates with little shape and size restrictions and minimal capital expenditure [35].

3.4. Characterization of NC Materials

In contrast to their bulk counterparts, nanocrystalline materials have distinct structural and functional characteristics due to their grain structure, which is defined by nanometer-sized particles. This is a quick summary of the characterization techniques that are frequently used to comprehend their structural and functional features. TEM provides high-resolution images to visualize individual grains and defects within the material. It allows for the measurement of grain size, grain boundary structure, and phase composition. SEM offers detailed surface imaging and topographical information. It is useful for observing grain morphology and distribution. XRD determines crystallographic structure and phase composition. It provides information on grain size through analysis of diffraction peak broadening.

4. Mechanical characteristics of alloys and metals with nanocrystals

This section reviews the primary motorized characteristics of (NC) metals, including yield stress, ductility, strain hardening, strain-rate sensitivity and active reaction, sneak, and weakness. It is necessary to note right away that porosity can conceal and/or distort characteristics, making it crucial. Early "bottom-up" synthesis techniques frequently left the grains with porosity and insufficient bonding [35].

4.1Strength of yield

It is well recognized that grain size significantly affects a material's mechanical behavior, particularly its yield stress [36]. In the usual polycrystalline range, the relationship between yield stress and grain size in metals is well-established (micrometer and bigger sized grains) [36]. Complete unhygienic density testing and micro- or nano-indentation, the mechanical characteristics of FCC metals with nano-range grain sizes have been evaluated. In order to minimize the impact of flaws such gaps that could negatively affect the material's mechanical response, micro-size tensile samples are frequently utilized [36]. Yield stress, σ_y, for materials with grain size *d*, is found to follow the Hall–Petch relation:

$$\sigma_y = \sigma_0 + kd^{-1/2}$$

Where k is a constant and σ0 is the friction stress. The power equation with exponent −n, where $0.3 \leqslant n \leqslant 0.7$, is a more general formulation that can be used instead of this approximation.

4.2 Ductility

In the traditional grain size regime, ductility typically increases as grain size decreases. Therefore, as the particle dimension gets closer to the nanoscale, one should anticipate a rise in malleability. Nonetheless, for the majority of particle sizes less than 25 nm, the ductility is low for metals that have tensile ductilitility between 40 and 60% elongation in the standard grain size [37].According to Koch there are three main reasons why nanocrystalline materials have poor ductility: (1) processing artifacts (such pores); (2) ductile uncertainty; and (3) crack nucleation or shear instability. Processing nanostructured materials without the artifacts that obscure their intrinsic mechanical qualities is challenging [38].

4.3 Hardening of the strain

Materials with ultrafine grains and nanocrystalline structure typically cannot withstand homogeneous tensile elongation. Several investigations indicate that, following an early period of fast stress hardening across a minor malleable pressure system (∼1-3%), essentially no strain hardening occurs. This reaction is distinct from that of coarse-grained polycrystalline metals. Due to either dynamic recovery or annihilation of displacements within the particle frontiers, the thickness of displacements in a NC sample saturates [39].

5. Functional Nanocrystalline Materials

5.1 Electrical properties

When grain boundary scattering is taken into account, the electrical resistivity of NC materials is found to be higher than that of coarse-grained materials with an identical chemical composition. This is due to the increased volume part of particles located at the grain boundaries. Additionally, it has been demonstrated that the electrical resistivity of NC materials is susceptible to several forms of synthesis-related defects and/or stressors in addition to particle frontiers. The electrical resistivity rises with decreasing grain size at constant temperature and increases with temperature for constant particle proportions, both of which are compatible with the theoretical explanation of electron scattering by grain boundaries. By adjusting the particle proportions of the electrically showing part, nanocomposites' electrical resistance (and hence conductivity) can be modified. For instance, the electrical conductivity in a nanocrystalline iron-silica combination can be altered by 14 orders of magnitude by varying the volume proportion of iron particles [40].

