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Influence of the composition of thermally sprayed (Al)CoCrFeNi(Mo) high-entropy alloy coatings in face turning and diamond smoothing

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Abstract
High-entropy alloys (HEAs) represent a relatively new material group with advantages like a high resistance to corrosion and wear. Concerning these properties and the elevated material costs their use as protection layers is aspired. In this regard, thermal spraying enables a wide range of different geometries. Due to the high initial roughness of the coatings, finishing is required.
For the investigations, layers of the HEAs CoCrFeNi, $Al_{0.3}$CoCrFeNi, and $Al_{0.3}$CoCrFeNi$Mo_{0.2}$ are generated by high velocity oxygen fuel (HVOF) spraying. Finishing of the coatings is realised by face turning with different cutting speeds in the range of 100 m/min to 400 m/min. Furthermore, experimental investigations regarding the surface modification by diamond smoothing with constant force (100 N) are carried out. After machining the cutting tool wear is analysed microscopically. The geometrical surface properties are determined using 3D laser scanning microscopy and tactile measurement. Additionally, the surface hardness is analysed.
Depending on the material composition a slight decrease of the surface roughness depth with increasing cutting speed can be detected. Simultaneously, the tool wear rises. Additionally, for the alloy compositions considered a strong decrease of the roughness values *Ra* and *Rz* as well as an increase of the surface hardness can be realised by diamond smoothing. The research expands the field of machining thermally sprayed HEAs. This promotes the use of these layers for wear or corrosion protection.

Diamond smoothing; High-entropy alloy; Thermal spraying; Turning

1. Introduction

HEAs are characterised by four or more elements with nearly equimolar proportions. Due to their high resistance to wear and corrosion and the comparatively high material costs wear protection layers are a possible field of application. Regarding the initial roughness after the coating process finish machining of the coatings is necessary to reach adequate tribological properties. Cutting processes with geometrically defined cutting edges enable a fast machining and a high geometrical flexibility. Besides the adaptions of the geometrical properties, an adjustment of the physical surface properties (e. g. hardness, residual stresses) is also possible. Subsequent smoothing allows for a further decrease of the surface roughness values, strong compressive residual stresses, and a grain refinement of the near-surface layer.

Machining of HEAs with geometrically defined cutting edges is rarely regarded until now. Guo et al. [1] analysed the machinability of selectively laser melted CoCrFeMnNi HEAs by different finishing processes. The initial roughness *Ra* of 30 µm could be reduced by grinding (to 4 µm), by EDM (to 3 µm), and also by milling (to 1 µm). The finishing processes also influenced the surface layer properties. Grinding resulted in a surface hardness of 350 HV, EDM led to about 400 HV, and milling entailed a hardness of about 450 HV. Due to the forces and the deformation while machining after milling (-700 MPa) and grinding (-400 MPa) respectively compressive residual stresses were detected. Clauß et al. [2] analysed the influence of the cutting speed and the feed in turning of an AlCoCrFeNiTi thermally sprayed HEA. Increasing the cutting speed in the range of 100 m/min to 400 m/min resulted in a decrease of the surface roughness values due to the reduced proportion of pulled-out coating material. However, for the range of 200 m/min to 400 m/min similar roughness values were measured. Additionally, the absolute values of the compressive residual stresses in the direction of feed motion rose with increasing cutting speed.

Burnishing or machine hammer peening of thermally sprayed coatings are rarely regarded until now. Rausch et al. [3] realised a surface modification of arc sprayed coatings (FeCrCMnSi with included WC particles) by machine hammer peening. As a result, the surface roughness values and the near-surface porosity were reduced. Additionally, the wear resistance of the coatings could be increased. Among others this was an effect of the strong compressive residual stresses introduced by the machine hammer peening process. Other researchers [4] machined atmospheric plasma sprayed iron-based coatings by turning and subsequent diamond smoothing. For the range of the smoothing force between 50 N and 150 N similar surface roughness values could be achieved for all feeds applied in pre-machining (0.05 mm – 0.15 mm). A further increase of the force resulted in higher values for *Ra* and *Rz* due to surface microcracks. Additionally, the absolute values of the compressive residual stresses in the surface layer could be increasingly enhanced with raising smoothing forces.

Currently, only a few studies regarding machining of HEAs are available. Heretofore, the influence of the specimens' chemical composition on the cutting process is not analysed. Smoothing processes enable a predefined modification of the surface properties. Until now there is almost no focus on thermally sprayed coatings in regard to HEAs. In addition to the influence of the cutting speed in turning the paper addresses the capability of diamond smoothing for machining of thermally sprayed HEAs.

