High-temperature wear behaviour of borided Inconel 718 HVOF coatings

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Abstract. Increasing demands on component performance and efficiency require continuous development efforts in existing material systems and concepts. In addition to the specific material properties, economic aspects have to be taken into account. Thermochemical treatments of iron- and nickel-base alloys allow for a distinct improvement of hardness and wear resistance. The process of boriding enables the highest hardness values and the formation of thermally stable precipitates. Especially nickel-base alloys are suitable for high temperature applications. An economic application of these alloys can be achieved by applying coating technologies and limiting the material usage to the surface. High-velocity oxygen fuel thermal spraying of the nickel-base alloy Inconel 718 and subsequent powder-pack boriding is conducted. Furthermore, the influence of a solution annealing step prior to the boriding process is investigated with the motivation to achieve a homogenisation of the coating. A successful diffusion enrichment and the formation of a precipitation layer could be achieved. The investigation of the resulting properties revealed a distinct increase of hardness and an improvement of wear resistance tested under reciprocating conditions in a wide temperature range.

1. Introduction
Nickel-base alloys are widely applied due to their mechanical properties especially at high temperatures. Furthermore, they exhibit a high corrosion resistance also under harsh environmental conditions e.g. in contact with seawater or at elevated temperatures. However, the wear resistance is rather limited [1–4]. Thermochemical treatments offer a possibility to enhance the property profile. Thermochemical processes can be divided into two main groups. Interstitial hardening can be achieved by nitrogen and carbon enrichment [5, 6]. For higher demands and the application at elevated temperatures precipitation hardening processes are used. The highest hardness values and the formation of thermally stable precipitates can be achieved by boriding. The most common process variant is powder-pack boriding. Besides nickel-base alloys this process is used for various steels and further recently developed materials [7–12]. However, thermochemical treatments are mainly limited to cast alloys. Due to the relatively high cost of nickel-base alloys, an economic application can be achieved by a functional division and the application of steel substrates.

Coating processes like laser cladding and thermal spraying are established for a wide range of technical applications, especially for wear and corrosion protection. Laser cladding of the nickel-base alloy Inconel 625 caused dilution of Fe resulting in a degradation of corrosion properties depending on the coating parameters [13]. Thermal spray processes enable the economic deposition of coatings within a wide range of coating thicknesses. Due to the relatively low thermal input and the absence of metallurgical bonding, the chemical composition of the feedstock material can be retained. For coating
processes conducted under normal atmosphere typical structural defects e.g. pores and oxides are formed. The extent of these defects can be reduced by the application of high kinetic processes. For the deposition of metallic coatings and cemented carbides the industrially most widely used process is high-velocity oxygen fuel (HVOF) thermal spraying [14, 15].

The process combination of boriding thermally sprayed coatings of various iron- and nickel-based coatings has been investigated in previous studies. However, the resulting property profile was only characterised at ambient temperature [16, 17].

In the current investigations a process combination of depositing the nickel-base alloy Inconel 718 by HVOF thermal spraying and thermochemical treatment by powder-pack boriding is considered. With the objective to improve internal bonding and reduce heterogeneities one group of the thermally sprayed coatings was solution annealed prior to the thermochemical treatment. An adapted boriding routine was applied to avoid the formation of silicon rich phases. The wear behaviour was investigated in a wide temperature range up to 900 °C under reciprocating conditions in dependence of the treatment condition.

