



Comparison of coating processes in the development of aluminum-based barriers for blanket applications



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HIGHLIGHTS

- Electrochemical processes ECA and ECX are suitable for Al deposition on RAFM steels.
- ECA and ECX are able to produce thin Al layers with adjustable thicknesses.
- All aluminization processes need a subsequent heat treatment.
- Scales made by ECA or ECX exhibit reduced thicknesses compared to HDA.
- ECX provides higher flexibility compared to ECA to produce scales on RAFM steels.

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ABSTRACT

Reduced activation ferritic-martensitic steels (RAFM), e.g. Eurofer 97, are envisaged in future fusion technology as structural material, which will be in direct contact with a flowing liquid lead–lithium melt serving as breeder material. Aluminum-based barrier layers had proven their ability to protect the structural material from corrosion attack in flowing Pb–15.7Li and to reduce tritium permeation into the coolant.

Coming from scales produced by hot dipping aluminization (HDA), the development of processes based on electrochemical methods to produce defined aluminum-based scales on RAFM steels gained attention in research during the last years. Two different electrochemical processes are proposed: The first one, referred to as ECA process, is based on the electrodeposition of aluminum from volatile, metal-organic electrolytes. The other process called ECX is based on ionic liquids.

All three processes exhibit specific characteristics, for example in the field of processability, control of coating thicknesses (low activation criteria) and heat treatment behavior. The aim of this article is to compare these different coating processes critically, whereby the focus is on the comparison of ECA and ECX processes. New results for ECX-process will be presented and occurring development needs for the future will be discussed.

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1. Introduction

Steel structures made of reduced activation ferritic martensitic steels (RAFM-steels) are proposed for application within the blanket system of a future fusion reactor. This claims development of suitable steels, functional scales and the qualification of them to fit the desired requirements in such challenging environments.

In the proposed HCLL (helium-cooled lead lithium) concept for a future fusion reactor, the envisaged breeding material is a flowing, liquid Pb–15.7Li melt, which has direct contact to the desired structural steel. The envisaged operating temperatures in this concept lie above 480 °C and reach in some designs 550 °C. Under these

conditions bare RAFM-steels such as Eurofer 97, Optifer IVa, Manet I and F82H-mod. suffer from uniform but significant corrosion attack [1,2]. For example Konys et al. reported relatively dramatic dissolution rates of Eurofer 97 of about 400 μm per year at operation temperatures of 550 °C and a Pb–15.7Li flow rate of 0.22 m/s [3]. To enhance the performance limits of such steels, the application of functional coatings is essential for the realization of a future blanket system. Coatings on RAFM-steels are envisaged to provide (a) reduced tritium permeation rates through the structural material and (b) corrosion protection properties, in contact with the liquid breeder material (Pb–15.7Li) [4]. To fulfill these requirements in Pb–Li systems, aluminum-based coatings on RAFM steels are seem to be advantageous since they are able to offer both properties at once. Thereby, an Al₂O₃ layer formed on the surface of aluminum-rich coatings acts as a barrier to provide reduced tritium permeation rates [5]. The ability of aluminum-rich coatings

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to reduce corrosion rates of RAFM steels in flowing Pb–Li melts compared to the bare steel material, were shown in studies by Glasbrenner et al. [6] and Konys et al. [7] for operating temperatures between 480 °C and 550 °C and flow rates up to 0.3 m/s.

In the past, several coating processes were under more or less intensive investigation to produce the desired aluminum-rich scales on RAFM steels. Each technique possesses inherent advantages and disadvantages with respect to plating rates, thickness distribution, coating of different geometries, scale formation, expenses and stability in the Pb–15.7Li environment. Besides chemical vapor deposition (CVD) and vacuum plasma spraying (VPS) [8], main research was focused on the hot dip aluminizing process (HDA) to produce Fe–Al scales on RAFM steels, in the beginning of these research activity [6,7,9].

Meanwhile, different processes based on electrochemical techniques which provide specific advantages, were introduced in the field of fusion technology and are still under intensive investigation. They seem to be an interesting new alternative to produce aluminum-based barriers for blanket applications [4,10] which additionally promise better technological and industrial relevance. The first one introduced, referred to as ECA process, is based on a commercialized Al electrodeposition process [11] with an additional heat treatment developed at KIT [10,12]. Unfortunately, this ECA process exhibited some disadvantages such as relatively high costs, due to complex and expensive equipment which is needed to fulfill encapsulation and safety requirements for the whole process, relatively low scattering behavior and low flexibility in adapting deposition parameters like current densities and deposition temperatures, to influence the microstructure of the deposited layer. The latter is essential, since the microstructure influences the formation of the Fe–Al scale during the subsequent heat treatment (HT) [13]. Therefore, another electrochemical deposition process based on ionic liquids (ECX process) was developed at KIT in the last years, to overcome some of the drawbacks of the ECA process [14].

