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Electrochemical techniques as innovative tools for fabricating divertor and blanket components in fusion technology

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Electrochemical techniques such as electroplating and electrochemical machining (ECM) are established processes in a variety of industrial applications, such as corrosion protection coatings on steel and high electro-conductive coatings in electrical industry. The main advantages of these processes are: Good controllability of coating thicknesses, cost-effectiveness and good scalability from scientific settings to industrial scale. For applications in fusion technology, material development needs to meet unique and specific requirements, e.g. high heat-flux properties and corrosion resistance to liquid metals. Therefore, uncommon materials like tungsten, CuCrZr, RAFM-steels have to be processed for which commercially available electrochemical processes show various limitations at the moment. To overcome these limitations, selected methods were introduced in research of divertor and blanket component manufacturing leading to promising electrochemical-based processes contributing their inherent advantages to a variety of fusion applications, such as the ECX process to fabricate Al-based corrosion barriers on RAFM steels resistant in flowing Pb-15.7Li. Others are the electrodeposition of metals fillers on tungsten and CuCrZr substrates for joining applications and the machining of tungsten surfaces by ECM in first wall applications. This paper provides an overview over these processes and describes the achieved benefits of using electrochemical processing techniques for applications in fusion technology.

Keywords: Electrochemical techniques, Al-based barriers, Tungsten, Joining, Blanket systems, Corrosion

1. Introduction

Over the last 150 years electrochemical processes such as electroplating, anodizing, electrochemical dissolution/ electropolishing, electroforming have been used in a variety of industrial processes. Despite electroforming, the main aim is to enhance the surface properties of a substrate material by electrodeposition of functional and/or decorative coatings or to improve the surface properties by defined electropolishing or electrochemical dissolution / machining, respectively. On the one hand especially electroplating of metals and electropolishing are widely established in industry with high volumes of mass products due to their cost effectiveness and versatility, including the superior understanding of scaling up processes compared to “dry” processes [1]. Main high volume applications of electroplating of metals are found for instance in automotive industry, where integrated electroplating facilities are used, e.g for electrodeposition of zinc or zinc-nickel alloy coatings on steel sheet for anti corrosion purposes [2,3]. Widely established are also the electrodeposition of chromium coatings for decorative purposes on bathroom fittings made of brass and even plastics and the deposition of hard chromium coatings. The latter provide unique material properties such as high melting point, good wear and corrosion resistance, low coefficient of friction and low wetting ability making it the preferred coating for many applications of highly stressed machine parts and tools of hardened steels [4]. Another important application field is selective electrodeposition of hard-gold and tin for example connectors in the electronic industry. Here optimized continuously working reel-to-reel plating lines are used for cost effective selective plating of removable connectors (hard-gold) and leadframes (Sn, for corrosion resistance and improved solderability) [5-7]. On the other hand also electrochemical processes that remove material such electropolishing (EP) and electrochemical machining (ECM) are established as surface improving processes and for material processing of difficult-to-cut materials (ECM) [8]. In case of EP, stainless steel components and other metals could be finished and mirror-like surfaces could be produced without introducing mechanical damages to the substrate material, achieving metal surfaces with enhanced properties [9-11].

Despite electrodeposition of coatings and electropolishing also electroforming, i.e. fabrication of complex shaped parts by electrodeposition, has achieved some industrial relevance where thick electrodeposits are applied on a pre-shaped mandrel which is removed after electrodeposition [12]. Due to its high reproducing ability of the mandrels surface quality electroforming gained some importance in fabrication of, e.g. reflectors and combustion chambers in aerospace industry and for wave-guides in high-frequency technology and also for molds in the automotive industry. Commonly used metals for electroforming, also in microstructures, are nickel and its alloys, copper [13-15] and to a lesser extent gold for jewelry applications [15].

However, as electrochemical processes are widely established in industry to produce, depending on the application, high quality surfaces with a variety of adjusted properties, the application of this often cost effective technique in fusion technology is up to date limited. Maybe this is due to the fragmented structure of this industry, despite of automatized large volume applications, and therefore a lack of knowledge and good will exists to transfer the extensive know-how of these

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electrochemical producers to new applications, especially when “uncommon” materials are used, as it is the case in fusion technology.

In this paper, three different electrochemical processes are described which were developed in the last decade at KIT refereeing to arising problems and their solution in special applications in fusion technology. The aim is to show that the transfer from established processes to new materials is possible and effective, showing the potential of electrochemical processes to transfer the advantages of these processes to future fabrication procedures in fusion technology.

