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Opinion

Upcycling and urban mining for nanomaterial synthesis

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ABSTRACT

Responsible processing of complex waste streams, especially through resource recovery and upgrading, is essential to lessen the environmental and societal impacts of materials production and disposal. Upcycling waste into materials that have higher value than their original form, and urban mining, the recovery of metals from electronic waste, are two recent strategies in waste management. Sustainable cradle-to-grave manufacturing means recovering resources that would otherwise contaminate the planet. Here, we discuss upcycling of plastic, rubber, and related carbon waste streams into graphene, as well as the recovery of precious metals and rare earth elements, while removing toxic heavy metals, all by using flash Joule heating. This article provides a brief overview of the current state of upcycling and urban mining as it applies to materials conversion, recovery, or nanomaterial synthesis, with a concluding perspective highlighting the considerations, opportunities, and challenges that accompany upcycling and urban mining research.

Introduction

As populations and living standards increase globally, and production costs of consumer goods broadly decrease, solid waste generated from plastics, electronics, batteries, solar cells, perfluoroalkyl substances, fly ash, rubber, automobiles and appliances produce large amounts of hazardous byproducts upon landfill [1]. These waste streams are resource rich, offering high concentrations of valuable metals or commodity chemicals [2]. In the United States, 140 million tons of solid waste were landfilled in 2018, and an estimated 67 million tons of plastics were recycled [3].

Recycling is challenged by the complexity and contamination of waste streams, requiring sorting, dismantling, washing, and other time-, labor-, and resource-intensive pre-processing steps. Waste is composed of dozens of different materials that are difficult to separate and recover in a cost-effective manner [4]. Upcycling, however, converts waste streams into products of higher value than their starting forms [5]. Urban mining is a term first introduced in the 1980s in Japan to describe the recovery of precious metals from municipal waste streams such as automobiles and appliances, electronics waste (e-waste), and batteries [6]. 'Urban mining' most often refers to e-waste, especially landfilled circuit boards or batteries, and the concept has steadily gained popularity. Often these waste streams have high concentrations of critical resources, however, device fabrication complicates the liberation of the metals so that they can not be efficiently separated. Recent research in nanomaterials and nanotechnology has focused on the use of upcycling and urban mining to lower the cost and environmental burden of high-value products, which we highlight in this prespective.

Flash Joule heating

Flash Joule heating (FJH) for the synthesis of graphene was described by Luong et al. in 2020, in which large amounts of current are discharged from a bank of capacitors, passing through a low-resistance carbon feedstock, reaching temperatures in excess of 3000 K in milliseconds while sustaining the high temperature for only seconds or less (Fig. 1**a**) [7,8]. This high temperature converts amorphous carbon into high quality graphene by providing sufficient energy to rearrange the carbon bonds to a thermodynamically favored sp^2-hybridization state. FJH is a scalable and efficient method of turbostratic graphene production without solvent, catalyst, or inert gas use [9]. Electricity required to transform 1 ton of

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amorphous carbon into graphene using FJH is calculated to cost $30 to $161 depending on the starting source [7]. A variety of other nanomaterials can also be produced by FJH, including boron nitride [10], carbon nanotubes [11], corundum nanoparticles [12], doped graphene [13], and more.

FJH for upcycling

Wastes such as food, mixed plastics, biochar, asphaltenes, and tires have been shown to produce graphene when subjected to FJH [7,14,15]. The FJH reaction can produce significant volatiles that

