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Bottom-Up and Top-Down Approaches for MgO

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Abstract

In this chapter, we present an overview of synthesis of MgO nanoparticles and thin films by using top-down and bottom-up approaches. The bottom-up approaches are generally utilized to grow nanoparticles by the methods that involve chemical reactions. Sometimes, methods based on these reactions are also able to grow thin films. The top-down approaches are preferred for growing thin films where bulk material is used for depositions. The methods, which are frequently used, are radio frequency sputtering, pulsed laser deposition, and molecular beam epitaxy and e-beam evaporation. Sometimes, methods like mechanical milling and high energy ball milling are used to grow nanoparticles.

Keywords: MgO, bottom-up approaches, top-down approaches

1. Introduction

Nanoparticles and thin films are very common form of materials for utilization in different applications [1–4]. Synthesis approaches play vital role to determine characteristics of nanoparticles [5] and thin films [6]. Thus, a number of methods are being developed to synthesize either nanoparticles [7–9] or thin films [10–12]. The motive behind to explore numerous methods is to look for reproducibility and cost effectiveness in terms of industrial utilization [13, 14]. Researchers are also working to get deep insights of involved phenomena during growth which persists a way to optimize for particular application [15–18]. The factors, which are considered during nanoparticle growth, are size [19], shape [20, 21] and size distribution [22, 23]. In case of thin films, these factors are nature of growth, morphology, stress, strain developed across films substrate interface [24–26].

While growing nanoparticles, one need to take care annealing treatment [27, 28] and stoichiometry [29, 30], however, process is rather typical in case of thin film technology. Choice of substrate [31], annealing temperature [32, 33], base pressure [34], target to substrate distance [35], deposition pressure [36, 37] and nature of gas during growth determine the nature of film [38]. Textured of grown thin film [39], stoichiometry [40] and nature of surface [41, 42] are another important parameter, which are considered during deposition. Thus, keeping in mind the necessity and challenges in the synthesis, synthesis approaches for growing nanoparticles and thin films are discussed by taking a simple inorganic system. However, magnesium oxide is known from long time [43] but recent advances in application of this

material motivated us to discuss these approaches for MgO [44]. In **Table 1**, a summary of properties of MgO are depicted [45–47].

While keeping in mind the importance of this material, we attempt to give an overview of synthesis of MgO nanoparticle and thin film. To grow nanoparticles, two kinds of approaches are used: (1) bottom-up approach and (2) top-down approach [48, 49]. These approaches are explained on the basis of following schematic diagram. In general, bottom-up approach is meant by synthesis of nanoparticles by means of chemical reactions among the atoms/ions/molecules (**Figure 1a**). Whereas top-down involves the mechanical methods to crush/ breaking of bulk into several parts to form nanoparticles (**Figure 1b**). In the next section both kind of approaches for growth of MgO nanoparticles and thin films are grown.

Properties/applications	Bulk [43, 45]	Nanoparticles [44]	Thin films [45]
Crystallite structure	Rocksalt	Rocksalt	Rocksalt
Lattice parameter (Å)	4.214	4.128	4.22
Optical band-gap (eV)	7.6	4–5	4–5

Table 1.
Properties and applications of MgO bulk, nanoparticles and thin films.

Figure 1.
Synthesis approaches for nanoparticles (a) bottom-up and (b) top-down approaches.

2. Bottom-up approaches

2.1 MgO nanoparticles

To initiate chemical reaction among the involved atoms/ions/molecules certain salts are taken as starting materials. These salts are mixed with each other to form a homogeneous solution along with a suitable chelating agent. Control of nature of solution also plays important role during synthesis process [50]. Thus, various methods are being developed by researchers to minimize annealing treatment, nature of chelating agent, pH value of solution. Some of these methods are depicted here.

Combustion synthesis is well known phenomena to synthesize nanomaterials of different kinds in its different variance [51, 52]. Most of the study utilizes solution combustion process for synthesizing nanoparticles. Typically, this method involves an oxidizer and fuel to initiate the reaction [53–55]. The most common oxidizers are metal nitrate/hydrates, ammonium nitrate and nitric acid. However, Urea, Glycine, Sucrose, Glucose, Citric Acid, Hydrazine based organic materials and Acetylacetone are frequently used as a fuel. The water, hydrocarbons and alcohols works as solvent for reactions involved in this synthesis [53].

Thus, combustion synthesis is able to produce nanoparticle of various materials both at research purposes as well as at industrial scale [51, 53–57]. Various kind of nanoparticles like titanates [58], ferrites [59], carbonates [60], hydroxide [61] and oxides [62] are grown using this approach. Combustion synthesis is utilized for growing different kind of MgO nanostructures [63, 64] and its derivative [65–67].

Our group utilizes, this method to synthesize MgO nanoparticles using combustion synthesis while taking magnesium nitrate as an oxidizer and citric acid as fuel [68]. This method shows reproducibility [69]. The following equation is expected during synthesis process.

Figure 2.
Synthesis of MgO nanoparticles from magnesium nitrate Ref. [69].

Figure 2 depicts schematic diagram of synthesis process. It is clear that synthesis takes place at low temperature, which reduces cost of synthesis. **Figure 3** shows representative X-ray diffraction (XRD) pattern of the nanoparticle synthesized at 500°C for 1 h. The method is able to produce nanoparticle with pure phase and no other crystalline phases are observed [70].