2. Experiments

2.1. Specimens

In the experimental investigations circular disc substrates of the steel EN 1.4404 (AISI 316L) characterised by a diameter of 40 mm and a thickness of 10 mm were used. To generate an adequate adhesive tensile strength between the substrate and the coating the discs were roughened by grit blasting at the plane surfaces. Afterwards, the coatings were applied by high velocity oxygen fuel (HVOF) thermal spraying according to Table 1. Inert gas atomised feedstock powder with a grain size of the fraction -50 + 20 µm of three different compositions (CoCrFeNi, $Al_{0.3}CoCrFeNi$ (referred to as AlCoCrFeNi in the following text), $Al_{0.3}CoCrFeNiMo_{0.2}$ (referred to as AlCoCrFeNiMo in the subsequent text)) was processed. A final layer thickness of approximately 500 µm was adjusted for all coatings. The coatings were characterised by a porosity of less than 1 %. The hardness of the coatings increased with higher number of elements (see chapter 3.3.) as a result of solid solution hardening.

Table 1 HVOF thermal spray coating parameters

Parameter	Value
O_2	850 l/min
Kerosene	22.5 l/h
Ar	2 x 11 l/min
Powder feed rate	2 x 35 g/min
Spraying distance	360 mm
Relative traverse speed	1.0 m/s
Spray path offset	5 mm

2.2. Tools

For face turning (pre- and finish machining) CBN-tipped (cubic boron nitride) indexable inserts were used. The cutting material consisted of 90 % - 95 % boron nitride particles with a grain size of 1 µm and a cobalt binder. The tools of the type CCGW 09T304 were characterised by a nominal rake angle of 0° and a sharp cutting edge (rounding < 5 µm). In connection with the tool holder used the cutting edge angle of the minor cutting edge was 5°. For diamond smoothing a tool with a MCD (monocrystalline diamond) spherical body exhibiting a radius of 2 mm was used.

2.3. Experimental Investigations

The finish machining experiments were carried out by face turning on a precision lathe SPINNER PD 32. Some of the specimens were diamond smoothed (DS) subsequently using the same machine tool. Regarding the initial roughness of the coatings pre-machining was also done by face turning. To provide a constant cutting speed during machining the specimens were prepared by drilling and internal turning between the axis of rotation and a diameter of 25 mm. Hence, in the experiments an annular area with a width of 7.5 mm (between the outer (40 mm) and the inner (25 mm) diameter) was machined. Additionally, the rotational speed was adapted to the actual diameter to keep the cutting speed unchanged while machining. For finish turning and turning prior to diamond smoothing the feed (0.05 mm) and the depth of cut (0.1 mm) were kept constant. Cutting was realised with different cutting speeds (v_c) in the range between 100 m/min and 400 m/min according to Table 2. For turning of the specimens used in diamond smoothing a cutting speed of 200 m/min was applied. Face turning was performed without cooling lubricant. For every experimental combination of material composition and cutting speed an unworn tool was used. The respective two specimens were machined without changing the tool.

Diamond smoothing was performed with the same feed like in turning experiments (0.05 mm). The smoothing force of about 100 N, which acts perpendicular to the machined surface, and the smoothing speed of 70 m/min were kept unchanged. To improve the sliding behaviour between the MCD tool and the specimens emulsion flood cooling was used. During finish machining the components of the resultant force were recorded by a three-axis dynamometer Kistler type 9257A.

Table 2 Number of machined specimens for turning experiments with different cutting speeds v_c and diamond smoothing (DS)

	CoCrFeNi	AlCoCrFeNi	AlCoCrFeNiMo
100 m/min	2	2	2
200 m/min	2	2	2
300 m/min	2	2	2
400 m/min	2	2	2
DS	1	1	1

2.4. Analysing of the experiments

The geometrical surface properties of all specimens machined were detected using a stylus instrument Mahr type LD 120. The roughness was measured in the direction of feed motion at five different sectors on the machined surface. The stylus used was characterised by a radius of 2 µm. The measuring length was 4 mm and the filtering of the profile was done in accordance to ISO 11562. Additionally, each specimen machined was measured at two different areas using a 3D laser scanning microscope (3D LSM) Keyence type VK-9700. The size of the analysed areas was 1 mm × 1 mm. The tool wear was detected by a Nikon measuring microscope type MM-400.

