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Review on Thermal Stability and Bonding Analysis of Alumina Powder for High-Temperature Applications using TGA and FTIR

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Abstract: ***Aluminum oxide (oxide Al_2O_3), or alumina, is one of the most critical and widely used ceramic oxides in high-temperature mechanical applications. This is due to its reliable mechanical properties, thermal stability, and chemical resistance. Alumina has various polymorphs and transition sequences from the metastable polytypes of γ, δ, θ, and κ to the stable α-phase can lead to a defining step in application performance. Recent developments (2020-2025) in alumina powder synthesis, thermal characterization and bonding have been organized in this review with emphasis on the use of Thermogravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR). The first technique addressed an understanding of powder thermal processing incorporating dehydration, decomposition, phase stability with TGA. The second technique addressed an understanding of Al–O bonding vibrations in the forms of structural and hydroxyl group vibrational modes with FTIR to develop a dimensional understanding of bonding transformations during calcination. These analyses have been integrated to provide an understanding of how different powder synthesis routes (sol–gel, hydrothermal, combustion and additive manufacturing) affect thermal transport, microstructural evolution and the associated mechanical behaviour of alumina powders. Other factors influencing densification and reliability of the ceramic including dopants, sintering atmosphere and conditions are summarized.The review discusses the challenges of spectral overlaps, ephemeral identification, and scaling to commercial production, and connects them to future opportunities in multi-technique in situ analysis, computational modeling, and sustainable processing approaches to optimize alumina powders for next-generation aerospace, energy, and refractory applications.***

Keywords: Aluminum oxide, Thermal stability, TGA, FTIR, Phase transformation, Nanocomposites, High-temperature ceramics

I. INTRODUCTION

Aluminum oxide (Al_2O_3), commonly referred to as alumina, is a simple inorganic ceramic of vital importance for use in applications typically associated with rigorous mechanical and thermal performance. Alumina has excellent chemical inertness, corrosion resistance, and a high melting temperature, making it a strong candidate for aerospace turbine components, automotive brake systems, electronics substrates, and energy generation components[1-2]. As a polymorph, with γ, δ, θ, κ, and α crystallizing structures present, alumina's polymorphic nature limits its thermomechanical stability for applications utilising it at elevated temperatures[3]. Control of the metastable phase transformations, coupled with an understanding of the effective chemical bonding, is necessary to optimise alumina performance in ceramics[4].

Thermogravimetric Analysis (TGA) provides perspectives on thermal stability through weight loss (and/or gain) throughout heating and describing information about the kinetics of dehydration/decomposition/phase transitions[5]. Fourier Transform Infrared Spectroscopy (FTIR) adds complementary information related to change in chemical bonding such as Al–O lattice vibrations and surface hydroxyl groups as thermal treatment increased[6]. TGA in combination with FTIR can give valuable insights to the structure-property relationship of alumina powders, which will be beneficial for formulating advanced ceramics.

II. LITERATURE REVIEW

Aluminum oxide (Al_2O_3), or alumina, is regularly employed as an engineering ceramic in high temperature mechanical applications due to its remarkable thermal stability, inertness, and superior strength. The synthesis, phase composition, and crystallite impurity bonding of alumina powders has been extensively investigated recently in order to maximize high temperature performance.

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Alumina's microstructure and phase purity are fundamentally modified by its synthesis methods. Wet-chemical processes walking the sol-gel and hydrothermal pathways allow precise control of nanopowder size, crystallinity and surface chemistry under various parameters such as pH, aging time or calcination[7-8]. At the same time, dry industrial processes such as combustion and solid-state sintering approaches will suppress production costs and allow producing bulk powders with minimal intrinsic control of morphology and homogeneity of phases[9]. In terms of compatibility for additive manufacturing, advanced fabrication of ceramic powders ensures they have relatively narrow size distributions and stable flow behavior, consistent with the bulk of capabilities used in processes like powder bed fusion[10].