Green synthesis techniques utilize natural extracts [71] as fuels/oxidizer. Some of the natural extracts for synthesizing MgO nanoparticles are Neem leaves [72], *Artemisia abrotanum* Herba Extract [73], orange fruit [74], Aqueous Eucalyptus globules leaf [75] and Medicinal Plant *Pisonia grandis* R.Br. Leaf [76].

Microwave synthesis utilizes microwave radiation rather than furnace heating in order to avoid longer duration of heating to precursor [77, 78]. This method was successfully applied to form MgO nanoparticles by number of researchers [79–81].

Other methods which are effectively used to grow nanoparticles are facile [82, 83] and miroemulsion synthesis [84–86].

2.2 MgO thin films

Spin coating method is well known tool for growing thin films which utilizes chemical reaction to form materials on the given substrate [87, 88]. Sol-gel chemistry is helpful to synthesize thin films of MgO of desired crystallographic orientation using spin coater [89, 90]. **Figure 4** shows schematic of sol-gel method utilizing a spin coater to grow thin film [91, 92].

Atomic layer deposition (ALD) method allows depositions with excellent uniformity and conformality, with a cost-effective methodology [93, 94]. Thickness and composition control are usually possible over large-area substrates. Thin films of MgO were deposited by atomic layer epitaxy (ALE) from bis(cyclopentadienyl) magnesium and water using soda lime glass and Si(100) as substrates [95]. In another study, MgO films have been grown by atomic layer deposition in the wide

Figure 3.
X-ray diffraction pattern of the nanoparticle synthesized at 500°C for 1 h.

Figure 4.
Schematic of sol-gel spin coating method to grow MgO thin films. This schematic is based on the method described Ref. [91].

deposition temperature window of 80–350°C by using bis(cyclopentadienyl) magnesium and H₂O precursors [96].

3. Top-down approaches

Top-down approaches are mostly utilized to grow thin films of inorganic materials. Some of these methods are discussed here.

3.1 MgO nanoparticle

Mechanical milling/high energy ball milling is well known method which utilizes bulk counterpart as starting material and used for growing nanoparticles of different kind of materials [97, 98]. Depending upon milling process, the milling machines are categories as follows: tumbler ball mills, vibratory mills, planetary mills, and attritor mills [99, 100]. In the ball milling process, powder mixture or bulk powder placed in the ball mill is subjected to high-energy collision from the balls for nanoparticle synthesis. **Figure 5** depicts the schematic of high energy ball milling system [101]. Though this technique is effective to synthesize oxide nanoparticles [102, 103], however, no report is available for synthesizing MgO.

3.2 MgO thin films

Top-down approaches for growing MgO thin films are depicted in this section.

Figure 5.
Schematic of ball-milling process. Redrawn from Ref. [101].

e-Beam evaporation method involves the evaporation of material target with e-beam energy [104]. Schematic of this method is shown in **Figure 6**. This method is effectively used to grow MgO thin films on different type of substrates like NaCl [105], Si [106], fused quartz [107] as well as on metallic layers [108, 109].

Figure 7 shows the MgO thin films on fused quartz substrate along with MgO powder. Both the films of thickness around 5 and 50 nm reveal almost amorphous

Figure 6.
e-Beam evaporation for growing MgO thin films. Schematic is based on the set-up used for growing MgO thin films in Ref. [107].

nature. Optical absorption spectra of MgO thin films exhibit onset of film formation (**Figure 8**). This method is also utilized to grow MgO thin films on Si substrate. Films grown on this substrate exhibits polycrystalline nature [110].

Figure 7.
X-ray diffraction pattern of MgO thin film grown on fused quartz using e-beam evaporation method.

Figure 8.
UV-Vis spectra of MgO thin film grown on fused quartz using e-beam evaporation method.

Molecular beam epitaxy (MBE) utilizes e-beam for growing thin films [111]. It provides better control over stoichiometry ratio but also helpful in epitaxial growth of MgO [112, 113].

Pulsed laser deposition (PLD) as a thin film growth technique was not much popular until the late 1980s, when it has been used to grow superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ films [114]. Since then, the amount of research involving this technique has increased significantly and a number of compositions have been stabilized in thin film successfully [115–122]. The schematic of PLD system is shown in **Figure 9**. In PLD, a pulsed laser beam (having wavelengths in UV range) strikes the surface of the target material to be deposited. For a short duration of laser pulse (~20 nanoseconds), enormous power (~10 MW) is delivered to the target material and absorption of energy leads to ablation before the thermodynamic equilibrium. The energy from

Figure 9.
Schematic of pulsed laser deposition setup.

Figure 10.
Schematic of radio frequency sputtering setup based on the work reported in Ref. [131].

Figure 11.
XRD pattern of MgO thin film grown using rf sputtering method.

the laser evaporates the target's surface and the ablated material forms the plasma plume which finally deposit on the substrate mounted in front of the target.

The main advantage of PLD is the stoichiometric transfer from the multicomponent target to the thin film form, which otherwise is hard to achieve with any other thin film growth technique such as thermal evaporation or sputtering. The pulsed nature of PLD allows precise control on the film growth rates. Some drawbacks of the PLD technique are small area deposition, growth of macroscopic particles (particulates) during the ablation process, defects produced during growth, etc.

These advantages of this method allow researchers to grow MgO films on yttrium stabilized zirconia (111) substrates [123], Si (100) [124] and Al_2O_3 (0001) substrates [125].

