Thermal Spray Processes for the Repair of Gas Turbine Components

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Gas turbine components are often operated in harsh conditions, which can lead to severe damage. As it is highly desirable from both an economical and an ecological point of view to restore these worn areas instead of manufacturing new components, repair technologies are of huge interest for companies supplying maintenance and overhaul of gas turbines. In this article, two thermal techniques are described that can be used for this application: cold gas spraying (CGS) and vacuum plasma spraying (VPS). The CGS process allows the deposition of metallic coatings with excellent mechanical properties; several examples including γ-TiAl, Inconel (IN) 718, and IN 738 are given. Essential for the deposition of high-performance coatings in CGS is to exceed the so-called critical velocity. This is discussed also with experimental findings. As a final topic, experiments that use VPS for the repair of single-crystal alloys are described.

1. Introduction

The repair of turbine blades and other large components has become of growing interest over the last 10–20 years to save resources and reduce the costs compared to the production of entirely new parts. These parts are usually used in harsh environments and due to the high mechanical and temperature loads experience creep, corrosion, fatigue, and wear damage. As high quality and reliability are required for each part, the production costs are quite high; e.g., the costs of a single turbine blade can be as much as $8000. Particularly, in the aerospace industry there is a demand for feasible and reliable methods to repair components to further extend the lifetime.

Cold gas spraying (CGS) is a cost-efficient and fast method to repair metallic components and restore their original shape due to the capability to deposit layers up to several millimeters in thickness in a relatively short amount of time. Another advantage is the low process temperature compared to other thermal spray techniques, which avoids oxidation, high thermal stresses, or phase transformations of the feedstock material and the substrate. Several applications for repair[4–6] and additive manufacturing[5,7,8] were already demonstrated and summarized by Raoelison et al.[9] and Yin et al.,[6] e.g., the restoration of a UH-60 helicopter gearbox, a UH-60 rotor transmission housing, and a flap transmission tee box housing.[10–11] The repair process of components can be separated into four steps. First, the damaged surface region is prepared for the deposition by processes such as milling, grinding, polishing, or grit blasting to remove the residual contamination (due to the service hours, e.g., in a turbine) and to smoothen the surface topography. Afterward, the prepared area is refilled by the cold-sprayed coating. As the coating surface has a certain roughness and the dimensions are not the desired ones, a postmachining step of the coated area is necessary to restore the original shape and dimensions of the component. Finally, the repaired component can be tested to ensure its quality.[6]

In the cold spray process, powder particles are accelerated with a hot process gas (nitrogen, helium, or a mix of both) under high pressure toward the substrate. Despite gas inlet temperatures of up to 1100 °C, the feedstock particles stay solid until the impact onto the substrate. For successful bonding in CGS, the particle velocity has to be higher than a material-dependent critical velocity \( v_c \). This concept was found in the 1980s by Anatoly Payrin and his colleagues from the Institute of Theoretical and Applied Mechanics (Novosibirsk, Russia) during experiments in a wind tunnel.[12,13] Assadi et al.[14] analyzed the particle impact by the finite element method. An important observation was a nonuniform development of strain and temperature at the interface velocities close to the experimentally observed critical velocity. As the particles deform at a very high strain rate and under adiabatic conditions, the plastic strain energy dissipates as heat, leading to a softening of the materials. Following, adiabatic shear instability (ASI) was suggested as a possible bonding mechanism in CGS. Grujicic et al.[15] observed the same phenomena and numerous finite element studies confirmed that the ASI is responsible for the bonding in CGS.[16–30] In their study, Assadi et al.[14] formulated an equation to estimate the critical velocity depending on the density, the melting
point, the ultimate strength, and the initial particle temperature. Schmidt et al. extended this work and developed a deposition window for the cold spray process. Here, they also describe the erosion velocity of the powder particle, above which the particle impact leads to dynamic erosion of the substrate material instead of deposition of the desired coating. Kamaraj and Radhakrishna analyzed experimental results from the literature and developed a cold spray coating diagram depending on the temperature dependence of the critical velocity, the particle temperature, and the particle impact velocity.

