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MICROSTRUCTURE OF DENSE ZIRCON SAND - ALUMINA REFRACTORY BRICK FROM BAKIN RUWA ZIRCON SAND USING LINE BLENDING METHOD FOR FOUNDRY APPLICATION

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ABSTRACT

Microstructural analysis of dense Zircon Sand-Alumna refractory brick from Bakin ruwa, Toro Local Government Area, Bauchi state, Nigeria using line blend method was conducted with scanning electron microscopy (SEM). A small specimen (typically 10–20 mm in size) is cut from the bulk brick using a diamond saw. Sampling locations should include: Surface region, core region and any visibly heterogeneous zones. Care must be taken to avoid introducing thermal or mechanical damage during cutting, as this may alter the microstructure. *The results revealed that the sample of the produced bricks ZK1-ZK9 contain different* microstructure observation such as Crystalline Structure, Grain Size Distribution, Porosity and Voids, Intergranular Bonding, Microcracking and Mineral Phase Distribution *which is in consistent with various ASTM standards and* Performance Implications that makes it suitable for high temperature industrial foundry application*s. This shows that bricks from Bakin ruwa zircon sand is suitable for foundry application.* It is recommended that both prospective entrepreneurs and government agencies invest in the development of indigenous refractory production. This initiative would serve as a sustainable local source for the refractory needs of foundry furnaces, conserve foreign exchange, generate employment for young people, and contribute significantly to national economic growth.

Keywords: Alumino-silicate, refractory, Binder, zircon sand, foundry, furnace, microcracking, intergranular,

1.0 INTRODUCTION

Refractory materials play a crucial role in enhancing the operational efficiency and thermal integrity of the foundry industry. These materials are defined as heat-resistant substances capable of withstanding extremely high temperatures without undergoing degradation. They are essential in the construction of furnaces, ladles, crucibles, kilns, and other high-temperature equipment commonly employed in metal casting and heat treatment processes. The primary functions of refractories include providing thermal insulation, resisting chemical attack from slags and molten metals, and maintaining structural stability under severe thermal and mechanical stress *(Silva et al., 2020).*

Refractories are generally characterised by their ability to endure temperatures typically exceeding 1,580 °C while being exposed to the physical and chemical effects of molten metal, slag, and furnace gases (Olade, 2019). Silica (SiO_2) and alumina (Al_2O_3) are the principal constituents of Alumino-silicate minerals, which form the basis of many refractory products. The performance of a refractory material in high-temperature applications largely depends on the properties of the raw materials from which it is produced. Improved property combinations can be achieved by blending two or more distinct materials. These materials exhibit both physical and chemical resistance to elevated temperatures. High-quality refractories are also characterised by their resistance to thermal fluctuations and their excellent insulating properties both thermal and electrical (Arnold, 2022).

Zircon sand is the major precursor to ZrO_2, one of the most refractory minerals known. It is composed of 59–63 percent zirconium oxide (ZrO_2), 32–38 percent silica (SiO_2), and occasionally about 1% hafnium, it has good abrasion, impact and thermal shock resistance. It is stable in chemical conditions and is non-magnetic as well as non-conductive. (Sari *et al.*, 2021).

Zircon has been recognized as a potential structural material because of its good combination of physical and mechanical properties, such as excellent chemical stability, thermal shock resistance, strength retention to high temperatures, low thermal expansion as well as low heat conductivity Owing to high demand of zircon (Routray *et al.*, 2021). The exploitation of different deposits which can be a potential source for this mineral is a priority at present. Hence, it has attracted a wide attention of, users to exploit zircons for end use, zircon is a proper material for foundry due to its several good properties such as high heat conductivity as a result, it has a faster cooling process compared to quartz-based foundry, not reactive to other metals, clean-surface, spherical form, this character is appropriate for all binder type, less binder material, stable grain size and neutral pH. (Silva *et al.*, 2020).

The Nigerian foundry industry, which is integral to the manufacturing and metallurgical sectors, continues to face significant challenges due to the limited availability, high cost, and substandard performance of refractory bricks used in high-temperature applications (Sullyman, 2018).