Radio frequency sputtering method: At present most desired application of MgO is its utilization as a barrier for magnetic tunnel junction and rf sputtering method is preferred choice [126, 127] as well as for other applications [128].

Figure 10 shows the rf sputtering setup for the fabrication of thin films.

MgO films on Si substrate are grown by number of researchers [129, 130] as well as by our group [42, 131]. Films grown on Si substrate are both amorphous [132, 133] and crystalline [134, 135] in nature depending upon the deposition time and annealing temperature. **Figure 11** shows the XRD pattern of the MgO thin film grown at substrate temperature of 350°C, deposition time of 400 min and annealing temperature of 800°C for 1 h followed by 300°C and 24 h.

Apart from this, number of groups utilizes **chemical deposition (CVD) method** to grow MgO thin films on different substrates [136–138].

4. Conclusions

Thus, an overview of bottom-up and top-down approaches for synthesis of MgO nanoparticles and thin films is depicted in this chapter. Chemical methods are effective to grow nanoparticles, however, later is successful to grow thin films.

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Conflict of interest

The authors declare no conflict of interest.

Notes/thanks/other declarations

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References

- [1] Salata OV. Applications of nanoparticles in biology and medicine. *Journal of Nanobiotechnology*. 2004;**2**:3
- [2] Khan I, Saeed K, Khan I. Nanoparticles: Properties, applications and toxicities. *Arabian Journal of Chemistry*. 2019;**12**:908-931
- [3] Mitsugi F, Nakamura A, Kodama Y, Ohkubo T, Nomoto Y. Preparation of inorganic solid state thin film for electrochromic application. *Thin Solid Films*. 2007;**515**:4159-4165
- [4] Cauduro ALF, dos Reis R, Chen G, Schmid AK, Méthivier C, Rubahn H-G, et al. Crystalline molybdenum oxide thin-films for application as interfacial layers in optoelectronic devices. *ACS Applied Materials & Interfaces*. 2017;**9**:7717-7724
- [5] Singh JP, Singh V, Pandey G, Sharma A, Chae KH, Lee S. Design of MgO nanostructures for diverse applications. *Heliyon*. 2020
- [6] Sanchez C, Boissière C, Grosso D, Laberty C, Nicole L. Design, synthesis, and properties of inorganic and hybrid thin films having periodically organized nanoporosity. *Chemistry of Materials*. 2008;**20**:682-737
- [7] Sharma D, Kanchi S, Bisetty K. Biogenic synthesis of nanoparticles: A review. *Arabian Journal of Chemistry*. 2019;**12**:3576-3600
- [8] Nyamukamba P, Okoh O, Mungondori H, Taziwa R, Zinya S. Synthetic methods for titanium dioxide nanoparticles: A review. *Titanium Dioxide - Material for a Sustainable Environment*, Dongfang Yang, IntechOpen. 2018;151-175
- [9] Fahmy HM, Mohamed FM, Marzouq MH, Mustafa ABE-D, Alsoudi AM, Ali OA, et al. Review of green methods of iron nanoparticles synthesis and applications. *BioNanoScience*. 2018;**8**:491-503
- [10] Chambers SA. Epitaxial growth and properties of thin film oxides. *Surface Science Reports*. 2000;**39**:105-180
- [11] Ramanathan, Shriram, editor. *Thin Film Metal-Oxides, Fundamentals and Applications in Electronics and Energy*. US: Springer; 2010
- [12] Barber ZH. The control of thin film deposition and recent developments in oxide film growth. *Journal of Materials Chemistry*. 2006;**16**:334-344
- [13] Kakade N, Kumar R, Sharma SD, Datta D. Cost effectiveness of silver nanoparticles over gold nanoparticles in nano-particle aided radiotherapy. In: *Proc. of 39th Annual Conference of Association of Medical Physicists of India (AMPICON 2018)*; Chennai City, India
- [14] Piccinno F, Gottschalk F, Seeger S, Nowack B. Industrial production quantities and uses of ten engineered nanomaterials in Europe and the world. *Journal of Nanoparticle Research*. 2012;**14**:1109
- [15] Singh JP, Kim SH, Won SO, Lim WC, Lee I-J, Chae KH. Covalency, hybridization and valence state effects in nano-and micro-sized ZnFe_2O_4 . *CrystEngComm*. 2016;**18**:2701-2711
- [16] Singh JP, Kim SH, Won SO, Lee IJ, Chae KH. Atomic-scale investigation of MgO growth on fused quartz using angle-dependent NEXAFS measurements. *RSC Advances*. 2018;**8**:31275-31286
- [17] Zhang J, Huang F, Lin Z. Progress of nanocrystalline growth kinetics based on oriented attachment. *Nanoscale*. 2010;**2**:18-34