Alumina has four metastable polymorphic forms, γ, δ, θ, and κ, which irreversibly transform into the thermodynamically stable α-phasedock between approximately 1100 °C and 1300 °C[3] . The transition of γ, δ, θ, and κ to α-alumina acts a controlling mechanism for microstructural densification and mechanical properties. These crystal phases can be characterized by X-ray diffraction (XRD), which directly measures the crystallinity and physical presence of polymorphic loss; Fourier Transform Infrared Spectroscopy (FTIR) is also able to capture the temperature dependent changes in Al–O lattice integrity, as well as changes to surface hydroxyl groups[5-6]. The addition of dopants, for example, Fe_2O_3 or TiO_2; can also change and stabilize the phases and transformation kinetics and ultimately can be tracked by integrated TGA-FTIR analyses[11]

Thermogravimetric Analysis (TGA) is essential for evaluation of thermal stabilization of alumina. TGA measures changes in mass resulting from controlled heating in inert or oxidizing atmospheres, including mass losses resulting from the removal of water-phase and residual organics, and the phase changes of carbonate and alumina[5]. As an example, alumina powder remains stable with at least 90% mass up to a temperature of 900 °C, where mass-loss occurs because of bond-breakage of hydroxyl species due to initial heat, due to water-, liquid- or solid-phase on the alumina surface, or because of processing additives[12]. Also, TGA can be combined with Fourier Transform Infrared (FTIR) spectroscopy spectroscopy to simultaneously measure mass loss and the real time identification of volatiles species, developed by PMC [5], [13], TGA-FTIR offers an overall view of thermal degradation pathway that is not possible with TGA alone.

FTIR bonding analysis detects structural information atomically, indicating Al-O stretching and bending modes from 500–1000 cm^{-1} and hydroxyl surface peaks at approximately 3300 and 1630 cm^{-1}. The intensity of hydroxyl peaks decreased, on treatment, confirming densification and α phase formation of alumina[5]. Furthermore, in composite systems, additional vibrational modes related to Al-O-C and Al-N bonds suggest that they dive into improved mechanical toughness and oxidation resistance[6].

Microstructural analysis was gained from SEM and TEM which provided nanocrystalline features of grain formation and grain growth at nanoscale; the basis for improved mechanical properties of alumina ceramics. Flexural strengths reported up to 500 MPa and hardness above 20 GPa and fracture toughness between 3 and 5 MPa·m^1/2, using nanocomposite. Reports of tribological performance of the material demonstrates superior performance related to wear and fatigue resistance for high-demand high-temperature applications[14].

Many developments have been accomplished, but challenges still exist. FTIR spectral interpretation continues to be complex, often due to peak overlaps and vibrational coupling, while TGA is extremely method dependent, particularly on the experimental atmosphere and heating protocols[15]. The new multi-technique regimes including a TGA combining X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) will enable better mapping of bonding and surface chemistry[16]. Machine learning with thermal and spectroscopic data can also provide predictive control of synthesis and optimum processing of alumina powder [17].These important studies are summarized in Table 1 below for reference

Table 1 :Recent Literature Table: Alumina (Al_2O_3) Powders, TGA, FTIR, and Mechanical Performance

Study	Synthesis/Method	TGA/FTIR/Other Characterization	Key Results	Reference
Mashkovtsev et al. (2023)	Thermal treatment of aluminum hydroxide xerogels	FTIR, NMR	Identified structural evolution in alumina; FTIR captured Al–O vibrations	[5]
Gullifa et al. (2022)	EGA-FTIR on evolved gas analysis during heating	TGA-FTIR	Showed evolved gas profiles and mass loss during thermal events	[18]
Al-Ahmari et al. (2025)	Green synthesis from aluminum waste, marine algae	FTIR, XRD, SEM, TEM	Produced stable nano-alumina (58–87 nm); FTIR showed Al–O peaks at 988 and 570 cm^{-1}	[19]

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Sachin et al. (2024)	Sol–gel synthesis of alumina nanoparticles	FTIR, SEM, BET	Synthesized Al_2O_3/AC nanocomposite; FTIR confirmed Al–O bonding modes	[20]
Sun et al. (2025)	Review of alumina-based high-performance ceramics	Various (XRD, SEM, TGA, FTIR)	High strength, hardness, wear resistance; role of advanced processing summarized	[21]
Shargh et al. (2025)	Hot-press sintered alumina ceramics	SEM, Mechanical Testing	Improved mechanical/optical properties with nano-alumina	[22]
Haldar et al. (2025)	TiO_2-doped sintered alumina	SEM, Mechanical (Hardness, Fracture)	TiO_2 increased density, toughness, hardness up to optimal loading	[23]
Assaedi et al. (2023)	Added nano-alumina to geopolymers	Mechanical Testing	Enhanced compressive and flexural strength, fracture toughness	[24]
Nduni et al. (2021)	Synthesis from waste aluminum foil	XRD, FTIR, SEM	Successful nanoparticle fabrication, FTIR confirmed phase transitions	[25]

