A Technical and Economic Evaluation of Etchant Choice for PCM of Copper and its Alloys

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Why am I presenting this talk today?

- The standard etchant of the PCM industry is ferric chloride.
- Disposal of spent ferric chloride etchant has become a costly process.
- I am continually being asked as to how disposal costs can be reduced.
- Is there an alternative etching strategy that might be less costly?
- Could alternative etchant cupric chloride be cheaper?
- Can waste byproducts become secondary products?
- Renewed interest **worldwide** for etchant regeneration*

* M Yu, X Zeng, Q Song, L Liu and J Li, Examining regeneration technologies for etching solutions: a critical analysis of the characteristics and potentials, Journal of Cleaner Production,113, 973-980, 2016.

How can we reduce the cost of PCM to remain competitive with rival processes?

1. Purchase less etchant – this implies that we should recycle waste etchant.
2. Dispose of less waste etchant to reduce disposal costs.
3. Extract dissolved metals from spent etchant to provide a secondary product with a retail value.
4. Purify spent etchant to provide a potential secondary product.

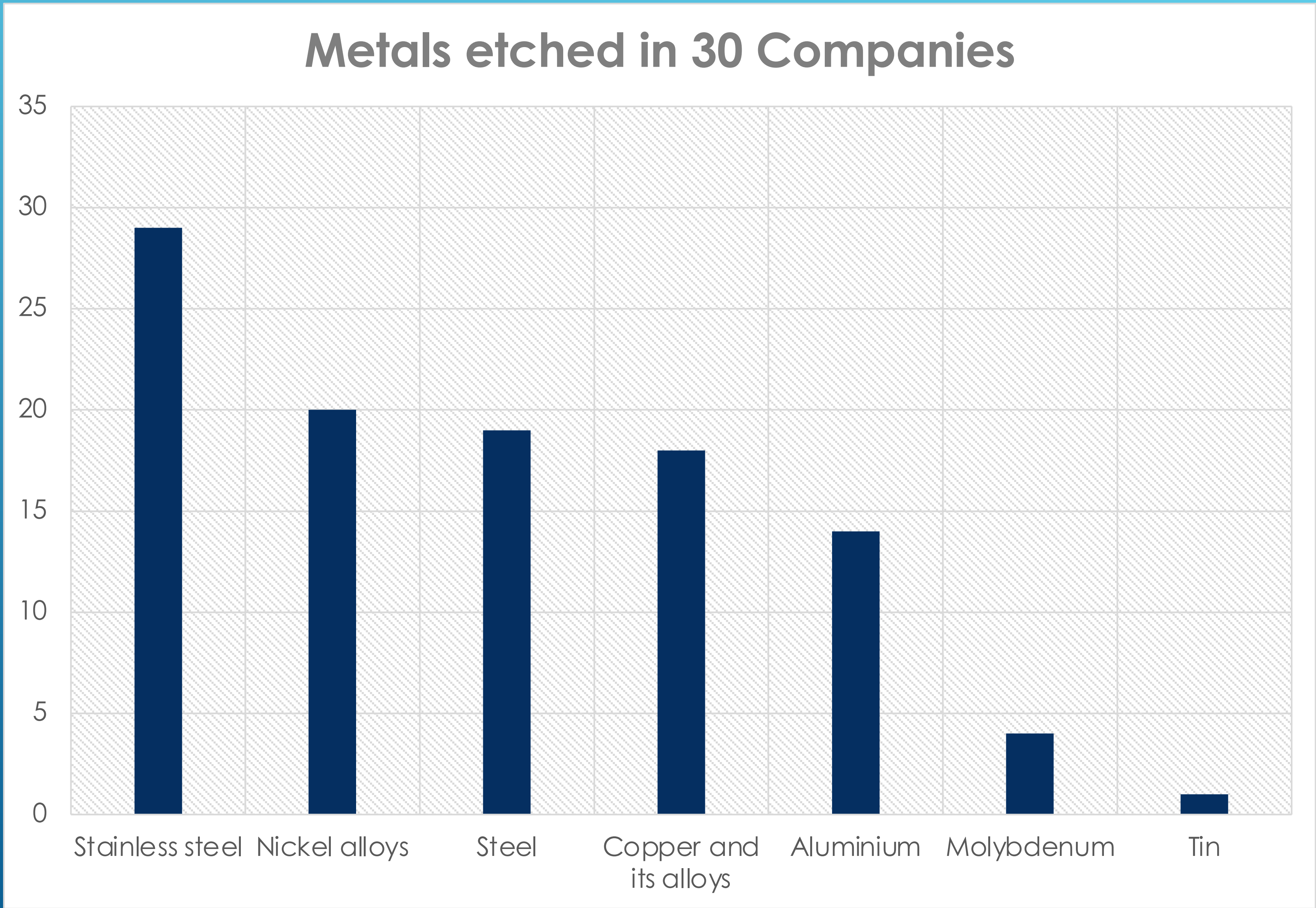
The ratio of etchant purchase cost to waste etchant disposal cost is increasing with time due to environmental pressures and subsequent financial instruments.

Comparative world costs of purchase and disposal for ferric chloride etchant

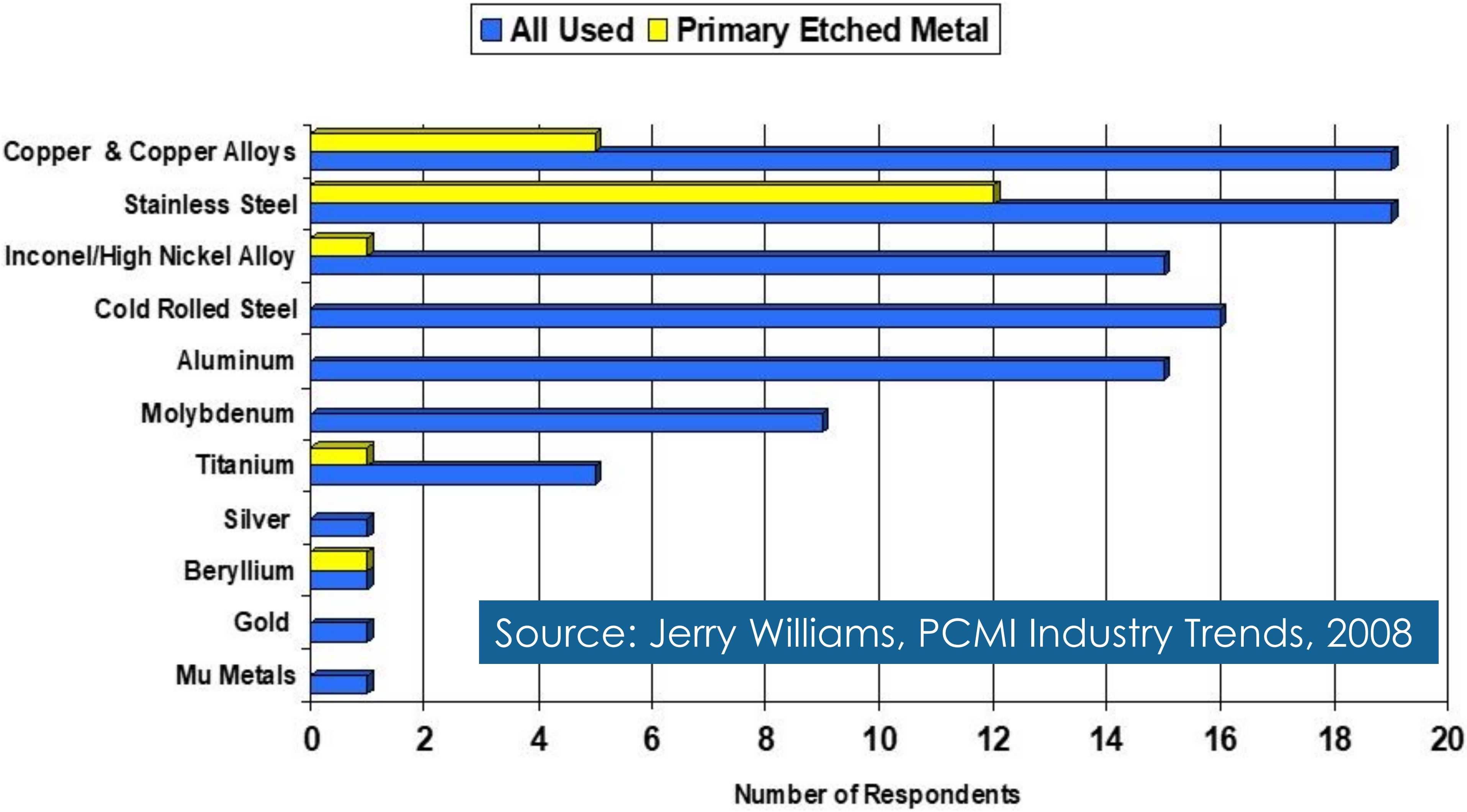
Location and date	Cost of purchasing $FeCl_3$	Cost of disposing waste $FeCl_3$	% of disposal cost compared to purchase price
USA average, March 2022	$3.75 /US gallon	$1.90 /US gallon	50.7%
Europe average, March 2022	€0.35 /litre	€0.50 /litre	143.0%