- [18] Yamamoto T, Adkar N, Okano Y, Ujihara T, Dost S. Numerical investigation of the transport phenomena occurring in the growth of SiC by the induction heating TSSG method. *Journal of Crystal Growth*. 2017;**474**:50-54
- [19] Kolhatkar AG, Jamison AC, Litvinov D, Willson RC, Lee TR. Tuning the magnetic properties of nanoparticles. *International Journal of Molecular Sciences*. 2013;**14**:15977-16009
- [20] Singh JP, Gagan D, Srivastava RC, Agrawal HM, Kumar R. Raman and Fourier-transform infrared spectroscopic study of nanosized zinc ferrite irradiated with 200 MeV Ag¹⁵⁺ beam. *Journal of Alloys and Compounds*. 2013;**551**:370-375
- [21] Khodashenas B, Ghorbani HR. Synthesis of silver nanoparticles with different shapes. *Arabian Journal of Chemistry*. 2019;**12**:1823-1838
- [22] Carney RP, Kim JY, Qian H, Jin R, Mehenni H, Stellacci F, et al. Determination of nanoparticle size distribution together with density or molecular weight by 2D analytical ultracentrifugation. *Nature Communications*. 2011;**2**:335
- [23] Zhang H, Minnich AJ. The best nanoparticle size distribution for minimum thermal conductivity. *Scientific Reports*. 2015;**5**:8995
- [24] Hu L, Hecht DS, Grüner G. Carbon nanotube thin films: Fabrication, properties, and applications. *Chemical Reviews*. 2010;**110**:5790-5844
- [25] Loureiro J, Santos JR, Nogueira A, Wyczisk F, Divay L, Reparaz S, et al. Nanostructured p-type Cr/V₂O₅ thin films with boosted thermoelectric properties. *Journal of Materials Chemistry A*. 2014;**2**:6456-6462
- [26] Ma C, Liu M, Chen C, Lin Y, Li Y, Horwitz JS, et al. The origin of local strain in highly epitaxial oxide thin films. *Scientific Reports*. 2013;**3**:3092-1-5
- [27] Shayesteh SF, Dizgah AA. Effect of doping and annealing on the physical properties of ZnO:Mg nanoparticles. *Pramana-Journal of Physics*. 2013;**81**:319-330
- [28] Singh JP, Srivastava RC, Agrawal HM, Kushwaha RPS, Chand P, Kumar R. EPR study of nanostructured zinc ferrite. *International Journal of Nanoscience*. 2008;**7**:21-27
- [29] Neagu D, Tsekouras G, Miller DN, Menard H, Irvine JTS. In situ growth of nanoparticles through control of nonstoichiometry. *Nature Chemistry*. 2013;**5**:916-923
- [30] Gunnarsson R, Helmersson U, Pilch I. Synthesis of titanium-oxide nanoparticles with size and stoichiometry control. *Journal of Nanoparticle Research*. 2015;**17**:353
- [31] Phillips JM. Substrate selection for thin-film growth. *MRS Bulletin*. 1995;**20**:35-39
- [32] Wu S-j J, Houng B, Huang B-s. Effect of growth and annealing temperatures on crystallization of tantalum pentoxide thin film prepared by RF magnetron sputtering method. *Journal of Alloys and Compounds*. 2009;**475**:488-493
- [33] Dualah A, Tétreault N, Moehl T, Gao P, Khaja Nazeeruddin M, Grätzel M. Effect of annealing temperature on film morphology of organic-inorganic hybrid perovskite solid-state solar cells. *Advanced Functional Materials*. 2014;**24**:3250-3258
- [34] García-Valenzuela JA, Andreu J, Bertomeu J. Effect of the base pressure

achieved prior deposition on the main properties of ZnO:Al films obtained by DC magnetron sputtering at room temperature for electrical contact use. *Journal of Vacuum Science & Technology A*. 2017;**35**:021603

[35] Hua Q, Ligang W, Ruijin L, Wenfeng Y. Influence of target substrate distance on the properties of transparent conductive Si doped ZnO thin films. *Optik*. 2014;**125**:3902-3907

[36] Wang C, Cheng BL, Wang SY, Lu HB, Zhou YL, Chen ZH, et al. Effects of oxygen pressure on lattice parameter, orientation, surface morphology and deposition rate of (Ba_{0.02}Sr_{0.98})TiO₃ thin films grown on MgO substrate by pulsed laser deposition. *Thin Solid Films*. 2005;**485**:82-89

[37] Agrawal A, Habibi HR, Agrawal RK, Cronin JP, Roberts DM, Caron-Popowich R'S, et al. Effect of deposition pressure on the microstructure and electrochromic properties of electron-beam-evaporated nickel oxide films. *Thin Solid Films*. 1992;**221**:239-253

[38] Pitt KEG, Howard AJ. The nature of residual gases during the deposition of resistive thin films. *Vacuum*. 1968;**18**:517-518

[39] Weber T P, Ma B, Balachandran U, McNallan M. Fabrication of biaxially textured magnesium oxide thin films by ion-beam-assisted deposition. *Thin Solid Films*. 2005;**476**:79-83

[40] Marton Z, Seo SSA, Egami T, Lee HN. Growth control of stoichiometry in LaMnO₃ epitaxial thin films by pulsed laser deposition. *Journal of Crystal Growth*. 2010;**312**:2923-2927

[41] Baeumer C, Xu C, Gunkel F, Raab N, Heinen RA, Koehl A, et al. Surface termination conversion during SrTiO₃ thin film growth revealed by

X-ray photoelectron spectroscopy. *Scientific Reports*. 2015;**5**:11829

[42] Singh JP, Ji MJ, Kumar M, Lee IJ, Chae KH. Unveiling the nature of adsorbed species onto the surface of MgO thin films during prolonged annealing. *Journal of Alloys and Compounds*. 2018;**748**:355-362

[43] Shand MA. Physical and chemical properties of magnesium oxide. In: Shand MA, editor. *The Chemistry and Technology of Magnesia*. John Wiley & Sons, Ltd. 2006

[44] Singh J, Chae K. d° Ferromagnetism of magnesium oxide. *Condensed Matter*. 2017;**2**:36

