

**MICROSTRUCTURE AND WEAR OF NI-AL₂O₃ COLD SPRAYED
COATING**

BY

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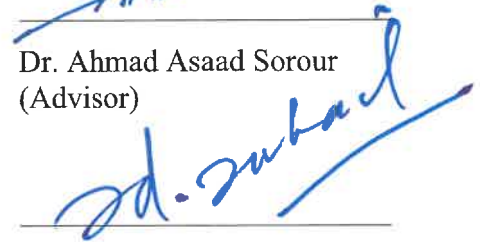


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2024

I dedicate this thesis wholeheartedly to my mother, father, wife, and entire family, whose
steadfast support has been my pillar through every circumstance.

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LIST OF ABBREVIATIONS

CS	Cold Spray
MMC	Metal Metrics Composite
KSS	Kinetic Spray Solution
LPCS	Low Pressure Cold Spray
HPCS	High Pressure Cold Spray
AM	Additive Manufacturing
SD	Stand-off Distance
304SS	304 Stainless Steel
SEM	Scanning Electron Microscopy
EDS	Energy-Dispersive X-ray Spectroscopy
OM	Optical Microscopy
COF	Coefficient of Friction

ABSTRACT

Full Name : [Raihan Abdulkarim Goriya]
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This study presents the fabrication of Ni-Al₂O₃ cold spray coatings on a 304SS substrate using a low-pressure cold spray technique. By employing the computational tool KSS for process parameter selection, extensive experimental trials were minimized. The investigation explored the effects of gas temperature (25°C to 600°C) and nozzle traverse speed (0.6 to 3.6 m/minutes) on microstructure, resulting in a uniform coating thickness with an average value of 130 µm under optimized conditions (400°C and 0.6 MPa). The utilization of Ni-Al₂O₃ cold spray coating via the low-pressure cold spray technique offers a promising alternative to hard chrome plating, providing superior wear resistance and environmental benefits. Incorporating Al₂O₃ ceramic particles into the nickel matrix significantly enhances the wear resistance of the coating, while the spraying process emits no toxic or hazardous gases, addressing environmental concerns. Nickel and high-entropy alloys have emerged as favored options for tribology applications due to their ability to form intermediate layers, such as tribolayers or semi-lubrication layers, which mitigate wear by reducing contact between sliding materials. This study investigated how Al₂O₃ concentration influences the wear properties of Ni-Al₂O₃ cold spray coatings through microstructural analysis and computational optimization of process parameters. Additionally, the study explored the mechanical properties of the coating and assessed the

impact of alumina concentration and the number of passes on coating performance, with a focus on wear properties and coating quality.

Keywords: Cold spray coating, Microstructure, Metal matrix composite, Process parameters

ملخص الرسالة

الاسم الكامل: ریحان عبدالکريم غورية

عنوان الرسالة: $Ni-Al_2O_3$ البنية الدقيقة وتآكل الطلاء بالرذاذ البارد

التخصص: مهندس ميكانيكى

تاريخ الدرجة العلمية: يونيو 2024

باستخدام تقنية الرش SS على ركيزة $Ni-Al_2O_3$ 304 تعرض هذه الدراسة تصنيع طلاءات الرش البارد لاختيار معلمة العملية، تم التقليل KSS البارد منخفض الضغط. من خلال استخدام الأداة الحسابية من التجارب التجريبية واسعة النطاق. استكشف البحث تأثيرات درجة حرارة الغاز (25 درجة مئوية إلى درجة مئوية) وسرعة اجتياز الفوهة (0.6 إلى 3.6 م/دقيقة) على البنية المجهرية، مما أدى إلى 600 سمك طلاء موحد بمتوسط قيمة 130 ميكرومتر في ظل الظروف المثلى (400 درجة مئوية و 0.6 ميغا عبر تقنية الرش البارد منخفض الضغط يوفر بديلاً $Ni-Al_2O_3$ باسكال). إن استخدام طلاء الرش البارد واعدًا لطلاء الكروم الصلب، مما يوفر مقاومة فائقة للتآكل وفوائد بيئية. يؤدي دمج جزيئات السيراميك في مصفوفة النيكل إلى تعزيز مقاومة التآكل للطلاء بشكل كبير، بينما لا تصدر عملية الرش أي Al_2O_3 غازات سامة أو خطرة، مما يعالج المخاوف البيئية. ظهرت سبائك النيكل والسبائك ذات الإنتروبيا العالية كخيارات مفضلة لتطبيقات علم الاحتكاك نظرًا لقدرتها على تكوين طبقات وسيطة، مثل طبقات ثلاثية أو طبقات شبه تشحيم، والتي تخفف من التآكل عن طريق تقليل الاتصال بين المواد المنزلقة. $Ni-Al_2O_3$ على خصائص التآكل لطلاءات الرش البارد Al_2O_3 بحثت هذه الدراسة في كيفية تأثير تركيز من خلال التحليل المجهرى والتحسين الحسابي لمعلمات العملية. بالإضافة إلى ذلك، قامت الدراسة، باستكشاف الخواص الميكانيكية للطلاء وتقييم تأثير تركيز الألومينا وعدد التمريرات على أداء الطلاء، مع التركيز على خصائص التآكل وجودة الطلاء.

CHAPTER 1

INTRODUCTION

Wear has emerged as a critical issue in modern industry, significantly impacting maintenance costs, causing downtime, and affecting machine efficiency and lifespan. To mitigate the wear problem, we have two main options: developing new materials or applying surface treatments to existing materials to enhance their durability and lifespan. The latter option is more practical, quick, and economical. By applying suitable surface coatings, we can significantly improve the wear resistance of these components, thereby extending their operational life and reducing maintenance costs. This problem is particularly pronounced in the context of landing gear applications as shown in Figure 1, where parts are exposed to harsh environments containing moisture, dirt, water, and other elements. For instance, the Boeing nose wheel steering actuator barrel is prone to corrosion and wear when exposed to these harsh conditions. However, being made from stainless steel, which offers high corrosion resistance, this material falls short in wear resistance, due to direct contact with the landing gear [1].

To effectively address this critical issue, surface modification of the material through cladding or coating with a suitable overlaying material offers a cost-effective and straightforward solution. Several coating techniques can be utilized, including Flame Spray, Arc Spray, High-Velocity Oxy-Fuel (HVOF), and Detonation Spray. Notably, a

new and emerging technique, Cold Spray Coating, is gaining significant attention. This method, a type of additive manufacturing similar to 3D printing, does not affect the substrate during the coating process, making it an attractive solution for enhancing the durability and performance of critical components such as the nose wheel steering actuator barrel.

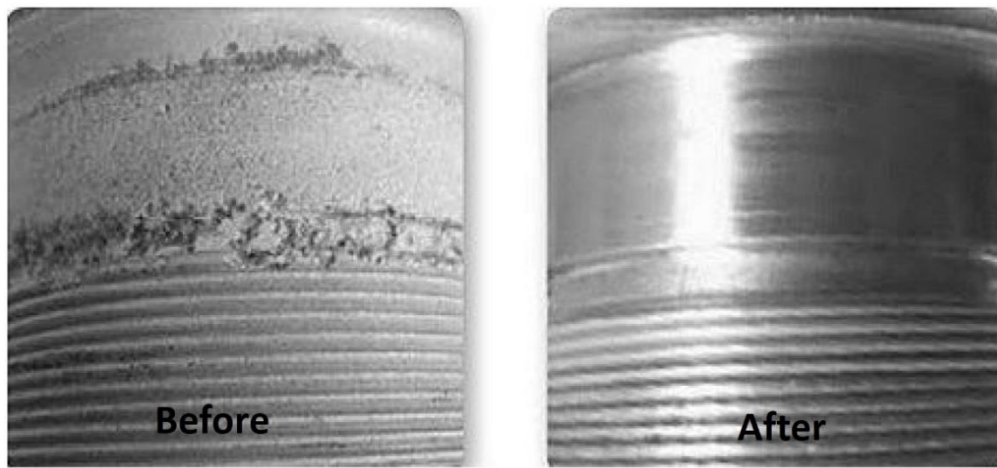


Figure 1 Boeing nose wheel steering actuator barrel: Before and After Coating [1]

In aerospace applications, particularly landing gear systems, there is a pressing need for advanced coatings that offer exceptional wear resistance, oxidation resistance, and specialized functionality. To address these challenges, there is a growing demand for Ni-Alumina ($\text{Ni-Al}_2\text{O}_3$) cold spray coatings in aerospace applications. These coatings have shown promising results in significantly enhancing surface micro-hardness, wear resistance, and yield stress of various metals and alloys. Unlike pure ceramic coatings, Ni- Al_2O_3 cold spray coatings offer superior performance and endurance, partly attributed to the reduced development of residual stress at the interface [2]. Thus, the development and implementation of Ni-Alumina cold spray coatings represent a crucial solution to

improving the efficiency, reliability, and longevity of components in aerospace applications, ensuring optimal performance under demanding operating conditions.

Ongoing development is centered on exploring the wear and corrosion resistance properties of cold-sprayed coatings. The influence of diverse matrix composite coatings on steel has yet to be thoroughly examined. This research aims to investigate the microstructure and wear resistance of Ni-Al₂O₃ cold spray coatings on steel substrates using various process parameters. The study will examine the effects of matrix material on coating hardness and wear resistance.

Outline

Chapter 1 introduces the research context and rationale, outlining the structure of the dissertation. Chapter 2 provides a comprehensive background and literature review on the processes, specifically focusing on the Ni-Al₂O₃ cold spray coating on 304 Stainless steel substrate and coating characterization. Chapter 3 establishes the primary objective, while Chapter 4 elaborates on the overall approach and methodology employed to achieve the research goals. Chapter 5 presents the results and discussion, and Chapter 6 encapsulates the conclusion, contribution to original scientific knowledge, and future recommendations.

CHAPTER 2

BACKGROUND AND LITERATURE REVIEW

This chapter introduces cold spray coating, coating parameters, and their impact on the properties of cold-sprayed samples. It further highlights the ability of cold-sprayed materials to be re-clad and their versatility in various applications.

2.1 Cold Spray Coating

Cold spray (CS) is a process in which solid powder particles (1 to 50 μm) are accelerated in a de Laval nozzle toward a substrate. If the impact velocity exceeds a threshold value, particles undergo plastic deformation and adhere to the surface. The origins of cold spray technology can be traced back to its development in Russia in the early 1980s. Cold spray deposition involves the deposition of a spray of particles onto a surface through plastic deformation induced by a supersonic gas jet without the need for melting any materials. The low process temperature in cold spray reduces thermal stress and substrate deformation [3]. Figure 2 displays a graph illustrating gas temperatures and particle velocities across various thermal spray methods. Over the past few decades, kinetic energy has been increasingly used instead of thermal energy in thermal spraying techniques. This shift has resulted in new advantages, such as the capability to produce uniform and dense coatings, since the cold spray process involves minimal oxidation. Furthermore, the low heat input during spraying preserves the substrate's characteristics and inhibits oxidation. An added benefit of utilizing the cold spray approach is the avoidance of phase transitions caused by

melting and developing of porosity resulting from solidification in other processes. [6]. This technique is especially advantageous for materials sensitive to heat [4].

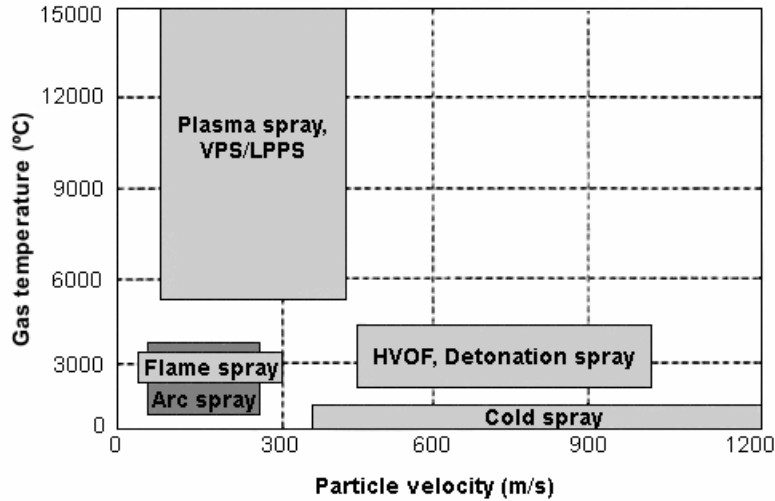


Figure 2 Particle velocity versus Gas temperature in the thermal spray processes [4]

Metallurgical bonding and/or mechanical interlocking occur as a result of the solid-state effect on the metallic substrate, which is essential for producing coatings. Powder particles are injected into the gas flow and then accelerated and sprayed onto the substrate through impact and drag. The speed of the powder particles plays a crucial role in determining the coating formation during cold spray. Each material has a unique critical velocity, represented by v_{cr} . At speeds below v_{cr} , only erosion and particle rebound occur without any coating formation, while speeds above v_{cr} cause the particles to stick to the substrate, leading to plastic deformation and coating development [13–17].

Compared to other thermal spraying methods, cold spraying is a relatively new technique with many advantages, including high deposition efficiency, minimal substrate heating, low residual stresses, phase stability, and compositional stability [10].

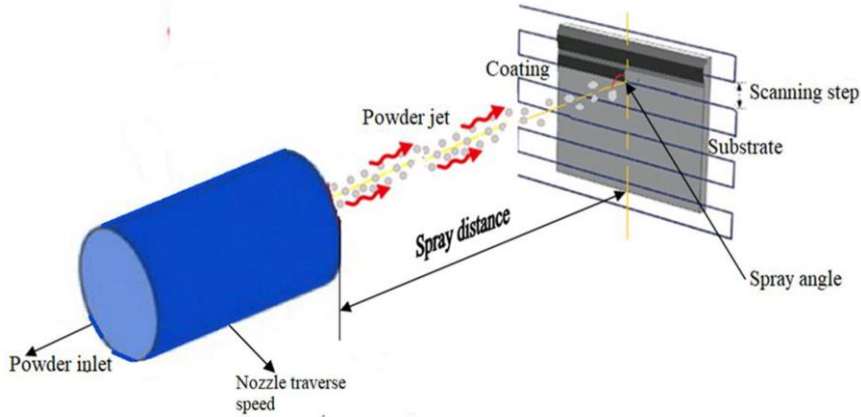


Figure 3 Schematic diagram outlining the process parameters involved in cold spray coatings [11]

To achieve high-quality coatings using cold spray technology, it is necessary to analyze the process parameters systematically. Robot-assisted spraying plays a vital role in this process by enabling precise control and manipulation of various kinematic parameters, including spraying trajectory, relative speed, stand-off distance (SD), nozzle angle, and scanning step.

In addition, to ensure the production of high-quality coatings, it is essential to monitor the coordination between the powder feeder, propulsive gas, and spraying nozzle. Although this can be challenging, understanding the relationships between process parameters and the resulting properties of the as-sprayed CS coatings, as shown in Figure 3, can be helpful. Considering these factors, an optimal CS fabrication strategy can be developed to achieve high-quality results [11].

Cold spray coating has been widely explored for various applications, presenting an advanced spray technology akin to 3D printing. It offers numerous advantages, including the capability to deposit thick coatings with high density, low oxygen content,

and superior mechanical properties on surfaces. Previous research has demonstrated its efficacy in repairing structures, enhancing asset sustainability, and refurbishing high-value products [12]. For instance, a study conducted by Wang et al. [13] utilized 3D X-ray Microtomography to characterize the porosity of cold-sprayed stainless steel coatings, revealing a low level of micro-sized and non-connected porosity. Omar et al. [14] examined the effect of Cold Sprayed Aluminum Coating on the corrosion behavior of Sponson Spar. Salt spray testing showed no rust on the coated area, supported by EDX results indicating a higher weight percentage of aluminum. The findings highlight corrosion mitigation as a crucial concern in the aircraft industry. Cavaliere et al. [15] employed cold spray for crack repair in Aerospace Aluminum Alloy Panel, demonstrating a more than sevenfold increase in crack resistance through the use of AA2198 for repair, contributing to the enhanced global fatigue life of cracked structures.

Benenati et al. [16] aimed to develop a Cold Spray deposition strategy for Additive Manufacturing, targeting residual stress reduction. It employed microscale ANSYS-AUTODYN simulations to analyze residual stress formation and macroscale static structural simulations based on Tsui and Clyne's model to optimize layer interactions for an effective deposition strategy. In one study a novel parametric investigation was conducted to analyze the residual stress formation in cold-sprayed deposited particles. It was found that impact velocity, the incident angle of impact, density, and yield stress of the materials involved significantly influence residual stress. Additionally, W.Y. Li et al. [17] investigated the impact of cold spraying on the microstructure and mechanical properties of optimized friction stir welded AA2024-T3 joints, revealing a maximum tensile strength of 420 MPa at a rotation speed of 600 rpm. Subsequently, an aluminum

coating was cold sprayed onto the optimized joint surface. Microstructural changes near the coated joint's surface were examined using transmission electron microscopy, electron back-scattered diffraction, and differential scanning calorimetry. The findings revealed a notable improvement in tensile strength, microhardness, and fatigue life of the cold-sprayed joint. Additionally, B. Aldwell et al. [18] assessed machinability of Al 6061 treated with cold gas dynamic sprayed additive manufacturing, examining parameters' influence on cutting forces, chip formation, and surface characteristics. Findings emphasized the significant impact of cold spray parameters on machinability, highlighting the need for tailored machining strategies. Q. Blochet et al. [19] investigated the effect of cold-sprayed aluminum coating-substrate interface morphology on bond strength in aircraft repair. It found that craters on the substrate surface significantly enhanced particle anchoring, supported by numerical simulations showing increased particle deformation on grit-blasted surfaces.

2.1.1 Key features of Cold Spray Coating

The key features of cold spray coating are shown in Figure 4. In this coating material deposition occurs in a solid state, distinguishing it from traditional thermal spray methods. This unique process offers several advantages [20]:

Cold spray coating operates at significantly lower temperatures compared to traditional thermal spray methods. Because the particles remain in a solid state throughout the process, there is minimal thermal exposure to the substrate and the feedstock material. This prevents the high-temperature-induced degradation of material properties, making cold spray particularly suitable for temperature-sensitive applications [21].

The cold spray process produces coatings with exceptionally low porosity. The high-velocity impact of particles onto the substrate results in a dense, well-bonded coating structure. Additionally, the absence of melting and rapid solidification reduces residual stress within the coating, enhancing the mechanical integrity and durability of the coated components [22]. Since the cold spray process is carried out at low temperatures, there is minimal oxidation of the feedstock material during deposition. This preserves the original properties of the materials and results in coatings that are free from unwanted oxide inclusions, which can be detrimental to the coating's performance in many applications .

The solid-state nature of the cold spray process, combined with the retention of the feedstock material's microstructure, results in coatings with outstanding mechanical, electrical, and thermal properties. These properties make cold spray coatings suitable for a wide range of applications, including wear-resistant surfaces, conductive layers, and thermal barriers.

One of the key benefits of the cold spray process is its ability to maintain the feedstock's original microstructure. The low-temperature deposition minimizes changes to the material's phase and grain structure, preserving its inherent properties. This is particularly beneficial for materials with complex or delicate microstructures that are essential for specific applications.

In conclusion, the cold spray coating process offers numerous advantages over traditional thermal spray methods. Its low-temperature, solid-state deposition results in coatings with low porosity and residual stress, minimal oxidation, and properties similar to those of the bulk material. These features make cold spray an attractive option for

applications requiring high-performance coatings with excellent mechanical, electrical, and thermal properties while maintaining the integrity of the feedstock material.

