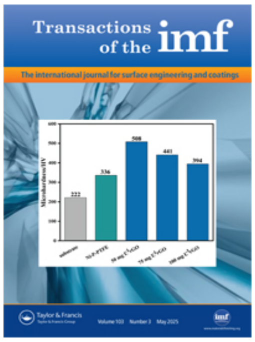

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BULLETIN

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Sustainable manufacturing in the finishing industries and the management of critical technology metals

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ABSTRACT

In the following article we review the discussion presented in a plenary session of the *Interfinish 2024* World Congress of the International Union for Surface Finishing (IUSF, Kowloon, Hong Kong). Here we describe some aspects of electrochemical materials finishing with a focus on sustainable manufacturing and on the dissolution and recovery of critical technology metals such as silver, gold, nickel, cobalt, silver and gallium. Such metals are often ubiquitous and essential for many modern technologies for example electronics (Ag, Au, Ni), energy conversion used in batteries or solar panels (Ag, Li, Co, Ni, Cu, Al) and even common light sources (Ga, In). However, they are usually present in low concentrations comprising a tiny proportion of an assembled device. We will cover some challenges and novel approaches in reclaiming these technology critical metals as well as exploring the technological synergies shared by the metal finishing and sustainable energy industries.

KEYWORDS
Deep eutectic; metal finishing; electronics; solar panels; batteries

Introduction

Many of the modern core technologies upon which we rely such as computing, phones, lighting, energy storage and conversion, and many more are now built on the supply and availability of a relatively small number of raw materials, without which they would not function. These are known as critical raw materials and include elements such as Si, Ge, Li, Ag, Au and Ga, to name just a few. Comprehensive lists of such materials are regularly published and maintained by governments and political organisations across the world, and are key metrics that guide and inform global social and economic strategies (see Note 1).

In a related trend, many associated manufacturing processes now rely heavily on surface finishing techniques to underpin these core technologies. The scope of such processes includes, but also goes far beyond, the traditional components of the surface finishing industry such as electroplating, polishing and organic surface treatment (paints, anti-corrosion, decorative and so on). It also includes technologies such as electronics manufacture/assembly, and a raft of thin-layer technologies including battery manufacture, low-power lighting (LED) and photovoltaic light harvesting devices (PV panels). Furthermore, commercial and political drivers are forcing the manufacturing agenda towards sustainability, zero waste and circular economic models and are demanding efficient management, recycling and reuse of resources. Against this background, there has been a spectacular growth in the global appetite for greener technologies such as electric vehicles (driven by lithium batteries), low power lighting (LEDs) and alternative energy sources including wind and photovoltaics. The combined shift in the patterns of use of core technologies, together with the pressures of sustainable manufacturing, have placed a heavy demand and stress on the markets for critical raw materials and, in particular, technology critical metals (TCMs).

The Centre for Sustainable Materials Processing (CSMP) is based in the School of Chemistry at the University of Leicester and comprises a group of academics and researchers at the interface between academia and industry. Over a period of ten years, the group has worked closely with many partners in the UK, Europe and across the world addressing technological challenges in surface finishing and in the development of greener, more sustainable manufacturing processing of materials. Most notably solvent technologies (deep eutectic solvents, DES) have been developed by the group which have attracted global attention and achieved high publication citation rates during this period.[1] These are non-aqueous liquids with highly tuneable properties, but which are formulated from widely available low-cost bulk commodity chemicals. Many of the DES liquids offer a greener and cleaner alternative to established aqueous chemical cocktails. Some of these general activities have been recently described in a review of the CSMP published in this journal.[2]

The challenges described above are addressed in this article which also focuses on a brief description of several projects within the CSMP as case studies to illustrate the group's strategies to tackle the sustainability demands on materials finishing manufacturing. Four separate projects in diverse parts of the surface finishing industry are described, and details are given of the group's approaches to not only improve resource usage and

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recover spent materials, but also to guide and inform the future design of technologies and materials to facilitate and enable end-of-life (EoL) recycling. This latter strategy is widely known as design-for-recycle.

