FRACTURE MECHANICAL BEHAVIOUR OF PLASMA-SPRAYED COATINGS

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ABSTRACT

Four different types of plasma sprayed coatings which differ significantly in chemical composition, microstructure and mechanical properties have been investigated. The coating materials are molybdenum, aluminium-silica and aluminium-silica with two different molybdenum percentages. In all cases the substrate material is a casehardened steel.
The microstructures of the coatings were characterized in the scanning electron microscope (SEM). Additionally nanoindentations were used to quantify local mechanical properties. The fracture toughness of the coatings was measured in special tests. Furthermore in-situ bend tests were carried out in the SEM to study the local failure behaviour and the crack path. The effect of the microstructure of the coating and the crack interaction with the substrate material on the fracture behaviour are discussed.

INTRODUCTION

In recent years coated materials have attracted increasing interest. On the one hand coatings can improve mechanical properties like wear resistance of the substrate material, on the other hand they are used to create special new properties for thermal or other applications. Especially wear resistance is of decisive importance in applications in the automobile industry. In this work the impact of different coatings on the in-service behaviour of coated materials designed for wear resistant applications has been studied.
Especially the behaviour of cracks perpendicular and parallel to the coating-substrate interface and the fracture processes in different coating systems have been investigated and shall be discussed.

MATERIALS

The four types of coatings used for the examinations are molybdenum, aluminium-silica and aluminium-silica with two different molybdenum percentages. In the present work they are denoted as Mo, AlSi, $AlSi/Mo_{low}$, $AlSi/Mo_{high}$. The parameters of the coating process and also the exact compositions of the coatings are literary property of the coating company Flametal S.p.A.. We used the casehardened steel 16MnCr5 as substrate material. The surface hardness after the casehardening process was more than 55HRC and the effective depth of hardening was 200-300µm.

Figure 1a. shows the SEM image of the rough surface which is typical for the investigated plasma sprayed coatings [1,2].

a. b.

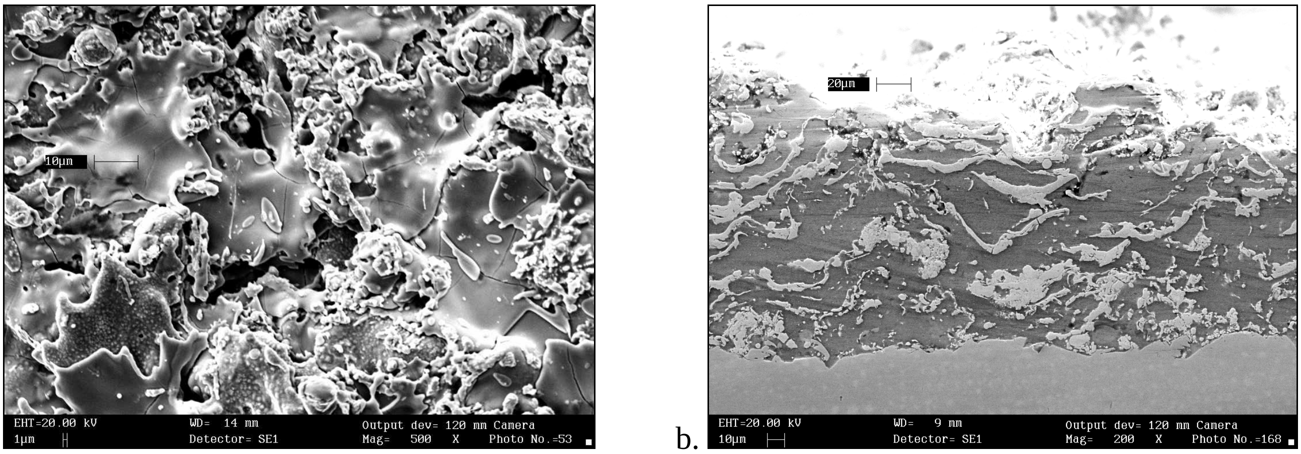


Figure 1: a. Surface of the pure Mo coating, **b.** Cross sectional view of polished AlSi/Mo_{high} coating

On cross sectional polished samples we studied the microstructure of the coatings in the SEM. Figure 1b. shows a typical SEM-micrograph of a plasma sprayed AlSi/Mo_{high} coating. The darker and the lighter phase correspond to the AlSi and the Mo splats respectively. On the bottom of the picture we can see the substrate material. On these cross sectional samples we measured local mechanical properties using a nanoindenter add-on device for our atomic force microscope (AFM). The results of these additional tests can be found in Table 3.