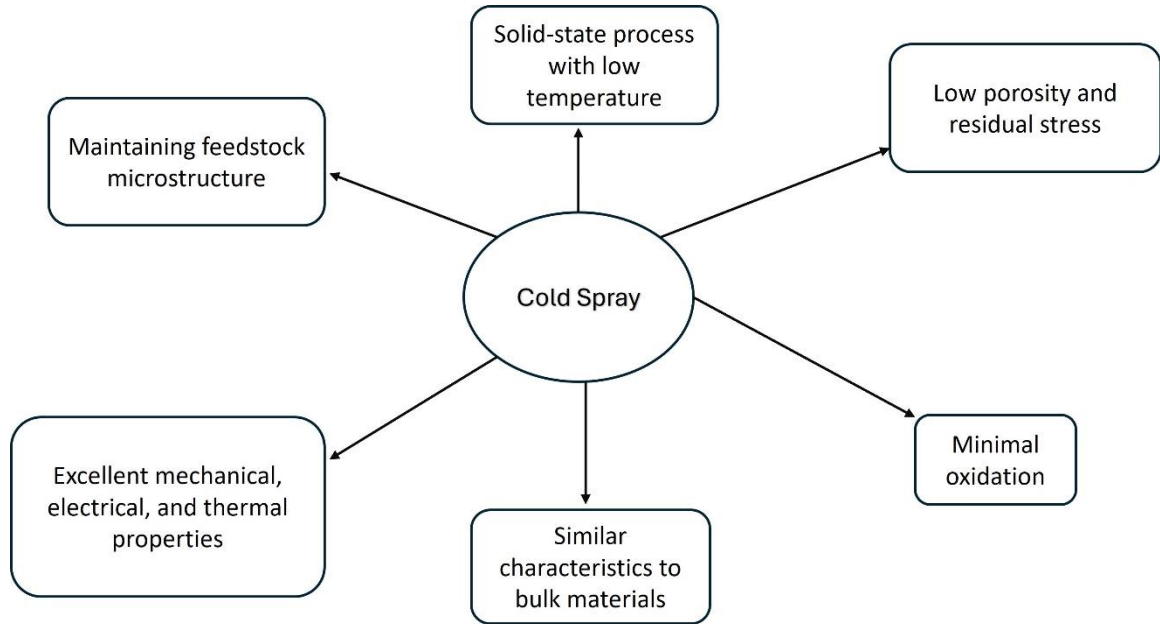


Figure 4 Key Features of Cold Spray Coating compared to other thermal spray coating

2.1.2 Limitations of Cold Spray Coating

Cold spray predominantly deposits composites or metals, ensuring ductility at low temperatures, with materials like Cu, Ti, Al, and ceramics like WC–12Co, Al–Al₂O₃, and Al–12Si alloy. While researchers explore expanding material ranges for cold spray, challenges persist in depositing certain exotic materials like SiC on Ni–Cr-based super alloys and Al–Ni intermetallic compounds, especially using alternative thermal procedures [23].

A robust substrate material ensures effective plastic deformation of incoming particles, fostering a strong bond [24]. Soft substrates, such as polyethylene, allow particles to pass without build-up, while brittle substrates, like carbon, experience erosion. To facilitate coating acceptance, substrates should be supportive and resilient. Cold spray processes demand high gas consumption, primarily employing nitrogen, helium, or air for propelling particles, with helium favored for its inertness and high velocity despite its cost and scarcity, leading to recycling initiatives. Research aims to develop cold spray methods compatible with air or nitrogen to reduce complexity, with nitrogen-operated systems reaching temperatures up to 1000°C. Careful assessment of operational and technical needs is crucial due to the significant gas consumption and associated costs when selecting a cold spray system.

Cold spray exhibits similarities with various thermal spray processes but differs significantly from others like physical and chemical vapor deposition and electroplating, making it a "line-of-sight" method [25]. The short standoff distance in cold spraying, typically only 10mm, poses challenges for spraying inner surfaces like internal pipe surfaces without altering the nozzle design, unlike traditional thermal sprays. This allows for easier design of nozzle assemblies to fit into cavities, making spray tasks feasible. Currently, commercial cold spray equipment manufacturers offer 90° nozzle assemblies capable of spraying pipes with up to a 90mm diameter.

2.1.3 Low Pressure Cold Spray Coating

In low-pressure cold spray, the accelerating gas—typically air or nitrogen—is preheated (up to 550°C) and pressurized (5–10 bar) to optimize its aerodynamic properties. This gas is then forced through a 'De Laval' or similar converging/diverging nozzle. On the

diverging side of the nozzle, the heated gas is accelerated to a speed range of approximately 300–600 m/s. While high-pressure cold spraying (HPCS) offers versatility across metal types, low-pressure cold spraying (LPCS) is primarily suited for easily deformable materials like tin, zinc, copper, aluminum, and nickel. However, through powder modifications such as adding ceramic to metal or mixing multiple metal powders, LPCS capabilities can be expanded beyond its inherent limitations.

It is important to note that Low pressure cold spray (CS) was originally developed for applying ductile metal powders, necessitating plastic deformation upon particle impact [26]. Consequently, metal powders and various metal-ceramic blends were predominantly used [27]. Ceramic powders serve to enhance coating mechanical and physical properties by facilitating peening and additional work hardening, leading to densification, reinforcement, and grain refinement [28]. This results in composite coating microstructures featuring a highly deformed matrix and non-deformed reinforcement, achieved through mechanical crushing and interlocking of ceramic particles within a soft matrix. Ceramic powder in the deposition process serves multiple roles: (i) preventing nozzle clogging in spray gun models lacking water cooling, (ii) activating surfaces by eliminating oxide layers and contamination, and (iii) enhancing surface roughness to augment contact area. However, using pure ceramic powder in cold spray typically necessitates a "soft" matrix. Recent research indicates that proper preparation of ceramic powders is crucial for successful deposition, with plastic deformation mechanisms possible in ceramics, particularly with fine particles [29]. Nanosized ceramic powder velocity can be increased by particle agglomeration, facilitated by powder production methods such as spray drying

or sol-gel, which leave behind organic additives acting as binders. These fragmented, agglomerated particles then bond to the substrate [30].

2.1.4 Parameters Affect the Cold Spray Coating

Various parameters impact the coating characteristics during the cold spraying technique, including particle impact velocity, gas temperature, gas pressure, powder feed rate, stand-off distance and nozzle traverse speed, spray angle, particle and substrate temperature, and surface preparation.

Particle velocity is a key factor in cold spray coating, as only particles with sufficient velocity can form a coating upon deposition. The gas pressure and temperature used to propel the particles affect their velocity, but high values should be avoided. Research has demonstrated that increasing gas pressure by a factor of two (from 1.5 to 3 MPa) can raise particle velocity by 15% while doubling the gas temperature (from 300 to 600 K) can increase it by 25%. However, at extremely high temperatures, the increase in particle velocity is less significant due to a decrease in gas density [31].

Gas temperature can significantly impact the critical velocity of powder particles. An increase in temperature can alter the material properties of the particles, potentially reducing their impact energy upon collision with the substrate. Consequently, temperature must be carefully controlled to ensure optimal coating adherence and deposition efficiency while minimizing particle oxidation [32].

Gas pressure plays a crucial role in propelling powder particles to the desired spraying velocity. Low pressure can hinder the particles from reaching this velocity, making it essential to maintain high pressure for effective deposition. Increasing the gas

pressure from 2 MPa to 2.8 MPa has been shown to reduce porosity from 18% to 1.5%. In a study conducted by Stoltenhoff, it was observed that increasing the pressure from 1.5 to 3 MPa resulted in a 15% rise in particle velocity, while the particle temperature remained nearly constant and unaffected by the pressure change. This indicates that higher pressure can significantly enhance coating quality by improving particle velocity without adversely affecting particle temperature [31].

Increasing the powder feed rate from 6.8 g/min to 10.1 g/min is expected to result in a significant improvement in cold spray coating properties. Specifically, the porosity is anticipated to decrease by approximately half, from $1.03 \pm 0.18\%$ to $0.55 \pm 0.08\%$, due to more efficient particle packing and reduced voids. Additionally, the micro hardness of the coating is expected to increase by 20%, driven by denser particle packing and stronger inter-particle bonding. These enhancements in porosity and hardness will lead to more durable and resilient coatings, offering improved performance in applications that require high wear resistance and mechanical strength [33].

The spraying distance or stand-off distance is between the nozzle and the substrate surface. This distance should not exceed 50 mm for cold spray techniques, whereas for other thermal spraying technologies, it can range between 120 and 300 mm [34]. The standoff distance, the gap between the nozzle and substrate, affects the impact velocity of powder particles during cold spray coating. Increasing the standoff distance can result in higher impact velocities due to shock wave attenuation, which is more noticeable at small distances. At these distances, a stronger bow shock reduces deposition efficiency. In contrast, the absence of a bow shock at larger distances allows for unhindered deposition.

However, this effect is only favorable up to a distance of 50 mm, beyond which the shock wave disappears, and the effect vanishes [31].

Nozzle transverse speed is critical in determining the amount of material deposited in a particular area. If the gun transverse speed is slower, more material will be deposited for a given number of passes. On the other hand, a higher gun transverse speed will result in less material being deposited [3].

Spray angle affects the deposition process significantly. When the impact occurs at an oblique angle, the particle velocity can be split into normal and tangential components. Lowering the spray angle reduces the normal component of particle velocity, potentially dropping it below the critical level and preventing deposition. Studies indicate that optimal deposition efficiency is achieved for copper at spray angles of 80 to 90 degrees, while for titanium, angles of 70 to 90 degrees are preferred [35]. Deposition efficiency decreases when the spray angle falls below these ranges. No particle deposition occurs when the spray angle drops below 40 degrees for copper and 50 degrees for titanium [36].

Higher initial temperatures of both the particle and substrate have been found to improve the physical characteristics, tribological-mechanical properties, and deposition efficiency of coatings. This observation has been reported in several studies, such as one by Li et al. in which a higher carrier gas temperature resulted in increased deposition efficiency during the LPCS process of injecting titanium feedstock particles. [36]. Raising the temperature during coating deposition can affect the coating properties and deposition efficiency in various ways. For instance, an increase in process gas velocity due to higher temperature can boost particle impact velocity, and higher gas or preheating temperatures can change material thermal behavior. This can lead to improved thermal softening and

chemical reactions that enhance the bonding and adhesion between the coating and substrate [37].

Enhancing the substrate surface roughness from a polished to a grit-blasted texture can significantly improve the deposition efficiency of metallic powders. This is because the particles impacting a rough surface undergo greater deformation, resulting in enhanced mechanical interlocking between the particles and the substrate. Mechanical interlocking is a widely accepted theory in cold spray coatings and is facilitated by the impact of particles on the substrate surface. Greater substrate roughness provides more crevices and indentations for cold spray particles to lodge, leading to increased bonding. These particles are further compacted by successive impacts on the substrate. Conversely, surfaces with low roughness offer minimal surface area for initial particle impact and binding, resulting in weaker bond strengths and lower deposited mass at the outset [31].

2.2 Ni – Al₂O₃ Cold Spray Coating

Ni-Al₂O₃ composite coatings are highly desirable due to their significant enhancement in surface micro-hardness and wear resistance. The addition of Al₂O₃ particles to a Ni-matrix results in coatings that demonstrate a remarkable increase in wear resistance and yield stress, up to two- and three-fold, respectively [38]. The incorporation of alumina showed increased hardness and wear resistance of the coatings. Raghavendra's study aimed to examine how coating parameters affect the mechanical, wear, and coating properties of Ni-Al₂O₃ composite coatings [39]. Ramesh investigated the impact of Si₃N₄ particles on the Ni matrix coating and found that the Al6061-Ni-P-Si₃N₄ composite had a reduced wear rate and friction coefficient compared to the matrix alloy [40]. Incorporating CeO₂ particles

into the Ni coating resulted in an increase in hardness of approximately 15%, according to Yusuf et al. [41]. Similarly, Gay et al. examined the effect of coating parameters and ZrO₂ particle inclusion in Ni coatings, and observed that the resulting Ni-P-ZrO₂ composite coating exhibited increased hardness, reduced friction coefficient, and good wear resistance. Introducing ZrO₂ particles resulted in notable changes to the crystal structure, surface roughness, thermal characteristics, and corrosion resistance of Ni-based coatings [42]. The bonding strength between nickel particles exceeds that of Ni-Al₂O₃ particles, resulting in a higher proportion of weaker ceramic-metallic bonds in coatings with greater ceramic particle concentrations, although the inclusion of ceramic particles aids in peening and roughening layers during deposition [43].

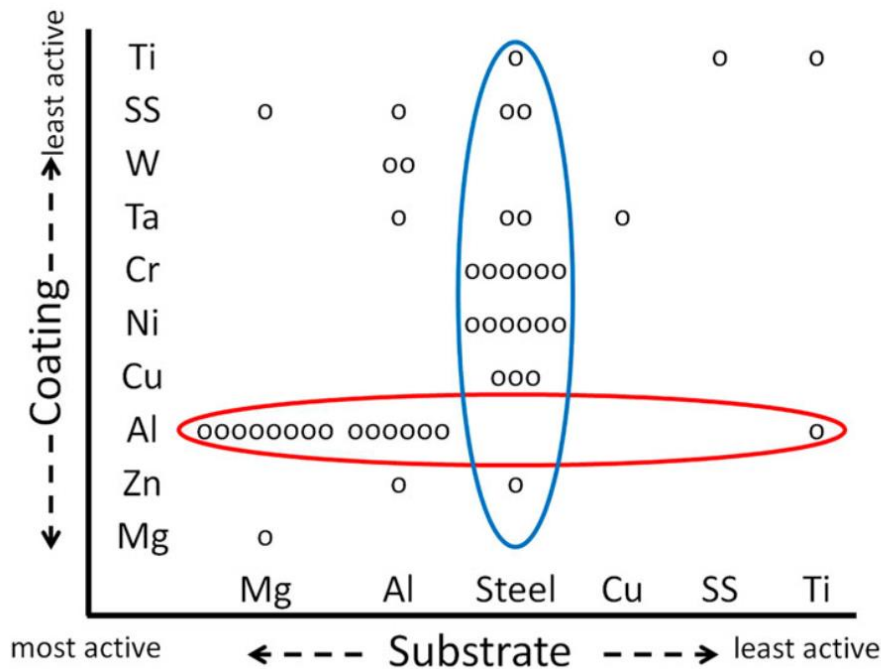


Figure 5 Coating powder and substrate material selection [44]

Nickel displays commendable resistance to corrosion across diverse corrosive environments owing to its exceptional electrical and thermal conductivity properties [44]. Cermets find extensive application in energy sectors because of their exceptional resistance to wear and oxidation. Al_2O_3 and $\text{Al}_2\text{O}_3\text{-TiO}_2$ composites are particularly suitable for gas turbine applications as they generate less residual stress at the interface compared to pure ceramics. Figure 5 shows that the substrate metal alloy or pure metal and the counterparts can be separated based on galvanic potential data. More noble metals, such as Zn metal or alloy powder, can protect SS (stainless steel) as a substrate. Cold spray is employed in the first scenario to add a protective layer, and the steel substrate acts as a sacrifice for protection when the zinc covering corrodes instead Al, Mg, and their alloys are highly active prone to corrosion and therefore require protection. Al and its alloys are often used as coatings due to their ability to produce oxides and form a passivation layer, leading to inhibited kinetics and offering corrosion protection.

2.2.1 Microstructure of the Coated Surface

During cold spray deposition, layers of deformed particles are generated, maintaining the fine microstructure of the feedstock powder produced during atomization. The resulting powder particles adopt an anisotropic pancake shape post-deposition, while still preserving the equiaxed grains and sub-grain structures, as illustrated in Figure 6.

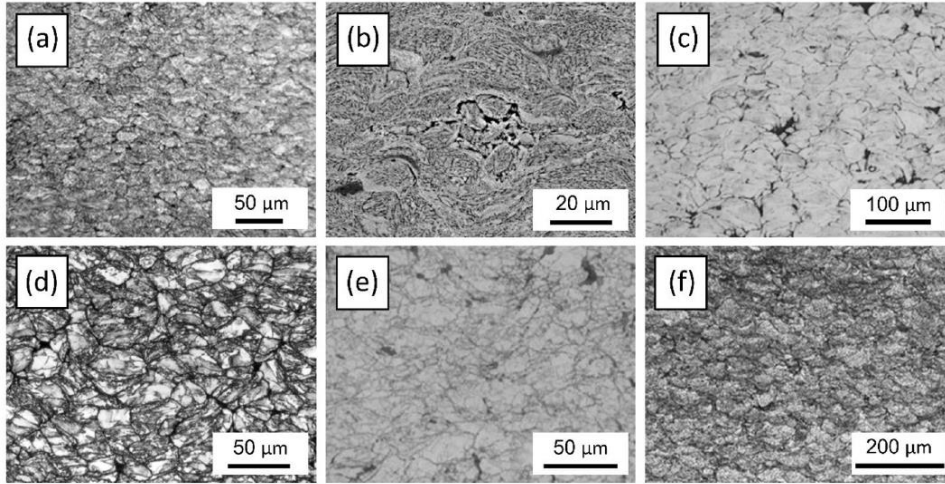


Figure 6 Optical images of cold spray coating [45]

High dislocation density at particle-particle interfaces is the result of cold working and dynamic recrystallization of particles brought on by the high strain rates observed during cold spray deposition. Recrystallization as a result of adiabatic shear instability and high strain rates is depicted in a theoretical model in Figure 7. When a particle is hit, its kinetic energy is transformed into thermal energy. Figure 7 depicts the grain and particle structures of sprayed metals and alloys. The powder particles become anisotropic and take on a pancake-like morphology during deposition, but their equiaxed grains and sub-grain structures remain unchanged [46].

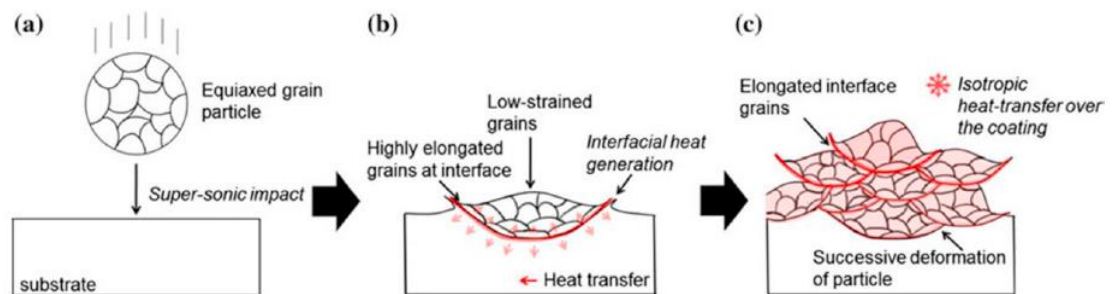


Figure 7 Particle deposition and grain morphology in cold spray coating [47]

Insufficient particle bonding in cold spray materials can lead to the formation of pores, which may act as initiation sites for damage, thereby diminishing the materials' static and cyclic properties. Inadequate handling, storage, or packaging can trap gas bubbles in a material, oxidations and hydroxides can form on the surface of Al powder, and adsorption of water can cause porosity. Increasing the impact energy of sprayed particles has been shown to decrease porosity. Porosity in gas processing is directly affected by nozzle length and gas temperature. Porosity can be reduced by modifying particle velocity and temperature [48].

Figure 8 shown the presence of micro-cracks within alumina ceramic particles and the evident occurrence of micro-pores within the coating. The cracks observed could have originated from either high-energy impacts during the arrival of splats or from the cooling process, potentially caused by a mismatch in the coefficient of expansion between Ni and Al_2O_3 [49]. Kaushal et al. taken the Ni+20% Al_2O_3 clad powder, reported the porosity percentage 0.97% in the microstructure of the cold spray coating and found negligible cracks in alumina at lower temperature [50]. Optimizing particle parameters and achieving critical velocity result in increased deposition efficiency and reduced porosity. Assadi et al. demonstrated that velocities ranging from 550 m/s to 670 m/s for copper particles led to higher deposition efficiency, with 80% of sprayed particles adhering to the substrate with negligible porosity percentage [51].