DM Allen, Regenerating Ferric Chloride Etchant and Waste Disposal Survey Update #2, PCMI Journal, 142, 204-210, 2023.

Metals etched in ferric chloride

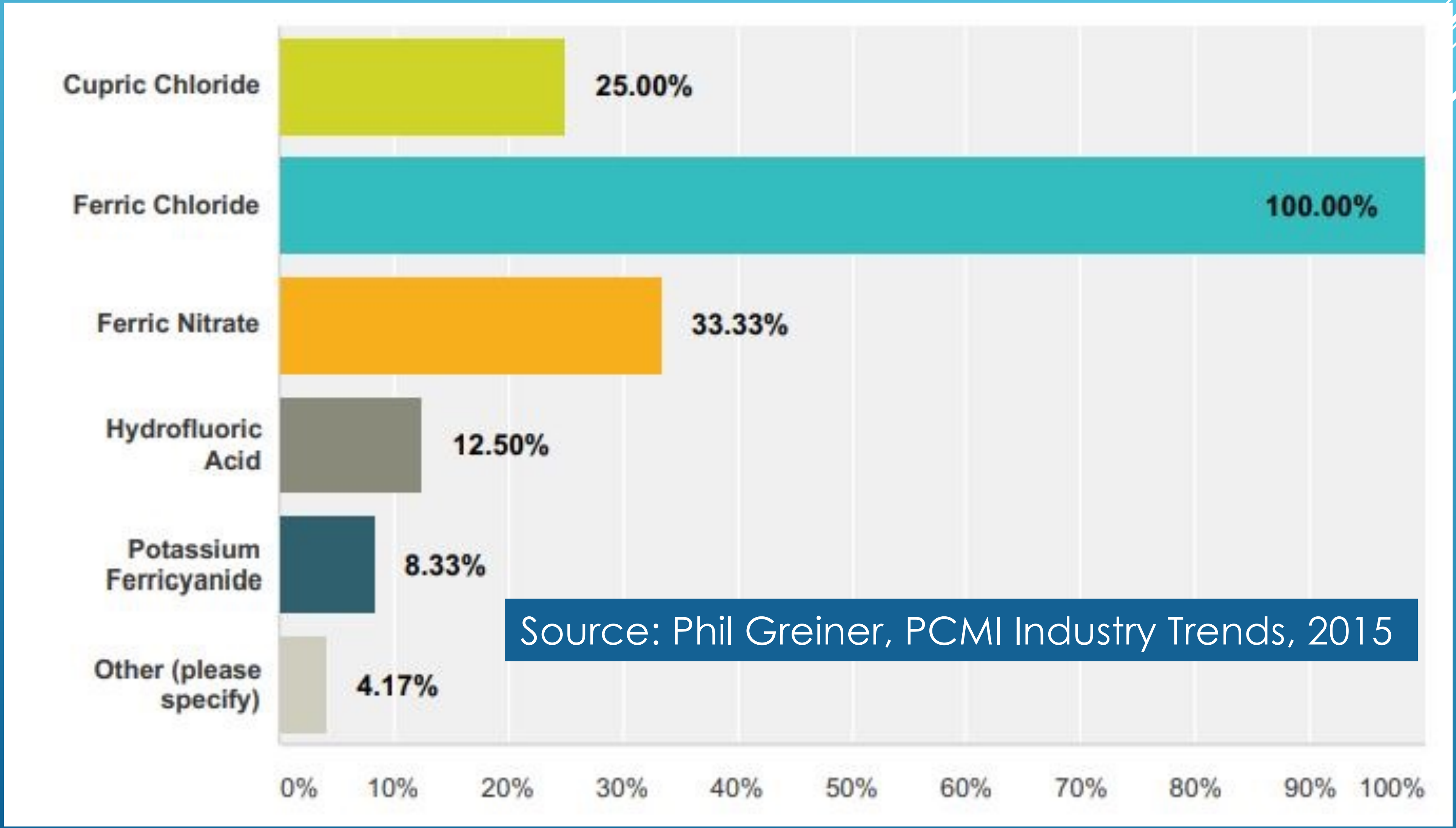


Typical photoetched metals



Source: Jerry Williams, PCMI Industry Trends, 2008

Etchant use for a specific year – in this case, 2015



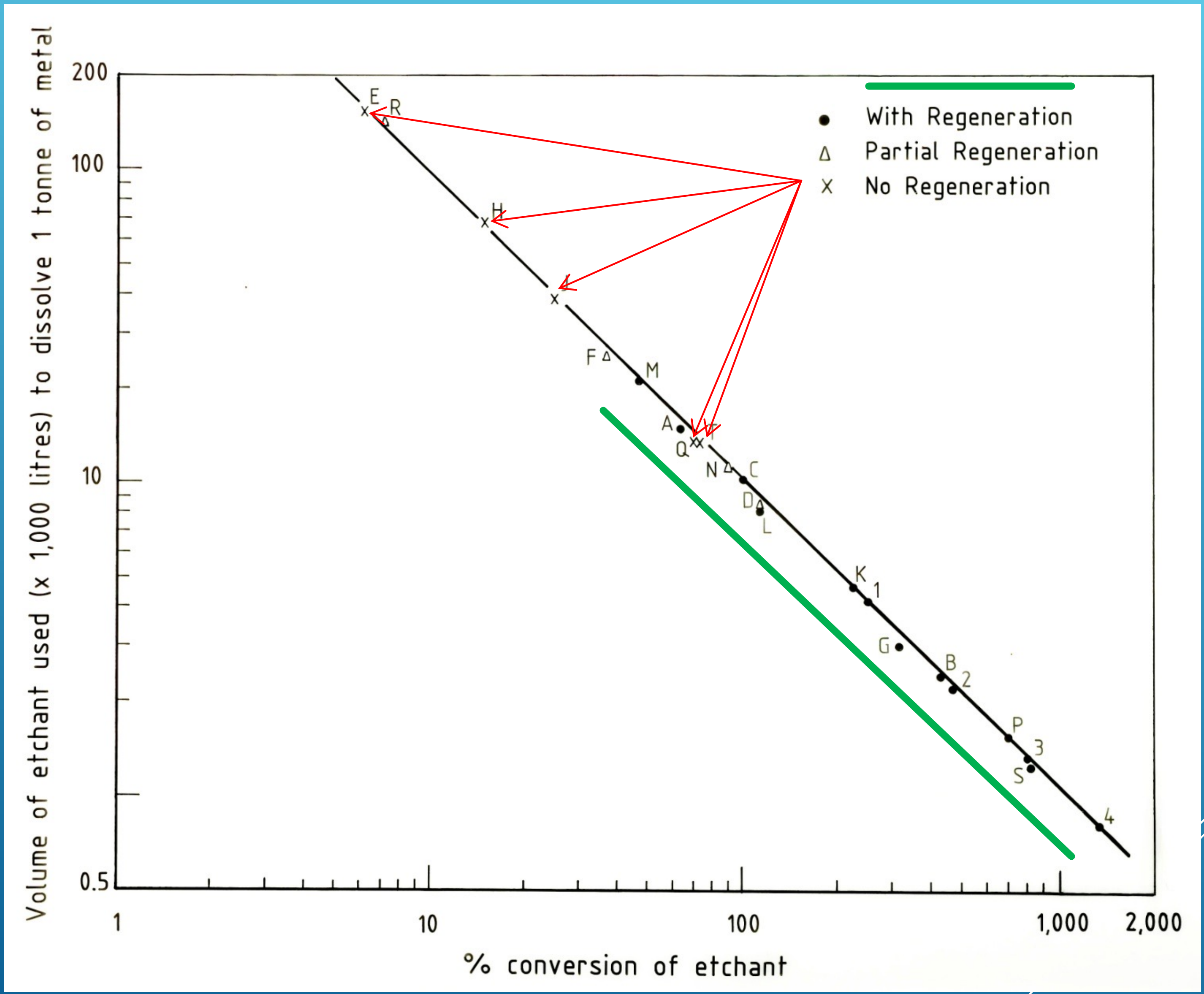
Why do we choose ferric chloride as our primary PCM etchant?

1. Relatively innocuous

2. Relatively cheap

3. Versatility

Environmental impact and cost of PCM is reduced by etchant recycling



Strategies of etching different metal mixtures

Product range	Etchant	Regeneration	Cost
Steels, Stainless steels, Iron-nickel alloys	Ferric chloride	Relatively easy	Medium
Above plus copper and its alloys	Diluted ferric chloride and cupric chloride mixture	Difficult	High
Copper and its alloys	Cupric chloride	Easy	Low

Cupric chloride?

Kevin Dubois (formerly of MD Designs by Metal Decor, Springfield, IL, USA) attended one of my Cranfield University short courses where I said cupric chloride could be a useful alternative etchant to ferric chloride when etching copper alloys.

Subsequently, he replaced one of his ferric chloride etching lines with a cupric chloride line to etch brass parts and stated:

"…I wish I had done that years ago, it is cheaper and so much easier to manage and gives excellent results."

