

https://doi.org/10.51885/3134-7983_CATMSP_2026_3_4

SRSTI 55.22.19

INFLUENCE OF OXYGEN PRESSURE DURING HVOF SPRAYING ON THE CORROSION AND THERMAL PROPERTIES OF AL-CU-FE QUASI-CRYSTALLINE COATINGS

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Keywords:

corrosion; Al-Cu-Fe; HVOF; TGA; SEM.

ABSTRACT

Al-Cu-Fe quasi-crystalline coatings were deposited onto AISI 321 stainless steel substrates using the high-velocity oxygen fuel (HVOF) spraying method at oxygen pressures of 3.0 and 3.5 bar. The effect of oxygen pressure on the phase composition, microstructure, porosity, corrosion behavior, and thermal stability of the coatings was investigated using X-ray diffraction (XRD), scanning electron microscopy combined with energy-dispersive spectroscopy (SEM/EDS), porosity analysis in ImageJ, electrochemical corrosion tests in a 3.5% NaCl solution, and simultaneous thermal analysis (TGA/DSC). XRD analysis revealed the formation of quasi-crystalline intermetallic phases alongside phases of Al, Fe₃Al₁₃, FeAl, Fe₃O₄, CuFe₂O₄, Cu₂O, and CuO. The coating deposited at a pressure of 3.5 bar exhibited the lowest porosity (5.37%), the most uniform microstructure, and the greatest residual coating thickness after corrosion testing. SEM and EDS analyses showed that corrosion predominantly initiated in pores, particle boundaries, and interphase boundaries, whereas the coating obtained at a pressure of 3.5 bar exhibited the most stable surface condition after exposure to a 3.5% NaCl solution. Thermal analysis showed that all coatings remained stable up to 900 °C. Sample (a) exhibited the lowest mass loss and the highest thermal stability, whereas sample (b) demonstrated the most favorable combination of structural integrity, phase order, coating density, corrosion resistance, and thermal stability. The results show that oxygen pressure is a critical parameter controlling the microstructure and functional properties of Al-Cu-Fe coatings deposited by the HVOF method, with 3.5 bar providing the most balanced set of properties.

INTRODUCTION

Improving the corrosion resistance of structural materials operating in aggressive marine environments remains a major challenge in materials science. Protective coatings are widely used to mitigate corrosion damage and extend the service life of engineering components



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[(Spiridonova et al., 2002), (Sukhova, 2009) & (Spiridonova et al., 1996)]. Among advanced protective materials, Al-Cu-Fe quasi-crystalline coatings have attracted considerable attention because of their high hardness, low friction coefficient, chemical stability, wear resistance, and corrosion resistance [Huttunen-Saarivirta et al. (2004) and Spurr et al. (1999)]. In addition, thermally sprayed zinc duplex coatings provide long-term corrosion protection for steel structures, while advanced Zn-based alloy coatings containing Al, Mg, Sn, and Bi have been developed to further enhance corrosion and mechanical performance [Liu et al. (2024) and Knudsen et al. (2019)]. Although previous studies have demonstrated that HVOF/HVAF spraying parameters strongly influence the phase composition, oxidation, porosity, and performance of Al-Cu-Fe quasi-crystalline coatings, the specific effect of oxygen pressure on their corrosion behavior remains insufficiently understood. Therefore, this study investigates the relationship between oxygen pressure during HVOF spraying, coating microstructure, and corrosion resistance.