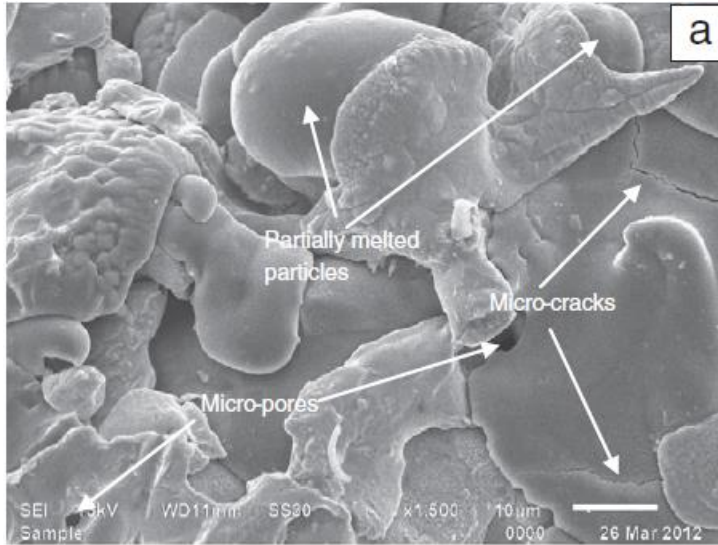


Figure 8 SEM micrograph of Ni-Al₂O₃ cold sprayed surface [49]

2.2.2 Microhardness of Ni-Al₂O₃ cold spray coating

As the composite coating incorporates more hard nano/micro particles, the microhardness of the coating increases accordingly [52]. The rise in hardness is attributed to particle strengthening, dispersion strengthening, and grain refinement [53]. Including hard particles leads to particle strengthening, particularly as the volume percentage increases. Simultaneously, incorporating nano-level fine particles facilitates dispersion strengthening while grain refinement occurs, marked by small grain nucleation on the coated surface. This structural refinement results in increased hardness by impeding dislocation movement. Raghavendra et al. [39] noted that embedded nanoparticles within the composite coating constrain crystal movement in Ni-Al₂O₃ coatings, thereby hindering dislocation motion. The microhardness exhibits an increase proportional to the particle content within the coating matrix. Enhanced microhardness is associated with larger grain boundary areas. The presence of embedded particles alters the crystalline structure of the coating, with significant modifications observed due to the presence of nanoparticles.

Benea et al. [54] The significant influence of 20 nm SiC particles on Ni nuclei and crystal growth was emphasized. Smaller sizes of reinforcement particles are associated with a greater reduction in Ni ion potential towards a positive potential, resulting in increased current due to larger surface area, potentially from heightened cathodic adsorption. Additionally, the enhanced microhardness is linked to smaller crystal grain sizes. In the evaluation of coating hardness, Li et al. measured a value of $173.1 \pm 33.3 \text{ Hv}_{0.2}$, which demonstrates a significant increase compared to the annealed pure Ni bulk 75Hv [28]. As indicated in a study [55], previous research on pure Ni coating revealed a microhardness of approximately $131 \pm 8 \text{ Hv}_{0.2}$. The significant increase in hardness arises from both the strain-hardening effect caused by Ni particles during cold spraying and the reinforcing influence of Al_2O_3 particulates in the composite coating, consistent with findings in other cold-sprayed Metal Matrix Composites (MMCs) [56].

2.3 Wear behavior of Ni- Al_2O_3 cold spray coating

The incorporation of alumina ceramic particles into the nickel matrix improved the wear properties of the coating. Goral et al. [57] noted enhanced wear resistance in Ni matrix coatings with the addition of nano Al_2O_3 particles, along with a reduced coefficient of friction compared to pure Ni coatings. They observed an abrasive wear mechanism attributed to adhesion, facilitated by the presence of hard nano Al_2O_3 particles and higher microhardness. Increasing the volume fraction of nanoparticles in the coating matrix resulted in further improved wear resistance, ultimately leading to decreased specific wear rates with the use of Ni- Al_2O_3 nanoparticle composite coatings [58]. In their study, Aruna et al. [59] found comparable results upon introducing Al_2O_3 particles, attributing the

heightened wear resistance to dispersion strengthening. This wear mechanism is a common observation when ceramic particles are incorporated into Ni matrix coatings [57]. The inclusion of hard nanoparticles in the composite coating changes wear behavior from adhesive to abrasive by creating elevated asperities, leading to a higher coefficient of friction caused by the formation of narrow, sharp grooves on worn surfaces [60]. Franco et al. [61] identified the presence of microcracks on the worn surface when SiC particles were incorporated into the Ni-P coating. The microcracks observed are ascribed to the load-bearing capacity of hard particles, leading to mechanical and thermal stresses. Similarly, Bajwa et al. [62] obtained coherent outcomes with Ni-based coatings incorporating different nanoparticles, noting a two-body abrasive wear nature in Ni-SiC, Ni- Al_2O_3 , and Ni- ZrO_2 coatings, likely due to their elevated hardness and susceptibility to plastic deformation during wear. Composite coatings outperformed pure Ni coatings in wear resistance, contrasting with bare Ni coatings showing adhesive wear. Plastic deformation in composite coatings occurs via debris spreading across surfaces and fatigue crack growth mechanisms. Higher volume percentages of Al_2O_3 particles in coatings improved wear resistance, owing to their heightened microhardness, bolstering load-bearing capacity at the cost of reduced ductility [63].

Furthermore, the higher bonding strength between the coating and the substrate contributes to increased wear resistance. In contrast, higher concentrations of Al_2O_3 particles in Ni- Al_2O_3 composite coatings tend to exhibit an adhesive wear nature. This leads to the formation of a greater number of grooves in the continuous wear track. Additionally, increased wear rates are often observed, indicating accelerated material loss. These observations are supported by Feng et al. [64], who found that Ni coatings exhibit

greater plastic deformation, resulting in larger debris sizes, as highlighted in Figure 9a. Conversely, a lower content of nanoparticles in the coating matrix facilitates particle pull-out, leading to the formation of pits on the worn surface, as depicted in Figure 9b,c. This effect is ascribed to increased abrasive action from elevated asperities formed by agglomerated particles, leading to their detachment from the surface and resulting in pits (see Figure 9d). Consequently, Ni- Al_2O_3 composite coatings exhibit minor abrasion grooves with heightened disorder and discontinuity.

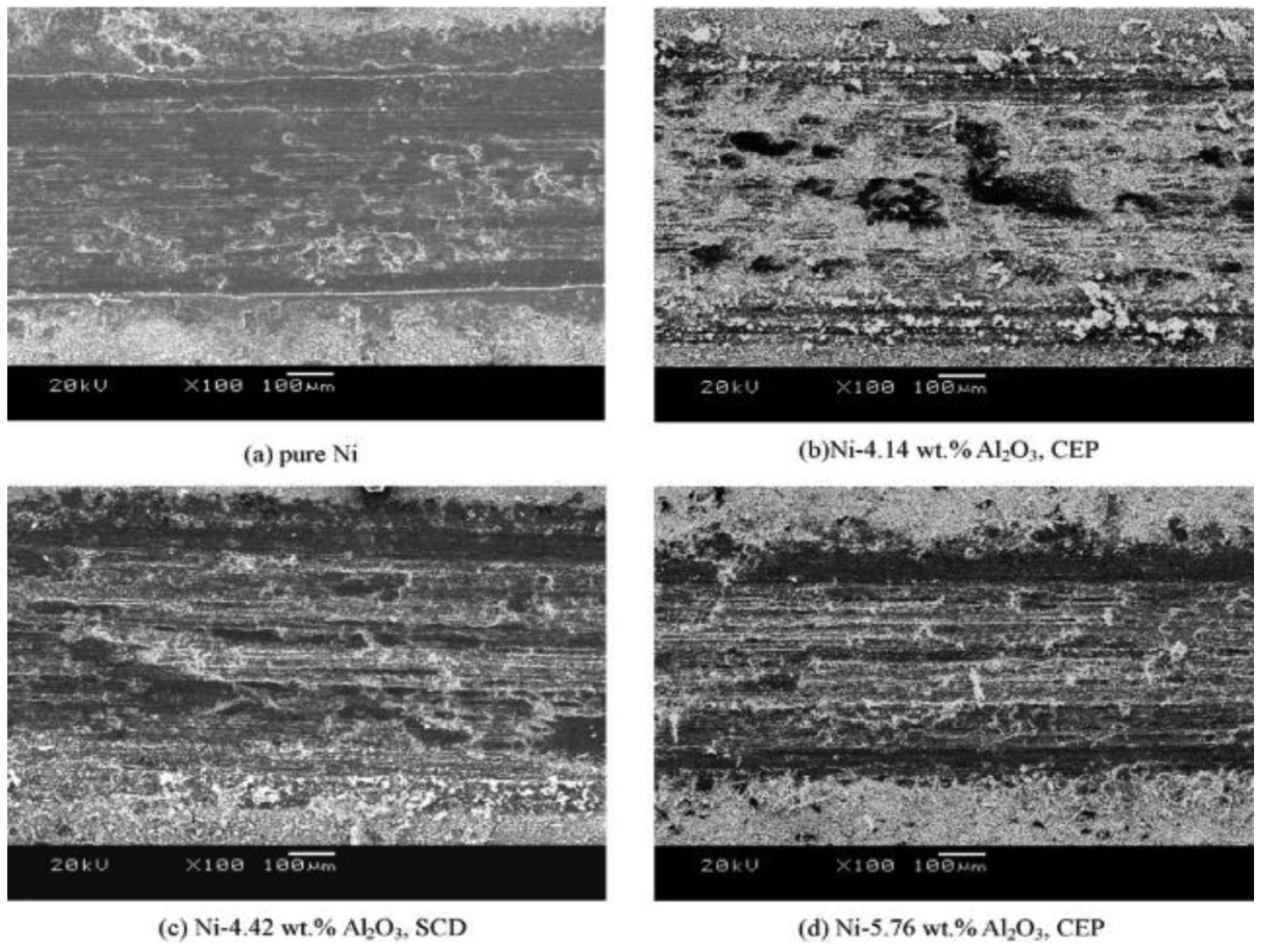


Figure 9 SEM images of coating wear track (a)Ni (b)Ni-4.14wt.% Al_2O_3 (c)Ni-4.42wt.% Al_2O_3 (d)Ni-5.76wt.% Al_2O_3 [64]

2.4 Summary of Literature Review

In summary, this provides a comprehensive overview of the literature review findings, highlighting significant discoveries and emerging trends. The exploration of coating technology research represents a fascinating journey through decades of scientific inquiry and innovation. It begins in 1998 with Dykhuizen et al.'s foundational work on one-dimensional isentropic gas flow modeling, inspiring subsequent investigations. By 2011, Assadi et al. expanded this model to analyze nozzle flow and the effects of copper particles, advancing our understanding of coating dynamics. In 2013, Cavaliere employed computational tools to study how process parameters affect pure metal coating quality, while researchers began exploring particle size and shape effects on coating properties. Despite these efforts, it became clear that pure metal coatings alone lacked significant performance improvements. In 2019, Grewal et al. introduced a groundbreaking approach by incorporating alumina particles into a nickel matrix, dramatically enhancing coating hardness. Subsequent research focused on optimizing coating compositions, varying metal and ceramic content to improve properties.

CHAPTER 3

AIM OF THE RESEARCH

3.1 Objective

Despite the extensive research conducted on pure metal coating, there are still gaps in our understanding of composite coating, particularly in Ni- Al_2O_3 cold spray composite coating, including microstructural changes, wear, and coating hardness. One of the gaps that requires attention is investigation the impact of process parameters such as Gas temperature, gas pressure, stand-off distance, traverse speed, and feed rate on microstructural changes during cold spray deposition. Additionally, further research is necessary to understand the relationship between the coating's microstructure and properties. To address these gaps in our understanding, we used a combination of experimental and computational methods to study the effects of different process parameters on the microstructure and properties of cold spray composite coatings.

Further investigation was required to explore the wear behavior of cold spray coatings. Additionally, the impact of coating thickness and Alumina concentration on wear resistance required attention as an area that needed further examination. More research was needed to explore the mechanisms governing the hardness of Ni- Al_2O_3 cold spray coatings at high temperatures. The coating's hardness was determined by a combination of factors, including its microstructure, Alumina concentration and defect structure. Understanding

the underlying mechanisms governing the hardness of the coating could provide insights into improving its performance at high temperatures.

Based on existing literature, cold spray coating has shown superior deposition characteristics compared to traditional thermal spray methods. This superiority is attributed to factors such as lower working temperatures, the presence of unmelted particles, and improved coating adhesion, quality, and wear resistance.

The primary goal of this research was to understand the microstructure and wear performance of Ni-Al₂O₃ coatings fabricated using cold spray onto 304 stainless steel using different process parameters.

The specific objectives were:

- i. To investigate the effect of process parameters (temperature, pressure, feed rate, and traverse speed) on microstructure and hardness of the coated surface using computational and experimental approach.
- ii. To study the wear behavior of the fabricated coatings and correlate the wear resistance with microstructure and hardness of the coating.

This can help other researchers to establish the relationship between process parameters, microstructure, hardness and wear performance for this composite cold sprayed material. Also, the behavior of wear resistance in cold-sprayed materials and in selecting suitable process parameters and substrate materials for specific applications.

CHAPTER 4

METHODOLOGY

4.1 Research Approach and Methodology

Figure 10 illustrates the comprehensive methodology employed in this study. The selected substrate material was 304 stainless steel and the coating material was Ni-Al₂O₃ powder. The cold spray coating process was employed to coat Ni-Al₂O₃ powder onto a 304 stainless steel substrate, and computational tools were utilized to select the process parameters. The coated surface underwent microstructure characterization, hardness measurements, and tribology testing. Experimental identification of phases was conducted through X-ray diffraction (XRD). Microstructure analysis involved optical microscopy (OM) and image analysis while scanning electron microscopy (SEM) was utilized to investigate the morphology and microstructure of the phases. Energy dispersive spectroscopy (EDS) facilitated qualitative elemental analysis, and micro-indentation and scratch test were employed to measure the coating's hardness. Ball-on-disk sliding rolling wear testing was performed to measure the coefficient of friction (COF) and wear resistance of the coating.

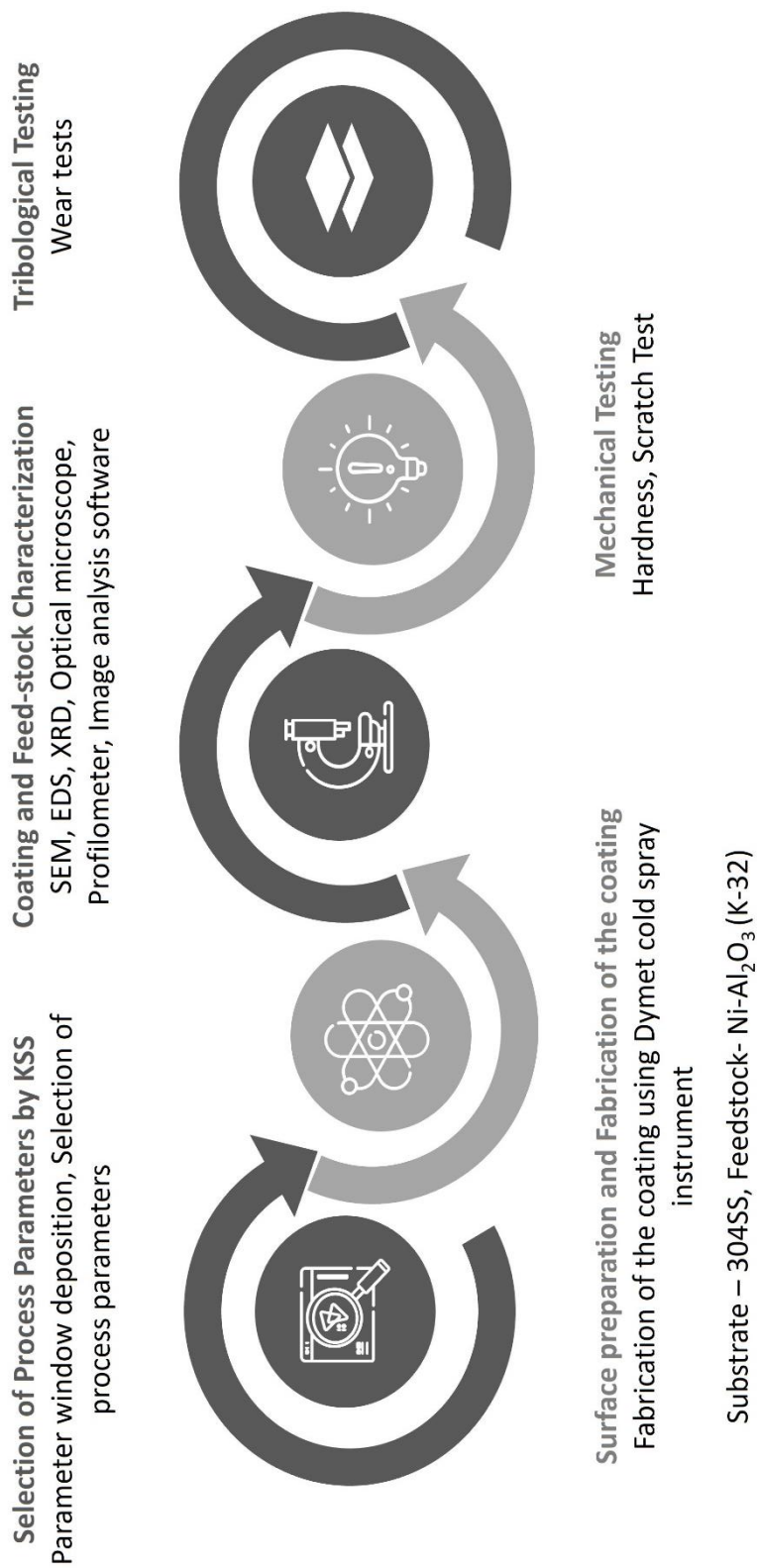


Figure 10 Schematic diagram of research methodology

4.2 Selection of Process Parameters

One-dimensional flow calculations possess limitations as they can only provide insights into gas dynamics within the nozzle and offer only an approximate view along the central axis of an actual three-dimensional nozzle. To gain a more comprehensive understanding of particle impact conditions, the utilization of advanced computational tools becomes crucial. These tools can perform finite volume two-phase flow analyses, providing detailed data on velocities and temperatures across the entire cross-sectional area of the nozzle. This analytical approach enables the examination of complex phenomena, such as interactions with ambient air in the free jet and the effects of the bow shockwave in front of the substrate.

Numerical techniques were utilized via the commercial software KSS (Version 1.4.2) to analyze propellant gas flow patterns inside and outside the nozzle, examining their impact on particle acceleration in cold spraying. Developed by Kinetic Spray Solution GmbH in Buchholz, Germany, the KSS software (version 1.4.2) plays a pivotal role in Selection of spray parameters for specific applications. Its primary function is to identify suitable sets of spray parameters based on intricate calculations involving particle impact conditions and critical velocities. These parameters are essential for achieving an optimal deposition process. Notably, the process employs air as both the carrier and process gas. To determine the optimal process parameters for Ni and Al_2O_3 , we selected a range of gas temperatures from 25°C to 900°C and a pressure range from 4 bar to 8 bar. This software is designed based on a flow model for pure metallic particles in a nozzle. In the upcoming section, we briefly demonstrate how particle velocity, critical particle velocity, and

deposition efficiency are calculated and utilized within the computational tool. The results are presented in terms of cohesive strength, deposition efficiency, and velocity ratio.

