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Preparation and Experimental Investigation of Lanthanum Hexa-Aluminate Calcinated Powders

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ABSTRACT

Lanthanum hexa-aluminate (LHA) has garnered significant attention as a high-performance material for thermal barrier coating (TBC) applications, primarily due to its exceptional thermal stability, low thermal conductivity, and resistance to high-temperature degradation. However, LHA's ability to retain its structural integrity under these extreme conditions. The goals of this work are to synthesize and characterize three different compositions of Alumina/Lanthanum hexa-aluminate (Alumina/LHA) ceramic powders. The synthesis process was carried out using chemical precipitation followed by filtration process. After synthesis, the ceramic powders were calcinated. The calcination was carried out at three different temperatures: 750°C, 900°C, and 1000°C for each sample. This thermal treatment enabled the researchers to study the evolution of the material's crystalline structure as a function of temperature and composition. X-ray diffraction (XRD) peak profile analysis was performed to determine phase transformations and investigate the crystallographic properties of the powders for changes induced by calcination. Field emission scanning electron microscopy (FESEM) was used to study the microstructure, while energy-dispersive X-ray spectroscopy (EDX) was performed to analyze elemental composition and distribution in the samples.

1. INTRODUCTION

Over the past decade, rare earth elements (REEs) have gained significant attention due to their essential role in a wide range of advanced technologies and industrial applications. These unique metals, which include elements like lanthanum, are fundamental to numerous fields, including electronics, medicine, renewable energy, and defense industries [1]. Their remarkable physical and chemical properties, such as high thermal resistance, magnetic behavior, catalytic efficiency, and electrical insulation, make them indispensable in modern technological advancements. As a result, the demand for REEs has been steadily increasing, driving research into their applications and synthesis techniques [2, 3]. Among these elements, Lanthanum hexa-aluminate ($LaAl_{11}O_{18}$) has emerged as a highly promising composite material. This compound is formed through the chemical reaction of lanthanum oxide (La_2O_3) and aluminum oxide (Al_2O_3), resulting in a stable structure with outstanding performance characteristics [4]. LHA has gained particular significance in high-temperature applications, primarily as TBC in aerospace and industrial sectors [5, 6]. Traditionally, Yttria-stabilized zirconia (YSZ) has been the most widely used material for TBC; however, it suffers from phase instability and degradation at elevated temperatures. In contrast, LHA offers superior high-temperature stability, making it a viable alternative to YSZ-based coatings. The unique crystal structure of LHA contributes to its exceptional thermal stability, allowing it to withstand extreme conditions found in gas turbines, jet engines, and other high-temperature environments. Its ability to endure prolonged exposure to intense heat without significant degradation extends the lifespan of coated components, reducing maintenance costs and improving operational efficiency. Additionally, LHA exhibits excellent electrical insulating properties, making it an ideal candidate for applications requiring both thermal and electrical isolation. This characteristic broadens its potential use in electronic components, power systems, and energy storage devices. Despite its promising attributes, the production of LHA nanoparticles presents significant challenges. The synthesis of LHA involves complex chemical processes that demand precise control over composition, temperature, and reaction conditions to achieve the desired nanoscale structure [7]. The high cost of raw materials,

energy-intensive processing, and the need for specialized equipment further add to the challenges associated with large-scale production. These factors make LHA an expensive material, limiting its widespread industrial adoption. To overcome these challenges, ongoing research and development efforts are focused on optimizing synthesis techniques to make LHA more economically viable. Scientists are exploring cost-effective and scalable production methods, such as sol-gel processing, combustion synthesis, and hydrothermal techniques, to enhance yield while maintaining material purity and performance. Additionally, advancements in nanotechnology are playing a crucial role in refining LHA's structural properties, further improving its efficiency in high-temperature applications. With continuous progress in manufacturing techniques and material engineering, LHA is poised to become a sustainable and widely adopted solution in industries that require superior thermal stability, corrosion resistance, and electrical insulation. Its potential to replace traditional materials in critical applications marks a significant step toward more efficient and durable high-temperature coatings, making it an essential focus of future research in the field of advanced materials.

