



Short communication

A new method for practical electrodeposition of aluminium from ionic liquids

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ABSTRACT

Successful electrodeposition of aluminium from ionic liquids has been long achieved scientifically. Nevertheless, standard industrial procedures have not yet been established due to the difficulties associated with using closed system filled with inert gas for the electrodeposition process. This paper presents a new methodology in which electrodeposition of aluminium is conducted in air, after preparation of ionic liquids in a glove box and covering them by a nonwater-absorbable layer of particular organic compound. This compound is stable and shows no reaction with the ionic liquid. Using this new methodology, functional aluminium layers were successfully deposited from a first generation ionic liquid $\text{AlCl}_3/1\text{-ethyl-3-methylimidazolium chloride}$ – [EMIm]Cl – (60/40 mol%) on low carbon steel. Cyclic voltammetry (CV) measurements revealed that there was no significant difference in electrochemical properties characteristic of Al deposition when conducted in open air. SEM/EDX assessments showed that uniform, dense and adherent Al layers were obtained. Adherence of Al to the steel substrate was improved via in-situ electrochemical etching.

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1. Introduction

Electrochemical studies of aluminium plating from room temperature ionic liquids have been the subject of a number of studies over the years, e.g. Refs. [1–10]. Nevertheless, the industrial processing of aluminium electrodeposition has not yet been established. A manufacturing pilot plant was developed by Nisshin Steel Co., Ltd., (Japan) based on this technology, but the plant was not economically viable [11]. The electrodeposition process must be performed inside an inert gas-filled glove box free of oxygen and humidity. Also, commercial aluminium electroplating processes from organic baths are too limited. According to the literature surveyed, only two processes – SIGAL (Siemens Galvano Aluminium) process [12,13] and REAL (Room-temperature Electroplated Aluminium) process [14] – have been reported. The SIGAL process is currently licensed to AlumiPlate, Inc. [11]. Although high quality Al can be electrodeposited by these processes, organo-aluminium baths are self-ignitable in air and have vigorous reaction with water [15,16].

Thus, the importance of our pioneer method [17] emerges to conduct electrodeposition of aluminium in air with the aim at facilitating the plating procedure of this technology. The present paper shows the possibility of electrodeposition of functional aluminium layers from $\text{AlCl}_3/[\text{EMIm}]\text{Cl}$ ionic liquid in ambient atmosphere, through insulating

the electrolyte from air with a layer of specific hydrocarbon compound. This enabled to electrodeposit uniform, dense and adherent Al layers on low carbon steel outside the glove box.

2. Experimental details

2.1. Chemicals

The chemicals used are listed as following: –i) aluminium chloride anhydrous – AlCl_3 – (Fluka $\geq 99\%$ purity), –ii) 1-ethyl-3-methylimidazolium chloride – [EMIm]Cl – (Fluka $\geq 95\%$ purity) with chemical formula $\text{C}_6\text{H}_{11}\text{ClN}_2$, and –iii) n-decane ($\text{C}_{10}\text{H}_{22}$, with Merck purity).

2.2. Equipment

Preparation of electrodeposition bath was conducted in a simple home-made glove box filled with argon. Cyclic voltammogram (CV) measurements and electrodeposition experiments were performed with Potentio-Galvanostat (model Wenking PGS 95) controlled by a PC, using a conventional three-electrode cell.

2.3. Procedure

Ionic liquid melt was made by stirring a mixture of 60 mol% of AlCl_3 and 40 mol% of [EMIm]Cl with stirring in a small beaker inside an argon-filled glove box. The mixture was completely converted into

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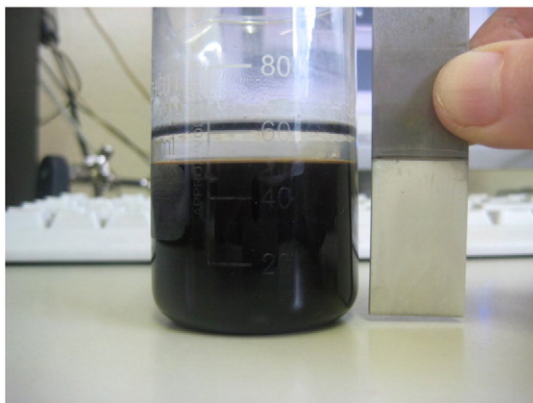


Fig. 1. Photograph of a beaker containing $\text{AlCl}_3/[\text{EMIm}]\text{Cl}$ ionic liquid insulated from air by a floating layer of decane, in addition to Al electroplated steel strip.

liquid in a few minutes as a result of the exothermic reaction between its ingredients. Then, a layer of decane was added which floated on the ionic liquid. This layer acted as a barrier preventing any reaction with air, see Fig. 1. This enabled to transfer the beaker containing the ionic liquid outside the glove box for electrodeposition experiments. The ionic liquid formed was used without further purification, in order to investigate the electrodeposition process in conditions easy to be applied in industry. All electrodeposition experiments were performed at room temperature, using a conventional three-electrode cell which consisted of the ionic liquid-contained beaker, in which two Al sheets ($80 \times 10 \times 1$ mm) were immersed and connected as anode. In between the Al sheets and about 15 mm far from each, a low carbon steel sheet ($80 \times 10 \times 2$ mm) was immersed and connected as cathode. The steel substrate was immersed to allow coating of area of 30×20 mm². Al wire was connected as a reference electrode. Pt sheet, with area of 1 cm², was used as working electrode for CV measurements. Surface preparation of electrodes was illustrated in Ref. [10].