4.2.1 Particle velocity

The provided equation matches the gas velocity values calculated through numerical methods, for any stagnation temperature (T_0) [65] .

$$v = v_1 = (c_1 + \frac{D^* - c_1 D^*}{D_e})^n a^* \quad (\text{Eq 1})$$

c_1 and n are fitting parameters.

$$c_1 = \exp (0.335k + 1162k^2) \quad (\text{Eq 2})$$

$$n = 0.85k^{-3} \quad (\text{Eq 3})$$

The fitting parameters are influenced by the process gas, with v_1 being independent of pressure. The form of the fitting function for particle velocity is chosen considering several factors. Firstly, the final gas velocity serves as an upper limit for the final particle velocity at the nozzle exit, ensuring that the particle velocity doesn't surpass the gas velocity during acceleration within the nozzle. Additionally, since gas velocity relies on T_0 rather than P_0 , it represents the maximum achievable particle velocity for a finite T_0 and infinitely large P_0 . Similarly, there's a second limiting velocity, $v_{\max}$, representing the highest attainable particle velocity for finite P_0 and infinitely large T_0 . The particle velocity at the nozzle exit is considered as a convolution of $v_{\max}$ and v_1 :

Particle velocity at the exit of the nozzle:

$$v_p = v_2 = f\left(\frac{1}{v_{max}} + \frac{1}{v_1}\right)^{-1} \quad (\text{Eq 4})$$

Considering the formation of a bow shock in front of the substrate, the actual velocity of particles upon impact tends to be lower. Accounting for this effect, modeling results have been observed to adhere to the following expression:

$$v_{pi} = v_2 \left(1 + \frac{\rho_0 \delta}{\rho_p d_p}\right)^{-1} \quad (\text{Eq 5})$$

Where, d is a fitting parameter of about 0.0007 m for the copper particle.

4.2.2 Critical Velocity

In cold spray coating, smaller particles exhibit higher critical velocities compared to larger particles, attributed to factors such as greater deviation from the adiabatic condition, leading to more effective heat diffusion from their surfaces. Critical impact velocity can be expressed in term of material properties and particle temperature:

$$v_{cr} = k_1 \sqrt{c_p(T_m - T_p) + 16 \frac{\sigma}{\rho_p} \left(\frac{T_m - T_p}{T_m - 293}\right)} \quad (\text{Eq 6})$$

Where, k_1 is a dimensionless fitting parameter typically ranging from 0.5 to 0.6.

4.2.3 Deposition Efficiency (DE)

Elevated pressures and temperatures are known to yield higher particle velocities, consequently leading to increased deposition efficiencies (DEs). Nevertheless, it's worth noting that the characteristic smooth variation of DE with temperature or pressure observed in cold spray experiments is also influenced by variations in particle size.

A broader size distribution results in a slower increase in DE with factors like temperature, pressure, mean particle size (v_{pi}), and v_{pi}/v_{cr} ratio. Caution is advised when interpreting DE plotted against v_{pi}/v_{cr} . Typically, particle size variance has minimal impact on powders, and fluid-dynamic effects introduce particle velocity variance, masking the influence of size variance. Hence, v_{pi}/v_{cr} remains a critical factor for DE prediction. Generally, for ductile materials, DE saturates around $v_{pi}/v_{cr} = 1.2$ and remains unchanged until erosive effects appear at $v_{pi}/v_{cr} = 1.5-3.0$ [65].

With the particle size distribution function $f(d_p)$, for a particular powder, deposition efficiency (DE) can be quantitatively determined as a function of process and material parameters.

$$DE = \int_0^{\infty} gf(d_p) dd_p \quad (\text{Eq 7})$$

Where, dd_p signifies the differential of d_p , and g is a size-dependent function derived concerning the characteristic particle velocities as follows:

$$g = \begin{cases} 1, & \text{if } v_{cr} < v_{pi} < v_{erosion} \\ 0, & \text{if } v_{pi} < v_{cr} \text{ or } v_{pi} > v_{erosion} \end{cases} \quad (\text{Eq 8})$$

In this context, $v_{erosion}$ denotes the upper limit for particle impact velocity, beyond which no deposition occurs.

4.3 Substrate, Feedstock Powder and Coating Fabrication

A commercial 304 stainless steel (SS304) was selected as a substrate material for this study. Table 1 presents the chemical composition of this alloy, as given by the manufacturer's data sheet. Commercially available Ni-Al₂O₃ powder was used for the cold spray coating process. It consists 55 to 59% nickel and 41 to 45% alumina, according to manufacturer's safety data sheet [66].

Table 1 Chemical composition of the substrate material 304SS

Element	C	Si	Mn	P	S	Cr	Ni	N	Fe
Wt.%	0.0220	0.4	1.53	0.031	0.0010	18.2	8	0.055	Balance

4.3.1 Analysis of Feedstock Powder

X-ray diffraction (XRD) was utilized for structural analysis of the powder, employing a Cu K α radiation source with a wavelength (λ) of 0.15416 nm, operating at 10 mA and 30 kV. Morphology and chemical composition analysis of the powder were conducted using a field emission scanning electron microscope (FESEM) model (Quanta 250 FEG manufactured by FEI in Oregon, USA) equipped with energy dispersive spectroscopy (EDS). The particle size distribution of the Ni-Al₂O₃ powder was assessed using ImageJ software.

4.3.2 Substrate surface preparation and coating Fabrication

The substrate surface was prepared by sandblasting using a Trinco dry blast machine. Sandblasting was performed at a pressure of 0.6 Mpa using 70 mesh-sized silica particles. Sandblasting was performed for 15 minutes at a blasting angle of 80 to 90 degrees. Surface

preparation followed the SA2½ standard. Blasted surface was cleaned using Alcohol to remove the foreign particles. And surface was preheated at 500 °C before coating.

Coatings were applied using a DYMET 423 from Dycomet, Europe. The setup was equipped with a heater and a circular outlet de Laval nozzle, specifically the CK20 insert from DYCOMET, featuring a circular outlet with , a divergent length of 120 mm, and an expansion ratio of 5.32 as described in [67]. Schematic diagram of cold spray coating setup is shown in Figure 11. Temperature controller (heater), feedstock and nozzle speed controller are shown in Figure.

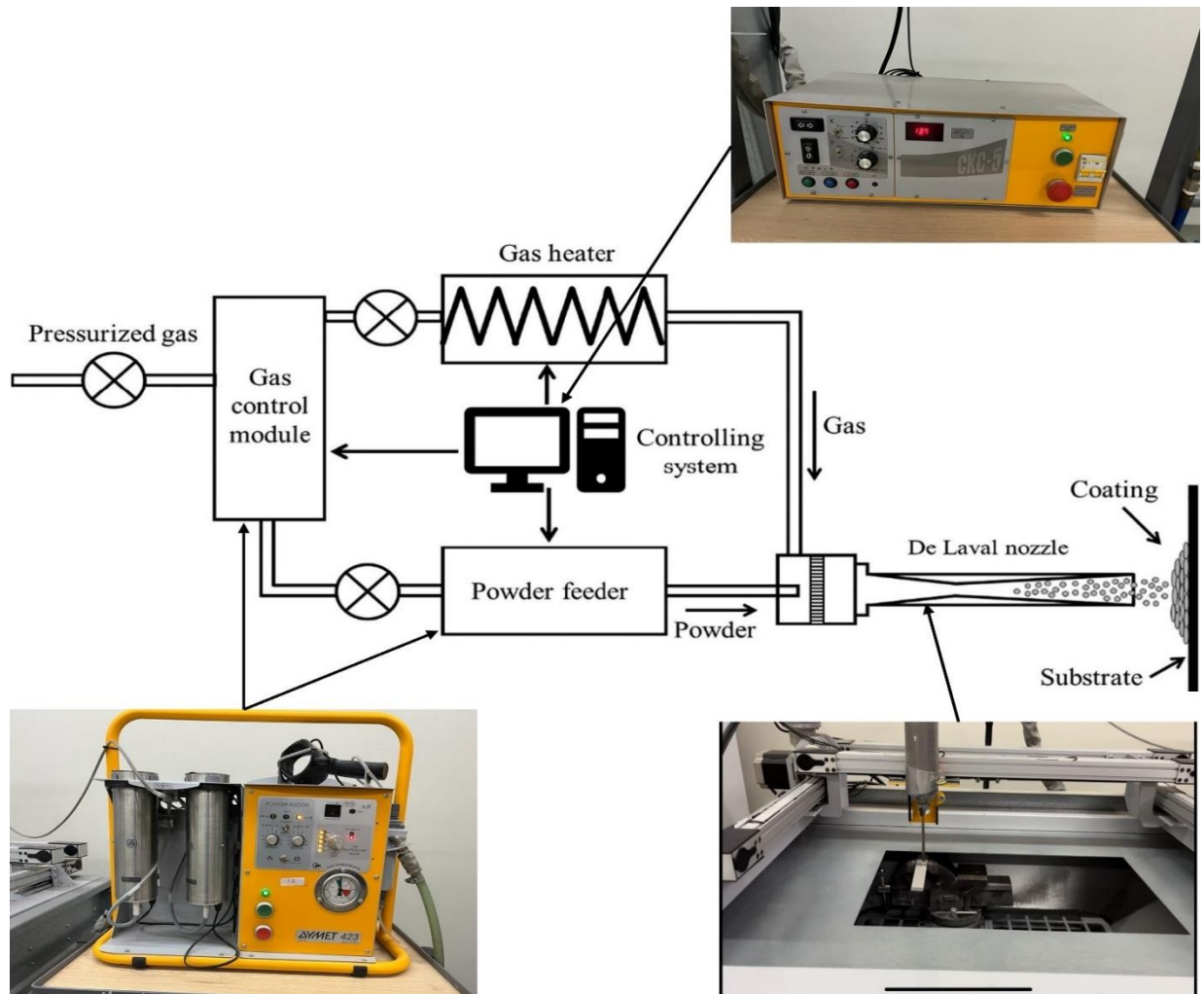


Figure 11 Schematic diagram and setup of cold spray coating instrument

4.4 Coating Sample Preparation and Testing

Coated sample was cut using a diamond cutter, taking precautions to prevent heat effects on the coating. To conduct cross-sectional analysis, the samples underwent sectioning and were subsequently mounted in epoxy powder using a hot mounting machine (Bainmount, Chennai Metco Ltd., India). The mounted samples were then ground using various grit sizes, starting with 220, followed by 400, 600, and 800 grit papers, and polished using a 0.5-micron alumina suspension. Coated samples and epoxy-mounted samples were used for various characterization and mechanical testing.

4.4.1 Phase and Microstructure characterizations

The characteristics of the top-coated surface were thoroughly examined using SEM,EDS and XRD, including the analysis of alumina concentration and micro-defects through cross-sectional (C/S) images. Additionally, a comparison of XRD spectra between the feedstock material and the resulting coating was conducted to assess phase composition and structural changes.

4.4.2 Optical Microscopy

Advanced Image Analysis Software, Stream Motion, was utilized to calculate various parameters from mounted samples. This included measuring the coating thickness, determining the alumina concentration percentage and assessing the porosity percentage in the cross-sectional coating.

4.4.3 Mechanical Testing

4.4.3.1 Hardness of the coating

Hardness testing using a Vickers indenter was performed. The experimental procedure involved subjecting coated samples to a load of 1 Kgf, followed by a dwell duration of 10 seconds. The hardness was tested at 5 different locations for each sample.

4.4.4 Scratch Test

The adhesion of each coating was evaluated using a scratch tester with a Rockwell C indenter featuring a 200 μm tip diameter. The indenter was moved linearly at a speed of 5 mm/min across the coated surface while incrementally increasing the applied load from 0.03 to 30 N at a rate of 15 N/min over a 5 mm distance. Critical loads and failure mechanisms were determined and analyzed through acoustic emission and optical micrographs of the scratch track.

4.4.5 Tribological Studies

Wear testing was conducted on the prepared samples using a Bruker UMT-3 tribometer following ASTM test standard G99-05 [32]. The samples were affixed to a turntable with adjustable rotational speed, while a 6.3 mm diameter alumina ball served as the counter-face. Tests were carried out at a normal load of 20 N and a constant sliding speed of 0.1 m/s, covering a total distance of 100 meters. Three wear tests were performed under each condition for repeatability, and the average coefficient of friction was recorded. Following each test, both the sample and counter-face ball were examined under an optical microscope to assess wear magnitude. All tests were conducted at room temperature

(25 ± 2 °C) and a relative humidity of $55 \pm 5\%$. Wear volume loss (V) was calculated using a profilometer (Profilom 3D,USA). Specific wear rate was calculated using Eq16.

$$K = V/W \times S \quad (\text{Eq16})$$

Where, W is normal load and S is the total sliding distance.

Chapter 5

RESULTS AND DISCUSSION

This chapter includes the results of the characterization analysis of the feedstock powder and coatings. It also covers the selection of process parameters and examines the impact of these parameters on the coating characteristics and performance.

5.1 Feedstock Powder Characterization

The cold spray powder exhibited a dendritic shape for nickel and an irregular sharpened shape for alumina. The morphology of the as-received powder is depicted in Figure 12. The particle size analysis reveals that the Ni- Al_2O_3 particle size falls within the 25 ± 10 μm range as shown Figure 13.

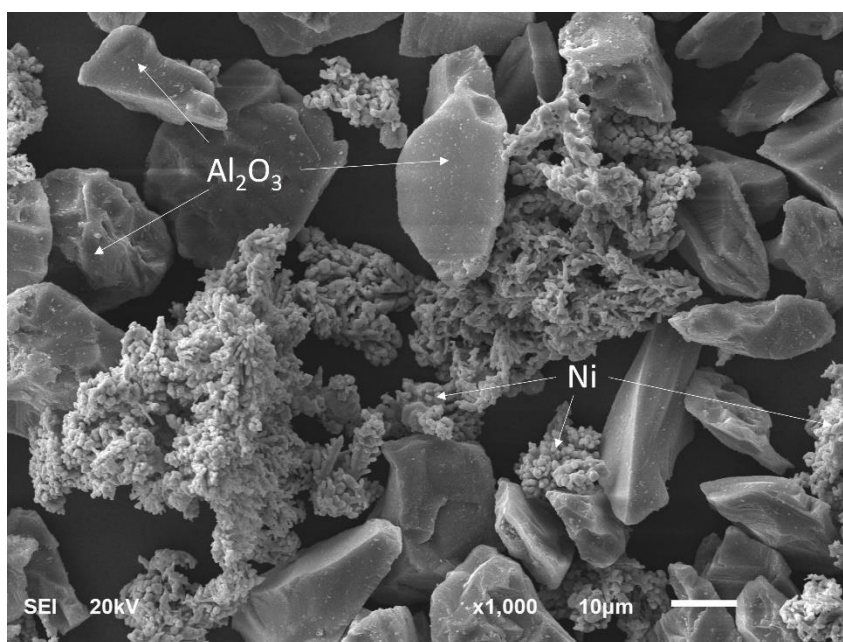


Figure 12 Morphology of feedstock powder Ni- Al_2O_3

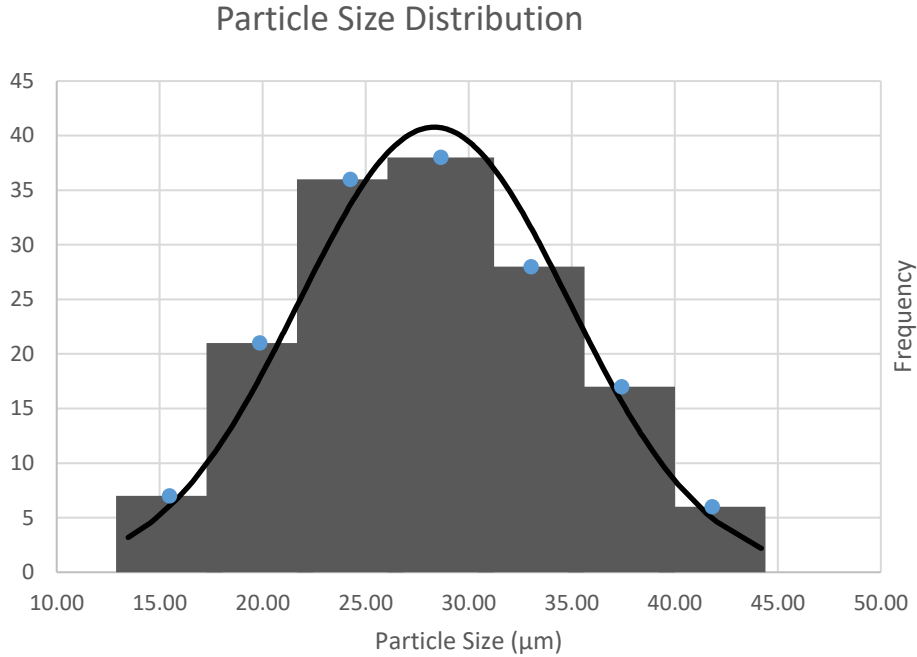


Figure 13 Feedstock powder particle size distribution

5.2 KSS Computational Results

5.2.1 Particle temperature

The graphical representation of Figure 14 clearly illustrates a direct correlation between the temperature of the gas and the particle temperature of nickel. Notably, as the gas temperature experiences an increase, the particle temperature of nickel follows suit, and this relationship holds consistently across the entire range of pressures examined. The linear progression of particle temperature with the rise in gas temperature is a conspicuous trend observed in the data, highlighting the sensitivity of nickel particles to variations in the gas temperature. Which is same as Assadi and colleagues illustrated that elevating the gas temperature results in a linear increase in particle temperature [65].

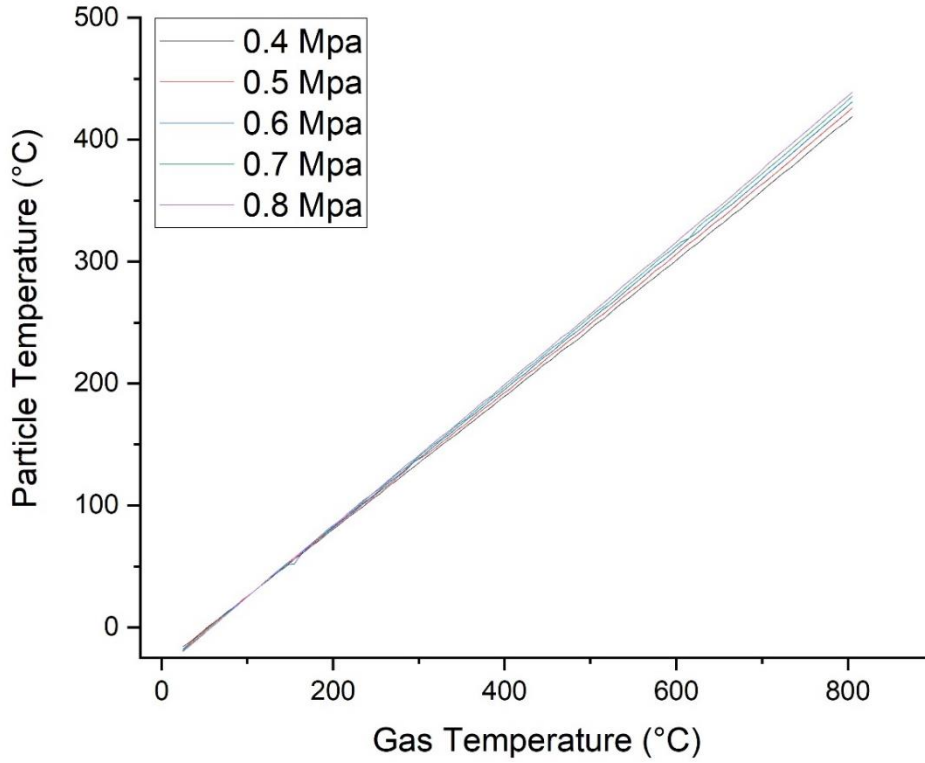


Figure 14 KSS result - Effect of Gas Temperature and Pressure on Particle Temperature

5.2.2 Velocity ratio η

The velocity ratio in Figure 15 displays a consistent linear relationship with particle temperature throughout the entire range of pressure settings. However, a noticeable distinction emerges when considering the data in the context of different pressure levels. At lower pressure conditions, it becomes evident that the velocity ratio exhibits slightly lower values in comparison to those observed at higher pressure settings, even when exposed to equivalent temperature values. This divergence highlights the influence of pressure on the response of the velocity ratio to temperature variations, with lower pressures resulting in a relatively modest reduction in the velocity ratio's value in response to equivalent temperature changes. This nuanced observation underscores the intricate

interplay between pressure and temperature in shaping the behavior of the velocity ratio, providing valuable insights into the underlying mechanisms governing this phenomenon.