In recent years, significant research efforts have been directed toward the development of advanced synthesis techniques for producing nano-sized powders with precise control over their morphology, particle size, and chemical composition. These nano powders have gained considerable attention due to their enhanced mechanical, thermal, and electrical properties, making them highly valuable for applications in ceramics, electronics, catalysis, and biomedical fields. To achieve the desired properties, researchers have explored various fabrication methods, each offering unique advantages in terms of particle uniformity, production efficiency, and cost-effectiveness. One such technique is shockwave consolidation, which utilizes high-energy shockwaves to compact fine powders into dense, uniform nanostructures. This method is particularly useful for preserving the intrinsic properties of the nano powders while achieving enhanced mechanical strength. Another widely used approach is ball milling, a mechanical process that involves the repeated impact of grinding media to break down bulk materials into nano-sized particles. This technique is cost-effective and scalable, making it suitable for mass production. Additionally, electroless plating has been employed to deposit thin metallic coatings on nano powders, improving their conductivity, corrosion resistance, and catalytic activity. This method is particularly beneficial for applications in electronics and surface engineering, where precise coating thickness is essential. Meanwhile, sol-gel processing has emerged as a highly effective wet-chemical technique for synthesizing nano powders with excellent purity and homogeneity. This method involves the transition of a sol (a colloidal solution) into a gel-like network, followed by drying and heat treatment to obtain the final nano powder. It is widely used in ceramic processing, optical coatings, and biomedical applications. Furthermore, chemical precipitation combined with filtration is another efficient approach for synthesizing nano powders. This method involves the controlled precipitation of solid nanoparticles from a liquid solution, followed by filtration and drying to obtain fine powders with tailored properties. It is particularly advantageous for producing nano powders with high purity and controlled stoichiometry, making it ideal for catalysts, pigments, and advanced functional materials. Collectively, these advanced synthesis techniques have revolutionized the production of nano powders, enabling researchers to fine-tune their properties for next-generation industrial and technological applications. Ongoing studies continue to explore innovative processing routes and hybrid techniques to enhance efficiency, reduce production costs, and improve the overall performance of nano materials in diverse fields [8-11]. These methods have been explored for their ability to produce fine powders with desirable properties, but their suitability often depends on factors like cost, scalability, and complexity. In the present study, the chemical precipitation and filtration method was chosen due to its cost-effectiveness and ease of implementation, making it a practical approach for synthesizing Alumina- LHA ceramic powders. Post-synthesis characterization is a critical step to ensure the desired properties of the powders, as materials at the nanoscale exhibit significantly altered behaviors compared to their bulk counterparts [12, 13]. Nano particles are particularly sensitive to changes in size, shape, and structure, which can drastically impact their physical and chemical properties [14]. To analyze these characteristics, researchers commonly employ advanced characterization techniques such as XRD, field emission scanning electron microscopy (FESEM), photoluminescence spectroscopy (PLS), and transmission electron microscopy (TEM). Among these, XRD stands out as a relatively simple and reliable method for determining the crystal size and phase composition of powders, making it an essential tool in nano powder research [15]. In short LHA is significant for high-temperature applications due to its unique properties like Thermal Stability, structural integrity and does not decompose at very high temperatures, often exceeding 1600°C. This makes it suitable for use in environments with extreme thermal demands. Low Thermal Conductivity is another important feature because of its crystal structure (hexa-aluminate phase) having inherent low thermal conductivity, making it an excellent thermal barrier material. This property is crucial for protecting components from high heat flux in aerospace engines and turbines. Compatibility with Composite Materials When combined with other materials, such as AZ91E magnesium alloys, LHA improves high-temperature performance by enhancing properties like creep resistance, oxidation resistance, and dimensional stability. Applications such as TBCs- Used in jet engines and gas turbines to protect metal components. Refractory Materials- Ideal for applications like furnace linings and other high-temperature industrial equipment. This study aims to address an important gap by investigating the effect of calcination temperature on different volume fractions of Alumina-LHA ceramic powders the work seeks to identify optimal processing parameters. This methodology offers a dual advantage: it deepens the comprehension of how the material responds to thermal treatment while simultaneously lowering the overall expenses associated with nano powder production. By analyzing the material's behavior under varying thermal conditions, this study provides critical insights that can lead to more efficient manufacturing processes. Additionally, the findings are expected to play a pivotal role in enhancing both the cost-effectiveness and functional performance of LHA-based materials. These improvements align with the growing industry demand for high-temperature-resistant and economically viable solutions, making this research highly relevant for industrial applications seeking optimized material performance at reduced costs.

2. MATERIALS AND METHODOLOGY

Aluminium oxide, Lanthanum oxide, Aluminium nitrate, Ammonium carbonate, Nitric acid, and Citric acid are used in the current study. Krish Met Tech Pvt. Limited in Chennai provided the supply of lanthanum oxide and high-purity aluminium oxide/alumina were used with a mean particle size of 50 microns. Three different compositions of Alumina/LHA powder varying from 20 to 60% LHA with a 20% LHA volume difference were produced using chemical precipitation and filtration technique. Figure 1 depicts pictorial representation of powders preparation. Stoichiometric Eqns. (1) and (2) were used to synthesize LHA powders [16]. Figure 1 gives a detailed Photographic Representation of Powder Preparation.

$$La_2O_3 + 6\,HNO_3 + 9H_2O - \rightarrow 2[La(NO_3)_3.6\,H_2O] \quad (1)$$

$$2[La(NO_3)_3.6\,H_2O] + 22\,Al\,(NO_3).9\,H_2O \rightarrow 2LaAl_{11}O_{18} + 72HNO_3 + 174H_2O \quad (2)$$

