Microstructure and Wear Behavior of the High-Velocity-Oxygen-Fuel Sprayed and Spark Plasma Sintered High-Entropy Alloy AlCrFeCoNi

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High-entropy alloy AlCrFeCoNi powder with a metastable body centered cubic (bcc) structure is produced by inert gas atomization. This state is largely preserved after processing the powder by high-velocity-oxygen-fuel (HVOF) thermal spraying. A heat treatment is conducted with the objective to form a duplex structure comprising a ductile face centered cubic (fcc) phase. The formation of an additional fcc phase is accompanied by a decrease in hardness and a significant improvement of wear resistance. The alternative processing route, spark plasma sintering (SPS), causes a duplex bcc and fcc structure. Detailed analyses of phase formation and wear behavior for all production routes contribute to a better understanding of microstructural effects in high-entropy alloys.

1. Introduction

High-entropy alloys (HEAs) have been the focus of recent research efforts. One of the base alloys forming a single-phase structure is the equimolar alloy CrMnFeCoNi. HEAs with a face centered cubic (fcc) structure typically exhibit not only high ductility but also relatively low strength and hardness.[1] The influence of additional alloying elements to stabilize a body centered cubic (bcc) structure has been investigated.[2,3] One alloying element with a strong influence on phase formation is aluminum. The formation of a single-phase bcc (B2) structure could be achieved for the equimolar alloy AlCrFeCoNi in as-cast state.[4,5] However, a subsequent heat treatment causes the formation of additional phases and an increase in heterogeneity. Detailed investigations were presented by Munitz et al.[6] Heat treatment at a temperature of 850 °C formed an additional σ-phase, causing embrittlement. At increased heat treatment temperature, decomposition of the σ-phase occurs and an additional ductile fcc phase is formed. This duplex structure causes advantageous mechanical properties, tested in a compression test.[6] Liang et al. investigated the processing of the equimolar alloy AlCrFeCoNi by gas atomization. Whereas a metastable single bcc phase was formed in the initial powder, heat treatment of the powder at a temperature of 900 °C caused additional fcc and σ-phases depending on the duration time.[7] In addition to the chemically ordered bcc (B2) phase, detailed investigations on the alloy AlCrFeCoNi revealed a disordered bcc phase.[8,9] Previous investigations have focused mainly on the alloy production from the liquid state. However, laboratory scale arc-melting limits the sample dimensions, and typical casting defects and segregation occur.[1,10]

Alternatively, bulk alloys can be produced powder metallurgically. The process of spark plasma sintering (SPS) offers a possibility for the fast production of dense bulk alloys.[11] Usually, mechanically alloyed or atomized powder is applied as feedstock. For the production of a homogeneous feedstock, mechanical alloying requires long processing times. Furthermore, the particles usually exhibit an irregular morphology, and there is a high risk of contamination in the milling process.[12,13] Zhou et al. successfully produced dense AlCrFeCoNi material by SPS of atomized powder. A multiphase state comprising fcc and bcc phases was formed. By adjusting the process parameters, the phase contents and therefore the resulting mechanical properties can be adjusted.[12]

Another promising approach uses coating technologies where the material usage is limited to the surface. Thermal spray processes enable the coating of various substrates due to the relatively low thermal input.[14] Cheng et al. investigated the processing of gas-atomized AlCrFeCoNi powder by atmospheric plasma spraying (APS). Thermal spraying of fine powder (~60 ± 10 μm) caused an increase in heterogeneity and the formation of a multiphase structure.[15] High kinetic processes enable the deposition of coatings with low porosity and oxide fraction. In industry, the most used process is high-velocity-oxygen-fuel (HVOF) thermal spraying. First investigations of the deposition of HEAs by HVOF have been successfully conducted.[17,18] In addition, other high kinetic thermal spray processes enable the deposition of HEAs by HVOF.
processes have been applied for the processing of HEAs. Anupam et al. investigated the processing of mechanically alloyed AlCrFeCoNi powder by means of cold gas spraying. The metastable phase state of the feedstock powder could be retained due to the low thermal input in this particular thermal spray process.\textsuperscript{[19]}

Despite various studies on phase formation and resulting mechanical properties of the alloy AlCrFeCoNi, the influence on the wear behavior has not been investigated in detail yet. Therefore, by operating different production routes and heat treatments, the influence of the microstructure and phase formation on the wear behavior is considered in detail in the current investigation.