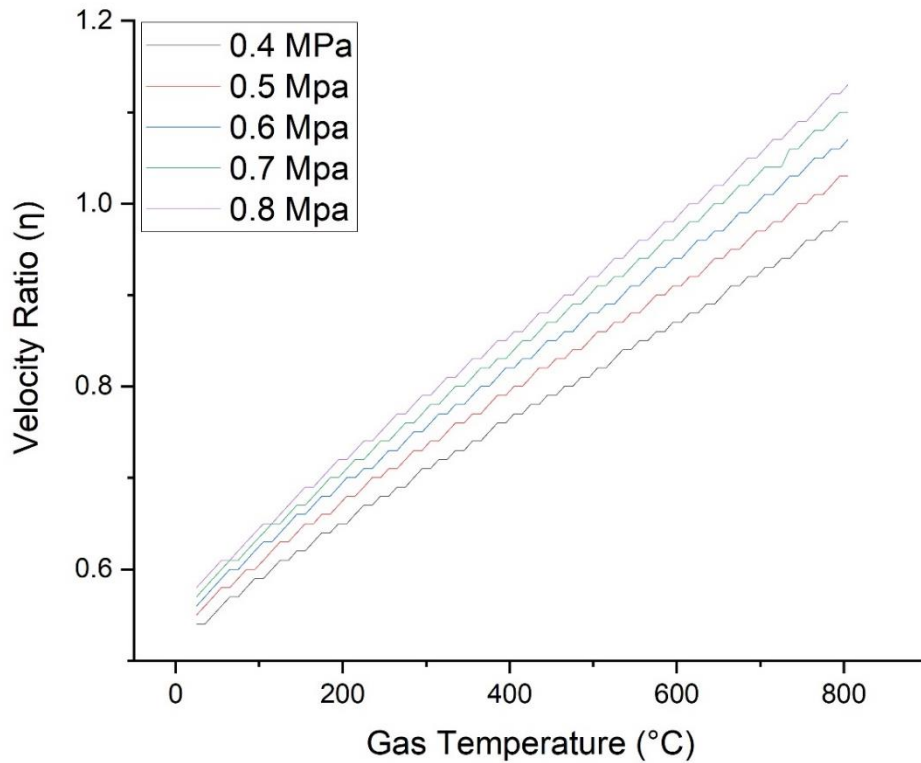


Figure 15 KSS result-Effect of Gas Temperature and Pressure on Velocity Ratio

5.2.3 Cohesive strength

Figure 16 depicts the relationship between gas temperature and the cohesive strength of the coating under different pressures. The cohesive strength consistently remained low at temperatures up to 350 °C. Conversely, between the temperature range of 400 to 600 °C, there was a significant and almost quadruple rise in cohesive strength across all pressure ranges. At temperatures below 350 °C, the ratio of particle impact velocity to critical particle impact velocity (η) was lower at 0.4 Mpa compared to 0.8 Mpa pressure. As a result, the cohesive strength was decreased for low pressure compared to high pressure.

The temperature of the gas also had an impact on this situation. An increase in temperature led to a corresponding increase in particle impact velocity compared to lower temperatures. Therefore, as indicated in Figure 16, at a lower temperature, 0.8 Mpa pressure exhibited higher cohesive strength than lower pressure.

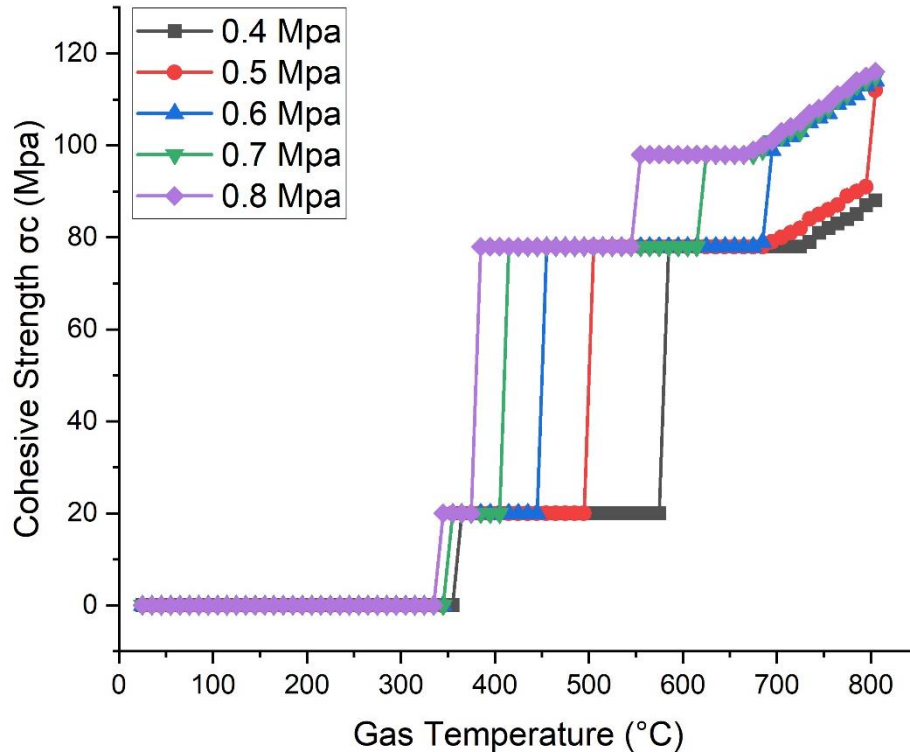


Figure 16 KSS result- Effect of Gas Temperature and Pressure on Cohesive strength

5.2.4 Deposition Efficiency

Figure 17 portrays the relationship between gas temperature and the deposition efficiency of the coating across various pressure settings. It is readily discernible from the graphical representation that, up to 400°C, the deposition efficiency remains consistently low. However, in the temperature range spanning from 400 to 600°C, a substantial and nearly threefold increase in deposition efficiency is evident across all pressure levels. This notable

surge can be linked to factors such as the size distribution of the nickel powder and the critical particle velocity of the nickel, which directly impact the thickness and quality of the coating.

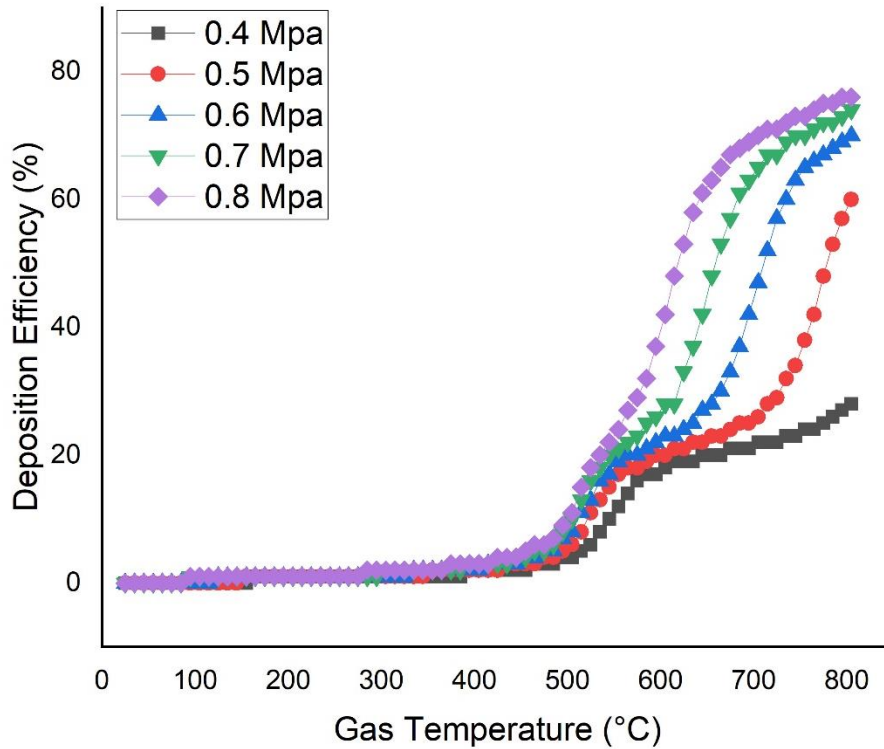


Figure 17 KSS result- Effect of Gas Temperature and Pressure on Deposition efficiency

5.2.5 Window of Deposition

As illustrated in the Figure 18, there is a discernible and noteworthy correlation between particle size and the window deposition. The key observation is that as particle size increases, it induces a notable reduction in the velocity of these particles. This reduction in particle velocity plays a crucial role in determining the deposition dynamics, as larger ceramic particles tend to exhibit a greater tendency to rebound during the deposition process.

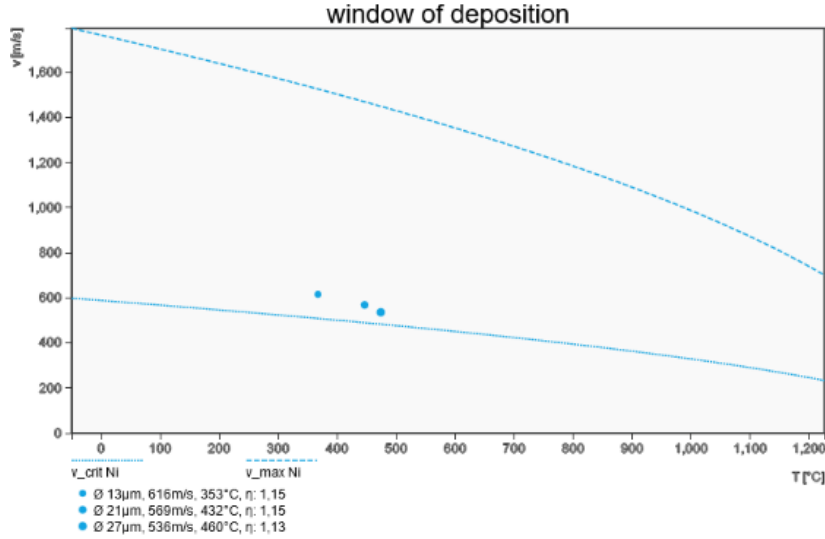


Figure 18 KSS result- Window of deposition for Ni Particle

The parallel pattern observed suggests that variations in temperature and velocity are synchronized between the particles and the compressed gas, indicating a close interaction and mutual influence between these components as shown in Figure 19. This observation highlights the complex dynamics within the system, where changes in one parameter, such as gas temperature or velocity, can directly impact the behavior and properties of the particles being sprayed. Understanding these interdependencies is crucial for optimizing the cold spray process and achieving the desired coating properties.

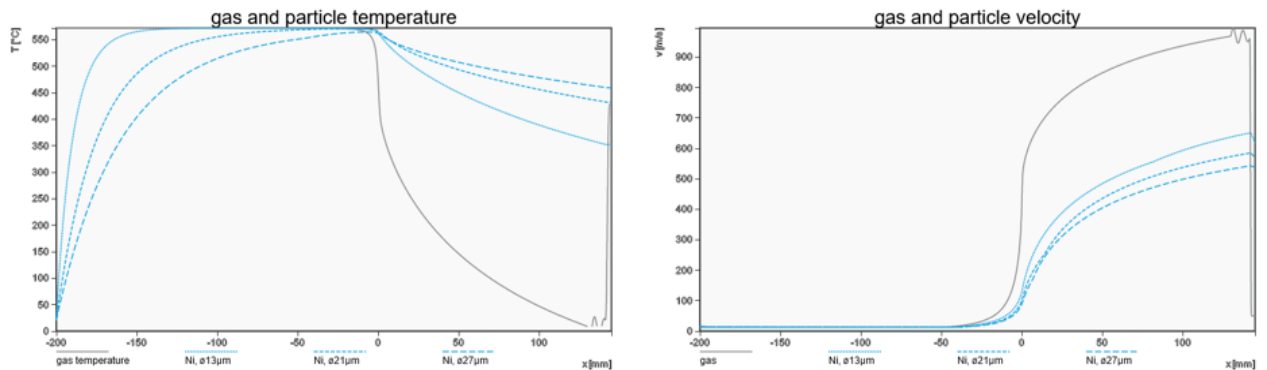


Figure 19 KSS result- Gas and size dependent Particle temperature and velocity

5.3 Effect of Gas Temperature and Gas Pressure

Based on the KSS results (cohesive strength and deposition efficiency), four different temperatures (room temperature, 200°C, 400°C, and 600°C) and two different pressures (6 bar and 7 bar) were selected for coating of Batch 1, as shown in Table 2, to investigate the effects of temperature and pressure on the microstructure of the coating. Air was used as the working gas, and the spacing between consecutive passes (step size) of the spraying gun was set at 2 mm. The constant values for the spray angle and stand-off distance throughout the spraying process were 90 degree and 15 mm, respectively. The total number of spraying runs carried out for each coating was 1.

Table 2 Coating parameters of batch-1

Spray Parameters_Batch-1				
Coating	Gas preheat temperature (°C)	Gas pressure (Mpa)	Powder feed rate (g/minutes)	Traverse speed (m/minutes)
T25P6	25	0.6	21.6	1.8
T25P7	25	0.7	21.6	1.8
T200P6	200	0.6	21.6	1.8
T200P7	200	0.7	21.6	1.8
T400P6	400	0.6	21.6	1.8
T400P7	400	0.7	21.6	1.8
T600P6	600	0.6	21.6	1.8
T600P7	600	0.7	21.6	1.8

Figure 20 depicts the effect of gas temperature on the coating thickness at 0.6 and 0.7 MPa gas pressure. The thickness of the coatings was noticeably increased when the gas temperature was elevated from 25 °C to 400 °C. The coating sprayed at 400 °C and 0.6 Mpa pressure (T400P6) stands out for its uniform structure with a thickness of about $127.96 \pm 1.77 \mu\text{m}$. The velocity ratio was lower at low gas temperatures, leading to reduced cohesive strength between the particles and, consequently, lower deposition efficiency as shown in Figure 16 and Figure 17, resulted in a thinner coating. At high temperatures and pressure, the increased velocity ratio (η) caused the sharp edges of alumina ceramic particles to erode the material, degrading the coating and reduced its thickness. The bulk of particles experienced rebounded, whereas just a small fraction successfully deposited onto the targeted surface. This observation is consistent with the study conducted by Assadi et al., which suggests that a decrease in velocity ratio results in no deposition, while an increase in velocity ratio leads to erosion of the coating material [51].

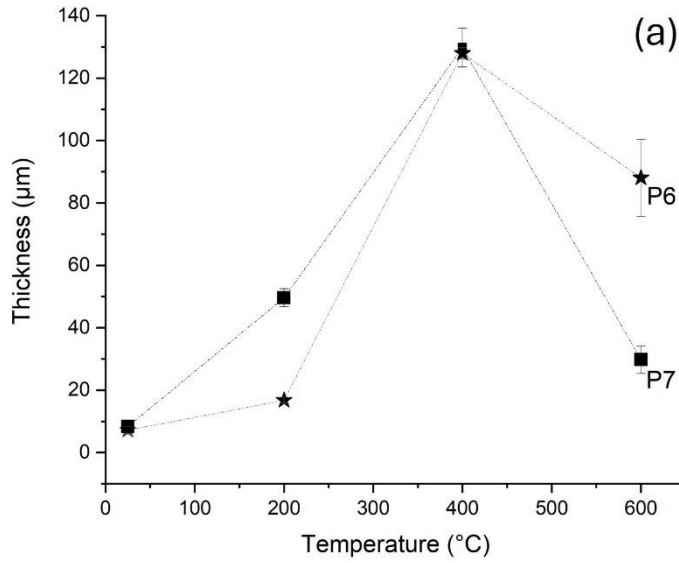


Figure 20 Effect of gas temperature and pressure on coating thickness for feed rate of 21.6 g/minutes and nozzle traverse speed of 1.8 m/minutes

Figure 21 illustrates the impact of gas temperature and pressure on coating thickness. At low temperatures, the coating thickness was minimal but increased with temperature up to 400°C. However, further increments in temperature up to 600°C resulted in a reduction in coating thickness.

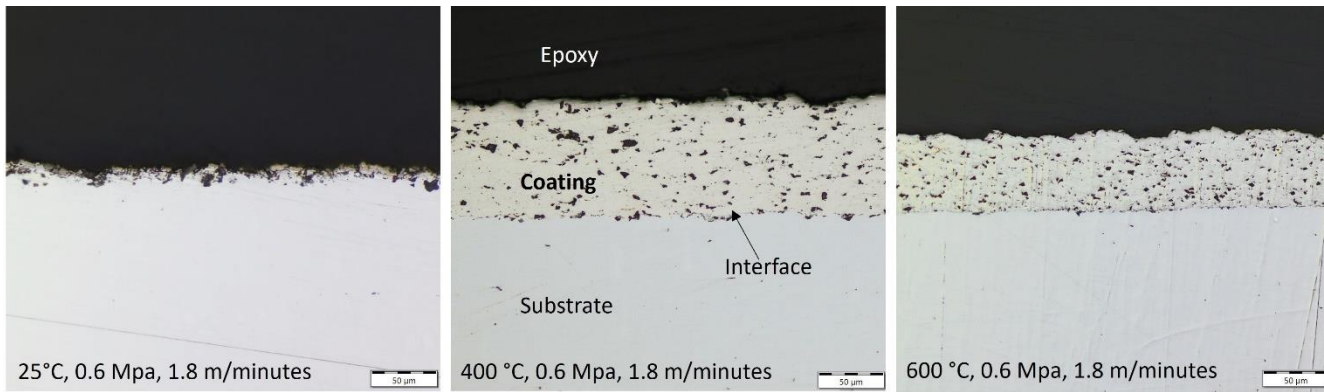


Figure 21 Effect of gas temperature on coating thickness for 0.6 and 0.7 Mpa pressure

The increase in gas temperature had a significant effect on the concentration of alumina ceramic particles in the coating, as shown in Figure 22. At higher pressures (P7) and high temperatures around 600°C, the coating exhibited reduced alumina content due to erosion effects, while lower temperatures below 400°C resulted in decreased alumina content due to lower deposition efficiency. The interaction between temperature, pressure, and alumina concentration possesses a significant role in determining the composition and quality of the coating.

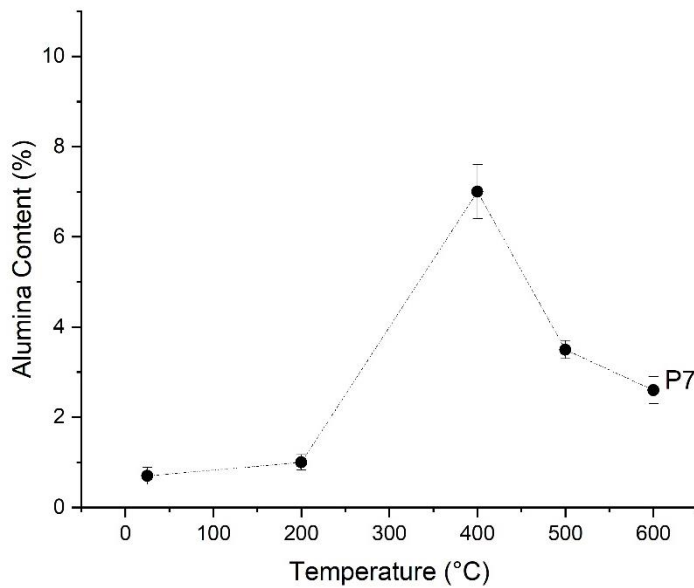


Figure 22 Effect of gas temperature and pressure on alumina concentration in coating

Figure 23 illustrates the impact of gas temperature on the porosity percentage measured from the coating microstructure. The average porosity percentage remained consistent at 3.8% across gas temperatures from 25 to 600°C, with a slight variation observed between the two pressures (0.6 and 0.7 Mpa). At 200°C, under pressure 0.7 Mpa, the rebounding effect was reduced compared to 25°C due to a higher impact velocity, leading to a reduction in porosity. At 400°C, a significant decrease was observed in

porosity percentage. This decline is due to the high cohesive strength and deposition efficiency for both pressures. At 600°C, erosion effect became significant, leading to an increase in porosity in the microstructure for both pressures. This complex interplay between gas temperature, pressure, and coating microstructure highlights the nuanced effects on porosity percentage observed throughout the temperature range studied.