Among quasi-crystalline systems, Al-Cu-Fe alloys are of particular interest because they readily form an icosahedral (i-) phase during solidification [Sukhova, O. V., & Syrovatko, Y. V. (2011)]. This phase exhibits low density, high hardness, and a high elastic modulus, making it suitable for protective coatings [Spyrydonova, I. M., et al (2012)]. However, corrosion studies on cast Al-Cu-Fe alloys in NaCl solutions have shown that corrosion resistance depends primarily on phase constitution and chemical composition rather than on the quasi-crystalline phase itself [Huttunen-Saarivirta and Tiainen (2004) and Sukhova et al. (2018)]. Uniform corrosion is generally observed, while copper-rich phases reduce the corrosion rate; potentiostatic tests also reveal susceptibility to pitting corrosion with porous copper-rich residues inside corrosion pits [Massiani et al. (1993) and Sukhova et al. (2018)]. Salt spray exposure similarly indicates no significant influence of quasi-crystalline phase content on the corrosion resistance of cast alloys [Rudiger, A., & Koster, U. (2018)].

In contrast, Al-Cu-Fe alloys produced under non-equilibrium conditions exhibit significantly different corrosion behavior, creating opportunities for improving performance through thermal spraying and vapor deposition techniques [Shaitura and Enaleeva (2007), Spiridonova et al. (1993), and Spiridonova et al. (1996)]. Plasma-sprayed coatings can reach thicknesses of up to 1 mm, although their corrosion resistance in acidic and alkaline environments may be inferior to that of cast alloys [Ryabtsev, S. I., et. al (2020)]. Thin films produced by magnetronspraying and related ion-plasma methods generally provide superior structural uniformity and purity [Sukhova et al. (2019), Milagre et al. (2019), and Dai et al. (2022)]. Their phase composition depends strongly on film thickness and substrate temperature, with amorphous structures transforming into crystalline or predominantly quasi-crystalline coatings after annealing or deposition above 400 °C [Parsamehr et al. (2019), Milman et al. (2023), Egorkin et al. (2020), and Ryabtsev et al. (2020)]. These coatings develop protective Al₂O₃ surface films, exhibit excellent environmental stability, and show ceramic-like wear behavior in aqueous media [Abaei et al. (2023), Sukhova et al. (2019), and Sukhova et al. (2019)].

Further improvements can be achieved using three-electrode ion-plasma spraying, which increases the kinetic energy of deposited atoms and promotes highly non-equilibrium structure formation [Ryabtsev et al. (2012), Dotsenko and Bashev (2001), Efremenko et al. (2017), and Grant et al. (1983)]. Since rapid cooling reduces the fraction of the quasi-crystalline phase [Holland-Moritz et al. (1997)], scandium addition has been proposed as a stabilizing element [Karpets and Milman (2005)]. The incorporation of approximately 0.5 at.% Sc increases the i-phase fraction by 20–25% and enhances the corrosion resistance of Al-Cu-Fe coatings in NaCl solutions [Ryabtsev (2009)]. These findings suggest that combining non-equilibrium deposition with appropriate alloying provides an effective strategy for developing corrosion-resistant quasi-crystalline coatings for aerospace and marine applications.

MATERIALS AND METHODS

The coatings were deposited using a Termika-3 HVOF thermal spraying system. The HVOF process provided particle velocities of $500\text{--}700\text{ m s}^{-1}$ and a maximum particle temperature of approximately $1500\text{ }^{\circ}\text{C}$. Oxygen and propane were used as fuel gases, with oxygen pressure of $0.8\text{--}1.0\text{ MPa}$ and propane pressure of $0.6\text{--}0.8\text{ MPa}$ [Cai, M., & Shen, J. (2020)]. The oxygen and propane flow rates were $30\text{--}40$ and $15\text{--}20\text{ L min}^{-1}$, respectively. Air cooling was applied during spraying at a flow rate of $500\text{--}700\text{ L min}^{-1}$ and a pressure of 0.6 MPa . The feedstock material was Al-Cu-Fe powder with a particle size of $-40 +15\text{ }\mu\text{m}$.

The spraying parameters were selected according to the operating characteristics of the HVOF process reported in [Cai, M., & Shen, J. (2020)]. A spray distance of 250 mm was chosen to balance particle temperature and impact velocity, preserving the quasi-crystalline structure while preventing excessive overheating. A propane pressure of 1.9 bar ensured stable combustion and uniform particle heating, whereas the oxygen pressure ($3.0\text{--}3.5\text{ bar}$) was varied to optimize particle kinetic energy, coating densification, and adhesion. An air pressure of 2.2 bar promoted complete combustion and minimized particle oxidation during flight.