2. Experimental

Coatings of the nickel-base alloy Inconel 718 were produced by applying the high kinetic thermal spray process HVOF. The commercially available powder NI-202-3 (Praxair Inc., Danbury, CT, USA) was used as a feedstock material. A particle size range of -45 +16 µm was specified by the manufacturer. The stainless steel EN 1.4404 (AISI 316L) was used as a substrate material. Pre-treatment was conducted by grit blasting using the blasting medium Alodur EK F 24 with a particle size of -850 +600 µm. A blasting pressure of 2.5 bar, a distance of 200 mm under an angle of 70° was applied. Subsequently, ultrasonic cleaning in ethanol has been carried out. For the coating process the liquid-fuelled HVOF system K2 was used (GTV Verschleißschutz GmbH, Luckenbach, Germany). The spraying parameters are summarised in Table 1.

| O2       | kerosene | λ     | nozzle | powder feed rate | spraying distance | relative traverse speed | spray path offset |
|----------|----------|-------|--------|------------------|-------------------|-------------------------|------------------|
| 900 l/min| 26 l/h   | 1.0   | 150/14 | 2 x 50 g/min     | 350 mm            | 1.0 m/s                 | 5 mm             |

A final coating thickness of approximately 270 µm was measured after 14 single-layers. For the thermochemical treatment one part has been considered in as-sprayed condition, whereas for the other group a heat treatment has been conducted prior to boriding with the objective to reduce heterogeneities and improve internal bonding. For the heat treatment the vacuum furnace Torvac 12 Mark IV (10^4 mbar) has been used. The samples were heated to a temperature of 1100 °C with a rate of 10 K/min. Holding the temperature for a duration of 1 h was followed by unregulated furnace cooling. Boriding has been conducted in a powder-pack process by the company BorTec GmbH & Co. KG with the parameters summarised in Table 2.

| boriding agent | temperature | duration | atmosphere |
|----------------|-------------|----------|------------|
| Ekabor® Ni     | 900 °C      | 5 h      | Argon      |

Cross-sections were prepared by standard metallographic procedures. Light microscopic investigations have been performed with an Olympus GX51 (Olympus, Shinjuku, Japan). For the visualisation of material contrast the scanning electron microscope (SEM) LEO 1455VP (Zeiss, Oberkochen, Germany) equipped with a backscattered electron (BSE) detector has been applied. The chemical composition of the feedstock powder and coatings has been measured by X-ray fluorescence spectroscopy (XRF) using
a Fischer X-Ray XAN (Fischer, Sindelfingen, Germany) operated with an acceleration voltage of 30 kV. The phase formation was determined by X-ray diffraction (XRD) with a D8-discover diffractometer (Bruker AXS, Billerica, MA, USA). Co Kα radiation and a diffraction angle range of 20° to 130° were used.

Elemental depth profiles were determined by glow discharge spectroscopy (GDOS) with a GDA 750 (Spectrum Analytik GmbH, Hof, Germany) using an anode diameter of 2.5 mm, 800 V, 25 mA and 3 hPa argon pressure.

Selective hardness measurements of the borided surface layer and the coating have been conducted by nanoindentation using an UNAT tester (ASMEC GmbH, Radeberg, Germany), equipped with a Berkovich B16 indenter (tip radius: 0.394 µm). A load of 10 mN has been applied for a holding time of 5 s. The Vickers hardness was deduced from the measured indentation depth using the InspectorX testing software version 3 (ASMEC GmbH, Radeberg, Germany). At least ten single measurements have been considered for the calculation of the average Vickers microhardness and the standard deviation.

The wear behaviour was investigated under reciprocating conditions in a wide temperature range using an SRV tribometer (Optimol Instruments GmbH, Munich, Germany) with the parameters summarised in Table 3.

| force | frequency | duration | amplitude | counter body | temperature |
|-------|-----------|----------|-----------|--------------|-------------|
| 26 N  | 40 Hz     | 900 s    | 0.5 mm    | Al₂O₃ (ø 10 mm) | 25 °C; 500 °C; 650 °C; 800 °C; 900 °C |

Three measurements have been conducted to determine the average value for every parameter set. The resulting wear tracks were investigated with the optical profilometer MikroCAD 3-D (LMI Technologies Inc., Burnaby, Canada).

3. Results and Discussion

3.1. Feedstock and coating characterisation

Prior to the coating process the feedstock powder was investigated in detail. The surface and cross-sections were investigated in SEM. Representative images are shown in Figure 1.