Project CRUPPAIL: cadmium replacement anti-corrosion coatings

The project CRUPPAIL (Cadmium Replacement Using Pulse-plating And Ionic Liquids) was designed to address the development of a non-aqueous electrolytic Zn-Ni coating as a practical and sustainable alternative to cadmium. Cadmium coatings are highly desirable in many applications resulting from the unique combination of lubricity and corrosion protection. However, because of the toxicity of Cd^{2+} solutions its use is proscribed for all but military purposes. Whilst Zn-Ni coatings have often been used as an alternative, the aqueous process suffers from other health and handling concerns and can be difficult to control and maintain. Leicester-based DES electrolytes were explored as a non-aqueous, more sustainable alternative and, additionally, pulse-plating methodologies were used to successfully exert control over coating morphology and composition.

In the CRUPPAIL project, the electroplating of Zn-Ni alloy on mild steel components from a modified deep eutectic solvent (DES), *Ethaline* (see Note 2), through a combination of barrel plating and pulse plating processes was demonstrated. The *Ethaline* electrolyte, a mixture of choline chloride (ChCl) and ethylene glycol (EG), was modified with propylene carbonate (PC) as a co-solvent, together with the addition of boric acid to control deposit morphology and to suit the barrel plating system. A coating of γ-phase Zn-Ni alloy with 12–16% Ni was formed uniformly on mild steel screws and small components, as shown in Figure 1(a). Qualitative and quantitative corrosion tests showed that the coatings performed well as a sacrificial anti-corrosion layer (Figure 1(b)). Coatings were successfully achieved both at small scale (1 L) in the laboratory and at medium pre-production scale (150 L) in a commercial electroplating workshop. The coatings were shown to be in the range 10–20 µm thick after 120 min plating, with a Zn-Ni composition in the range 12–16% Ni, a typical γ- phase Zn-Ni alloy. The Zn-Ni coating was shown to cover the steel screws and other components uniformly and general appearance, morphology and adhesion were excellent. These findings were reported as part of an Innovate UK-funded collaboration between the University of Leicester and industry.[3]

From both the laboratory bench-scale experiments and the pilot demonstrator trials (at E.C. Williams Ltd. – see Figure 1(c)), it was concluded that the barrel plating of components using these materials and methods is very effective on a medium-scale and that the specification and performance of the coating is comparable with that from aqueous media. This fulfils many aspects of the intended project brief, however, further development of this