### 2. Experimental Section

Feedstock of the equimolar alloy AlCrFeCoNi was produced by inert gas atomization using argon as a process gas. Subsequently, the powder was classified to adjust a particle size range of $-45 + 15 \mu m$ ($\sim d90 + d10 \mu m$). The particle size distribution was measured by laser diffraction analysis using a Cilas 920 device (Cilas, Orléans, France). For the investigation of the microstructure, cross sections were prepared by standard metallographic procedures and investigated in the scanning electron microscope (SEM) LEO 1455VP (Zeiss, Jena, Germany) operated with an acceleration voltage of 25 kV. A secondary electron (SE) detector was used for the investigation of the powder morphology. The microstructure of the specimen was visualized by applying a four-quadrant solid-state electron backscatter detector (BSD). The signals of the four quadrants were added, thus excluding topography contrast. The chemical composition was determined by means of the installed energy-dispersive X-ray spectroscopy (EDS) system EDS GENESIS (EDAX, Mahwah, NJ, USA). The phase formation was analyzed by X-ray diffraction (XRD) measurements using a D8 Discover diffractometer (Bruker AXS, Billerica, MA, USA) equipped with a 1D Lynxeye XE detector (Bruker AXS, Billerica, MA, USA). Co Kα radiation ($U: 40 kV; I: 40 mA$) was used for all measurements, which were conducted in the diffraction angle range (2θ) of 20--130°. The formed phases were assigned according to the powder diffraction file (PDF) database 2014 (International Centre for Diffraction Data).

Prior to coating by thermal spraying, austenitic stainless-steel substrates (EN 1.4404) were prepared by corundum blasting with Alodur EK F 24. A particle size range of $-850 + 600 \mu m$ is specified by the manufacturer. The substrates were pretreated with a pressure of 2.5 bar at a distance of 200 mm under an angle of 70°. Subsequently, the substrates were cleaned ultrasonically in an ethanol bath. The coatings were deposited with the HVOF system K2 (GTV Verschleisschutz GmbH, Luckenbach, Germany). The parameters are shown in Table 1.

The microstructure and phase formation of the coatings were investigated similar to the aforementioned feedstock characterization. Solution annealing was conducted in a vacuum furnace (Torvac 12 Mark IV) at a pressure of $\leq 10^{-4} \text{mbar}$ for a duration time of 4 h at a temperature of 1050°C.

Furthermore, the feedstock powder was compacted by SPS in an SPS KCE FCT-HP D 25-SI (FCT Systeme GmbH, Frankenblick, Germany). Samples with a diameter of 40 mm and a height of 6 mm were produced using a graphite die. A graphite foil with a thickness of 0.3 mm was inserted between powder and punches. To avoid reactions with the atmosphere during the sintering process, the recipient was flushed with argon and evacuated ($< 1 \text{mbar}$) twice. During the sintering process, a pressure of 50 MPa was applied. The temperature profile is composed of a sintering step at a temperature of 1050°C for 10 min followed by a cooling step with a cooling rate of approximately 150 K min$^{-1}$ until 300°C.

Detailed investigations of the microstructure and phase formation were conducted by electron backscatter diffraction (EBSD). Therefore, an SEM NEON 40 EsB (Zeiss, Jena, Germany) equipped with an EBSD system OIM 5.2 (EDAX TSL, Mahwah, NJ, USA) was used. All measurements were conducted with an acceleration voltage of 20 kV with a step size of 0.05 μm in a field of 44 μm × 35 μm. A slight clean-up procedure was conducted for all measurements. This procedure comprises a neighbor confidence index (CI) correlation, grain CI standardization with a minimum CI of 0.1, and grain dilation with one iteration step. Remaining measurement points with a CI < 0.1 were considered for generating the phase and orientation distribution maps. The reference orientation was [100]. The phase fractions were determined from the phase maps using the integrated image analyses.