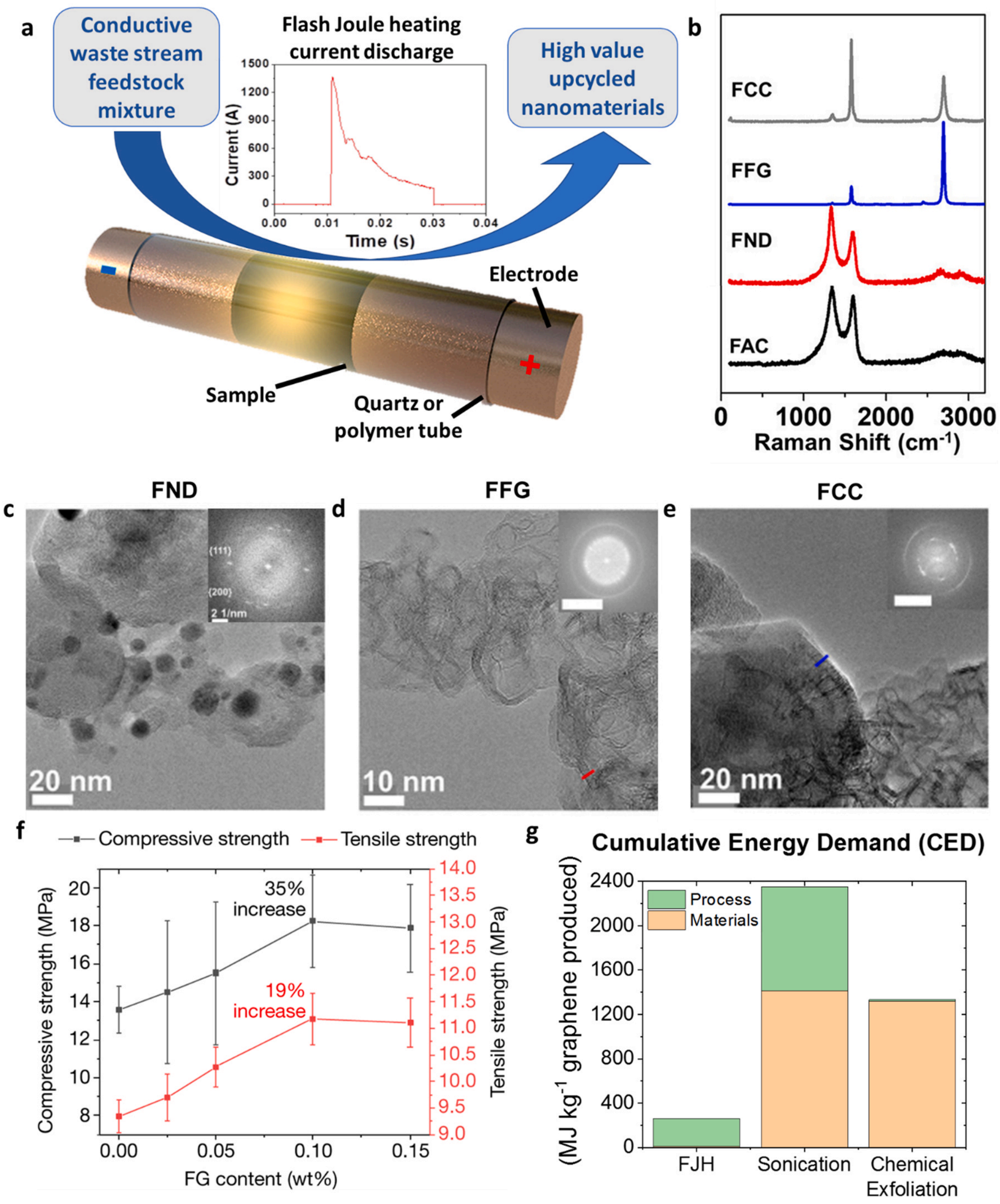

Fig. 1. Upcycling by flash Joule heating. a) A scheme showing a general strategy to upcycle conductive waste streams into high value turbostratic graphene or other nanomaterials using flash Joule heating. **b)** Raman spectra characterizing PFAS contaminated waste upcycled into nanomaterial products through flash Joule heating, where fluorinated amorphous carbon, fluorinated nanodiamond, fluorinated flash graphene, and fluorinated concentric carbon are abbreviated FAC, FND, FFG, and FCC, respectively. **c-e)** Transmission electron microscope images of PFAS contaminated waste upcycled into nanomaterials products through flash Joule heating. An inset fast Fourier transform (FFT) is inlaid in each image. **f)** Tensile and compressive strength testing of flash graphene (FG) added into cement at various loadings. **g)** Cradle-to-gate life cycle assessment showing the cumulative energy consumption of the upcycling of end-of-life vehicle waste plastic into graphene by FJH, compared to graphene produced by other methods. (**a**) Printed with permission [67]. Copyright 2021, American Chemical Society. (**b-e**) Printed with permission [19]. Copyright 2021, American Chemical Society. (**f**) Printed with permission [7]. Copyright 2020, Springer Nature. (**g**) Printed with permission [21]. Copyright 2022, Springer Nature.

outgas when lower carbon-content feedstocks are used. Plastic and tire pyrolysis ashes have been tested and shown to produce higher mass conversions of graphene since those materials have already been subjected to volatilization [16,17]. Machine learning can be used to map the FJH reaction parameter space through model based optimization, obtaining graphene qualities that are superior to human optimized methods [18]. Polyfluoroalkyl substances (PFAS) are hazardous materials that are usually removed from water through sorption onto activated carbon. FJH treatment upcycles PFAS and other fluorine compounds into fluorinated products including amorphous carbon, nanodiamonds, graphene, and concentric carbons, which are onion-like carbon spheres made of stacked sp^2-hybridized layers [19]. The phase, morphology, and fluorine content of the product is controllable by tuning the FJH reaction parameters such as duration, peak current, and precursor material (Fig. 1**b-e**).

