# Metamaterials in the World of *Materionics* Overview of Fabrication Processes

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**Abstract**: *Advances in materials science and electronic engineering, especially in overlapping subjects and phenomena, have inspired the introduction of a new term for researchers in these fields to conduct a common dialogue throughout the world. The new term is Materionic Engineering or Materionics, analogous to Mechatronics. Furthermore, in the field that will be known as Materionics, combined research effort in 'materials science and engineering' and 'electronic engineering' has contributed significantly to the development of 'metamaterials'. Herein, we present an overview of the fabrication methods for metamaterials, i.e., top-down and bottom-up approaches involving lithography and self-assembly, respectively. In a nutshell, the future achievements in the field of Materionics will be driven mostly by the use of the bottom-up approaches.*
**Keywords** – *bottom-up, electronics, materials, self-assembly, top-down*

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## I.    Introduction

The discovery of new materials and structures has often helped in addressing global challenges faced by humankind. The fast-evolving fields of modern materials science and electronics have led to the development of many functional materials and devices. Over the years, different names have been coined for describing these functional materials as they have become increasingly available for performing certain functions; for example, electro-ceramics, electro-polymers, piezo-electrics, semi-conductors, nano-biomaterials, etc. In this article, *Materionics* is introduced as a new term to build a universal vocabulary to describe the cooperation and collaboration between the two fields of materials and electronics, encompassing all the relevant terms. The new term *Materionics* is analogous to the term Mechatronics, which was coined by Tetsuro Mori in 1969, and has been quite widely used in both academia and industry [1]. We believe that the new term *Materionics* will become mainstream much quicker, because of the significant overlapping research in the two aforementioned fields of engineering.

Historically, the understanding and applications of different materials have become important keystones for delineating human civilization; for example, the Stone Age, the Bronze Age, and perhaps the Nanotechnology Age for the 21st century. Everything we use, touch, or watch in our daily lives is made out of some sort of material. Some of the greatest creations of the 21st century, such as smart phones and smart watches, are composed of advanced electronic materials to utilize their functional properties, enabled by the understanding, manipulation, and controlling of electron flow within them. This multidisciplinary approach has led to the creation of numerous subfields involving electronic materials with specific functional applications: magnetic, optoelectronic, photonic, piezoelectric, thermoelectric, electro-polymeric, semiconductors, sensors and actuators, superconductors, metamaterials, etc. [2]. Notably, excellent research and development in the fields of materials science and engineering and electronic engineering have made the development and advancements of materials possible. Advances in materials can be limitless; therefore, we believe that the ever-growing interdisciplinary activities in the relevant subfields of engineering demand a universal vocabulary to unify the various subfields. The term we introduce herein is *Materionics*, which is a combination of the two words **materi**als and elect**ronics**. Analogous to the term mechatronics, we believe that the term *Materionics* will become mainstream amongst the scientific and engineering communities around the world in the future. Furthermore, it will become a standalone academic degree, again similar to mechatronics, in many undergraduate and graduate programs in different universities around the world.

## II.     Metamaterials and their Fabrication

In the field of *Materionics*, one remarkable example of a functional material is metamaterials, which have shown properties not found in naturally occurring materials. The Greek prefix 'meta', which stands for beyond, has been used to indicate that they are artificial materials with unusual properties. Almost all known materials have positive electrical permittivities ($\varepsilon$), as well as positive magnetic permeabilities ($\mu$). In the artificially fabricated metamaterials, both values are negative. When these two terms are simultaneously negative, the refractive index (n) is also negative, yielding a material with unique electromagnetic properties. These new artificially fabricated materials have thus been termed as negative-index materials (NIM) or left-handed (LH) materials. Notably, their properties arise from microscopic and nanoscopic structures or meta-atoms assembled in unusual combinations, and their applications range from aberration-free lenses, nanoscale optical devices, to invisibility cloaks [3].

Perhaps the most fascinating feature of metamaterials is their application in invisibility cloaks. This remarkable ability may soon be exploited in the extremely rich industries and markets of art, entertainment, and movie making. Further understanding of how metamaterials function may help scientists observe and study dark matter as well. Perhaps the existing dark matter in space has a slick covering of a metamaterial layer, or its entire structure is metamaterial in nature. The fact that scientists claim its existence but it remains yet to be observed may require the development of a proper understanding of its metamaterial structure or determination of the right frequency range for its natural observation.

There have been many reports on the importance of metamaterials; however, an article providing an overview of their fabrication methods, along with their most important structures, is lacking. Therefore, in this article, an overview of the fabrication methods for metamaterials has been presented, classified into two general approaches: top-down and bottom-up. The top-down approach involves slicing or systematically and preferentially cutting a bulk material into the final desired nano-sized shape/geometry; on the other hand, in the bottom-up approach, the buildup takes place from the bottom atom by atom, molecule by molecule, or cluster by cluster, escalating upward to the final desired shape. The top-down approach used for the fabrication of nano-sized shapes and geometries that is the most prevalent in the electronic industry is known as lithography. Several subcategories of the lithographic technique have been reported for the fabrication of metamaterials. On the other hand, in the bottom-up approach, only one technique, known as self-assembly, is used. A discussion of these techniques, along with the most important resulting structures, viz. fishnet, carpet, and gyroid structures, is presented herein.