![Figure 1. SEM images of Inconel 718 powder: a) surface (SE) and b) cross-section (BSE).](image)

The gas atomized powder has a characteristic spherical shape. A dendritic structure can be observed in the cross-section. Due to the low porosity the powder is well suited for the HVOF process.
The microstructure of the coating in as-sprayed state was investigated in cross-sections by light microscopy. Furthermore, the influence of the additional heat treatment step was investigated. Cross-sections of the coatings in dependence of the treatment condition are shown in Figure 2.

![Figure 2. Light microscopy of Inconel 718 HVOF coatings: a) as-sprayed state and b) solution annealed state.](image)

A typical structure occurs for the coating in as-sprayed state. Single spray particles can be observed, which are flattened due to the high kinetic energy of the HVOF coating process. These particles are well bonded. At the boundaries oxides appear due to the interaction with the atmosphere in the coating process. Only a minor content of porosity is formed.

Solution annealing causes an altered coating structure. Particle boundaries are dissolved and the oxide lamellae coagulate, resulting in the reduction of coating defects and a more homogeneous state.

The chemical composition was measured for the feedstock powder and the coating in as-sprayed as well as solution annealed state by XRF. The results are summarised in Table 4.

|               | Ni   | Cr   | Fe   | Nb  | Mo  | Ti  |
|---------------|------|------|------|-----|-----|-----|
| powder        | 52.5 | 19.2 | 18.5 | 5.3 | 3.5 | 0.9 |
| coating (AS)  | 52.5 | 19.7 | 19.0 | 4.7 | 2.9 | 1.2 |
| coating (SA)  | 53.2 | 16.9 | 19.3 | 4.8 | 3.1 | 2.6 |

Processing of the powder by HVOF thermal spraying and subsequent solution annealing causes only minor changes of the chemical composition. The chemical composition is within the specification of the manufacturer. For the coating in as-sprayed state the molybdenum and niobium content are slightly reduced in comparison to the feedstock powder. The chromium concentration is decreased after the solution annealing treatment. The heat input in the coating and heat treatment process result in diffusion, oxidation or evaporation of individual components and minor changes of the chemical composition.

In general, the changes in the composition are small and the composition of the alloy Inconel 718 has been largely preserved.

3.2. Boriding treatment
The microstructure of the boron-enriched surface layer was investigated in cross-sections using an SEM equipped with a BSE detector to show material contrast. Representative images of the coatings borided in as-sprayed and solution annealed state are shown in Figure 3.
Figure 3. Cross-sections of borided Inconel 718 HVOF coatings: a) borided in as-sprayed state and b) borided after solution annealing.

For the coating borided in as-sprayed state, a uniform dark layer with a thickness of approximately 20 µm can be observed at the surface. The low intensity of backscattered electrons indicates a high concentration of elements with a low atomic number. Thus, an enrichment with boron at the surface can be assumed. The enriched layer has a mostly homogeneous thickness and is well bonded to the underlying material. However, the structure of the thermally sprayed coating comprised of single particles and structural defects can still be observed. In the underlying material the dendritic structure of the initial feedstock powder is retained.

For the coating borided in solution annealed state also a diffusion enriched surface layer is formed. The typical structure of thermally sprayed coatings is partially dissolved after the additional annealing treatment. In contrast to the coating borided in as-sprayed state no dendritic structure occurs, showing that the heat treatment caused homogenisation.

For the investigation of the elemental distribution, depth profiles have been determined by GDOS measurements. The depth profiles of boron and the main alloying elements nickel and chromium are shown in Figure 4.

Figure 4. Elemental depth profiles determined by GDOS for borided Inconel 718 HVOF coatings: a) as-sprayed coating and b) solution annealed coating.