A 250 mL glass beaker filled with distilled water and weighed Ammonium Carbonate, (NH4)2CO3 of 19.21716 g is added to it. The solution is stirred for some time to form a solution of 0.2M Ammonium Carbonate. In beaker 1, 0.2382g of Alumina powder, Citric acid of 0.05 wt% and appropriate water is added to form a stable solution thereafter magnetic stirring is done for15 minutes. In beaker 2, 1.0720 g of Lanthanum Oxide powder, 0.8229 mL of Nitric acid is added with sufficient amount of distilled water to form Lanthanum Nitrate. 27.152 g Aluminum Nitrate is weighed and added to beaker 2. The solution is thoroughly blended followed by 15 minutes of heating. The solution formed in beaker 2 was added to 0.2M of Ammonium Carbonate solution in a drop-by-drop pattern to form precipitate. This precipitate solution is filtered using a Wattman 41 filter paper to get Al_2O_3-LaAl11O18 powder after drying overnight. Finally, the prepared powders ranging from 20 to 60 vol% LHA were calcinated at 750°C, 900°C, and 1000°C for duration of 2 hours with a controlled heating rate of 4–8 0C/min to prevent cracking, ensure homogeneity, and promote proper phase transformation. Post-calcination, XRD is used to confirm the formation of the desired LHA phase.

Using MINIFLEX 600 machine, in a 2θ range from 3 to 90° with step size of 0.02°. XRD data is extracted for prepared powders followed by analysis of peak using Match software. As the phases between Lanthanum and Alumina are more signifying powders are properly blended with one another. Lanthanum aluminate ($AlLaO_3$) and LHA (Al11.5La0.85O18.5) peaks are identified which matches with the JCPDS cards 31-0022 and 33-0699 with magneto plumbite phase which confirms with the literature [17, 18].