Particular attention was paid to preserving the quasi-crystalline structure of the $\text{Al}_{65}\text{Cu}_{20}\text{Fe}_{15}$ coating. The influence of oxygen pressure on coating formation, microstructure, and corrosion performance was systematically investigated.

The surface morphology and elemental composition of the coatings were characterized using scanning electron microscopy (SEM) (Tescan Vega 4, Brno, Czech Republic). The phase composition of the deposited coatings was analyzed by X-ray diffraction (XRD) using a Malvern Panalytical X'Pert PRO diffractometer (Philips, Amsterdam, The Netherlands) with a Cu anode ($\lambda = 1.5406\text{ \AA}$, Cu $K\alpha$ radiation). All measurements were performed at room temperature ($25\text{ }^{\circ}\text{C}$) using an operating voltage of 40 kV and a current of 30 mA . Diffraction patterns were recorded over a 2θ range of $20.01^{\circ}\text{--}89.99^{\circ}$ with a step size of 0.02° and a counting time of 2 s per step. Phase identification and analysis were conducted using the HighScore Plus software (Malvern Panalytical).

Prior to corrosion testing, the coated samples were mechanically polished, cleaned, and dried. Electrochemical measurements were performed using a P-20X potentiostat-galvanostat (Russia) in a step potentiostatic mode. The tests consisted of 10 potential steps (1.5 mV per step, 30 s dwell time) within a total potential range of 2 V . The maximum current was 1500 mA , the minimum detectable current was 200 nA , and the acquisition rate was $3.33\text{ points s}^{-1}$. Corrosion behavior was evaluated in $3.5\text{ wt.}\%$ NaCl solution ($\text{pH } 6.9\text{--}7.1$) at room temperature using a two-electrode configuration, with the coated sample as the working electrode. The total test duration was 300 s .

Thermal analysis was carried out using an STA-200 instrument under a nitrogen atmosphere. Samples were heated from room temperature to $900\text{ }^{\circ}\text{C}$ at a heating rate of $5\text{ }^{\circ}\text{C min}^{-1}$. Thermogravimetric analysis (TGA) was used to evaluate the thermal stability and mass changes of the coatings. The thermal effect temperatures and corresponding mass changes were determined from the recorded TGA curves using the instrument software.

RESULTS AND DISCUSSION

The selected spraying parameters ensured stable combustion, uniform deposition, and the formation of high-quality $\text{Al}_{65}\text{Cu}_{20}\text{Fe}_{15}$ coatings on an AISI 321 stainless steel substrate. The optimized particle size and spray distance ensured sufficient particle heating and kinetic energy, resulting in a homogeneous coating with low porosity.

The low porosity indicates effective particle deformation and limited oxide formation, which is characteristic of the optimized HVOF process. Prior to corrosion testing, a clearly defined coating/substrate interface was observed; the coating thickness was $43.97 \pm 3.0\text{ }\mu\text{m}$, $101.89 \pm 13.5\text{ }\mu\text{m}$, for samples (a), and (b), respectively.

After corrosion testing, all coatings exhibited significant degradation, leaving only a thin residual layer on the substrate. The residual coating thickness was $13.05 \pm 1.65 \mu\text{m}$ for sample (a), $31.5 \pm 3.5 \mu\text{m}$ for sample (b). Among the coatings studied, sample (b) retained the greatest residual thickness, indicating the highest corrosion resistance and structural integrity. This behavior is consistent with its lower porosity and more uniform microstructure, obtained at an oxygen pressure of 3.5 bar (Fig. 1).