EXPERIMENTAL METHODS

To measure the fracture toughness of the coating we produced special samples with a geometry as shown in Figure 2. The dimensions of the samples are: width W = 22.5 mm, thickness B = 9 mm, height 2h = 15 mm, notch depth a = 7 mm.

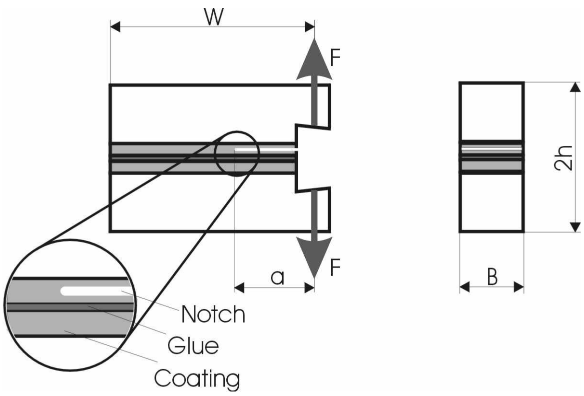


Figure 2: Sample geometry for the special fracture toughness tests

Two coated metal beams were glued together. For the gluing we used the CIBA-GEIGY multi-purpose adhesive ARALDITE® 2011. In one of the combined coatings we produced a notch by spark erosion over a length marked by the letter a. Using a wire of 50µm diameter we managed producing a notch which lies only within the coating. The coating thickness varied between 200 and 400µm. The load line is marked with two arrows and the letters F. The loading speed was 0.4 mm/min. Such tests were also performed in-situ in the SEM.

In order to study the crack propagation perpendicular to the coating-substrate interface, in-situ 3 point bend tests were carried out in the SEM. Because of the banded structure of the coatings different fracture behaviours in different crack propagation directions are to be expected. The sample geometry for these tests can be seen in Figure 3 (height H = 5 mm, width B = 3 mm, span width L = 23 mm). In these samples we prepared a "precrack" into the coating using a special racer-blade polishing method. The crack propagation under load was examined in-situ in the SEM.

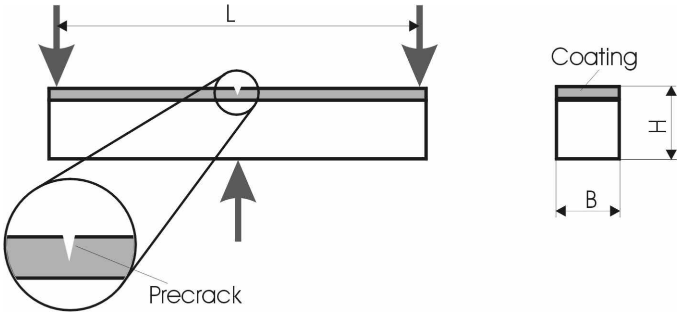


Figure 3: Geometry for bend test samples with the "precrack"

RESULTS AND DISCUSSION

Fracture toughness tests

Table 1 shows the results of the special fracture toughness tests (mode I loading of cracks parallel to the interface).

