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Characterization of new powders used in ecological technology of shell moulds fabrication

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ABSTRACT

This work summarizes the results of powders characterization in new ecological method of ceramic shell moulds fabrication for precision casting process of aircraft turbine blades using nickel superalloys. The article is first part of articles group describing whole method and complete characterization of: powders, binders, ceramic slurries, ceramic shell moulds and final summary. In the paper two types of powders were investigated: base powders and stuccos for prime coat and backup of the moulds. This method involves powders based on aluminium oxides, zirconium silicates and cobalt aluminate. To characterization of their properties SEM observations, grain size, X-ray diffraction, X-ray fluorescence and Zeta potential methods were widely used. Studies were proven that chosen powders exhibit suitable and optimal properties and met the assumed requirements for ceramic shell moulds fabrication.

Keywords: ceramic casting moulds, aircraft turbine, binders, nickel superalloys.

INTRODUCTION

The "dip and stucco" method represents one of the oldest and most enduring techniques in the manufacturing of molds, with its origins tracing back to ancient times when artisans crafted functional tools and decorative objects. Its longevity can be attributed to its inherent simplicity, versatility, and efficiency. Over the centuries, this method has undergone significant transformations, driven by advancements in material science and engineering. Today, it is a cornerstone of precision casting, particularly for high-performance applications such as turbine blades for aircraft engines and complex industrial machinery components, where dimensional precision and surface integrity are critical [1–3]. Originally, the "dip and stucco" method employed rudimentary tools and naturally sourced materials. Early craftspeople utilized primitive slurries and coarsely ground powders to create molds capable of producing detailed yet functional castings. These early innovations provided the foundation for modern precision casting technologies, which now incorporate highly engineered materials and processes [4]. The evolution of this method reflects a continuous effort to enhance accuracy, efficiency, and scalability, aligning it with the demanding requirements of modern industries. In contemporary applications, the method revolves around the use of wax patterns that replicate the geometry of the final product. These wax models, often integrated with complex gating and pouring systems, serve as a substrate for constructing multilayer ceramic molds. The chemical composition and mechanical properties of the wax are of paramount importance, ensuring dimensional stability, minimal contraction, and compatibility with ceramic slurries. These attributes enable the creation of intricate designs while minimizing the risk of deformation or chemical interactions during processing [5, 6]. The fabrication of ceramic shell molds through the "dip and stucco" method involves a highly controlled multi-step process. Initially, the wax model is immersed in a finely tuned ceramic slurry, which adheres uniformly to its surface. This is immediately followed by stuccoing, where the model is coated with ceramic powder. The layer is then subjected to drying and hardening to ensure its stability before subsequent layers are applied [7–9]. Depending on the required thermal and mechanical properties, the molds typically consist of five to eight layers. Each additional layer reinforces the structure, enhancing its durability under the high temperatures and pressures encountered during casting. After the layering process, the ceramic molds undergo a rigorous heat treatment regimen designed to prepare them for metal casting. This involves three distinct stages:

- Wax extraction, removed in an autoclave using superheated steam at 170 °C and 0.86 MPa; this step ensures the integrity of the ceramic shell while leaving a hollow cavity for casting.
- Pre-sintering, heated to a temperature range of 700–900 °C, depending on the ceramic composition, to eliminate residual wax and consolidate the ceramic structure.
- Final sintering, conducted at 1000–1500 °C, this phase solidifies the ceramic material and transforms the binder into a robust gel-like phase; the temperature is tailored to the specific application:
  - 1000 °C – suitable for conventional castings,
  - 1250 °C – used for thin-walled polycrystalline components such as low-pressure turbine blades,
  - 1500 °C – required for monocrystalline castings, including high-performance turbine blades.
- Post-sintering, the molds are filled with molten metal, allowed to cool, and subsequently broken to retrieve the final casting; additional processes, such as surface finishing and separation of gating systems, yield a near-net-shape component, reducing the need for extensive machining [10–13].

The adaptability and precision of the "dip and stucco" method make it indispensable for producing components from high-melting-point alloys, such as nickel-based superalloys. These materials are critical in aerospace and energy industries due to their ability to withstand extreme temperatures and mechanical stresses. The method's capability to produce intricate geometries with minimal material waste supports its extensive use in both mass and serial production [14, 15]. Modern research has enhanced this technique by addressing traditional challenges such as uneven layer deposition, sedimentation of slurries, and thermal stresses during sintering. Innovations in binder formulations, particularly nanoparticle-based systems, have significantly improved layer uniformity, reduced drying times, and enhanced structural integrity. These advancements ensure the consistent production of high-quality castings while maintaining cost-efficiency [16]. Historically, the reliance on alcohol-based binders in the "dip and stucco" method posed substantial health and environmental risks, including exposure to harmful VOC emissions. Regulatory frameworks, particularly in Europe, have necessitated the transition to more sustainable alternatives. Water-based colloidal silica binders now dominate the field, offering several advantages:

- Environmental compliance – drastically reduced VOC emissions align with stringent regulations.
- Worker safety – elimination of toxic fumes improves workplace conditions.
- Enhanced properties – superior thermal stability, mechanical strength, and gas permeability enhance casting quality [17].

Solutions like Keysol and Matrixsol exemplify how eco-friendly materials can achieve or surpass the performance of traditional systems while supporting industrial sustainability. The adoption of water-soluble binders is also driven by economic factors. Facilities transitioning from ethanol-based systems have reduced costs associated with air filtration and protective equipment. Furthermore, the development of advanced powders, including aluminum oxide, zirconium silicate, and cobalt aluminate, has tailored the process to meet the specific needs of modern casting. Analytical techniques such as SEM, XRD, and Zeta potential measurements validate these materials' efficacy, ensuring their suitability for high-precision applications [18].

As the "dip and stucco" method continues to evolve, research is expected to focus on:

- Advanced eco-friendly materials – development of binders and powders with lower environmental impact.

- Scalability – adaptation of the process for a broader range of industrial applications.
- Customization for advanced alloys – optimization of binder systems to accommodate emerging high-performance materials [19].

The “dip and stucco” method, rooted in historical practices, has been transformed through centuries of innovation to meet the demands of modern manufacturing. Its blend of tradition and technological advancement underscores its ongoing relevance in producing high-quality, complex castings. As industries increasingly prioritize sustainability and precision, the method’s adaptability ensures it remains a cornerstone of advanced manufacturing, well-suited to the challenges of a rapidly evolving industrial landscape [20]. According to studies by Rao et al. [21] and Frueh et al. [22], silica-based systems have been explored extensively for their inertness and thermal stability. However, earlier systems using colloidal silica suffered from issues like sedimentation and uneven layer deposition on complex geometries. The use of nanoparticles in the new binder system addresses these issues by ensuring uniformity and reducing the drying time, a critical advancement over previous generations of water-soluble binders. Studies by Liu et al. [23] and Du et al. [24] on ceramic mold materials emphasize the importance of controlling thermal expansion and conductivity to prevent stress-related defects. The new binder systems’ performance up to 1200 °C, as noted in the current research, aligns with these findings, confirming the importance of controlled sintering to reduce defects during casting. Other researchers, such as Ismael et al. [25], have highlighted the environmental advantages of using colloidal silica binders in comparison to ethanol-based systems. The results from this study corroborate these environmental benefits, specifically showing that the new water-soluble binders reduce volatile organic compounds (VOCs) and improve worker safety without sacrificing casting performance.