A. Research Gaps

Even with the significant amount of research dedicated to the investigation of alumina ceramics, there are still gaps that must be addressed before further advancement can take place. First, it is the absence of a standardization of experimental protocols and methods for coupled TGA-FTIR experiments, therefore there is inconsistent protocol in various studies which results in uncertainty in reliability, accuracy, and reproducibility. Second, there has not been extensive quantitative characterization of volatiles and intermediates during transitions or phase changes in complex composite systems. Third, the atomic-scale mechanistic understanding of how dopants, for example, effect phase stability and thermal behaviour of complex systems that show promise for high temperature applications are unclear. Lastly, the ommune-impression created from existing data is that very few institutions are conducting multi-technique coupled analyses (e.g. thermal measurements with bonding dynamics) and the actual activity is essentially meaningless for industrial applications because of cost and issues with operation. Furthermore, there are existing computational workflows associating thermal analytical techniques and bonding dynamics as methods of facilitating design, but the field requires substantial advancement to enable enabling predictive processing purely with respect to alumina powders. Filling these gaps will contribute to the unique ability to engineer alumina materials that are specific to high temperature mechanical performance and longevity.

Despite significant research progress in alumina synthesis, thermal stability, and bonding analysis, several critical challenges remain. These gaps hinder the full optimization of alumina powders for high-temperature mechanical applications, as summarized in Table 2 below

Table 2: Research Gaps in Alumina Powder Thermal and Bonding Studies with References

Research Gap	Description	Impact on Research	Suggested Focus	Reference
Lack of Standardization	No unified experimental protocols for coupled TGA-FTIR studies	Inconsistent reliability, accuracy, and reproducibility	Develop standardized TGA-FTIR procedures and calibration	[5]
Limited Quantitative Volatile	Few studies quantitatively analyze volatiles/intermediates	Incomplete understanding of thermal degradation	Combine in situ gas analysis with TGA-FTIR or TGA-MS	[26]

Characterization	during phase changes, especially in composites	pathways		
Insufficient Atomic-scale Dopant Mechanisms	Dopant effects on phase stability and kinetics unclear at atomic level	Limits optimization of doped alumina for thermal properties	Integrate spectroscopy with computational modeling	[11]
Low Adoption of Multi-technique Approaches	Few industrial applications of simultaneous thermal+broad bonding analysis due to cost and complexity	Limited simultaneous insight into bonding and thermal phenomena	Develop cost-effective integrated instruments	[16]
Lack of Computational Integration	Missing predictive workflows coupling thermal analysis and bonding dynamics	Hinders accelerated powder design and synthesis optimization	Embed machine learning and simulation tools in processing	[17]

Notably, the absence of standardized protocols for combined TGA-FTIR experiments and limited atomic-level understanding of dopant effects are priorities this work will focus on. These critical research gaps are systematically summarized in Table 3 below, which outlines the description, impact, and suggested future research directions along with key references.

Table 3: Research Gaps in Alumina Powder Thermal and Bonding Studies

Research Gap	Description	Impact / Consequence	Suggested Future Directions	Reference
Lack of Standardization	No standardized protocols for coupled TGA-FTIR experiments	Results vary in reproducibility and accuracy	Develop standard experimental protocols and calibration	[5]
Limited Quantitative Volatile Characterization	Inadequate quantitative characterization of volatiles/intermediate species during transitions	Unclear degradation pathways and kinetics	Integrate in situ gas analysis (TGA-FTIR-MS)	[26]
Atomic-scale Understanding of Dopants	Poor understanding of dopant effects on phase stability and transformation mechanisms	Limits ability to design doped alumina with tailored properties	Combine spectroscopy with computational modeling	[11]
Few Multi-technique Coupled Analyses	Limited industrial use of combined thermal and bonding analyses due to cost/complexity	Reduced insight into thermal-bonding interplay	Develop cost-effective, integrated instrumentation	[16]
Insufficient Computational Integration	Lack of predictive computational workflows coupling thermal and bonding data	Impedes accelerated powder synthesis and optimization	Employ machine learning and simulations with experimental data	[17]