When the FJH feedstock is not conductive, such as insulating plastics, amorphous carbons such as carbon black or metallurgical coke can be added to allow current discharge required for FJH. The amount of conductive additive can be decreased to as low as 5 wt% when a two-step FJH process is used to upcycle [20]. First a low-current discharge carbonizes the plastics, which undergoes a pyrolysis-like process where volatile monomers, oligomers, and syngas are emitted. This leaves an amorphous carbon char that is then conductive enough to undergo high current FJH. This two-step process can upcycle mixed and dirty waste plastics into high quality turbostratic graphene [20,21]. Introduction of alkali metal salts to act as blowing, etching and pore proppant agents allows for the conversion of mixed waste plastics into high surface area holey and wrinkled graphene [22]. Specific surface areas up to 874 m^2/g are possible, compared with surface areas of 50–240 m^2/g for non-activated flash graphene.

Applications of graphene products upcycled from waste materials through FJH include composites with cement, polyurethane foam, vinyl esters, and epoxies (Fig. 1**f**). Holey and wrinkled flash graphene has been studied as a competitive metal-free hydrogen evolution reaction electrocatalyst, and as a Li-metal battery anode that affords rapid cycling because of its high surface area and porosity [22,23]. Flash graphene, compared with commercial graphenes, is many times more dispersible in composites as a result of the turbostratic stacking of the layers and can be used as an effective lubricant [7,16,24]. FJH can cyclically upcycle graphene containing polymer-nanocomposites without impacting the quality of the product [21]. The total resource consumption and environmental impact of synthesizing 1 kg of graphene by FJH has been compared with commercial production methods using physical or chemical exfoliation of graphite (Fig. 1**g**). FJH was determined to use up to 88% less energy, 97% less water, and emit up to 85% less emissions than other graphene production methods [21].

FJH for recycling, upcycling and resource recovery is being scaled up industrially and is becoming more widespread in research. For example, Huang et al. used fluorine-contaminated hazardous carbon waste from aluminum production as a feedstock for FJH [25]. Dong et al. leveraged FJH to regenerate Li-ion battery anode graphite, removing the solid electrolyte interface, repair defects, and increase conductivity to levels higher than commercial graphite [26]. Fundamental studies of the FJH process through simulation of heat distribution, flux, and energy consumption during the reaction and upon scaling up has also been carried out [27]. Qiu et al. produced a synergistic Ag, Co, and C electrocatalyst for oxygen reduction reactions using FJH [28], while Wei et al. used CuRu alloy decorated CNTs synthesized by FJH for efficient water splitting [29]. Graphene-ferroferric oxide nanocomposites were prepared from lignin by FJH and used for electromagnetic wave adsorption by Chen et al [30]. Zhu et al. synthesized N-doped graphene by FJH for use in high-stability supercapacitors [31].

FJH for urban mining

Metal-containing solid waste streams can become sustainable resources because they often contain precious and rare earth metals [32]. The concentration of Au in e-waste is higher than that in mined Au ores [33]. The recovery of these valuable metals from solid wastes offers an alternative as critical resources become exhausted, and mitigates the negative environmental impact of traditional mining from ores and downstream solid waste disposal [34]. Urban mining faces many refining challenges similar to the mining of mixed virgin ores. Traditional urban mining methods use extractive metallurgy, mainly pyrometallurgical and hydrometallurgical processes to liberate metals from encased platforms like printed circuit boards [35,36]. Hydrometallurgical processes are more selective and leach metals from waste materials using specific strong acid mixtures known as lixiviants, but secondary aqueous waste streams of strong acid are generated [37].

The FJH process enables the ultrafast, water-free recovery of precious metals and removal of hazardous heavy metals from e-waste [38]. Metal recovery by FJH is composed of three steps: the FJH reaction for metal evaporation, gas transport by a vacuum system, and condensation of volatiles in a cool zone (Fig. 2**a**). Printed circuit board e-waste is finely ground and mixed with carbon black or metallurgical coke as the conductive additive (Fig. 2**b**). The sample temperature ramps to ~3400 K in milliseconds during FJH, affording evaporative separation of precious metals (Fig. 2**c**). The use of salts as additives converts metals into their halides, lowering their resulting vapor pressures, and producing recovery yields of > 80% for Rh, Pd, Ag, and > 60% for Au (Fig. 2**d**). The toxic heavy metals in electronic waste, including Cr, As, Cd, Hg, and Pb, are also removed (Fig. 2**e**), leaving a final waste with minimal metal content, acceptable even for agricultural soil use. Urban mining by FJH has an energy consumption of ~939 kWh ton^{-1}, which is 80 × to 500 × less energy consumptive than traditional smelting furnaces.