5.2 Magnetic properties

Saturation magnetization measurements M s of nanocrystalline iron (a-Fe) showed a decrease from 220 e.m.u., g-1 to roughly 130 e.m.u., g-1. The crystal size of the iron is 6 nm. M s is only decreased to roughly 215 e.m.u, g-1 in metallic iron glasses (extrapolated to pure iron), indicating a distinct atomic and magnetic structure for glassy and nanocrystalline (NC) iron. The three commonly observed magnetic transitions are replaced by a unique low-temperature transition to superparamagnetic behavior with nanocrystalline erbium (grain size 10-70 nm). On the other hand, with nanocrystalline (NC) erbium with greater grain dimensions, the typical magnetic transitions resurface at a different temperature and the low-temperature superparamagnetic behavior endures [41].

5.3 Catalytical properties

Furthermore, NC materials are employed in chemical reactions as catalysts. Bauer et al.'s experiments [42] produced NC PtSn, PtPb, PtBi, $FePt_3$, PtSb, PtSn, and Cu_3Pt by reacting NC platinum, $Al2O_3$, and CeO_2 metal with metal salts. Directly in the solution, these chemical reactions produced alloys and nanoparticles that had the catalytic ability to start or speed up the chemical reaction [43].

5.4 Optical properties

5.4.1 Materials' Optical Characteristics, Including Quantum Structures

Models employed to examine the optical characteristics of nanostructures are often depends on EM theory; however, quantum processes need to be taken into account when a semiconductor nanocrystal's diameter is less than the de Broglie wavelength. The electron wavefunction's coherence length is known as the de Broglie wavelength [44].Numerous studies on quantum structures are conducted in various labs due to their intriguing electrical and luminous characteristics. Quantum dots (QDs) are structures with a three-dimensional spatial resolution of only a few nanometers. Because the energies of the electron and hole can only take on specific values, QDs behave like atoms [45]. The size of the QDs and the characteristics of the semiconductor being employed affect both their absorption and luminescence spectra. With photoluminescence quantum efficiency reaching 80%, they have the potential to be incredibly effective light emitters [46]. Numerous studies on quantum structures are conducted in various labs due to their intriguing electrical and luminous characteristics. Quantum dots (QDs) are structures with a three-dimensional spatial resolution of only a few nanometers. Because the energies of the electron and hole can only take on specific values, QDs behave like atoms [45].In addition to already-existing uses, like multiple quantum wells63 for lasers and detectors, the research of quantum structures will lead to further significant uses in the future. QDs can be utilized, among other things, to create biological tags. They have been utilized more recently to create white LEDs with good efficiency [47].

5.4.1 Photonic crystal

Light is either transmitted or reflected through the sample in the case of optical coatings. Conversely, diffraction structures are typically employed to controllably disperse light in various directions throughout the space. Control over the propagation directions and spectral dispersal of optical waves in universe is made possible by the combination of diffractive and interference structures in three-dimensional parts. Specifically, the photon lifetime can be used to modify the propagation in the structure and the local field. The structure's normal dimensions are in the range of a partial wavelength [48]. These materials with periodic structure are also called photonic crystals because light waves act in a way that is comparable to the wave function of electrons in crystals. Utilizing the thickness of optical methods, which Purcell defined as the thickness of modes for electrons in solids, is another method to explain the behavior of light in such materials [49]. Metamaterials with optical qualities that are unattainable with nanostructured materials include photonic crystals. For instance, the structure may act as though it were made of a substance with a refractive index of zero or even lower. The research of metamaterials is a focus of many labs and businesses because they have the potential to be used in key applications in the near future, such as cloaking, stealth, or completely stigmatic optics. It is intriguing to think about multilayer mirrors as both a diffractive element and an interference system [50].

6. Applications

Materials are a sophisticated class of materials with improved mechanical qualities that are simple to make and treat in large quantities. These characteristics make NC materials useful for an extensive variety of applications, like those in the chemical, aerospace, structural, nuclear, and automotive industries. A few NC material applications are depicted in Figure 3. Automobiles and aircraft can be made with high-strength, lightweight materials like NC Aluminum, which increases their load-bearing capacity and reduces fuel consumption significantly. NC materials are helpful in military and defense applications outside of the automobile industry, such as ballistic materials and armored military vehicles [51].

6. Magnetic application

The enhancement of magnetic characteristics in magnetic materials can be greatly facilitated by nanostructuring. Numerous inquiries have also been carried out to observe the magnetic characteristics of nanocrystalline (NC) materials for magnetic applications when the base metal is Fe or Ni. Additionally, scientists have been working on the synthesis of nanocrystalline (NC) superconducting ceramics that would use the motorized vibrations of aeroplanes to generate electricity. Both sensors and semiconductor applications make extensive use of NC materials [52].

