Microwave Irradiation Assisted Synthesis of Gallium Aluminum Oxide Using Metal Acetylacetonate Precursors, Peculiar Observations and Safety Guidelines for Young Scientists

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Abstract. Microwave-assisted nano-particle synthesis has seen a boom in recent decade. Different variants are being tested all over the world, be it microwave-irradiation-assisted solvothermal synthesis, thin-film deposition using microwave plasma-assisted CVD, microwave-assisted combustion synthesis, or mist CVD. This technology induces self-catalysis, faster reaction time, less energy and is considered safer from other purely chemical variants. Gallium-Aluminum Oxide is an oxide of vital importance. Gallium Oxide in its most stable form, $\beta$-Ga$_2$O$_3$, has a band-gap of ~4.8 eV and cannot be used for deep ultra-violet (UV) applications (i.e., below 250 nm). Aluminum oxide is a wide band-gap material. The ionic radius of Aluminum ions is similar to that of Gallium ions. Therefore, according to Hume-Rothery rule, Aluminum ion can substitute Gallium in its lattice structure and vice-versa. This gives the possibility of band-gap engineering from 4.6 eV (band-gap, $\beta$-Gallium Oxide) to 8.8 eV (band-gap, $\alpha$-Aluminum Oxide), extending the application of Gallium Oxide to deep UV applications. In present work, three different compositions of Gallium-Aluminum Oxide thin films (varied by mass) are deposited, using microwave-irradiation-assisted solvothermal thin-film deposition technique and metal acetylacetonate precursors. A stoichiometric ratio of ~5:1 is obtained for 90:100 variation. For 50:50 and 10:90, the stoichiometric ratio remained close to 5:5 and 1:9, respectively, showing good agreement with the Gallium to Aluminum ratio in precursor solution. The investigation is done by using energy dispersive X-Ray analysis. A particle variation from 5 nm to 500 nm was obtained as calculated from Field Emission Scanning Electron Microscopy (FESEM) results. In second part of the work, the corresponding author wants to strongly argue that “Microwave in kitchen is not same as microwave in lab” and report some of the observations from lab to pave a way towards a truly Green or safer technology.

1. Introduction
Microwave-assisted synthesis and deposition have drawn much curiosity among researchers worldwide. Many of the questions are still unanswered, and with these unanswered questions, the technology is slowly maturing. Early researchers reported cracking of vessels, generation of sparks, formation of hot-spots, super-heating phenomenon, faster reaction time, plasma generation inside a microwave, and attempted to explain these phenomena using different theories [25, 26]. Slowly microwave-assisted technology started to be used widely in laboratories, especially in calcination of ceramics, in organic chemistry, fusing glass rods, nano-particle synthesis and thin-film deposition.
Today microwave-assisted synthesis is a rapidly evolving technology for drug discovery, material processing and is being more extensively used in chemistry and nanotechnology. With more sophisticated instruments available, it is easier to tune/monitor the properties of materials (to be synthesized or deposited).

The advantages of microwave-assisted synthesis over other thin-film deposition methods are (a) Faster reaction time [22, 23, 29] (b) comparatively lower cost than CVD (Chemical Vapor Deposition), ALD (Atomic Layer Deposition) and other high-quality thin-film deposition methods (c) higher yield [25, 26] (d) deposition on dielectric or non-metallic surface [27, 28, 29] (e) high-quality thin-film deposition, specifically in case of metal oxides, metal sulfides [31, 32] or metal ion-substituted metal oxides (e.g., ZnO [30], Ga2O3 [22], ITO [27]).

Gallium-Aluminum Oxide is an important oxide for deep UV applications. With variation of composition, if right stoichiometric ratio can be obtained, there is a possibility of band-gap engineering, i.e., using Gallium-Aluminum Oxide for applications beyond 200 nm, which is currently not possible with Gallium-Oxide alone.