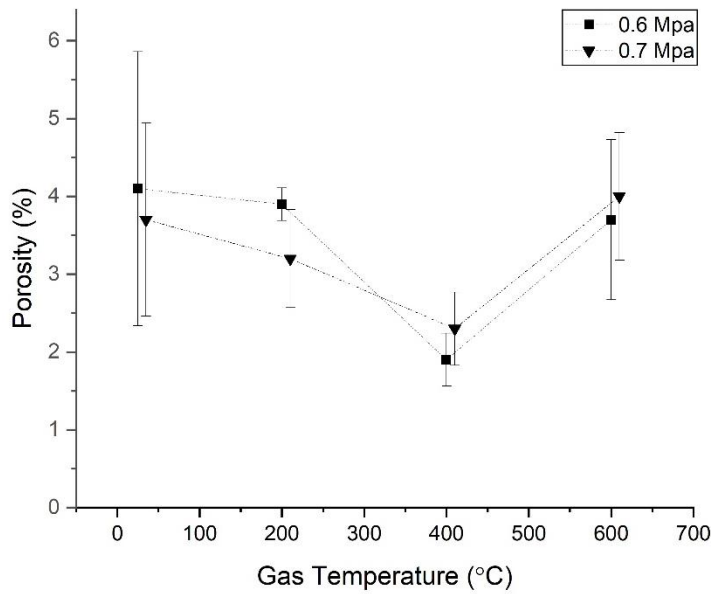


Figure 23 Effect of gas temperature on cross sectional coating porosity for two different pressures at F3 feed rate and 1.8 m/minutes nozzle traverse speed

Figure 24 displays the XRD results of the as-received feedstock powder and the Ni- Al_2O_3 cold spray coating applied using the parameter T400P6. All coatings with other parameters exhibited a similar XRD pattern to T400P6. The XRD pattern revealed a nickel matrix with distinct peaks at various 2θ angles aligned with (JCPDS No. 00-003-1051). It was observed that the peaks corresponding to Al_2O_3 (PDF No. 42-1468) were significantly weaker in coating compared to the feedstock powder. This phenomenon can be attributed to the protective effect of the clad Ni matrix, which effectively shields the underlying Al_2O_3

particles during XRD investigation as shown in Figure 25, a proposition that is in line with the findings presented by Li et al [68] and Tey et al [69]. Also, the reduction in Al_2O_3 peak patterns may indicate a decline in ceramic particle amounts in the coating. The decrease in Al_2O_3 particles is attributed to inadequate cohesive particle strength at temperatures below 400°C and accelerated erosion at approximately 600°C due to elevated velocity ratios, consistent with findings reported by Assadi et al. [65].

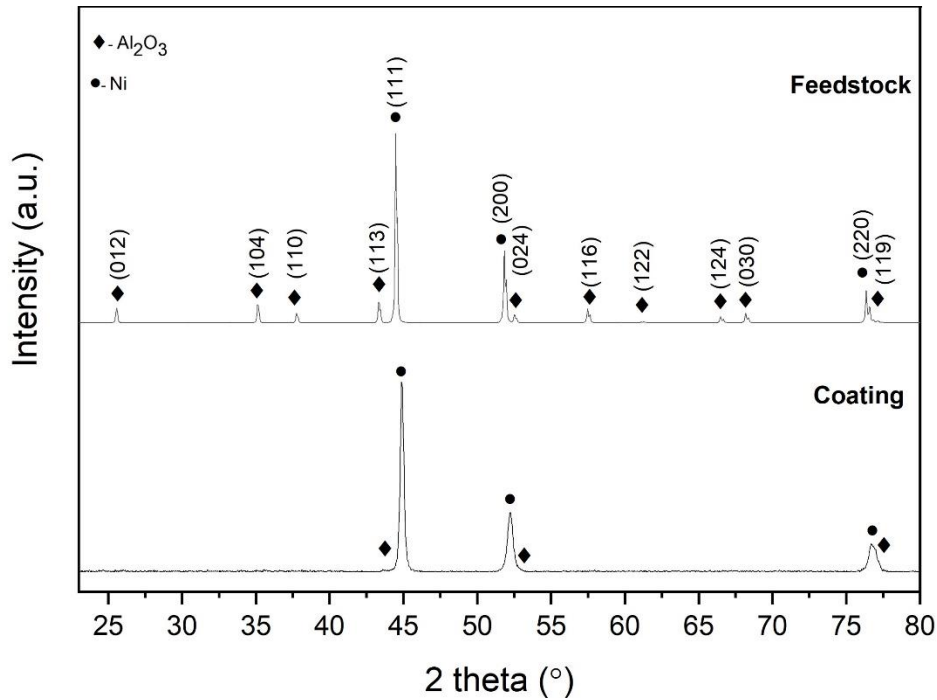


Figure 24 XRD pattern of the feedstock powder and as-sprayed Ni- Al_2O_3 cold spray coating of sample T400P6

Figure 25 (a,b) shows the SEM micrographs of the top surface of the fabricated Ni- Al_2O_3 cold spray coating. EDS analysis confirms the formation of the Ni matrix (Figure 25c) and Al_2O_3 particles (Figure 25d), maintaining their distinct identity within the Ni-matrix. Ni particles were fully deformed, whereas alumina ceramic particles appeared embedded in the matrix. Al_2O_3 particles retain the angular morphology and attributes of

the initial feedstock powders. Some fine fragments of alumina were observed within the Ni-matrix as illustrates in Figure 26b.

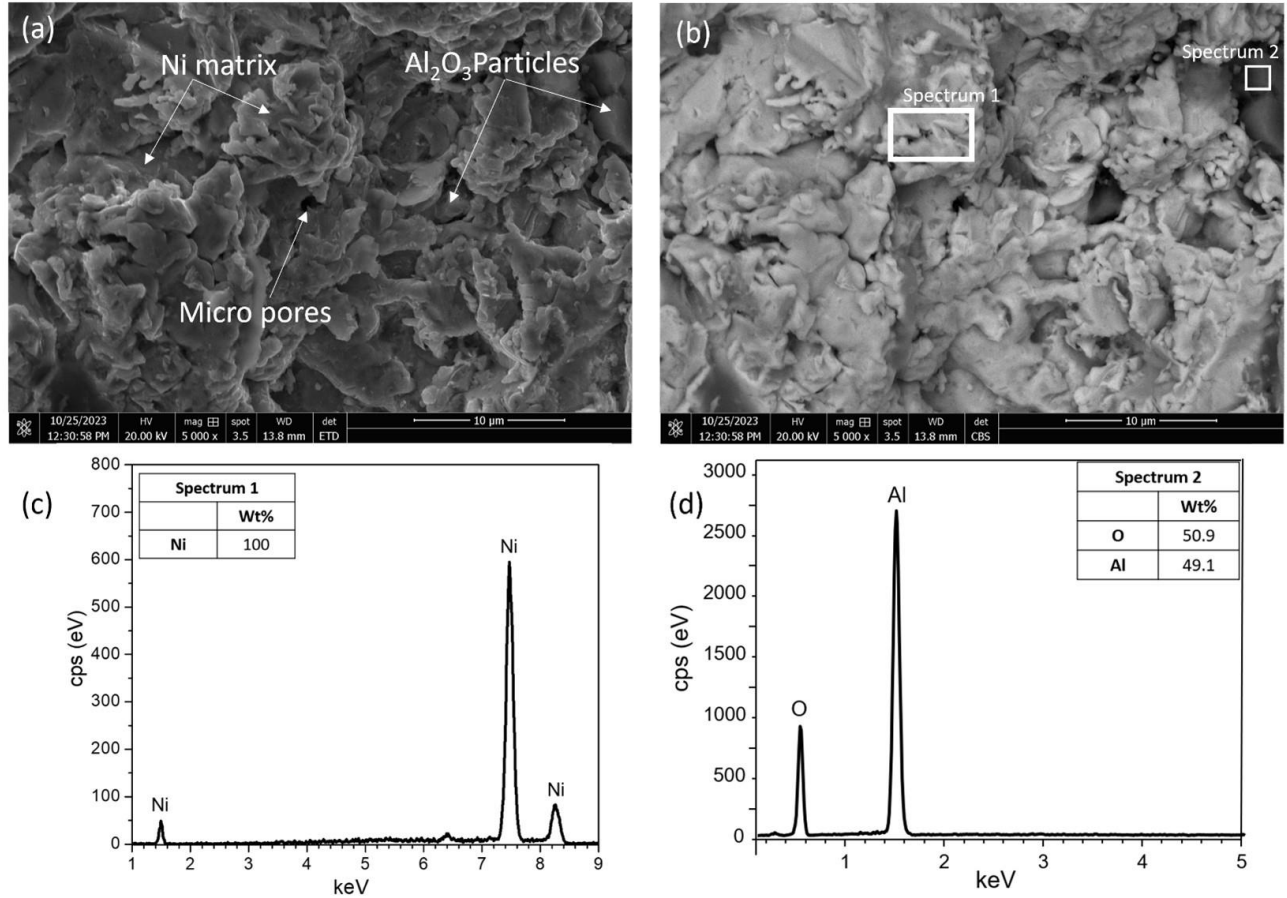


Figure 25 SEM and EDS micrograph of top surface Ni-Al₂O₃ cold spray coating in T400P6

However, at high pressure and temperature, the high particle velocity ratio caused the Al₂O₃ ceramic particles to break down, reducing their size from an average of 30 ± 10 μm to less than 10 ± 5 μm. Also, cracks are found in ceramic particles at high temperature and pressure as depicted in Figure 26a. These findings are in line with the results reported in [43]. The sprayed surface became highly rough due to the bombardment of hard ceramic Al₂O₃ particles during the cold spray coating process. Also, it was found that alumina splats exhibited strong bonding to the nickel matrix [68], [70].

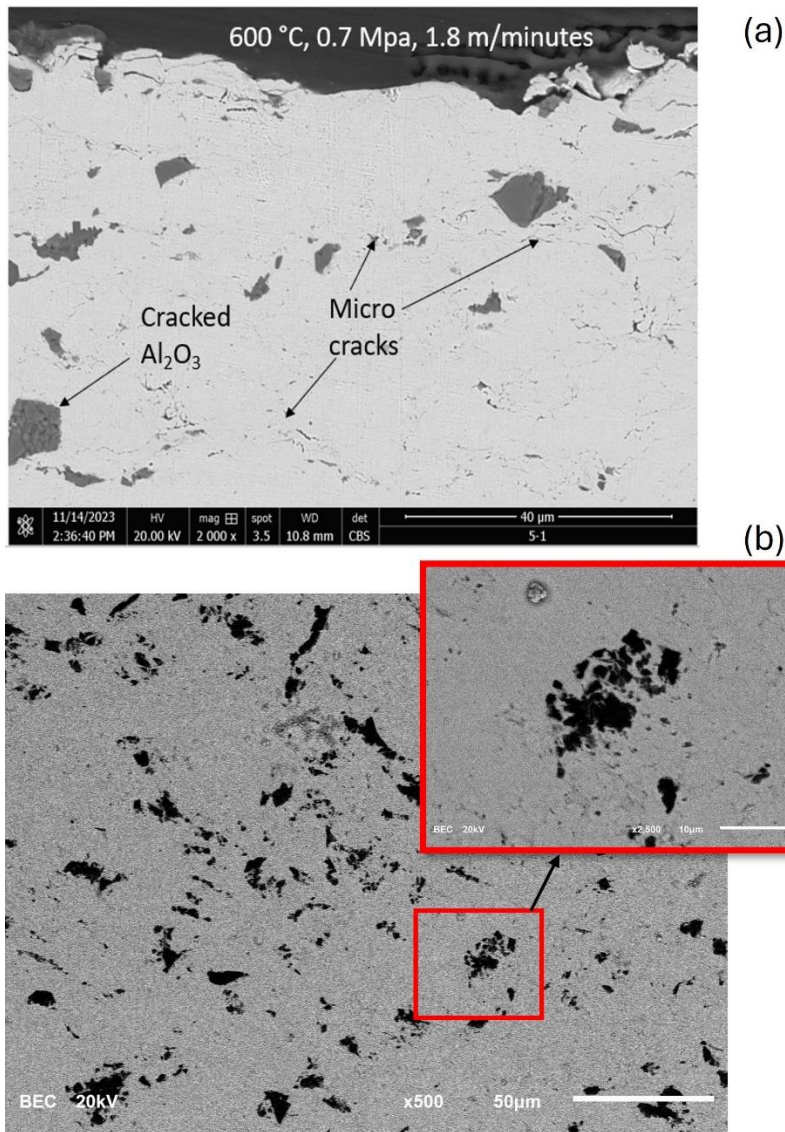


Figure 26 BSE micrograph of T600P7 showing (a) cracked Al₂O₃ and (b) fine ceramic particles at high magnification, illustrating the effect of gas pressure

In summary, it was observed that T400P6 exhibited a uniform microstructure with minimal micro-defects and had the highest coating thickness compared to the other coatings. Based on this observation, a gas temperature of 400°C and a gas pressure of 0.6 MPa were selected to investigate the effect of feed rate and nozzle traverse speed on the coating's microstructure and performance.

5.4 Effect of Feed rate and Nozzle Traverse Speed

Effect of feed rate and nozzle traverse speed was evaluated using the coating process parameters as mentioned in Table 3. Stand-off distance, step size, and spray angle were kept constant throughout the process, with values of 15 mm, 2 mm, and 90 degrees, respectively. A single coat was applied for each condition.

Table 3 Coating parameters of batch- 2

Spray Parameters_Batch-2				
Coating	Gas preheat temperature (°C)	Gas pressure (Mpa)	Powder feed rate (g/minutes)	Traverse speed (m/minutes)
F3V1	400	0.6	21.6	0.6
F6V1	400	0.6	27.6	0.6
F3V3	400	0.6	21.6	1.8
F6V3	400	0.6	27.6	1.8
F3V6	400	0.6	21.6	3.6
F6V6	400	0.6	27.6	3.6

The effect of feed rates and the nozzle traverse speed on the coating thickness is shown in Figure 27. As the nozzle traverse speed increases, the coating becomes thinner for both feed rates. These findings align with the research conducted by Irrisou et al. [23], emphasizing that the cermet's alumina content does not exceed 25 weight percent. At a traverse speed of 3.6 m/minute, the average thickness was measured at its minimum value of around 20 μm for both feed rates. In contrast, a decrease in nozzle traverse speed, specifically when using feed rate F3, yielded the maximum coating thickness of around

450 μm . This outcome aligned with the findings by Kumar et al. [71], indicating that an increase in powder feed rate decreased particle velocity, resulting in a reduction in coating thickness due to a low-velocity ratio. Furthermore, a study indicated that changes in the powder feed rate significantly impacted the flow dynamics of propulsive gases and powder particles within the nozzle [72].

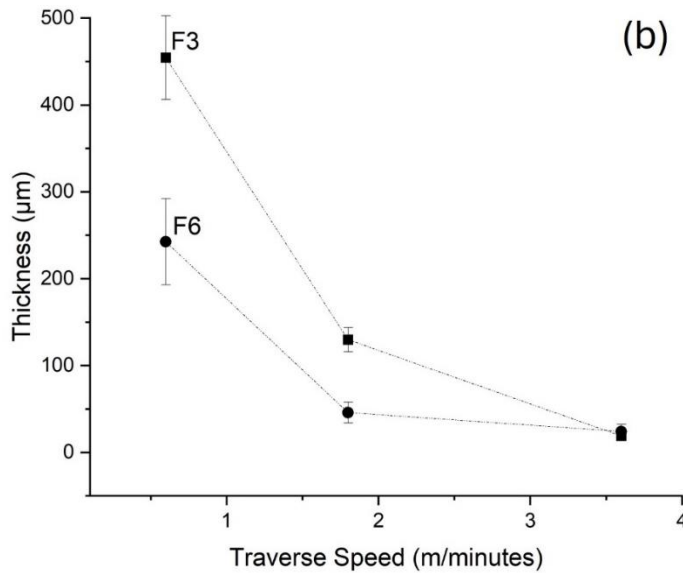


Figure 27 Effect of feed rate and nozzle traverse speed on coating thickness for gas temperature of 400 °C and pressure of 0.6 Mpa

Figure 28 illustrates the influence of nozzle traverse speed and feed rate on the porosity. The statistical analysis revealed that, for both feed rates F3 and F6, the average porosity percentages were nearly identical at nozzle traverse speeds of 1.8 and 3.6 m/minutes. However, a noteworthy disparity emerged at low nozzle traverse speeds, where the average porosity percentage reached 5.5%, considerably higher than at high nozzle traverse speeds for both feed rates. Consequently, it can be inferred that the feed rate had

a negligible impact on porosity percentage under varying nozzle traverse speeds. These findings align with the trends described by Abubakar et al. in [29].

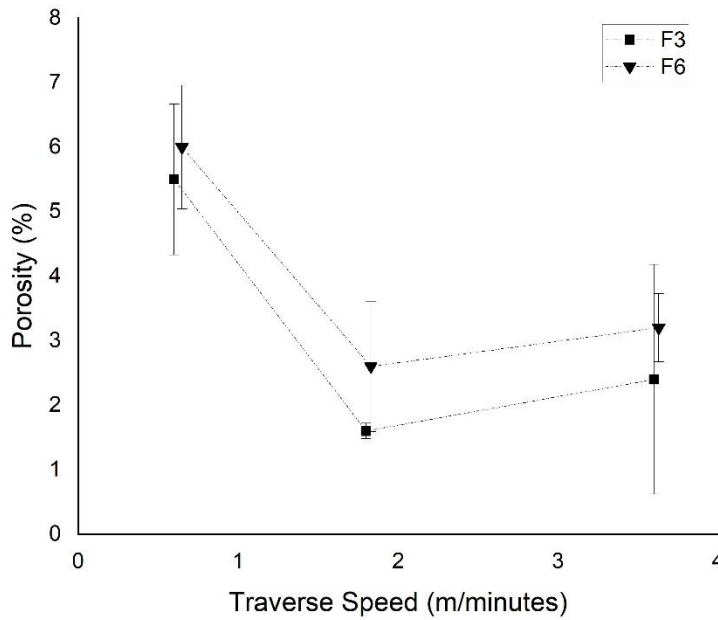


Figure 28 Effect of nozzle traverse speed on cross sectional coating porosity for two different feed rates at 400 °C gas temperature and 0.6 Mpa gas pressure

Figure 29a presents the distribution of the alumina phase that appeared relatively uniform within the Ni-matrix for sample 400 °C, 0.6 Mpa, and with 1.8 m/minute nozzle traverse speed (T400P6). The microstructure of the coating was significantly affected by the nozzle traverse speed. A reduction in traverse speed led to increased thickness but was associated with the presence of microcracks and micropores, as shown in Figure 29b. Low traverse speed induced high residual stress, contributing to the formation of microcracks and porosity in the microstructure. This observation aligned with the findings reported by Kumar et al. [73].