6.1 Chemical applications

Additionally, they investigated the optical characteristics of nanoparticles and suggested uses for chemical sensing and solar cell technologies. Utilizing thin films for windows made from CdS chemical precipitation which can function as planetary cell is another usage for solar technology. Reports on the erosion behavior of NC nickel-base alloys have been published [53]. The typical amount of Ni dissolution was found to be greater than that of the coarse-grained material, as anticipated. Due to their fine particle dimension and similarity, the nanocrystalline (NC) materials, on the other hand, showed superior localized corrosion resistance. Grain mass and sponginess in the samples can be precisely regulated to obtain the total surface area available, since most synthesis processes yield nanocrystalline materials in powder form. The normal amount of Ni dissolution was found to be greater than that of the coarse-grained material, as anticipated [53].

6.2 Sensory application

Optical characteristics of nanoparticles and suggested uses for them in chemical sensing and solar cell technologies [54]. Another application for solar technology is the production of thin films for windows from CdS chemical precipitation, which can also serve as solar cells. NC Ni for (micro-electromechanical system) MEMS usage in the automobile industry was produced by Baghbanan et al. Using the electrodeposition technique [42].To describe the mechanical characteristics of the micro-systems, they ran a number of tests.

Table 1. Different types of Nanocrystalline materials.

Designation	**Dimensionality**	**Typical methods of synthesis**
Clusters	Zero dimensional	Sol-gel method
Layered (lamellar)	One dimensional	Vapour deposition
Filamentary	Two dimensional	Chemical vapour deposition
Crystallites Equiaxed	Three dimensional	Gas condensation Mechanical alloying