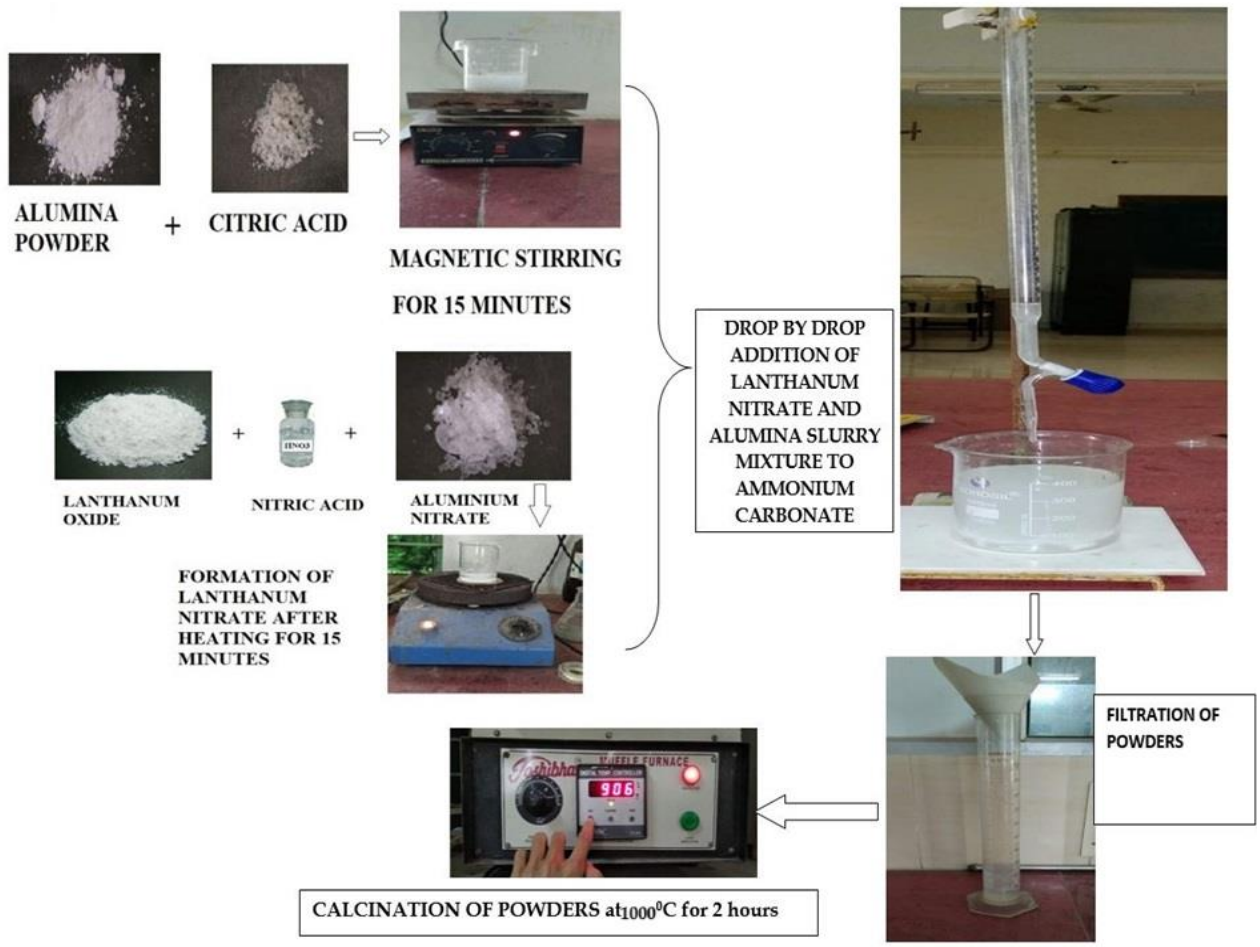


Figure 1. Photographic representation of powder preparation

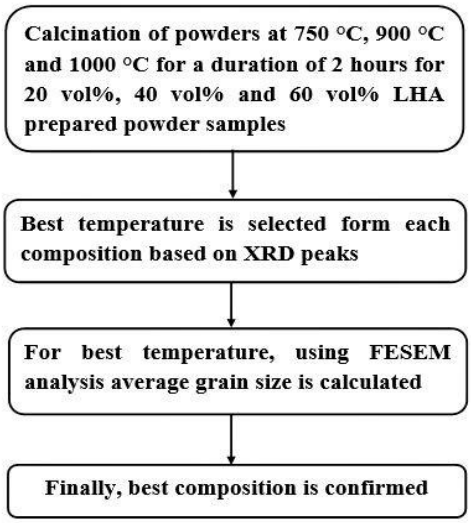


Figure 2. Methodology of present work

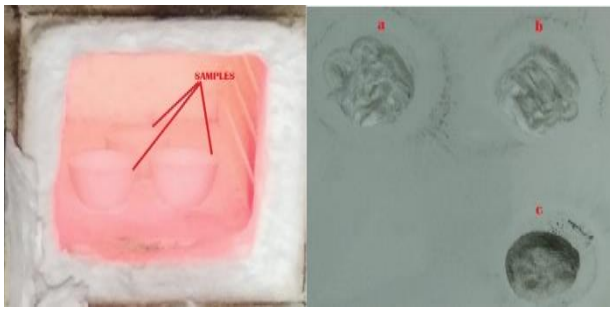


Figure 3. Calcination process with in a muffle furnace is carried out at 1000℃ for a) 20%LHA b) 40%LHA c) 60%LHA

Figure 2 depicts the methodology of present work for the identification of optimal processing parameters. Figure 3 depicts the powders undergoing Calcination process with in a muffle furnace. Based on the number of XRD peaks corresponding to the identified phases at each temperature, the optimal temperature is determined. At the optimal temperature the samples namely 20% LHA,40 %LHA,60% LHA are held in a muffle furnace for further investigation as shown in Figure 3.

3. RESULTS and DISCUSSIONS

3.1 XRD analysis

Figure 4 depicts the XRD patterns of LHA powder prepared for 20-60 vol% LHA and calcinated at 750℃, 900℃ and 1000℃ for 2 hours. As the calcination temperature is raised from 750 to 1000℃, the peaks of the XRD are increased this shows LHA powder have higher degree of crystallinity of the nano particles. Higher temperatures generally produce sharper and more intense peaks, indicating better crystallinity. The XRD intensity and peak sharpness vary with increasing preparation temperature. After the XRD analysis, it revealed that over the all-selected ratios for 40 vol% and 60 vol% LHA samples having the greater number of matched peaks of $Al_{11.5}La_{0.85}O_{18.5}$ at 900℃. An increase in peak intensity indicates enhanced crystallinity of the material. As the calcination temperature rises, thermal energy facilitates the growth of well-ordered crystalline domains, reducing structural defects. Higher peak intensity also suggests the dominance of the desired phase, such as the hexa-aluminate phase in LHA. Sharper peaks correspond to larger crystallite sizes and reduced lattice strain. This indicates that the material transitions from a fine, nanocrystalline or strained structure to a more stable, well-formed crystalline state. The diminishing amorphous background with increasing calcination temperature suggests the conversion of disordered precursor phases into the crystalline hexa-aluminate phase [19]. A reduction in amorphous content also reflects complete decomposition of organic binders or volatile impurities. Changes in peak sharpness and intensity reflect microstructural evolution, such as grain growth, reduction in lattice distortions, and improved homogeneity. Monitoring these changes allows optimization of calcination parameters to achieve desired properties for high-temperature applications.