## III.     Top-down Approach

### 3.1. Lithography

### 3.1.1. Electron Beam Lithography (EBL)

Electron beam lithography (EBL) is a patterning technique using an electron beam impinging on an electron-sensitive substance called a resist polymer. The direction and motion of the electron beam is determined by a complex combination of lenses, beam distortions, and orifices, thereby focusing the radiation on the resist polymer. The EBL method is suitable for structures smaller than 100 nm. Therefore, this method is a suitable approach for production of metamaterials of smaller sizes, comparable to the wavelengths of visible light [4].

Typically, the EBL device consists of a chamber, column, and objective lenses. Other electronic parts include power supply, vacuum pump, computer, and control units. The EBL device has been schematically depicted in Fig. 1.
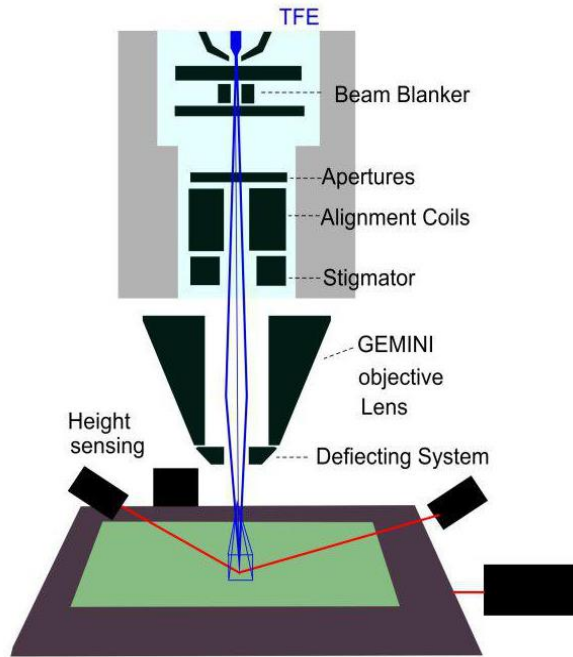
**Fig. 1.** Schematic of components of EBL device [5]

Various factors are involved in determining the quality of the pattern created in the resist polymer, including the electron beam spot size, exposure dose, and beam step size. The beam spot size is critical for making small patterns: smaller beam spot sizes can create smaller patterns. The level of exposure of the resist polymer also determines the intensity of the incoming load of electrons. The beam step size is a physical criterion for the physical distance between each electron beam, any change in which alters the quality of the patterns created on the resist polymer [5]. The EBL process is schematically shown in Fig. 2.
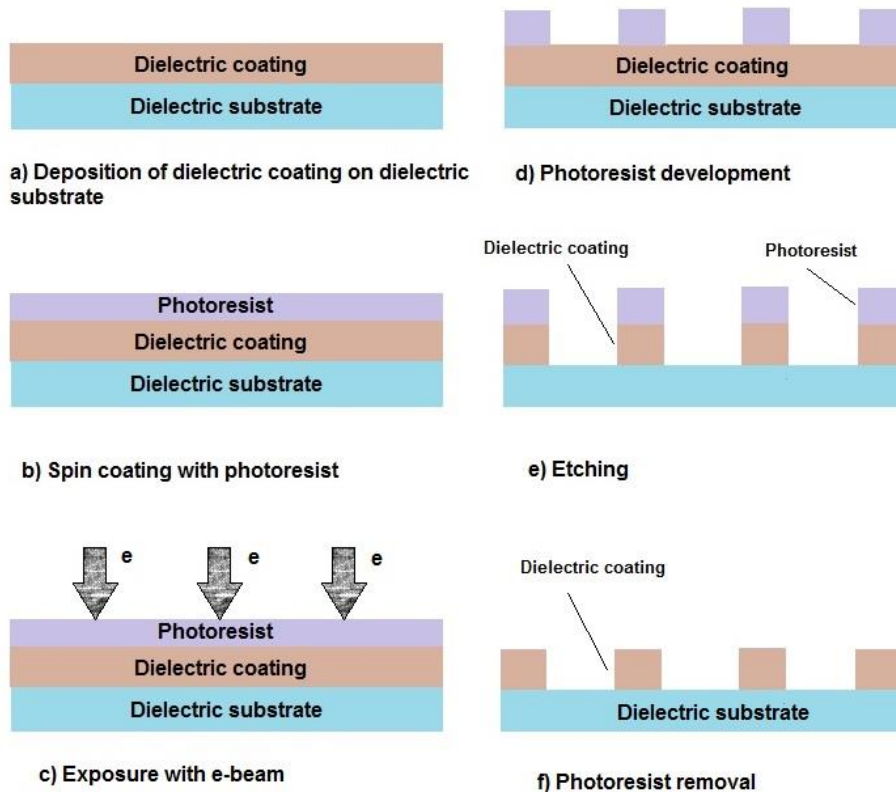