[45] Plócienni P, Guichaoua D, Zawadzka A, Korcala A, Strzelecki J, Trzaska P, et al. Optical properties of MgO thin films grown by laser ablation technique. *Optical and Quantum Electronics*. 2016;**48**:277

[46] Choudhury B, Choudhury A. Microstructural, optical and magnetic properties study of nanocrystalline MgO. *Materials Research Express*. 2014;**1**:025026

[47] Raja AME, Jayachandran M, Sanjeeviraja C. Fabrication techniques and material properties of dielectric MgO thin films—A status review. *CIRP Journal of Manufacturing Science and Technology*. 2010;**2**:92-113

[48] Iqbal P, Preece JA, Mendes PM. Nanotechnology: The “top-down” and “bottom-up” approaches. In: Gale PA, Steed JW, editors. *Supramolecular Chemistry*. John Wiley & Sons, Ltd. 2012

[49] Wang Y, Xia Y. Bottom-up and top-down approaches to the synthesis of monodispersed spherical colloids of low melting-point metals. *Nano Letters*. 2004;**4**:2047-2050

- [50] Sierra-Pallares J, Huddle T, García-Serna J, Alonso E, Mato F, Shvets I, et al. Understanding bottom-up continuous hydrothermal synthesis of nanoparticles using empirical measurement and computational simulation. *Nano Research*. 2016;**9**:3377-3387
- [51] Patil KC, Aruna ST, Mimani T. Combustion synthesis: An update. *Current Opinion in Solid State and Materials Science*. 2002;**6**:507-512
- [52] Chae S, Lee H, Pikhits PV, Kim C, Shin S, Kim DH, et al. Synthesis of terraced and spherical MgO nanoparticles using flame metal combustion. *Powder Technology*. 2017;**305**:132-140
- [53] Varma A, Mukasyan AS, Rogachev AS, Manukyan KV. Solution combustion synthesis of nanoscale materials. *Chemical Reviews*. 2016;**116**:14493-14586
- [54] Li F-t, Ran J, Jaronie M, Qiao SZ. Solution combustion synthesis of metal oxide nanomaterials for energy storage and conversion. *Nanoscale*. 2015;**7**:17590-17610
- [55] Orante Barrón VR, Oliveira LC, Kelly JB, Milliken ED, Denis G, Jacobsohn LG, et al. Luminescence properties of MgO produced by solution combustion synthesis and doped with lanthanides and Li. *Journal of Luminescence*. 2011;**131**:1058-1065
- [56] Aruna ST, Mukasyan AS. Combustion synthesis and nano-materials. *Current Opinion in Solid State & Materials Science*. 2008;**12**:44-50
- [57] Hwang CC, Tsai JS, Huang TH, Peng CH, Chen SY. Combustion synthesis of Ni-Zn ferrite powder—Influence of oxygen balance value. *Journal of Solid State Chemistry*. 2005;**178**:382-389
- [58] Sukpanish P, Lertpanyapornchai B, Yokoi T, Ngamcharussrivichai C. Lanthanum-doped mesostructured strontium titanates synthesized via sol-gel combustion route using citric acid as complexing agent. *Materials Chemistry and Physics*. 2016;**181**:422-431
- [59] Singh JP, Won SO, Lim WC, Lee I-J, Chae KH. Electronic structure studies of chemically synthesized MgFe_2O_4 nanoparticles. *Journal of Molecular Structure*. 2016;**1108**:444-450
- [60] Singh JP, Ji M-J, Shim C-H, Kim SO, Chae KH. Effect of precursor thermal history on the formation of amorphous and crystalline calcium carbonate. *Particuology*. 2017;**33**:29-34
- [61] Singh JP, Kim SH, Lim WC, Won SO, Chae KH. Local electronic structure investigation of the sol-gel processed calcium hydroxide material. *Advanced Materials and Processes*. 2018;**3**:377-381
- [62] Bhardwaj R, Singh JP, Chae KH, Goyal N, Gautam S. Electronic and magnetic structure investigation of vanadium doped ZnO nanostructure. *Vacuum*. 2018;**158**:257-262
- [63] Li S. Combustion synthesis of porous MgO and its adsorption properties. *International Journal of Industrial Chemistry*. 2019;**10**:89-96
- [64] Nassar MY, Mohamed TY, Ahmed IS, Samir I. MgO nanostructure via a sol-gel combustion synthesis method using different fuels: An efficient nano-adsorbent for the removal of some anionic textile dyes. *Journal of Molecular Liquids*. 2017;**225**:730-740
- [65] Sangeeta M, Karthika KV, Ravishankar R, Anantharaju KS, Nagabhushana H, Jeetendra K, et al. Synthesis of ZnO, MgO and ZnO/MgO by solution combustion method: Characterization and photocatalytic studies. *Materials Today: Proceedings*. 2017;**4**:11791-11798