Refractory bricks are critical for lining furnaces, ladles, and other thermal equipment, and their quality directly impacts energy efficiency, production costs, and operational safety. However, Nigeria largely depends on imported refractory products due to the underdevelopment of local refractory raw material processing and manufacturing capabilities (Ndanusa, 2005).

Despite the country's substantial natural reserves of refractory-grade minerals such as kaolin, bauxite, and zircon sand, these resources remain grossly underutilized due to inadequate beneficiation technologies, poor funding for research and development, and weak industrial linkages (Nigerian geological survey agency NGSA (20013).

2.0 MATERIALS AND METHODS

2.1 Principle of Microstructural Determination Using SEM

Scanning Electron Microscopy (SEM) is a high-resolution imaging technique in which a focused beam of electrons scans the surface of a specimen to generate detailed images of its morphology and composition. It is widely employed in the study of ceramic and refractory materials due to its ability to resolve microstructural features at micrometer to nanometer scale.

In refractory bricks, SEM enables the identification of grain size and morphology, distribution of crystalline and glassy phases, porosity and pore connectivity, microcracks and bonding characteristics. However, it is important to note that SEM provides primarily two-dimensional surface information, which may not fully represent the three-dimensional internal structure.

2.2 Sampling from Refractory Brick

2.2.1 Procedure

A small specimen (typically 10–20 mm in size) is cut from the bulk brick using a diamond saw. Sampling locations should include: Surface region, core region and any visibly heterogeneous zones. Care must be taken to avoid introducing thermal or mechanical damage during cutting, as this may alter the microstructure.

2.2.2 Sample Preparation

Sample preparation is the most critical stage, as improper handling can distort the microstructure. SEM requires specimens that are stable under vacuum and electrically conductive.

2.2.3 Drying and Stabilisation

Refractory materials often contain residual moisture or volatile phases. The sample was: oven-dried or air-dried, alternatively freeze-dried to preserve pore structure. Drying methods significantly influence particle orientation and microstructural integrity, especially in clay-based systems.

2.2.4 Mounting

The specimen was mounted on an aluminium stub using conductive adhesive (e.g., carbon tape). For friable samples, embedding in resin may be required before polishing.

2.2.5 Grinding and Polishing

To obtain a flat and smooth surface: Sequential grinding using silicon carbide papers (coarse to fine grit) Polishing with diamond paste (down to ~1 µm or finer). This step exposes internal microstructural features such as grain boundaries and pores.

2.2.6 Conductive Coating

Refractory bricks are typically non-conductive ceramics; hence: A thin conductive coating (gold, platinum, or carbon) was applied via sputtering. This prevents charge accumulation under the electron beam, which would otherwise distort imaging.

2.3 SEM Examination

2.3.1 Instrument Setup

Vacuum chamber was evacuated and accelerating voltage (typically 5–20 kV) is selected, working distance and magnification are adjusted

Imaging Modes

Secondary Electron (SE) imaging

Reveals surface topography

Backscattered Electron (BSE) imaging

provides compositional contrast (heavier elements appear brighter)

2.3.2 Magnification Range

Microstructural examination was conducted at multiple scales, for example, low magnification (≈100×–500×): overall texture and pore distribution, medium magnification (≈1000×–5000×): grain arrangement, high magnification (>5000×): intergranular bonding and microcracks.

2/3.3Microstructural Analysis

The SEM images are interpreted to determine key characteristics:

2.3.4 Grain Morphology and Phase Distribution

Identification of crystalline phases (e.g., mullite, silica, alumina) and observation of glassy phases formed during firing

2.3.5 Porosity

Size, shape, and distribution of pores, degree of densification. Porosity decreases and particle bonding improves with increasing firing temperature, leading to enhanced mechanical properties.

2.3.6 Intergranular Bonding

Degree of sintering between particles and Presence of neck formation between grains

2.3.7 Defects

Microcracks and inclusions or impurities

3.0 RESULTS AND DISCUSSION

The table below shows microstructure result of dense refractory bricks of 90% zircon and 10% Kaolin (ZK1) using scanning electron microscopy.