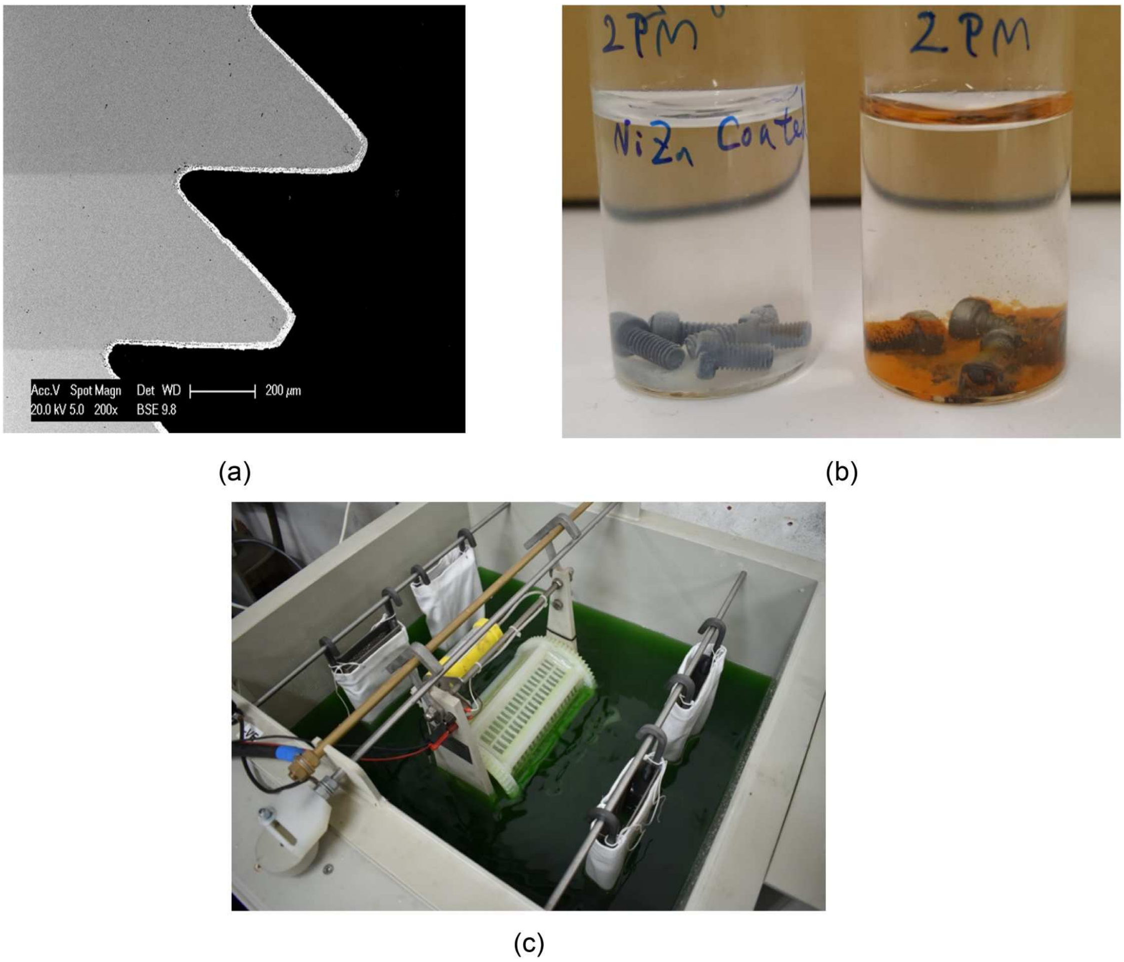

Figure 1. (a) Cross-sectioned SEM image of Zn-Ni coated steel screw showing coating distributed across the threaded features of the screw; (*b*) Corrosion tests of Zi-Ni coated screws (part (a)) coated (*left*) and uncoated (*right*) screws immersed in aqueous 0.2M $MgCl_2$ solution for two weeks; (*c*) Scale-up trials showing the operation of the pilot plating tank (150 L) at *E.C. Williams Ltd.* with barrel drive assembly and associated soluble Zn anodes. Reproduced with permission[3].

process may be limited by low current efficiency and the high economic cost (relative to water) of DES media.

Project APOLLO: sustainable recycling of PV panels

Photovoltaic panels are one form of sustainable energy source whose implementation is rapidly increasing as an alternative to fossil fuels. The panel modules contain both structural elements such as an aluminium frame and laminated glass support, polymers *etc.*, but also the functional PV panel wafer.[4,5] The latter is essentially a crystalline silicon wafer upon which various metallic coatings are patterned. Metals commonly used include Al, Cu and Ag variously implemented as electrical contact materials (where the specific work function of the metal is relevant to its function). The manufacturing processes include masking, photolithography and metallisation and are shared with many aspects of electronics falling easily within the remit of metal/materials finishing. The lifetime of PV panels in use is estimated to be around 20 years and so there is both an existing legacy of spent materials, together with a growing stock of materials to consider as they approach end of life. Considering the wafer component only, Figure 2(a), the overall mass is dominated by silicon but contains up to 1% wt. silver. Since both Ag and Si are critical raw materials (CRM) and technology-critical materials (TCM), these have to be recovered, separated and reused. Other TCMs are also present including Al and Cu.

Project APOLLO (*A Proactive Approach to the Recovery and Recycling of Photovoltaic Modules*) is a European consortium comprising a large group of universities, technical institutions, manufacturers, policy makers and end-users focused on analysing the materials present in waste PV panels and on developing methods for the efficient extraction, recovery, labelling, redistribution and remanufacture of the components (see Note 3). Current recycling practices for photovoltaic (PV) waste modules are unrefined and not very efficient. To be economical and sustainable the