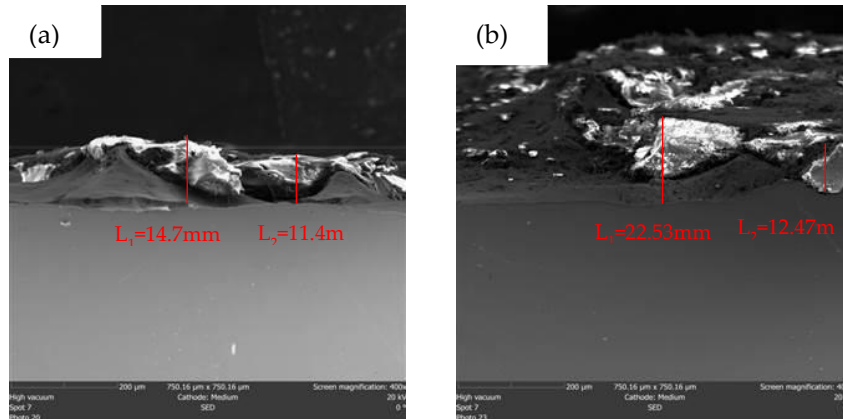


Figure 1. Micrographs of the thickness of Al-Cu-Fe coatings after corrosion: samples (a-b)

XRD analysis showed that the phase composition of the HVOF-sprayed Al-Cu-Fe coatings strongly depends on the oxygen pressure used during deposition. The diffraction patterns revealed the presence of Al, $\text{Fe}_3\text{Al}_{13}$, FeAl , Fe_3O_4 , CuFe_2O_4 , Cu_2O , and CuO , indicating the formation of intermetallic and oxide phases characteristic of thermally sprayed Al-Cu-Fe coatings (Fig. 2).

The diffraction peak intensity and sharpness varied with oxygen pressure, reflecting changes in crystallinity, phase ordering, and oxidation during spraying. These results indicate that oxygen pressure significantly influences phase formation and the structural stability of the coatings during the HVOF process.

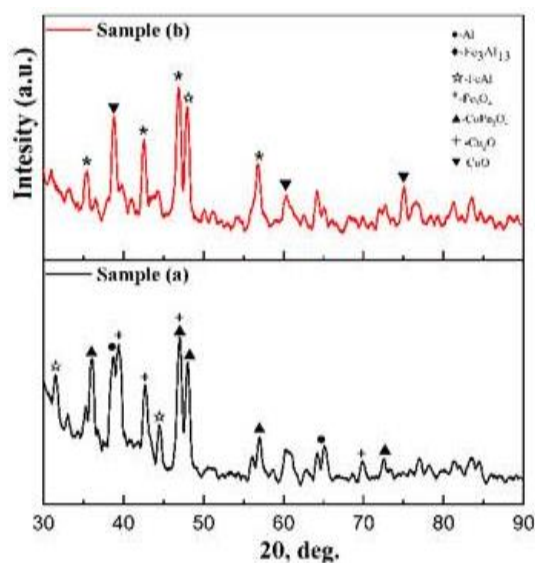


Figure 2. X-ray diffraction analysis

As shown in Figure 3, exposure to 3.5 wt.% NaCl solution resulted in localized corrosion characterized by micro-pitting, inter-splat degradation, and heterogeneous surface dissolution. Corrosion preferentially initiated at coating defects such as pores, splat boundaries, and compositionally inhomogeneous regions.