To optimize the process for the individual coating/substrate system several parameters have to be controlled carefully, e.g., the spray distance, the robot velocity, the powder feeding rate, the inlet gas temperature, and the inlet gas pressure. Each parameter plays a crucial role and the influence on the coating properties such as adhesion, hardness, and porosity has to be understood. Other possibilities to further improve the deposition condition and enhance the adhesion are to increase the substrate temperature, to optimize the substrate preparation, and to pretreat the feedstock.

In this study, we will discuss CGS of three different technologically important alloys: 1) Inconel (IN) 718, 2) IN 738, and 3) Ti–48Al–2Cr–2Nb (or 48–2–2). Due to the frequent use in aerospace applications, the repair of components made out of these alloys is of high interest. The different microstructures and properties of each alloy have a crucial impact on the sprayability of the feedstock.

IN 718 and IN 738 are Ni-based alloys that are often used for high-temperature applications. IN 718 has good mechanical properties up to 650°C in combination with appropriate weldability. In recent years, several authors have studied CGS of IN 718 and successfully deposited coatings with layer thicknesses of several hundred micrometers. Applications that require higher thermal stability typically IN 738 is used. A disadvantage of this alloy is the low weldability due to the high volume fraction (about 40%) of the fine dispersed γ' precipitates, which makes repair processes more challenging.

Due to their high specific strength, titanium aluminides (\(\rho = 4 \text{ g cm}^{-3}\)) are considered lighter-weight substitutes for nickel-based superalloy structural materials (\(\rho > 8 \text{ g cm}^{-3}\)) in gas turbine engines. An example is Ti–48Al–2Cr–2Nb (or 48–2–2), which has been used since 2011 as a low-pressure turbine blade material in commercial aircraft engines. Its implementation led to a significant reduction in fuel consumption, noise, and NOx emissions. CGS is a promising repair process for 48–2–2 because it avoids phase transformation and keeps the oxidation at a minimum. Unfortunately, the properties of Ti–48Al–2Cr–2Nb, namely, the 1) low density, 2) high brittle-to-ductile transition temperature (750°C), and 3) high strain rate sensitivity and yield stress anomaly, impede the deposition of coatings by CGS, as reported by Bakan et al.

With the CGS process, only polycrystalline components can be restored, but another challenge is the repair of single-crystalline turbine blades. As an alternative to laser cladding, Kalfhaus et al. used vacuum plasma spraying (VPS) and demonstrated its potential capability to repair single-crystalline turbine blades. In VPS a direct current arc between the anode and the nozzle cathode of the torch generates a plasma. The feedstock particles are injected into the plasma jet via a carrier gas. In contrast to CGS, the feedstock particles immediately melt after injection into the plasma jet due to the very high temperatures of up to about 10,000°C. The resulting droplets are accelerated toward the substrate, solidify rapidly on the surface, and form splats. The coating is built up layer by layer and the flattening ratio of the splats depends on the Reynolds number. Important process parameters are the gun power, powder feeding rate, the spray distance, and the nozzle velocity. Here, the oxidation is minimized by using a low-pressure protective gas atmosphere (around 60 mbar), usually argon, within the deposition chamber. In addition, by increasing the substrate temperature the heat flux from the impacting molten particles into the substrate can be influenced. This leads to a decrease in the solidification rate and could be beneficial for the repair of single-crystalline turbine blades.

For the applications in industry, the chosen processes have to be well understood and reliable to fulfill the standards on an everyday basis. Thus, the fundamental understanding is of great importance. Different diagnostic systems are available to monitor the particle size, particle velocity, and particle temperature during the plasma spray and cold spray process. This information can later be used to shed light on the underlying physics and to optimize the fabrication of coatings with the desired microstructure and properties.

The results of this article consist of four parts. First, methods to analyze the particles in-flight during the cold spraying process and to identify the critical velocities \(v_{cr}\) are presented. The focus lies on the cold spray meter EVOLUTION, which is available in Jülich. In the second part, the challenge of depositing Ti–48Al–2Cr–2Nb by CGS for jet engine applications is described. The influences of the spray parameters as well as heat treatments of the feedstock powder are discussed. CGS of IN 718 and IN 738 repair coatings are then described in the third part. In the end, the repair of single-crystalline substrates is investigated. A possible application is to restore single-crystalline turbine blades by VPS in combination with a post heat treatment using hot isostatic pressing (HIP).

