



# Synthesis and characterization of superalloy coatings by cathodic arc evaporation



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## ABSTRACT

Superalloy targets were produced from Ni-(Al-C-Co-Cr-Mo-Ta-Ti-W) powders by spark plasma sintering technology. The crystalline structure of the as-produced targets was investigated by XRD analysis and compared with the evolution of phases resulting from the operation of the cathodic arc at the target (cathode) surface. Coatings were synthesized at superalloy substrates utilizing these superalloy targets in non-reactive and reactive evaporation processes. Synthesized coatings and target surfaces were compared with respect to chemical composition and crystal structure. The interface between coating and superalloy substrates was investigated by TEM. As an example, a complete layer stack was synthesized by cathodic arc evaporation starting from the superalloy substrate - superalloy coating interface to a fully oxidized superalloy coating showing epitaxial growth in the interface to the superalloy substrate.

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## 1. Introduction

The research for materials utilized at high temperature and in oxidizing and corroding environments has been an ongoing effort for applications in aircraft, gas turbine and combustion engines [1–10]. Despite the different final utilization and the difference in design and dimensions, the trend in these industries is going towards the same goal, which is a continuous improvement of engine efficiency to reduce fuel consumption but also to comply with more strict regulations concerning CO<sub>2</sub> emission. This implies running the engine at higher temperatures, thus increasing consequently the need for more robust, stable and resistant base materials operated in harsh environment at different sections of a turbine engine. Even with the use of the most advanced materials such as superalloys or composites, coating technologies cannot be by-passed when it comes to improving the lifetime of the components by increasing the resistance to oxidation, wear, erosion and corrosion at high operating temperatures [11–13]. Despite the fact that the coating technologies introduced many decades ago are well established and continuously being improved utilizing new processes and new coating materials, the coating systems produced on engine

components require increased complexity. Therefore, the interactions e.g. between layers, the methods for surface preparation, heat treatments and diffusion issues become increasingly important [14]. Moreover, the requirements of the next generation engines are quite challenging for these existing technologies due to their limitations and the inability to provide the required bulk material properties. Typical coating systems in gas turbines are made of several layers, generally consisting of a bond coat, a thermally grown oxide and a top ceramic layer. Bond coats, which are used to protect the turbine against oxidation, are typically produced either by diffusion processes for PtAl, electron beam physical vapour deposition (EB-PVD) or low pressure plasma spraying for MCrAlY [15,16]. There have been also attempts to utilize Physical Vapour Deposition (PVD) by cathodic arc for the bond coat [17,18]. The bond coat and the top ceramic layer form the so-called thermal barrier coating (TBC). The top ceramic coating is produced by atmospheric plasma spraying as a porous coating or EB-PVD as a columnar structured coating [19–22]. The design of the bond coat is challenging because it has to realize two sophisticated interfaces: the one to the superalloy substrate to guarantee mechanical stability for a wide temperature range, and the other one to the porous oxide providing an excellent oxygen barrier. This implies not only an intelligent design of the bond coat, but it also requires high reproducibility in the fabrication of the coating system (layer stack).

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In this work, a bond coat concept is investigated which is sketched in Fig. 1. The approach is based on the formation of a “substrate-identical” interface (IF-1) to the bulk superalloy substrate (SA-S) and a subsequent transition from IF-1 to a partially or fully oxidized coating ending in a second interface (IF-2). This IF-2 may be an oxygen diffusion barrier and/or a nucleation layer for a porous oxide as it is utilized in the design of a TBC [23]. It could also be an oxygen diffusion strengthened coating or a mixture of oxides which are formed during the oxidation of the superalloy vapour. The whole layer stack is synthesized in one process under vacuum conditions typical for PVD. Non-reactive and reactive arc evaporation is utilized to produce this coating design by *in-situ* processing. This work discusses the manufacturing of targets from powder more or less identical in chemical composition with the bulk superalloy, examines the influence on the chemical composition and phase evolution at target surface for the evaporation by arc under non-reactive and reactive (in oxygen) conditions, and characterizes the coatings obtained under these conditions. A layer stack was synthesized for which the transition from IF-1 to a fully oxidized superalloy coating is demonstrated. Main emphasis of this work was the investigation of IF-1 and the substrate-identical superalloy coating.

## 2. Experimental

The substrates as well as the targets were produced from powders with the chemical composition listed in Table 1 (2nd column). The composition of the powders was analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS). It corresponds to the elemental composition of the superalloy PWA1483 (proprietary alloy of Pratt & Whitney). For the experiments, the substrates as well as the targets were fabricated by spark plasma sintering (SPS) at approximately 1200 °C and 30 MPa (PLANSEE Composite Materials GmbH). This technology has been increasingly utilized during the last decade to synthesize difficult to fabricate materials with dedicated properties [24,25]. In our studies, it was required to achieve a mechanical stable and smooth target surface after arc operation under non-reactive and reactive conditions. Because of the method of manufacturing, it is likely that this material differs from the industrially utilized PWA1483 bulk material produced by melting and casting [26]. Considering this, we will further denominate this material as SA-S (if it is utilized as substrate) and SA-T (if it is used as target for the evaporation). Small discs (Ø 60 mm) were produced from this material and machined to the size of (30 mm × 10 mm × 5 mm) for the SA-S. In identical processes, the SA-T discs (Ø 150 mm) were fabricated.