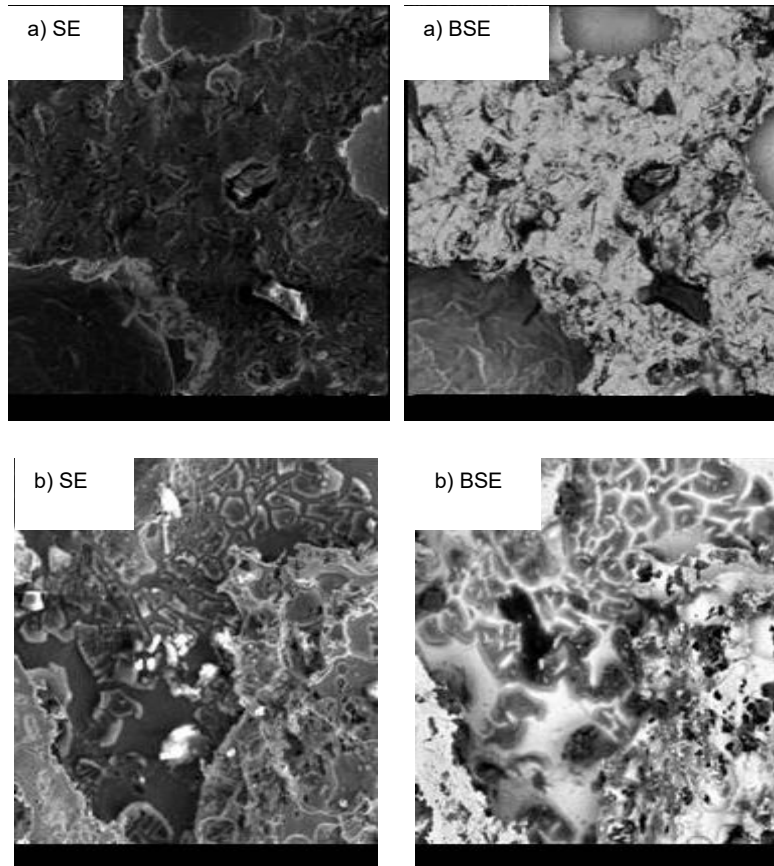


Figure 3. SEM images of the samples after corrosion in a 3.5% NaCl solution at room temperature

EDS analysis conducted after electrochemical corrosion tests revealed significant differences in the surface composition of the coatings studied. Among all the samples, sample (b) demonstrated the best corrosion resistance and the most stable surface condition after exposure to the aggressive environment (Fig. 4).

Similarly, sample (a) exhibited the highest iron concentration (48.2 wt.% / 22.1 at.%), indicating the most severe degradation among the coatings studied. The chromium and nickel contents remained relatively high, while the oxygen concentration reached 3.1 wt.% / 4.9 at%. Trace amounts of aluminum (0.8 wt.% / 0.7 at%), titanium (0.2 wt.% / 0.1 at%), and chlorine (0.9 wt.% / 0.6 at%). The presence of chlorine confirms the penetration of chloride-containing electrolyte into the coating structure, which accelerated localized corrosion processes.

For sample (b), the EDS spectrum showed a high carbon content (71.0 wt.% / 78.9 at%) along with a moderate oxygen concentration (21.0 wt.% / 17.5 at%). The aluminum content remained detectable (6.6 wt.% / 3.3 at%), while the iron concentration was very low (1.3 wt.% / 0.3 at%). The copper content was below the EDS detection limit. The preservation of aluminum, along with the limited amount of corrosion products, is consistent with the improved corrosion characteristics of sample (b).