2. Principles of electrochemical techniques

The main principle of electrochemical processes is schematically depicted in figure 1: By applying an external electrical current between two electrodes, metal species dissolved in the surrounding electrolyte could be reduced i.e. deposited at the surface of a substrate that acts as cathode (electrodeposition, electroforming) to form the intended coating. In case of EP and ECM, the workpiece acts as anode and the metal surface is anodically dissolved into an electrolyte. In both cases the applied electrical current/charge is proportional to the deposited or removed mass of metal species according to Faraday’s law (equation 1), whereby M is the molar mass of the metal, F the Faraday constant (96500 C/mol), z the amount of electrons, I the flown current in a distinct time t:

$$m=\frac{Q\cdot M}{F\cdot z}=\frac{I\cdot t\cdot M}{F\cdot z}$$

Through this relationship the mass changes i.e. the amount of deposited (coating thicknesses) or removed metal could easily be controlled by measuring and controlling the parameters I and t, which represents one of the main advantages of electrochemical techniques. The applicable current densities are limited due to specific diffusion coefficients of metal species in the electrolyte depending on, e.g. temperature, concentrations (salt solubility in electrolyte). In practice current densities and therefore deposition/dissolution rates can be further increased by orders of magnitude for economic reasons by adjusting electrolyte agitation and/or lateral movement of the workpieces. As a consequence deposition rates of up to 100 µm/min could be reached [16]. Articles reported current densities exceed 200 A/dm² in industrial continuously working electrogalvanizing lines which equals to deposition rates of approx. 1 µm/s and above [3,17].

Due to the dependence on the electrical field also complex shaped parts could be treated by electrochemical techniques. For example inner walls of tubes are accessible by special anode/cathode configurations. Last but not least electrochemical processes could be upscaled under moderate costs from lab scale to industrial scale [2,17-19]. This was shown several times in the past and a lot of experience exists in this field in the industry, for example electrodeposition of Cu and electropolishing of the inner wall of up to 10 meter long tubes are reported [19,20].

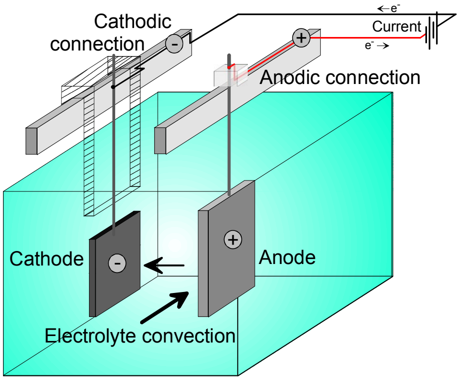

Fig. 1: Scheme of the principle of electrochemical deposition / dissolution.

3. Electroplating for joining applications in fusion technology

3.1. Motivation

Electroplating of nickel interlayers is successfully used to realize sufficient joining of parts in many applications, e.g. assisting diffusion bonding of ceramics [22], brazing of WC-Co and stainless steel [23,24], in friction stirrer weldings of Ti or Al alloys and steel [25,26] and direct joining of dissimilar metals such as Cu and Al [27] and Be and stainless steel [28] or Cu [29]. However, in fusion technology some special requirements have to be considered during material selection. One of these special materials is tungsten, which has to be joined with the RAFM steels for divertor, or copper or copper alloys, e.g. CuCrZr in low temperature heat sink applications. The application of conventional brazing tools and fillers showed weaknesses ranging from, e.g. wetting of tungsten up to embrittlement of fillers under operation conditions. Consequently, a process is required to find a solution and close the gap between the requirements of the fusion technology and the practical implementation as a component. The electroplating processes offer alternatives for these needs.

3.2. Joining of W-W and W-steel by electroplating assisted brazing

For application in a future divertor, W as armor material has to be joined with W-based alloys and steel as structural material. The operating temperatures are nearly 1200°C and 700°C. Ni-based fillers, e.g. STEMET [30,31] had shown their potential but some unwanted behavior as well. A direct homogeneous application of the filler material on the workpieces that have to be joined by electroplating would improve the manufacturing and cost efficiency. The main problem in the development of electroplating assisted brazing was the development of a sufficient pretreatment of tungsten

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Fig. 2: Electroplating assisted brazing of two tungsten parts, by electroplating of Cu and Ni layers as brazing fillers on W parts: Joined W parts after brazing (left) and after shear testing (right).

surfaces to activate them to ensure a good adhesion of the electroplated metal coatings, e.g. Cu, Ni and Pd. An alkaline etching solution based on potassium cyanate was found to be optimal for electroplating sufficient thicknesses of Ni and Cu on W as filler materials. The joined parts of W-W and W-steel could be achieved by brazing at 1100°C while the electrodeposited elements Ni and Cu diffuse into each other to form a solid solution [31] and make a strong metallurgical bonding to the substrates. The mechanical strength was tested for some parts and in case of W-W joints the failure occurs in the tungsten base material outside of brazing zone (see figure 2). Further development of electroplated filler materials for brazing focused on the substitution of Ni as alloying element by Pd instead. For this purpose a commercial Pd electrodeposition procedure was adapted for fusion relevant substrates and W-W and W-steel joints could be successfully fabricated by brazing in combination with electroplated Cu layers as filler materials [31,32].

