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Coating and prototyping of single-phase iron aluminide by laser cladding

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Laser cladding with powder blends of iron and aluminium was performed using a 500-W diode laser and a coaxial powder nozzle. Mild steel, pure nickel and aluminium alloy were used as substrate materials. The results were investigated by scanning electron microscopy, energy dispersive X-ray spectroscopy and X-ray diffraction. Completely crack-free, single-phase Fe_3Al coatings could be achieved on steel. The coating on nickel consisted of FeAl. Despite the high cooling rates, which normally occur during laser cladding, the grains within the coatings were rather coarse. Laser cladding on aluminium, on the other hand, led to cracking. Furthermore, it could be shown in this work that it is possible to prototype 3-D iron aluminide structures by *in situ* laser cladding.

1. Introduction

The intermetallic phases FeAl (B2) and Fe_3Al ($D0_3$) have been investigated for many decades and are known to possess good corrosion and oxidation resistance and low density (5.4-6.7 g/cm^3) at a low cost [1]. The mechanical properties of iron aluminides, especially the ductility at room temperature, strongly depend on many factors like Al-content, further alloying elements, testing environment and processing route [1,2].

Laser cladding is a process that can be used for protective coating, repair welding and rapid manufacturing [3]. In the last decades, many works on laser cladding with different substrate and powder material combinations have been published. It has been shown that some phases or alloys can be produced directly in the resulting coating or structure when using elemental powder blends instead of pre-alloyed powder. This process is called *in situ* laser cladding. The first experiments on laser cladding of single and stacked iron aluminide tracks on steel were performed by Abboud et al. [4].

It is the aim of this work to investigate if it is possible to produce single-phase iron aluminide coatings on different substrates by *in situ* laser cladding, using elemental powder blends. Crack-free laser cladding can render protective coatings possible for components under harsh environments and wear as well as 3-D object manufacture.

2. Materials and methods

2.1. Materials

Square mild steel (S235JR) samples 50 mm × 50 mm × 10 mm in size, aluminium wrought alloy (AW-2007) samples with the same dimensions and round technically pure nickel (LC-Ni 99) samples with a diameter of 50 mm and a height of 5 mm were used as substrate materials. Different substrates were used to show their influence on the resulting coatings. S235JR and AW-2007 are typical examples for common, low-alloy engineering materials. The motivation for the use of pure nickel in these first experiments is that industrially used nickel alloys have a high amount of further alloying elements which complicate the interpretation of the formation of the resulting phases. The sample surfaces were ground with 500# SiC abrasive paper and ultrasonically cleaned in isopropanol before the laser-cladding process.

Aluminium (99.5% purity) and iron (99% purity) powders with particle sizes between 20 μm and 50 μm were mixed in a custom-built stainless steel powder mixer at a speed of 60 rpm for 1 h to achieve a 45/55 at% ratio, respectively. This ratio was chosen since, according to the Fe-Al equilibrium phase diagram (cited in [5]), the temperature stability for FeAl is highest (up to 1310 °C). Furthermore, the composition in the coatings might still lie within the single-phase area of the phase diagram, should a shift in composition arise during laser cladding.

2.2. Laser cladding

For the laser-cladding experiments, the same facility as in Bax et al.[6], consisting of a 500-W diode laser (LDM 500-20, Laserline), a powder feeder (TWIN-10-C, Sulzer Metco), a coaxial powder nozzle (ILT Aachen), a three-axis CNC system (Bosch-Rexroth) and a heating block, was used.

The laser treatment parameters scanning speed, laser power, powder flow rate, shielding gas flow and heating temperature were optimized for the different substrates to improve the quality of single tracks, coatings and 3-D structures. The main goals in this context were the avoidance of cracks and a compromise between a small amount of dilution and a sound bonding to the substrate. Only the results for the most successful experiments are described and discussed in Section 3. The sample

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description and the varying parameters are listed in Table 1. Defocus distance (5 mm), carrier gas flow (5 Nl/min helium), shielding gas flow (5.8 Nl/min) and track overlap (50%) were identical for samples I to VI. When superimposing many layers to achieve cuboidal 3-D structures (samples V and VI), a scanning strategy described by Kelbassa [7] was used: every layer was commenced and finished with a frame which was filled by scanning the whole area. The main scanning direction was always perpendicular to the one of the previous layer. A *z*-offset of 0.3 mm was chosen since this was the average thickness of the single layers when using a nominal laser power of 500 W.

2.3. Sample characterisation

Metallographic cross sections of the coatings were prepared by cutting them perpendicular to the main laser scanning direction. The samples were embedded, ground and polished down to a 0.04-μm silica suspension. Optical microscopy was performed on an Axiovert 200 MAT (Zeiss). A focussed ion beam (FIB)/scanning electron microscope (SEM) dual beam workstation (Helios NanoLab 600, FEI), equipped with an energy dispersive X-ray (EDX) detector, was used for microstructural and chemical analysis. Microhardness tests were performed on an automatic hardness tester (Durascan, emcotest) using a load of 0.981 N and a loading time of 10 s.

As a preparation for X-ray diffraction (XRD) investigations, the coatings were ground down to be plane-parallel to the bottom side of the sample by a few tens of μm to circumvent the roughness and to remove oxide layers which both evolved during the laser-cladding process. The samples were polished down to a 1-μm diamond suspension. Phase analyses were performed on an X'Pert MPD Pro (PANalytical) using Cu-Kα radiation. Texture analysis was performed on an X'pert MRD (PANalytical) using Cr-Kα radiation.