The main purpose of these research was to change old technology based on alcohol binders. New European requirements caused limitations in use alcohol binders for water soluble binders. For this reason, technology of ceramic shell moulds fabrication must be changed. The second purpose was to eliminate alcohol vapors from laboratory conditions. Employees exhibits basic symptoms of intoxication of organism. For financial reason the better way was to eliminate alcohol-based binders than investments in expensive and modern filtering devices and personal protection of employees.

MATERIAL AND EXPERIMENTAL

The research focused on the characterization of ceramic powders used in an ecological method for fabricating ceramic shell moulds intended for the precision casting of turbine blades made from nickel superalloys. The materials were divided into two categories: powders for prime coats and powders for backup layers.

Materials:

- Prime coat powders:
  - Aluminum oxide (Al_2O_3): grades 200# and 325#,
  - Zirconium silicate ($ZrSiO_4$),
  - Cobalt aluminate ($CoAl_2O_4$).
- Backup layer powders (stuccos):
  - Aluminum oxide 80#,
  - Molochite with three granulometries: 16/30, 30/80, and 120.

All powders were tested in dry form, and also prepared in aqueous suspensions for analyses that required dispersion.

Before analysis, powders were stored in a desiccator to avoid moisture absorption. For slurry-related tests, suspensions were prepared using deionized water. Each sample was ultrasonicated for 15 minutes to ensure homogenization. No dispersants were used, to allow for intrinsic electrokinetic behavior to be observed. Microstructures of powders was examined using Hitachi SU-70 scanning electron microscope, 5 kV, 10 kV and 20 kV of accelerating voltage in secondary electron detector. Samples were glued on carbon films. For SEM investigations, multiple images (typically three to five) were taken at various magnifications and different regions of each sample to ensure representativeness of the observed morphology. For each powder type, at least five images were taken at various magnifications (×1000 to ×10,000).

For particle size analysis using the Horiba LA 950 analyzer using the LALLS (low angle laser light scattering) method. Three parallel measurements were conducted for each powder type, with the average values and standard deviations reported. The use of aqueous dispersion ensured uniform suspension and minimized aggregation, which was verified between runs. Measurements were conducted in aqueous medium with three repetitions per sample. Similarly, zeta potential measurements were carried out three times per powder in a controlled aqueous environment, following sample sonication to achieve dispersion homogeneity. Only fine powders used in the prime coat were tested, as the equipment limitations prevented accurate analysis of coarser stucco materials. The tests were carried out in an aqueous environment. The reported average values are accompanied by standard deviations. Zeta potential was analyzed for the prime coat powders using a Zetasizer Nano ZS. Measurements were carried out in water, with pH adjusted from 2 to 10 using HCl and NaOH. Each powder was tested three times, and the isoelectric point (IEP) was determined from the ζ-potential vs. pH curve. Stucco powders (Molochite and Al_2O_3 80#) were excluded due to particle size limitations of the instrument.

Measurements of chemical and phase composition were obtained using a Bruker S4 Explorer WDXRF X-ray fluorescence spectrometer and a Bruker D8 Advance powder diffractometer. The Bruker S4 Explorer wavelength-dispersive XRF spectrometer was equipped with a Rh tube, copper anode and Cu, Pb, Al filters and for XRD Cu anode was used. Scan parameters: 2θ = 5°–80°, step size = 0.02°, scan speed = 1.2°/min. In the case of X-ray fluorescence (XRF) and X-ray diffraction (XRD) analyses, each sample was subjected to at least three scans to verify the consistency of phase and elemental composition. This was particularly important for confirming the purity of synthetic powders and detecting any trace contaminants. In the present study, all key measurements, including particle size distribution, zeta potential, phase composition (XRD), chemical composition (XRF), and morphological observations (SEM), were conducted with a minimum of three independent repetitions per sample. This approach was implemented to account for potential variability in measurement conditions, sample preparation, and instrument performance.

RESULTS

Prime coat of ceramic shell powders investigation

Results of microstructure analysis of powders used on prime coat of ceramic shell moulds are shown in Figures 1–4.

Analyzed powders exhibit a typical particle morphology used in the process of ceramic shell moulds fabrication. There are characterized by irregular shape, sharp edges and expanded surface. The obtained distribution of mean particle diameter of the powders used for prime coat are shown in Figure 5 and Table 1.

It was established that powders are characterized by the following features:

- Al_2O_3 (200 #) – the powder has a wide particle size range of 3.4 to 200 μm; the average particle diameter is 57.2 ± 2.10 μm; the resulting distribution is symmetrical and has one maximum – d = 58.95 μm.
- Al_2O_3 (325 #) – the powder has a wide range of particle size from 5.8 to 229 μm; the average particle diameter is 27.6 ± 1.02 μm; the resulting distribution is asymmetrical and has 2 maximums – d = 11μm and d = 34 μm, which may indicate the occurrence of agglomerates,
- Cobalt Blue ($CoAl_2O_3$) – powder has a narrow particle size range from 2.2 to 29 μm; the average particle diameter is 8.4 ± 0.40 μm; the resulting distribution is asymmetrical and has 2 maximums – d = 4.4 μm and d = 8.8 μm, which may indicate the occurrence of agglomerates,
- Zirconium silicate ($ZrSiO_4$) – the powder has a narrow range of its size from 2 μm to 55 μm; the average particle diameter is 20 ± 0.92 μm; the resulting distribution is asymmetrical and has 3 maxima – d = 3.3 μm, d = 13.3 μm and d = 39.2 μm, which may indicate the occurrence of agglomerates.

Zeta potential values of powders used for prime coat fabrication are shown in Figure 6 and Table 2.

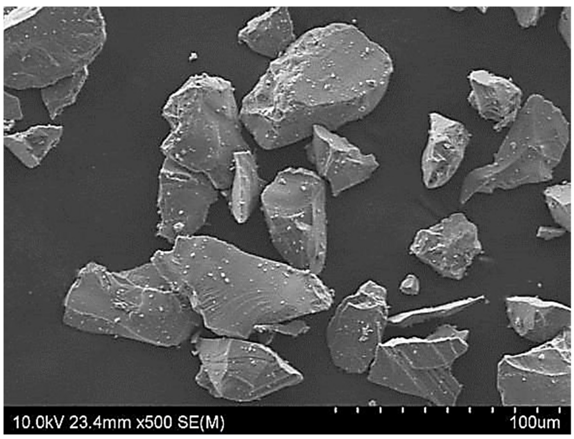

Figure 1. SEM microstructure of Al_2O_3 200#

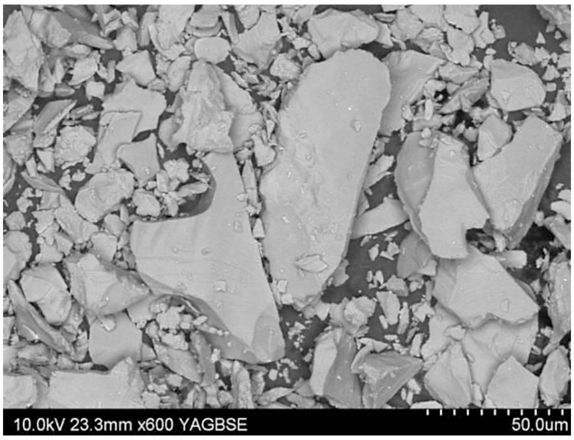

Figure 2. SEM microstructure of Al_2O_3 325#

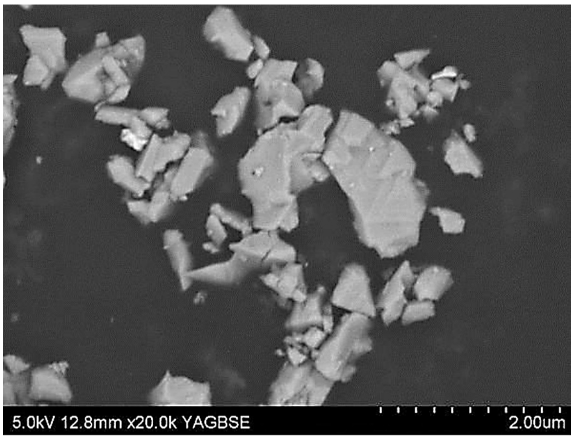

Figure 3. SEM microstructure of $CoAl_2O_4$

Large particle sizes made it impossible to carry out of powder used to manufacture of back-up layers. The obtained values of the isoelectric point are consistent with the literature data [26–29]. Analysis of the obtained results allows to conclude that:

- the isoelectric point for Al_2O_3 200 # powder is present at pH ≈ 2.8; for pH > 3.0 the zeta potential is negative, it reaches the maximum value: -32.5 mV.
- the isoelectric point for powder Al_2O_3 325 # occurs at pH ≈ 9.1; for pH > 9.5, the zeta potential is negative, it reaches the maximum value: -45.1 mV.
- the isoelectric point for $CoAl_2O_4$ powder is for pH ≈ 5.2; for pH > 5.2 the zeta potential is negative, it reaches the maximum value: -38.3 mV.
- the isoelectric point for $ZrSiO_4$ powder is for pH ≈ 3.1; for pH > 3.1 the zeta potential is negative, it reaches the maximum value: -51 mV.

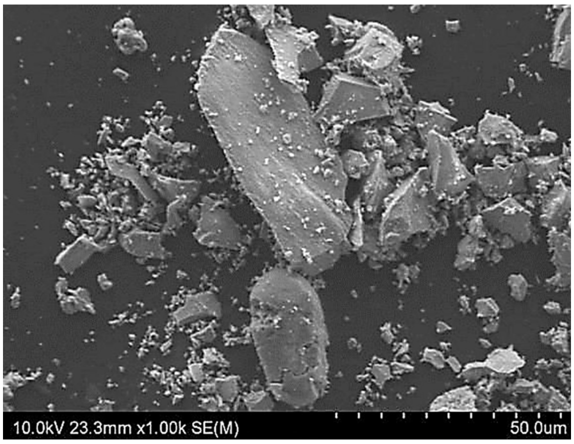

Figure 4. SEM microstructure of $ZrSiO_4$

Zeta potential is a critical parameter in evaluating the electrostatic stability of ceramic suspensions, particularly in water-based systems used for the fabrication of precision shell molds. It reflects the degree of electrostatic repulsion or attraction between particles in a suspension and significantly influences the homogeneity, rheological behavior, and long-term stability of the slurry. In this study, zeta potential measurements were performed for the fine powders used in the prime coat – namely Al_2O_3 200#, Al_2O_3 325#, $CoAl_2O_4$, and $ZrSiO_4$ – due to equipment limitations with coarser particles. The results revealed distinct isoelectric points (IEPs) for each powder, indicating differences in their surface charge behavior depending on the pH of the suspension. Al_2O_3 200# showed an IEP at pH ≈ 2.8, while Al_2O_3 325# exhibited a significantly higher IEP at pH ≈ 9.1. This indicates that Al_2O_3 325# will have a strong negative surface charge in mildly alkaline environments, promoting good dispersion and reducing the likelihood of agglomeration. Similarly, $CoAl_2O_4$ presented an IEP at pH ≈ 5.2, while $ZrSiO_4$ had an IEP near pH 3.1, suggesting lower stability in acidic conditions. These findings are highly relevant to the practical processing of ceramic shell moulds. High absolute values of zeta potential (typically $|\zeta| > 30$ mV) are indicative of stable suspensions, as strong electrostatic repulsion prevents particle aggregation. In the context of investment casting, such dispersion stability is crucial during the "dip and stucco" coating process, as it ensures uniform layer formation, reduces the risk of slurry sedimentation, and minimizes the formation of surface defects after drying and sintering. As noted by Ferenc et al. [2] and Zhao et al. [7],

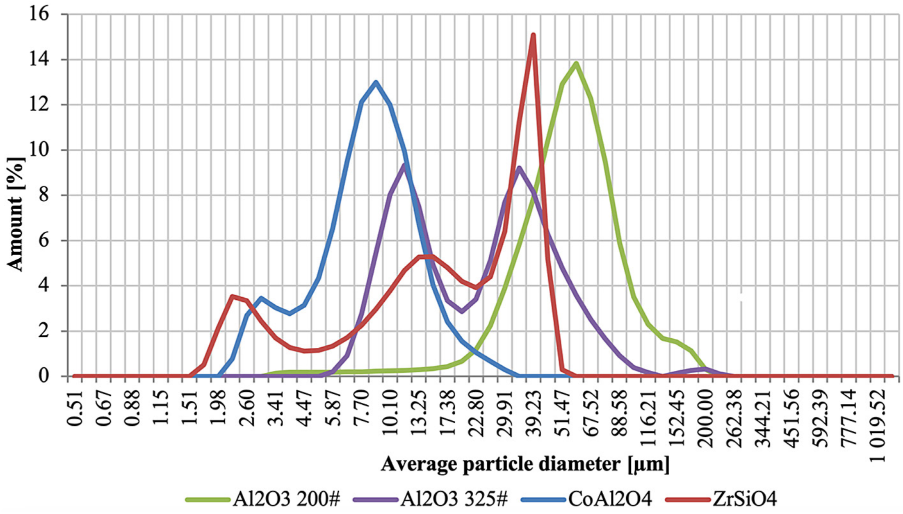

Figure 5. Distribution of the average particle diameter of powders used for prime coats – Al_2O_3 200#, Al_2O_3 325#, $CoAl_2O_4$, $ZrSiO_4$

Table 1. Comparison of the average particle diameter of the tested powders

Powder	Test environment	Average particle diameter [µm]
Al_2O_3 200#	WATER	57.20 ± 2.10
Al_2O_3 325#	WATER	27.60 ± 1.02
$CoAl_2O_4$	WATER	8.40 ± 0.40
$ZrSiO_4$	WATER	20.00 ± 0.92

optimizing zeta potential through pH adjustment or surface modification of powders directly improves slurry stability and casting surface quality [2, 7]. Moreover, a well-dispersed ceramic slurry facilitates better infiltration around complex wax patterns, leading to more accurate reproduction of intricate geometries. The relationship between zeta potential and casting quality has been well established in literature. For instance, Huang and Lin [34] demonstrated that ceramic suspensions with a controlled zeta potential yielded coatings with fewer microcracks and better resistance to thermal shock during firing [34]. Similarly, Richards and Bowen [18] emphasized the importance of optimizing electrokinetic stability to achieve consistent mold strength and dimensional accuracy [18]. In this study, the powder Al_2O_3 325# emerged as the most electrostatically stable in alkaline conditions, making it particularly suitable for use in modern, eco-friendly colloidal silica binders, which typically exhibit pH values in the 9–10 range. This compatibility reduces the need for additional dispersants or stabilizers, simplifying the formulation and reducing production costs. On the other hand, $ZrSiO_4$'s lower zeta potential and narrow stability range may contribute