3.2. Joining of tungsten to Cu-based materials by electroplating assisted diffusion bonding

For low temperature heat sink applications W has to be joint to Cu-based materials such as CuCrZr. Therefor high brazing temperatures could not be applied. As for brazing applications the joint parts could efficiently electroplated by Cu and Pd. It was found that the Cu-based work piece could be Pd plated effortless due to high hydrogen overvoltage and no H-embrittlement of Pd occurred and the W part is coated by Cu. The parts are joined in vacuum at 700°C under a load of 2 N/mm². Reported measured shear strength of these joints reached 100 MPa depending on bonding conditions [33].

4. Electroplating for fabricating Al-based coatings for corrosion protection in Pb-Li

4.1 Motivation

For liquid breeder concepts such as HCLL, DCLL, WCLL, Al-based coatings had been identified to solve corrosion related problems such as precipitation of corrosion products / tube plugging [34] coming from poor corrosion resistance of RAFM steels in flowing breeder Pb-Li, which could exceed 0.4 mm/a at high flow velocities [35]. Additionally, Al-based coatings are considered to reduce permeation of tritium. However, coatings made by, e.g. CVD, PVD to a lesser extent HDA revealed some unfavorable corrosion behavior in Pb-15.7Li [36-39]. In addition, coatings made by HDA revealed local corrosion attack of the coating itself [39] and poor coating thickness distribution leading to crack formation during the mandatory HT, causing reduced tritium permeation factors [37,38].

4.2. Al-based coatings by electrodeposition and a subsequent HT

A favorable coating thickness control and distribution and lower overall thicknesses (low activation criteria) compared to HDA coatings are expected by applying electroplating as coating technique. However, Al can be deposited only from non-standard water-free electrolytes due to its electronegativity. Therefor two electroplating processes had been developed in the last decades for application in fusion technology: the first was based on toluene based electrolytes with Al-alkyls as metal source [40]. This process had already gained some industrial relevance for some applications in automotive [18,41], aircraft and space industry [42] in the past, but showed some disadvantages/safety concerns resulting from the volatile and combustible components and the limited variability in process parameters [43]. Furthermore, coatings made by this so called ECA process, were to a lesser extent, locally attacked in flowing Pb-15.7Li [44]. To overcome the limitations of ECA process, the ECX process was developed in the last decade which is based on electrodeposition of Al from ionic liquids and a subsequent heat treatment. The coating properties, e.g. surface morphologies [43], corrosion behavior are superior of ECA coatings and corrosion rates in flowing Pb-Li are reported to be < 10 µm/a after 10,000 h of exposure [45]. Figure 3 illustrates the effect of Al-based coatings made by ECX process on material loss in flowing Pb-Li in contrast to bare Eurofer, underlining the great benefits of ECX coatings for future fusion applications.

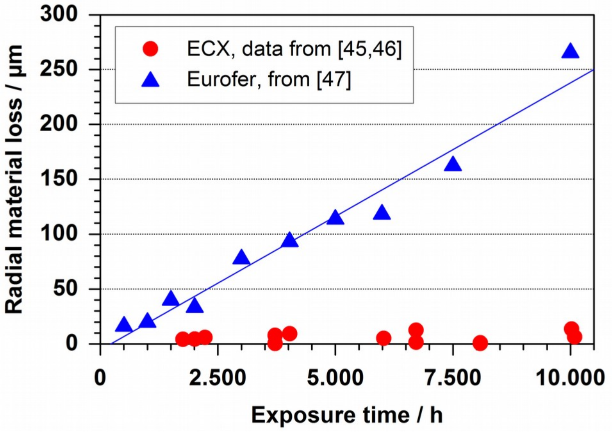

Fig. 3: Comparison of determined radial material losses of bare Eurofer (blue) and ECX coated Eurofer (red) in flowing Pb-Li at 550°C and 0.1m/s. Data from [45-47].