For the coatings borided in as-sprayed state a high concentration of boron was determined at the surface. Until a surface distance of approximately 20 µm a high level of boron concentration is retained. At higher depths a sharp decline occurs. With decreasing boron concentration, the concentration of the main alloying elements nickel and chromium increases. However, a continuous transition from the boron-enriched layer to the thermally sprayed nickel-base coating occurs.
For the coatings borided in solution annealed state similar elemental depth profiles were recorded. A high concentration of boron was determined at the surface, which is retained until a surface distance of approximately 20 µm.

For the evaluation of the phase formation in dependence of the conducted treatment, phase analyses by XRD have been conducted. The resulting diffractograms are shown in Figure 5.

![Diffractograms of Inconel 718 HVOF coatings in dependence of the treatment condition.](image)

**Figure 5.** Diffractograms of Inconel 718 HVOF coatings in dependence of the treatment condition.

For the coating in as-sprayed state solely diffraction peaks of a phase with fcc structure occur. Solution annealing results in no distinct changes of the diffractogram, showing that no phase transformation occurred. However, in comparison to the as-sprayed state narrower diffraction peaks can be observed. This behaviour is caused by grain coarsening as a result of the heat treatment.

Boriding of the coatings results in a distinct change of the diffractograms. No diffraction peaks of the fcc phase can be observed after diffusion enrichment with boron. For the coating borided in as-sprayed state high intensity diffraction peaks of the tetragonal precipitates Ni$_2$B appear. Furthermore, diffraction peaks with low intensity are present, which can be assigned to the orthorhombic Ni$_3$B phase. Partially overlapping of the diffraction peaks with other phases occurs. The formation of the phase Ni$_4$B$_3$, which was proven in previous studies for the borided bulk Inconel 718, could not be detected [18]. Additional minor diffraction peaks are not assignable, showing that minor contents of additional phases were formed. According to the phase analyses by XRD the formation of silicides was prevented by the application of a process routine adapted for nickel-base alloys. A successful formation of precipitates in the boron-enriched surface layer could be achieved.

The additional solution annealing step prior to the boriding did not cause distinct changes of the respective diffractogram. Hence, the phase formation was not influenced.

To investigate the influence of the treatment condition on the resulting properties, hardness measurements have been conducted. The results are summarised in Table 5.
Table 5. Microhardness HV 0.001 of Inconel 718 HVOF coatings borided in as-sprayed and solution annealed state.

|                | as-sprayed coating | solution annealed coating |
|----------------|--------------------|---------------------------|
| B-rich layer   | 2110 ± 120         | 2010 ± 80                 |
| unaffected material | 500 ± 40         | 440 ± 20                  |

Boriding causes a distinct increase of hardness in comparison to the unaffected material due to the successful formation of a precipitation layer. For the coating borided in as-sprayed state a high hardness of 2110 HV 0.001 was determined. A high standard deviation occurs due to the presence of structural defects in the precipitation layer. For the coating, solution annealed prior to the boriding treatment, the hardness is slightly reduced. Also the standard deviation is reduced indicating the formation of a more homogeneous state and the reduction of structural defects, which was proven by microscopic investigations. Solution annealing reduces the hardness measured in the unaffected material, which can be caused by grain coarsening and the reduction of residual stress.

3.3. Wear behaviour

The results of the wear investigations under reciprocating conditions in dependence of the test temperature and treatment condition are summarised in Figure 6.

![Figure 6](image)

Figure 6. Results of reciprocating wear investigations for Inconel 718 HVOF coatings in a temperature range of 25 °C to 900 °C in dependence of the treatment condition.

The investigations at room temperature reveal a similar wear resistance for the as-sprayed and solution annealed coating. Boriding causes a reduction of the wear depth and hence an increased wear resistance. However, the wear depth exceeds the thickness of the diffusion enriched surface layer, which was determined by SEM and GDOS measurements, showing that failure occurred.