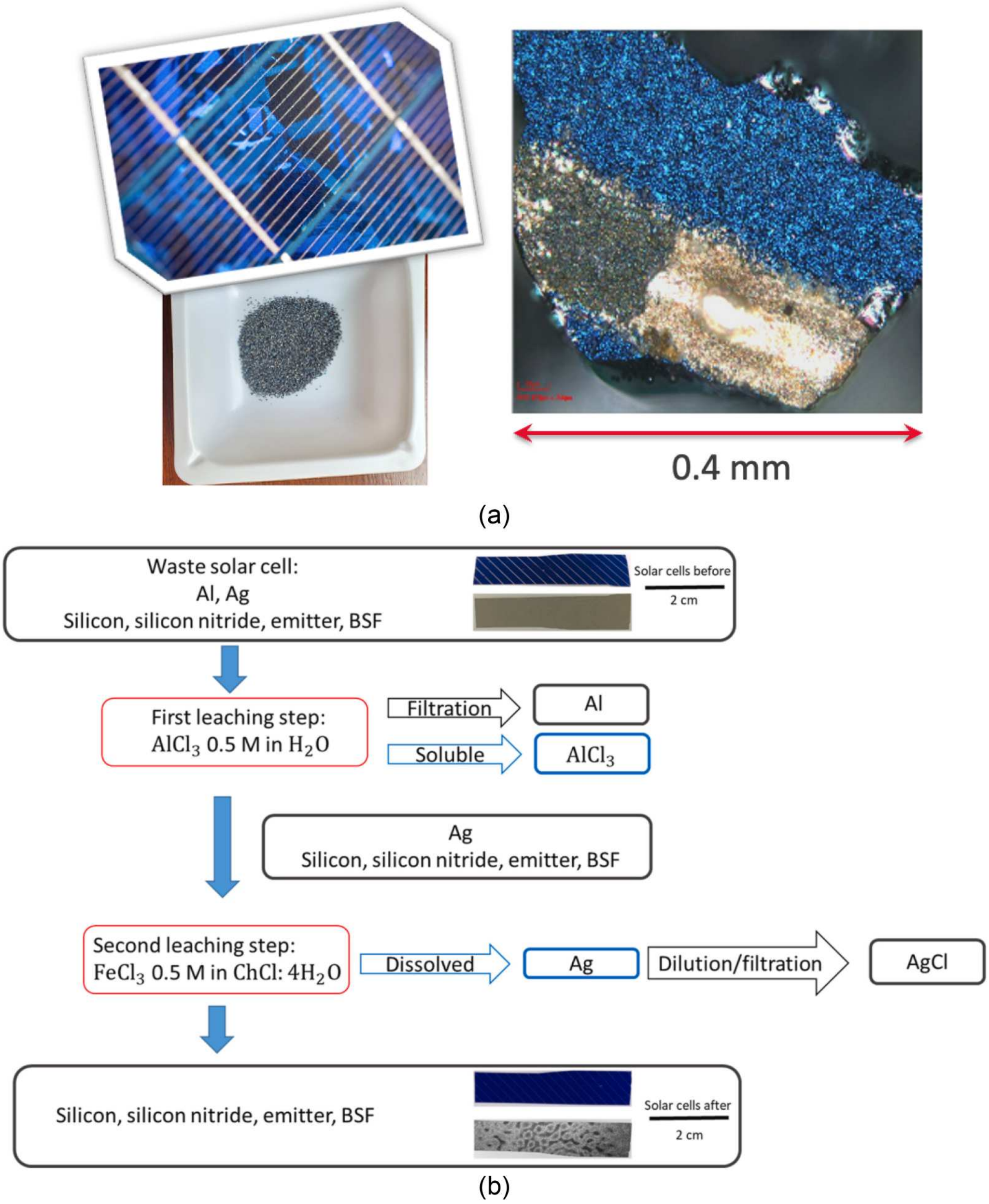

Figure 2. (a) Shredded PV panels containing Si, Ag, Cu and Al. A fragment is shown where the Ag contact track is clearly visible on the surface; (b) the recycling flowchart for selective extraction of metals. Reproduced with permission[6].

recycling of PV waste needs to recover all of the material constituents at a quality suitable for the reuse in new PVs, particularly the TCM content. APOLLO is aimed at creating a circular approach to facilitate legacy recycling, but also to inform future production, and hence future recycling. One aim of the project is to construct a pilot scale recycling line where incoming modules are disassembled, segregated and reprocessed allowing the recovery of high-quality Si, glass and TCMs such as Ag as a feedstock for remanufacture. As part of this, the present authors have developed solvent technology for the extraction and separation of Al, Cu and Ag from PV wafers.[6] This combines common oxidising agents such as $FeCl_3$ with DES electrolytes to extract the metals into solution, Figure 2(b). Once in solution, the mixtures of Cu and Ag can be selectively and sequentially obtained either by precipitation, or by electrochemical reduction i.e. electroplating, or else by galvanic displacement reactions – also known as electroless deposition. These two techniques, of course, draw on the core manufacturing legacy and expertise of the traditional materials finishing industries. Aluminium can subsequently be isolated by chemical precipitation. The ongoing aim is to optimise the efficiency of proof-of-concept dissolution protocols, for example with the use of ultrasound to overcome mass transport limitations, to maximise space–time yield for the extraction and recovery of these valuable resources.