- [66] Ning P, Zhang F, Wang LJ, Zhou Y, Wang YJ, Wu YY, et al. Sol-gel derived AgMgO films for antibacterial and bioactive surface modification of niobium metal. *Materials Chemistry and Physics*. 2020;**243**:122646
- [67] Vahid BR, Haghighi M. Urea-nitrate combustion synthesis of MgO/MgAl₂O₄ nanocatalyst used in biodiesel production from sunflower oil: Influence of fuel ratio on catalytic properties and performance. *Energy Conversion and Management*. 2016;**126**:362-372
- [68] Singh JP, Won SO, Lim WC, Shim C-H, Chae KH. Optical behavior of MgO nanoparticles investigated using diffuse reflectance and near edge X-ray absorption spectroscopy. *Materials Letters*. 2017;**198**:34-37
- [69] Singh JP, Chae KH. Local electronic structure perspectives of nanoparticle growth: The case of MgO. *ACS Omega*. 2019;**4**:7140-7150
- [70] Singh JP, Lim WC, Won SO, Song J, Chae KH. Synthesis and characterization of some alkaline-earth-oxide nanoparticles. *Journal of the Korean Physical Society*. 2018;**72**:890-899
- [71] Iravani S. Green synthesis of metal nanoparticles using plants. *Green Chemistry*. 2011;**13**:2638-2650
- [72] Krishna Moorthy S, Ashok CH, Venkateswara Rao K, Viswanathana C. Synthesis and characterization of Mgo nanoparticles by neem leaves through green method. *Materials Today: Proceedings*. 2015;**2**:4360-4368
- [73] Suresh J, Yuvakkumar R, Sundrarajan M, Hong SI. Green synthesis of magnesium oxide nanoparticles. *Advanced Materials Research*. 2014;**952**:141-144
- [74] Ganapathi Rao K, Ashok CH, Venkateswara Rao K, Shilpa Chakra CH, Akshaykranth A. Eco-friendly synthesis of MgO nanoparticles from orange fruit waste. *International Journal of Advanced Research in Physical Science*. 2015;**2**:1-6
- [75] Jeevanandam J, Chan YS, Ku YH. Aqueous *Eucalyptus globulus* leaf extract-mediated biosynthesis of MgO nanorods. *Applied Biological Chemistry*. 2018;**61**:197-208
- [76] Joghee S, Ganeshan P, Vincent A. Ecofriendly biosynthesis of zinc oxide and magnesium oxide particles from medicinal plant *Pisonia grandis* R.Br. Leaf extract and their antimicrobial activity. *BioNanoScience*. 2019;**9**:141-154
- [77] Gerbec JA, Magana D, Washington A, Strouse GF. Microwave-enhanced reaction rates for nanoparticle synthesis. *Journal of the American Chemical Society*. 2005;**127**:15791-15800
- [78] Motshekga SC, Pillai SK, Ray SS, Rui KJ, Krause WM. Recent trends in the microwave-assisted synthesis of metal oxide nanoparticles supported on carbon nanotubes and their applications. *Journal of Nanomaterials*. 2012;**2012**:691503
- [79] Makhluf S, Dror R, Nitzan Y, Abramovich Y, Jelinek R, Gedanken A. Microwave-assisted synthesis of nanocrystalline MgO and its use as a bactericide. *Advanced Functional Materials*. 2005;**15**:1708-1715
- [80] Ribeiro DV, Paula GR, Morelli MR. Use of microwave oven in the calcination of MgO and effect on the properties of magnesium phosphate cement. *Construction and Building Materials*. 2019;**198**:619-628
- [81] Mirzaei H, Davoodnia A. Microwave assisted sol-gel synthesis of MgO nanoparticles and their catalytic

activity in the synthesis of Hantzsch 1,4-dihydropyridines. Chinese Journal of Catalysis. 2012;**33**(9-10):1502-1507

[82] Marwaha N, Gupta BK, Verma R, Srivastava AK. Facile synthesis and characterization of pH-dependent pristine MgO nanostructures for visible light emission. Journal of Materials Science. 2017;**52**:10480-10484

[83] Chamack M, Mahjoub AR, Hosseinian A. Facile synthesis of nanosized MgO as adsorbent for removal of Congo red dye from wastewater. Nanochemistry Research. 2018;**3**:85-91

[84] He Y. MgO nanostructured microspheres synthesized by an interfacial reaction in a solid stabilized emulsion. Materials Letters. 2006;**60**:3511-3513

[85] Li S, Zhou B, Ren B, Xing L, Dong L, Li J. Preparation of MgO nanomaterials by microemulsion-based oil/water interface precipitation. Materials Letters. 2016;**171**:204-207

[86] Bumajdad A, Al-Ghareeb S, Madkour M, Al Sagheer F, Zaki MI. Synthesis of MgO nanocatalyst in water-in-oil microemulsion for CO oxidation. Reaction Kinetics, Mechanisms and Catalysis. 2017;**122**:1213-1229

[87] Kelso MV, Mahenderkar NK, Chen Q, Tubbesing JZ, Switzer JA. Spin coating epitaxial films. Science. 2019;**364**(6436):166-169

[88] Yoon J-G, Kwag YJ, Kim HK. Structural characterization of sol-gel derived MgO thin film on Si substrate. Journal of the Korean Physical Society. 1997;**31**:613-616

[89] Jung HS, Lee J-K, Kim JY, Hong KS. Synthesis of nano-sized MgO particle and thin film from

diethanolamine-stabilized magnesium-methoxide. Journal of Solid State Chemistry. 2003;**175**:278-283

[90] Shin D-Y, Kim K-N. Electrical and optical properties of MgO films deposited on soda lime glass by a sol-gel process using magnesium acetate. Journal of Ceramic Processing Research. 2009;**10**:536-540

[91] Balta AK, Ertek Ö, Eker N, Okur İ. MgO and ZnO composite thin films using the spin coating method on microscope glasses. Materials Sciences and Applications. 2015;**6**:40-47