**Fig. 2.** Schematic of the EBL process [5]

For patterning, selected parts of the photoresist polymer are exposed to radiation, which passes through a mask that allows the passing of electrons according to the desired pattern. Radiation-induced reactions occurring in the resist polymer due to radiation cause molecular and structural alterations. The substrate coated with the photoresist polymer is then washed in a suitable solvent, and the desired pattern is created by etching. The repetition of this process leads to the formation of complicated patterns on the surface [6]. During the experimental fabrication of a dielectric metamaterial in the mid-infrared band [7], a thin film of Te was first deposited on barium fluoride ($BaF_2$) substrate, followed by patterning using EBL. Fig. 3 shows that a patterned sample possessing 1.53-µm Te cubes can be obtained after reactive ion etching.
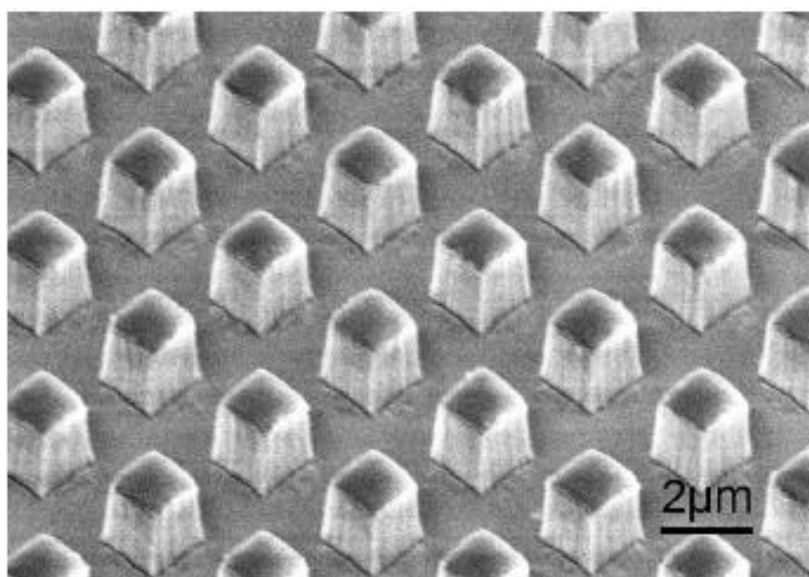


**Fig. 3.** SEM image of 1.53-µm Te cubes on a $BaF_2$ substrate patterned by EBL method

### 3.1.2 Focused Ion Beam Lithography (FIBL)

Since the discovery of the source of liquid metal ions in 1957, the focused ion beam (FIB) method has advanced significantly and developed into an interesting tool for lithography, etching, deposition, and doping. In general, liquid metal ion sources of Ga and Au–Si–Be alloys are used because of their longevity and stability. Therefore, sub-micrometre-size electronic devices can be produced via the FIB method. The advantages of this method include high radiation to resist polymer, high sensitivity compared to EBL, negligible dispersion of ions in resist polymer, and low backscattering from the substrate. However, the FIB method has some disadvantages, such as low performance and extensive damage to the substrate. Generally, the FIB method is suitable for cases where damage to the substrate is not important [8].

The FIB method has many benefits for the creation and processing of magnetic nanostructures, compared to the EBL method. Because the ions are larger than electrons, the FIB method is less affected by magnetic fields. This technique is suitable for small patterns and sheet substrates. Furthermore, no mask is used in this method [9]. This method is schematically presented in Fig. 4. An ion gun contains the liquid metal source heated to its melting point. A wire exposed to a voltage of 2–10 keV causes evaporation of the ionized metal. The evaporated ions are accelerated by applying a high voltage (30–50 keV) focused on the substrate, on which the desired patterns are created by FIB milling, leading to removal of the unnecessary material [10].
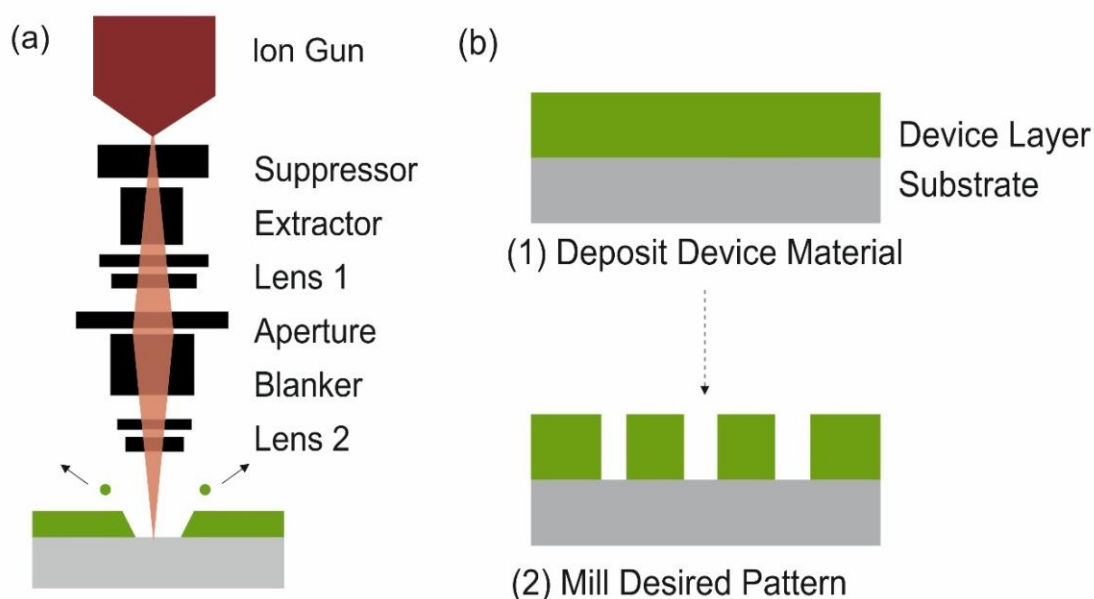
**Fig. 4.** a) Schematic of the FIB device. The ion beam passes through the lenses and apertures before colliding on the sample surface. b) Schematic of the FIB process: the device layer is first deposited, followed by milling to create the desired patterns