Project ReGalL: sustainable recycling of gallium from spent LEDs

Project ReGalL (Recovery of Gallium from Ionic Liquids) was implemented to develop a recovery process of Gallium from bulk-sourced end-of-life (EoL) LEDs to supply the uptake of Gallium Nitride (GaN) semiconductors in the remanufacture of LED light sources. The aim in developing this process has begun to lay a foundation for upscaling the recovery of GaN in a range of EoL devices.

GaN is mostly used in LEDs but is increasingly being adopted in power electronics due to its superior qualities as a semiconductor compared to silicon counterparts. GaN has a wider band gap, switches faster, loses less heat and takes up less space. These qualities make it very desirable as a transistor for all sectors of interest (aerospace, automotive, energy and industrial drives), making it a desirable material to urban mine and build a supply chain for. Gallium's superior qualities contribute to its economic value, but they also make the material difficult to recycle. There exist recycling processes for gallium metal with optimal leaching efficiency of 99%, but they are currently only recovered from fine dust that is produced during manufacturing (as by-product), not from end-of-life products.

The innovation focus here was to create a circular supply chain of Gallium building on established recycling methods and expanding it to encompass bulk sourced EoL LEDs. The recovered Gallium could then be used to create a sustainable supply chain, avoiding virgin mining, increasing the supply chain resilience, and laying the foundation for establishing the EoL recycling process.

Currently, supply chains are very linear, with few OEMS having implemented EoL and circular strategies for Ga containing LED

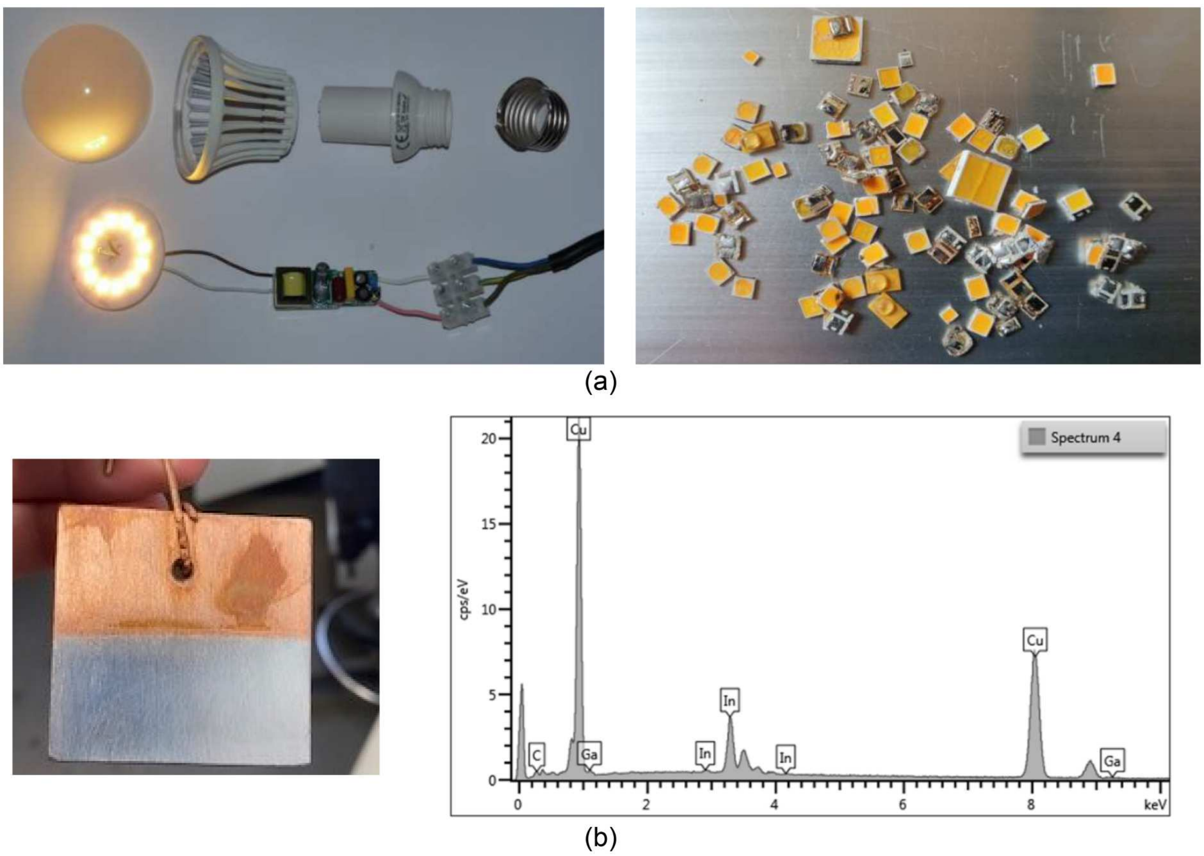

Figure 3. Gallium recovery from spent LEDs in the ReGalL project: (a) a disassembled LED light bulb showing the structural and electronic components (*left*) as well as the active LED elements (*right*) that contain the Ga and other TCMs; (b) a Ga coating on a copper panel (*left*) electroplated from Ga-containing DES electrolyte together with EDX analysis (*right*).