Al2O3 and Ga2O3 exist in multiple phases. Al2O3 has six known phases, α, γ, δ, κ, ε and θ (including other debatable phases). The phase, α-Al2O3 is thermodynamically stable phase and ε, κ are polar analogous to Ga2O3. β-Ga2O3 (thermodynamically stable), α-Ga2O3, γ-Ga2O3, and δ-Ga2O3 ε-Ga2O3, and κ-Ga2O3 phases (meta-stable phases) are the known phases of Ga2O3 [1-5]. The ε-Ga2O3 phase is the second most stable phase of Ga2O3 and is polar in nature.

Attempts to deposit Gallium-Aluminum Oxide have been done in past [6-9]. Some of the very recent attempts are shown in table 1.

Table 1. Recent attempts to deposit Gallium Aluminum Oxide thin-films

| Product                        | Substrate         | Method                  | Remarks                                                                 | Year of Publication and References |
|--------------------------------|-------------------|-------------------------|------------------------------------------------------------------------|------------------------------------|
| γ-(AlxGax)2O3 thin-films       | Defect spinel     | Mist CVD                | Band gap of film varied up to 6 eV on increasing Al content (lattice matching with misfit locations, reported). | 2020 [10]                          |
| β-Ga2-xAlxO3 and intermediate α, β polymorphs | No Substrate-Powder | Solvothermal Synthesis Method | Transformation of γ-Ga2-xAlxO3 into monoclinic β-Ga2-xAlxO3 for 0 ≤ x ≤ 1.3 above 750 °C, intermediate α and β polymorphs for 1.3 < x < 1.8 and α-Ga2-xAlxO3 for x=1.8 | 2020 [11]                          |
| Aluminum-Gallium Oxide thin-films | c-plane Sapphire | Pulse Laser Deposition (PLD) | Average roughness of thin-film decreases to 0.69 nm, and 0.70 nm after annealing in Oxygen and Nitrogen atmosphere, respectively, at 900°C. Also, optical band-gap increases from 5.14 eV to 5.41-5.46 eV. | 2020 [12]                          |
| ε-(AlGa)xO3                   | Sapphire (0001)   | Pulse Laser Deposition (PLD) | Solar-blind photo-detector, fabricated by depositing a pure phase of ε-(AlGa)xO3 on sapphire substrate. | 2021 [33]                          |
| β-(GaAl)xO3                   | c-Sapphire        | Thermal Annealing       | Al substitution up to 0.81, highly homogeneous ternary thin-films obtained at temperature ≤1200 °C as reported. | 2021 [13]                          |
Attempts to deposit Gallium-Aluminum Oxide films by microwave-assisted solvothermal method, has not been reported till the date of reporting this work, to best of author’s knowledge. In this work, an attempt to deposit thin-films of $\text{(Ga}_{x}\text{Al}_{1-x})_2\text{O}_3$ (where, $x \in \{0.90, 0.50, 0.10\}$) and maintain stoichiometric ratio thereof is attempted, on Silicon p-type (400) substrate, to explore the possibility of this low cost and easily accessibele deposition technology.

2. Method

The experiment is divided into two parts; the precursor preparation and the thin-film deposition. The primary aim is to understand thin-film deposition assisted using microwave-irradiation and possibility of composition variation.

2.1. Precursor Preparation

The precursors are prepared by adding 0.085 M, 0.047 M, 0.009M, of Gallium (III) Acetylacetonate (CAS-14405-43-7, Sigma Aldrich, 99%) to 0.011 M, 0.053 M, 0.096 M, of Aluminum (III) Acetylacetonate (CAS-13963-57-0, Sigma Aldrich, 99%) respectively. The solutions are prepared in 30% Ethanol and 70% of MilliQ ultra-pure DI (De-ionised) water. Acetic acid (99.9%), at stirring condition of 500 rpm, at 50°C, is added drop-wise to these solutions until a clear liquid is obtained. These solutions (now termed as precursors) are used for deposition and named as Ga90Al10, Ga50Al50 and Ga10Al90, for the purpose of documentation. The composition is varied by mass to ensure scalability and repeatability of experiments.