3. Results and discussion

3.1. Coating on mild steel

The iron-aluminium powder blend can successfully be applied on the mild steel substrate over a relatively wide range of processing parameters. Sample I is an example of a sound and completely crack-free coating. The cross-sectional micrograph is presented in Fig. 1A. The height of the coating is approximately 215 μm, and the depth of the molten zone is approximately 135 μm, which corresponds to a dilution value of about 38.5%. Columnar grains can be found in the main part of the coating, and no secondary phase appears in the cross section. The absence of a second phase is confirmed by a uniform distribution of the elements iron and aluminium within the coating which was investigated by EDX mapping. The chemical composition of the coating is 75.8 at% iron and 24.2 at% aluminium. The shift in composition in comparison to the initial powder composition can be caused by two phenomena: the dilution of the substrate material which is mainly iron and/or a shift in powder composition during the mixing or laser-cladding process. The dilution itself whose value is given above would lead to a resulting aluminium content of approximately 28 at%. Therefore, it can be concluded that a shift in the chemical composition of the powder has taken place during the process. This can either result from uneven mixing or, more likely, from unequal deposition efficiencies of the two elements during the laser-cladding process. It shall be mentioned here that the overall deposition efficiency of single tracks with the given parameters, calculated by $P_e = A_c S \rho_p / F$, where A_c corresponds to the cross-sectional clad area, S to the scanning speed, ρ_p to the average powder density and F to the feed rate [8], was only 45%. A possible explanation for the difference in deposition rates among iron and aluminium is their differing physical properties (e.g., melting point, boiling point, absorbance at 940 nm) and their powder shapes.

At the given composition, the Fe-Al equilibrium phase diagram (cited in [5]) shows an α to B2 transition and a B2 to D0$_3$ transition. Since cooling rates are very high in laser cladding (up to 10^3 to 10^5 K/s [9]), the ordered structures do not necessarily have to be present at room temperature and knowledge of the chemical composition is not sufficient to determine the existing phase. Thus, XRD was performed and is presented in Fig. 1B. Peaks which can only correspond to the high order D0$_3$ and cannot be found in patterns of the less ordered B2 phase and α solid solution ({311}, {331}) are present in the pattern. Thus, the coating consists of Fe_3Al with a lattice parameter of 5.795 Å (calculated from the {440} peak position). The higher microhardness in the coating (see Fig. 1C) underlines the presence of an intermetallic phase. The ratio of peak intensities differs from the ones in the powder diffraction file (00-050-0955), which indicates that the coating is textured. This can be attributed to the columnar growth which can be seen in the cross sections. Since the sample was rather small and the grain size within the coating was rather high, we produced a larger sample with a base area 40 mm × 40 mm for XRD-texture analysis. The process parameters were the same as for sample I, preheating omitted. The results of the analysis can be seen in Fig. 2: the sample shows a strong cube texture with the {h00} planes being nearly parallel to the sample surface.

3.2. Coating on pure nickel

The process window for applying crack-free iron aluminide coatings on pure nickel is smaller than in the case of steel substrate. Nevertheless, it is possible to achieve crack-free coatings as illustrated in Fig. 3A. The SEM micrograph shows elongated single-phase grains. The average chemical composition was determined to be 54.1 at% iron, 24.4 at% aluminium and 21.5 at% nickel. A shift in the original iron to aluminium ratio can be observed with nickel substrates, excluding dilution as a cause. According to the ternary Fe-Ni-Al phase diagram [10], the resulting chemical composition corresponds to B2–(Fe,Ni)Al at temperatures above 950 °C. Below this temperature, a multiphase region, consisting of B2-(Fe,Ni)Al and α-Fe, has been found for the given composition. Since only one phase is present within the coating according to the backscatter micrograph and the XRD pattern (Fig. 3A and B), it can be assumed that a non-equilibrium phase composition exists in the coating which is probably caused by the fast cooling which generally occurs during the laser cladding process. The present phase was identified as B2-(Fe,Ni)Al with a lattice parameter of 2.876 Å (calculated from the {220} peak position). The presence of the {100} peak in the XRD-pattern and the high hardness values (Fig. 3C) exclude the existence

Table 1
Laser treatment parameters for different samples.

	Sample I	Sample II	Sample III	Sample IV	Sample V	Sample VI
Substrate material	Steel	Nickel	Aluminium	Steel	Steel	Steel
Shape	Single layer	Single layer	Single layer	5 layers	3-D structure	3-D structure
Base area	10 mm × 15 mm	10 mm × 4 mm	10 mm × 6 mm	5 mm × 5 mm	8 mm × 13 mm	30 mm × 13 mm
Laser power (nominal)	300 W	500 W	500 W	500 W, 300 W for the 1st layer	500 W, 300 W for the 1st layer	500 W, 300 W for the 1st layer
Preheating temperature	300 °C	500 ° C	500 °C	300 °C	300 °C	300 °C
Scanning speed	200 mm/min	150 mm/min	200 mm/min	200 mm/min	200 mm/min	200 mm/min
Powder feed rate	0.36 g/min	0.36 g/min	0.72 g/min	0.36 g/min	0.36 g/min	0.36 g/min