Sad to say, his company closed in October 2020 after failing to survive the Covid pandemic.

Personal experience of using cupric chloride etchant

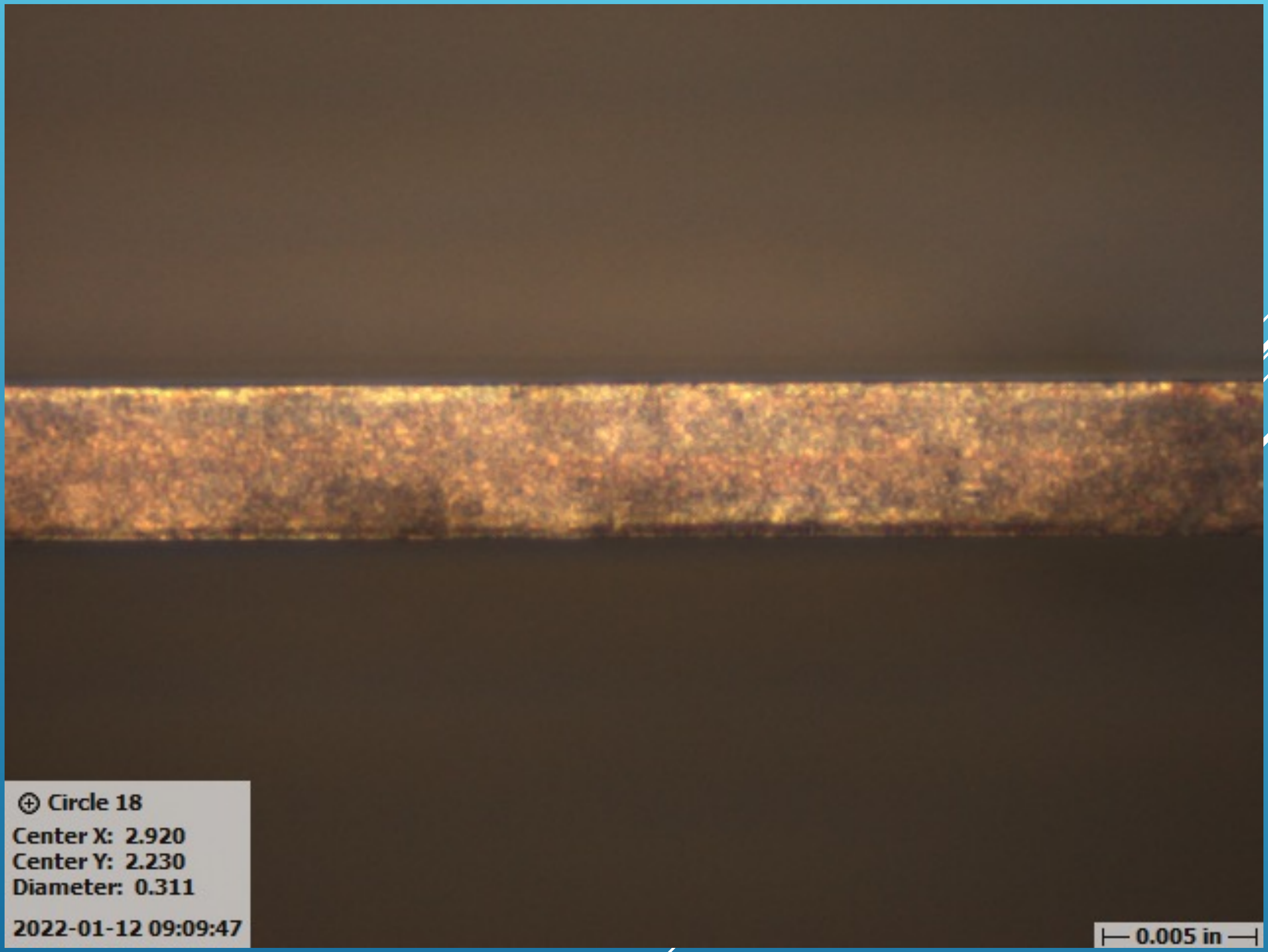
1. DM Allen and O Cakir, Copper etching economics, PCMI Journal, 52, 4-7, 1993.
2. DM Allen, O Cakir and HJA White, Chemical regeneration of waste metal etchants, Proceedings of 1st International Conference on Environmental Engineering, Volume I, 1993.
3. DM Allen and O Cakir, Photochemical machining of copper alloys using cupric chloride etchant, Proceedings of 6th International Machine Design and Production Conference, 357-365,1994.
4. DM Allen and O Cakir, The photochemical machining of brass with cupric chloride etchant and a technique for the partial recovery of dissolved zinc, Proceedings of the Symposium on High Rate Metal Dissolution Processes, Volume 95-19, 305-315, October 1995.
5. DM Allen and O Cakir, The effects of concentration, temperature and HCl additions on the photochemical machining of brass, Proceedings of 7th International Machine Design and Production Conference,191-199, 1996. (**Reprinted** in PCMI Journal, 138, 66-75, 2021).
6. DM Allen and O Çakır, Comparison of $FeCl_3$ and $CuCl_2$ in the photochemical machining of brass, 6th Biennial Conference on Engineering Systems Design and Analysis, pp.1-7, , 8th-11th July 2002.