The surface hardness before and after machining was measured using a micro-hardness tester type TUKON 1102 with a Vickers indenter. For each indent, a load of 4.905 N (0.5 kp) was applied for a time of 10 s. The resulting Vickers hardness values were calculated using the diagonal lengths of the indents. Each hardness value presented corresponds to the mean value of 10 indents, uniformly arranged on a defined radius of the machined surface. Regarding the coating thickness after machining of about 200 µm and the indentation depth of about 12 µm no significant influences of the substrate material and the surface roughness on the hardness measurements were expected. The hardness of the unmachined specimens was measured at ground surfaces.

3. Results and discussion

3.1. Tool wear and components of the resultant force

The tool wear after machining two specimens by face turning is summarised in Table 3. For the specimens from all alloys machined, an increase of the cutting speed results in a larger flank wear land width. This occurs due to two dominant effects. Firstly, increasing the cutting speed entails a rise of the shear zone temperature as a consequence of the higher cutting power. Hence, the strength of cutting material, especially the binder, is reduced. These effects were also shown in [5, 6], where a constant decrease of the cutting material hardness and strength with increasing temperature was detected. Furthermore, this decrease is stronger in the case of using CBN with a metallic compared to a ceramic binder. Secondly, the kinetic energy of the coating particles including hard phases (e. g. oxides) raises. Both simultaneously acting mechanisms generally result in a growth of the tool wear with increasing cutting speed.

Table 3 Influence of the cutting material and the cutting speed on the flank wear land width (in µm), occurance of built-up edges (BE) and cutting edge chipping (C) after machining two specimens

v_c (m/min)	CoCrFeNi	AlCoCrFeNi	AlCoCrFeNiMo
100	25 (BE + C)	10	5
200	35 (BE + C)	25	20 (BE)
300	30 (BE)	65 (BE)	20 (BE + C)
400	55 (BE + C)	100 (BE + C)	75 (BE + C)

For all cutting speeds, after machining CoCrFeNi specimens built-up edges occur due to the least hardness of the alloys regarded. For low cutting speeds the highest tool wear was detected after machining this thermally sprayed coating. However, the increase of the flank wear land width with rising cutting speed is smaller compared to machining of AlCoCrFeNi and AlCoCrFeNiMo. This slighter rise of the tool wear is a result of the lower hardness of the coating consisting of four alloy elements. Comparing the two harder coating layers, in the analysed range of the cutting speed a higher flank wear land width occurs after machining of two specimens of the alloy AlCoCrFeNi. Besides the abrasive wear diffusion effects between the cobalt binder and the iron of the alloy depending on the iron content have also to be considered, but were not detected. Furthermore, the highest flank wear land width is measured after machining the AlCoCrFeNi alloy with a cutting speed of 400 m/min. In this case, besides the already mentioned effects the strongest increase in hardness after machining is detected (see chapter 3.3.), which results in a higher tool wear. Generally, regarding the total cutting length of about 30.8 m the wear is comparatively high. Concerning the similar surface properties after facing (see chapter 3.2 and 3.3) if possible a lower cutting speed should be chosen.

While machining the components of the resultant force were detected. The three single values are calculated in the range of theoretically constant cross-section of undeformed chip. The cutting force represents the highest component of the resultant force with mean values between 14.2 N to 18.5 N, followed by the passive force (12.3 N – 15.1 N), and the feed force (4 N – 5.4 N). For all three components no clear influence of the material composition and the cutting speed is visible. Generally, a slight decrease of the components of the resultant force with increasing cutting speed as a result of the raising shear zone temperature and concomitant softening of the material is expected. Additionally, growing tool wear with increasing cutting speed involves an increase of the respective components. These simultaneously acting mechanisms result in the nearly constant values for the different cutting speeds and materials machined.

3.2. Geometrical surface properties

Figure 1 represents the influence of the cutting speed on the arithmetic mean surface roughness *Ra* and the surface roughness depth *Rz* depending on the alloy composition. The mean values are calculated from all tactile measurements of the specimens machined with the same conditions. The standard deviation is represented by the error bars. Generally the surface roughness after turning is higher than the calculated kinematic roughness (0.78 µm). This is a result of the cutting edge chipping in consequence to the tool wear and the specific morphology of thermally sprayed coatings. The particle structure of the coating layers and the microstructure inherent pores lead to effects like opening of pores, pull-out of coating material while machining (see Figure 2), and the occurrence of microcracks. However, due to the low porosity, the effect of the pore-opening on the surface roughness should be minor.