2.2. Substrate Preparation

Substrates used for deposition are cleaned by RCA-2, Piranha (3:1) solution, including cleaning by acetone, IPA (Isopropyl alcohol) solution and in-between with the jet of ultra-pure DI water. No additional pre-treatment is used on the substrate.

2.3. Thin-film Deposition Set-up

Fig.1. shows the thin-film deposition set-up used in this experiment. The microwave used for the experiment is single mode domestic microwave with magnetron frequency 2.45 GHz (IFB, Model No. 17PMMEC1) and cavity of $31.5 \times 19.8 \times 29.4 \text{ cm}^3$. Round bottom flask with 24/29 connector (Borosil®, 4380C21) is used as a reaction vessel. The vessel-condenser assembly is sealed with Teflon-tape to avoid any leakage inside/outside the chamber.

![Figure 1. Experimental set-up for solvothermal based microwave-assisted deposition](image)

2.4. Thin-film Deposition

The films are deposited by immersing the substrate in the corresponding solutions (Ga90Al10, Ga50Al50, and Ga10Al90). The cleaned substrate is immersed in the solution and irradiated by 2.45
GHz, 700W microwave power for 12 minutes, as shown in Fig.1, to ensure a film of sufficient thickness can be obtained and can be analyzed by accessible characterization tools.

3. Characterization

3.1. X-Ray Diffraction (XRD) Spectroscopy

The X-Ray Diffraction (XRD) spectroscopy is an important tool to understand crystal structure, phases and approximate particle size of thin-films deposited. XRD results of deposited films along with p-type Silicon substrate are shown in Fig.2. The high-intensity broad doublet peak of p-type Silicon Substrate at 69.03° and 69.23° corresponds to (400) plane [14]. The peak at 32.85° and 32.94° corresponds to forbidden (200) plane reflections [15], the intensity of these peaks is such that they are nearly undetectable in presence of 69.03° and 69.23° doublet peaks.

The peaks at 61.58°, 65.77° and 65.88°, intensity 29.119 cps, 24.26 cps and 18.96 cps, respectively, are low intensity peaks with an additional peak of 68.74° with intensity 532 cps are assigned to substrate. These peaks are likely to originate from multiple reflections coming back from detector and diffracted by multiple planes of substrate due to very high intensity 69.03° and 69.23° doublet peak (~10^6) (Fig. 4).

Since most of the peaks of blank substrate match with corresponding or shifted peaks of different phases of Ga_2O_3 and Al_2O_3 (JCPDS 00-046-1215, 01-086-1410, 01-074-1776), it was important to detect all the reflections from the blank substrate. Also the obtained data and results are reported ‘as obtained’ in Fig.2, due to following reasons (a) peak positions appear to be shifted after using smoothening function, (b) change in peak intensity, in different thin-films, was not detectable if normalization function is being used, (c) scaling up leads to peak broadening and scaling down to narrowing and (d) peaks of substrate and the peaks of deposited thin-films are very closely related and 20 corrections may lead to misinterpretations.

With thin-film deposition, the intensity of XRD peaks increased, and the peaks appear to be shifted, as can be seen from Fig.2 and Fig.3.

![Figure 2. X-Ray Diffraction pattern of deposited thin-films on p-type Silicon substrate along with diffraction pattern of substrate (Yellow)](image)

The shift in peaks can be attributed to increased compressive strain. Aluminum ions are smaller than Gallium ions, and if substituted in lattice, they most likely will cause compressive strain, i.e., reduced spacing between planes, thus increase in 20 value. Also, due to d-block contraction, the atomic radius...
of Gallium atom is comparable to Aluminum atom (radii (Al) = 1.1667Å, radii (Ga)= 1.3514Å) [16]; ionic radii too follows the same trend, radii (Al\(^{3+}\))=0.468 Å and (Ga\(^{3+}\))=0.547 Å (Shanon’s Radii) [17]. The reason XRD peaks of same phase of Al\(_2\)O\(_3\) and Ga\(_2\)O\(_3\) are close together can be derived from here, (during substitution the strain in lattice is lower, thus lesser change in lattice structure).