Cupric chloride –an alternative etchant for etching copper and its alloys

Summary of **technical** aspects:

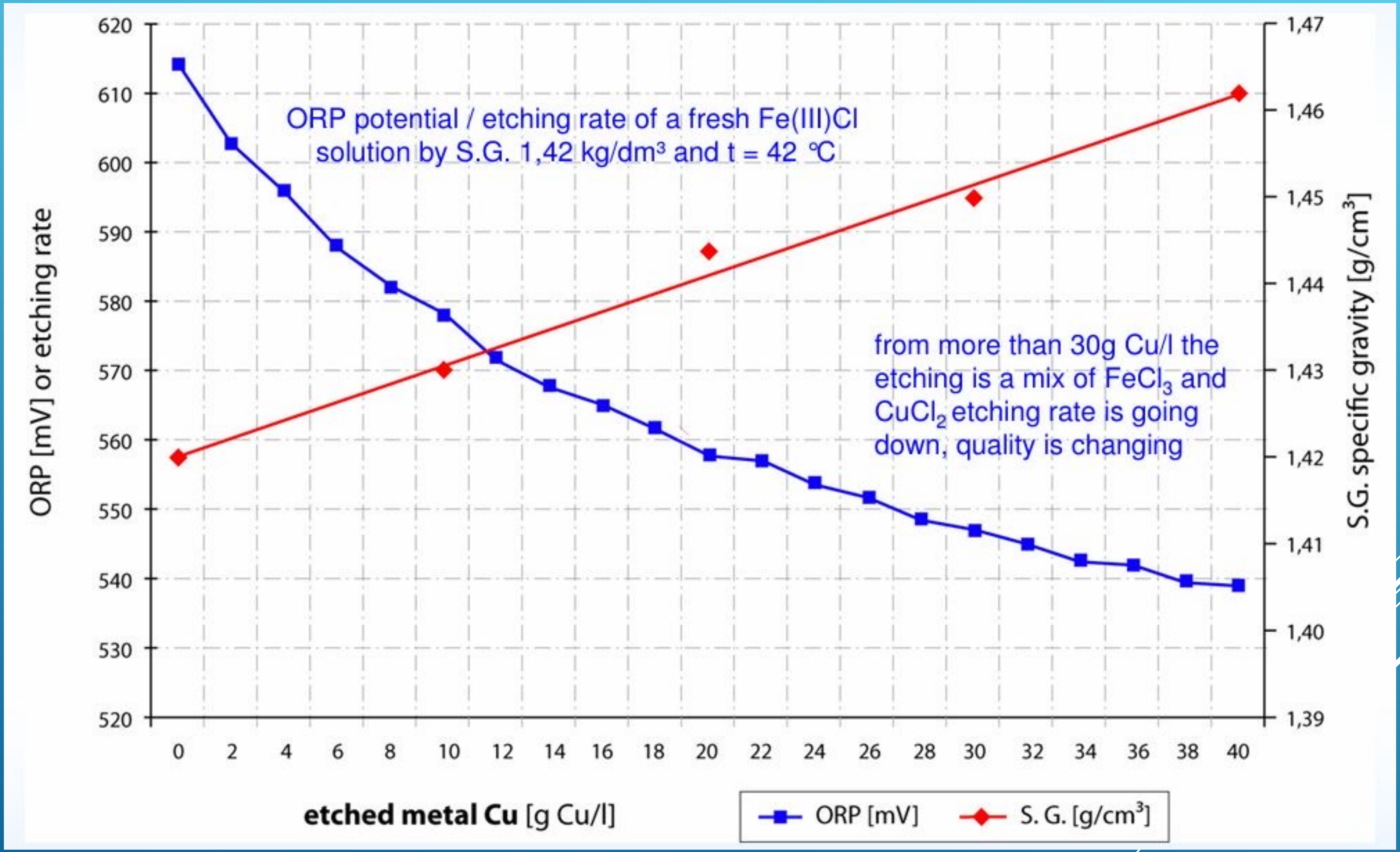
- Etch rate was faster using ferric chloride
- Undercut was lower using cupric chloride
- Etch factor was higher for cupric chloride
- Chemistry of etchant regeneration was less complex for cupric chloride
- Etchant of choice was cupric chloride with additions of hydrochloric acid (≤ 3N) to increase etch rate.

Effect of high etch factor on quality



Smooth edge of annealed 0.005" beryllium-copper part etched in cupric chloride (Courtesy of Orbel Corp., Easton, PA, USA)

Effect of dissolved copper in $FeCl_3$ on ORP [mv] and specific gravity



Courtesy of Hans-Jurgen Schmitz, KSD Innovations GmbH, Hattingen, Germany

Implications for etchant control and regeneration of cupric chloride

Metal / Etchant	Pros and Cons
Brass, copper & copper alloys (e.g. Be-Cu, Cu-Ni-Zn)	Easier regeneration with no Fe^{3+} or Fe^{2+} ions in solution to complicate the chemistry but build-up of other alloy metal ions, such as Be^{2+}, may need special attention for disposal.

$2Fe^{3+} + Cu \rightarrow 2Fe^{2+} + Cu^{2+}$ but both Fe^{3+} and Cu^{2+} will etch Cu

$Cu^{2+} + Cu \rightarrow 2Cu^{+}$ is a simpler system that eliminates Fe^{n+}

Note: Regeneration is essential for economic viability as cupric chloride is more expensive than ferric chloride.

Regeneration chemistry is simple: $Cu^{+} \rightarrow Cu^{2+} + e^{-}$

Etch rates and etch factors for various copper alloys

Alloy	% Copper	% Zinc	% Lead	% Iron	% Nickel	% Other
Brass CZ106	68.5-71.5	Rem. (≈30)	0.05	0.05		
Brass CZ108	62.5-65.0	Rem. (≈35)	0.30	0.20		
Brass CZ128	58.5-61.0	Rem. (≈38)	1.5-2.5	-		
Brass CZ120	58.0-60.0	Rem. (≈40)	1.5-2.5			
Muntz metal	60	Rem. (≈40)		trace		
Bronze	88					12 Tin
Monel 400	32			2.5	63	2 Mg

Alloy	Etch rate (μm/min)	Etch factor	Etch depth (μm)
Brass CZ106	11.1	3.86 ± 0.01	111 ± 5
Brass CZ108	10.3	1.94 ± 0.05	103 ± 1
Brass CZ128	10.1	2.10 ± 0.02	101 ± 3
Brass CZ120	9.8	2.50 ± 0.10	98 ± 2

The role of hydrochloric acid in ferric chloride etchant ***in addition*** to prevention of hydrolysis

Reaction at the metal surface:

$$FeCl_3 + Cu \rightarrow FeCl_2 + CuCl$$

but CuCl is relatively insoluble and will retard etching of the underlying copper surface.

However, hydrochloric acid can complex cuprous chloride and render it soluble, viz.