Table 2: Microstructure result of ZK1

Microstructure observation	**O**bservation from SEM	**A**STM Standard	**P**erformance implications
Grain Bonding and Density	Dense grain structure with tightly packed crystals.	**ASTM C27 (Classification)**	Ensures high mechanical strength and low porosity. Essential for load-bearing and wear resistance.
Needle-Like Crystals	Prominent needle-shaped structures indicating strong bonding.	**ASTM C133 (Modulus of Rupture Test)**	Contributes to improved flexural strength and resistance to mechanical stress.
Porosity and Voids	Visible gaps in some regions, suggesting controlled porosity.	**ASTM C20 (Apparent Porosity)**	Porosity levels should be below **20%** for dense refractory bricks to maintain durability and heat resistance.
Glassy Phase Presence	Small regions of vitrified (glassy) material observed	**ASTM C621 (Slag Resistance Test)**	Limited glassy phases are acceptable but excessive amounts may reduce thermal shock resistance.
Micro-Cracks and Debris	Cracks and fragmented particles are present.	**ASTM C704 (Abrasion Resistance Test)**	Excessive micro-cracking can reduce resistance to thermal shock and mechanical impact.

The dense grain structure aligns with ASTM C133 standards for modulus of rupture, supporting improved load-bearing performance. The tightly packed grains improve heat conduction aligning with ASTMC201guidelines.

The moderate porosity ensures better insulation properties, reducing heat loss. The vitrified phase improves chemical resistance but may require control to prevent excessive brittleness.

The table below shows microstructure result of dense refractory bricks of 80% zircon and 20% Kaolin (ZK2) using scanning electron microscopy.

Table 3: Microstructure result of ZK 2

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Grain Structure	The structure shows dense, closely packed grains with a rough texture.	**ASTM C27 (Classification)**	This indicates strong mechanical strength and low porosity, key for heat resistance and durability.
Intergranular Bonding	Clear bonding between grains with minimal voids is observed.	**ASTM C133 (Modulus of Rupture Test)**	Ensures structural integrity under mechanical stress. Strong bonding reduces crack propagation.
Porosity and Voids	Some micro-voids are visible but appear minimal.	**ASTM C20 (Apparent Porosity Test)**	Controlled porosity aligns with ASTM standards, ensuring better thermal insulation and strength.
Crystalline Phases	Crystals with a fibrous and elongated texture are present.	**ASTM C71 (Crystalline Phase Analysis)**	The presence of such phases enhances thermal shock resistance and improves chemical stability.
Glassy Regions	Limited areas of vitrified (glassy) material.	**ASTM C621 (Slag Resistance Test)**	A controlled amount of vitrification can improve chemical resistance, though excessive glassy phase may reduce mechanical strength.

The fibrous crystalline phase structure enhances resistance to rapid temperature changes. Dense packing and solid bonding suggest good compressive strength as required by ASTM C133**.** Limited vitrification aligns with ASTM guidelines, maintaining strong resistance to aggressive slag attack.

International Journal of Global Affairs, Research and Development (IJGARD), Vol.4, No.1, 2026, 528-542, ISSN 2992-2488

The table below shows microstructure result of dense refractory bricks of 70% zircon and 30% Kaolin (ZK3) using scanning electron microscopy.

Table 4: Microstructure result of ZK3

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Grain Morphology	The SEM image shows irregular-shaped particles with a mix of angular and rounded grains. Some grains display smooth surfaces while others appear porous.	**ASTM C92 (Fineness of Refractory Materials)**	Varied grain morphology enhances mechanical strength, but excessive porosity can reduce durability.
Porosity and Voids	Significant void spaces are visible between particles. The presence of both interconnected and isolated pores is evident.	**ASTM C20 (Apparent Porosity and Bulk Density)**	High porosity may reduce mechanical strength but can improve thermal insulation properties.
Crystalline Phases	The presence of distinct crystalline structures, possibly mullite or corundum phases, suggests effective firing.	**ASTM C71 (Refractory Terminology)**	Crystalline formations enhance thermal stability and strength under high temperatures.
Particle Bonding	Particles appear loosely bonded with minimal glassy phase presence.	**ASTM C133 (Modulus of Rupture Testing)**	Weak bonding may reduce mechanical strength and require optimization of firing conditions.
Microcracking	Some microcracks are visible, likely formed during cooling or due to thermal stresses.	**ASTM C832 (Thermal Shock Resistance Test)**	Excessive microcracking can reduce thermal shock resistance. Optimized cooling cycles may improve performance.