## 2. Experimental Section

The repair of the following alloys are discussed: Ti–48Al–2Cr–2Nb, IN 718, IN 738, and CMSX-4. Some specifics of the used feedstock powders are given in Table 1. Before the spray experiments, the table

| Table 1. Description of the used feedstock materials. |
|-------------------------------------------------------|
| \(\text{Particle size [} \mu\text{m]}\) & \(d_{10}\) & \(d_{50}\) & \(d_{90}\) & Manufacturer & Remarks |
| IN 718 | 10 | 14 | 20 | Oerlikon Metco & Additional NiAl phase |
| IN 738 | 30 | 38 | 50 | Oerlikon Metco & Amdry 1718 |
| IN 718 | 60 | 78 | 100 | Oerlikon Metco & Amdry 718 Cl. B |
| Ti–48Al–2Cr–2Nb | 28 | 42 | 61 | Helmholtz–Zentrum |
| Ti–48Al–2Cr–2Nb | 18 | 30 | 45 | Helmholtz–Zentrum |
| CMSX-4 | 28 | 40 | 56 | TLS Technik GmbH |
surfaces of the substrates were pretreated, typically by grit blasting. Exceptions were the CMSX-4 single-crystalline substrates. These specimens were cut by electron discharge machining and later prepared by grinding (SiC papers) and polishing with diamond solutions (final step was 1 μm) to mirror-like quality. The grit blasting was performed with F36 (420–600 μm) Al₂O₃ particles under an angle of approximately 90° using a pressure of 0.3 MPa.

The cold-sprayed coatings were fabricated with the Kinetics 8000/52 kW system by Oerlikon Metco (Switzerland; see Figure 1a) and the Impact 5/11 system (Impact Innovations GmbH, Germany). While the Kinetics system can be operated up to a maximum gas pressure of 4 Mpa (40 bar) and a maximum inlet gas temperature of 1000 °C, the impact system offers higher temperature and higher gas pressure, up to 1100 °C and 5 Mpa (50 bar), respectively. These systems were equipped with a water-cooled D-24 de-Laval type converging–diverging nozzle, and nitrogen was used as propellant gas. The spray gun was connected to an industrial robot, which allowed the variation of spray distance, spray angle, gun velocity, and step size.

CGS deposition of 48–2–2 was investigated in Jülich from an intermetallic feedstock powder.[49] The powder was manufactured at Helmholtz-Zentrum Geesthacht (Geesthacht, Germany) by electrode induction gas atomization (EIGA). The oxygen content of the feedstock was 0.079 wt% and due to high solidification rates in atomization, the particles showed a dendritic microstructure. CGS experiments were performed with the Kinetics 8000/52 kW system (Oerlikon Metco AG, Wohlen, Switzerland) using N₂ as process gas. Gas temperature and pressure were 950 °C and 4 MPa, respectively. Powders with two different particle size distributions (d₅₀ = 30 μm and d₅₀ = 42 μm; see Table 1) were deposited at a short (20 mm) and a large (60 mm) spray distance. The robot velocity and feed rate for the coating deposition were 500 mm s⁻¹ and 15 g min⁻¹, respectively. Furthermore, the feedstock was heat-treated at 700 °C for 1 h under vacuum (10⁻³ mbar) to transform nonequilibrium α into α₂ and γ equilibrium phases. Conditions of the heat treatment were defined based on high-temperature X-ray diffraction (XRD) analysis of the powder, and the details are described elsewhere.[49] The deposition with the heat-treated feedstock powder also was performed with a selected parameter set.

An IN 738 repair coating was produced with the Impact Spray System 5/11. A fine IN 738 powder with a dₜ₀ of 8 μm was sprayed at the maximum gas temperature of 1100 °C, at a pressure of 5 MPa, and with nitrogen as a process gas.

For the deposition of CMSX-4 powder on a single-crystalline CMSX-4 substrate a VPS system from Oerlikon Metco, using a F4VB spray gun with a nozzle diameter of 7 mm, was chosen. During the process, the pressure of the argon atmosphere inside the chamber was kept at 60 mbar. The plasma gas consisted of 10 normal liters per minute (NLPM) hydrogen and 50 NLPM argon. As an additional parameter, the substrate temperature was changed using a specifically designed substrate holder, which allowed a temperature variation up to 1000 °C. Afterward, a multistep posttreatment of the coated specimens was performed starting with an HIP-solution heat treatment at a temperature of 1310 °C for 2 h under a pressure of 100 MPa (performed at the Ruhr University in Bochum). Following, two precipitation heat treatments were undertaken, one by HIP and one in a furnace under a protective argon atmosphere. More experimental details can be found in Kalfhaus et al.[56]

As the feedstock kinetic energy plays a key role in CGS, in-flight particle velocity measurements are appropriate to investigate and monitor the process. Yin et al.[69] gave a detailed review of these experiments.
comprehensive summary of previous experimental works on particle diagnostics in CGS. In this work, particle velocities were measured by means of the cold spray meter EVOLUTION (Tecnar Automation Inc., St. Bruno, QC, Canada).