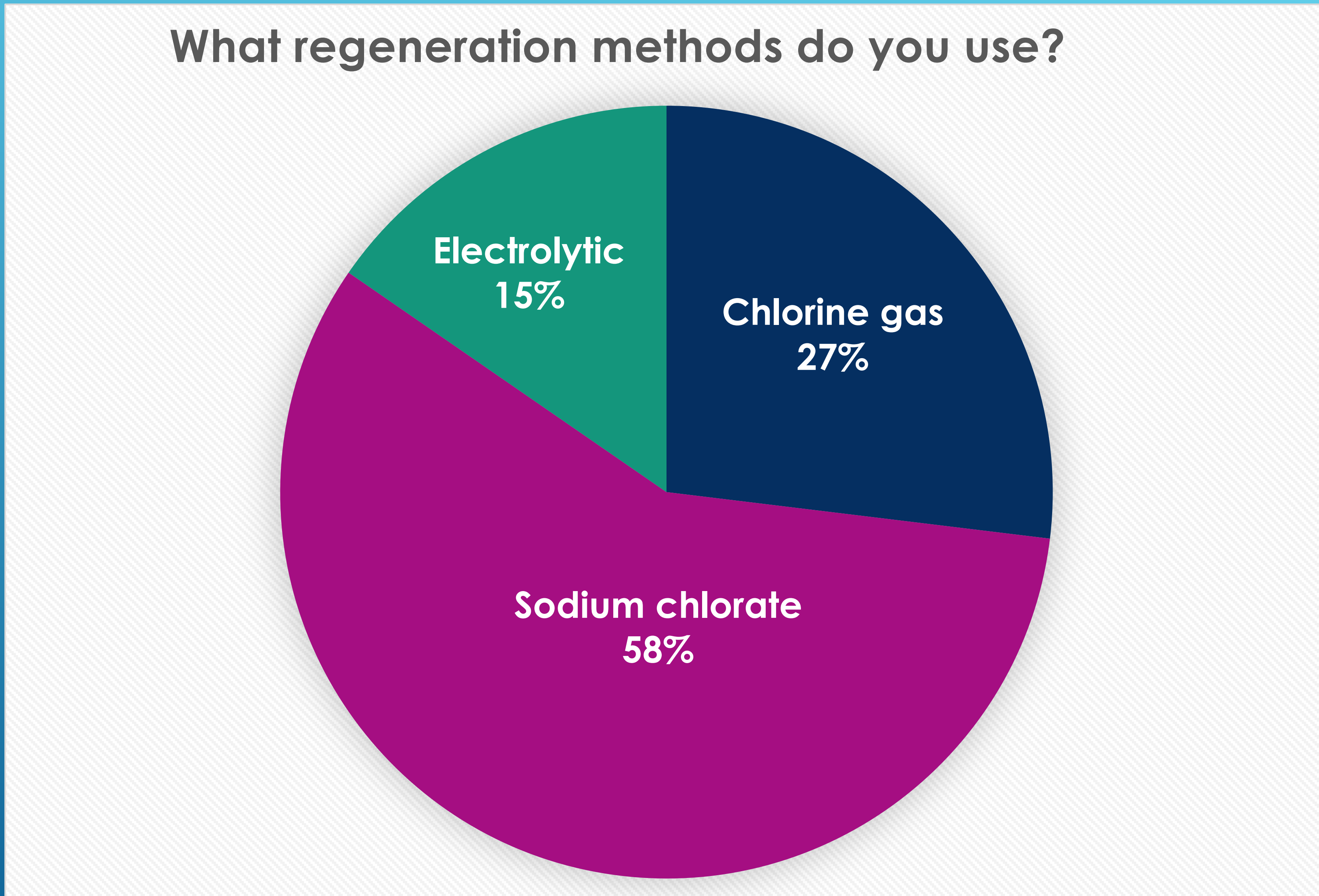
$$2HCl + CuCl \rightarrow H_2CuCl_3$$

Thus, additions of HCl to the ferric chloride etchant are beneficial in increasing the etch rate. In solution

$$FeCl_3 + H_2CuCl_3 \rightarrow FeCl_2 + CuCl_2 + 2HCl$$

and at the metal surface $CuCl_2 + Cu \rightarrow 2CuCl$

Regeneration methods used by 24 companies, some of which use more than one method



DM Allen, Regenerating ferric chloride etchant and waste disposal, Survey #2, PCMI Journal, 142, 204-210, 2023.

Etchant regeneration leads to lower PCM process cost

Can regeneration of cupric chloride etchant be as cost-effective as regeneration of ferric chloride?

Ferric chloride can be regenerated by:

- Chlorine gas (cheapest)
- Sodium chlorate – hydrochloric acid (most popular)
- Electrolytic oxidation
- Ozone gas (expensive)
- Oxygen gas (slow) and
- Air (very slow).

What are the equivalent processes for cupric chloride?

Regeneration of spent ferric chloride

Oxidising agent	Hazards	Challenges	Cost
Chlorine gas	Toxic	Location restrictions	
Sodium chlorate	Explosive and fire hazard	Bulk storage limit?	
Electrolytic	Inflammable H_2 gas at cathode	Electrical power costs. Large footprint. Metal extraction?	
Ozone gas	Toxic and explosive	On-site generation: O_3 gas cannot be stored.	
Oxygen	Compressed gas	Slow*	
Air	None	Very slow*	

* DM Allen and P Jefferies, An economic, environment-friendly oxygen-hydrochloric acid regeneration system for ferric chloride etchants used in photochemical machining, Annals of the CIRP, 55/1, 205-208, 2006.

Electrolytic RotReg NE type regeneration unit to recover copper and other metals from ferric chloride

Courtesy of Hans-Jurgen Schmitz, KSD Innovations GmbH, Hattingen, Germany

Fe-Ni deposition from spent ferric chloride used to etch lead frames

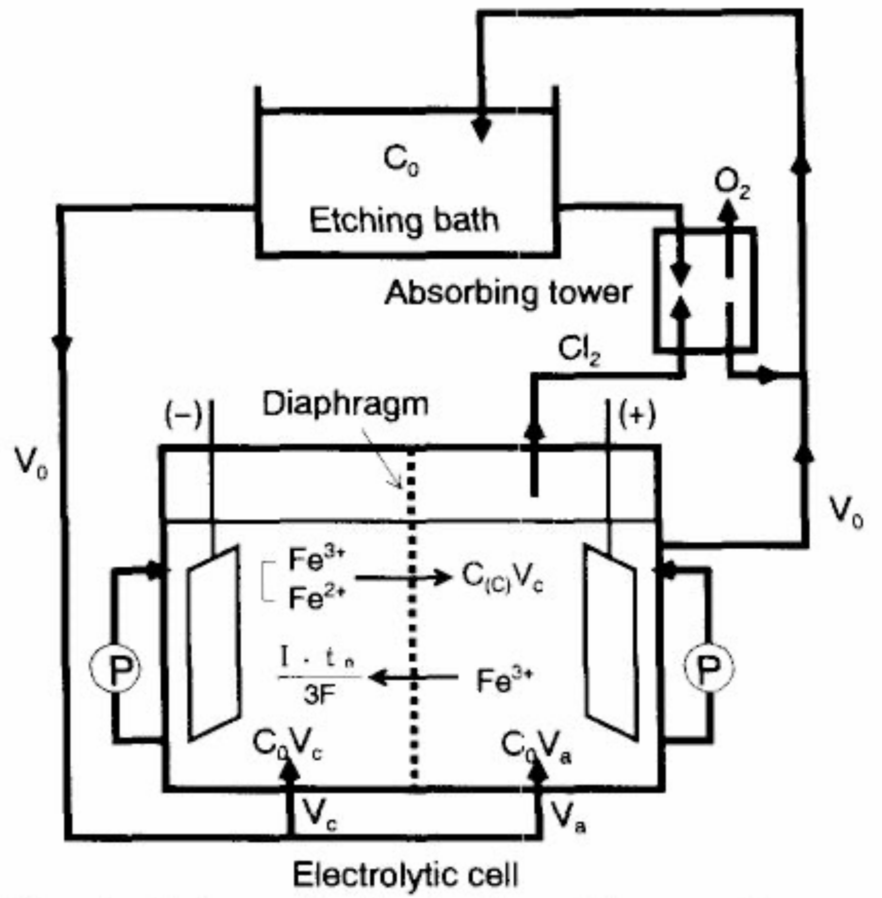


Fig. 1 Schematic illustration of the continuous recycling process.