In addition to precious metals, rare earth elements (REEs) are critical materials in electronics and renewable energy technologies [39]. REEs recovery from secondary wastes, compared with mining virgin REEs, enables a circular economy [40]. The applicable wastes include coal combustion residual coal fly ash (CFA), bauxite residue (red mud) from aluminum production, and e-waste [41,42]. Present methods for REEs recovery suffer from lengthy purifications, low extractability, and high waste stream impacts. The FJH process acts as an activation strategy for improving REEs extractability from wastes [43]. Before FJH, the solid waste is mixed with carbon as the conductive additive. The REE leachability from FJH-activated CFA is improved by 187–206% in mild acid leaching conditions (1 M HCl, 85 ºC) and, most importantly, FJH enables a high REE recovery yield even by dilute acid leaching (e.g., 0.1 M HCl, Fig. 2**f**). For individual REEs, acid leachability is improved between 170% and 230% (Fig. 2**g**). FJH thermally converts hard-to-dissolve REE phosphates to REE oxides with high thermodynamic solubility (Fig. 2**h**). The rapid heating and cooling also cracks the glass that encases REEs in CFA formed during the coal burning process, rendering the REEs aqueous-acid-accessible. FJH can also recycle graphite anodes, recovering Ni, Co, Mn, and Li while producing value-added graphite anodes, which outperform current recycled graphite [44].

While FJH presents a highly efficient method for synthesis, upcycling, purification, and urban mining, some disadvantages and challenges remain to be solved. As discussed in Section 2.1, conductive additives may need to be mixed with non-conductive feedstocks such as plastics, which may increase the cost. Because of the rapid kinetics of the FJH reaction, the particle size of the feedstock can significantly impact the quality of produced nanomaterials or the recovery yield of metals. Therefore, some milling or grinding of the feedstock is an important step to optimize the process and