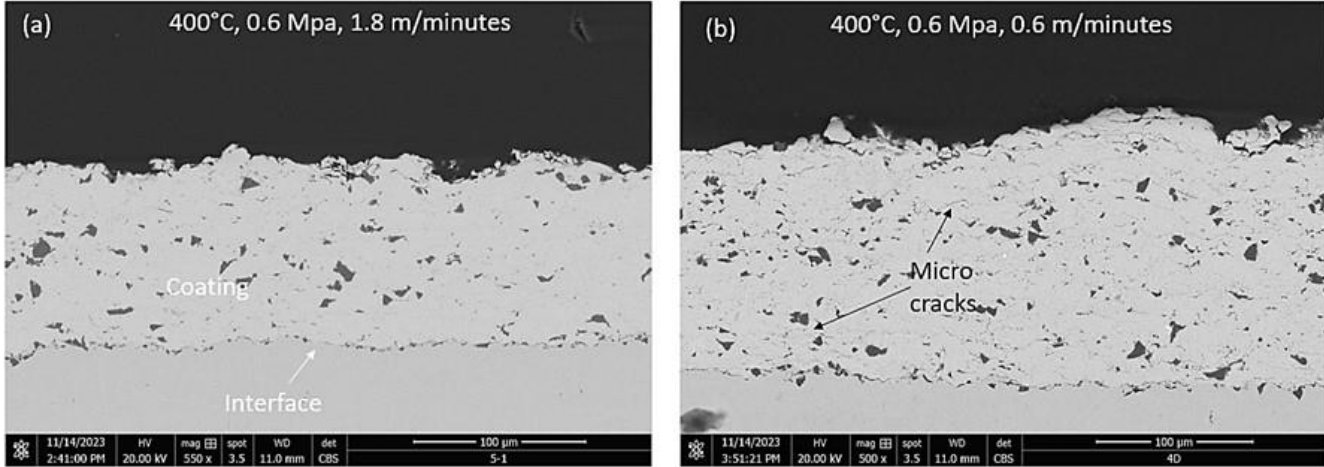


Figure 29 SEM micrograph of Ni-Al₂O₃ cold spray coating showing the effect of nozzle traverse speed at (a) 1.8 m/min and (b) 0.6 m/min for a feed rate of 21.6 g/min

The measured surface roughness of the cold-sprayed surface exhibited variations corresponding to changes in nozzle traverse speed as shown in Figure 30. A reduction in nozzle speed resulted in a significant increase in surface roughness (Ra) for sample F3V1 (nozzle traverse speed 0.6 m/minute), with a Ra value of 0.88 µm, while higher traverse speeds produced a smoother coating surface. As previously mentioned, at temperatures below 400°C, the low impact velocity resulted in the alumina particles bouncing back, causing the surface to become rough and develop voids on the coated surface. On the other hand, at temperatures over 400 °C, the surface experienced erosion and roughening as a result of the high impact velocity of alumina.

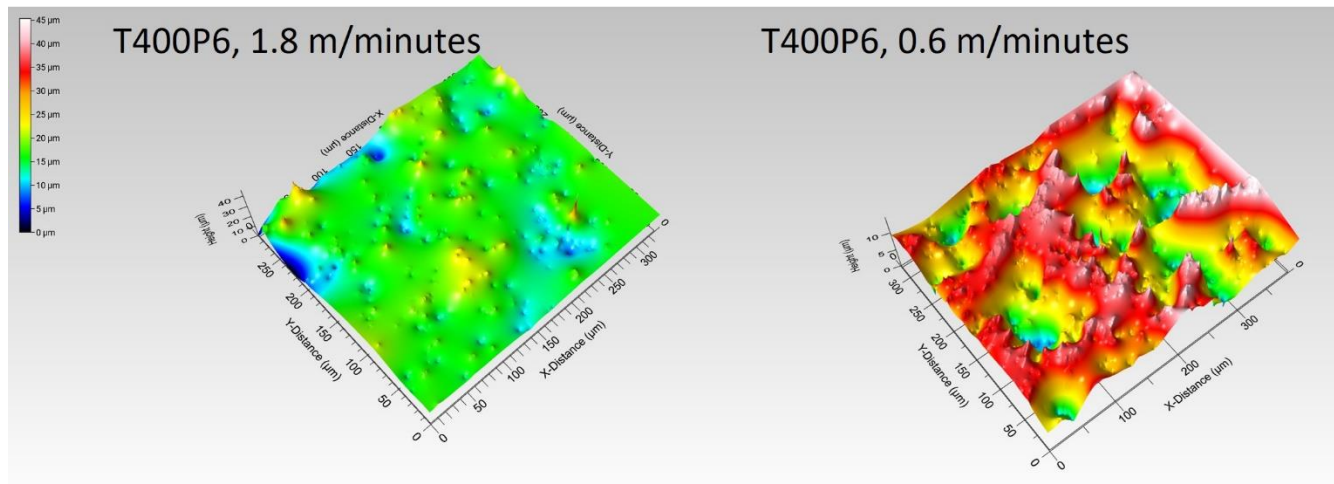


Figure 30 Effect of nozzle traverse speed on surface roughness of cold spray coating

In summary, it was observed that F3V3 exhibited a uniform microstructure with minimal micro-defects compared to the other coatings. Based on the observations, the T400P6 and T200P7 parameters were selected to investigate the effect of multiple passes on the coating's microstructure.

5.5 Effect of Multiple Passes on the coating

The effect of multiple passes was investigated using the parameters listed in the Table 4.

Table 4 Coating parameters of batch-3

Spray Parameters					
Coating	Gas preheat temperature (°C)	Gas pressure (Mpa)	Powder feed rate (g/minutes)	Traverse speed (m/minutes)	Number Spraying runs
T400P6A	400	0.6	21.6	1.8	2
T200P7B	200	0.7	21.6	1.8	3

The average coating thickness for T400P6A remained relatively consistent at $190.37 \pm 17.26 \mu\text{m}$ after two passes, while for T200P7B, the coating thickness was notably lower at $85.42 \pm 14.37 \mu\text{m}$ after three passes, as shown in Table 5 and Figure 30. Due to the low velocity ratio (η), particles in T200P7B were unable to adhere effectively because of their low cohesive strength, resulting in the majority of particles rebounding. Consequently, the alumina content in the T200P7B coating remained low at $4.14 \pm 0.6 \%$, compared to T400P6A, where the alumina content significantly increased to around $8.32 \pm 0.92 \%$, as depicted in Table 5. The rebounding effect directly impacts the coating, creating voids on the surface and affecting surface roughness. As a result, sample T200P7B exhibited higher surface roughness, with an average value of $25.34 \pm 6.21 \mu\text{m}$. In contrast, T400P6A displayed a smoother surface compared to T200P7B, with an average surface roughness of $8.15 \pm 1.34 \mu\text{m}$. These results are consistent with the study reported by Kumar et al in [74].

Table 5 Coating characteristics

Coating characteristics	Coating	
	T400P6	T200P7
Coating Thickness (μm)	190.37 ± 17.26	85.42 ± 14.37
Alumina Content (%)	8.32 ± 0.92	4.14 ± 0.60
Coating surface roughness S_a (μm)	8.15 ± 1.34	25.34 ± 6.21
Porosity (%)	1.03 ± 0.12	2.36 ± 0.76

Analysis of images revealed that the T400P6A coating exhibited relatively low porosity, estimated at approximately $1.03 \pm 0.12 \%$, while the porosity of the T200P7 coating was higher, measured at around $2.36 \pm 0.76 \%$. Despite their overall compactness,

larger pores were discernible within the T200P7 coating, particularly evident upon closer examination in Figure 31d. Additionally, interlamellar cracks, marked by black arrows in Figure 31d, were observed within the coating structure, indicating a lack of robust bonding between adjacent deposited particles and suggesting potential weaknesses in the coating.

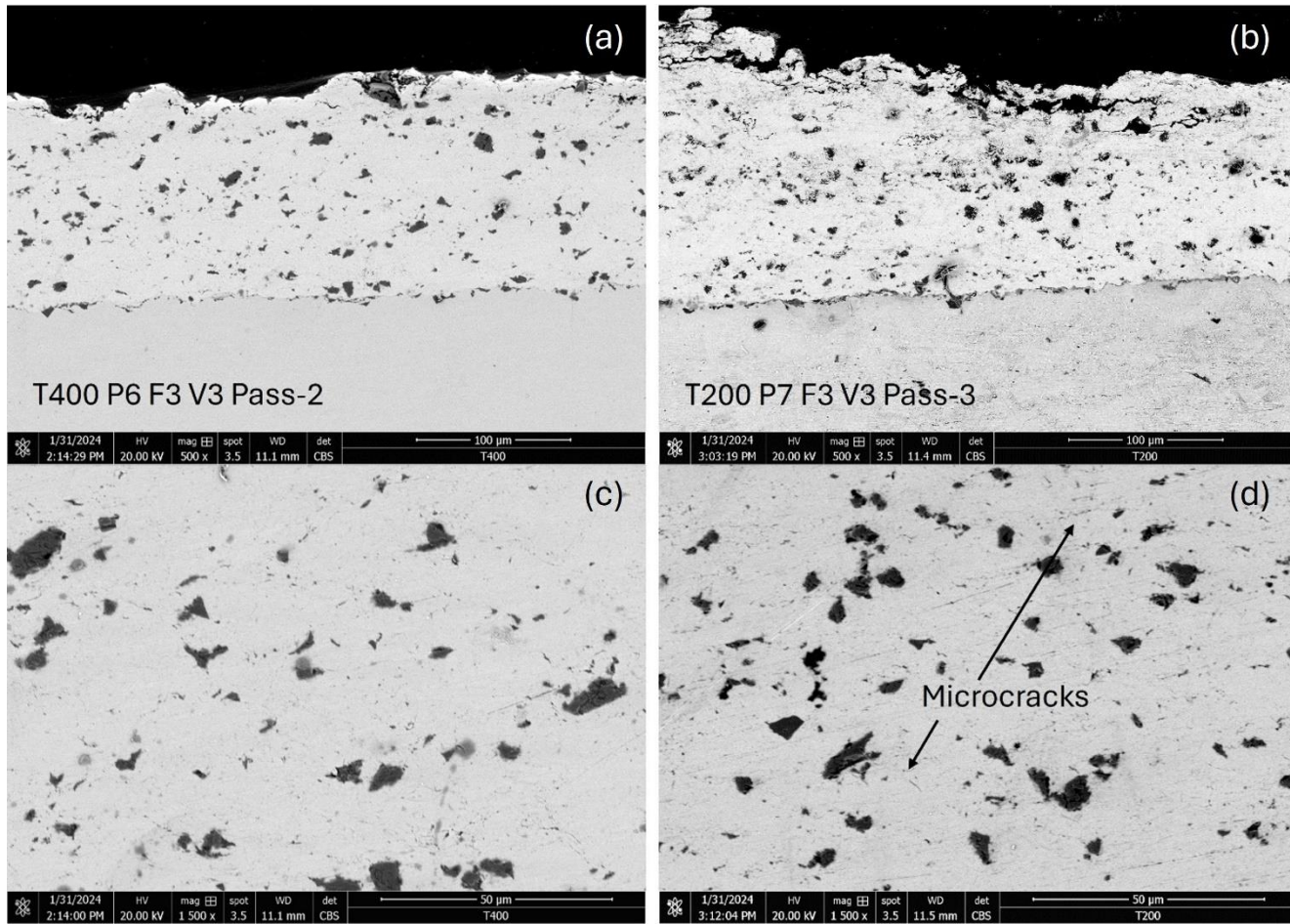


Figure 31 Effect of multiple pass on coating thickness and micro-defects (a and c) T400P6A (b and d) T200P7B

In summary, in both samples, T400P6A and T200P7B, no cracks or defects were observed between the passes. And T400P6A exhibited a higher thickness and a defect-free, uniform coating compared to T200P7B.

5.6 Hardness of the Coating

The hardness of the coated surface, when compared to the bare 304SS substrate, demonstrated a notable increase. This enhancement in hardness is a crucial aspect as it signifies the effectiveness of the coating in providing improved mechanical properties and resistance to wear and abrasion as depicted in Figure 32. The coating with a higher concentration of Al_2O_3 exhibited the maximum microhardness on its surface, with a recorded value of $740 \pm 68.14 \text{ HV}_1$. While the average microhardness in the coating with lower Al_2O_3 concentration was observed at $534 \pm 45.88 \text{ HV}_1$. It was also noted that at locations where Ni and Al_2O_3 particles/splats were in close proximity, the hardness values were exceptionally high. This can be attributed to the resistance offered by the Al_2O_3 particle against the plastic deformation of the Ni matrix. The highest average microhardness was recorded in a sample coated at 400°C and 0.6 Mpa compared to the sample coated at 25°C at 0.6 Mpa and 600°C at 0.7 Mpa , attributed to the presence of the higher alumina particles in coating. A higher alumina content in the coating corresponded to increased overall hardness, consistent with findings reported elsewhere [130].

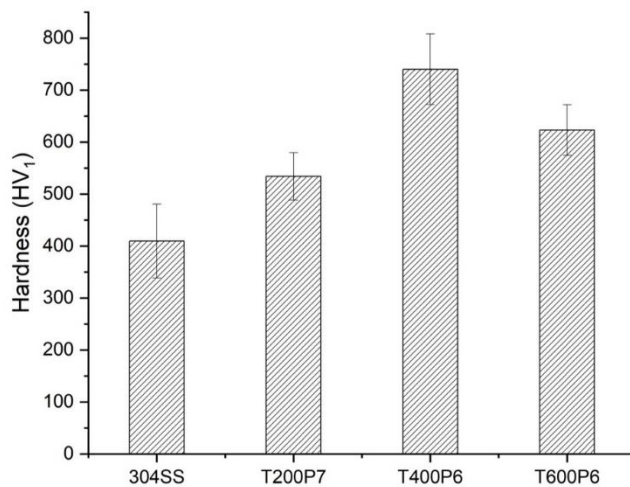


Figure 32 Hardness of the coating

5.7 Scratch Resistance of the Coating

The strength of the Ni-Al₂O₃ cold spray coating was assessed through the implementation of a progressive scratch test. Figure 33 illustrates the relationship between the penetration depth and the gradually applied load, while Table 6 presents the critical load and the coefficient of friction (COF) of the coating. The depth of penetration is dependent on the hardness of the coating. As the resistance of the coating decreases, the penetration depth of the indenter increases. The acoustic signal serves as an indication of the generation of elastic waves, which in turn signifies the occurrence of irreversible fracture formation within the coating-substrate system. Generally, the initiation of these cracks occurs within the coating, subsequently propagating towards the interface, resulting in the delamination of the coating. The point at which the internal crack within the coating first appears is referred to as the cohesive strength (L_{c1}), whereas the point at which the coating separates from the substrate is termed the adhesive strength (L_{c2}).

Table 6 Critical load and coefficient of friction of Ni- Al₂O₃ cold sprayed coating

Characteristic		Results	
		200 °C, 7bar	400 °C, 6bar
Critical Load (N)	L_{c1}	0.4	1.9
	L_{c2}	0.6	4.8
Coefficient of friction		0.32 ± 0.04	0.36 ± 0.06

Table 6 presents the values of L_{c1} and L_{c2} for each of the coatings. The results revealed that the values of L_{c1} and L_{c2} decreased in the sample subjected to spraying gas temperatures of 200 °C compared to the sample sprayed at 400 °C. This decline might be attributed to the reduced concentration of alumina inside the sprayed coating, as indicated

earlier. The experimental results indicated that the presence of alumina led to an increase in the hardness of the coating. The sample subjected to a temperature of 400°C exhibited substantial durability in its coating, evident as the initial crack did not instantaneously result in the detachment of the coating. On the contrary, the coating demonstrated significant resistance to the propagation of cracks, with delamination occurring at elevated loads. The impact of a work-hardened surface near the interface during the beginning periods of the test on the hardness of the coating was noted at a temperature of 200 °C. Nevertheless, when the magnitude of the applied load increased, the impact of this factor gradually decreased.

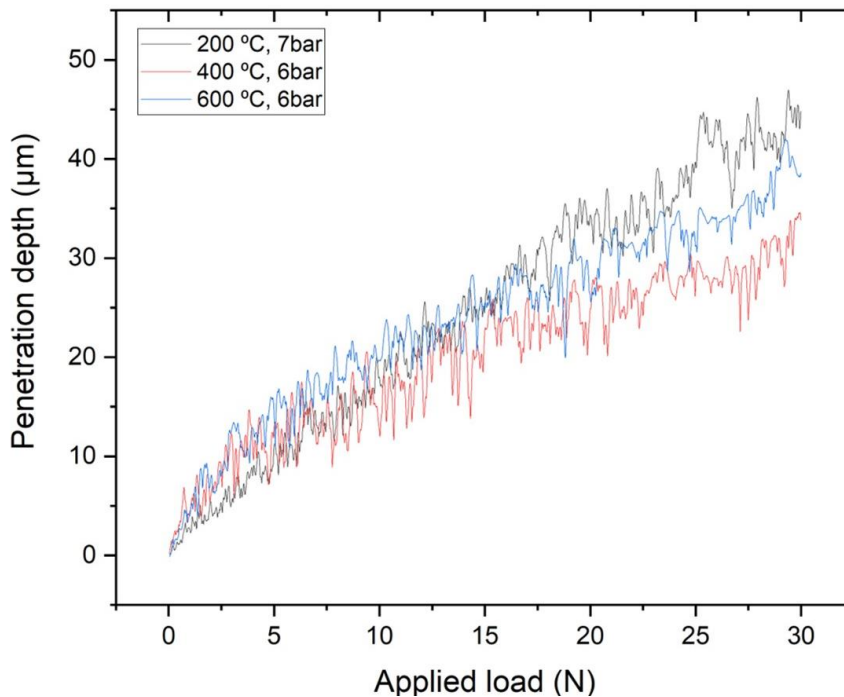


Figure 33 Penetration depth as a function of applied load for Ni-Al₂O₃ cold spray coating for different parameters

Figure 34 illustrates the failure mechanisms, showcasing the complete scratch track and high-magnification SEM pictures of sections extracted from the track. These images provide insight into the distinctive form of failure. The delamination of the coating was seen following cohesive failure, as depicted in Figure 34b. This failure mechanism was found to occur at higher loads. Figure 34d depicts the occurrence of both cohesive and adhesive spallation failure, which signifies the intermittent failure of the coating subsequent to L_{c1} . The phenomenon of continuous adhesive spallation may be noticed with the escalation of the normal load, as depicted in Figure 34d. As the applied force reaches around 5 N, the degree of adhesive spallation increases and expands further from the track.