As can be seen from fig.3, the peaks are closely matching with Al\(_{12}\)Ga\(_2\)O\(_24\) (JCPDS 00-022-0615), a=b=5.60, c=22.71, α=β=90, c=120, space group P63/mmc). Using simulation results it was observed that the 2θ values of Ga10Al90 corresponds closely to cell parameters a=b=5.97, c=21.7, α=β=90, γ=120 (space group P63/mmc), the 2θ values were obtained by varying ‘c’ with respect to constant ‘a’ and ‘b’ vice-versa. No clear 2θ shift can be obtained in between Al\(_2\)Ga\(_2\)O\(_24\) and simulated results. The shifted peaks of Aluminum Oxide are also present in Ga10Al90, which is equally likely as Aluminum to Gallium ratio is higher (can also be seen from EDX results para 1, section 3.2.).

In Ga50Al50, the peaks 33.05°, 68.78°, 69.13° corresponds to Al\(_{12}\)Ga\(_2\)O\(_24\), where 69.13° is highest intensity peak of both i.e., thin-film and Al\(_{2}\)Ga\(_{2}\)O\(_{24}\). In 2010 Y. Wang et al., reported nano-nets [18] with matching peak positions of Al\(_{12}\)Ga\(_{2}\)O\(_{24}\) (Ga\(_{2}\)O, 11Al\(_2\)O\(_3\)), (according to FESEM results similar nano-nets with much smaller size (~7-21 nm) can be seen in cracked microstructures, in Ga50Al50 thin-films (Fig. 6 (d)). The peaks are shifted from the original values, citing towards the fact that there is a possibility of change in formula or possibility of change in crystal structure. The peak 61.7° corresponds to β-Al\(_2\)O\(_3\) (JCPDS 10-0414), the other corresponding peaks from fig.2. are assigned to Al\(_{2}\)Ga\(_{2}\)O\(_{24}\) and AlGaO\(_3\) (as shown in fig.3).

![Figure 3. High Resolution X-Ray Diffraction spectra of deposited thin films with respect to silicon p-type substrate (blank)](image-url)

Ga90Al10 peaks are lower in intensity than Ga10Al90 and Ga50Al50, the morphology of this thin film is also different (as shown in fig. 9.), the peaks are assigned to β-Al\(_2\)O\(_3\) (JCPDS 10-0414) and Al\(_{12}\)Ga\(_{2}\)O\(_{24}\) as shown in fig.3. The peak 32.87° cannot be assigned directly to any phase or JCPDS card number, but it can be assigned closest to ε-Ga\(_2\)O\(_3\) (JCPDS 06-0509, 33.268) or β-Ga\(_2\)O\(_3\) (JCPDS 11-0370, 33.525°). The peak 29.401° corresponds to ε-Ga\(_2\)O\(_3\) (JCPDS 06-0509) which is not present in any other phases of Ga\(_2\)O\(_3\) or Al\(_2\)O\(_3\), and also absent in blank, a shifted peak of the same can be seen in Ga10Al90 (29.416°) and Ga50Al50 (29.401°), which indicates towards possibility of presence of localized substituted phases of ε-Ga\(_2\)O\(_3\) along with substituted Al\(_{12}\)Ga\(_{2}\)O\(_{24}\) and AlGaO\(_3\) oxides.
3.2. Energy Dispersive X-Ray (EDX) Spectroscopy

The EDX spectra were obtained using Bruker SDD (Silicon Drift Detector)-EDX configured with Nova NanoSEM 450 FESEM. The results obtained for thin-films are tabulated in Table 2.

Larger percentage of SiK was observed in thin-films with cracks (Ga50Al50), as larger Silicon area was exposed due to crack formation. In smaller area the percentage of Gallium, Aluminum and Oxygen remained smaller, however with larger area the percentage of Gallium, Aluminum and Oxygen was higher, this observation is due to presence of larger number of atoms in larger area and vice-versa.