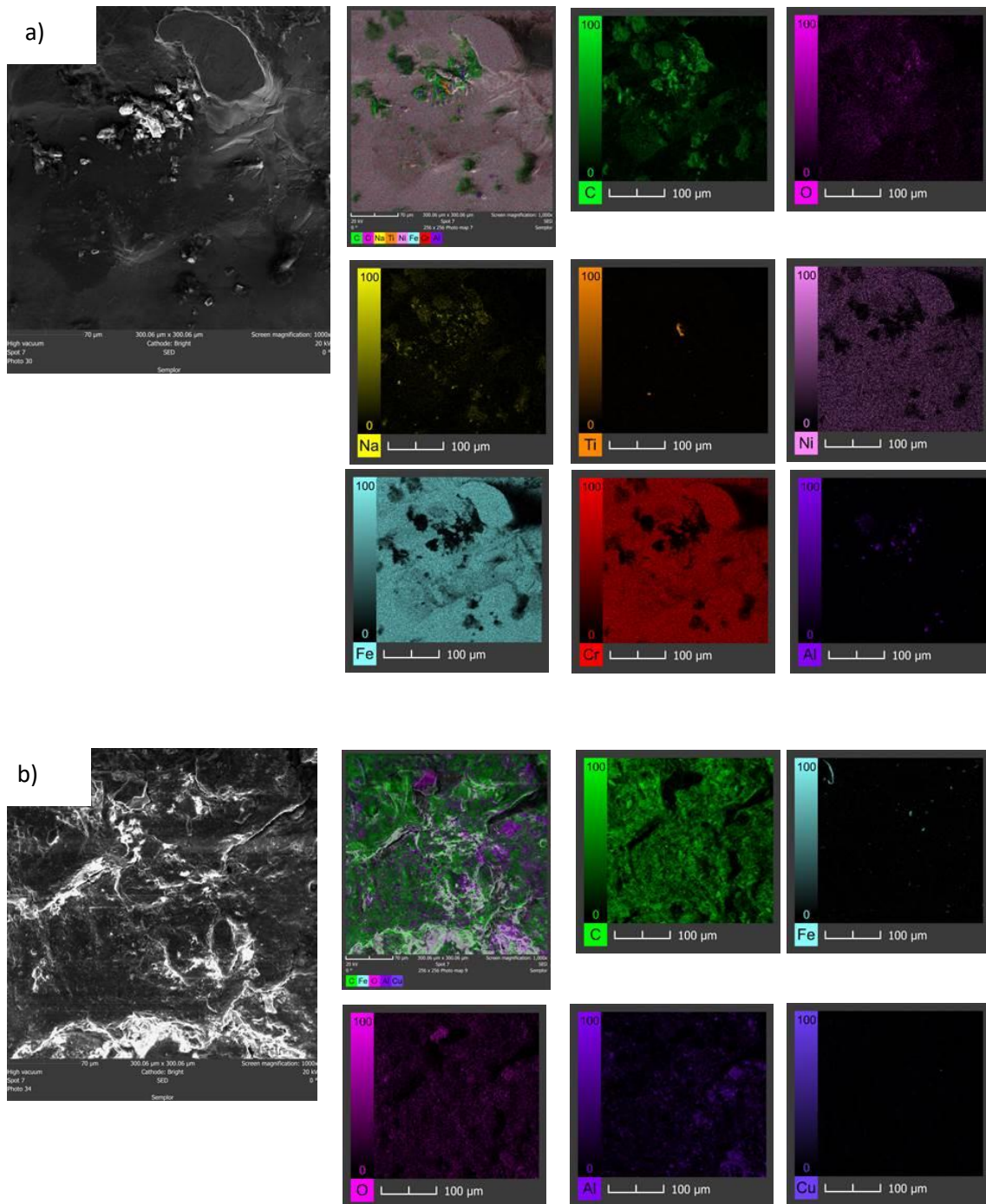


Figure 4. EDS Analysis after Corrosion Testing

The porosity of the coatings was determined prior to corrosion testing using SEM micrographs and ImageJ software. The measured porosity values were 8.61% and 5.37% for samples (a) and (b), respectively (Fig. 5). The coating applied at an oxygen pressure of 3.5 bar exhibited the lowest porosity, indicating a denser and more uniform microstructure. The higher porosity observed for sample (a) may reduce its barrier properties and corrosion resistance, confirming that an oxygen pressure of 3.5 bar provides favorable conditions for high-speed gas-flame spraying to produce dense Al-Cu-Fe quasi-crystalline coatings.