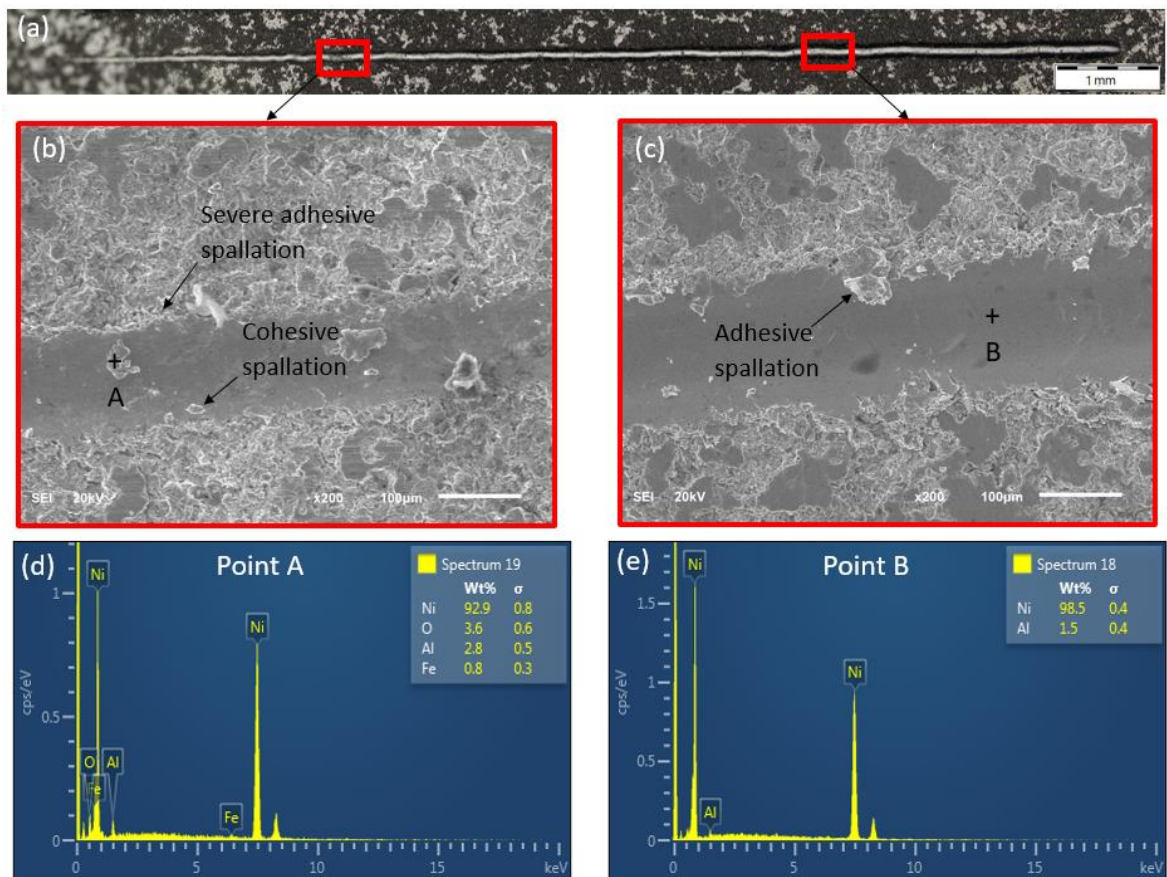


Figure 34 (a) Optical image of scratch track (b, c) high magnification SEM images from scratch track (d, e) EDS analysis at point A and B from (b and c)

5.8 Wear test of the Ni-Al₂O₃ cold spray coating

Figure 35a illustrates the coefficient of friction (COF) curve over the distance traveled. The bare 304 stainless steel substrate displayed a higher COF than the T400 and T200 cold spray-coated steel samples. The bare 304SS sample initially showed a lower COF (~0.2) due to asperity smoothening. However, after the run-in period, the COF sharply increased (~0.58) due to the increased contact area. In contrast, the Ni-Al₂O₃ coated samples maintained a stable and low COF (~0.32) throughout the test. During this phase, nickel oxidation formed a third body in the tribo-system. The COF of the Ni-Al₂O₃ cold spray coated 304SS was nearly 62% lower than that of the bare steel substrate during the steady-state of the test, indicating the enhanced mechanical characteristics of the 304SS surface after coating with Ni- Al₂O₃ cold spray. Applying the coating led to a reduction in both the friction coefficient and wear rate of the surface, attributed to the increased hardness [75]. Harder materials experienced less plastic deformation at the contact point compared to softer materials with higher contact [76]. So, it was observed that higher Al₂O₃ concentration resulted into lower the COF.

Figure 35b depicts the comparison of specific wear rates between the Ni-Al₂O₃ cold-sprayed coated 304SS and the bare 304SS surface. The specific wear rate of the cold-sprayed coated surface was observed to be $7.25 \times 10^{-5} \pm 1.5 \text{ mm}^3/\text{Nm}$ for sample T400P6, which was lower than for T200P7. The specific wear rate for the bare 304SS surface was observed to be $29.83 \times 10^{-5} \pm 1.5 \text{ mm}^3/\text{Nm}$, which was four times higher than for the coated surface. With a higher Al₂O₃ concentration in coating T400P6, the strength of the coating increased, along with higher cohesive strength between nickel-nickel and nickel-alumina particles, resulting in a lower specific wear rate compared to sample T200P7.

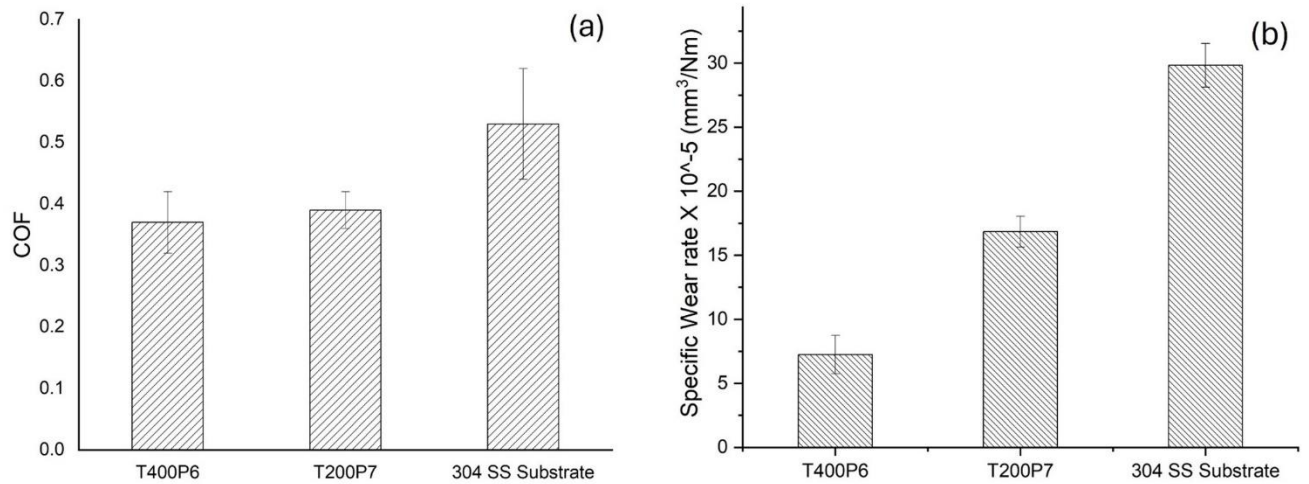


Figure 35 Comparison of (a) COF (b) Specific wear rate of the coated and bare substrate

Figure 36(a and b) shows the 3D optical profilometer images revealing the complete wear track and the corresponding wear scar of the alumina ball used in the wear test. Notably, the wear track on the uncoated 304SS and the wear scar on the corresponding ball appeared to be more severe than those on the Ni-Al₂O₃ coated 304SS and its counterpart ball. The wear track on the uncoated SS and the wear scar on its counterface ball exhibited a series of highly directional grooves and wear depth, indicative of the three-body wear phenomenon, which increased the abrasive component of wear between the contacting surfaces. In contrast, the coated surface showed negligible grooves on both the track and the counterpart wear scar Figure 36b.

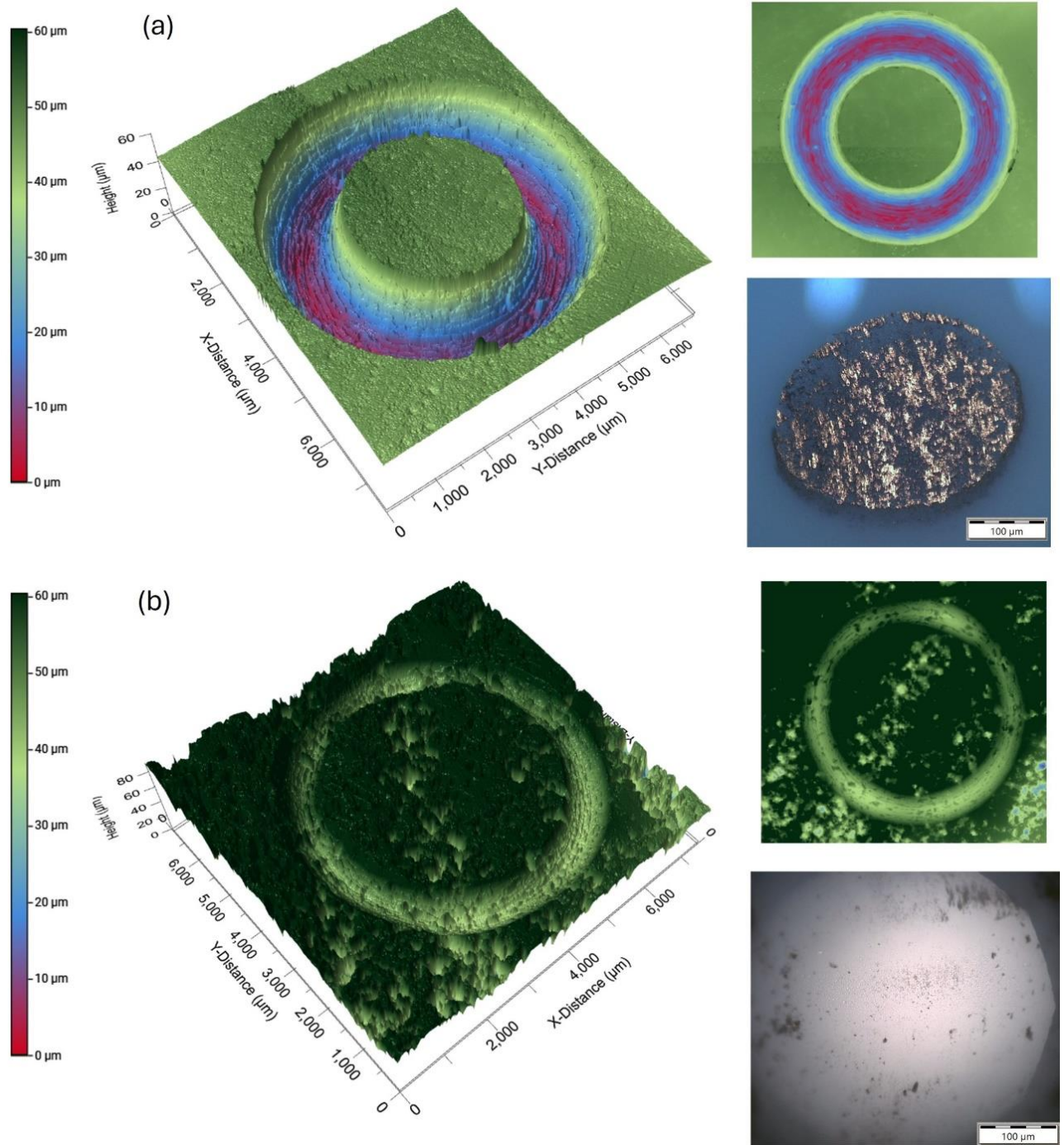


Figure 36 Optical profilometry images of the full wear track and the corresponding wear scar of the ball counterpart for (a) bare 304SS and (b) Ni-Al₂O₃ coated 304SS.

Typical wear track micrographs of Ni-Al₂O₃ coated and bare 304SS surfaces are shown in Figure 37. These SEM micrographs also reveals the presence of worn debris. The macro-observation indicated light wear debris, differing in color from the coating material, suggesting oxidation during the test. Some wear debris was ejected outside the wear tracks, while the rest remained between the coating and the counter material (ball). These wear products acted as the “third body” in the tribo-system.

Figure 37 shown the morphology of the worn traces of coatings T400P6 and T200P7. The surface morphology investigation of the wear traces in sample T200P7 coatings revealed microgrooves and ridges along the wear tracks as a result of micro-abrasion and micro-ploughing. Additionally, plastic deformation of the nickel particles was observed, but no cracks were detected in the wear tracks for the sample T400P6 compared to the T200P7. The substrate was not exposed, and the coating remained intact in all the tests performed.

It is generally accepted that a portion of the frictional work during sliding is converted into heat, leading to an increased interface temperature [77]. This temperature rise can induce surface oxidation and/or the formation of an oxide-based tribo-layer at the sliding interface. In our investigation, oxygen and nickel were detected in the wear tracks using EDS chemical analysis Figure 37b, with oxygen levels around 22–25%, originating from the oxidation of the coating. Thus, the main wear mechanism was micro-abrasion (specifically micro-ploughing), accompanied by plastic deformation and oxidation of nickel.

During the sliding process, oxidation reactions occurred on the real contact areas under the combined action of contact stresses and tribological heat. Part of the debris was trapped between the two sliding bodies, forming a protective layer. This film prevented direct contact between the MMC coatings and the ceramic counterface, thereby decreasing the wear loss of the coating [78]. The absence of aluminum indicated no material transference from the counter ball to the coating. Secondary electron micrographs, representative of track morphology, displayed brighter and darker areas of the wear tracks. As confirmed by backscattered micrographs and EDS analysis, these areas did not consist of different phases and showed no variation in chemical composition.

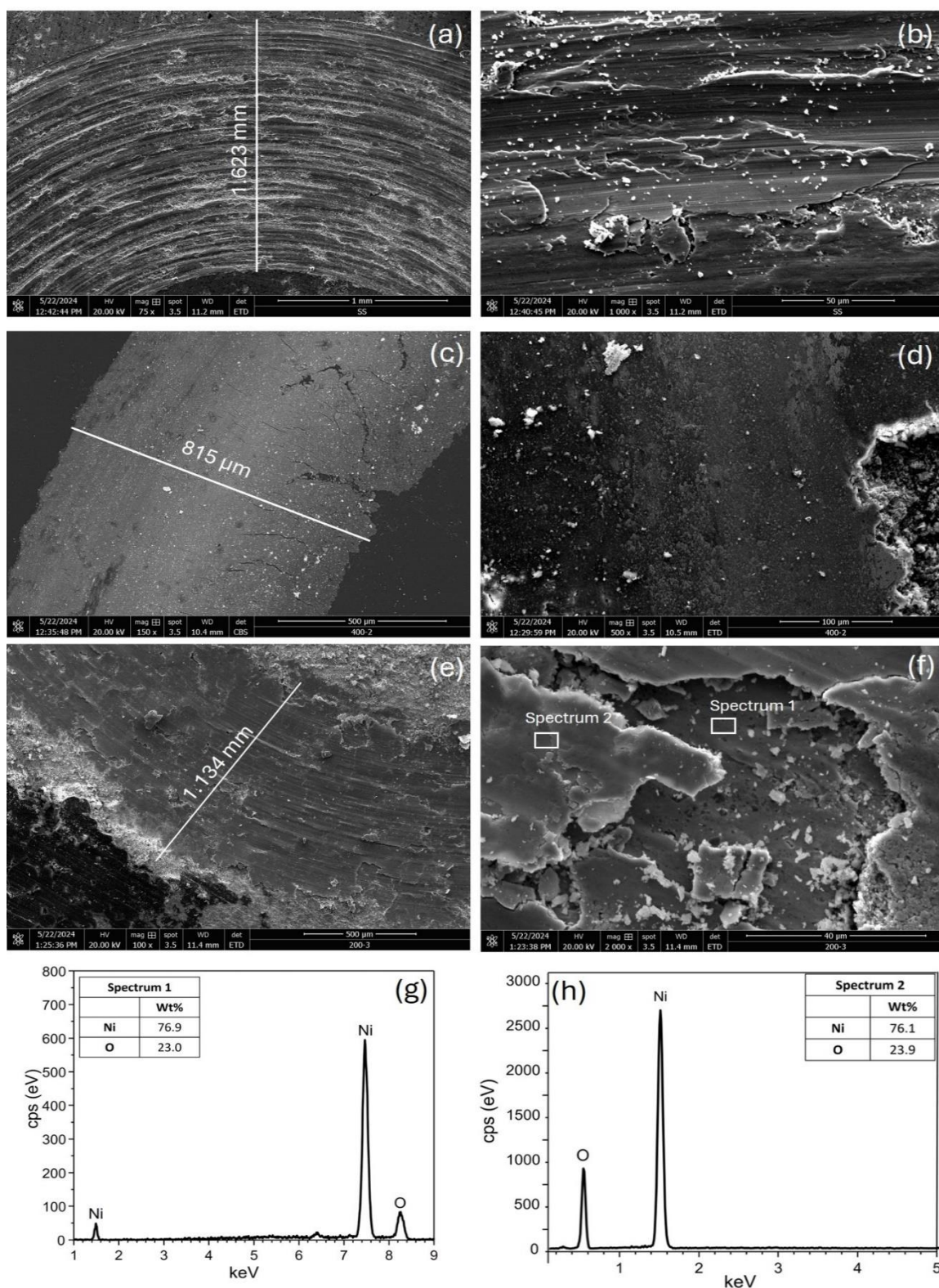


Figure 37 SEM micrograph of wear track of (a,b) bare 304SS (c,d) T400P6 (e,f) T200P7 EDS analysis from the (f)

CHAPTER 6

CONCLUSION AND FUTURE WORK

6.1 Conclusion

This study underscored the significance of using advanced computational tools to select process parameters and minimize experimental trials. It also emphasized the effect of process parameters on alumina concentration, which directly impacts the hardness and wear resistance of the coating.

1. Computational results from Kinetic Spray Solution (KSS) showed a significant increase in cohesive strength and deposition efficiency of the coatings with increasing gas temperature and gas pressure.
2. The sample coated with parameters of 400°C gas temperature, 0.6 Mpa pressure, 21.6 g/minutes feed rate and 1.8 m/min nozzle speed showed a uniform microstructure compared to other samples. Additionally, its high alumina concentration resulted in greater hardness and wear resistance than the other samples.
3. The lowest porosity was observed in the sample T400P6, with an average value of $1.6 \pm 0.4 \%$, which is lower than that of coatings applied using other techniques like HVOF and Plasma Arc.
4. SEM micrographs of the cross-section of the coatings highlighted the presence of interlamellar cracks and larger pores, indicating potential weaknesses within the coating structure for T200P7.

5. The study demonstrates that Ni-Al₂O₃ cold spray-coated 304SS samples exhibit lower coefficient of friction (COF) compared to bare 304 stainless steel substrates, indicating enhanced surface characteristics after coating.
6. The specific wear rate of the bare 304SS surface is four times higher than that of the Ni-Al₂O₃ cold spray-coated surface, indicating that the higher Al₂O₃ concentration significantly increased the coating's strength.
7. The SEM micrographs of Ni-Al₂O₃ coated and bare 304SS surfaces revealed light wear debris and microgrooves from ploughing, with oxidation of nickel contributing to wear. The coating remained intact with the substrate, preventing substrate exposure, and reducing wear loss by forming a protective tribo-layer.

6.2 Contributions to Original Scientific Knowledge

This section outlines the primary contributions of this research to current scientific knowledge.

1. This study represents the initial endeavor to investigate the behavior of composite powder during the spraying process. By analyzing the intricate dynamics and interactions of composite powders during spraying.
2. Previous studies lacked comprehensive computational analysis for selecting parameters of composite powders; however, this study rigorously employed such methods. Additionally, unlike previous research focused on single metals with traditional shapes, this study unveiled unique morphologies: nickel displayed a dendritic shape, while alumina exhibited a sharper form.

3. An advanced computational tool was utilized to optimize process parameters, thereby mitigating the need for extensive experimental trials. This approach not only saves time and resources but also ensures a systematic and thorough exploration of the parameter space, leading to more informed decision-making and better overall outcomes in the experimental process.
4. Advanced image analysis software was employed to meticulously examine various aspects of the material, including its microstructure, particle distribution, coating uniformity, and porosity levels. This state-of-the-art tool enabled detailed scrutiny of the material's internal features, facilitating a comprehensive assessment of its structural integrity and quality.

6.3 Recommendations for Future Research

The Ni-Al₂O₃ cold spray coating explored in this study demonstrated significant potential to streamline experimental trials and resource utilization through the selection of process parameters by KSS, facilitating easy fabrication using the proposed parameters. Based on the findings of this study, future research directions of these coating can be suggested to further understand this coating as follows:

1. Developing computational models for metal matrix composite (MMC) coatings is crucial to optimize their design and performance. Current models primarily focus on pure metal coatings, overlooking specific analysis for composites. Extending existing algorithms to consider parameters like particle size and distribution could enhance understanding and optimization of MMC coatings, benefiting various industrial applications such as aerospace and automotive.

2. Exploring high-pressure process parameters is recommended to comprehend their impact on coating microstructure and mechanical properties, facilitating a thorough evaluation of coating performance for varied applications and operational settings.
3. Future research should explore different substrates with varying hardness levels to comprehensively evaluate coating quality and performance, offering insights into substrate characteristics' influence on coating properties across diverse material compositions and surface conditions.

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“Microstructure and tribological behavior of Multilayer Ni-Al₂O₃ cold sprayed coating”
by **Raihan A. Goriya**, Ahmad A. Sorour. **(Under preparation)**

“Effect of heat treatment on the tribological properties of PVD coated TiAlN coatings on stainless steel substrate” Results in surfaces and interfaces, **(Under review)**

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