While in other thermal spray processes the thermal emission of the hot particles in the plume is sensed, this is not sufficiently detectable in CGS. Thus, the cold spray meter applied in this work was equipped with a continuous diode laser source to illuminate the particle plume. A dual-slit photomask was used so that the light scattered by particles passing in front of the sensor generated two-peak signals. The particle velocity was then obtained from the distance between the two slits and the measured time from peak to peak. The particle diameters were estimated from the measured energies assuming that the scattered radiation intensity corresponds to the surface area of a spherical particle. More details on the diagnostics procedure can be found in previous studies.

In these experiments, the sensor head was always focused at a spray distance 60 mm from the nozzle exit and at the radial coordinate of the highest flux of triggered particles. During each measurement run, 10,000 single particles were recorded, ensuring that the statistics were sufficiently safe.

For the microstructure investigations of the coatings, a Zeiss Ultra 55 field emission gun-scanning electron microscope (SEM) equipped with EDS INCA and the Crystal Analysis System from Oxford Instruments was used. Electron back scatter diffraction (EBSD) measurements were conducted with a Quanta FEI 650 ESEM (Hillsboro, USA) equipped with a Hikari XP camera (EDAX, AMETEK) to analyze the crystallographic orientations and grain sizes of the substrates and coatings.

The coating porosity was evaluated with a confocal laser microscope Keyence (Osaka, Japan) VK-9710 using a wavelength of 408 nm. The obtained cross-section images were analyzed quantitatively with the analySIS pro software.

3. Results and Discussion

3.1. IN 718 Diagnostics

Three IN 718–type powders were used in the experiments. Subsequently, they are referred to as fine, coarse, and very coarse. Details are given in Table 1. The powders were inert-gas-atomized and had an overall globular morphology. The working gas temperature was varied between 450 and 950 °C and the pressure was set to 3 and 4 MPa. The measured single-particle velocities were weighted by the particle volumes obtained from the corresponding individually measured particle diameters assuming spherical shapes. From these volumetric distributions, the median values were calculated. In Figure 2, these were plotted for the three investigated powders as functions of the propellant gas temperature and pressure. There is an increase of the particle velocities with increasing working gas temperatures, which is steeper for the fine powder than for the coarse one. The particle velocities rise at increasing gas pressure as well, but not that pronouncedly and in a similar order of magnitude for all investigated powders. Furthermore, the particle velocities perform inversely with the particle diameters. These qualitative developments could be expected and agree well with literature data (e.g., Pardhasaradhi et al. [70]).

Assuming that all the particles impacting at a velocity of \( v_p \geq v_{cr} \) are bonded on the substrate, \( v_{cr} \) can be determined from the frequency distribution \( Q_3(v_p) \) of the measured particle velocities. As the frequency distribution is cumulative, each data point designates a volume fraction comprising all those particles with velocities below a specific particle velocity \( v_p \). Focusing in particular on \( v_p = v_{cr} \), the corresponding particle volume fraction \( Q_3(v_{cr}) \) can be interpreted as the part of the feedstock that is not bonded due to too low impact velocities. The complementary fraction \( 1 - Q_3(v_{cr}) \) is however bonded and corresponds...
immediately to the deposition efficiency \( DE \). Thus, it can be inferred that \( 1 - DE \equiv Q_3(\nu_c) \) (see Figure 3). In the experiments, the \( DE \) data was obtained based on the coatings’ weights and the feedstock powder mass flow.