Diaphragm: Polyester filter cloth TR G800K from Nakao Filter Media Corp., Japan.

Electrodes: DSA (Dimensionally Stable Anode titanium plate coated with ruthenium and tin oxides for chlor—alkali electrolysis TDK Corp, D230) and lead (Pb) plates were used as anode and cathode.

Y Tanimura, T Itoh, M Kato and Y Mikami, Electrolytic regeneration of iron (III) chloride etchant II. Continuous electrolysis, Denki Kagaku, 64, (4), 301-306, 1996.

The role of hydrochloric acid in cupric chloride etchant

Reaction at the metal surface:

$$CuCl_2 + Cu \rightarrow 2CuCl$$

but CuCl is relatively insoluble and will retard etching of the underlying copper surface.

However, hydrochloric acid can complex cuprous chloride and render it soluble, viz.

$$2HCl + CuCl \rightarrow H_2CuCl_3$$

Thus, additions of HCl to the cupric chloride etchant are beneficial in increasing the etch rate.

In summary, $CuCl_2 + Cu \rightarrow 2CuCl \rightarrow 2H_2CuCl_3$ and on regeneration e.g. $2H_2CuCl_3 + Cl_2 \rightarrow 2CuCl_2 + 4HCl$

Regeneration of cupric chloride

Cupric chloride can be regenerated by:

- Chlorine gas
- Sodium chlorate – hydrochloric acid
- Hydrogen peroxide – hydrochloric acid
- Electrolytic oxidation (rare in PCM?)
- Electrolytic closed-loop (used for PCBs)
- Ozone gas
- Oxygen gas (viable) and
- Air (slow).

Regeneration chemistry of etching copper with cupric chloride

1. Chlorine gas:

$$Cu + CuCl_2 \rightarrow 2CuCl \xrightarrow{Cl_2} 2\,CuCl_2$$

2. Sodium chlorate plus hydrochloric acid

$$Cu + CuCl_2 \rightarrow 2CuCl \xrightarrow{1/3\,NaClO_3 + 2HCl} 2CuCl_2 + 1/3NaCl + H_2O$$

3. Hydrogen peroxide plus hydrochloric acid

$$Cu + CuCl_2 \rightarrow 2CuCl \xrightarrow{H_2O_2 + 2HCl} 2CuCl_2 + 2H_2O$$

NB. Doubling of [$CuCl_2$] in all cases requires water additions to maintain original concentration*

* D Ball, Chemcut Corporation, *Process Guidelines for Cupric Chloride Etching*, Technical Information Bulletin 8, 2010.

Cost of regeneration of cupric chloride with sodium chlorate and hydrochloric acid / lb Cu

Materials	Gm	Gm/mole	Moles	Molar ratio	Gm required	Lbs required	Lbs/ US gallon	US gallon
1 lb of copper to be etched	454	63.546	7.144	1				
46 wt.% Sodium chlorate		106.44	2.381	1/3	253.4	0.558	**5.371**	0.104
Hydrochloric Acid (HCl)		36.46	14.29	2	521.0	1.15		
Muriatic Acid (31% HCl)						1.15	**3.093**	0.372

630 lb drum of chlorate contains 54.2 US gallons and costs $1,237.71

0.104 US gallon of 46% $NaClO_3$ will cost **$2.37**

Cost of muriatic acid is $0.58/lb

0.372 US gallon of muriatic acid will cost **$2.07**

To etch 1lb copper, regeneration cost of $NaClO_3$ and HCl = $4.44

Cost of regeneration of cupric chloride with hydrogen peroxide and hydrochloric acid

Materials	Gm	Gm/mole	Moles	Molar ratio	Gm required	Lbs required	Lbs/ US gallon	US gallon
1 lb of copper to be etched	454	63.546	7.144	1				
50 wt.% H_2O_2		34.01	7.144	1	243.0	0.535	**4.978**	0.108
31 wt.% H_2O_2		34.01	7.144	1	243.0	0.535	**2.869**	0.186
Hydrochloric Acid (HCl)		36.46	14.29	2	521.0	1.15		
Muriatic Acid (31% HCl)						1.15	**3.093**	0.372

50% hydrogen peroxide costs $13.74/US gallon

0.108 US gallon of 50% H_2O_2 costs **$1.48**

31% hydrogen peroxide costs $14.44/US gallon

0.186 US gallon of 31% H_2O_2 costs **$2.69**

Muriatic acid is $0.58/lb. 0.372 US gallon of muriatic acid will cost **$2.07**

To etch 1lb of copper, regeneration cost of H_2O_2 and HCl = $3.55 - $4.76

Cost comparisons between sodium chlorate and hydrogen peroxide regeneration of spent cupric chloride

Regeneration method	Cost		
	US$/lb (Wible)	UK£/kg (Decker)	US$/lb (Allen)
Chlorine gas	0.24-0.58	0.19	-
$NaClO_3$ / HCl	0.43-0.70	0.40	4.44
H_2O_2 / HCl	0.77-1.04	0.56	3.55-4.76

PM Wible, Regeneration of etchants, PCMI Journal, 6, 711, 1981.

G Decker, Various methods of etchant regeneration – advantages, processes, equipment and cost analysis, undated *Technical Note,* (Chemcut GmbH).

DM Allen, Calculations from prices of chemicals supplied to Chemcut Corp, State College, PA, USA, March 2025 (Courtesy of Kirk Lauver)

Regeneration chemistry of etching copper with cupric chloride

4. Oxygen and air:

$$Cu + CuCl_2 \rightarrow 2CuCl$$

$$2CuCl + 2HCl + \tfrac{1}{2}O_2 \rightarrow 2CuCl_2 + H_2O$$

Regeneration of spent cupric chloride with oxygen gas

A scaled-up reactor of 0.5 m^3 volume and estimated monovalent copper oxidation rate of 20 kg/h was constructed as shown right.

D Kopyto *et al*, Environmentally friendly method for regeneration of copper chloride acidic solutions used in etching of printed circuits, E3S Web of Conferences, 18, 01021, 2017.