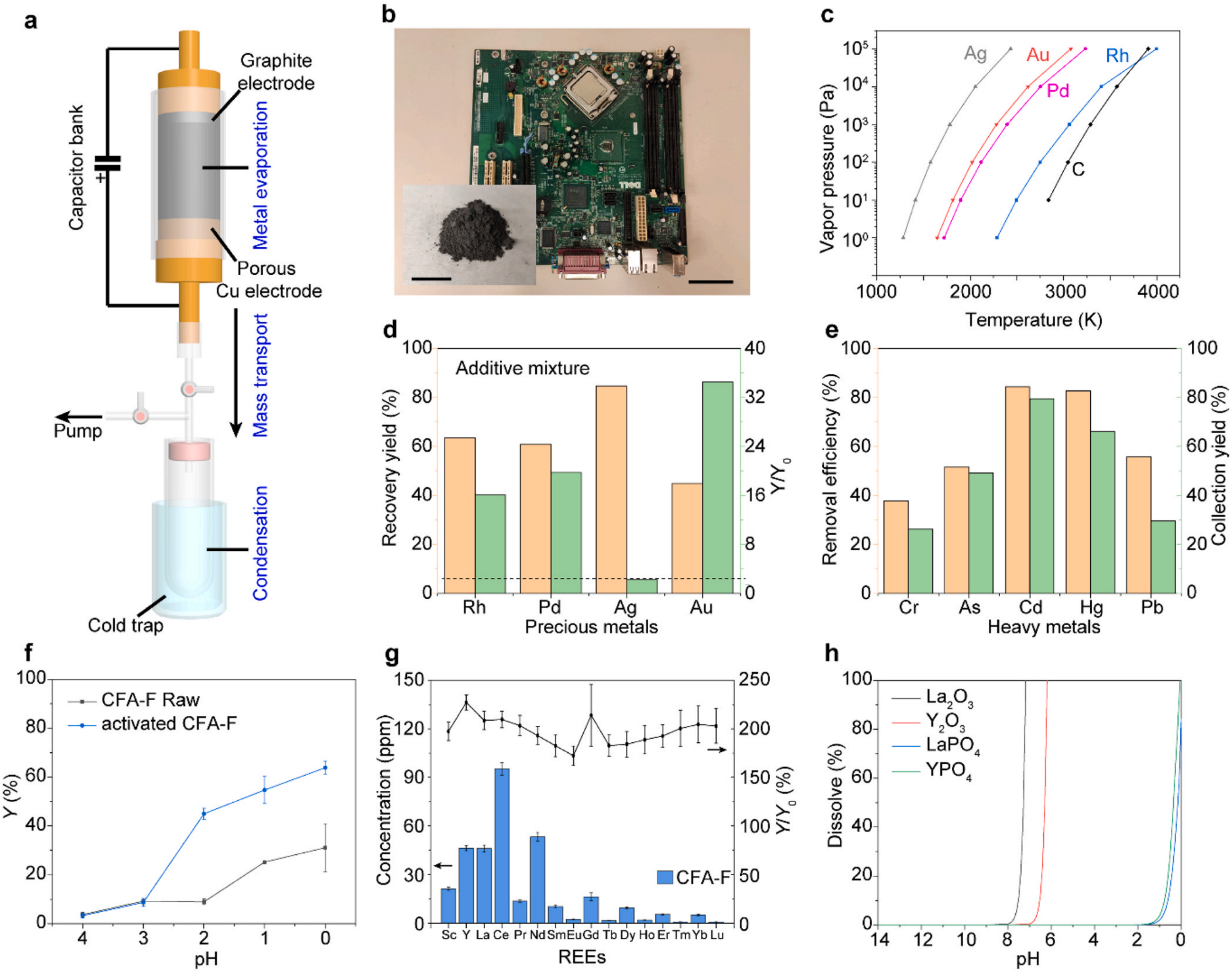

Fig. 2. Urban mining by flash Joule heating. (**a**) Schematic of the evaporative separation system composed of FJH apparatus for metal evaporation, vacuum system for mass transport, and cold trap for volatiles condensation. (**b**) Picture of a discarded PCB. Scale bar, 5 cm. Inset, the mixture of carbon black with PCB powder. Scale bar, 2 cm. (**c**) Vapor pressure-temperature relationship of precious metals and carbon. (**d**) Recovery yield of precious metals by using the mixture of NaF, NaCl, and NaI as additive. The recovery yield (Y) is the recovery yield of precious metal with halide additives, Y_0 is recovery yield without the halide additives, so Y/Y_0 indicates the improvement of yield with the halide additive. The dashed line represents $Y/Y_0 = 1$. (**e**) Removal efficiency and collection yield of heavy metals. The removal efficiency is calculated by: $1 - (\frac{\textit{Metals remaining in e}-\textit{waste}}{\textit{Total initial metals in e}-\textit{waste}})$. The collection yield is calculated by: $\frac{\textit{Metals recovered in cold trap}}{\textit{Total initial metals in e}-\textit{waste}}$. The collection yield is higher than the removal efficiency due to the metals condensing in areas other than the cold trap. For **d** and **e**, the yields are the average of three independent FJH reactions. (**f**) pH-dependent REEs leachability from the Class F CFA (CFA-F) raw materials and the activated CFA-F. (**g**) HCl-leachable individual REEs contents (1.0 M, 85 °C) from activated CFA-F and the increase in recovery yield. Y_0 represents the yield by 1.0 M HCl leaching the CFA raw materials, and Y represents the yield by HCl leaching the activated CFA. Error bars in **f** and **g** represent the standard deviation where N = 3. (**h**) Calculated dissolution curves of Y_2O_3, YPO_4, La_2O_3, and $LaPO_4$ with a mass **of** 1 g in 100-mL solution. Cl^- is used to balance the charge. (**a**-**e**) Printed with permission [38]. Copyright 2021, Springer Nature. (**f**-**h**) Printed with permission [43]. Copyright 2022, American Association for the Advancement of Science.

improve product homogeneity. Lastly, process scale-up to multi-kilograms per day has so far only been demonstrated for graphene synthesis from coal, not waste materials or urban mining processes, which may require further optimization.

Recent upcycling and urban mining trends

The upcycling of waste plastics, biomass, or biochar into graphitic carbon materials such as graphene, carbon nanotubes, bamboo-like carbon nanofibers, or graphite using thermal annealing, arc discharge, pyrolysis, microwave-assisted decomposition [45], and other techniques has become quite popular (Fig. 3**a**) [46–50]. Particular focus should be given to scalable methods that do not require the sorting of plastics or the use of solvents, catalysts, or inert gases, to reduce process burdens and external costs. E-waste, such as printed circuit boards, is being upcycled to produce Au, Ag, Cu, metal oxides, and other materials for a variety of demonstrated applications (Fig. 3**b**) [51–56]. Urban mining and upcycling of other metal-rich waste streams such as pickling liquor and furnace slag from steel making, CFA, and solar cell, cable, or battery waste, is also being explored [57–61]. Bauxite residue is also being investigated as a source for metal nanoparticles and nanowires on a laser-induced graphene support [62].