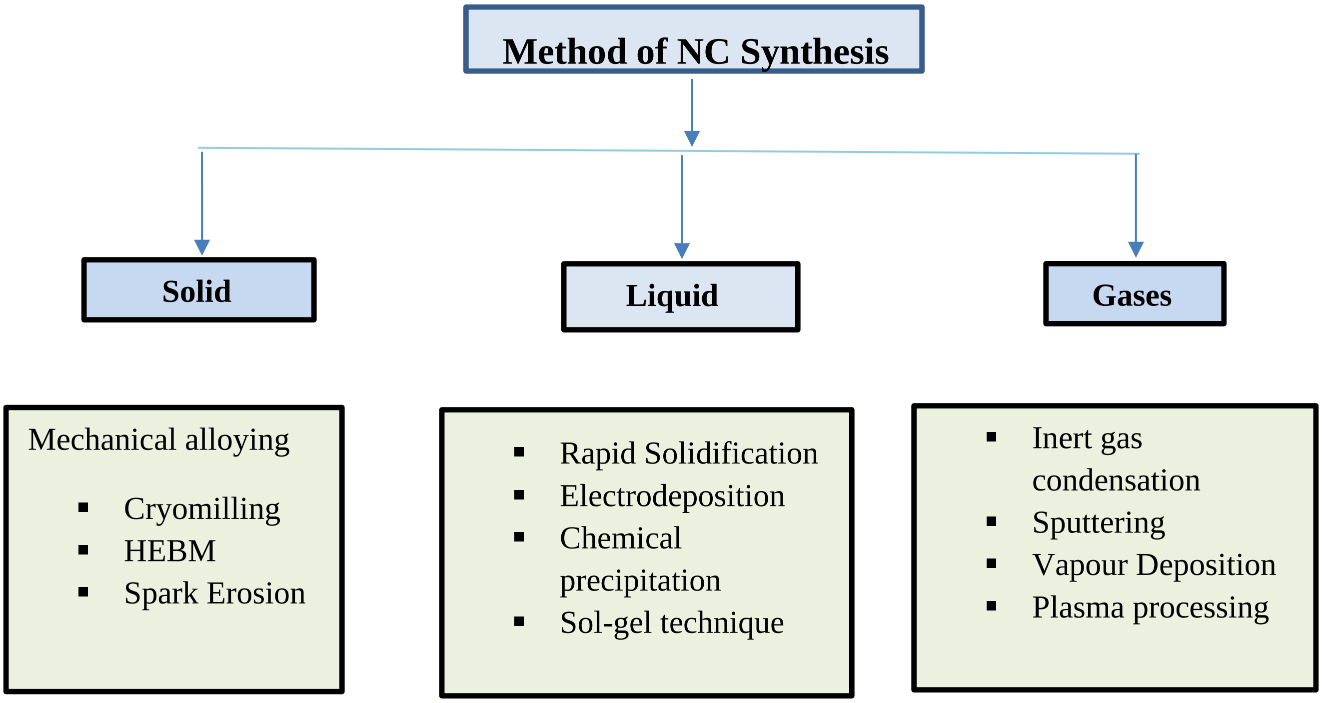

Figure 1. Different techniques for synthesis of NCM's

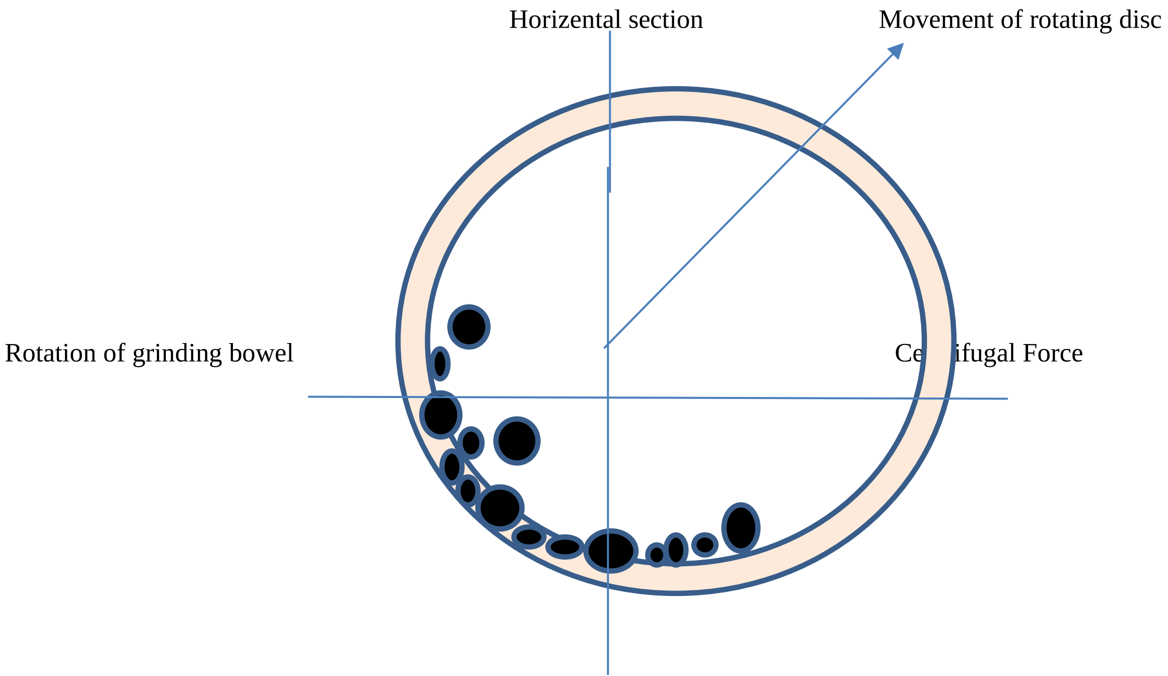

Figure 2 .Ball milling technique

Table 2. Compaction yield strength of NC Pd and Cu produced using IGC.

Hardness/3 (GPa)	σ_y (GPa)	Grain size (nm)	Density (% theor.)	Compaction temperature (°C)	Sample
1.0	1.15	54	98.5	335	Pd1
1.1	1.10-1.13	38	97.9	183	Pd2
0.75	0.75	24	95.3	RT	Pd3
0.77	0.65	19	92.5	106	Cu1
0.87	0.85	20	84.4	106	Cu1