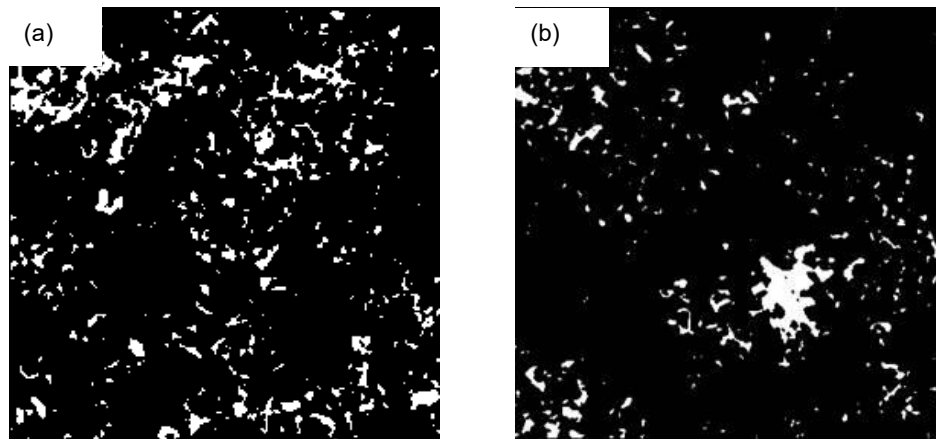


Figure 5. Porosity analysis of HVOF-sprayed Al–Cu–Fe quasi-crystalline coatings using ImageJ software based on SEM surface micrographs: (a) sample (a); (b) sample (b). White regions indicate pores identified during image processing

Thermal treatment of Al–Cu–Fe coatings deposited by HVOF spraying at 550–600 °C was reported by Wang Peng et al. (2023) to increase the proportion of the quasi-crystalline I phase, eliminate the β phase, increase hardness to 674 HV, and improve corrosion resistance in 3.5% NaCl. Pore sealing further enhanced the protective properties of the coatings [Feitosa, F. R. P., et al (2018)]. These findings indicate that the oxygen pressure during HVOF spraying can affect the coating microstructure and phase composition. In the present study, the coating produced at an oxygen pressure of 3.5 bar exhibited a favorable combination of coating density and phase composition compared with the sample prepared at 3.0 bar.

Ryabtsev et al. (2019, 2020) demonstrated that the addition of 0.5 at.% Sc improves the corrosion resistance of Al–Cu–Fe ion-plasma films in NaCl solutions. They also reported that, in addition to the icosahedral I phase, cast Al–Cu–Fe alloys may contain λ -Al₁₃Fe₄, β -AlFe(Cu), τ -AlCu(Fe), and θ -Al₂Cu phases, whereas nanostructured films exhibit high corrosion resistance associated with their ultrafine grain size (~3 nm).

The corrosion rate, determined from mass loss during electrochemical testing in 3.5% NaCl solution over 300 s, was $2.03 \cdot 10^{-4} \text{ g s}^{-1}$ for the uncoated substrate and $4.0 \cdot 10^{-5} \text{ g s}^{-1}$ and $1.13 \cdot 10^{-4} \text{ g s}^{-1}$ for samples (a) and (b), respectively. These results confirm the protective effect of the Al–Cu–Fe coatings, with sample (a) exhibiting the lower corrosion rate.

According to Wehner, B. I., & Köster, U. (2000), high-temperature oxidation of Al–Cu–Fe alloys is accompanied by the formation of an oxide layer and follows linear or parabolic oxidation kinetics. Teplov et al. (2021 and 2023) reported that the thermal stability of polymer composites containing Al–Cu–Fe strongly depends on the surrounding atmosphere, whereas metallic quasi-crystalline systems remain stable over a considerably wider temperature range. A similar behavior was observed in the present study.

TGA analysis revealed differences in the thermal behavior of the studied coatings (Figure 6). Sample (a) exhibited the highest thermal stability, with two stages of relatively minor mass loss of approximately 2.67% and 2.93%, followed by a nearly stable mass up to 900 °C. Sample (b) showed slightly lower thermal stability, with an initial mass loss of approximately 2.33% below 350 °C, followed by a gradual mass loss of 3.27% up to 900 °C. Nevertheless, both coatings demonstrated relatively stable behavior at elevated temperatures, indicating their resistance to significant thermal degradation under the applied testing conditions.