Figure 4 gives the \( DE \) data as functions of the ratios of measured particle velocities to experimentally derived critical velocities \( \nu_p/\nu_c \). According to Assadi et al., key characteristics of the coatings such as deposition efficiency and cohesive coating strength can be expressed as unique functions of this ratio. It was referred to as quality parameter \( \eta \) and is commonly acknowledged as a basic factor for parameter selection maps. In this study, such a unique relationship of \( DE \) and \( \eta \) is experimentally confirmed for IN 718 as all the data points for various temperatures and particle sizes are allocated along parts of almost identical curves.

The measured particle velocities showed the expected dependencies on the process parameters such as propellant gas temperature and pressure as well as on particle size. The critical velocities could be identified by applying the experimental deposition efficiencies on the volumetric cumulative distributions of the measured particle velocities. Furthermore, it could be confirmed that the deposition efficiency builds a unique function of the ratio of particle impact and critical velocities for various spray parameters and particle sizes.

### 3.2. Ti-48Al-2Cr-2Nb

In titanium aluminides, titanium and aluminum are combined to produce intermetallic Ti-Al (hexagonal) and TiAl (face-centered tetragonal) compounds which are named \( \alpha_2 \)-aluminide and \( \gamma \)-aluminide, respectively. Typically, these ordered intermetallics possess low ductility and poor fracture toughness, which makes them susceptible to brittle fracture. Extensive research led to the development of so-called gamma titanium aluminide alloys, such as 48–2–2, where a mixture of \( \alpha_2 \) and \( \gamma \) compounds yields acceptable ductility at room (1–3%) and operating temperatures (5–12%) provided that the microstructure is also optimized with controlled heat treatments.

The properties of the 48–2–2 feedstock may deviate substantially from those of the bulk material due to quenching in the gas atomization process. Oxidation during the gas atomization process is another critical issue for 48–2–2 because the work of Lamirand et al. showed that the room temperature ductility of 48–2–2 was reduced when the oxygen content in the alloy was greater than 0.1 wt%.

In general, CGS of intermetallic compounds has not been extensively studied yet in the literature. Cinca et al. studied the CGS deposition of a Fe–40Al from the intermetallic feedstock. Novoselova et al. investigated depositing coatings from TiAl powder mixtures and transforming the composition into intermetallic titanium aluminide by subsequent two-stage heat treatment. They showed that the \( \alpha_2 \) phase appears upon heating; however, the porosity of the coatings also markedly increases at the same time possibly due to the large difference in the diffusivities of Ti and Al.

The XRD pattern of the feedstock was mainly indexed with the \( \alpha_2 \) phase but without superstructure reflections (Table 2). This was also attributed to nonequilibrium conditions in the atomization process because according to the phase diagram, the investigated composition has a mixture of \( \alpha_2 \) and \( \gamma \) phases at room temperature. This disordered \( \alpha_2 \) phase is referred to as

| Condition | Phase composition | Oxygen content | Indentation hardness$^a$ [GPa] |
|-----------|-------------------|----------------|-------------------------------|
| As-atomized | 0/100 [wt%] | 0 [wt%] | 6.9 ± 0.3 |
| Heat-treated at 700 °C, 1 h, 10$^{-5}$ mbar | 24/76 [wt%] | 0.145 | 9.5 ± 0.3 |

$^a$Average of measurements taken from 20 particles from each powder via nanoindentation.

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Figure 3. Identification of critical velocities \( \nu_c \) from cumulative frequency distributions \( Q_3(\nu) \) of measured particle velocities \( \nu_p \); the index ‘3’ indicates that these are frequencies by particle volumes, not by particle number. The example is for the very coarse powder at a process gas pressure of 3 MPa and a temperature of 750 °C.

Figure 4. Experimentally determined deposition efficiencies \( DE \) as functions of the ratios of measured particle velocities \( \nu_p \) and experimentally derived critical velocities \( \nu_c \). (The dashed lines are only a guide for the eye.)
Although a lower critical velocity is expected at the shorter spray distance due to higher particle temperatures, particle velocities are also anticipated to be lower as the gas is still accelerating the particles at this distance. In contrast, shorter distance also implies a higher substrate temperature, 

Figure 5. Cross-section SEM images of 48–2–2 deposits (950 °C, 4 MPa) on 48–2–2 substrates. a) \( d_{50} = 42 \mu m, \) spray distance = 60 mm; b) \( d_{50} = 30 \mu m, \) spray distance = 60 mm; c) \( d_{50} = 42 \mu m, \) spray distance = 20 mm; d) \( d_{50} = 30 \mu m, \) spray distance = 20 mm. Dashed lines show the coating–substrate interface.\(^{[49]}\)