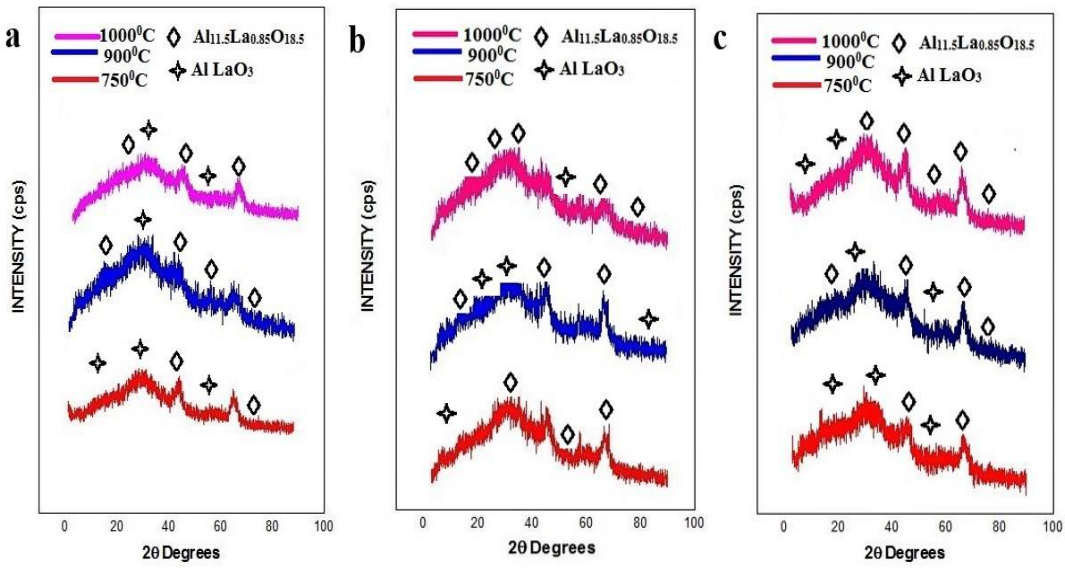


Figure 4. XRD patterns of Al2O3-LHA ceramic powder samples with volume fraction a.) 20% b.) 40% c.) 60% percentage calcinated at 750°C, 900°C and 1000°C

3.2 Hardness measurement

Proper preparation is essential to ensure that the mechanical properties accurately reflect the intrinsic characteristics of the material. One of the critical steps in this process is ensuring that the powder used for pellet preparation is completely free from moisture. This is achieved by pre-drying the powder at approximately 100℃ for a duration of 1–2 hours. The removal of moisture prevents inconsistencies in pellet formation and ensures uniform mechanical properties. For effective pellet preparation, several key factors must be considered. These include maintaining uniform pressure distribution during compaction, achieving the desired pellet thickness, properly preparing the pellet surface, and avoiding over-compaction, which may introduce internal stresses or defects in the sample. Once the pellets are formed, they are subjected to heat treatment at an optimal temperature. Specifically, samples containing different compositions—20% LHA, 40% LHA, and 60% LHA are placed inside a muffle furnace. The controlled heating process enhances the structural integrity of the pellets, and after the required time, the powders are carefully extracted following a cooling period. To assess the hardness of the prepared pellets, a Vickers hardness tester is employed. The testing is conducted on a polished surface to ensure accuracy and consistency in the measurements. A controlled load of 5–10 kg is applied for a dwell time of 2–4 seconds at multiple points across the sample surface. This approach ensures the reliability and repeatability of the hardness readings. The properly prepared powder pellets, intended for hardness measurement, are depicted in Figure 5.

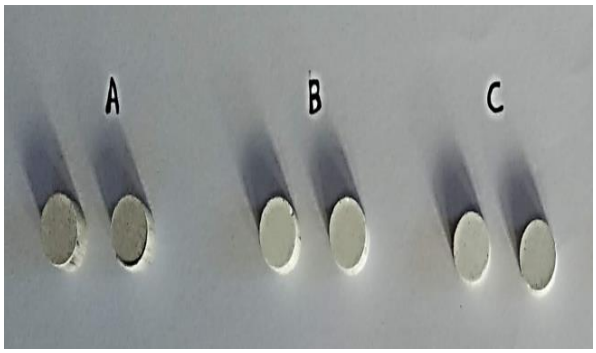


Figure 5. Prepared pellets for a.) 20% b.) 40% c.) 60%LHA percentage calcinated at 1000℃

For the hardness measurement of the prepared samples, the Vickers hardness test was conducted using a Vickers hardness tester (Model: HVS 1000B). This test was performed to evaluate the mechanical strength of the material by measuring its resistance to indentation. The test was carried out on a polished surface to ensure accuracy and consistency in the recorded values. A controlled load was applied, and the indentation size was measured to determine the hardness values. The results of the hardness test indicate that at 20% LHA, the hardness value was measured at 118 ± 6 HV. However, as the LHA content increased, the hardness values initially decreased before showing a sudden rise again at 60% LHA. This fluctuation in hardness can be attributed to variations in particle size and their effect on material densification. Smaller particles generally contribute to higher hardness due to an increased surface area, which promotes better compaction and densification during pellet formation. The higher packing efficiency of fine particles results in fewer voids, leading to improved mechanical strength. However, when the particle size becomes excessively small, there is a tendency for particles to agglomerate. Agglomeration hinders uniform compaction and creates localized weak spots, which can reduce the overall hardness of the material [20, 21]. This explains the initial decrease in hardness observed as the LHA content increased beyond 20%. On the other hand, at 60% LHA, an increase in hardness was noted, which may be attributed to improved crystallinity. At higher temperatures, the material undergoes structural reorganization, enhancing crystallinity and leading to a more ordered atomic arrangement. This increase in structural order strengthens the material, thereby increasing its hardness. In summary, the variation in hardness values observed in the LHA samples is influenced by a combination of particle size effects, compaction behaviour, agglomeration tendencies, and crystallinity improvements. These factors collectively determine the mechanical performance of the material under different LHA compositions. Summary of hardness measurement for various compositions are presented in Table 1

Table 1. Summary of hardness measurement for various compositions

Composition	Temperature	Hardness (HV) [nm]
20 vol% LHA	1000℃	118±6 HV
40 vol% LHA	1000℃	109±3 HV
60 vol% LHA	1000℃	114±3 HV

3.3 FESEM/EDX analysis

FESEM analysis is a critical technique for characterizing the microstructure, surface morphology, and particle size of materials like LHA.