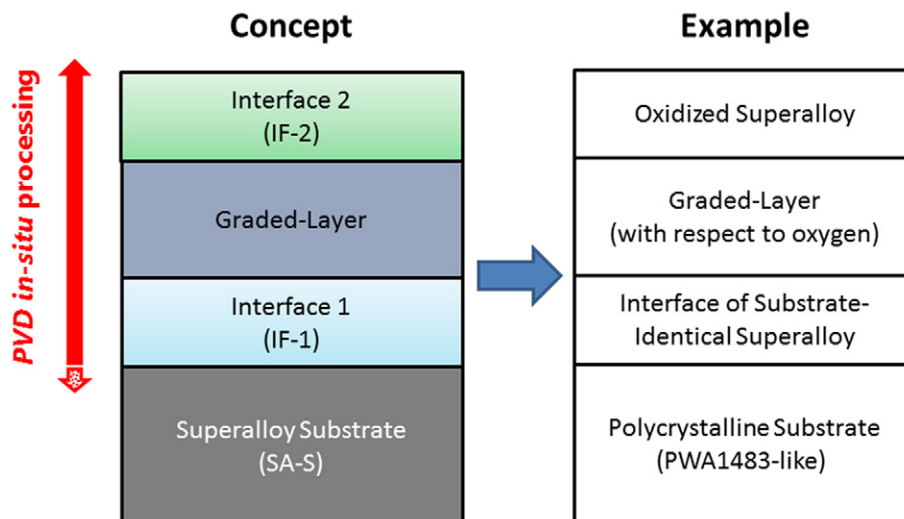
**Table 1**

Elemental composition (measured on the target surface) of the as produced target and the targets after utilization in evaporation processes A and B in comparison with the original powder composition utilized for target manufacturing.

Element	Powder composition (ICP-OES and ICP-MS) [wt%]	Target composition (EDX)		
		As produced [wt%]	Process A [wt%]	Process B [wt%]
C	0.07	n.a.	n.a.	n.a.
Al	3.6	3.8	3.2	1.6
Ta	5	8.3	5.5	4.6
W	3.8	4.8	3.6	4.7
Mo	1.9	2.0	1.3	1.6
Ti	4.1	3.8	3.1	2.2
Cr	12.2	11.1	14.3	11.8
Co	9	8.7	9.1	9.6
Ni	60.33	57.5	59.9	63.9

Table 2 lists the main process parameters utilized in the cathodic arc evaporation using the SA-T as cathodes. Before deposition, the process chamber was evacuated below  $0.2 \times 10^{-3}$  mbar and standard heating and etching steps were performed to ensure a sufficient coating adhesion to the substrate. In process A, the total pressure drops during deposition slightly due to gettering of residual gases by the metallic vapour. The total pressure of process B starts with  $0.3 \times 10^{-3}$  mbar (shorter gettering) and reaches finally  $25 \times 10^{-3}$  mbar if stabilized in oxide synthesis. A net deposition time of 45 min was chosen for the non-reactive process (metallic vapour only) and was increased to 240 min for the reactive processes in oxygen. This is due to the reduced evaporation rate of the SA-T in pure oxygen reactive gas, resulting in coating thicknesses of 1.5 µm (non-reactive process A) and 2.2 µm for whole layer stack of process B, respectively. The cathodes were operated with DC arc currents of 140 A, either in metallic vapour only, or with a gas flow of 800 sccm oxygen (reactive processes) using an INNOVA batch-type production system of Oerlikon Surface Solutions AG. SA-S together with sapphire substrates were coated at substrate temperatures of approximately 550 °C. Only one arc source was utilized for deposition. A symmetric bipolar bias voltage of –40 V with a frequency of 25 kHz and a negative pulse length of 36 µs and 4 µs positive pulse length was applied to the substrate during processing in oxygen.

The target surface was analyzed in a LEO 1530 scanning electron microscope (SEM). The chemical compositions of the SA-T and SA-S were measured by energy-dispersive X-ray spectroscopy (EDX) in the SEM. The errors in this standard-less EDX analysis is estimated to be 3 wt%. The small amount of C in the target as well as in the coating, and the C



**Fig. 1.** Schematics of the new bond coat concept (left) and an example (right) realized in this work by *in-situ* processing utilizing cathodic arc evaporation.

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