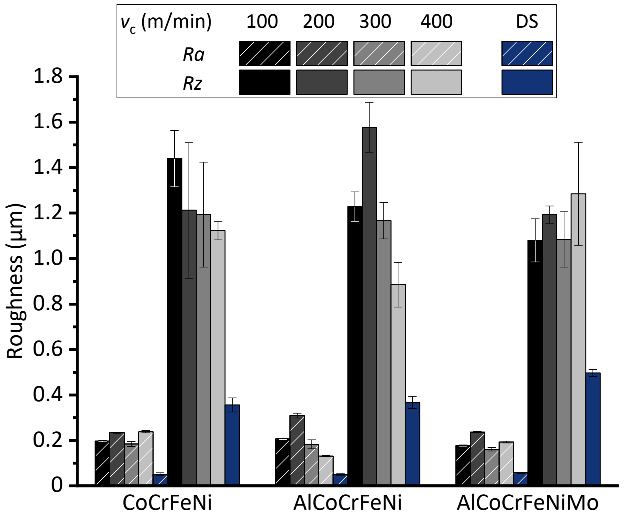

Figure 1. Influence of the material and the cutting speed in face turning as well as the effect of diamond smoothing on *Ra* and *Rz*

Regarding the mean values of *Rz* a slight tendency of decreasing with increasing cutting speed is visible for machining CoCrFeNi. This could be the result of the lower proportion of pulled-out coating particles, which is also visible in Figure 2, due to an increased shear zone temperature and shear angle. Excepting the values after machining with a cutting speed of 200 m/min, the slight trend is also recognisable for AlCoCrFeNi. Regarding the alloy with additional molybdenum this relationship is not noticeable, maybe due to the increased hardness. Summarising and especially taking the deviations into account similar surface roughness values are measured in the analysed range of cutting speed for all coating materials. Additionally, the weak tendencies mentioned are only partly visible regarding the values for *Ra*.

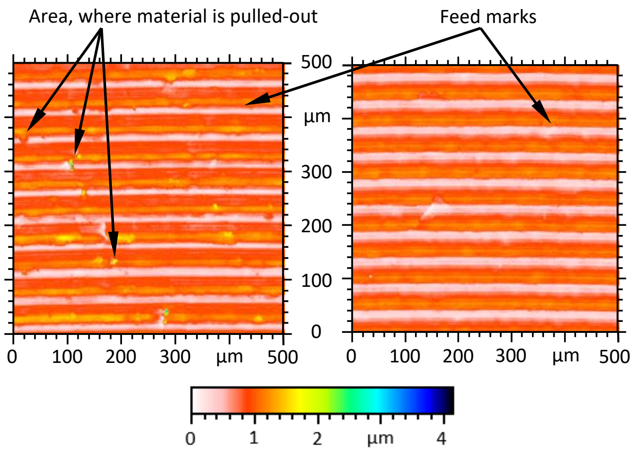

Figure 2. Surfaces detected by 3D LSM after machining CoCrFeNi with a cutting speed of 100 m/min (left) and 400 m/min (right)

For all alloys regarded, a significant decrease of the *Ra* and *Rz* values is reached by diamond smoothing. While machining the peaks of the roughness profile are lowered by plastic deformation and the valleys of this profile are lifted. Additionally, the specific microstructure of thermally sprayed coatings also enables a compaction of the material by reducing the near-surface porosity. The surfaces after smoothing are shown in Figure 3. Generally, the waviness is similar to turning. Only for diamond smoothing of CoCrFeNi the waviness is slightly higher compared to facing the softest alloy specimens. However, a small adjustment in smoothing force should lead to comparable results. The *Rz* values after diamond smoothing of AlCoCrFeNiMo coatings are considerably higher (0.497 µm) than after smoothing of the two other alloys regarded (0.356 µm and 0.367 µm). This is a consequence of the higher hardness and the expected higher strength of the alloy consisting of six alloy elements. Thus, smoothing results in a lower deformation. The surface profile of the smoothed CoCrFeNi specimen (Figure 3a) is characterised by valleys corresponding to the radius of the tool used (2 mm) and a distance corresponding to the feed. Additionally, smeared coating material is also visible which results in an increase of the surface roughness values. By diamond smoothing of AlCoCrFeNi the kinematic roughness profile after turning is hardly visible. The peaks and valleys of the turning profile are nearly completely deformed. However, the mentioned smoothing profile, characterised by the tool diameter and the feed is not completely visible. After diamond smoothing of the hardest coating the largest heights of the profile are measured. With the smoothing force chosen the peaks and valleys of the kinematic roughness profile are not completely lifted or lowered. To reach comparable results to CoCrFeNi and the expected smoothing profile an increased smoothing force is required. Furthermore, the results of the smoothing process depend on the surface properties after turning (shown in [4]). Surface imperfections, like pulled-out particles after turning cannot or only partly be compensated or closed by smoothing.