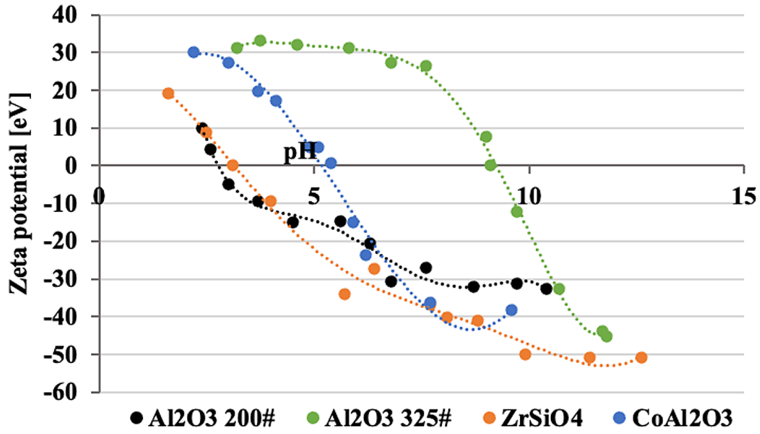

Figure 6. Distribution of Zeta potential of ceramic powders – Al_2O_3 200#, Al_2O_3 325#, $ZrSiO_4$, $CoAl_2O_4$

Table 2. Comparison of the isoelectric points of the tested powders – Al_2O_3 200#, Al_2O_3 325#, $ZrSiO_4$, $CoAl_2O_4$

Powder	Isoelectric points
Al_2O_3 200#	2.8
Al_2O_3 325#	9.1
$CoAl_2O_4$	5.2
$ZrSiO_4$	3.1

to increased risk of particle flocculation, uneven layer thickness, and potential casting defects – an issue also highlighted by Kaiser et al. [4] in their study on zircon decomposition and slurry instability [4]. Therefore, understanding and controlling zeta potential is not only essential for suspension stability but also for ensuring the mechanical integrity and surface quality of ceramic molds, particularly when transitioning to water-based, sustainable binder systems. The current findings reinforce the importance of this parameter in the design and optimization of casting materials and processes.

Diffraction diagrams of powders used to fabricate prime coats are shown in Figure 7. The phase analysis results of the each powders compositions are shown in Table 3.

Analysis of the phase composition of Al_2O_3 powders showed that exhibit two-phase of materials - Al_2O_3 aluminum oxide and $NaAl_{11}O_{17}$ sodium aluminate (often called beta alumina). It should be emphasized that the test powders are primarily characterized by a high content of α oxides All of tested powders exhibit of large value of Al_2O_3 amount over than 99%. Rest of the substances are a pollution. Analysis of the phase composition of the tested powders showed that exhibit single-phase materials. The aluminate powder is composed only of $CoAl_2O_4$ cobalt aluminate and $ZrSiO_4$ powder contains only $ZrSiO_4$ zirconium silicate. Cobalt blue sample revealed the large amount of CoO and Al_2O_3. It can be assumed that sum of this substances is over than 98.76%. However, 1.21% are the pollution amount. Results of tested zirconium silicate are comparable. Nevertheless, pollution amount is larger and equal 6.05%. Bases of the zirconium silicate are ZrO_2 59.25% and SiO_2 – 34.70%.

Backup layers powders investigation

Microstructures of researched powders used for backup layers fabrication are shown in Figure 8–11. Analysis of the obtained SEM images shows that the powders have a typical morphology of the particles obtained in the high-temperature kaolin calcining process. This is followed by crushing and fractionation. The shape of the

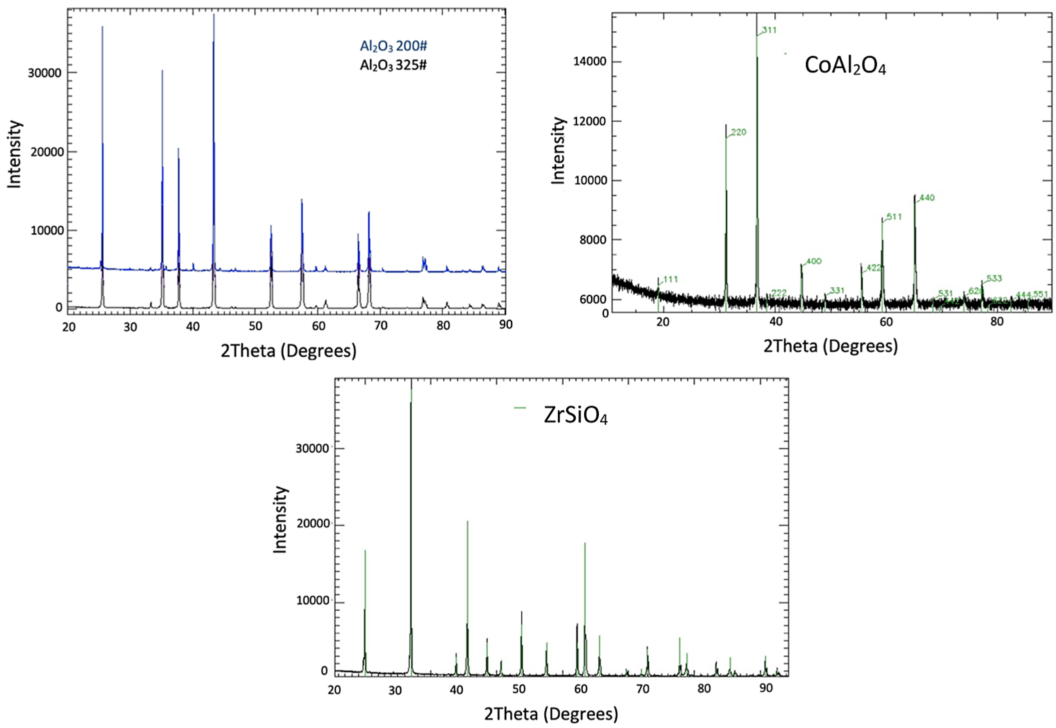

Figure 7. Diffraction diagrams of the powders

Table 3. Chemical composition of XRF tests for tested powders

Al_2O_3 200#		Al_2O_3 325#	
Stoichiometric formula	Amount [%]	Stoichiometric formula	Amount [%]
Al_2O_3	99.34	Al_2O_3	99.54
P_2O_5	0.293	P_2O_5	0.271
CaO	0.175	CaO	0.152
SiO_2	0.15	Fe_2O_3	0.0315
Fe_2O_3	0.0287	Ga_2O_3	0.0101
Ga_2O_3	0.0097		
$CoAl_2O_3$		$ZrSiO_4$	
Stoichiometric formula	Amount [%]	Stoichiometric formula	Amount [%]
CoO	49.39	ZrO_2	59.25
Al_2O_3	49.40	SiO_2	34.70
SiO_2	0.64	HfO_2	1.51
P_2O_5	0.25	Al_2O_3	2.92
CaO	0.16	CaO	0.39
NiO	0.08	P_2O_5	0.38
SO_3	0.05	Y_2O_3	0.24
Fe_2O_3	0.03	TiO_2	0.47
		Fe_2O_3	0.09

particles is irregular with sharp edges with a strongly expanded surface. This guarantees good adhesion of powders in the process of fabrication a ceramic shell moulds. For the Molochite 120 matrix powder, two fractions of powder particles were observed – fine and coarse. This is typical for the ceramic matrix of subsequent structural layers of foundry molds.