components. Additionally, implementing changes to a product or process is difficult once they are in place. It is therefore important to be aware of this process at its inception to encourage the circular adoption. Keeping with current, linear models wastes a magnitude of valuable materials and exposes OEMs to environmental, ethical and supply risks. As gallium is also of high economic value, OEMs have an opportunity to leverage the remaining GaN in their components.

The consumer LED light bulb has many different formats (e.g. GU10) and contains structural conformal materials including glass, plastic, contact metals *etc*. as well as drive electronics (see Figure 3(a)). The Ga containing active LED elements represent only a very small proportion of total mass (and volume). Once again, this complicates the recovery of TCMs as the supporting structures have to be physically isolated and separated from the LED elements. This is more challenging for some formats than others but the ReGalL project dealt with consumable GU10 and also some commercial lighting (e.g. from airport runway systems) sourced by project partners. However, once the LED active elements were recovered, the focus of the project was to design a suitable liquid electrolyte to both etch out the available Ga and then facilitate the recovery by electrowinning. Here the LED elements were first milled to a fine powder and then a DES electrolyte was used together with an oxidising agent. Here the oxidising agent can be molecular iodine, which is soluble in *Ethaline*, but not in water. Additionally, no strong acid was used and the iodide formed can be regenerated electrolytically during electrowinning.[7] Ga was extracted into the DES most probably as $GaCl_3$ although the project timescale did not allow for detailed quantification and optimisation of the process. Electroplating of Ga metal on a copper cathode was subsequently demonstrated from a solution of $GaCl_3$ in *Ethaline* (Figure 3(b)). In this sense a demonstration of concept was reached showing a possible route to the recovery of Ga. This was the original objective of the project. The concept can be further extended to include indium as another TCM which is also often present in these devices and other consumer electronics.

Project ReLiB: recycling of Li-ion batteries

Battery manufacture is another technology area that inherits much of its core manufacturing principles and operations from the surface finishing industries. The battery anode and cathodes often comprise a metal foil support (e.g. aluminium or copper) with thin-layer (50–100 µm) coatings containing the active materials. The fabrication and assembly of the active battery electrodes combines electrochemical processes of the charge storage elements with organic additives and binder chemistries inherited from the paints industry. The operation of a rechargeable battery often relies on sophisticated electroplating and stripping processes shared with the metal plating and polishing sectors together with electrolyte chemistries used to control the deposit morphology and prevent the formation of metal dendrites (see Note 4).

In recent years the consumer electronics market has become dominated by the presence of lithium-ion batteries for mobile charge storage, and this has been recently augmented by the acceleration in production of electric vehicles.[8] The latter has been driven by the political will of many developed nations to invest in and promote green technologies (judged at point-of-use). This market is now entirely reliant on Li-ion technology. Lithium-ion batteries have many variants with different chemistries (particularly electrolyte and cathode materials) but commonly contain many essential technology-critical metals including lithium, nickel, manganese, cobalt, copper, aluminium and others. In order to maintain both the green credentials of these power storage devices as well as the supply of critical raw materials for a rapidly growing market, the CRMs must be recovered from end-of-use batteries and reused in downstream manufacture. Currently there is a large legacy stock of spent Li-ion batteries for which there is an inadequate capacity to recycle and reuse. Additionally, current recycling methods are often wasteful; whole battery units are often shredded necessitating downstream physical separation and chemical segregation of material types. Pyrometallurgy methods are also commonly used which are both inefficient and can generate toxic waste (including HF from the combustion of fluorinated polymer binders and electrolytes).[8,9]