TABLE 1

RESULTS OF THE FRACTURE TOUGHNESS TESTS

Spec. Nb.:	K_Q [MPa√m]	Crack Path	Spec. Nb.:	K_Q [MPa√m]	Crack Path
Mo					
K_11a	1.4493	iC	K_11c	2.0197	iC
K_11b	2.0451	iC	K_12b	2.0071	iC
K_12	0.4004	iG	K_12c	1.9527	iC
K_13	0.6753	iG	K_13b	0.2362	iG
K_14	0.6533	iG	K_13c	2.2278	iC
K_15a	1.7032	iC			
K_15b	1.7108	iC			
K_15c	1.6869	iC			
K_15d	1.7484	iC			
K_15e	1.7229	iC			
AlSi Coating					
K_21a	1.7263	iI	K_21c	1.8829	iI
K_21b	2.1835	iI	K_21d	2.1956	iI
K_22	1.8305	iI	K_22b	2.2085	iI
K_23	0.2331	iG	K_22c	1.9663	iI
K_24	1.8226	iI	K_22d	1.9410	iI
AlSi/Mo_{low} Coating					
K_31a	2.2876	iI	K_31c	2.4917	iI
K_31b	2.2484	iI	K_31d	2.1804	iI
K_32	2.3098	iG-iI	K_32b	2.5052	iG-iI
K_33	0.1307	iG	K_32c	1.7740	iI
K_34	0.1405	iG	K_32d	1.7949	iI

AlSi/Mo_{high} Coating

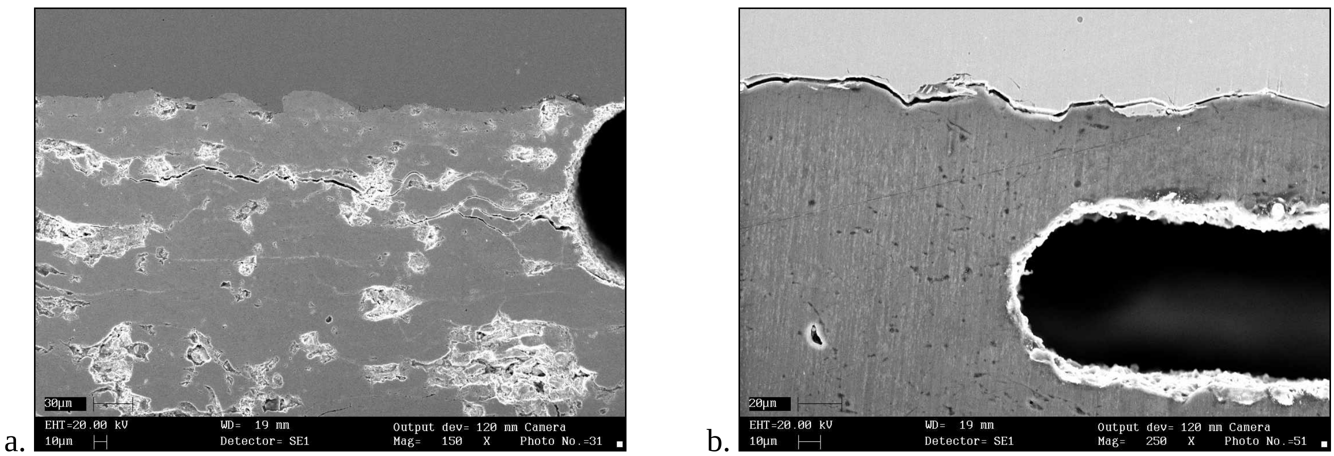
K_41a	2.2798	iG	K_41c	2.3651	iG-iI
K_41b	2.1337	iG	K_41d	2.5139	iG-iI
K_42	2.1627	iG	K_42b	2.0473	iG
K_43	0.4208	iG	K_42c	0.5038	iG
K_44	1.2557	iG	K_42d	2.6070	iG

The crack path propagation type was characterised in all samples and is listed in Table 1.
Four different types could be distinguished:

- the crack propagates in the coating (iC)
- the crack propagates in the glue (iG)
- the crack propagates in the coating-substrate interface (iI)
- the crack propagates in the glue and also in the interface (iG-iI)

For the calculation of fracture toughness values simple equations for homogeneous materials were used. For the calculation of the real local stress intensity factor additional information like the thermal, elastic and the plastic mismatch would have to be taken into account [3,4]. For example the residual stresses which are caused by the thermal mismatch between the coating and the substrate. We used the maximum force during the test to calculate a K_Q value. For this calculation we used standard equations for K_{IC} tests. [5]