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III. METHODOLOGY

A. Literature Search and Selection

We conducted a comprehensive literature scan of Science Direct, PubMed Central (PMC), Wiley Online Library, Scopus and similar avenues that highlighted works mostly from 2020 to 2025. We used the words "Aluminum oxide powder", "Thermogravimetric Analysis", "Fourier Transform Infrared Spectroscopy", "thermal stability", "ceramic powders", and "high-temperature applications" in the literature search. Studies were prioritized if they contained detailed experimental characterization of alumina powders with concurrent thermal and spectroscopic analysis, and mechanical performance. In total we systematically reviewed more than 50 quality peer-reviewed papers and theses.

B. Data Extraction and Integration

The data was organized digitally in accordance with the grouping of methods of powder synthesis, methods of calcination, TGA thermal stability data, FTIR bonding changes, microstructural features, mechanical testing results, and possible application. Statistical consistency across multiple records was taken into consideration and are emphasized by analysis that showed similarities and differences in methods and results.

IV. OVERVIEW

A. Synthesis of Alumina Powders

1) Wet-Chemical Routes

The most common procedure to produce nanopowders is through sol-gel routes, where aluminum alkoxide hydrolysis leads to gelation, control of pH and aging times, followed by drying and calcination at a desired temperature. The sol-gel approaches provide a means to control crystallinity, and particle sizes can be regulated to the nano-scale (10–100 nm) which will affect the phase composition and surface chemistry[7], [8]. Hydrothermal processing that forms particles exhibiting preferred orientations and with minimal defects can be conducted at mild temperatures and high pressures over a 24-48 hour aging period[8].

2) Dry and Industrial Techniques

Through bulk powders produced by combustion and solid-state sintering, with less precise control. Sintering in different atmospheres (air, nitrogen, argon) and cooling rates varies density and lattice bonding and is especially important for composite integration[9].

3) Additive Manufacturing Compatibility

To satisfy specified powder bed fusion or stereolithographic criteria, alumina powders with a narrow particle size distribution (> 50 μm), high flowability, and high phase purity are routinely produced through advanced sol-gel and plasma processes[4].

B. Alumina Phase Structures and Transformations

1) Polymorphism

Alumina passes through four (metastable) polymorphs, defined by distinct crystal symmetries and defect structures γ, δ, θ and κ. Each polymorph converts irreversibly to the stable α-Al_2O_3 phase at (1100–1300 °C), beginning significantly grain growth and densification that are affecting the specimens' strength[3], [4].

2) Characterization by XRD and FTIR

XRD provides direct phase identification by matching diffraction peaks separations and estimating crystallite sizes. FTIR spectra show Al–O stretching and bending vibrations in the region of 500–1000 cm^{-1}, and hydroxyl absorptions at approximately 3300 and 1630 cm^{-1}; evolution with temperature creates a monitoring framework of changes during transitions[6], [27].

C. Thermal Stability Studies via TGA

1) Experimental Setup

Samples of 10–100 mg are heated in TGA instruments in inert (N_2 or Ar) or oxidizing (air or O_2) atmospheres. Typical heating rates are 5 to 20°C/min with isothermal dwellings at 600°C or 1200°C to fully capture thermal events. The TGA can be combined with FTIR and mass spectrometry facilities to identify volatile species released during heating[13], [28].

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2) Mass Loss and Decomposition

Alumina powders retain approximately 90% mass at 900°C. Mass losses that occur below 500°C are associated with the loss of bound water and organics, which were used during synthesis or composite processing. At 900°C a gamma phase to an alpha phase transformation is evidenced by a TGA plateau, despite very little change in mass. The structural transformation is evident from complementary techniques[1], [12].