The present paper describes both electrochemical processes developed at KIT in more detail and presents scales produced electrochemically. The results are discussed in respect to scales produced by the HDA process in the past.

2. Aluminization

2.1. Hot-dip aluminization

In the hot-dip aluminization process (HDA), the parts to be plated are immersed into a melt of aluminum (e.g. $T = 700$ °C) for a certain time, whereby short dipping times of 30 s are to be favored ensuring complete wetting of the steel parts but also minimizing the amounts of aluminum reacting with the steel (low activation criteria). Afterwards the plated parts are removed from the Al melt and cooled naturally [1,8]. Besides an aluminum layer on top of the sample, an aluminum-rich reacting zone consisting of mostly brittle Fe_2Al_5 phase is formed on the substrate material, e.g. RAFM steel, due to relatively high temperatures of the melt [8]. The conversion to suitable barriers is done by performing a heat treatment subsequently.

2.2. Aluminization by electroplating

2.2.1. General electroplating

Compared to other plating techniques such as hot-dip metallization or CVD, electrochemical metal deposition in general provides some favorable properties:

Table 1

Comparison of process parameters and properties of electrochemical Al deposition processes ECA and ECX.

	ECA	ECX
Metal source	$\text{NaF-Al}(\text{C}_n\text{H}_{2n+1})_3$	AlCl_3
Electrolyte	e.g. Toluene, xylene	[Emim]Cl, [Bmim]Cl
Operating temperature	95–103 °C	<100 °C
j_{mean}	10 mA/cm ²	10–20 mA/cm ²
Current efficiency	Ca. 100%	Ca. 100%
Deposition rates	12 μm/h (10 mA/cm ²)	25 μm/h (20 mA/cm ²)
Pulse-plating	Yes [18]	Yes [15–17]
Vapor pressure	High	None/very low
Inflammability/sensitivity	High/sensitive to oxygen, hydrogen	Low/sensitive to hydrogen

- Good controllability of layer thickness, from nanometer to millimeter scale by controlling current density and time (*Faradays law*).
- Thickness distribution is controllable by choosing special shaped anodes and/or adjusted deposition parameters (current density (j), pulse-plating) → complex shaped parts could be plated.
- Low temperature process (e.g. <100 °C) → low energy consumption

Another advantage is the long history of commercialized plating processes and a long experience for plating different metals on a variety of substrates.

However, these well described processes are mainly based on aqueous electrolytes, and therefore reached some limitations in the past. For example, the plating of hydrogen sensitive materials such as high strength steels is difficult and the deposition of and on highly electronegative metals is impossible due to hydrogen evolution during the plating process or incomplete reduction to metallic state. To overcome these limitations new classes of water-free electrolytes are under development to introduce electrochemical processes with their advantages to new technological applications, such as electrochemical aluminum deposition which was possible earlier only from high temperature molten salts.

2.2.2. Electrochemical deposition of aluminum

Due to its very negative standard potential of about -1.7 V vs. NHE (normal hydrogen electrode), aluminum plating from water-based electrolytes, is not possible [11]. Therefore, non-aqueous electrolytes are required for aluminum electrodeposition. Besides the electrodeposition from molten salts, different low temperature electrodeposition processes (<100 °C) were developed in the past.

The older process, referred to as ECA process is a commercialized plating process based on metal-organic electrolytes were Al-alkyl compounds (as aluminum source) are dissolved in a volatile organic solvent, e.g. toluene [10,11,15]. Due to the use of highly volatile solvents, and highly water sensitive organoaluminum, the plating process has to be done in a very good protective environment.

A second, more recent plating process referred to as ECX in the field of fusion technology is based on ionic liquids (IL). This is a relatively new class of electrolytes that gained a lot of attention in the field of metal deposition during the last decade [16]. Ionic liquids are defined as ionic compounds that are liquid at temperatures below 100 °C.

Electrodeposition of aluminum from such electrolytes mainly based on eutectic mixtures of an imidazolium-type IL and AlCl_3 as metal source, whereof 1-ethyl-3-methylimidazolium chloride ([Emim]Cl) and 1-ethyl-3-butylimidazolium chloride ([Bmim]Cl) are the most commonly used ILs for aluminum deposition [17–19]. The main process parameters and properties for ECA and ECX process are summarized and compared in Table 1.

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