In a reported study [11], Cr and Ag/SiO$_2$layers were placed on a silicon nitride substrate by sputter coating to create a metamaterial with a window-shaped structure. The samples were treated with the FIB method from the substrate side on the bottom Cr layer by the localized elimination of the substrate and the creation of a window pattern of dimensions 10 μm ×10 μm. The mentioned operation was also performed on the upper layer. The cross-sectional SEM image of the structure created by the FIB method is shown in Fig. 5.
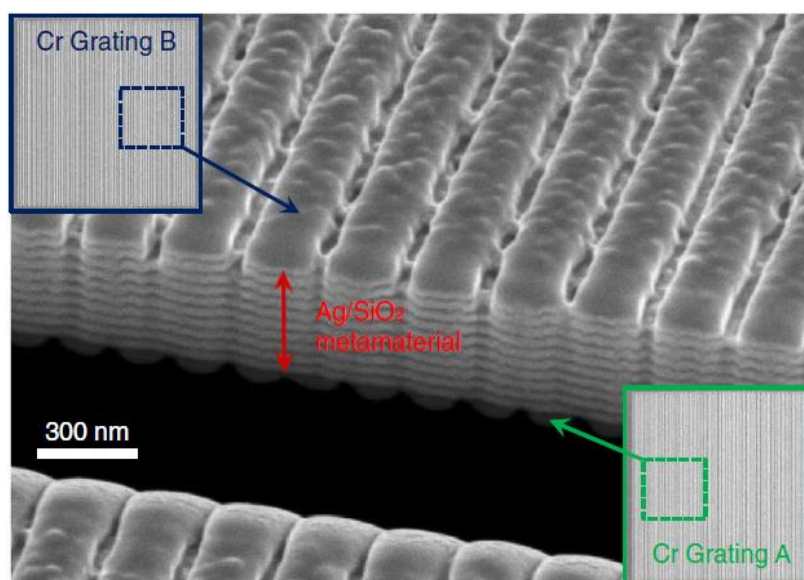


**Fig. 5.** Cross-sectional SEM image showing the inside of metamaterial prepared by the FIB method

### 3.1.3 Nanoimprint Lithography (NIL)

In this method, a mould with a predefined topological pattern is brought into physical contact with a substrate previously covered by a thin layer of imprint resist polymer, and is subjected to a pressure of 5−10 MPa [12-14]. For the polymer to flow and fill the pores of the mould, it is heated 90–100°C above its glass transition temperature (T$_g$). After drying and relieving the pressure, the mould can be removed without any damage. Eventually, a thin polymer layer remains in the pressed area, which is removed by reactive ion etching

[15]. The NIL process is classified into thermal, UV, and roll-to-roll. A schematic of the thermal method is presented in Fig. 6.
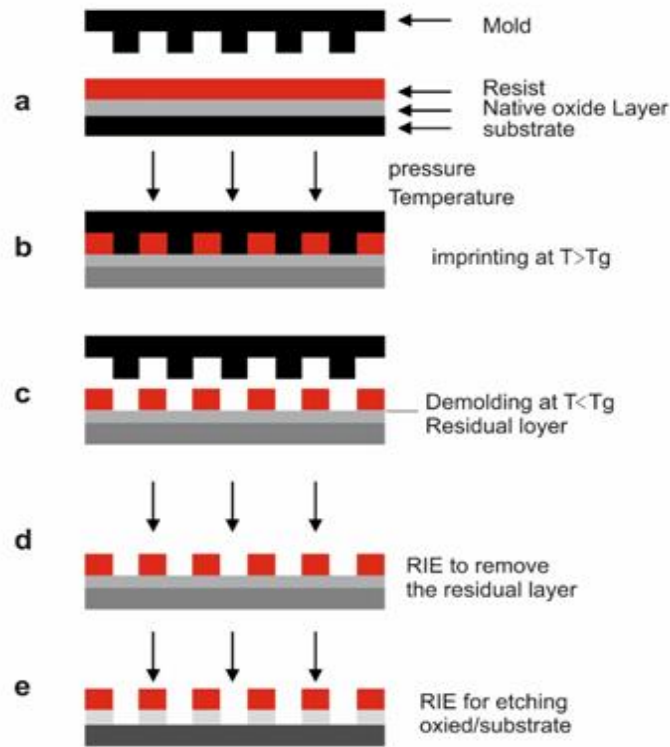


**Fig. 6.** Schematic of the stages of the NIL process: a) Main components, b) Stamping process under pressure and heat, c) Removal of the mould, d) Etching for removal of the residual resin polymer, and e) Elimination of the oxide layer [16]

A metamaterial surface created by the NIL method is presented in Fig. 7. Application of the NIL method caused formation of a uniform template [17].
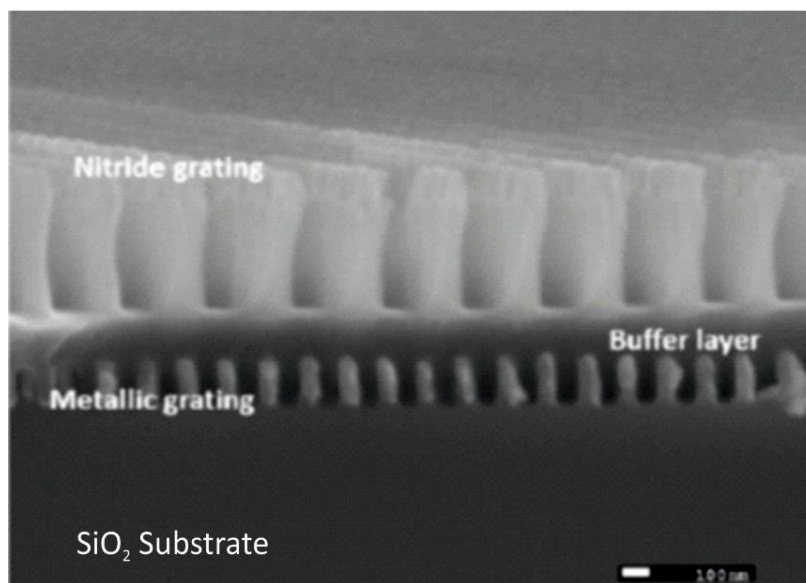