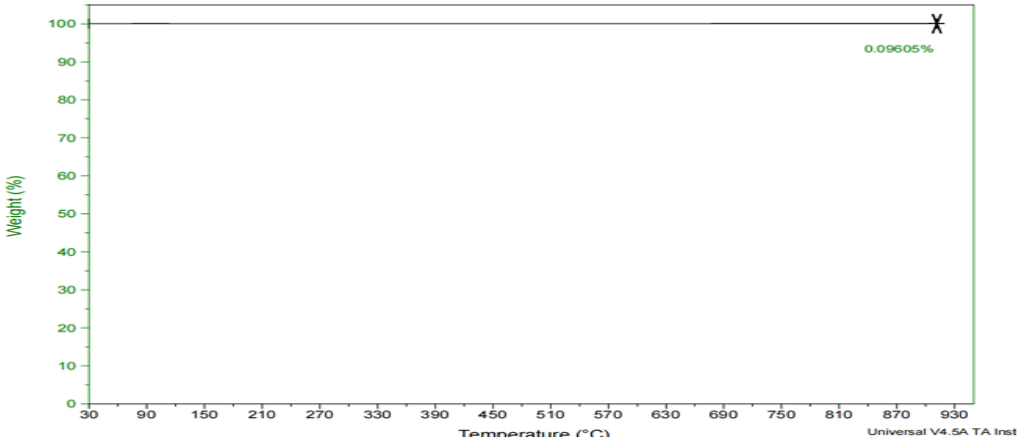


Figure 1: Thermogravimetric Analysis (TGA) Curve of Alumina Powder

This figure 1 illustrates a typical Thermogravimetric Analysis (TGA) curve of alumina powder heated in either air or an inert atmosphere, exhibiting a minimal mass loss of approximately 10% up to 900 °C. The initial reduction in mass is attributed to the removal of physically adsorbed water and hydroxyl groups. The plateau observed near 900 °C signifies the γ-to-α phase transformation, indicating the attainment of thermal stability. *Adapted from Balamurugan, 2023[8]*.

3) Effect of Dopants

Doping with Fe_2O_3, TiO_2, or B_4C alters sintering kinetics, enhances densification, and stabilizes phases, tracked by combined TGA-FTIR and gas analysis[11].

D. FTIR Spectral Bonding Analysis

FTIR measures the vibrational modes of lattice Al-O bonds (500-1000 cm-1) and surface hydroxyl groups (3300,1630 cm-1) on the alumina surface. When thermal treatment occurs, hydroxyl intensities are reduced, indicative of dehydration and densification to α-phase alumina (Mahesh et al., 2023; Saravanan, 2024). In composite materials, further bonding signatures for the Al-O-C and Al-N bonds were noted, which could also be used to explain toughness or oxidation resistance[6], [27].