Total cost of etchant utilisation

Debit

- Purchase cost of etchant
- Recycling strategy
- Waste disposal cost for spent etchant

Credit

- Secondary products

(One man's waste is another man's starting material: the concept of **Waste Exchange**)

One man's waste is another man's starting material

Spent cupric chloride etchant can be reused as:

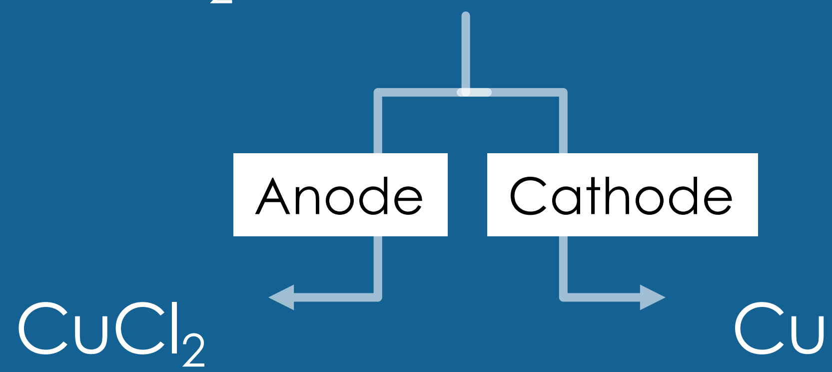
- A leaching agent to dissolve copper from recycled Waste Electrical and Electronic Equipment (WEEE) Directive waste
- A pigment for colouring plastic*
- A flocculant/coagulant
- A source of valuable copper metal

*DM Allen and O Cakir, Copper etching economics, PCMI Journal, 52, 4-7, 1993.

Regeneration chemistry of etching copper with cupric chloride

5. Electrolytic processing summary:

$$Cu + CuCl_2 \rightarrow 2CuCl$$



Value of reclaimed copper can be up to 75% price of grade A copper

NB. The [$CuCl_2$] remains at its original concentration

Electrolytic regeneration of spent cupric chloride etchant

This is a little-used process that dates back to at least 1987 when Dr Markus Bringmann (Chema Technologien GmbH, Sprockhövel, Germany) published the following paper in German:

M Bringmann, Wirtschaftliche und umweltfreundliche Regeneration von gebräuchlichen Atzmedien auf electrolytischem Wege, Galvanotechnik,10(78), 2-6, 1987.

The title translation is:

“Economical and environment-friendly regeneration of common etchants by electrolytic means”

The process did not appear to be an attractive one for PCM companies.

Electrolytic regeneration background

CHEMA Technologien GmbH, Germany

F Dorrenbach, Electrolytic regeneration processes of common etchants, PCMI Journal, 44, 16-17, 1991.

Electrochemical Design Associates, Inc., USA (EDA)

D Brackenberry *et al*, Fregen™ - Electrochemical regeneration of ferric chloride and cupric chloride etch solutions, PCMI Journal, 78, 1723, 2000.

Cemco-FSL, UK (Licensed from EDA)

P Watson and A Brown, Improved inner layer etching by simultaneous cupric chloride regeneration and copper recovery system, www.PCB007.com , March 2008.

Case history at Tri-Star Technologies Co. Inc., MA from Toxics Use Reduction institute, University of Massachusetts Lowell, USA

Cupric Chloride Etch Regeneration, Technical Report, 45, 1997.

Electrolytic recovery of dissolved copper from printed circuit boards

J Sallo and T Ricks, Recovery of copper metal in the continuous regeneration of cupric chloride etchant, Proc. IPC Printed Circuits Expo, APEX and The Designers Summit, 2008.

Abstract

An electrochemical device has been developed that **deposits non-adherent copper metal powder at the cathode and generates chlorine gas at the anode**. The chlorine is contained within the system and is used to regenerate the etchant in a continuous closed loop process. Chlorine is generated only when the electrochemical process is underway. There is never any stored chlorine gas.

…..The current high price of copper metal has made this technology very economically viable.

Sallo & Ricks: equipment functionality

Figure 1 Cathode Arrangement

Chlorine gas is evolved at DSA® (Dimensionally Stable Anodes) titanium anodes coated with mixed metal oxides based on Ir, Ru, Pt, Rh and Ta.

The chlorine gas is fed back into the spent (cuprous chloride) etchant to regenerate fresh cupric chloride

Regeneration of spent cupric chloride

Oxidising agent	Hazards	Challenges	Cost
Chlorine gas	Toxic	Location restrictions	
Sodium chlorate	Explosive; fire hazard	Bulk storage limit?	
Hydrogen peroxide	Unstable	Shelf-life; bulk storage limit?	
Electrolytic closed-loop system	Large Direct Currents	Location critical for cheap electricity. Large footprint.	
Ozone gas	Toxic and explosive	On-site generation -gas cannot be stored.	
Oxygen	Compressed gas	Viable etch rate[1]	
Air	None	Viable etch rate of 8μm/min by immersion[2]	

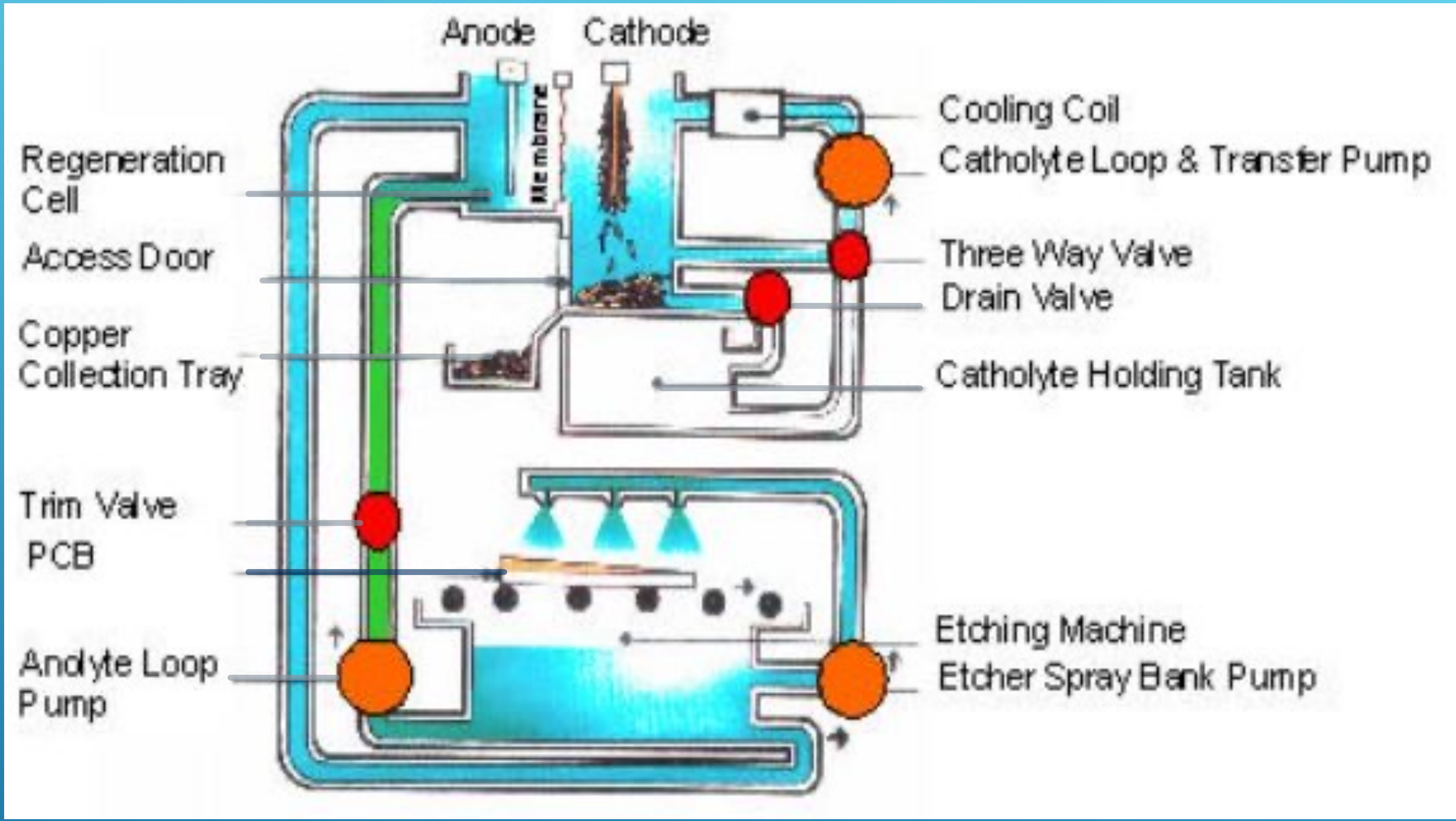
1. D Kopyto *et al*, Environmentally friendly method for regeneration of copper chloride acidic solutions used in etching of printed circuits, E3S Web of Conferences 18, 01021, 2017.
2. B Fadhil, Copper etching in air regenerated cupric chloride solution, Journal of Engineering, Number 3, Volume 16, September 2010.