Traditional hydrometallurgical and pyrometallurgical processes are being improved by adding advanced photocatalysis or using new thermodynamic reactions. Chen et al. reported the selective recovery of precious metals through photocatalysis [63]. Acetonitrile, dichloromethane, and TiO_2 powder photocatalytically extract precious metals from e-wastes and ores with high selectivity and high purity > 98% (Fig. 4**a-b**). The process has been scaled up to kilograms, with the catalyst reused > 100 times, demonstrating potential industrial compatibility. Stinn et al. proposed the selective

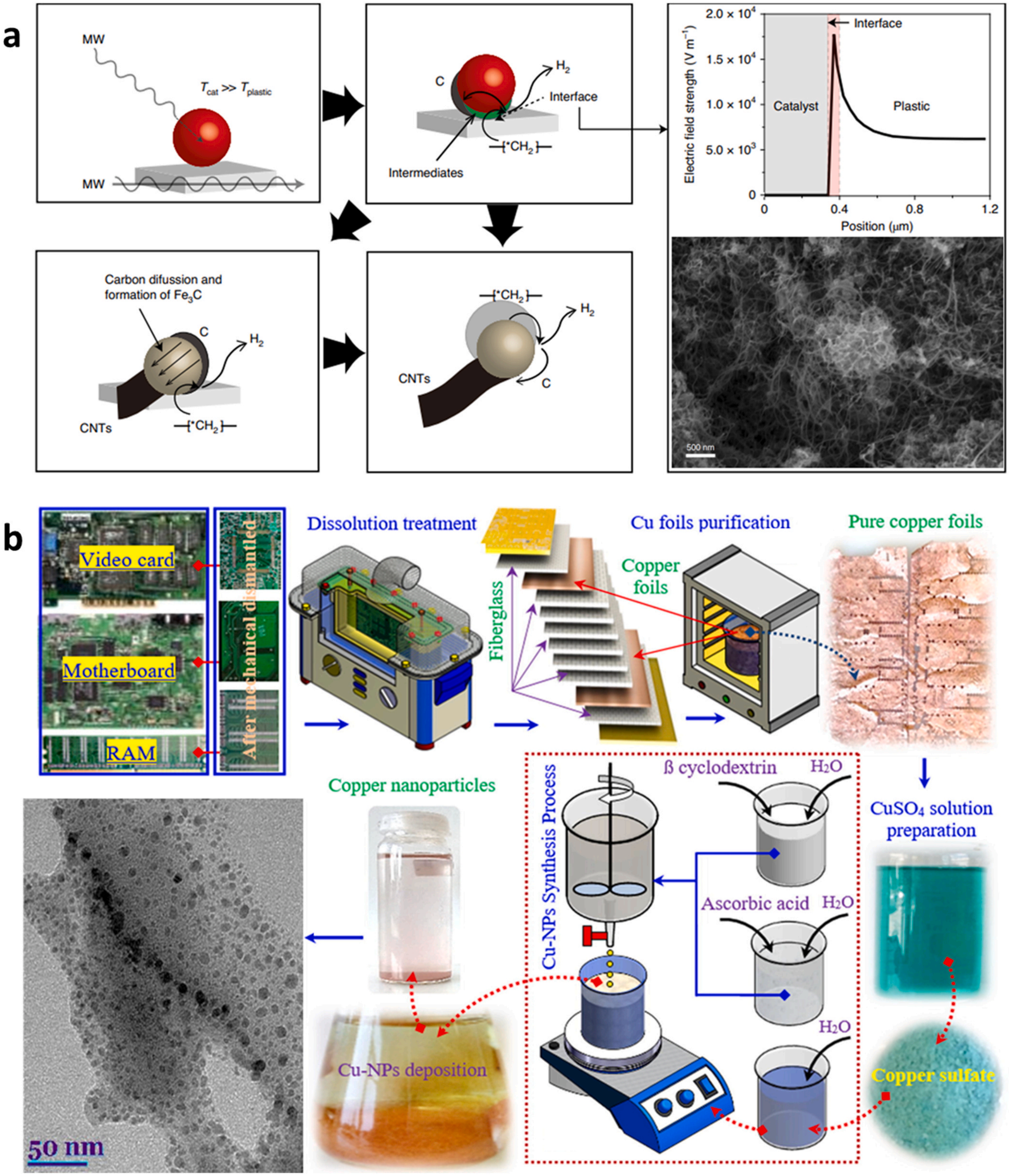

Fig. 3. Upcycling processes of waste plastics and electronic waste. **a**) Schematic of a proposed mechanism in which microwaves (MW) and FeAlO$_x$ upcycle high density polyethylene into hydrogen gas and multiwalled carbon nanotubes. A simulation of the temperature of the system during MW heating, and a scanning electron microscope (SEM) image of the produced multiwalled carbon nanotubes are shown on the right side. **b**) Schematic of a method to upcycle electronic waste into copper nanoparticles using ultrasonic separation of copper foil from the printed circuit boards, followed by use of environmentally available chemicals to synthesize the copper nanoparticles, at a 6 times lower cost than commercial methods. A TEM image of the produced copper nanoparticles is shown in the bottom left. (**a**-**e**) Printed with permission [47]. Copyright 2020, Springer Nature. (**f**-**h**) Printed with permission [52]. Copyright 2018, Elsevier.