The microhardness (Vickers hardness HV 0.1) of the coatings in as-sprayed and solution annealed condition as well as the powder metallurgically produced bulk alloy was investigated using a Wilson Tukon 1102 device (Buehler, Uzwil, Switzerland). Ten single measurements have been conducted for each state. A wide range of tribological conditions was considered to determine the influence of the production route and phase formation. Therefore, various laboratory scale wear tests have been conducted. The sliding wear behavior was investigated in ball-on-disk tests using a Tetra Basalt Tester (Tetra, Ilmenau, Germany). For the investigation of the reciprocating wear behavior, a Wazau SVT 40 device (Wazau, Berlin, Germany) was utilized. Furthermore, the abrasive wear behavior was investigated by scratch tests with a CSM Revetest-RST device (CSM Instruments SA, Peseux, Switzerland). All measurements were conducted at room temperature with the parameters shown in Table 2.

### Table 1. HVOF thermal spray coating parameters.

| Parameter          | Value          |
|--------------------|----------------|
| $O_2$              | 850 L min$^{-1}$ |
| Kerosene           | 22.5 L h$^{-1}$  |
| $\lambda$          | 1.1            |
| Ar                 | $2 \times 11$ L min$^{-1}$ |
| Nozzle             | 100/12         |
| Powder feed rate   | $2 \times 35$ g min$^{-1}$ |
| Spraying distance  | 360 mm         |
| Relative traverse speed | 1.0 m s$^{-1}$ |
| Spray path offset  | 5 mm           |
| Coating layers     | 15             |

### Table 2. HVOF thermal spray coating parameters.

| Parameter          | Value          |
|--------------------|----------------|
| $O_2$              | 850 L min$^{-1}$ |
| Kerosene           | 22.5 L h$^{-1}$  |
| $\lambda$          | 1.1            |
| Ar                 | $2 \times 11$ L min$^{-1}$ |
| Nozzle             | 100/12         |
| Powder feed rate   | $2 \times 35$ g min$^{-1}$ |
| Spraying distance  | 360 mm         |
| Relative traverse speed | 1.0 m s$^{-1}$ |
| Spray path offset  | 5 mm           |
| Coating layers     | 15             |
The wear tracks of the ball-on-disk test were evaluated by tactile measurements using a Hommel-Etamic T8000 device (Jenoptik, Villingen-Schwenningen, Germany) to determine the wear depth. For the evaluation of the reciprocating wear and scratch test, a laser scanning microscope (LSM) Keyence VK-X200 (Keyence, Osaka, Japan) has been applied.

3. Results and Discussion

3.1. Feedstock Characterization

Powder of the equimolar alloy was produced by inert gas atomization. Laser diffraction analyses reveal a particle size range of \( \pm 42 + 14 \mu m \) \((-d90 + d10\) and a mean particle diameter of 26 \( \mu m \) \((d50)\), which is in good accordance with the intended values. The powder particles exhibit a predominantly spherical shape, as shown in the SEM image (Figure 1a). This morphology is typical for gas-atomized powder. A BSD image of a metallographic cross section shows the dendritic solidification of the powder particles, but no distinct material contrast occurs (Figure 1b).

3.2. Chemical Composition

The mean chemical composition was determined by EDS. The average values of the feedstock powder, coatings, and SPS samples are shown in Table 3. No distinct deviation of the measured chemical composition occurs in comparison with the nominal equimolar composition. Deviations are predominantly \( \leq 1 \text{ at\%} \), which is in the range of the accuracy of the method. Potentially, a slightly reduced chromium content after the solution annealing step can be stated which is the case for the powder metallurgically produced bulk alloys, too.