The presence of substantial porosity indicates potential suitability for insulating applications.
also the visible crystals suggest good high-temperature stability, and the loose particle bonding suggests potential for mechanical failure under stress conditions. The observed microcracks may indicate reduced resistance to rapid temperature changes.

The SEM analysis indicates that while the refractory bricks exhibit good crystalline structure development and thermal insulation potential, improvements are necessary to enhance bonding strength and reduce microcracking. Aligning the manufacturing process more closely with ASTM standards such **as** ASTM C20**,** ASTM C133**,** and ASTM C832 would improve overall performance.

The table below shows microstructure result of dense refractory bricks of 60% zircon and 40% Kaolin (ZK4) using scanning electron microscopy.

Table 5: Microstructure result of ZK4

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Crystalline Structure	The image reveals a blend of sharp-edged crystalline formations and amorphous phases.	**ASTM C71 (Standard Terminology Relating to Refractories)**	Well-developed crystals suggest controlled firing and effective phase development.
Grain Size Distribution	The presence of varied grain sizes is evident, with some large, blocky grains mixed with finer particles.	**ASTM C92 (Fineness of Refractory Materials)**	A balanced grain distribution enhances thermal shock resistance and mechanical strength.
Porosity and Voids	The structure shows notable porosity, with interconnected voids and fine cracks.	**ASTM C20 (Apparent Porosity and Bulk Density)**	Increased porosity may reduce strength but can improve thermal insulation. Controlled porosity is essential to balance these factors.
Intergranular Bonding	Particles appear tightly bonded with minimal detachment zones.	**ASTM C133 (Modulus of Rupture Testing)**	Strong bonding improves mechanical strength and resistance to stress under load.
Microcracking	Some microcracks are visible but appear minimal.	**ASTM C832 (Thermal Shock Resistance Test)**	Limited microcracking enhances resistance to rapid temperature fluctuations.

The presence of crystalline structures with minimal microcracks indicates resilience under thermal cycling. The observed voids are consistent with materials designed for thermal insulation applications, which ensures improved load-bearing capacity and structural stability.

The SEM analysis suggests that the fired dense refractory bricks demonstrate a well-formed crystalline structure, controlled porosity, and limited microcracking characteristics that align well with ASTM standards for refractory materials. Enhancing porosity control and refining firing conditions could further improve performance.

The table below shows microstructure result of dense refractory bricks of 50% zircon and 50% Kaolin (ZK5) using scanning electron microscopy.

Table 6: Microstructure result of ZK5

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Crystal Formation	Large, well-defined crystalline structures are present, indicating effective mineral phase development.	**ASTM C71 (Crystalline Phase Analysis)**	Strong crystalline phases improve mechanical strength, slag resistance, and stability at elevated temperatures.
Intergranular Phases	Distinct gaps and intergranular boundaries are visible.	**ASTM C133 (Modulus of Rupture Test)**	Clear phase boundaries help manage thermal expansion, reducing stress build up.
Porosity and Voids	Some voids are apparent between crystalline grains, but they appear controlled.	**ASTM C20 (Apparent Porosity Test)**	Controlled porosity ensures thermal insulation without compromising mechanical strength.
Microcracking	Limited evidence of microcracking suggests stable thermal expansion characteristics.	**ASTM C832 (Thermal Shock Resistance Test)**	Few microcracks indicate the brick's ability to withstand rapid temperature changes.
Grain Shape and Size	Presence of large, blocky grains with smooth surfaces.	**ASTM C113 (Refractory Testing for Spalling Resistance)**	Larger grains reduce overall surface energy, improving resistance to spalling under thermal stress.

The presence of large, well-formed crystalline phases aligns with ASTM standards for high-temperature applications. The visible voids appear minimal, promoting better insulation while maintaining strength. this ensures the material maintains structural integrity under repeated thermal cycling and strong intergranular bonding contributes to improved flexural and compressive strength

International Journal of Global Affairs, Research and Development (IJGARD), Vol.4, No.1, 2026, 528-542, ISSN 2992-2488

The table below shows microstructure result of dense refractory bricks of 40% zircon and 60% Kaolin (ZK6) using scanning electron microscopy.