The ReLiB project (Reuse and Recycling of Li-ion Batteries) comprises a consortium of UK universities, stake holders and end-users brought together to examine the supply chain for Li-ion batteries, characterise the sources of legacy (end-of-life) waste and design new and efficient processes to recover and reuse the critical raw materials. An important part of this has also been to understand the construction and manufacturing materials and processes, and to design future-proof technologies that can be recycled and reused more easily and effectively *i.e. design for recycle*. ReLiB is funded by the Faraday Institution of the UK. One of the roles of the present authors in the ReLiB project has been to examine the binder chemistries used in electrode manufacture. The binder is a polymer adhesive used to generate coherent, functional surface coatings from active electrode materials such as graphitic carbon, or metal oxide powders. In current (and legacy) manufacture fluorinated polymers such as PVDF are used. These act as a very effective adhesive for the CRM-containing active materials but are very difficult to remove. This makes recovery and separation of the CRMs from end-of-life cells using anything other than organic solvents very challenging. There are alternatives to PVDF, including cellulose-based materials, but these are often highly cross-linked and difficult to separate. Here the group has developed alternative binder materials using bio-based polymers such as gelatine and alginate; the latter is derived from seaweed. The use of alginate, in particular, when combined with DES electrolytes such as *Gyceline* (choline chloride and glycerol), see Figure 4(a), gives a binder which is, under limited testing, functionally comparable to PVDF.[10] The advantage of this alginate-DES composite is that it is easily and quickly removed from the electrode substrate in aqueous media using low-power ultrasound. This is illustrated in Figure 4(b). The images here show an anode sheet prepared on a Cu foil using alginate-DES as a binder for the graphitic carbon active. When the anode assembly is immersed in water with agitation from low-power ultrasound, all the graphite is easily delaminated from the surface and can be reused.[11] The half-cell capacity test