Quantitative XRD analysis of the heat-treated feedstock revealed an increase in the \( \gamma \)-phase content to 76 wt% (Table 2). Superstructure peaks of the \( \alpha \) phase were still missing after the treatment, indicating that ordering in this phase requires longer time and/or higher temperature treatments. This could not be done in the vacuum furnace because the oxygen content of the feedstock was already increased up to 0.145 wt% after the short treatment performed. When the hardness of the particles before and after nanoindentation was compared, it was found that the hardness was significantly increased after the heat treatment (Table 2). Either phase transition or oxidation during the treatment seemingly leads to this hardening, which is not clear at this stage of the investigation. In agreement with this result, however, the number of bonded heat-treated particles on the substrate was found to be inferior to that of bonded as-atomized particles that were sprayed at the same conditions (Figure 6). Obviously, reduced ductility is not favorable for bonding and leads to brittle fracture of the sprayed particles upon impact or adiabatic shear instabilities cannot be reached due to deformation at higher strain rates. Further investigations on the deposition of 48–2–2 are ongoing with the Impact Spray System 5/11 using \( N_2-\)He mixtures as propellant gas as well as heat-treated feedstock with minimum oxygen contents.

Figure 6. Confocal laser microscopy images of 48–2–2 single particles sprayed (950 °C, 4 MPa, 20 mm) on polished 48–2–2 substrates. a) As-atomized feedstock, b) heat-treated feedstock. The arrow indicates the traveling direction of the gun.\(^{[49]}\)

### 3.3. Cold Spray of IN 718 and IN 738 for Repair Applications

#### 3.3.1. IN 718

For the CGS of this alloy, the maximum pressure (4 MPa) and temperature (950 °C) of the Oerlikon system with nitrogen as the only propellant gas was used.\(^{[42]}\) With this condition, highly dense coatings (porosity below 2%) could be produced. SEM micrographs of the coatings sprayed with a stand-off distance of 40 and 80 mm using coarse grit-blasting alumina grit (average particle size of 420–600 \( \mu m \)) and a rectangular (90°) spraying direction are shown in Figure 7.

Increased deposition efficiency is obtained for reduced stand-off distance. The deposition performance should be reduced if one considers the bow shock effect, which is the shock wave resulting from the supersonic gas flow impacting on the substrate.\(^{[78]}\) It is expected that the strength of this bow shock, which should reduce the velocity of the impinging particles, is getting larger for reduced spraying distance. One explanation for the increased efficiency at low stand-off distance might be the slightly higher substrate temperatures at 40 mm compared to 80 mm (240 °C compared to 220 °C).

The spray angle has a more pronounced effect than the stand-off distance. A reduction from 90° to 60° reduced the coating thickness by about 50%. This effect is at least partly a result of the reduced particle velocity perpendicular to the substrate.
In addition, the influence of different surface treatments of the substrates such as grit blasting and polishing was investigated. It turned out that the grit-blasting process gives better results for the coarser grit media. However, also in this study, it was obvious that the grit-blasting process led to alumina inclusions at the interface, which can reduce the bonding strength considerably. This was confirmed in more recent studies, also showing a large compressive stress level due to the blasting in the near-surface regions of the substrates.

In a further study, the mechanical properties of the coatings have been investigated.\(^{[43]}\) Vickers hardness, as well as indentation modulus, increased with coating thickness whereas porosity stayed rather constant. It was assumed that already-deposited splats are further affected by a peening effect of later-deposited particles, improving their adhesion. Astonishing here is that no pronounced effect on the porosity level could be observed. For the thickest coatings, Young's modulus values of 185 GPa and above were measured, which are close to the bulk value of 200 GPa.

As another property, the bonding strength was measured as it is of major importance for repair applications. It turned out that the bonding strength decreased considerably with increased coating thickness. For thin coatings in the thickness range of several hundred micrometers, the strength exceeded the strength of the used glue for the adhesion tests, which was about 70–80 MPa.