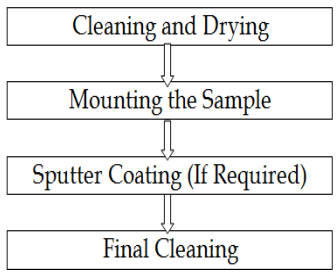


Figure 6. Sample preparation for FESEM technique

Table 2. Summary of average grain size (D) for various compositions

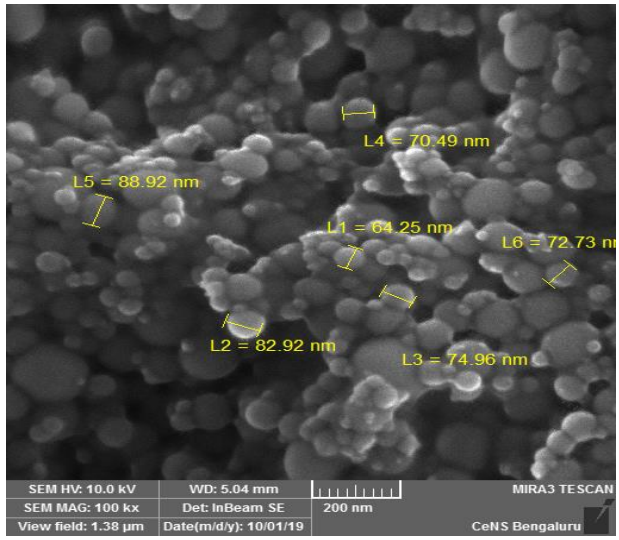
Composition	Temperature	Average Grain Size [nm]
20 vol% LHA	900℃	75.71
40 vol% LHA	1000℃	85.64
60 vol% LHA	1000℃	111.425

Figure 6 depicts the sample preparation for FESEM technique. LHA powder samples were examined using (Model: TESCAN-MIRA 3 LMH) FESEM images are captured at a magnification of 500x, at an accelerating voltage of 50 V to 30 KV in steps of 10 V coupled with (Model: QUANTAX 200 with XFlash BRUKER) Energy Dispersive X-ray spectrometer (EDX/EDS).

From The structural and morphological characterization of LHA powders with different volume fractions (20 vol%, 40 vol%, and 60 vol%) was performed using XRD and FESEM.

The XRD analysis confirmed the phase composition of the synthesized powders, while FESEM analysis was conducted to evaluate the microstructural features, particularly the average grain size of the LHA samples after calcination at 1000℃. Figures 7-9 present the FESEM micrographs and energy-dispersive EDX spectra of the 20 vol% LHA ($Al_{11.5}La_{0.85}O_{18.5}$) powder sample calcined at 1000℃. From the FESEM micrographs, it is evident that the LHA particles exhibit significant sintering behavior at this temperature, leading to grain growth and increased densification. The observed grain coalescence suggests that at 1000℃, the material undergoes partial densification, where grain boundary diffusion and particle necking contribute to the overall microstructure formation. The mean grain size of the 20 vol% LHA sample at 1000℃ was determined from the micrographs and is summarized in Table 2. The EDX analysis of the 20 vol% LHA powder sample provides insight into its elemental composition, revealing that aluminum (Al) exhibits the highest weight percentage, while lanthanum (La) is present in a relatively lower proportion, alongside oxygen (O). This result aligns with the expected stoichiometry of the synthesized $Al_{11.5}La_{0.85}O_{18.5}$ phase, confirming the accuracy of the synthesis process and elemental distribution within the sample. Similarly, the FESEM micrographs of the 40 vol% and 60 vol% LHA powder samples calcined at 1000°C show a trend of increasing grain size with LHA content. As the LHA concentration increases, the microstructure reveals a higher degree of particle agglomeration, which is a consequence of enhanced sintering at elevated temperatures. This is indicative of increased grain boundary mobility, which facilitates the coalescence of fine particles, thereby contributing to larger grain formations. The EDX studies of the 40 vol% LHA powder sample confirm a high weight percentage of aluminum (Al), accompanied by a significant oxygen (O) content, while lanthanum (La) remains in a lower proportion. This suggests a uniform elemental distribution and the successful formation of the LHA phase in the sample. Likewise, the 60 vol% LHA powder sample exhibits a similar elemental trend, with aluminum as the predominant element, followed by oxygen, and a comparatively lower concentration of lanthanum. This indicates that even at higher LHA concentrations, the synthesis method maintains consistent elemental composition and phase purity. The summary of the mean grain sizes obtained from the FESEM analysis for different LHA compositions is presented in Table 2. The observed microstructural trends suggest that an increase in LHA content leads to enhanced particle sintering and grain coarsening, which can influence the mechanical and thermal properties of the material. These findings provide valuable insights into the phase stability, grain morphology, and elemental composition of LHA-based materials, which are crucial for optimizing their performance in high-temperature applications such as thermal barrier coatings, ceramics, and refractory materials. With the gradual rise in calcination temperatures, the effect of calcination temperatures on the mean grain size of the powders increases steadily [22]. The grains grow gently at a low calcination temperature, while grains grow faster at a higher temperature and form agglomerations. Grain Growth caused at Higher temperatures provide the energy needed for grains to grow through Ostwald ripening, where larger grains grow at the expense of smaller ones. Grain growth reduces the number of individual particles, leading to agglomeration of the remaining larger grains [23].