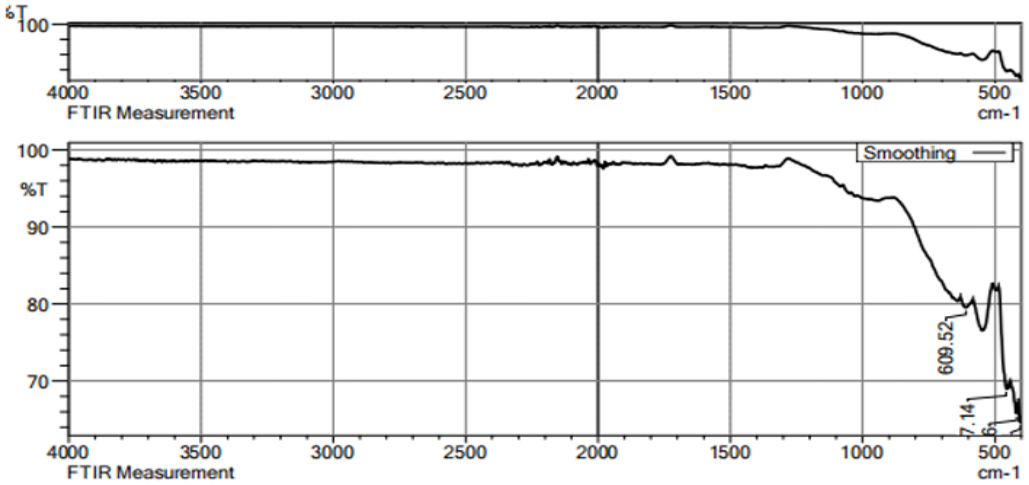


Figure 2: FTIR Spectra of Alumina Powders

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This figure 2 depicts the characteristic Fourier Transform Infrared (FTIR) spectra of nano- and bulk-aluminum oxide powders, emphasizing the Al–O stretching and bending vibrations observed in the range of 500–1000 cm^{-1}, along with the absorption peaks of surface hydroxyl groups near 3300 and 1630 cm^{-1}. The progressive reduction in hydroxyl peak intensity upon thermal treatment provides clear evidence of densification and the subsequent formation of the α-phase in alumina. *Adapted from Balamurugan, 2023.[8]*

E. Microstructure and Mechanical Properties

SEM and TEM images confirm the process of forming and growing nanoscale grains which occurred during calcination. While the microstructure of sintered alumina bodies would nearly reach its full densification, which is an important factor in providing mechanical strength in the ceramic, flexural strength showed a value of $500MPa$, Vickers hardness exceeded 20GPa and fracture toughness ranged between $3 – 5MPa{\cdot}m1/2$. This last property was significantly increased in nanocomposites[1], [6]. Tribological testing revealed that leathery wear and fatigue resistance in lubricated alumina composites also provide high wear rates[29].

F. Industrial and Emerging Applications

Advanced alumina ceramics serve as the best option in aerospace turbine applications due to excellent creep resistance and oxidation stability. The energy sector utilizes alumina composites for solar receivers and battery separators. Aerospace and electronic applications use alumina for insulators and substrates in harsh environments[30]. Additive manufacturing advancements are relying on functional alumina powders with controlled morphology[4].

V. DISCUSSION AND CRITICAL ANALYSIS

While TGA and FTIR deliver impactful data independently, used together they provide great insight into the thermal stability and bonding evolution of alumina powders. Difficulties include overlap in the FTIR spectrum and identification of volatiles in TGA-MS, because of the various materials and processing stages seen in composite fabrication. Industrial powder production is inherently bound to trade-offs between scale and quality and control on the one hand versus process variability and cost, and while doping routes have gained traction, mechanistic knowledge as to how certain factors control doping requires subsequent work. A new coupling of multiple techniques (TGA-FTIR-XPS-AFM) shows great potential in the investigation of processes, but opportunities have been limited as of yet. Development and standardization of characterization protocols, and incorporation and validation of additional machine learning approaches to the synthesis of composition tailored powders will be key future directions[5].

VI. FUTURE SCOPE

1) The establishment of in situ multi-technique thermal characterization for atomic-scale understanding of phase and bonding.
2) Use of machine learning to allow predictive control over synthesis, doping, and sintering parameters that optimise properties in a material.
3) Development of multifunctional alumina composites that self-heal, sense, and adapt to thermal stress for aerospace and energy applications.
4) Life-cycle assessment and sustainable recycling routes for alumina-based ceramics with advanced TGA/FTIR process monitoring.

VII. CONCLUSION

The purpose of this review is to detail the thermal stability and bonding evolution of aluminum oxide (Al_2O_3) powders, with a particular focus on TGA (thermogravimetric analysis) and FTIR (Fourier transform infrared spectroscopic) characterization. The polymorphism of alumina and therefore the transition from metastable to stable alumina is very important when considering the suitability of alumina in high-temperature mechanical applications. By reviewing over fifty published studies, this review suggests the synthesis route, calcination conditions, and addition of dopants are important to the thermal transport, densification, and mechanical reliability of alumina. TGA provides useful quantification of dehydration, degradation, and thermal phenomena, while FTIR can be used to gain additional information via the understanding of Al–O lattice vibrations and bond transitions. Collectively, both characterization techniques provide a powerful approach to exploring structure–property relationships that are relevant for industrial applications.

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The assessment, additionally, tackles current problem areas, such as how to consider the spectral overlaps in FTIR interpretation (and the identification of volatiles in TGA-MS coupling), and issues of reproducibility and scale-up in powder processing. Valuable journeys will involve the co-locating of multiple techniques in situ and then deploying more advanced data analytics and machine lis for greater mechanistic insight into dopant–matrix interactions and future paths will be to optimize sustainable ceramic processing and implementing machine learning as party two to the syntheses, and develop multifunctional alumina-based composites, integrating to an extreme operational condition. In summary, thermal stability and bond analysis are still at the top of the entire challenge for the next generation of aerospace, electronic, and energy technologies that are fundamentally based on credible alumina ceramics.