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5. Electrochemical Machining (ECM) of tungsten based plasma facing components

5.1. Motivation

Tungsten is used in a variety of applications within a fusion reactor, e.g. as plasma facing material of the divertor and blankets, respectively. However, machining of hard and brittle materials such as tungsten or NiCr alloys by turning/milling or electrochemical discharge machining (EDM) have been reported as relatively difficult as formation of unacceptable micro-cracks at the machined surfaces occurred leading to crack propagation during thermal cycling in divertor components [48,49]. Besides the implanted mechanical failures, methods like turning or milling are often not suitable for mass production of work pieces of difficult-to-cut materials, e.g. W or Inconel 718 due to high costs resulting from high tool wear and high time consumption [8,50,51]. Non-mechanical techniques such as ECM are able to make such machining more efficient as it is already shown. in aerospace industry, where ECM techniques have already successfully replaced milling and turning in the fabrication of integral bladed rotors and turbine wheels due to lower costs and a higher accuracy [52]. Another important application is the surface structuring of hard to machine surfaces in the micrometer range with high accuracy and high aspect ratios. Mask assisted electrochemical machining has already shown some encouraging results in the fabrication of ink-jet nozzles in industry [53] and for microstructuring of materials such as Si and Ti [54-56]. However, as in the case of electroplating on tungsten substrates (see section 3) the natural tungsten oxide layer prevents the direct use of existing ECM processes as known from steel processing [57,58]. Therefore, a new approach has been followed to enable ECM of tungsten workpieces for application in fusion technology.

5.2. Results of ECM of tungsten materials and structuring of tungsten surfaces

The breakthrough of ECM for tungsten materials was the development of a new alkaline two-component ECM electrolyte enabling the sufficient dissolution of anodically removed, oxidized tungsten products [59]. Subsequently, the potential of ECM of tungsten was demonstrated for fusion applications by Holstein et al., by adapting two standard ECM techniques for fusion related requirements. On the one hand copying-tool ECM was used to fabricate different geometries of up to 1 mm depth (removal rate 10 µm/min) on tungsten plates using different tool designs (parallel slots and 16-fold star) and electrochemical parameters [58,59]. For the latter it was shown that pulsed electrical currents drastically improved the sharpness of edges at high aspects ratios [59]. On the other hand standard through-mask ECM was used to apply arrays of spherical and cubic microstructures of a size of 100 µm to 500 µm on tungsten surface to enhance the adherence of vacuum plasma sprayed (VPS) W-Fe gradient coatings on tungsten for blanket applications. Therefor different kinds of masks and photo resists for UV lithography were tested, while best results with respect to shaping accuracy were achieved by using laser-structured lithography masks [60]. Figure 4 depicts such a W-surface with an accurate array of cubic structures (100 µm x 100 µm) on an overall test area of 2.5 cm^2 produced by the enhanced through-mask ECM, showing the potential of this effective structuring technique.

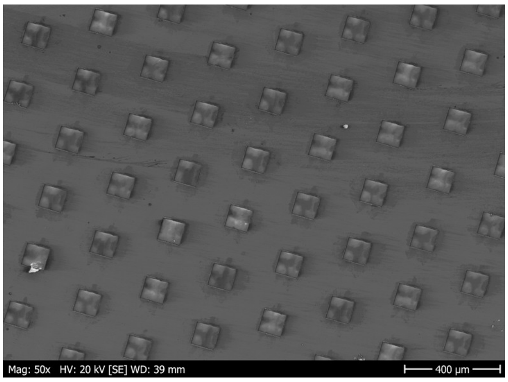

Fig 4: SEM image of a micro-structured W surface processed by an enhanced ECM process developed at KIT.

6. Conclusions

This paper presented three examples where and how electrochemical processes could bring in their specific advantages such as excellent control of coating thicknesses or removal rates in to selected applications in fusion technology. The intention of this paper is also to show the great potential of electrochemical techniques in general for manufacturing proposes, as they are widely established in a variety of applications in industry from large scale plating lines to single part manufacturing in aerospace industry. Unfortunately, the fragmentation of this industry branch with its highly specialized, usually rather small companies, makes it sometimes hard to find appropriate collaborators for projects in fusion technology development. However, materials manufacturing in fusion technology should not oversee these interesting powerful techniques, because as it was shown in the paper, the established processes could be adapted for fusion purposes and requirements easily. Indicating that these often cost efficient, flexible and up-scalable processes can contribute to the cost sensitive manufacturing of diverse components, e.g. tungsten manufacturing due to the benefits they provide. On the other hand, process development is also ongoing in the field of electrochemical processing of materials, where additive manufacturing by electrodeposition [61] is under investigation. Material engineers also should lay an eye on the developments in the field of new electroplating processes including upscaling of recently developed processes to industrial scale [19] and the intensified reasearch in the field of the electrodeposition from ionic liquids. Here new attempts are made to improve the properties of "common" metal layers e.g. Pd with lower embrittlement [62], or to electrodeposite refractory metals such Cr [19], Zr [63], Nb and Ta [64,65], which are, in most cases, until now only accessible be electrodeposition from molten salts at high temperatures. Thus, these new developments might be of

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interest for future applications in fusion technology too, as it was shown during the successful development of Al-based barrier based on electrodeposition of aluminum (ECA and ECX-process) in the past. However, material developers in fusion technology should be open to these or any new developments in this field even when electrochemical manufacturing techniques are often not in the curriculum during the training of e.g. mechanical engineers.

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