Table 2. The EDX results obtained from thin-films (Ga10Al90, Ga50Al50, Ga90Al10) on p-type Silicon Substrate

| Elements | Ga10Al90 | Ga50Al50 | Ga90Ga10 |
|----------|----------|----------|----------|
|          | Weight % | Atomic % | Weight % | Atomic % | Weight % | Atomic % |
| OK       | 41.835   | 56.345   | 9.696    | 16.532   | 26.25    | 51.528   |
| GaL      | 3.345    | 1.03     | 8.57     | 3.348    | 51.19    | 23.075   |
| AlK      | 17.1     | 13.645   | 3.976    | 4.028    | 3.58     | 4.1675   |
| SiK      | 37.725   | 28.975   | 77.756   | 76.088   | 18.98    | 21.233   |

Fig.4. The pie-chart obtained by plotting GaL, AlK values, from three composition variations.

Figure 4. The percentage of Gallium to Aluminum in deposited thin-films (a) Ga10Al90 (b) Ga50Al50 and (c) Ga90Al10

However the results obtained may seem encouraging from Fig.4, but the concept of percentage can be misleading. Thus, revisiting Table 2, the ratio of Ga to Al atoms in different compositions can be re-written as ~ 1:13, ~1:1.20, and ~5.5:1 for Ga10Al90, Ga50Al50 and Ga90Al10, also presence of Hydrogen cannot be detected with EDX.

3.3. Field Emission Scanning Electron Microscopy (FESEM)

FESEM is a powerful technique to analyze surface morphology of thin-films; the images are captured using Nova NanoSEM 450 FESEM. The films deposited are not entirely uniform, but are reported as it, such that it can be understood what can be directly obtained from a domestic microwave oven, using present solvent system.

Fig.5. shows the FESEM image of Ga10Al90 thin-films, the average grain size distribution ranges from ~2.7 nm ~34 nm. The fine grain-like structures can be seen in the grown thin-films, however haphazardly grown structures 75 nm to 272 nm, with an average thickness of 141 nm are also obtained as can be seen from Fig.5 (left). To understand the morphology better 3D plots of the thin-film surface are obtained using ImageJ 1.53c [21], with the help of inverted structure it can be seen, that the growth
is likely to occur in net like structures. The grains follow a nano-hump like patterns and the spaces around are following inverted-hump (net-like) patterns (fig.5 (right)). As XRD results indicate towards possibility of presence of nano-nets, all the images obtained were thoroughly searched for net-like morphology, however Ga10Al90 showed nano-humps like morphology with size as small as 2.7 nm.

**Figure 5.** The FESEM image of Ga10Al90, 3D surface plot and corresponding inverted plot

Ga50Al50 thin-films are shown in fig. 6., the films at lower magnification where appeared to be nearly uniform, on magnification, two kinds of cracks were observed, smaller cracks (fig.6.(b), ~400nm) and bigger cracks (fig.6. (c),~2 μm). It is important to understand which morphology can be obtained if the microstructures could be removed, to understand this in better context image (fig. 6 (d) and fig.6 (e)) were zoomed in, and the results are shown in fig.7.

**Figure 6.** FESEM images of Ga50Al50 ((a) (5000 x); (b), (c) (10000 x); (d), (e) (200 000x))

**Figure 7.** (a) Sectional zoom fig. 6 (d); (b) sectional zoom fig.6 (e)

Fig.7(a) shows net like structures with diameter varying from ~5 nm to 27 nm. The films deposited below the cracks are of similar grain-like morphology as that of Ga10Al90 (fig. 5) thin-films (fig. 7(b)). Ga90Al10 thin-films are shown in (fig.8). Ga10Al90 and Ga50Al50 show similar kind of deposition in the form of debris/ microstructures, but rod and flower like microstructures (length varying from ~500 nm to ~4 μm) are observed in Ga90Al10.