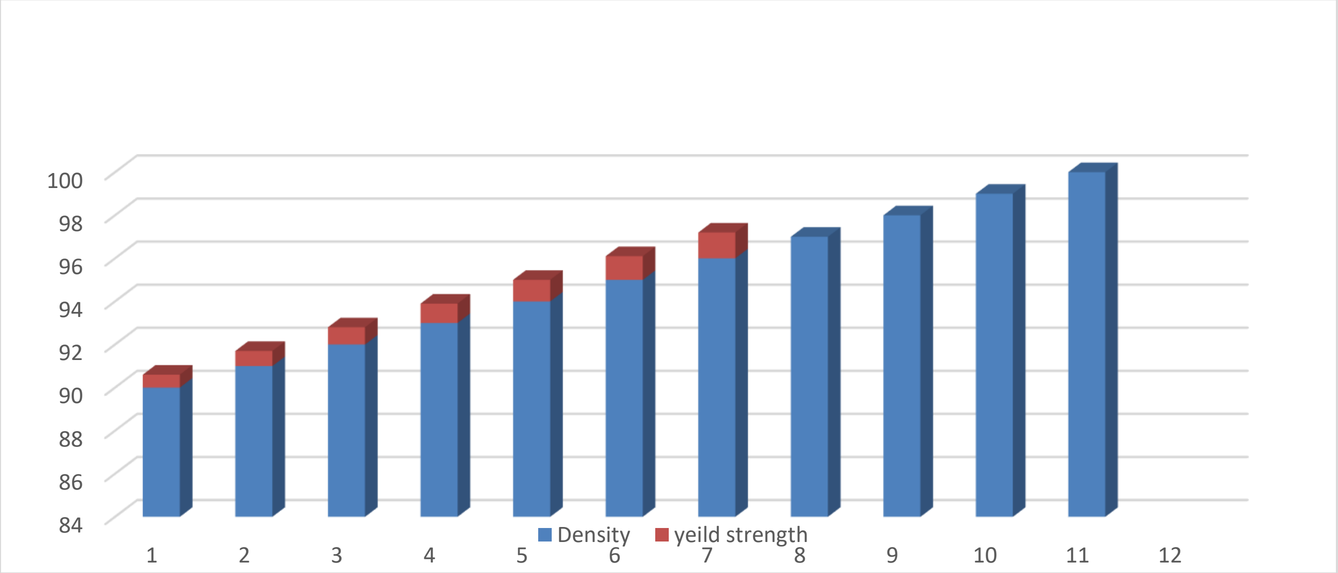

Figure 3. compressive yield strength of Cu and Pd as a function of consolidation density.