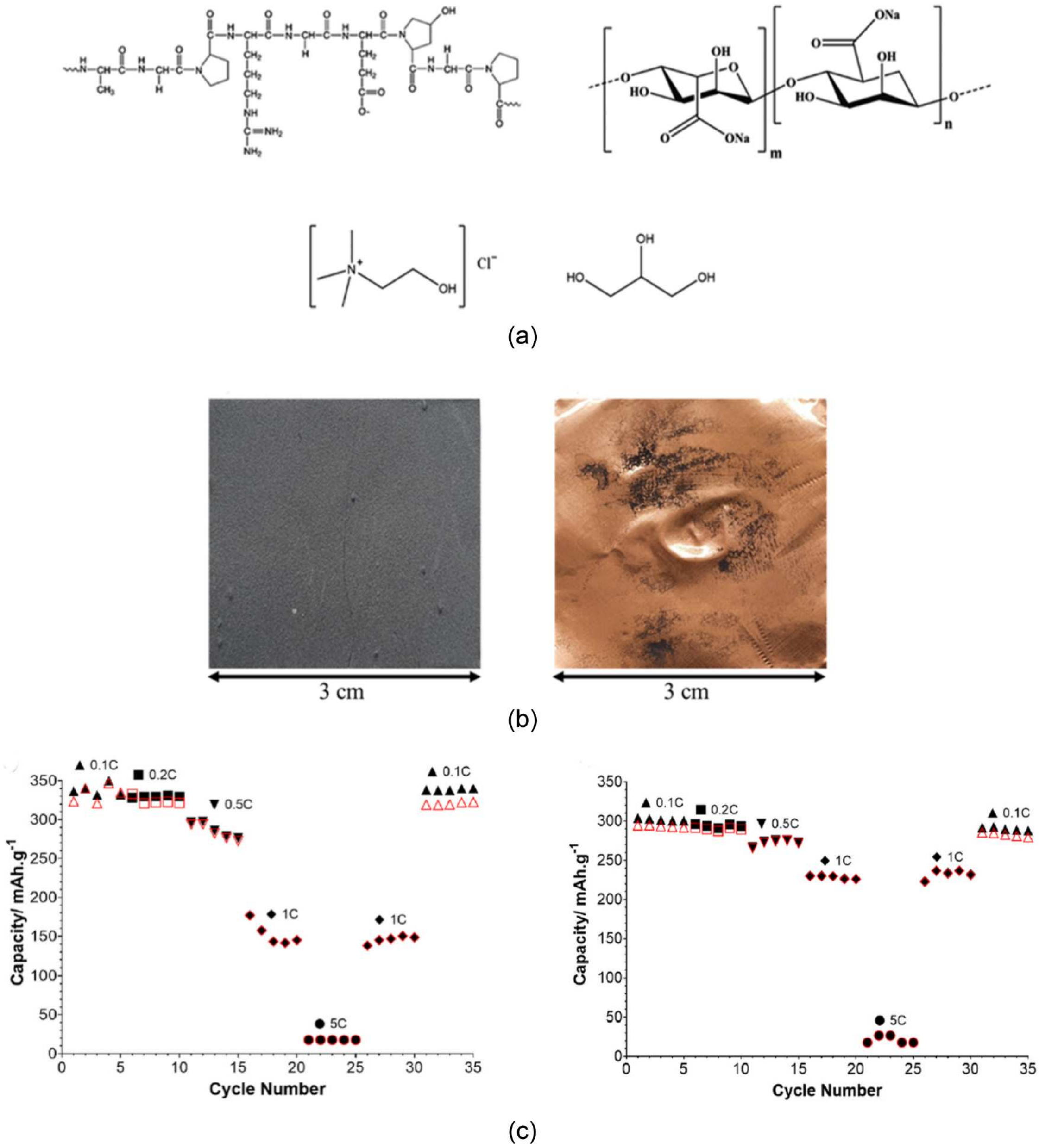

Figure 4. (a) Molecular structures of gelatine, sodium alginate, and DES components choline chloride and glycerol (*left to right, top to bottom*), reproduced with permission[11]; (b) Images showing graphite-coated anodes before (*left*) and after (*right*) processing with ultrasonication at room temperature, reproduced with permission[11]. The binder used was a DES alginate composite; (c) Waterfall plots showing how the c-rate applied to the half-cells affects the cell capacity (mA h g^{-1}). Black symbols show graphite charging, red outlines show graphite discharging. (*left*) PVDF binder, (*right*) NaAlginate binder, reproduced with permission[10].

data presented in Figure 4(c) are for an anode material constructed using conventional PVDF binder in comparison to the alginate-DES binder. The data were recorded over various charge/discharge rates (c-rate – see Note 5) and show a very similar pattern of functional behaviour.[10] In this case the use of the alginate bio-polymer has produced a functional alternative to other binders that can be integrated into current manufacturing processes, but which facilitates easy recovery of electrode components and CRMs at end-of-life.

Conclusions

This article has considered four examples of important modern technologies: electroplating for corrosion protection, photovoltaic panels, low-power LED lighting, and batteries. All of these technologies and the manufacturing processes that sustain them are underpinned in different ways by the expertise and background of surface finishing industries. In these and many other industries, the commercial, social and political drivers are forcing the sustainability agenda, demanding efficient management, recycling and reuse of resources. In the four examples discussed here, the authors have described how these demands can be addressed and conclude that sustainability challenges can be tackled in principle by the use of alternative process solutions using modern DES electrolytes. The tuneability of these systems enables optimisation of a process such as alloy plating. Additionally, the technologies which have embedded thin-layer processing and fabrication can be enhanced to improve recovery, recycling and reuse rates using the methods described here. These include, but are not limited to, gallium extraction from waste LEDs, TCM (Ag, Cu, Al) from PV panels, and a range of TCMs and other materials from Li-ion batteries. In all these aspects, surface finishing know-how is critical in the approach to sustainability and the implementation of a design for recycle philosophy. Finally, it has been shown that as well as considering existing feedstocks of legacy material containing CRMs and TCMs for recycling, thoughtful design and use of sustainable materials and approaches can lead to advances that are readily integrated into existing processes. This enables more efficient direct recycling methods and subsequent manufacturing.

Notes

1. Critical raw materials lists first published by the European Union in 2011, https://single-market-economy.ec.europa.eu/sectors/raw-materials/areas-specific-interest/critical-raw-materials_en, and updated regularly since; *Communication From The Commission To The European Parliament, The Council, The European Economic And Social Committee And The Committee Of The Regions*; Critical Raw Materials Resilience: Charting a Path towards greater Security and Sustainability, Brussels, 3.9.2020 COM(2020) 474.
2. *Ethaline* is the trivial name given to a stoichiometric mix of choline chloride and ethylene glycol, often in a 1:2 mol ratio.
3. *A Proactive Approach to the Recovery and Recycling of Photovoltaic Modules*; https://www.apolloproject.eu/.
4. Metal dendrite formation in battery cells during charging can lead to short circuit pathways. This represents an important failure mechanism and also a safety concern.
5. A c-rate of unity is defined as the current density required for complete charge / discharge of a cell in 1 h. A c-rate of 2, is discharged in half the time, hence twice the current density.

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