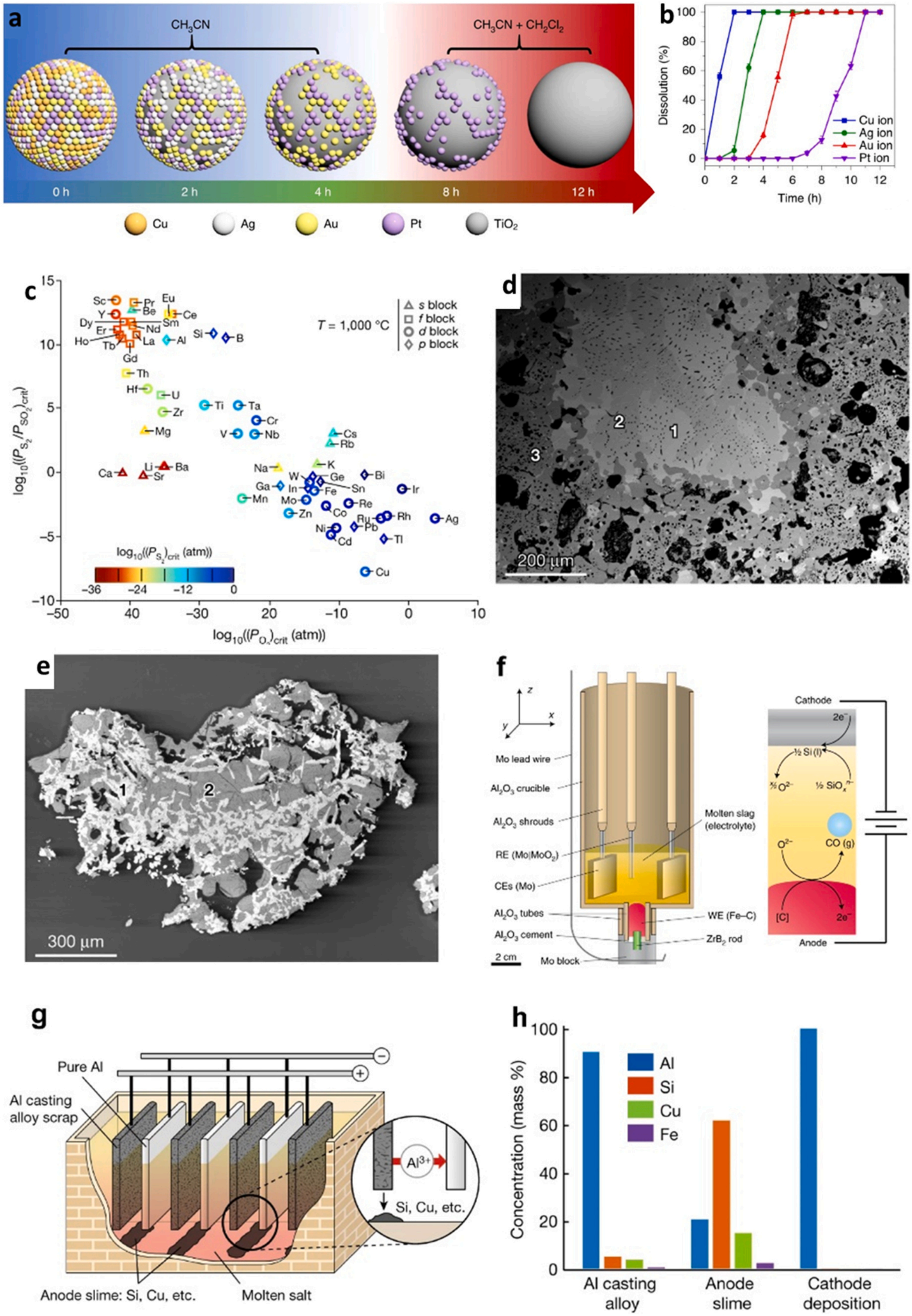

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Fig. 4. Advanced techniques for metals recycling and upcycling. (**a**) Schematic of the TiO_2 photocatalytic selective dissolution of metals under 365 nm ultraviolet illumination with CH_3CN and CH_2Cl_2 as the solvent. (**b**) The dissolution percentages of metals obtained by selective photocatalysing metal catalysts at different reaction times. (**c**) Oxide-sulfide anion exchange chemistry exacerbates the thermodynamic differences between metal compounds. (**d**) Selective sulfidation for lithium-ion battery recycling with image showing $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ sulfidized, which separates into Ni-rich sulfide (**1**), Co-rich sulfide (**2**), and Mn oxysulfide (**3**). (**e**) Selective sulfidation of rare earth magnet recycling with image showing (Nd,Pr,Dy)-Fe-B magnet sulfidized, which separates into Nd-rich regions (**1**) and Fe-rich sulfide phases (**2**). (**f**) Schematic illustration of the electrochemical cell and electrorefining process for direct decarburization of molten iron. WE, working electrode; CEs, counter electrodes; RE, reference electrodes. (**g**) Schematic of the solid-state electrolysis process for upcycling of aluminum scrap. (**h**) The composition analysis showing that the typical alloying elements were separated into the anode slime. (**a-b**) Printed with permission [63]. Copyright 2021, Springer Nature. (**c-e**) Printed with permission [64]. Copyright 2022, Springer Nature. (**f**) Printed with permission [65]. Copyright 2021, Springer Nature. (**g**) Printed with permission [66]. Copyright 2022, Springer Nature.