Agglomeration of LHA particles at higher calcination temperatures is driven by sintering, grain growth, surface energy reduction, and phase transformations. While it may improve mechanical stability for some applications, it can also hinder properties like surface area and homogeneity. Proper process optimization is essential to balance these effects based on the material's intended use.



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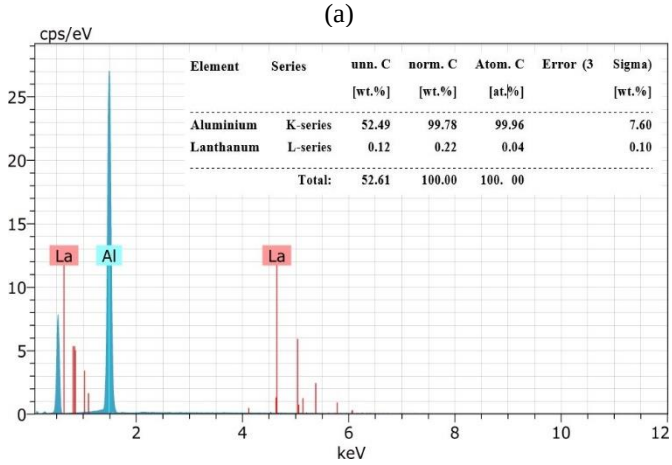
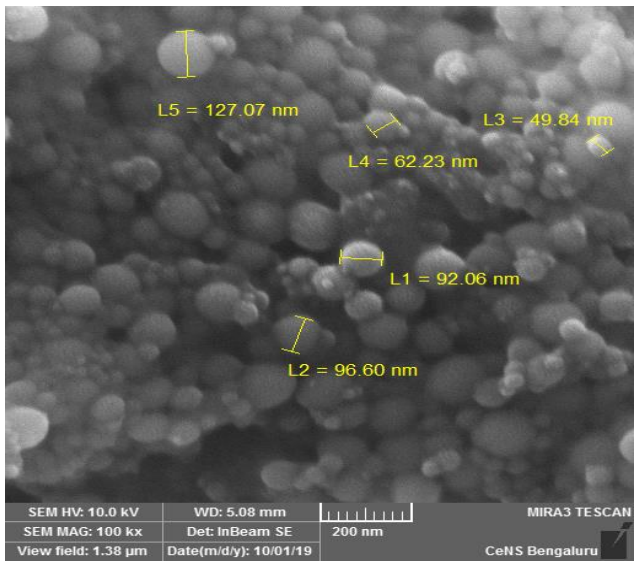


(b)

Figure 7. (a) FESEM Grain size measurement and EDX report for 20 vol% LHA sample calcinated at 1000℃, (b) EDX report for 20 vol% LHA sample calcinated at 1000℃



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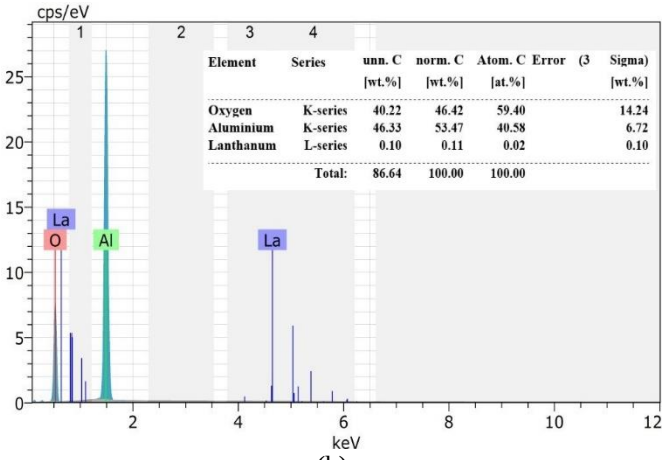
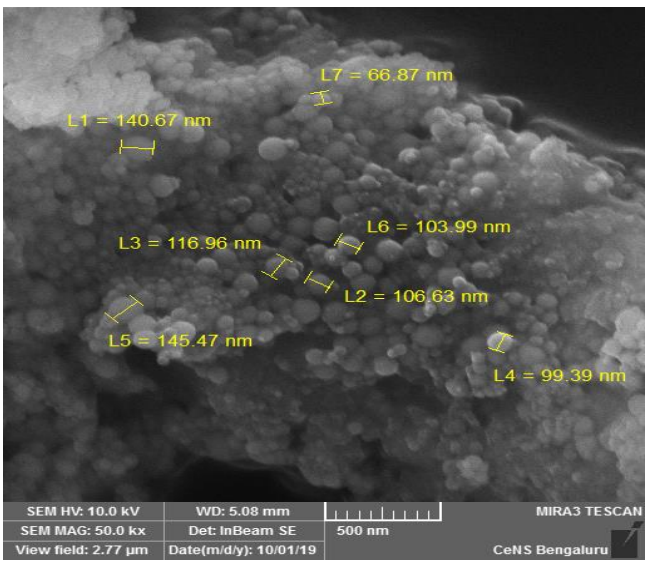