**Fig. 7.** Cross-sectional SEM image of the metamaterial produced by the NIL method

# IV. Bottom-up Approach

## 4.1. Self-Assembly

Self-assembly is a process in which an irregular system of components is converted to an organized patterned structure via local interactions among the primary components without applying any external force. Because the driving force for this process ends to reduce its Gibbs free energy and balances the opposing forces of repulsion and attraction, the system reaches a general and local thermodynamic equilibrium after the formation of the structure [18]. To scientifically define the process, it should be stated that self-assembly is a bottom-up method in which atoms or molecules arrange themselves into a regular nanostructure. The formation of salt crystals and snowflakes with complex structures are two common natural examples of self-assembly [19].

In self-assembly, arrangement of nanoparticles or molecules into ordered nanostructures occurs because of their unique electronic, magnetic, catalytic and/or other physical and chemical properties [20]. There are two types of self-assembly processes for nanostructure components: physical and chemical. Physical self-assembly is a simple process for creating non-complex structures, and expectedly, there is a lack of long-term stability in thus-assembled nanoparticles [21, 22]. On the other hand, in chemical self-assembly, a strong chemical bonding/coupling usually exists within the nanoparticles and even with the substrate surface, whether functionalized or non-functionalized. For instance, the location of nanoparticles on functionalized surfaces can be controlled to design complex nanostructures on the surface for specific applications, such as biosensors. In general, interactions leading to the self-assembly of nanoparticles may be covalent in nature, but mostly non-covalent interactions like electrostatic interactions, hydrogen bonding, and various host–guest interactions are involved [23, 24]. A visible-wavelength metamaterial fabricated by self-assembly is schematically shown in Fig. 8. For this fabrication, nano-size polystyrene (PS) spheres were periodically arranged on an indium tin oxide (ITO) glass substrate, followed by deposition of a silver layer into the gaps among the PS spheres. The PS spheres were removed by washing with chloroform and alcohol, leaving behind a perforated silver film. A polyvinyl alcohol (PVA) solution was spin-coated onto the silver film and solidified in a dry box. Then a second layer of PS spheres was periodically arranged on the solidified PVA film, followed by depositing a second silver layer in the gaps. Again, the PS spheres were removed by washing with chloroform and alcohol, resulting in a three-layered Ag-PVA-Ag sandwich nanostructure [24].
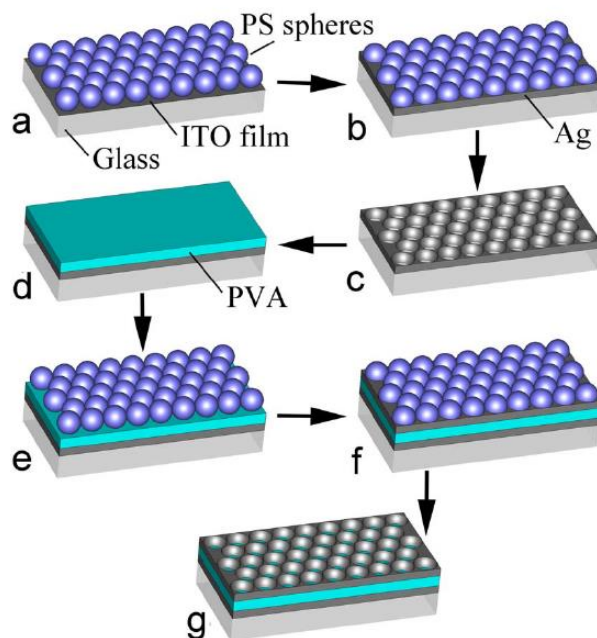


**Fig. 8.** A three-layered Ag-PVA-Ag sandwich nanostructured metamaterial prepared by self-assembly method

# V. Structure

## 5. 1. Fishnet Structure

The fishnet structure is a multilayer structure containing two or more metal layers separated by dielectric layers and without any vertical connections between the layers [25], schematically shown in Fig. 9.
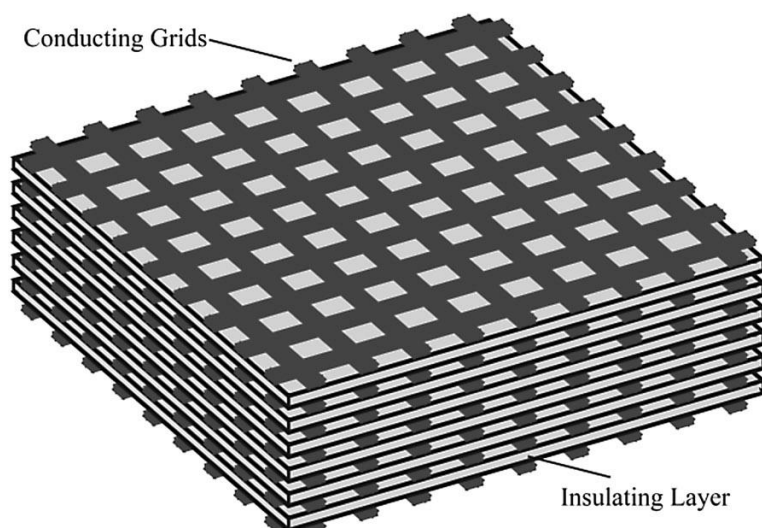