In the molybdenum coating in most cases the crack propagates directly from the root of the notch throughout the coating. When we assume that we always have a few cracks like defects in the ground of the notch then we can treat the calculated K_Q for this coating as a nominal valid K_{IC} value.
For the pure AlSi coating we observed in most cases a failure of the coating-substrate interface. Here the crack started somewhere in the vicinity of the root of the notch caused by the higher stress intensities in this region. For these coatings the calculated K_Q can't be treated as a valid K_{IC} value but only gives an estimate of the stress intensity at which debonding of the AlSi coating from the substrate material takes place.
In the AlSi/Mo_{high} coating the crack propagates nearly without exception within the glue starting in the region of the ground of the notch. Here the calculated K_Q can be used to estimate the bonding strength of the glue to the coating. For a few AlSi/Mo_{high} coated samples we also found failure of the coating-substrate interface which means that for this coating the bonding strength between the coating and substrate is nearly the same as that between the glue and the coating. For the AlSi/Mo_{low} we found almost the same results as for the AlSi/Mo_{high} coated samples but with a higher amount of samples breaking by coating-substrate interface failure. For all the AlSi coatings we can deduce from the tests that their K_{IC} value have to be higher than the calculated K_Q but can not be examined with this kind of test.
For all coating materials we observed few tests which break at a very low load level due to a weak bonding of the glue to one of the two sample halves. This can be explained by a badly cleaned coating surface before the gluing process. Figures 4a., 4b., 5 show some examples of observed crack paths during the loading in the SEM .



a. b.

Figure 4: a. Crack path in pure molybdenum coating, **b.** Debonding of AlSi coating

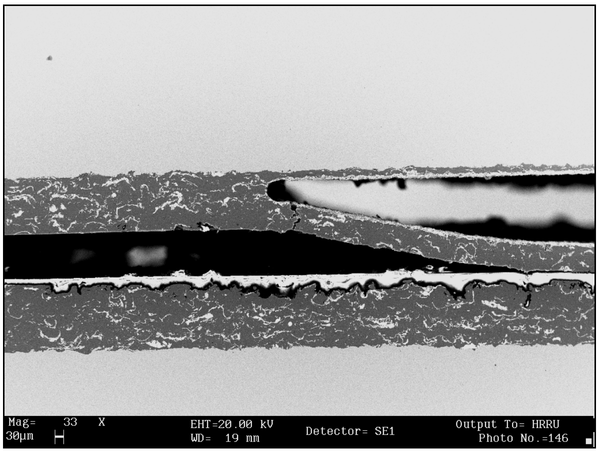


Figure 5: Glue failure in AlSi/Mo$_{low}$ coating system

Bend tests

In the bend tests we also found different behaviours of failure in the coating systems. For the AlSi coatings we observed in all three cases a debonding of the coating from the substrate material. For the molybdenum coating we could not find any debonding of the coating from the substrate. The forces for the debonding or the failure of the coating in case of the molybdenum coating can be seen in Table 2.

We measured a failure force where we found debonding of the coating in case of the AlSi coatings and where the cracks propagated through the coating into the substrate material in case of the molybdenum coating.

TABLE 2

RESULTS OF THE BEND TESTS

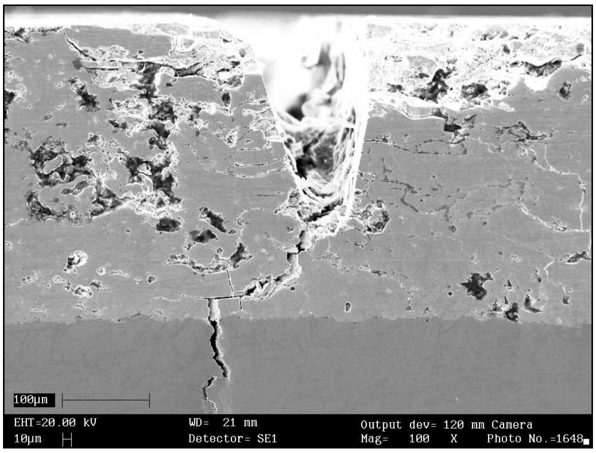
Spec. Nb.:	a [mm]	Coating Thickness [mm]	P [N]	Coating
t1	0.22	0.34	~3000	Mo
t2	0.23	0.35	~3000	Mo
t6	0.16	0.33	~2000	AlSi
t7	0.13	0.32	~2000	AlSi
t5	0.09	0.28	~1500	AlSi/Mo$_{low}$
t3	0.16	0.31	~1000	AlSi/Mo$_{high}$
t4	0.17	0.32	~1000	AlSi/Mo$_{high}$