The behavior was explained by the rather high residual stress levels in the coatings. It was found that these stresses are typically compressive due to the peening effect during the CGS. The stresses in the coatings were measured by two different techniques. At first, a bending method was applied. Here, the curvature of a rather thin substrate after the coating was determined. By the Atkinson equation,\(^{[79]}\) the mean stress in the coating can be calculated. In addition, stress profiles have been measured by the hole-drilling method.\(^{[80]}\) Here, the relaxation of the strain was determined by strain gauges and from that based on calibration data and the materials' elastic constants the stresses were evaluated. In coatings with a thickness of about 700 \(\mu\)m, compressive stresses of 200 and 300–400 MPa were calculated using the bending and the hole drilling method, respectively.

These stresses can release during the debonding of the coating and promote crack growth. For a thicker coating, although the stress level remains rather constant, the amount of energy that releases (the so-called energy release rate) increases linearly with the coating thickness, which leads to the mentioned thickness dependence of the adhesion strength. A detailed study on this topic will be published in a separate article.

### 3.3.2. Cold Spray Repair of IN 738 with Laser-Structured Substrates

The strain energy release rate (G) is the driving force for spontaneous debonding of the coating and depends on the coating thickness and the in-plane stresses.\(^ {\text{[81]}}\) G rises linearly with the coating thickness, which leads to the delamination at a critical thickness. Singh et al.\(^ {\text{[43]}}\) found that this critical coating thickness for cold-sprayed IN 718 with grit-blasted substrates is about 1 mm. To improve the adhesion of the sprayed coatings, the rather new laser structuring technique of polished substrates, introduced by Kromer et al.\(^ {\text{[41]}}\) was tested. The roughness increases through the laser structuring process to a \(R_a\) value of 7.15 \(\mu\)m.

The resulting IN 738 repair coating with a thickness of more than 3.2 mm is shown in the confocal laser microscope (Keyence, Vh-9710) image in Figure 8. The reason for the excellent adhesion properties of the coating is correlated to the coating adhesion strengthening. Kromer et al. give a more detailed explanation.\(^ {\text{[41]}}\) The crack propagation through the interface kinks out if the recess angle of each crater is about 60° and the crack propagates into the coating. This leads to a cohesive and adhesive mixed-mode failure of the interface and the coating itself. The coating has a porosity of 1.6% (image analysis) and the interface shows good material intermixing and no cracks. The craters of the laser pretreatment are highlighted in the interface region.

### 3.4. Vacuum Plasma Spray Repair of CMSX-4

The following part introduces different approaches for the repair of single-crystalline turbine blades using VPS. The first section
examines the polycrystalline repair of single-crystalline CMSX-4 with a focus on the influence of the substrate temperature. The second part covers the single-crystalline repair using a directional annealing approach.

### 3.4.1. Polycrystalline Repair

To reduce the solidification rate of the splats, a heated sample holder was used during the coating process of a polished single-crystalline substrate at a temperature of 1000 °C.[56] The microstructure of the interface was analyzed by SEM and EBSD. **Figure 9a** represents an image of the forescatter diodes which reveals the low porosity level of the coating. The crystal orientation of the microstructure of the same area is shown in the EBSD mapping in **Figure 9b**. As already described in the literature,[82] molten powder particles nucleate at the single-crystal substrate due to the reduced solidification rate.

In parts of the coating, epitaxial crystal growth occurs. Some regions show the same crystal orientation as the substrate and reach several micrometers into the coating. No texture was observed in the entire layer. The determined grain sizes revealed no significant dependence on the spray parameters. However, the porosity increased when spray parameters that lead to a thicker bead were used.

Heat treatments combined with HIP and subsequent rapid quenching were conducted. The three-stage heat treatment, consisting of a solution annealing and two precipitation annealing steps, reduced the porosity below 1%. Grain growth occurred and coarsened the microstructure. The epitaxially solidified areas grew by several 10 μm into the repair coatings. The typical desired γ/γ’-microstructure is uniformly present, both in the repair layer and in the single-crystalline substrate. In further microstructure investigations, oxides were identified as Al₂O₃ and the impurity phase on the grain boundaries as a μ-type topologically close-packed phase.[56]

The measured oxide content can be related to the grain size by an oxygen analysis of the heat-treated samples. The samples with spray parameters that lead to a thick bead have a lower oxide content. During spray processes with thicker beads, a smaller surface area of the deposited layer is exposed to oxygen uptake from the ambient atmosphere. The higher oxide content at thin beads suppresses grain growth during heat treatments and results in grains in the 5 μm range. Samples prepared with a thick bead grew during the heat treatment to a grain size larger than 10 μm.[56]