**Fig. 9.** Multilayer metamaterial with fishnet structure

In addition, the fishnet structure has micrometre-level dimensions in the terahertz frequency range, which facilitates the creation of a structure in the micrometre size range. Each fishnet layer can be considered a network of inductors connected by capacitors. For a wave propagating perpendicular to the fishnet plane, the capacitors and inductors are the series and parallel elements, respectively [26]. Unlike split-ring resonator (SRR) surface arrays, μ and ε and a negative refractive index can be simultaneously obtained in fishnet structures [27]. In SRR structures, elevated frequency produces increased losses in the negative refractive index, limiting their application to the microwave range. Therefore, one of the structures causing an effective negative refractive index in the millimetre-wave and visible wavelength ranges is the fishnet structure [28]. Generally, independent control of μ and ε is an interesting feature of metamaterials with fishnet structures.

Sharp et al. [29] evaluated the production and characterization of fishnet structure on a polymer layer. In the NIL method, a poly(methyl methacrylate) (PMMA) polymer layer was used as the substrate and three $Ag/MgF_2$ layers were used. In addition, a pressing machine was used to create columnar holes. The resultant structure is schematically presented in Fig. 10.
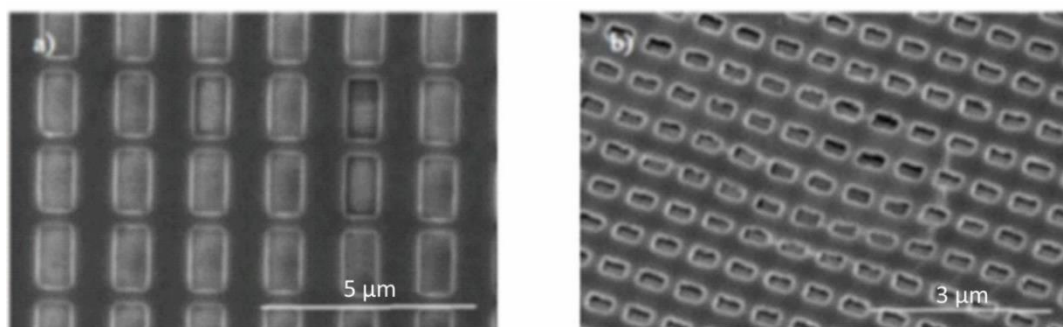


**Fig. 10.** Imprinted metal-dielectric metal fishnets with rectangular pillars pressed into PMMA. a) Planar view of fishnet and pillars, b) Fishnet viewed at a shallow tilt

**5. 2. Carpet Cloak Structure**
    The carpet cloak concept, along with an actual carpet cloak sample, is shown in Fig. 11.
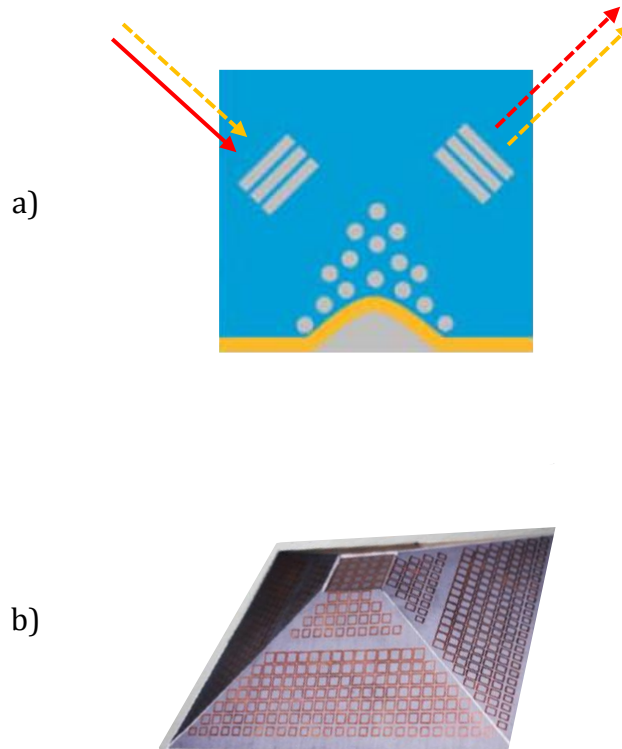


**Fig. 11.** a) Schematic of the carpet cloak concept [10], b) A carpet cloak sample shaped as a truncated pyramid, tilt angle of 24.6°, and dimensions of 176 mm × 176 mm × 34 mm [30]