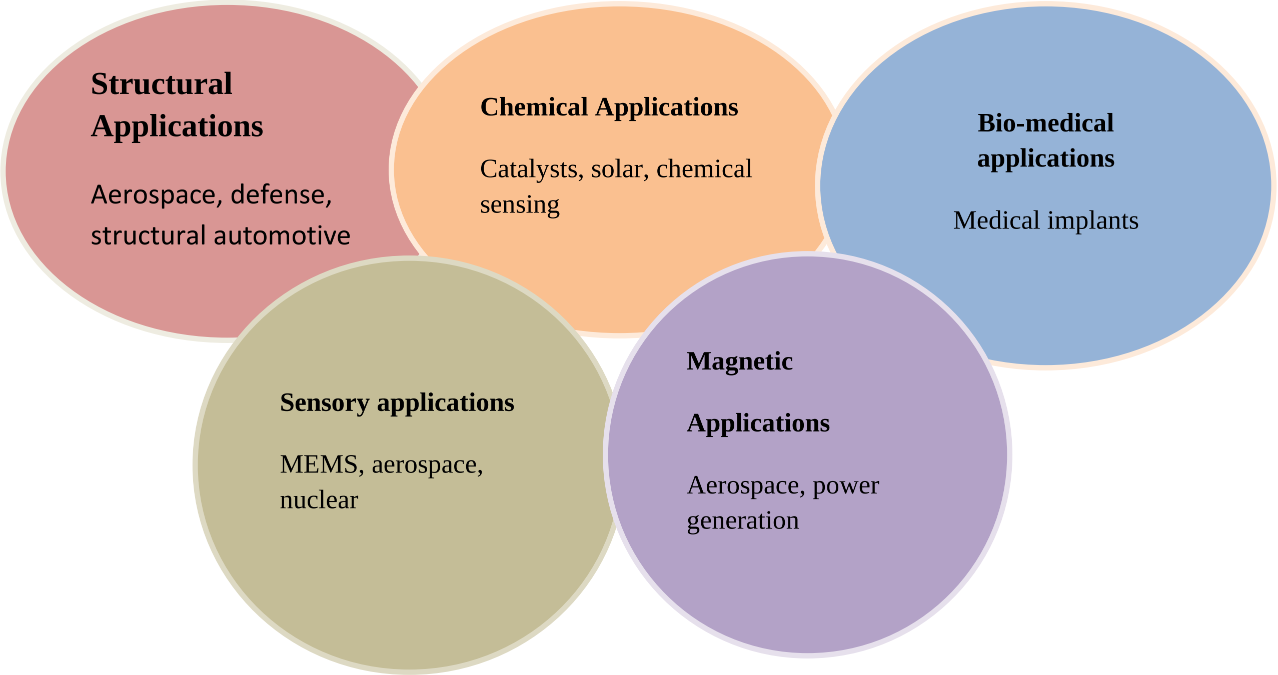

Figure 4. Applications of NCM's in different fields.

Their findings demonstrate that the MEMS parts have better cross-sectional homogeneity and toughness. NC SrMnO3, a perovskite-type oxide compound, was recently produced by Doroftei et al. Using a novel self-combustion process combined with the sol-gel approach. This compound has an average crystallite size of 88.9 nm and is intended for use in resistive and or capacitive humidity sensor applications [55].

6.3 Structural application

It is well known that the microscopic dispersion of a second phase in ceramics can significantly increase their fracture toughness [56]. Micro/nanohybrids are predicted to provide a new class of ultrastrong and ultratough ceramics since decreasing particle dimensions to nanometer (nm) sizes can boost strength and rigidity. These mixtures have a nanocomposite matrix reinforced with submicrometer-sized particles, such as stubbles, platelets, or extended fibers. While the characteristics of coarse-grained composites often deteriorate at high temperatures, these hybrids exhibit improved fracture toughness and strength. Furthermore, through the suppression of grain boundary sliding, nanoreinforcements raise the creep resistance [57].

7. Challenges

Many studies have been conducted in the last 20 years to create NC materials and the processing methods for them in order to enhance their mechanical qualities [58]. But there are also some difficulties. Preventing metal powder contamination is the first potential problem during the cryomilling process. Researchers have not yet addressed this issue. In addition to this process-specific problem, NC structures also have another common problem. Post-processing of the material is necessary for every processing pathway that produces NC material. Because of grain boundary relaxation and grain accumulation, the grain borders of NC materials are frequently vulnerable to nucleation and grain growth during this post-processing when heat is applied. One ongoing problem that academics are tackling is restricting this grain expansion. Maintaining the distinctively better qualities of nanocrystalline (NC) materials requires achieving the thermal stability of nanocrystalline (NC) grain boundaries. This is necessary to put nanocrystalline (NC) material usage into vibrant commercial manufacturing processes and applications. According to recent research, utilizing a modest cooling rate throughout the post-processing stages may assist prevent grain growth [59].

8. Future scope

The Hall-Petch relation states that when grain size decreases, a material's strength tends to rise. But it's also crucial to remember that, according to the inverse Hall-Petch connection, the material softens when the particle size falls under a particular critical value. It is easy to vary the mechanical characteristics by limiting the grain size by modifying the process parameters or presenting another stage to limit the expansion and reduction of the particle frontiers. According to recent research, the Zener pinning effect may limit grain growth when a secondary element is doped near the matrix grain boundary [60]. Due to the great promise of NC materials, a great deal of research is being done in this field. Since the ductility of NC materials is naturally restricted, future efforts should concentrate on increasing it. Since the field of NC materials is expanding, researchers may also be looking for new potential uses for it in other, undiscovered fields [61].

9. Conclusions

This review paper provided a thorough explanation of bulk nanostructuring of metals and alloys. A complete conversation was held on different bulk-nanostructuring techniques, including cryomilling, mechanical alloying (including HEBM), chemical precipitation, chemical vapor deposition, fast solidification, and chemical precipitation. This article highlights the procedure and lists its advantages over alternative processing techniques. Grain growth and recovery is emphasized as a problem**.** The possibility of powder contamination from the grinding media through the cryomilling procedure is another important concern. Because of its exceptional qualities, NC materials undoubtedly have a wide range of possible uses, which should be thoroughly investigated soon. Better understanding of various synthesis techniques and selection of suitable process parameters can be gained from this review article, leading to the production of bulk NC materials with superior mechanical properties**.**

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