This repair process can be used to produce polycrystalline repair layers on damaged parts with small structural deviations of the original component dimensions and a low mechanical load regarding the layer thickness. In addition, the formation of interdiffusion phases due to the use of the same alloys is excluded. The layers have a porosity below 1% and a grain size between 5 and 11 μm.
3.4.2. Single-Crystalline Repair

Annealing experiments were conducted to investigate the influence of temperature on grain boundary mobility. The samples were subjected to a HIP heat treatment below the γ'-solution temperature to reduce the porosity before the actual experiment. The grain size only increased slightly, due to suppressed grain growth. The actual annealing experiments were conducted above the γ'-solution temperature in a temperature range between 1292 and 1301 °C at varying durations. In this temperature range, abnormal growth was observed, resulting in bimodal grain size distributions.

A directional annealing experiment was set up to stimulate grain growth from the SX substrate into the repair coating. The necessary boundary conditions to achieve this require a temperature range with high grain boundary mobility (abnormal grain growth), a temperature gradient, and a movement of the repair coating into a hot zone.[81] In CMSX-4, this is defined by the isotherm of the γ'-solution temperature, as abnormal grain growth begins at this temperature.[84,85]

In a first attempt, the desired boundary conditions could be fulfilled within the transition zone of a tube furnace. A set-up consisting of an isolated sample attached to a heat conductor was pushed to several defined positions within the furnace with the aim to create a movement into the hot zone. An EBSD analysis shows a successfully directed annealing, resulting in a columnar grain structure in the upper left of Figure 10. The largest grain has grown from a grain size of 5 μm to a length of several 100 μm and an average width of more than 100 μm. Due to the unprecise implementation, grain growth could not be initiated at the SX substrate. As already shown during the development of the crystal structure of turbine blades consisting of Ni superalloys, the columnar crystal structure is a prestige for the SX crystal structure.[86]

For the SX repair, directed grain growth must begin in the SX substrate. Normally, oxides at an interface are a problem for grain growth. For the repair coatings introduced before, the oxide concentration at the interface is low since epitaxially grown areas are present. A further reduction of the oxide concentration would have a positive effect on the propagation of the epitaxially grown areas. For the technical implementation, the velocity of the γ'-isotherm must be adapted to the maximum grain boundary velocity; otherwise the grain structure will only coarsen (lower part of Figure 10). Calculations resulted in a maximum grain boundary velocity of several millimeter per minute for grain sizes in the range of several micrometers, which defines the maximum feasible velocity of the γ'-isotherm.

In case of severe damage to the turbine blades, their repair requires the application of a thick layer to enable them to withstand high thermal loads. Such damage could be repaired by VPS and directional recrystallization, stimulating grain growth from the SX substrate.

4. Conclusions

In this study, we have demonstrated the potential of repair coatings produced by CGS and VPS for polycrystalline and single-crystalline aerospace components, respectively. Technologically important alloys with different properties were investigated and the following conclusions can be drawn: 1) The indentation modulus of cold-sprayed IN 718 coatings was close to the bulk values. The measured relatively high residual compressive stresses of 200–400 MPa were identified as a possible reason for the observed coating thickness dependence of the adhesion strength. 2) The preparation of the IN 738 substrate material with laser structuring improved the adhesion of the repair coating significantly and enabled the deposition of a 3 mm thick layer. 3) The deposition of Ti-48Al-2Cr-2Nb by CGS is a challenging task, but nevertheless, thin coatings of several tens of micrometers were deposited. Annealing of the feedstock material to change the feedstock properties did not lead to an improvement of the coatings and further investigations are necessary to increase the deposition efficiency. 4) VPS in combination with a post heat treatment has the potential to repair single-crystalline CMSX-4 turbine blades in the future. After the deposition process locally epitaxial grown grains are present in the coating. These regions can grow during a post heat treatment and potentially lead to a single-crystalline repair coating.

Furthermore, the in-flight particle velocities of three spherical IN 718 powders with different particle size distributions were determined depending on inlet gas pressure and inlet gas temperature. The critical velocities were identified and it was found that the deposition efficiency builds a unique function of the ratio of particle impact and critical velocities for various spray parameters and particle sizes.

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