    A conventional carpet cloak system, which can conceal any object located on a perfect planar surface, was first introduced by Li and Pendry [31]. Other systems have been introduced over the years, such as concealing any object on rough surfaces for more general and practical purposes [32]. A schematic of the fabrication of a conventional carpet cloak is presented in Fig. 12. Deposited on a 3-μm-thick $SiO_2$ substrate, the 250-nm-thick Si layer serves as a two-dimensional (2D) optical waveguide, where the light is confined in the vertical dimension but can freely propagate in the other two dimensions. The fabrication involves using the FIB to make the mirror facet, holes, and gratings onto the Si layer. The holes are milled with a constant diameter of 110 nm, aligned to the mirror facet, for creating the refractive index pattern. Two gratings are also milled for coupling the light into and out of the Si-waveguide. The next step involves placing a PMMA coat on top of the sample and depositing a 100-nm-thick Au layer onto the Si mirror facet to form the reflecting surface. The PMMA coat prevents Au deposition on the top of Si, which would degrade performance.
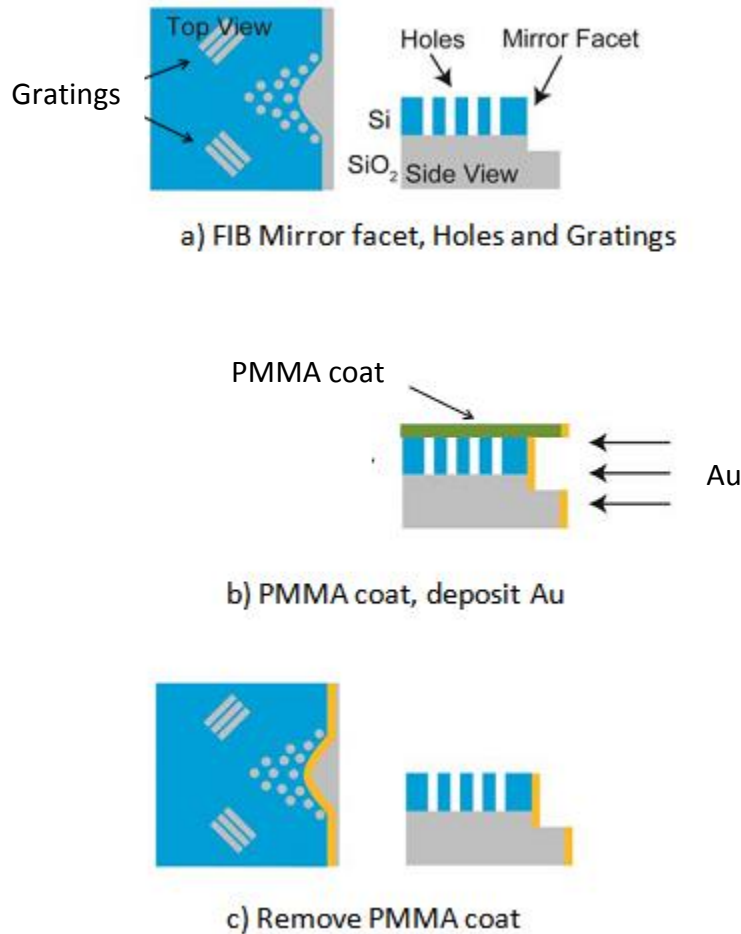
a) FIB Mirror facet, Holes and Gratings



b) PMMA coat, deposit Au



c) Remove PMMA coat

**Fig. 12.** Schematic of carpet cloak fabrication. a) The FIB is used to mill the Si layer to create the mirror facet, hole profile, and gratings. b) PMMA is coated on the top of the sample followed by depositing Au onto the Si mirror facet to form the reflecting surface. c) The PMMA coat is removed to complete the fabrication process

**5. 3. Gyroid Structure**

The word 'gyroid' was first coined in 1970 [33]. The gyroid morphology, i.e. a continuous three-dimensional structure with a constant mean curvature and chiral directions, is generated by the self-assembly of block copolymers. Block copolymers, which inherently self-assemble in selective solvents [34], consist of two or more chemically different polymers. In the gyroid morphology, the blocks forming the interwoven networks are composed of distinct polymers, denoted as minority phases, each forming a so-called single gyroid. The single gyroid has strong chirality in certain directions. It has been proposed [23] that a chiral structure can offer a unique route to a negative refractive index even when both the permittivity and permeability are positive.

The first chiral metamaterial was proposed by Pendry [31]. The complex structure of chiral metamaterials has posed a significant challenge in their miniaturization to the nano-metric scale. Standard top-down approaches such as lithography techniques are unsuitable for the fabrication of complex three-dimensional chiral structures, whereas the self-assembly of gyroid block copolymers is an ideal option to produce a chiral metamaterial.

Generally, the gyroid structure is divided into three non-intersecting parts, including a central section, that involves the largest part, and two other non-intersecting parts. As shown in Fig. 13, the gyroid production process by self-assembly method involves the following stages [35]:
1. Production of gyroid co-polymers by self-assembly
2. Selection and removal of the minor phase
3. Filling the site of the removed minor phase with a metal
4. Elimination of the remaining polymer mould

The fabrication of a gyroid co-polymer by self-assembly and replacement of the minor phase with Au were first performed by Yufa [19]. A schematic of a gyroid sample fabrication is presented in Fig. 13, and the SEM images in Fig. 14 show a metal gyroid from different orientations.
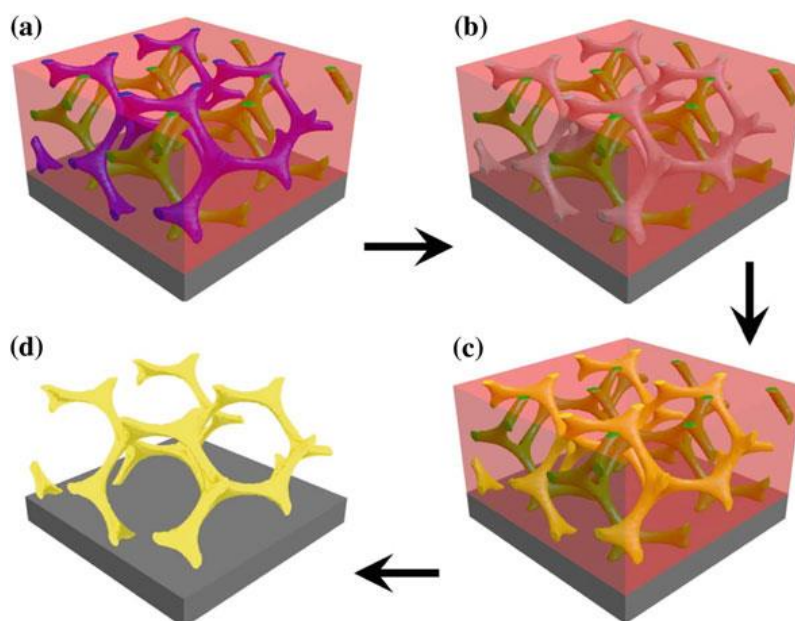