The obtained distribution of mean particle diameter of the powders used for backup layers are shown in Figure 12 and Table 4. It was established that the powders were characterized by the following features:

- Al_2O_3 80 # – the powder has a relatively narrow size range of its particles from 67 μm to 300 μm; the obtained distribution is symmetrical and has one maximum – d = 174.42 μm; the average diameter of the powder particles is 184.5 ± 6.20 μm,
- Molochite 120 – powder has a wide particle size range from 2.60 μm to 262.68 μm; the average diameter of the powder particles is 51.22 ± 2.24 μm; the resulting distribution is asymmetrical and has 3 maximums – d = 10.10 μm, d = 39.23 μm and d = 88.58 μm, which may indicate the occurrence of agglomerates,

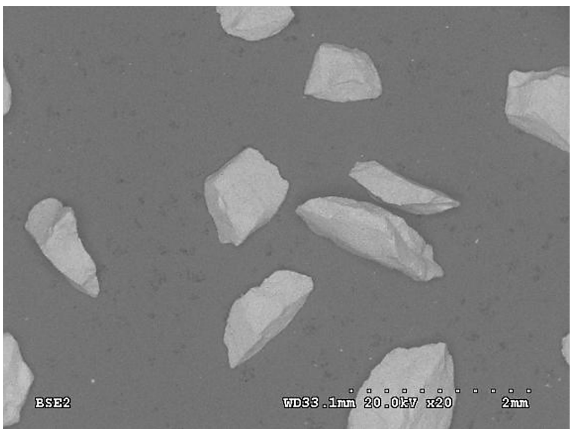

Figure 8. SEM microstructure of Molochite 16/30

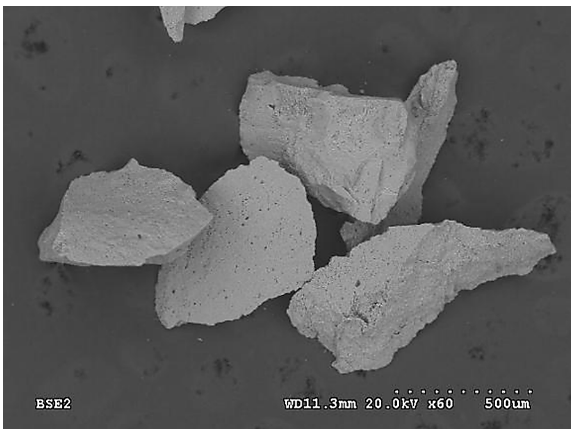

Figure 9. SEM microstructure of Molochite 30/80

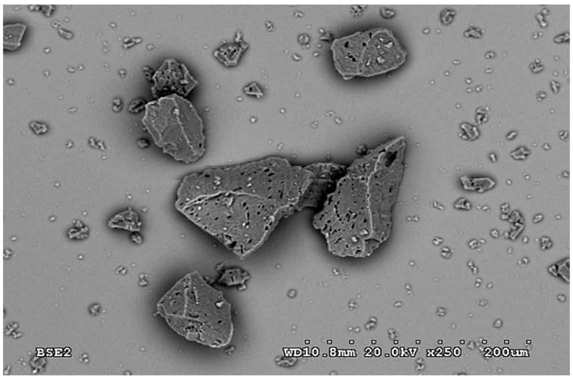

Figure 10. SEM microstructure of Molochite 120

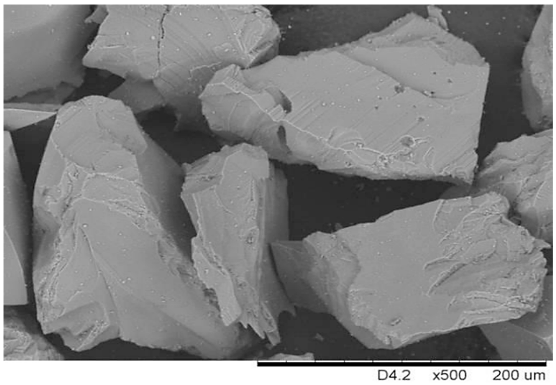

Figure 11. SEM microstructure of Al2O3 80#

- Molochite 30/80 – powder has a wide particle size range from 152.45 µm to 890.12 µm; the average particle diameter of the powder is 479.74 ± 15.78 µm; the obtained distribution is symmetrical and has one maximum – d = 517.20µm,
- Molochite 16/30 – the powder tested has a wide particle size range from 300.52 µm to 1754.61µm; the average particle diameter of the powder is 479.74 ± 23.53 µm; the obtained distribution is symmetrical and has two maximums – d = 870.12µm and d = 1167.73 µm, which may indicate the presence of agglomerates.

Diffraction results of the backup powders are shown in Figure 13. Analysis of the phase composition showed in Table 5 obtained for all Molochite tested powders, revealed a single-phase material, composed only of $Al_6Si_2O_{13}$ – mullite aluminosilicate. Higher intensity for Molochite 120 is caused by increased exposure time – 15 s. In addition, the effect caused by the presence of an amorphous phase is visible on the diffractogram of the Molochite 120 powder. All tested samples exhibit the larges amount of SiO_2 and Al_2O_3 over than 95%. It can be assumed that the pollution amount is nearby 5%. In contrast with the powders for prime coat, backup powders exhibit large value of pollution and its final cost is lower than prime coat powders.

DISCUSSION

The results of the conducted research on the characteristics of new powders used in environmentally friendly technology for producing

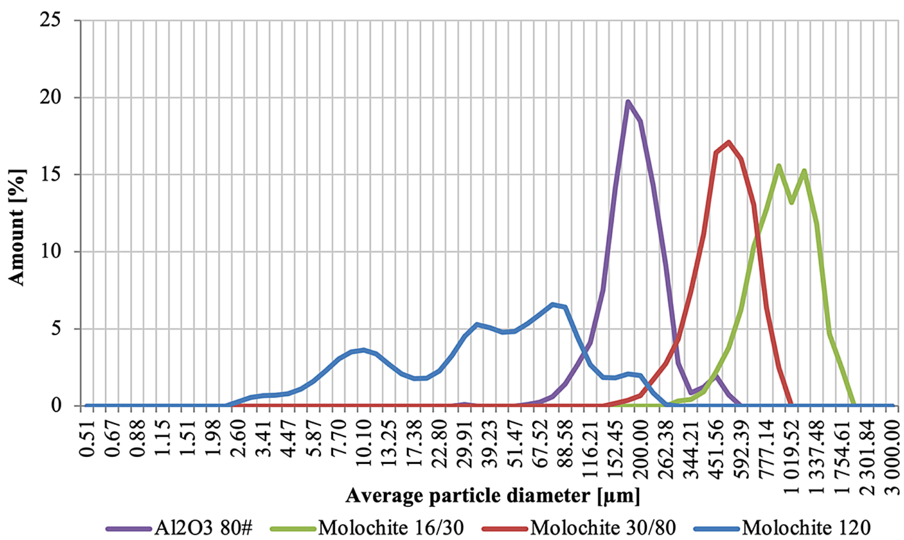

Figure 12. Distribution of the average particle diameter of powders used for backup layers – Al_2O_3 80#, Molochite 16/30, Molochite 30/80, Molochite 120

Table 4. Comparison of the average particle diameter of the tested powders

Powder	Test environment	Average particle diameter [μm]
Al_2O_3 80#	WATER	184.5 ± 6.20
Molochite 16/30	WATER	902.8 ± 23.53
Molochite 30/80	WATER	479.7 ± 15.78
Molochite 120	WATER	51.2 ± 2.24

ceramic shell molds for precision casting of aircraft turbine blades have provided valuable insights into their microstructure, chemical composition, particle size, and stability in aqueous solutions. The obtained data indicate that the tested powders meet key technological requirements and may serve as an effective alternative to traditional materials used in foundry applications. SEM analysis revealed that the investigated powders exhibit irregular particle shapes, which is advantageous in terms of adhesion during the formation of multilayer ceramic structures. The presence of sharp edges and an extended particle surface area is particularly important, as it may contribute to better layer adhesion and increased mold strength. Zeta potential measurements provided information on the electrostatic stability of powder suspensions in water. The results indicate