**Figure 8.** The FESEM images of Ga90Al110 in different magnification

**Figure 9.** Sectional zoom fig. 8 (d) and fig. 8 (b)

These flowers like structures can also be seen buried inside uniform looking part of thin-film (fig.8 (a)), indicating towards a different morphology that other two counterparts. An important point to note
here is that while the substrate is dipped in the solution and irradiated by microwaves the actual-film growth is completed within first few seconds/minutes of irradiation (depending upon the reaction mixture used, irradiation time, irradiation power, volume of solution and the substrate) and a saturation point is reached wherein the excess precursor not used in the film formation starts participating in deposition and falls and collects over the actual film surface in the form of debris. These debris have much looser compaction and are much thicker and bigger in size than actual thin-film. The thin-films grown show clear sign of this over-deposition.

3.4. Solvent System and UV-Visible Spectroscopy

The aim of study was to use minimum number of solvents during the study. Thus, solution for deposition was firstly prepared with ethanol and metal acetylacetonates only. The solubility of metal acetylacetonate was found to be poor with water and even with water-ethanol solution.

Since, solubility affects the quality of thin-film deposited therefore acetic acid was used as a solvent. High dielectric constant of Ethanol helped the solvent system to get increased heat, due to increased dielectric heating. Both are polar protic solvents and used on purpose to stabilize the carbo-cations. Water is used to ensure lab safety.

![Image](image_url)

**Figure 10.** UV-Visible Spectra of thin-films with and without acetic acid (Left), change in FWHM (nm), Wavelength (nm), Absorbance (Right)

The percentage of ethanol (99.9%) should be below 50% of the solution, to avoid any burning, explosion or spilling of solution inside microwave, when operating at high power and high-temperature conditions, increased ratio of water helped with the same. The solutions prepared, by using metal acetylacetonates and 30% ethanol-water solution with 12 minute, 2.45 GHz, 700 W microwave-irradiation, are represented by Ga10Al90_1, Ga50Al50_1 and Ga90Al10_1 in fig.10.

The UV Visible spectroscopy gives very important details about electronic configuration of a material. Therefore, the solutions prepared before and after adding acetic acid with 12 minute microwave-irradiation, were tested and the results are shown in fig. 10; with addition of acetic acid the Full Width Half Maxima (FWHM) increased and the UV-Visible plots shifted by 17.18 nm on an average towards a lower wavelength i.e., towards higher energy.

4. Discussion, Conclusion and Future Scope

The study aimed to find the possibility of composition variation using microwave-assisted solvothermal synthesis method, the study resonates with the possibility. The XRD peaks of various phases of Ga2O3 and Al2O3 are very close to each other. Taking blank as reference the prominent peaks of Ga10Al90, Ga50Al50 and Ga90Al10, are shifted by 0.12, 0.10 and 0.03 20 degrees (on average) as can be seen from fig.3. With decrease in Aluminum content the change in 20 degrees is also
decreasing, which indicates towards Aluminum substitution in Gallium lattice (para. 3, section 3.1).

But, as the peak intensity decreases, the change in 2θ values of peaks become nearly indistinguishable.

During substitution it is difficult to tell exactly which phase and which compound has been formed using XRD i.e., XRD alone is insufficient technique for thin-film characterization of Gallium-Aluminum Oxide, due to overlapping of peaks in different phases of Ga$_2$O$_3$ and Al$_2$O$_3$.

Uniform films obtained below the microstructures (debris) as can be seen from fig. 5, 7 and 8, providing strong indication of uniformly grown thin-films up to a saturation point. The evidences of nano-humps beneath cracks (fig. 6. (e)) also provide a strong indication of a uniformly grown layer. Due to current limitations of detection a sufficiently thick layer was grown, however authors want to look forward the possibility of deposition of thin-films and observe crystal structures thus formed using same solvent system at lower deposition power and time of irradiation. Also, study the effect of post-processing including ultrasonication to remove sub-micron/micro structures.

The microwave-assisted solvothermal synthesis has potential to grow metal-substituted metal oxide thin-films with current metal acetylacetonates, acetic acid and ethanol-water based system. Future work may look forward in improving the thin-film quality and use this low cost microwave technology for device application, as demonstrated by Piyush et al’s group in 2018 [22] with single solute-multiple-solvent system.