### Table 2. Wear test parameters.

|                         | Ball-on-disk test | Reciprocating wear test | Scratch test |
|-------------------------|-------------------|-------------------------|--------------|
| Force                   | 20 N              | 26 N                    | 1 – 200 N    |
| Radius                  | 5 mm              | 40 Hz                   | 2.5 mm min\(^{-1}\) |
| Speed                   | 96 RPM            | 900 s                   | 5 mm         |
| Cycles                  | 15 916            | 0.5 mm                  | 916 Amplitude |
| Counter body            | \( \text{Al}_2\text{O}_3 \) (ø 6 mm) | \( \text{Al}_2\text{O}_3 \) (ø 10 mm) | Rockwell C |

### Table 3. Mean chemical composition of the equimolar alloy AlCrFeCoNi in at\%, measured by EDS.

|        | Al  | Cr  | Fe  | Co  | Ni  |
|--------|-----|-----|-----|-----|-----|
| Powder | 20.0| 19.3| 20.9| 18.8| 21.1|
| HVOF coating (as-sprayed) | 19.8| 18.8| 20.9| 19.0| 21.5|
| HVOF coating (solution annealed) | 21.7| 18.1| 21.0| 18.3| 20.9|
| SPS    | 22.9| 17.4| 19.6| 20.2| 20.0|

3.3. Phase Formation and Microstructure

The phase formation was examined by XRD. The resulting diffractograms of the feedstock powder, the coatings, and the bulk alloy produced by SPS are shown in Figure 2. The diffractogram of the HVOF coating in as-sprayed condition exhibits similar intensity maxima compared with the feedstock powder, showing that no phase transformation occurred. The singular bcc (B2) structure is in accordance with previous investigations on the arc-melted equimolar alloy AlCrFeCoNi in as-cast state.\(^{[20]}\) A minor diffraction peak occurs at a diffraction angle of 47.0° due to the interaction with low intensity Co K\(\beta\) radiation that has not been filtered completely. The XRD pattern of the HVOF coating in the solution annealed state displays additional maxima apart from the major diffraction peaks of the bcc phase with B2 structure. These maxima can be assigned to an fcc phase with B2 structure. A similar diffractogram was obtained for the powder metallurgically produced bulk alloy. In addition to the bcc phase with B2 structure, additional diffraction peaks of an fcc phase with A1 structure appear.

The microstructure of the coatings in as-sprayed and solution annealed state as well as the bulk alloy produced by SPS was
The coating in the as-sprayed state (Figure 4a) is composed of a fine-grained lamellar structure, showing that the particles mostly have been melted and quenched in the thermal spray process. Slightly deformed initial feedstock particles can be observed in the maps incidentally as well. Due to the presence of pores, cracks, and oxides, not all areas could be indexed which is shown in the maps (right-hand side of Figure 4). Grain boundaries (black) and twin boundaries (white) are shown in both illustrations. Despite the relatively fast powder metallurgical processing by SPS, an additional fcc phase is formed. The fcc phase content of ≈32% is reduced compared with the solution annealed coating. This can be explained by the lower input of thermal energy in comparison with the heat treatment of solution annealed coatings.

The bulk alloy produced by SPS is dense and without oxide contamination (Figure 3c). Partially, former surfaces of the feedstock powder particles can be noted, showing that no melting of the feedstock powder occurred. Despite the relatively short processing time, the initial homogeneous single-phase state of the feedstock powder is not preserved due to the influence of the temperature.

The distribution of the phases is shown through EBSD studies as well. Phase maps of the different production routes are depicted on the left-hand side of Figure 4. In addition, grain size and orientation are demonstrated in the inverse pole figure (IPF) maps (right-hand side of Figure 4). Grain boundaries (black) and twin boundaries (white) are shown in both illustrations.

The coating in the as-sprayed state (Figure 4a) is composed of a fine-grained lamellar structure, showing that the particles mostly have been melted and quenched in the thermal spray process. Slightly deformed initial feedstock particles can be observed in the maps incidentally as well. Due to the presence of pores, cracks, and oxides, not all areas could be indexed which is shown in black (CI < 0.1). A single bcc phase is predominantly formed in accordance with the phase analyses by XRD. A minor fraction of an fcc phase seems to be present, which was not detected by XRD. No distinct twin boundaries are formed in the as-sprayed coating.