3. Results and discussion

A typical cyclic voltammogram (CV) recorded onto Pt electrode in $\text{AlCl}_3/[\text{EMIm}]\text{Cl}$ (60/40 mol%) ionic liquid protected from air by decane layer is shown in Fig. 2. The potential of Pt was scanned from the open circuit potential (OCP) towards the negative potential to -500 mV with a rate of 10 mV/s. Increasing cathodic current density corresponding to the Al deposition started at -200 mV. The reverse

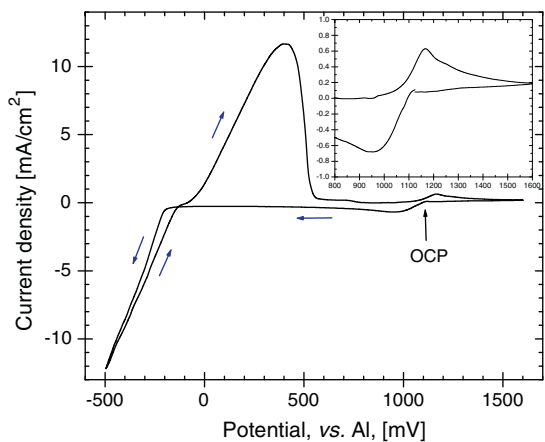


Fig. 2. The typical cyclic voltammogram for Pt sheet electrode in $\text{AlCl}_3/[\text{EMIm}]\text{Cl}$ (60/40 mol%) ionic liquid protected from air by a layer of decane at ambient atmosphere with scan rate of 10 mV/s. Inset: Higher resolution of the right part of CV.

scan monitored a current loop indicating that the rate of Al deposition is mostly nucleation controlled, which is characteristic of Al deposition in such ionic liquids [9,15,16,18]. After the nucleation loop, an oxidation peak due to Al stripping was recorded with a maximum anodic current density at 400 mV. The close observation of the right part of voltammogram (encompassed in a separated diagram in Fig. 2) revealed two additional peaks: cathodic one at 950 mV which can be ascribed to the underpotential deposition (UPD) of aluminium; and the other corresponding peak apparent on the reverse scan at 1160 mV that can also be attributed to stripping of UPD Al. In a comparison observation with CV of Pt in such ionic liquid systems reported in literature, it is clear that the CV of Pt electrode in the IL studied in air had the same features as that reported for ILs studied inside the glove box and reported elsewhere [9,15,16]. All that can be distinguished are small shifts in peak positions that can be ascribed to using the IL without purification [10], in contrary to previously purified ILs used in the literature.

Fig. 3 shows the CV of the aforementioned ionic liquid conducted outside the glove box onto a low carbon steel substrate. The potential of steel electrode was scanned from about 250 mV more positive than the OCP towards to negative potentials. By this anodic polarisation part (250 mV overpotential), iron ions dissolved into the ionic liquid and the steel surface was activated, thereby leading to excellent adhesion with Al deposit, as will be seen below. With continued scanning towards the negative potentials, a weak cathodic current peak (shown in inset of Fig. 3) was observed at $+80$ mV, which is presumably caused by reduction of iron ions. Differently to CV of Pt in Fig. 2, there was also a gradual decrease in cathodic current between -50 mV and -220 mV. This gradual current decrease is likely to be due to deposition of Fe–Al alloy. Thereafter, bulk Al deposition started depicting a sharp increase in current density. As in the case of Pt electrode, a significant current loop attributed to nucleation was depicted for bulk deposition of Al. After the nucleation loop, an oxidation peak due to Al stripping was recorded with maximum anodic current density at 320 mV. There was also a further side peak overlapped with the Al stripping peak, and ended at 580 mV. This side peak was presumably attributed to dissolution of Fe–Al alloy [10,16]. The following peak centred on 650 mV is most likely correlated to the dissolution of deposited Fe. As a conclusion, it can be stated that the CV of steel in $\text{AlCl}_3/[\text{EMIm}]\text{Cl}$ (60/40 mol%) IL conducted outside the glove box exhibited also the same general features recorded on steel in the same IL system executed inside the glove box, compare with Ref. [16].

In order to take the advantage of the rewarding impact of the in-situ pre-treatment through anodic polarisation, potentiostatic

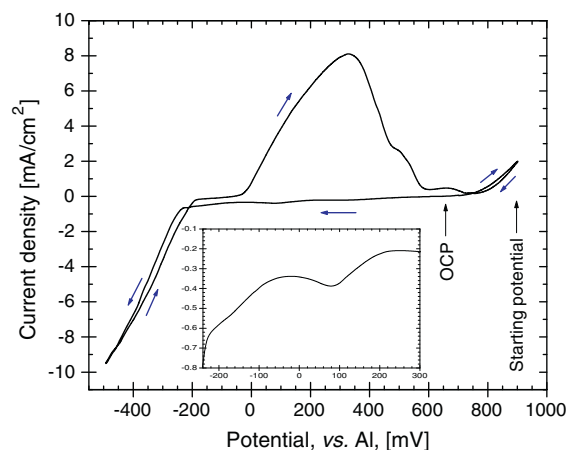


Fig. 3. The typical cyclic voltammogram recorded on low carbon steel substrate in $\text{AlCl}_3/[\text{EMIm}]\text{Cl}$ (60/40 mol%) ionic liquid protected from air by a layer of decane at ambient atmosphere with scan rate of 10 mV/s, started from anodic part. Inset: Higher resolution of the forward cathodic polarisation part preceding bulk Al deposition.

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