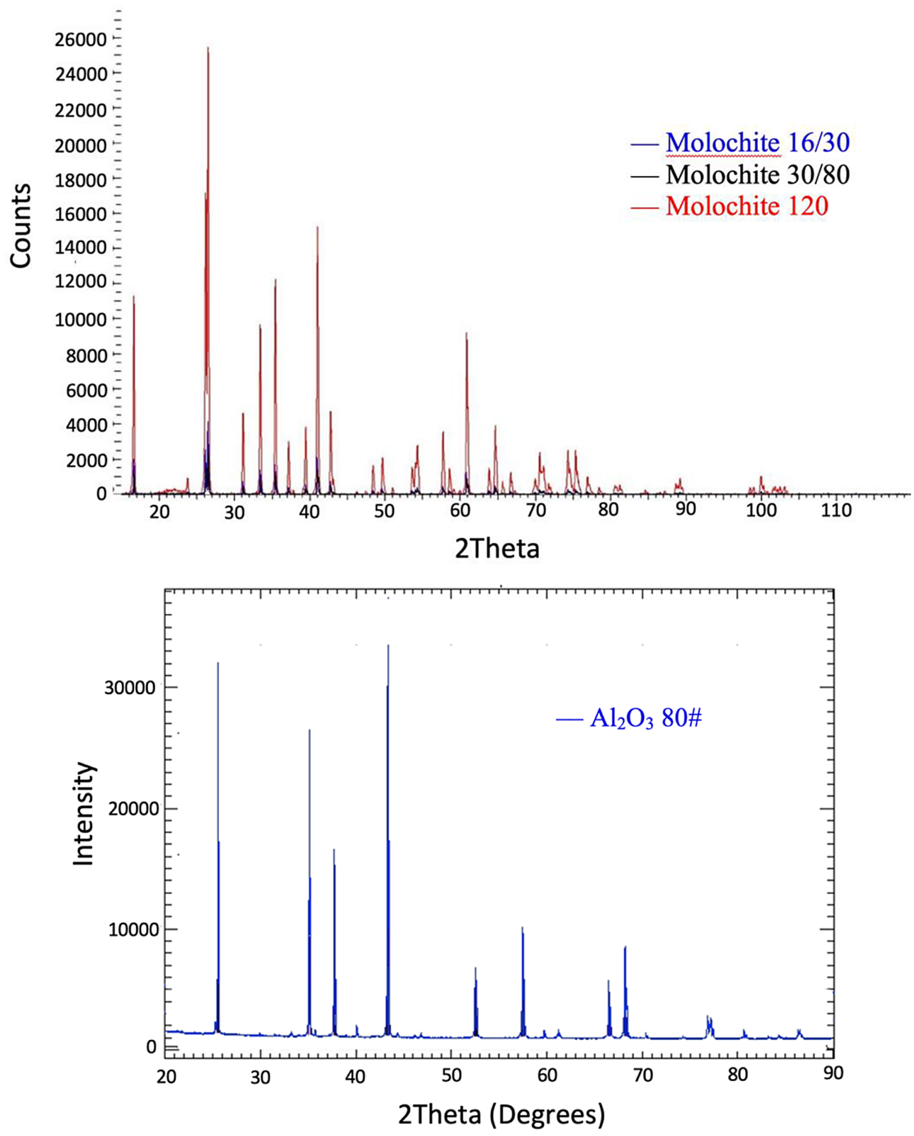

Figure 13. Diffraction diagrams of Molochite 16/30, Molochite 30/80, Molochite 120 and Al_2O_3 80#

Table 5. Chemical composition of XRF tests for tested powders Molochite 16/30, Molochite 30/80, Molochite 120 and Al_2O_3 80#

Molochite 16/30		Molochite 30/80		Molochite 120		Al_2O_3 80#	
Stoichiometric formula	Amount [%]	Stoichiometric formula	Amount [%]	Stoichiometric formula	Amount [%]	Stoichiometric formula	Amount [%]
SiO_2	50.8	SiO_2	51.1	SiO_2	51.1	Al_2O_3	99.41
Al_2O_3	44.6	Al_2O_3	44.2	Al_2O_3	44.3	P_2O_5	0.318
K_2O	2.27	K_2O	2.31	K_2O	2.30	CaO	0.222
Fe_2O_3	1.544	Fe_2O_3	1.562	Fe_2O_3	1.453	Fe_2O_3	0.0285
P_2O_5	0.418	P_2O_5	0.419	P_2O_5	0.360	Ga_2O_3	0.0184
CaO	0.223	CaO	0.233	CaO	0.218		
TiO_2	0.071	TiO_2	0.0811	TiO_2	0.0741		
Rb_2O	0.0447	Rb_2O	0.0449	Rb_2O	0.0387		
SrO	0.0434	SrO	0.0443	SrO	0.0375		
ZrO_2	0.0270	ZrO_2	0.0275	ZrO_2	0.0236		
Ga_2O_3	0.010	CuO	0.009	Ga_2O_3	0.008		
CuO	0.008	Ga_2O_3	0.009	CuO	0.008		

that the selected powders have different isoelectric points, which may influence their behavior in suspension. It was determined that Al_2O_3 325# exhibits an isoelectric point at pH 9.1, meaning that in a more alkaline environment, its particles will carry a negative charge, improving suspension stability. In contrast, $ZrSiO_4$ has an isoelectric point at pH 3.1, suggesting that it may be less stable in acidic conditions.

The findings of this study are consistent with publications on materials used in casting processes. Ferenc et al. [30] emphasized the crucial role of controlling the rheological properties of ceramic suspensions through the appropriate selection of organic modifiers. In this study, zeta potential analysis provided similar information, confirming that the electrostatic stability of suspensions is key to maintaining the uniformity of ceramic mold layers. Kaiser et al. [31] analyzed the thermal stability of zircon ($ZrSiO_4$) and found that it is susceptible to decomposition at high temperatures, which may lead to surface defects in castings. The results of this study confirm these observations, indicating that the use of $ZrSiO_4$ requires strict control of process conditions to prevent degradation and interactions with cast alloys. Gao and Bai [32] highlighted the need for optimizing the chemical composition and microstructure of powders to achieve the desired mechanical and thermal properties in castings. The findings of this study confirm that Al_2O_3 and $CoAl_2O_4$ powders can be effectively used in precision casting of aerospace components due to their chemical stability and appropriate particle morphology. Lee and Oh [33] demonstrated that the use of nanoparticles in ceramic systems can improve layer uniformity and increase mold resistance to thermal cracking. The results of this study on particle distribution and its impact on mold structure confirm these findings, emphasizing the importance of precise powder selection in minimizing casting defects. Zhao et al. [34] identified the development of environmentally friendly binders as a key factor in modernizing casting technologies. In the context of this study, the application of aqueous binder systems can significantly enhance the environmental aspect of ceramic mold production by reducing volatile organic compound emissions and improving working conditions in foundries. Huang and Lin [35] investigated the optimization of mechanical strength and thermal stability of ceramic molds, confirming the importance of precise control over sintering parameters and the chemical composition of powders used. The results of this study, particularly regarding the thermal stability of Al_2O_3 and its ability to limit interactions with metal alloys, align with these findings.