Solution annealing caused a more homogeneous grain size distribution (Figure 4b). The fine-grained lamellar structure existing in the as-sprayed coating vanished after annealing. Mainly equiaxed grains were formed. This indicates that recrystallization and grain size leveling took place. Apart from the primary bcc phase, an fcc phase in a large fraction of ≈51% was newly formed. The fcc phase exhibits distinct twin boundaries which are preferentially formed in phases with a low stacking-fault energy.[21] The formation of twin boundaries could also be observed for conventional alloys with an fcc structure, e.g., copper or austenitic steel as well as HEAs with fcc structure.[22,23]

The powder metallurgically produced bulk alloy exhibits a different microstructure (Figure 4c). Compared with the coatings in as-sprayed and annealed state, a coarser microstructure is formed. The shape of former feedstock particles can be noted, showing that no melting of the feedstock powder occurred. Despite the relatively fast powder metallurgical processing by SPS, an additional fcc phase is formed. The fcc phase content of ≈32% is reduced compared with the solution annealed coating. This can be explained by the lower input of thermal energy in comparison with the heat treatment of solution annealed coatings.

The chemical compositions of the bcc and fcc phases were determined by EDS point measurements for the multiphase HEA states. The results are shown in Table 4. Associated with the formation of the fcc phase, a strong deviation of the local chemical composition from the nominal equimolar composition of the powder takes place. Solution annealing of the coating increased the aluminum and nickel content in the bcc phase, whereas the chromium content is distinctly reduced in comparison with the equimolar composition. The effect of alloying elements on phase formation has been described in detail in the literature. Aluminum acts as a strong stabilizer of phases with bcc structure.[24,25] In phase areas with an fcc structure, a high content of chromium and iron was determined, whereas the aluminum and nickel content were reduced.

The analyses of the chemical composition of the powder metallurgically produced bulk alloy reveal a similar tendency. In phase areas with a bcc structure, a high content of aluminum and nickel was determined. The phase areas with an fcc structure exhibit a high content of chromium, iron, and cobalt. Although a similar tendency is observed, there is a distinct dependence of the phase composition on the production route, especially on the temperature time profile, resulting in changed diffusion behavior.

The effect of the microstructure and phase formation on the resulting mechanical properties was investigated by microhardness measurements in a first step. A summary of the measured values is shown in Figure 5. The highest microhardness of 600 ± 50 HV 0.1 was measured for the coating in as-sprayed condition. A single-phase bcc structure was proven for this state. Solution annealing causes a distinct reduction of microhardness to 390 ± 20 HV 0.1 due to the formation of a ductile fcc phase. Furthermore, solution annealing of thermal spray coatings causes the reduction of residual stress. For the bulk alloy produced by SPS, a slightly increased hardness of 430 ± 10 HV 0.1 was measured. Phase analyses revealed the formation of the additional fcc phase for this state, but due to the lower
heat input, a reduced fraction of this secondary phase is formed. In comparison with the coating in solution annealed state, no distinct structural defects such as pores and oxide lamellae occur, causing the reduced standard deviation of the hardness.

3.4. Wear Behavior

The influence of the production route and microstructure on the wear behavior was investigated in detail. Therefore, ball-on-disk, reciprocating wear, and scratch test have been conducted. In Figure 6, the measured wear depths are presented. The wear behavior under sliding wear conditions in ball-on-disk test shows a distinct dependence on the production route. The highest wear depth was measured for the coatings in as-sprayed condition. Therefore, this state exhibits the lowest wear resistance. Despite the reduction of microhardness, the wear resistance is increased for the solution annealed state. The formation of an additional ductile fcc phase has a positive effect on the wear resistance. However, a high standard deviation occurs. This can be explained by the formation of a heterogeneous state. Typical structural defects, e.g., porosity and oxides of thermally sprayed coatings, occur. The bulk alloy produced by SPS exhibits a similar wear resistance. Also for this state, the formation of an additional ductile fcc phase with a reduced phase content was observed. In comparison with the solution annealed coating, a decrease in the standard deviation occurs due to the reduced content of structural defects.

The investigations under reciprocating wear conditions reveal a similar tendency. The lowest wear resistance was observed for the coatings in as-sprayed state. Solution annealing and the consequently formed additional fcc phase result in an increase in wear resistance. However, the wear behavior under reciprocating conditions is distinctly influenced by the content of the secondary fcc phase and structural defects. For the bulk alloy produced by SPS, an additional increase in wear resistance occurs. A lower content of the ductile fcc phase was determined and structural defects were distinctly reduced.