With an increase of temperature, the wear depth distinctly increases for the coating in as-sprayed and solution annealed state. This effect can be caused by thermal softening. For temperatures of ≥ 800 °C the wear depth is reduced. This effect is most pronounced for the coatings in solution annealed state, resulting in an improvement of wear resistance in comparison to the as-sprayed state. Possible reasons are the enhanced internal bonding and the reduction of heterogeneities.
The coatings in borided state show a less distinct dependence on test temperature. This behaviour indicates that thermally stable precipitates were formed. For temperatures of $\geq 800 ^\circ C$ the wear depth is reduced. This effect is most pronounced for the coatings in solution annealed and borided state.

For the investigation of the underlying wear mechanisms the wear tracks were investigated by SEM using a BSE detector to visualise material contrast. Representative images in dependence of the treatment condition and test temperature are shown in Figure 7.

![Figure 7](image-url)

**Figure 7.** Surface (SEM/BSE) of the HVOF Inconel 718 coatings after reciprocating wear investigations at room temperature (left), 650 °C (middle) and 900 °C (right): a) solution annealed state, b) borided state and c) solution annealed and borided state.

The wear tracks of the coatings in solution annealed state exhibit a distinct material contrast. Dark areas represent a low average atomic number. These areas can be assigned to oxides, whereas the bright appearing areas can be assigned to the metallic coating. The surface is partially covered with oxides showing a rough surface. Hence, a permanent formation and removal of oxides can be assumed. At increased temperature of 650 °C, where the maximum wear depth was determined, the wear track exhibits a changed appearance. The surface is still partially covered with oxides. However, a relatively smooth oxide layer with grooves in wear direction is formed. With a further increase of test temperature (900 °C) the content of the surface covered with oxides increases, enabling a protection of the underlying coating. Partially breakouts and failure of the oxide layer occurs.

For the coating borided in as-sprayed state, the investigations at room temperature result in a distinctly changed appearance. Partially oxides are formed. Furthermore, cracks can be observed,
showing that reciprocating load causes failure of the surface layer. For the test conducted at a temperature of 650 °C the wear track is partially covered with oxides, showing a relatively smooth surface. Higher test temperatures result in the formation of a widely closed oxide film. Grooves in wear direction appear, indicating abrasive wear of the oxide film. However, protection of the underlying material could be achieved. For the coatings borided after initial solution annealing of the coating, distinct oxide formation was observed after testing at room temperature. However, a rough surface appears showing that permanent formation and removal of oxides occurs. At elevated temperature wide areas are covered with a smooth oxide layer, protecting the underlying material.

4. Summary and Conclusions
The process combination of powder-pack boriding thermally sprayed Inconel 718 coatings was successfully realised. A homogenisation of the coating could be achieved by an additional solution annealing step. The chemical composition could be retained by the chosen process route. Powder-pack boriding resulted in the formation of a homogeneous precipitation layer mainly comprised of the tetragonal phase Ni_{2}B. No delamination occurred and the formation of silicon-rich phases could be prevented by the application of an adapted boriding routine. A significant increase in hardness could be achieved by the thermochemical treatment. Detailed investigations on the wear behaviour under reciprocating conditions have been conducted in a wide temperature range up to 900 °C. A distinct improvement of wear resistance in the investigated temperature range was determined. Furthermore, the temperature dependence was reduced for the borided coating, showing that thermally stable precipitates were formed. However, the wear depth exceeded the thickness of the precipitation layer, hence an adaption of the boriding process is required to enable a higher thickness of the precipitation layer. The combination of solution annealing and subsequent boriding resulted in a further increase of wear resistance. Detailed investigations of the wear tracks revealed the formation of stable oxide films at elevated temperatures, enabling the protection of the underlying material.

The results show that the investigated process combination is a conceivable option for the replacement of bulk nickel-base alloys. Further studies have to be conducted to determine the effect of the investigated process combination on the resulting corrosion properties. A distinct reduction of process cost and duration can possibly be achieved by the integration of the solution annealing treatment in the thermochemical process.

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