a...................depth of the precrack (notch)

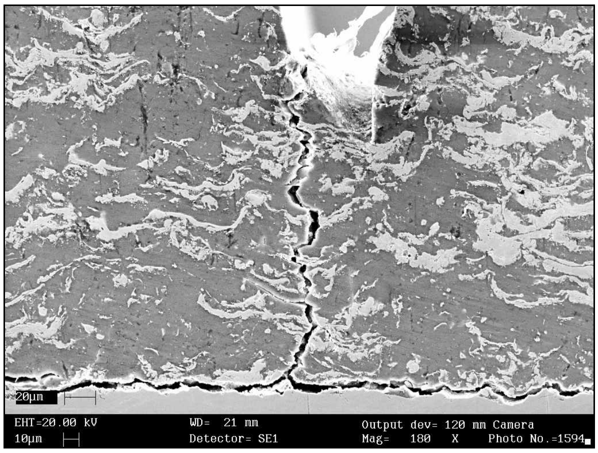
P ....failure force

In these tests the crack started in all cases from in the immediate vicinity of the root of the “precrack” and propagated in direction to the coating-substrate interface. In the Mo coating we observed a lot of micro cracks in the coating between the molybdenum splats before we got a total failure of the coating. These micro cracks (seen in Figure 6c.) lead to a lateral distraction of the main crack. This can be seen in Figure 6a. In the AlSi coatings we observed in most cases a breaking of the AlSi splats perpendicular to the coating-substrate interface which leads to a relatively straight crack propagation (see Figure 6b.). Only the molybdenum splats in the AlSi/Mo$_{low}$ and the AlSi/Mo$_{high}$ coatings can sometimes lead to a bigger lateral distraction of the main crack (see Figure 6d.).

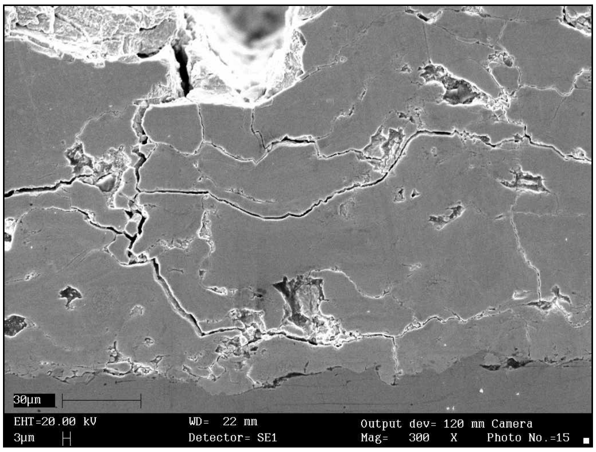
As we can see in Table 2 the forces which are necessary to get debonding of the pure AlSi coating are larger compared to that of the other AlSi coatings. This can be explained by a larger amount of micro cracks in this coating system which may lead to a stress relaxation. This effect is similar to microcrack crack-shielding in materials like ceramics.



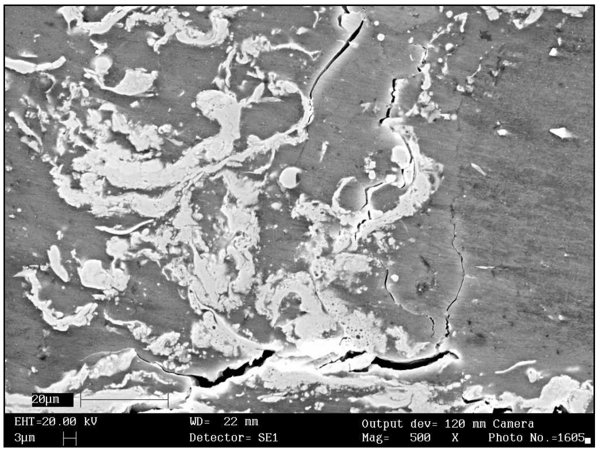
a.



b.



c.



d.