(b)

Figure 8. (a) FESEM Grain size measurement and EDX report for 40 vol% LHA sample calcinated at 1000℃, (b) EDX report for 40 vol% LHA sample calcinated at 1000℃



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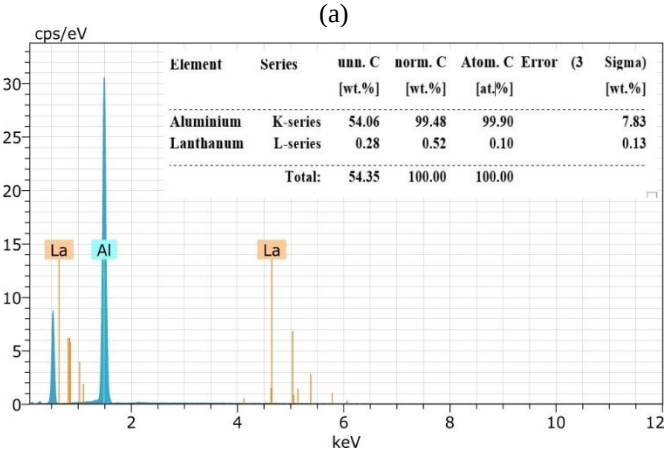

(b)

Figure 9. (a) FESEM Grain size measurement and EDX report for 60vol% LHA sample calcinated at 1000℃, (b) FESEM Grain size measurement and EDX report for 60vol% LHA sample calcinated at 1000℃

4. CONCLUSIONS

In the current study, three distinct compositions of Alumina/Lanthanum hexa-aluminate (Al_2O_3/LHA) powder were synthesized, with LHA content varying from 20 to 60 volume percent. The synthesis process was carried out using the chemical precipitation method, followed by filtration, to ensure uniform particle distribution and high purity of the prepared powders. Chemical precipitation was chosen due to its ability to produce fine, homogeneously mixed particles with controlled stoichiometry, which is crucial for achieving the desired phase composition and enhancing material performance. To thoroughly analyze the structural and morphological characteristics of the synthesized powders, a comprehensive characterization was performed. Phase identification and crystallinity of the powders were examined using powder XRD, which provided insights into phase formation, phase purity, and crystallite size. Additionally, FESEM was utilized to investigate the surface morphology and particle size distribution of the synthesized powders, offering a detailed understanding of their microstructural features. To complement the morphological analysis, energy dispersive EDX was conducted to confirm the elemental composition and ensure the successful incorporation of lanthanum into the alumina matrix. These characterization techniques collectively provided a deeper understanding of the phase evolution, microstructure, and chemical homogeneity of the synthesized Al_2O_3/LHA powders, which is essential for their potential application in high-temperature and wear-resistant environments.

• After preparation of Alumina-Lanthanum Hex-aluminate ceramic powders these were subjected to calcination temperatures at 750°C, 900°C and 1000°C for 2 hours.

• Out of all 20 vol% LHA composition at 1000℃ is opted as best one as the number of peaks obtained are more and it is conformed using EDX analysis also.

• It is observed that at 20% LHA hardness value is 118+6 HV but the values are decreased and then again increased at 60% LHA this sudden increase or decrease is due to the smaller particles typically lead to higher hardness due to increased surface area and higher compaction density.

• The mean grain size of 20% LHA at 1000℃ is 85 nm.it is observed that as the temperature is high due to formation of agglomerations the grain size is more at 60% LHA.

Furthermore, LHA is an emerging material with significant potential for high-temperature applications, making it a promising candidate for various advanced engineering solutions. One of its key applications lies in thermal barrier coatings, where its superior thermal stability and insulating properties can effectively protect underlying components from extreme heat, thereby extending their lifespan and performance in harsh environments. Additionally, the powders synthesized through this study can serve as reinforcement agents when combined with other base materials. By incorporating these LHA-based powders into composites, it is possible to enhance critical tribo-mechanical properties, such as wear resistance, friction reduction, and overall mechanical strength. This makes LHA a valuable addition to industries requiring durable, heat-resistant materials, including aerospace, automotive, and energy sectors, where both high performance and cost efficiency are paramount.

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