**Fig. 13.** Schematic of gyroid structure fabrication: a) Three different colours (phases) are observed, where the blue colour denotes the isoprene block, b) Isoprene has been removed, c) Isoprene site has been filled with gold by electrodeposition. d) The final structure, a 3D continuous network of gold, has been obtained by plasma etching removal of the two remaining polymer blocks [35]
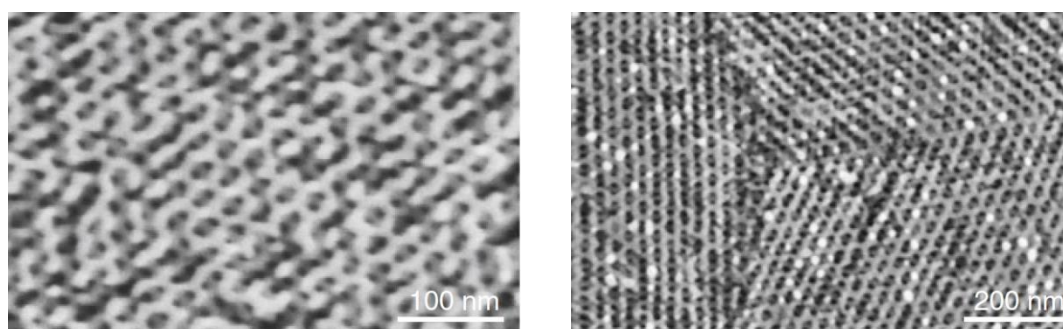


**Fig. 14.** SEM images of metal gyroid structure from different orientations [35]

## VI.     Discussion

The methods for fabricating metamaterials, along with the most important structures, are summarized in Table 1. The most important structures can be categorized into three groups: fishnet, carpet, and gyroid. Fishnet is a multilayer structure consisting of alternate metal layers separated by dielectric layers. As observed in Table 1, almost all of the current methods (EBL, FIB, NIL, roll-to-roll NIL, and self-assembly) can be used to obtain the fishnet structure. Another structure is the carpet cloak, which can be fabricated by EBL, FIB, or a combination of both these techniques. The fabrication of the gyroid requires the self-assembly method because of the significant complexity of this structure and inadequate ability of the other methods to realize the same. For this reason, future achievements in the field of *Materionics* are expected to rely on the use of the bottom-up approach.

**Table 1.** The most important structures along with their fabrication methods

| Structure | Fabrication Method | Source |
|---|---|---|
| Fishnet | Focused Ion Beam Milling | [36], [37] |
| Fishnet | Nanoimprint Lithography | [29], [38], [39] |
| Fishnet | Nanoimprint Lithography (Roll to Roll) | [40] |
| Fishnet | Electron Beam Lithography | [41] |
| Fishnet | Self-Assembly | [24] |
| Carpet | Focused Ion Beam Milling | [42] |
| Carpet | Electron Beam Lithography | [43], [44] |
| Carpet | Electron Beam Lithography Focused Ion Beam Milling | [45] |
| Gyroid | Self-Assembly | [19] |

## VII.    Prospective

Nanotechnology has seen rapid advances in recent decades, with exponential growth in some cases, especially the top-down approach. Some of the methods in the top-down approach include electron beam lithography (EBL), focused ion beam lithography (FIBL), and nanoimprint lithography (NIL). While these techniques play a significant role in the fabrication of nanomaterials, one of their notable disadvantages is the lack of access to miniaturisation. In addition, these methods are extremely slow and require very expensive equipment to obtain the desired small size and/or structures. On the other hand, none of these disadvantages are observed in the nascent bottom-up approach, which has laid the foundation for accessing ever-smaller and more complicated structures. As the name implies, the bottom-up approach starts from atoms or molecules to build up and fabricate structures with smaller sizes and units, the design of which involves the use of self-assembly features. Some of the advantages of the self-assembly technique include obtaining materials/structures that cannot be fabricated by the previous techniques, production and creation of homogeneous nanostructures on large substrates; and the ability to create truly 2D and 3D structures. Another important advantage of self-assembly is the ability to combine some production methods, so that all the advantages of the constituent methods can be realised. Thus, structures with larger sizes can be fabricated by using much smaller amounts of materials at reduced costs.

Another significant observation is that the bottom-up approach is inspired by nature, as observed in the natural formation of structures with larger sizes from cells and DNA. Bottom-up self-assembly opens up a new opportunity for the fabrication of metamaterials with significantly smaller sizes, which has not been possible by the traditional top-down techniques. Consequently, the last significant observation revolves around the fact that future achievements in the field of *Materionics* will depend on the use of bottom-up approaches.

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