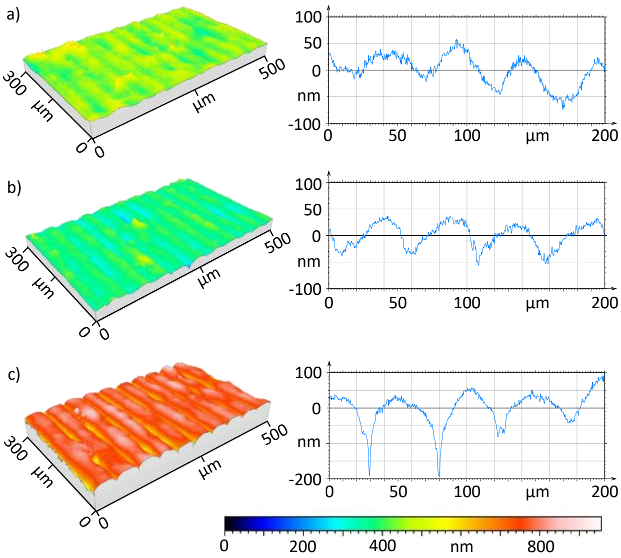

Figure 3. Surfaces detected by 3D LSM after diamond smoothing of CoCrFeNi (a), AlCoCrFeNi (b), and AlCoCrFeNiMo (c) (2D profiles are detected in the direction of feed motion)

3.3. Hardness

The results of the hardness measurements at the surfaces are summarised in Table 4. For the analysed coatings an increase of the number of alloy elements results in a higher hardness. Generally, there is a higher variation of the values in the initial state and after turning compared to smoothing. Regarding the alloy CoCrFeNi a raise of the surface hardness after turning is visible. In the analysed range of cutting speed similar hardness values are reached. The hardness increase during machining of sprayed coatings is based on simultaneously acting mechanisms. The components of the resultant force, especially the passive force lead to a deformation of the near-surface particles and a material compaction. Additionally, the temperatures in the shear zone while machining and the subsequent cooling could result in a raising hardness, e. g. due to strain hardening. For the coatings consisting of five or six alloy elements no hardening effect by turning (except AlCoCrFeNi with v_c = 400 m/min) is detected. This could be a consequence of the higher initial hardness and the lower deformation while facing in connection with the highest cutting speed and shear zone temperature. For all alloys smoothing resulted in a hardness increase. The force acting perpendicular to the surface is significantly higher than during turning. While sliding only a minor heating is expected. Due to the stronger deformation and compaction while smoothing the hardness increase shown can be explained.

Table 4 Influence of the material, the cutting speed in turning, and the effect of diamond smoothing (DS) on the surface hardness (mean value and standard deviation) compared to the initial state (IS) (in HV 0.5)

	CoCrFeNi	**AlCoCrFeNi**	**AlCoCrFeNiMo**
IS	349 ± 17	373 ± 13	402 ± 25
100 m/min	378 ± 9	385 ± 17	414 ± 7
200 m/min	375 ± 12	370 ± 29	396 ± 20
300 m/min	368 ± 5	368 ± 23	393 ± 16
400 m/min	370 ± 13	406 ± 20	392 ± 27
DS	427 ± 5	447 ± 8	472 ± 4

4. Summary and outlook

The experimental investigations shown represent the first results published analysing the influence of the composition of thermally sprayed HEAs in turning and diamond smoothing. Considering the tool wear a general increase of the flank wear land width with raising cutting speed is detected. In the cutting speed range regarded similar mean values of *Ra* and *Rz* are reached after machining. However, a small decrease of the mean values of *Rz* with increasing cutting speed could be detected for the alloys CoCrFeNi and AlCoCrFeNi. For all HEAs a significant reduction of the surface roughness values by diamond smoothing is possible. However, differences in the resulting surface profiles and roughness values due to the coating hardness are visible. Additionally, an increase of the surface hardness after diamond smoothing is determined.

Further analyses include residual stress measurements and an analysis of the microstructure at cross-sections for an enhanced understanding of the mechanisms in turning and diamond smoothing of HEA coatings. Additionally, enlarged experimental investigations regarding the process parameters in diamond smoothing should indicate the potentials and achievable properties. The results shown represent a next step for the industrial application of HEAs as protection layers.

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