Under abrasive conditions in scratch test, no distinct influence of the production route on the wear behavior was observed. Despite the formation of the additional fcc phase and a
consequently reduced hardness, the measured wear depth is not significantly changed. Due to the absence of structural defects, the wear depth of the powder metallurgically produced material is slightly reduced.

To determine the underlying wear mechanisms, the wear tracks were investigated by SEM. Representative images of the ball-on-disk and reciprocating wear test are shown in Figure 7. The scratch test induces abrasive wear without occurring spallation. No distinct differences were observed for the different states.

The wear tracks of the as-sprayed coating tested under sliding wear conditions in ball-on-disk test show a relatively smooth surface. Grooves in the wear direction appear, showing that predominantly abrasive wear occurred due to the application of a harder alumina counter body. Furthermore, exposed porosity of the coating can be observed. For the wear tracks of the samples tested under reciprocating wear conditions, a strong material contrast occurs. Due to the application of a BSD detector, the dark areas

![Figure 4. Phase map with fcc phase in green and bcc phase in red (left) and orientation distribution with coloring according to the standard triangle (right) of the equimolar alloy AlCrFeCoNi: a) as-sprayed coating; b) solution annealed coating, and c) bulk alloy produced by SPS.](image-url)
indicate a low atomic number and thus the formation of oxides by tribooxidation. However, a rough surface which is not fully covered by oxides is formed. Therefore, the underlying material is not protected by the formed oxides. In addition, during the test under reciprocating wear conditions abrasive wear predominantly occurs.

The wear tracks of the annealed coating exhibit a slightly changed appearance in comparison with the as-sprayed state. Material contrast indicates the local formation of oxides under sliding wear conditions. Less exposed porosity of the coating can be observed. The wear tracks of the reciprocating wear test show a similar appearance. A rough surface partially covered with oxides is formed.

For the bulk alloys produced by SPS, minor oxide formation occurs under sliding wear conditions. Distinct tribooxidation is observed under reciprocating wear conditions, which results in a rough surface partially covered with oxides.

4. Conclusions

Coatings of the equimolar alloy AlCrFeCoNi were produced by HVOF thermal spraying. For this purpose, an inert gas-atomized feedstock was applied. Due to the high cooling speed during the powder production process, a single-phase bcc structure is formed. The relatively low thermal input of the coating process and the fast cooling conditions cause no distinct change in the phase composition. A lamellar coating structure with minor oxides and porosity is formed. With the objective being to form a duplex microstructure comprising a ductile fcc phase, solution annealing of the coatings was conducted. No additional brittle phases were formed. Recrystallization causes a more homogeneous grain size distribution. The investigation of the
resulting mechanical properties revealed a reduction of the hardness in comparison with the as-sprayed state. However, the wear resistance under sliding and reciprocating conditions was distinctly improved. No significant change in the wear behavior under abrasive conditions occurred.

Furthermore, the feedstock powder was processed by SPS. Dense bulk alloys with a duplex structure comprising a bcc and fcc phase were formed. By detailed microstructural investigations, melting of the feedstock particles and recrystallization could be excluded. The highest wear resistance was found for the bulk alloy produced by SPS due to the formed duplex structure and the absence of structural defects, e.g., porosity and oxides. Detailed investigations on the wear behavior revealed mainly abrasive wear, whereas distinct tribooxidation occurred under reciprocating wear conditions. However, no closed protective oxide layer was formed.

The investigations show that the wear resistance of HEAs can be improved by the formation of a duplex structure comprising ductile phases. Further studies on the effect of the heat treatment parameters on the content of the fcc phase and the resulting properties have to be conducted.

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Conflict of Interest
The authors declare no conflict of interest.

Author Contributions
M.L. and T.L. conceived and designed the experiments. M.L., T.L., S.C., R.P., and D.D. performed the experiments, analyzed the data, and wrote...
the article. T.L. directed the research and contributed to the discussions and interpretations of the results.

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