Figure 6: a. Breaking of molybdenum coating in a bend test sample, **b.** Failure during a bend test in AlSi/Mo_{high} coating sample, **c.** Micro cracks in Mo, **d.** Lateral crack distraction in AlSi/Mo_{high}

Nanoindentation tests

With a nanoindenter add-on device for the AFM we investigated the local mechanical properties of the different coatings. Such indentations allow measuring of reduced local Young's moduli [6,7] which are listed in Table 3.

TABLE 3

RESULTS OF NANOINDENTATION TESTS

Coating	E_{min} [GPa]	E_{max} [GPa]	E_{av} [GPa]	Number of measurements
Mo	231	337	289	9
AlSi	73	92	82	11
Substrate Steel	203	261	239	12

E_{min} .... lowest measured Young's modulus
E_{max}.................. highest measured Young's modulus
E_{av}.................... average value over all measurements

CONCLUSION

In the present experimental study we have investigated the critical stress intensity factors in plasma sprayed coatings in different crack propagation directions: in the plane parallel to the coating-substrate interface and perpendicular to the interface. The studied coating materials are molybdenum, aluminium-silica and aluminium-silica with two different molybdenum percentages. The observed failure mechanisms were breaking of the coating or debonding of the coating from the substrate material.

In the Mo coating:

- for the mode I loading of a crack parallel to the interface the crack propagates through the coating mainly between the molybdenum splats. The nominal fracture toughness for this propagation direction was ~ 1.84 MPa√m.
- for the mode I loading of a crack perpendicular to the interface the crack propagates through the coating directly into the substrate material. Before we got a total failure of the coating we found a lot of micro cracks between the splats which lead to a lateral distraction of the main crack.
- for the molybdenum coating we couldn't find any debonding of the coating.

In the AlSi coating:

- for the mode I loading of a crack parallel to the interface the crack propagates within the coating-substrate interface starting in the vicinity of the produced notch. For this coating we could not calculate a nominal fracture toughness. The interface failed at a "stress intensity" ~ 1.95 MPa√m.
- for the mode I loading of a crack perpendicular to the interface we observed debonding of the coating from the interface.

In the AlSi with Mo coatings:

- for the mode I loading of a crack parallel to the interface the crack propagates within the coating-substrate interface or within the glue. The calculation of a nominal fracture toughness was not possible. The samples failed at a "stress intensity" ~ 2.20 MPa√m.
- for the mode I loading of a crack perpendicular to the interface we observed debonding of the coating from the interface. The crack propagates mainly by breaking of the AlSi splats perpendicular to the coating-substrate interface which leads to a relatively straight crack propagation, only the molybdenum splats in these coatings can lead to a bigger lateral distraction of the main crack

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REFERENCES

1. Heimann, R.B. (1996). *Plasma-Spray Coaing*. Wiley/VCH, Weinheim.
2. Müller, K.-P. *Praktische Oberflächentechnik*. Vieweg, Göttingen.
3. Jiang, X., Matejicek, J., Sampath, S. (1999) *Materials Science and Engineering* **A272**, 189.
4. Suresh, S. and Mortensen A. (1998). *Fundamentals of Functionally Graded Materials*. IOM Communications Ltd, London
5. Schwalbe, K.-H. (1980). *Bruchmechanik metallischer Werkstoffe*. Hanser, München Wien
6. Menèik, J., Munz, D., Quandt, E., Ludwig, A. (1999) *Z. Metallkd.* **90 10**, 766.
7. Göken, M., Kempf, M. (1998). *Acta mater*. **Vol. 47 No. 3**, 1043