[92] Lee J, Jeong T, Yu SG, Jin S, Heo J, Yi W, et al. Secondary electron emission of MgO thin layers prepared by the spin coating method. Journal of Vacuum Science and Technology B. 2001;**19**:1366-1369

[93] George SM. Atomic layer deposition: An overview. Chemical Reviews. 2010;**110**:111-131

[94] Leskelä M, Mattinen M, Ritala M. Atomic layer deposition of optoelectronic materials. Journal of Vacuum Science & Technology B. 2019;**030801**:37

[95] Putkonen M, Sajavaara T, Lauri NE. Enhanced growth rate in atomic layer epitaxy deposition of magnesium oxide thin films. Journal of Materials Chemistry. 2000;**10**:1857-1861

[96] Vangelista S, Mantovan R, Lamperti A, Tallarida G, Kutrzeba-Kotowska B, Spiga S, et al. Low-temperature atomic layer deposition of MgO thin films on Si. Journal of Physics D: Applied Physics. 2013;**46**:485304

[97] Indris S, Amade R, Heitjans P, Finger M, Haeger A, Hesse D, et al. Preparation by high-energy milling, characterization, and catalytic properties of nanocrystalline TiO₂.

The Journal of Physical Chemistry. B. 2005;**109**(49):23274-23278

[98] Salah N, Habib SS, Khan ZH, Memic A, Azam A, Alarfaj E, et al. High-energy ball milling technique for ZnO nanoparticles as antibacterial material. *International Journal of Nanomedicine*. 2011;**6**:863-869

[99] Yadav TP, Yadav RM, Singh DP. Mechanical milling: A top down approach for the synthesis of nanomaterials and nanocomposites. *Nanoscience and Nanotechnology*. 2012;**2**:22-48

[100] Suryanarayana C. Powder Metal Technologies and Applications. *ASM Handbook*, Vol. 7. Materials Park, OH: ASM International; 1998. pp. 80-90

[101] Synthesis of nanomaterials by high energy ball milling. Available from: <http://www.understandingnano.com/nanomaterial-synthesis-ball-milling.html> [Accessed: 30 December 2016]

[102] Amirkhanlou S, Ketabchi M, Parvin N. Nanocrystalline/nanoparticle ZnO synthesized by high energy ball milling process. *Materials Letters*. 2012;**86**:122-124

[103] Hosseini SG, Ayoman E. Synthesis of α -Fe₂O₃ nanoparticles by dry high-energy ball-milling method and investigation of their catalytic activity. *Journal of Thermal Analysis and Calorimetry*. 2017;**128**:915-924

[104] Singh V, Abhilash SR, Behera BR, Kabiraj D. Fabrication of thin self-supporting platinum targets using evaporation techniques. *Nuclear Instruments and Methods in Physics Research A*. 2011;**635**:20-23

[105] Aboelfotoh MO. Epitaxy of MgO on alkali halides with NaCl-type structure. *Journal of Applied Physics*. 1978;**49**:2770-2776

[106] Lee MJ, Park SY, Kim SG, Kim HJ, Moon SH, Kim JK. Effect of stress and density on the electrical and physical properties of MgO protecting layer for alternating current-plasma display panels. *Journal of Vacuum Science and Technology A*. 2005;**23**:1192-1196

[107] Singh JP, Sulania I, Prakash J, Gautam S, Chae KH, Kanjilal D, et al. Study of surface morphology and grain size of irradiated MgO thin films. *Advanced Materials Letters*. 2012;**3**:112-117

[108] Diao Z, Feng JF, Kurt H, Feng G, Coey JMD. Reduced low frequency noise in electron beam evaporated MgO magnetic tunnel junctions. *Applied Physics Letters*. 2010;**96**:202506-1-3

[109] Singh JP, Raju M, Asokan K, Jai P, Kabiraj D, Abhilash SR, et al. Magnetization in MgO based multilayers fabricated by e-beam evaporation. *AIP Conference Proceedings*. 2012;**1447**:749-750

[110] Singh JP, Chen CL, Dong CL, Prakash J, Kabiraj D, Kanjilal D, et al. Role of surface and subsurface defects in MgO thin film: XANES and magnetic investigations. *Superlattices and Microstructures*. 2015;**77**:313-324

[111] Yadavalli S, Yang MH, Flynn CP. Low-temperature growth of MgO by molecular-beam epitaxy. *Physical Review B*. 1990;**41**:7961-9963

[112] Niu F, Hoerman BH, Wessels BW. Epitaxial thin films of MgO on Si using metalorganic molecular beam epitaxy. *Journal of Vacuum Science and Technology B*. 2000;**18**:2146-2152

[113] Niu F, Meier AL, Wessels BW. Epitaxial growth and strain relaxation of MgO thin films on Si grown by molecular beam epitaxy. *Journal of Vacuum Science and Technology B*. 2006;**24**:2586-2591