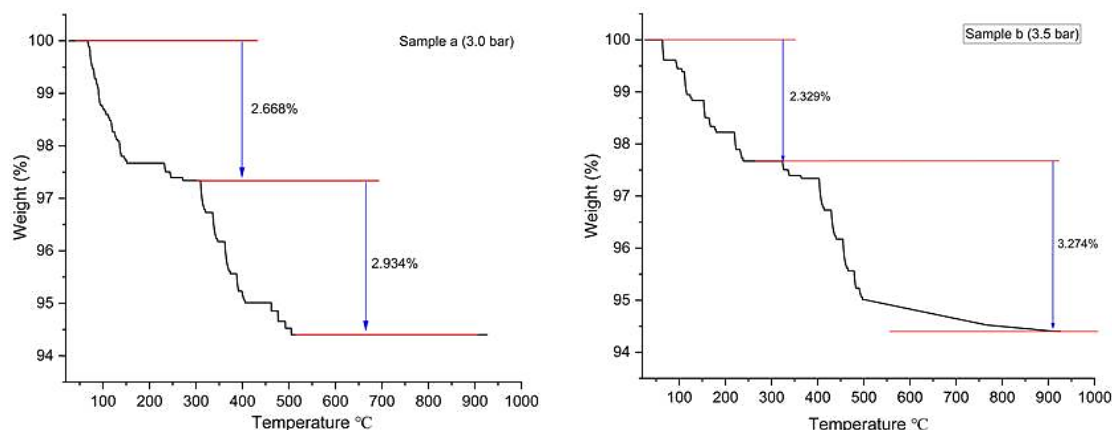


Figure 6. TGA curves for samples: (a); (b)

Overall, based on the TGA results, the thermal stability of the coatings decreased in the following order: Sample (a) > Sample (b). These results indicate that Sample (a) exhibited the highest resistance to thermally induced mass loss and compositional changes under the applied testing conditions.

CONCLUSIONS

Al-Cu-Fe quasi-crystalline coatings were successfully deposited on AISI 321 stainless steel by the HVOF process using different oxygen pressures. The effect of oxygen pressure on the phase composition, microstructure, porosity, corrosion behavior, thermal stability, and microhardness was investigated.

XRD analysis confirmed the formation of quasi-crystalline-related intermetallic structures along with Al-, Fe-, and Cu-containing phases. Oxygen pressure affected the phase composition and coating characteristics. The measured porosity values were 8.61% and 5.37% for coatings deposited at 3.0 and 3.5 bar, respectively.

Corrosion tests in 3.5 wt.% NaCl demonstrated that degradation preferentially initiated at pores, splat boundaries, and phase interfaces. SEM observations further showed that the oxygen pressure influenced the corrosion behavior and structural integrity of the coatings after exposure to the chloride-containing environment.

Overall, increasing the oxygen pressure from 3.0 to 3.5 bar resulted in reduced porosity and improved coating characteristics, including corrosion resistance, thermal stability, and mechanical properties. These results demonstrate the significant influence of oxygen pressure on the performance of Al-Cu-Fe quasi-crystalline coatings for protective applications in chloride-containing environments.

CONFLICT OF INTEREST: The authors declare no conflict of interest.

FUNDING: This research is funded by the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP26103943).

INSTITUTIONAL REVIEW BOARD STATEMENT: *Not applicable.*

INFORMED CONSENT STATEMENT: *Not applicable.*

DATA AVAILABILITY STATEMENT: *The data supporting the findings of this technical study are available from the corresponding author upon reasonable request; alternatively, no new experimental data were generated as the work was based on numerical modeling and theoretical analysis*

ACKNOWLEDGEMENTS: none

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE TECHNOLOGIES: The authors declare that no artificial intelligence (AI) tools were used in the preparation of this article.

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