Richards and Bowen [36] conducted research on the impact of ceramic suspension rheological parameters on the final strength of molds. Their analyses showed that the appropriate selection of particle size and rheological stabilizers can significantly improve mold structure uniformity and enhance its resistance to cracking. Turner and Bell [37, 38] indicated that the use of synthetic powders such as Al_2O_3 and $CoAl_2O_4$ can reduce chemical inhomogeneities in ceramic molds. In this study, it was confirmed that the application of these powders leads to increased thermal and mechanical resistance of molds, which aligns with their conclusions. Martinez and Singh [39, 40] carried out extensive research on the influence of heat treatment processes on the microstructure of ceramic molds. They determined that precise control of sintering thermal parameters allows for the reduction of porosity and improvement of dimensional stability in castings, which corresponds with the results obtained in this study. Patel and Kumar [41] analyzed various types of protective coatings applied to ceramic mold surfaces. Their research demonstrated that the addition of metal oxide-based protective layers can significantly enhance mold durability and reduce the risk of defects in castings. Wang and Xu [42] highlighted the impact of ceramic powder grain size on its mechanical properties. Their studies showed that reducing powder particle size leads to better structural densification of the mold and increased resistance to mechanical stresses.

In summary, the results of this study provide a valuable contribution to the development of environmentally friendly technologies for producing ceramic shell molds. The obtained data indicate a high potential for using Al_2O_3 and $CoAl_2O_4$ powders in innovative casting processes, which may contribute to improving casting quality and reducing environmental impact. Use of alumina and alumina silicates powders increases yield of the final aircraft parts. Nowadays most of the alloys uses to fabricate turbine blades contain a very reactive alloy element- hafnium. Kaiser and all describe the decomposition reaction of zirconium silicate. The reaction with the components of the liquid alloy is synonymous with the formation of defects on the surface of the precise casting. In addition, a gradual increase in the $ZrSiO_4$ price is currently observed – 1.8 ÷ 1.9 euro/kg. Therefore, precision foundries are trying to replace silicate with synthetic products of considerable chemical purity e.g. corundum. The results of the tests have confirmed that the corundum does not degrade during the tests. Use of synthetically manufactured compounds, i.e. alumina as a consequence reduces the above-described process. Manufacturer receives the same purity all the time, not as in the case of $ZrSiO_4$, which is a mineral.

The development of this new technology for ceramic shell mold fabrication is strongly driven by environmental concerns and regulatory requirements, particularly within the European Union. Traditional methods in investment casting have relied heavily on ethanol- or methanol-based binders, which release substantial amounts of volatile organic compounds (VOCs) into the atmosphere during the drying phase. These substances pose serious health risks to workers and contribute to air pollution, thereby prompting a shift toward more sustainable and safer alternatives.

In this context, the adoption of water-based colloidal silica binders significantly enhances the environmental profile of the ceramic mold fabrication process. By completely eliminating the use of alcohol-based binders, the proposed method effectively reduces VOC emissions by more than 90%, leading to a substantial improvement in indoor air quality and worker safety. As highlighted by Zhao et al. [7] and Kumar and Singh [10], water-soluble binders not only comply with current environmental legislation but also reduce the need for costly air filtration systems and personal protective equipment [7, 10].

Furthermore, this environmentally friendly approach contributes to energy savings at multiple stages of production. First, water-based systems eliminate the fire hazard associated with alcohol vapors, allowing for simpler and less energy-intensive ventilation infrastructure. Second, improvements in slurry stability, driven by optimized powder selection and zeta potential control, result in more uniform shell layers and reduced drying times. This leads to lower overall energy consumption during the drying and sintering phases, as fewer defective molds require reprocessing or additional heating cycles.

The use of synthetic ceramic powders such as high-purity alumina (Al_2O_3) and cobalt aluminate ($CoAl_2O_4$) further enhances the environmental sustainability of the process. Unlike natural zirconium silicate, which may contain impurities and require energy-intensive purification steps, synthetic powders offer consistent chemical composition and eliminate the need for complex refining operations. Additionally, the declining availability and rising cost of $ZrSiO_4$ due to environmental limitations on its mining and processing reinforce the need for alternative raw materials with lower ecological footprints.

The new technology also aligns with the principles of circular economy by reducing waste and improving material utilization. Improved layer adhesion and reduced defect rates contribute to higher casting yields, meaning that less metal is wasted during the post-casting rejection of defective components. This is particularly critical in aerospace applications, where high-performance nickel superalloys are expensive and resource-intensive to produce.

Finally, the elimination of hazardous substances from the manufacturing process creates a more sustainable working environment and supports long-term occupational health. Employees are no longer exposed to chronic inhalation of alcohol vapors, and the process becomes safer and more acceptable in laboratory and industrial settings alike. As noted by Ismael et al. [25], the use of colloidal silica as a binder not only improves technical performance but also significantly lowers the ecological and human health burden associated with ceramic mold production.

In conclusion, the developed ecological technology represents a significant advancement in sustainable manufacturing for the casting industry. It integrates material innovations with environmentally responsible practices, offering a high-performance alternative that reduces emissions, minimizes waste, and ensures compliance with modern environmental and safety standards.

To fully evaluate the performance and industrial relevance of the developed ceramic shell mold technology, it is important to compare it with existing solutions currently utilized in the aerospace industry. Conventional shell mold systems for turbine blade casting predominantly rely on zirconium silicate ($ZrSiO_4$) as the base ceramic powder, combined with ethanol-based binders. These systems have been in use for decades due to their relative affordability and established process compatibility. However, several drawbacks – including thermal instability, environmental concerns, and inconsistent raw material quality – have prompted a shift toward more advanced alternatives.

The newly developed system, based on synthetic high-purity alumina (Al_2O_3) and cobalt aluminate ($CoAl_2O_4$) powders combined with water-soluble colloidal silica binders, offers multiple advantages over traditional solutions. Firstly, the chemical composition of the synthetic powders is more consistent and free from naturally occurring contaminants that are often present in mined zirconium silicate. As confirmed through XRF and XRD analysis in this study, Al_2O_3 powders used in the developed solution contain more than 99.5% pure alumina, while $ZrSiO_4$ powders often exhibit variable concentrations of hafnium, iron oxides, and other impurities. These impurities can react adversely with superalloy components – particularly hafnium-containing nickel alloys – during high-temperature casting, leading to surface defects and casting rejections [4].

Secondly, from a mechanical and thermal performance perspective, the new powders show superior behavior under extreme temperatures. Alumina and cobalt aluminate are both thermally stable above 1500 °C, which is critical for casting monocrystalline turbine blades. In contrast, zircon decomposes at elevated temperatures, generating silica and zirconia, which can promote unwanted reactions at the mold-metal interface. This thermal degradation compromises the dimensional stability of the mold and increases the risk of ceramic inclusions in the final casting [5; 34].

The stability of ceramic suspensions in water-based systems is another area where the proposed solution outperforms traditional technologies. The high absolute zeta potential values observed in Al_2O_3 325# and $CoAl_2O_4$ powders in alkaline pH conditions suggest enhanced dispersion and reduced agglomeration, which translates to more uniform coating layers. This is particularly important in aerospace applications, where tight dimensional tolerances and defect-free surfaces are essential for component performance and safety. In comparison, traditional $ZrSiO_4$ suspensions are less stable, requiring the addition of rheological modifiers and stabilizers to maintain acceptable processability [2].

From an economic standpoint, while synthetic alumina may have a higher unit cost than natural zircon at first glance, this is offset by the higher yield and reduced defect rate observed in the casting process. The need for fewer mold repairs and reduced casting rejections translates to lower overall production costs. Moreover, the market price of zirconium silicate has been steadily increasing due to mining restrictions and geopolitical factors, making it a less reliable long-term option for high-volume aerospace casting operations.

Environmentally, the new system also presents a substantial improvement. The use of water-based binders eliminates the health risks and VOC emissions associated with ethanol-based systems.

Additionally, the ability to work with high-purity synthetic powders reduces the environmental impact of mining and mineral processing, aligning the technology with modern sustainability goals and regulatory frameworks [7, 25].