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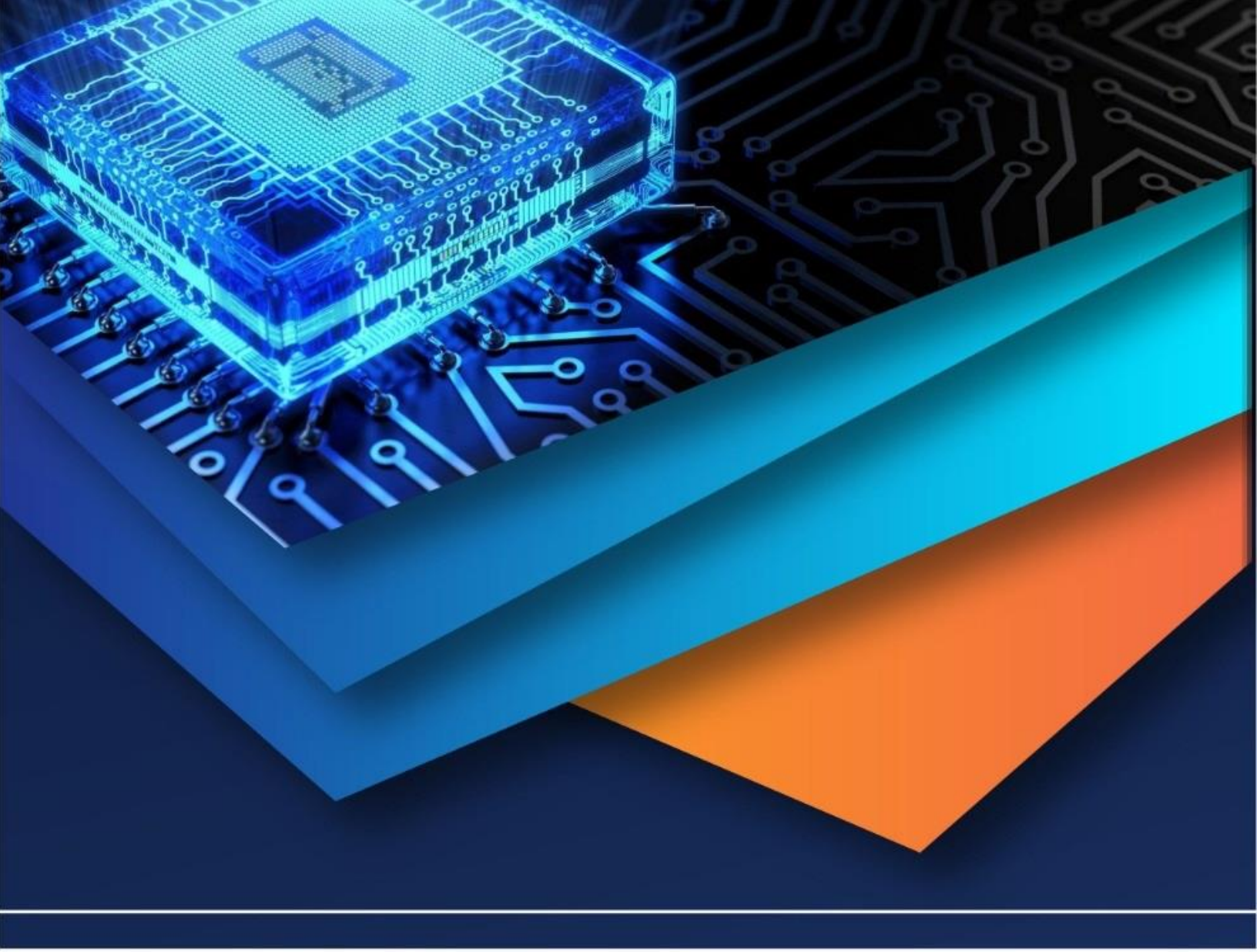
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