The change in UV-Vis spectra with addition of new solvent (fig. 10) indicate towards role of solvents in defining property of material inside microwave. It will be interesting to explore more about the effect of addition of new solvents and change in band-gap of deposited thin-films thus obtained.

5. Peculiar Observations and Lab Safety Guidelines

The experiments used in this research work and subsequent observation in working with microwave, multi-solvent systems and three different substrates over a period of more than 18 months has provided with following observations crucial for lab safety, and are reported in this work for the greater benefit of scientific community.

5.1. Cracking of Vessels and Effect of Contamination

a). Not only reaction mixture but the substrate also plays an important role in synthesis of nanoparticle in microwave-assisted synthesis and variants i.e., everything inside microwave matters, even the substrate used for deposition, ignoring this fact may lead to accidents inside microwave/or in the lab. The vessel (chemical resistant) shown in fig.11 (f) cracked as Silicon was used for deposition instead of Glass or cloth material. The crack took place within 30 seconds of irradiating substrate with 2.45GHz, 700W irradiation.

![Figure 11.](image-url)
b). Contamination may change a microwave transparent material to microwave absorbing material. Thus, working with microwaves one needs to be aware of any possible source of contamination. While accidently using a Quartz crucible (used previously for heating human hair (at 800°C in a heating furnace)), inside microwave for coating Glass substrate with a modified microwave-assisted thin-film deposition method, the Teflon tape wrapped outside the nozzle burned instantly after 30 seconds of 2.45 GHz, 700 W irradiation. The temperature inside microwave reached above 370°C (ignition temperature of Teflon) and the microwave coating burned (Fig. 11 (c)).

c). After 15 minutes when the incident was reported to head-supervisor and microwave power as turned off instantly, the microwave door was opened as the door was opened the glass slide fused inside the Quartz boat (Fig. 11 (a)) with a crackling sound, the temperature of Quartz boat was still above 50°C i.e., the temperature inside microwave would had gone much higher than 370°C, as when the microwave power was closed instantly after that incident the glass inside was red hot, as can be seen from microwave door inside, due to safety reasons the door was kept closed for a calculated time.

5.2. Commercial Microwave, Modification and Lab-safety

a). Commercial/Domestic microwaves come with lab/domestic safety guidelines and ensure maximum customer safety. However before attempting a major experiment, or while self-modifying system, make sure the coating inside is intact and test the equipment ‘under expert supervision’.

b). If using vapors or liquid inside microwave, make sure the periodic structures in the doors of microwave system are free of contamination, when they are being used again; especially when microwave has been used to deposit different chemicals over a long period of time, or is been used too frequently (if any part of microwave, especially door is heating frequently, there may be chances of microwave leakage and a need to discard the existing equipment).

c). Vapors coming out of microwaves should be treated with same precautions as fumes coming out of a chemical reactor are being treated, especially when dealing with heavy metal compounds. Have a close look in changing health patterns while working with microwave, early detection can help one to take additional precautions.

d). Do not use domestic microwave equipments, or chemical resistant borosilicate petri-dishes for heating bare Silicon/Sapphire substrate inside microwave for chemical experiments, the petri-dishes may crack instantly or while attempting to open microwave door, this can be dangerous.

e). Do not directly heat the substrate/material, obtained after microwave irradiation, the material may explode or the vessel/dish in which it is to be heated may crack.

f). Always arrange and wear proper safety equipments, P-100 respirators or N-95 masks, proper chemical resistant safety goggles and necessary PPE kits while performing an experiment in microwave.

Corresponding author wants to strongly argue that lab safety is equally important in microwave-irradiation assisted synthesis, as everything inside microwave plays an important role and any carelessness or ignorance may lead to a major accident or affect the health of researcher severely over time. The corresponding author has skipped accidents nearly on modifying and experimenting with new materials inside microwave. Thus, as a moral responsibility these safety guidelines are included while reporting this research work.

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