Table *7*: Microstructure result of ZK6

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Grain Structure	The microstructure reveals large, dense grains with rough surfaces. The grains appear tightly packed, contributing to overall mechanical strength.	**ASTM C92 (Fineness of Refractory Materials)**	Dense grains enhance mechanical strength and thermal stability.
Porosity and Pore Distribution	Some isolated pores are present, but overall porosity appears controlled. The structure indicates a relatively low apparent porosity.	**ASTM C20 (Apparent Porosity and Bulk Density)**	Lower porosity improves mechanical strength, resistance to slag penetration, and thermal conductivity.
Intergranular Phases	There is evidence of intergranular bonding with minimal visible cracks at grain boundaries.	**ASTM C133 (Modulus of Rupture Testing)**	Strong intergranular bonding improves flexural strength and resistance to thermal cycling.
Microcracks and Defects	Minimal microcracks are observed, likely due to optimized firing conditions or controlled cooling rates.	**ASTM C832 (Thermal Shock Resistance Test)**	Reduced microcracking enhances thermal shock resistance and long-term durability.
Inclusion of Impurities/Secondary Phases	Some distinct white deposits suggest possible secondary mineral phases or impurities.	**ASTM C704 (Abrasion Resistance of Refractory Materials)**	Secondary phases may influence abrasion resistance and thermal performance, depending on composition.

The observed structure suggests strong mechanical performance and durability. Indicates good thermal shock resistance and controlled firing conditions. The visible white inclusions may impact chemical stability, especially in acidic or alkaline environments.

The SEM analysis indicates that the refractory bricks exhibit a well-controlled microstructure with dense grains, minimal porosity, and reduced microcracking. This aligns well with ASTM standards such as **ASTM C20, ASTM C133, C704.** However, addressing the presence of impurities may further improve the material's performance.

The table below shows microstructure result of dense refractory bricks of 30% zircon and 70% Kaolin (ZK7) using scanning electron microscopy.

Table 8: Microstructure result of ZK7

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Crystalline Phase Formation	The image reveals distinct crystalline structures with sharp, well-defined edges. Such formations are typical in refractory bricks that have undergone sufficient sintering and vitrification.	**ASTM C71 (Standard Terminology for Refractories)**	Strong crystalline phases improve resistance to high temperatures and chemical corrosion.
Glass Phase and Matrix	The presence of a glassy matrix indicates partial melting, which is common in dense bricks designed for high-temperature resistance.	**ASTM C830 (Standard Test Methods for Refractory Brick Porosity and Density)**	The glass phase enhances the material's ability to withstand thermal shock and resist slag infiltration.
Porosity and Pore Structure	Fine interconnected porosity is observed, suggesting well-controlled firing conditions with minimized excessive pore growth.	**ASTM C20 (Apparent Porosity and Bulk Density)**	Controlled porosity optimizes thermal insulation properties without compromising mechanical strength.
Microcracks and Defects	Limited evidence of microcracks indicates good thermal stability and crack resistance.	**ASTM C704 (Abrasion Resistance of Refractory Materials)**	Fewer microcracks improve resistance to spalling and mechanical wear.
Mineral Phase Distribution	The SEM reveals distinct mineral phases that appear segregated yet stable.	**ASTM C133 (Modulus of Rupture Testing)**	Stable mineral phases ensure improved structural strength and thermal endurance.

The results above Indicate improved mechanical strength and resistance to thermal deformation.
Helps reduce porosity while enhancing resistance to chemical attack. Sign of effective sintering and firing protocols.

The SEM results indicate a well-developed microstructure with desirable characteristics like controlled porosity, crystalline phase growth, and minimal microcracking. These align with ASTM standards such as **ASTM C20, ASTM C830**, and **ASTM C704,** confirming the brick's suitability for high-temperature and abrasive environments which is in consistence with the work (Okoli et al.,2023).

The table below shows microstructure result of dense refractory bricks of 20% zircon and 80% Kaolin (ZK8) using scanning electron microscopy.