- [114] Chrisey DB, Hubler GH. Pulsed Laser Deposition of Thin Films. New York: Wiley Interscience; 1994
- [115] Kumar M, Phase DM, Choudhary RJ, Lee HH. Structure and functionalities of manganite/cuprate thin film. *Current Applied Physics*. 2018;**18S**:33-36
- [116] Kumar M, Choudhary RJ, Shukla DK, Phase DM. Metastable magnetic state and magnetotransport in disordered manganite thin films. *Journal of Applied Physics*. 2014;**115**:163904
- [117] Kumar M, Choudhary RJ, Phase DM. Valence band structure of YMnO_3 and the spin orbit coupling. *Applied Physics Letters*. 2013;**102**:182902
- [118] Kumar M, Choudhary RJ, Phase DM. Magnetic and electronic properties of $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3/\text{h-YMnO}_3$ bilayer. *Journal of Vacuum Science and Technology A*. 2016;**34**:021506
- [119] Kumar M, Choudhary RJ, Phase DM. Growth of different phases of yttrium manganese oxide thin films by pulsed laser deposition. *AIP Conference Proceedings*. 2012;**1447**:655
- [120] Devi V, Kumar M, Choudhary RJ, Phase DM, Kumar R, Joshi BC. Band offset studies in pulse laser deposited $\text{Zn}_{1-x}\text{Cd}_x\text{O}/\text{ZnO}$ hetero-junction. *Journal of Applied Physics*. 2015;**117**:225305
- [121] Devi V, Kumar M, Kumar R, Singh A, Joshi B C, Band offset measurements in $\text{Zn}_{1-x}\text{Sb}_x\text{O}/\text{ZnO}$ hetero-junctions. *Journal of Physics D: Applied Physics*. 2015;**48**:335103
- [122] Panchal G, Choudhary RJ, Kumar M, Phase DM. Interfacial spin glass mediated spontaneous exchange bias effect in self-assembled $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3:\text{NiO}$ nanocomposite thin films. *Journal of Alloys and Compounds*. 2019;**796**:196-202
- [123] Matsuzak K, Hosono H, Susak T. Layer-by-layer epitaxial growth of polar $\text{MgO}(111)$ thin films. *Physical Review B*. 2010;**82**:033408-1-4
- [124] Kaneko S, Ito T, Soga M, Motoizumi Y, Yasui M, Hirabayashi Y, et al. Growth of nanocubic MgO on silicon substrate by pulsed laser deposition. *Japanese Journal of Applied Physics*. 2013;**52**:01AN02
- [125] Susaki T, Kumada S, Katase T, Matsuzaki K, Miyakawa M, Hosono H. Fabrication of flat $\text{MgO}(111)$ films on $\text{Al}_2\text{O}_3(0001)$ substrates by pulsed laser deposition. *Applied Physics Express*. 2009;**2**:091403-1-4
- [126] Villegle JC, Radparvar PM, Yu LS, Faris SM. RF-sputter-deposited magnesium oxide films as high-quality adjustable tunnel barriers. *IEEE Transactions on Magnetics*. 1989;**25**:1227-1230
- [127] Chen X, Freitas PP. Magnetic tunnel junction based on MgO barrier prepared by natural oxidation and direct sputtering deposition. *Nano-Micro Letters*. 2012;**4**:25-29
- [128] Lee JH, Eun JH, Park SY, Kim SG, Kim HJ. Hydration of r.f. magnetron sputtered MgO thin films for a protective layer in AC plasma display panel. *Thin Solid Films*. 2003;**435**:95-101
- [129] Kaneko S, Funakubo H, Kadowaki T, Hirabayashi Y, Akiyama K. Cubic-on-cubic growth of a $\text{MgO}(001)$ thin film prepared on $\text{Si}(001)$ substrate at low ambient pressure by the sputtering method. *Europhysics Letters*. 2008;**81**:46001

- [130] Nakano T, Fujimoto T, Baba S. Measurement of surface roughness and ion-induced secondary electron emission coefficient of MgO films prepared by high-pressure sputter deposition. *Vacuum*. 2004;**74**:595-599
- [131] Singh JP, Kumar M, Lee IJ, Chae KH. X-ray reflectivity and near edge X-ray absorption fine structure investigations of MgO thin films. *Applied Science Letters*. 2017;**3**:47-52
- [132] Singh JP, Lim WC, Chae KH. An interplay among the Mg²⁺ ion coordination, structural order, oxygen vacancies and magnetism of MgO thin films. *Journal of Alloys and Compounds*. 2019;**806**:1348-1356
- [133] Singh JP, Lim WC, Lee J, Song J, Chae KH. Surface and local electronic structure modification of MgO film using Zn and Fe ion implantation. *Applied Surface Science*. 2018;**432**:131-139
- [134] Singh JP, Lim WC, Lee I-J, Won SOK, Chae KH. Surface structure of MgO thin films revealed from X-ray reflectivity and near-edge X-ray absorption fine structure measurements. *Science of Advanced Materials*. 2018;**10**:1372-1376
- [135] Singh JP, Kumar M, Lim WC, Lee HH, Lee YM, Lee S, et al. Growth of MgO on Si(001) by radio-frequency sputtering. *Journal of Nanoscience and Nanotechnology*. (In Press)
- [136] Wang WB, Yang Y, Yanguas-Gil A, Noe NC, Girolami GS, Abelson JR. Highly conformal magnesium oxide thin films by low-temperature chemical vapor deposition from Mg(H₃BNMe₂BH₃)₂ and water. *Applied Physics Letters*. 2013;**102**:101605
- [137] Ko JB, Kim SM. Preparation and electric characteristics of MgO films deposited by plasma-enhanced chemical vapor deposition. *Journal of Ceramic Processing Research*. 2009;**10**:643-646
- [138] Sartori A, Habra NE, Bolzan M, Rossetto G, Sitran S, Barreca D, et al. Stability study of a magnesium β-diketonate as precursor for chemical vapor deposition of MgO. *Chemistry of Materials*. 2011;**23**:1113-1119