sulfidation of metal compounds to improve their separation factors [64]. By controlling parameters including gas partial pressure, flowrate, and carbon addition, the target metal from a mixed metal-oxide feed can be selectively sulfidized (Fig. 4**c**), enabling the improved separation of metals compared with liquid methods. This has been applied to recycling of Ni-Mn-Co lithium ion battery cathodes and (Nd,Pr,Dy)-Fe-B magnets (Fig. 4**d-e**) and the sulfidation process potentially leading to 60–90% reduction in greenhouse gas emissions.

Recycling of Fe and Al is critical to closing material supply loops and increasing sustainability, but impurities during remelting degrade the recycled product quality, resulting in downcycling. Judge et al. reported the electrorefining of molten Fe [65]. In the electrorefining process, the molten Fe is decarburized by applying an electromotive force between it and a slag electrolyte (Fig. 4**f**), evolving CO and producing ultra-low-carbon steels using little energy and no reagents. Lu et al. proposed solid-state electrolysis for upcycling Al scrap [66]. In the process, Al scrap acts as the anode, pure Al as the cathode, and a molten salt composed of a mixture of alkali chlorides and alkaline earth metal chlorides as the electrolyte (Fig. 4**g**). During electrolysis, Al is dissolved from the anode and the refined Al, with purity comparable to virgin Al, is deposited on the cathode, while Si, Cu, and Fe impurities are separated out (Fig. 4**h**). Moreover, the energy consumption of the recycling process is 50% less than primary Al production.

Summary and Outlook

Upcycling and urban mining by FJH and other techniques represent a powerful new paradigm in the synthesis of nanomaterials. Many challenges remain, but some important considerations can be applied by academic and industrial chemists, engineers, or materials scientists when leveraging upcycling or urban mining:

1. Process scalability, simplicity, reproducibility, and overall cost are directly tied to the likelihood of eventual implementation. Use of authentic waste streams rather than lab generated substitutes can illuminate unforeseen complications that could arise during implementation. For example, commercially obtained plastics pyrolysis ash can be contaminated with heavy metals, which impacts the process optimization and economic viability of its upcycling.
2. Life cycle assessment and technoeconomic analysis are powerful tools to calculate burdens, such as energy and water use or greenhouse gas emissions, associated with materials recycling or production process. Burdens for new or proposed processes should be compared with current methods to identify steps that require further optimization and inform scale-up considerations. Similarly, products of upcycling and urban mining should be compared with current methods and commercial products to benchmark quality. When possible, application of the upcycled product should be demonstrated to display value of the process and quality of the product.
3. Public policy and grant funding focused on simple and scalable upcycling and urban mining of realistic waste streams could further encourage advancement of the field. Collaboration and communication between researchers and waste generating, collection, and processing industries could speed reclamation of emerging waste streams and improve understanding of current methods, challenges, and opportunities. For example, recent collaboration between environmental scientists, funding agencies, and chemists has accelerated efforts to remediate and upcycle hazardous perfluorinated alkyl substances (PFAS) [25].
4. Planned design and assembly of electronics, batteries, automobiles, and other products with a focus on better end-of-life processibility for recycling, upcycling, and urban mining or the use of fewer materials, can lower the barrier of entry for resource recovering processes. For example, separation of Li-ion battery components is a bottleneck for recycling efforts complicated by different pouch or cylinder constructions. Battery components should be constructed to ease disassembly.

In conclusion, waste materials present an abundant and low-cost resource to upcycle into nanomaterials or to supply valuable metals. Recent use of FJH represents a simple and multipurpose method for upcycling and urban mining, but require further advancement and study to realize a future of responsible waste management and resource recovery.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Universal Matter Inc. has licensed FJH approaches to graphene from Rice University. J.M.T. is a stockholder in this company, but not an employee, officer, or director. Potential conflicts of interest are mitigated through regular disclosures to and compliance with Rice University's Office of Sponsored Programs and Research Compliance. The authors declare no other competing interests.

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