Table 9: Microstructure result of ZK8

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Porosity Distribution	Highly interconnected pores are evident, indicating incomplete densification.	**ASTM C830 (Porosity and Density Test)**	High porosity can compromise thermal resistance and mechanical strength.
Crystal Growth (Needle-like Structure)	Distinct needle-like crystal formations are visible, indicating certain phases formed during sintering.	**ASTM C133 (Modulus of Rupture Test)**	Needle-like crystals improve mechanical integrity but may become brittle under stress.
Inter-granular Bonding	Loose particle interfaces suggest weak bonding, which may reduce strength.	**ASTM C1161 (Flexural Strength Test)**	Weak bonding leads to lower mechanical resilience.
Crack Formation	Visible micro-cracks are observed near porous zones.	**ASTM C704 (Abrasion Resistance Test)**	Cracks indicate potential weak points under thermal shock or abrasion.

This SEM image suggests that while some beneficial crystal growth has occurred, the high porosity, crack presence, and weak particle bonding indicate insufficient firing or material preparation. Following ASTM standard testing and refining the firing process will improve the brick's overall durability and performance.

The table below shows microstructure result of dense refractory bricks of 10% zircon and 90% Kaolin (ZK9) using scanning electron microscopy.

Table 10: Microstructure result of ZK9

Microstructure Feature	Observation from SEM	Relevant ASTM Standard	Performance Implications
Crystalline Structure	The image reveals a blend of sharp-edged crystalline formations and amorphous phases.	**ASTM C 71 (Standard Terminology Relating to Refractories)**	Well-developed crystals suggest controlled firing and effective phase development.
Grain Size Distribution	The presence of varied grain sizes is evident, with some large, blocky grains mixed with finer particles.	**ASTM C92 (Fineness of Refractory Materials)**	A balanced grain distribution enhances thermal shock resistance and mechanical strength.
Porosity and Voids	The structure shows notable porosity, with interconnected voids and fine cracks.	**ASTM C20 (Apparent Porosity and Bulk Density)**	Increased porosity may reduce strength but can improve thermal insulation. Controlled porosity is essential to balance these factors.
Intergranular Bonding	Particles appear tightly bonded with minimal detachment zones.	**ASTM C133 (Modulus of Rupture Testing)**	Strong bonding improves mechanical strength and resistance to stress under load.
Microcracking	Some microcracks are visible but appear minimal.	**ASTM C832 (Thermal Shock Resistance Test)**	Limited microcracking enhances resistance to rapid temperature fluctuations.

The SEM analysis suggests that the fired dense refractory bricks demonstrate a well-formed crystalline structure, controlled porosity, and limited microcracking characteristics that align well with ASTM standards for refractory materials. Enhancing porosity control and refining firing conditions would further improve performance.

The presence of crystalline structures with minimal microcracks indicates resilience under thermal cycling. The observed voids are consistent with materials designed for thermal insulation applications, which ensures improved load-bearing capacity and structural stability.

4.0 CONCLUSION AND RECOMMENDATIONS

4.1 Conclusion

Based on these findings above, it is concluded that the production process, which employed both pressing and line blending methods, yielded nine brick samples (ZK1–ZK9), all of which were subjected to rigorous microstructural analysis. The microstructural analysis reveals that all the nine samples of refractory bricks contain the key characteristics such as Crystalline

Structure, Grain Size Distribution, Porosity and Voids, Intergranular Bonding, Microcracking and Mineral Phase Distribution which align with different ASTM standard of refractory bricks. This provides various performance implications with relevant standards which qualified them to become refractory bricks

4.2 Recommendations.
In view of the above findings, the following recommendations were drawn.

i. Promotion of Indigenous Refractory Production
It is recommended that both prospective entrepreneurs and government agencies invest in the development of indigenous refractory production. economic growth.

ii. Optimization of Zircon-Kaolin Blending Ratios
Empirical evidence supports the use of 60:40, 50:50, and 40:60 zircon-to-kaolin blending ratios for the production of high-performance, durable refractory materials suitable for foundry applications.

iii. Utilization of Characterised Zircon Sand
The locally characterised zircon sand should be actively exploited as a raw material in the production of refractory bricks.

iv. Advanced Binder and Temperature Studies
Additional research should focus on evaluating the performance of alternative binders in various blending ratios under different sintering temperatures.

v. Establishment of SME Refractory Enterprises
Efforts should be directed towards establishing small and medium-scale refractory production firms, particularly in regions where raw material deposits have been identified.

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