In summary, when compared with existing market solutions, the developed ceramic shell mold system offers higher chemical purity, better thermal and mechanical stability, improved slurry performance, and lower environmental impact, making it a superior alternative for aerospace applications. Its compatibility with current casting infrastructure and potential for process scalability further support its adoption in industrial settings, particularly in sectors requiring the highest standards of precision and material performance.

CONCLUSIONS

The research conducted in this study represents a significant contribution to the field of ecological ceramic shell mold fabrication for precision casting, particularly in the production of aircraft turbine blades. The comprehensive characterization of new powders used in this process has led to an improved understanding of their microstructure, chemical composition, particle size distribution, and stability in aqueous environments. The transition from traditional alcohol-based binders to water-based alternatives has not only addressed stringent environmental and occupational safety regulations but has also resulted in the optimization of the ceramic shell mold fabrication process. The tested powders, primarily composed of alumina (Al_2O_3), zirconium silicate ($ZrSiO_4$), and cobalt aluminate ($CoAl_2O_4$), have demonstrated excellent properties suitable for ceramic shell mold fabrication. The SEM analysis confirmed their irregular particle morphology, which is advantageous for layer adhesion, ultimately improving the mechanical integrity of the molds. The use of Al_2O_3-based powders, particularly in prime coatings, has shown significant potential in replacing traditional zirconium silicate-based powders, thereby enhancing the thermal stability and chemical purity of the molds. X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysis have confirmed the high purity and stability of the selected powders. The primary phases detected in Al_2O_3-based powders ensure their resistance to high-temperature decomposition and reactions with molten alloys. Unlike $ZrSiO_4$, which tends to degrade at elevated temperatures, Al_2O_3 and $CoAl_2O_4$ maintain their structural integrity, making them more suitable for high-performance applications. The zeta potential measurements provided critical insights into the electrostatic stability of the powder suspensions. The data revealed that Al_2O_3 325# exhibits an isoelectric point at pH 9.1, resulting in enhanced dispersion stability in an alkaline medium. In contrast, $ZrSiO_4$, with an isoelectric point at pH 3.1, is prone to instability in acidic conditions, making it less suitable for prolonged suspension in aqueous environments. The shift towards synthetic Al_2O_3-based powders addresses both cost and environmental concerns. The rising cost of $ZrSiO_4$, estimated at 1.8–1.9 EUR/kg, makes it an increasingly less viable option. Additionally, the elimination of alcohol-based binders from the manufacturing process significantly reduces VOC emissions, improving workplace safety and compliance with European environmental regulations. The transition to water-based binders has further resulted in cost savings related to reduced air filtration and personal protective equipment requirements. One of the most critical findings of this study is the role of powder morphology in minimizing casting defects. The presence of sharp edges and an expanded surface area in Al_2O_3 and $CoAl_2O_4$ powders enhances adhesion between layers, reducing the likelihood of delamination during sintering and metal casting. This contributes to a lower defect rate in turbine blade production, ultimately improving the reliability and efficiency of aerospace components.

The findings of this research offer valuable insights for the investment casting industry, particularly in the aerospace sector, where the demand for high-performance turbine blades is increasing. The adoption of Al_2O_3 and $CoAl_2O_4$ powders as primary materials in ceramic shell molds ensures improved mechanical and thermal stability, making them a superior alternative to $ZrSiO_4$. Furthermore, the successful integration of water-based binders into the process aligns with sustainability goals, reducing environmental impact while maintaining production efficiency. The use of advanced characterization techniques in this study has established a strong foundation for future improvements in ceramic mold fabrication. The observed advantages of synthetic Al_2O_3-based powders suggest the potential for widespread industrial adoption, offering a sustainable and cost-effective solution for precision casting.

While this study has demonstrated the feasibility of using water-based binders, further research is required to optimize their formulation and application parameters. Studies focusing on nanoparticle-based binders could enhance layer uniformity, reduce drying times, and further improve mechanical strength. Combining different ceramic powders, such as Al_2O_3 with yttria-stabilized zirconia (YSZ), may enhance the thermal and mechanical properties of shell molds. Investigating such hybrid compositions can provide insights into their potential advantages over conventional materials. Conducting long-term studies on the durability and performance of ceramic molds made from Al_2O_3 and $CoAl_2O_4$ powders under actual casting conditions will provide deeper insights into their reliability. Assessing their performance in high-temperature environments over multiple casting cycles will be essential for validating their industrial applicability. The integration of additive manufacturing techniques, such as 3D printing, with ceramic mold fabrication may offer further advancements in precision and customization. Research in this area could lead to the development of innovative mold designs optimized for complex aerospace components.

The results of this study provide a strong scientific basis for the transition to environmentally friendly, high-performance ceramic shell mold fabrication. The demonstrated advantages of Al_2O_3 and $CoAl_2O_4$ powders in combination with water-based binders confirm their potential for large-scale industrial implementation. As the aerospace industry continues to demand superior casting quality, these findings pave the way for future advancements in sustainable precision casting technologies. The successful adoption of these materials and processes will not only enhance the efficiency and quality of turbine blade production but also contribute to reducing the environmental footprint of the foundry industry. The elimination of hazardous substances, coupled with improvements in mold integrity and defect reduction, establishes a new benchmark for modern investment casting practices. With further research and technological refinement, these advancements will continue to shape the future of ecological manufacturing in high-performance applications.

The research conducted in this study confirms the suitability and advantages of using synthetic ceramic powders – namely alumina (Al_2O_3) and cobalt aluminate ($CoAl_2O_4$) – in combination with water-based colloidal silica binders for the ecological production of ceramic shell molds in the precision casting of turbine blades. The key findings can be summarized as follows:

- The tested Al_2O_3 powders exhibited high chemical purity (> 99.5%), favorable particle morphology, and thermal stability above 1500 °C.
- $CoAl_2O_4$ and $ZrSiO_4$ powders demonstrated distinctive zeta potential behaviors that directly influenced slurry stability and casting layer uniformity.
- The application of water-based binders significantly reduced VOC emissions and improved workplace safety.
- Enhanced dispersion of powders, particularly Al_2O_3 325#, resulted in better layer uniformity, reduced agglomeration, and minimized surface defects.
- The developed system reduced mold failure rates and improved the mechanical integrity of the ceramic shells.
- Increased chemical stability led to fewer interactions between the mold and reactive elements (e.g., hafnium) in nickel-based superalloys, resulting in cleaner, defect-free castings.
- The elimination of alcohol-based binders and the use of synthetic materials contributed to lower environmental burden and ensured compliance with modern EU safety and sustainability regulations.
- Improved mold performance and higher casting yield reduced waste and production costs.
- While the results are promising, several areas remain open for further investigation to optimize the performance and industrial scalability of the proposed system.
- Future studies should explore the use of nanoparticle-enhanced colloidal silica binders to improve mechanical strength, reduce drying time, and increase green strength of the ceramic layers.
- Investigating the synergistic effects of Al_2O_3 with other ceramics, such as yttria-stabilized zirconia (YSZ) or spinel-based materials, may lead to enhanced thermal shock resistance and dimensional accuracy.
- Long-term studies should be conducted to evaluate the durability of molds under repetitive thermal cycling and high-temperature exposure, simulating real industrial casting conditions.

- Combining this material system with 3D printing technologies could allow for rapid prototyping of complex shell geometries and further reduce material waste.
- The application of real-time process monitoring, including rheology and viscosity measurements during slurry preparation and coating, could improve reproducibility and automation potential.
- A comprehensive life cycle assessment (LCA) of the new system compared to traditional methods would provide valuable insights into its full environmental impact and industrial feasibility.

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