Metal extraction from spent ferric chloride and spent cupric chloride etchants

Etchant	Dissolved metals	Extraction method	Cost
Ferric chloride	Iron from carbon steels	Not financially viable	Not applicable
Ferric chloride	Nickel and chromium from stainless steels	Solvent extraction?[1]	Yet to be determined
Ferric chloride	Copper & Fe/Ni	Electrolytic	Viable
Cupric chloride	Copper	Electrolytic	
Cupric chloride	Copper and zinc from brass	Electrolytic?[2]	Yet to be determined

1. P Jefferies and DM Allen, Solvent extraction of dissolved nickel and chromium from spent ferric chloride etchants, PCMI Journal, 139, 30-48, 2022.
2. DM Allen and O Cakir, The photochemical machining of brass with cupric chloride etchant and a technique for the partial recovery of dissolved zinc, Proc. Symposium on High Rate Metal Dissolution Processes, Volume 95-19, The Electrochemical Society, 305-315, 1995.

Cemco-FSL: electrolytic Cu regeneration



BENEFITS

- No chemical oxidising agent is required
- No storage of fresh and spent etchant
- Low copper level in etchant promotes sludge-free working
- The only byproduct is copper powder
- A profit over total running costs is made

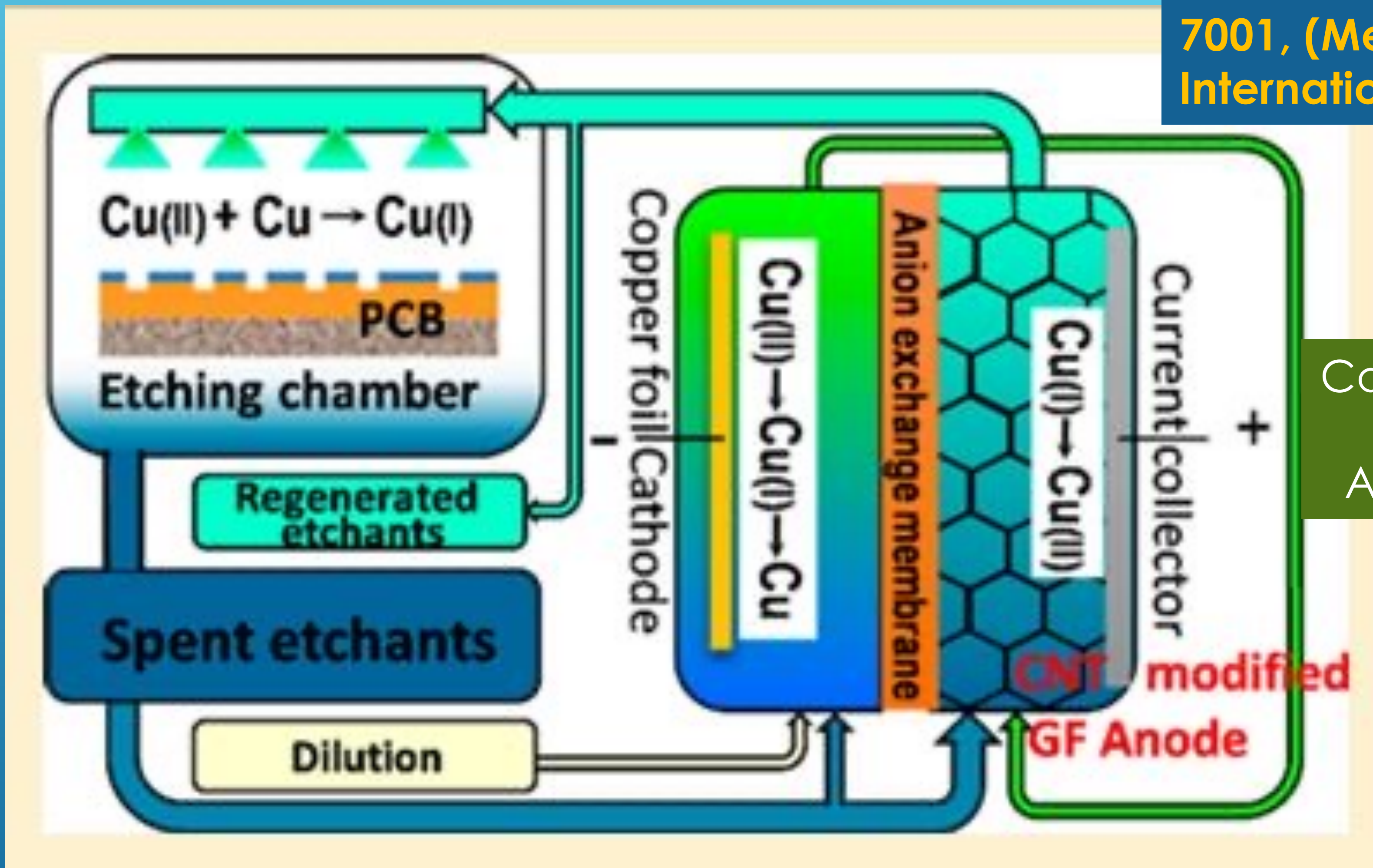
Metallic copper is removed at a maximum rate of 8 kg/hr at the cathodes of the system and sold as scrap. The power requirements of this system are 8,000 amps DC at 6-10 V.

Novel electrode system: abstract to paper by Y Chang *et al*

Developing effective technologies for treatment of spent etchant in printed circuit board industries is paramount for sustainable copper reuse and reducing copper discharge. We developed a novel closed-loop electrochemical cell for on-site regeneration of spent acidic cupric chloride etchant. It does not have any emissions and recycles all the copper using a three-dimensional graphite felt (GF) anode decorated with carbon nanotubes (CNT). The CNT/GF anode oxidizes Cu(I) to Cu(II) so that the spent cuprous chloride can be converted to cupric chloride and reused. The decorated CNT layer with abundant oxygen-containing functional groups significantly enhanced the electrocatalytic activity for Cu(II)/Cu(I) redox. The $CuCl_3^{2-}$ is oxidized to CuCl+ at the anode and the CuCl+ is reduced to Cu(0) at the cathode. The closed-loop cycle system converts the catholyte into the anolyte. On average, the energy consumption of Cu(I) oxidation by CNT/GF is decreased by 12%, comparing to that by untreated graphite felt. The oxidation rate of Cu(I) is determined by the current density, and there is no delay for the mass transport of Cu(I). This study highlights the outstanding electrocatalytic performance, the rapid mass-transfer kinetics and the excellent stability of the CNT/GF electrode and provides an energy-efficient and zero-emission strategy for the regeneration of etchant waste.

Closed-loop system schematic

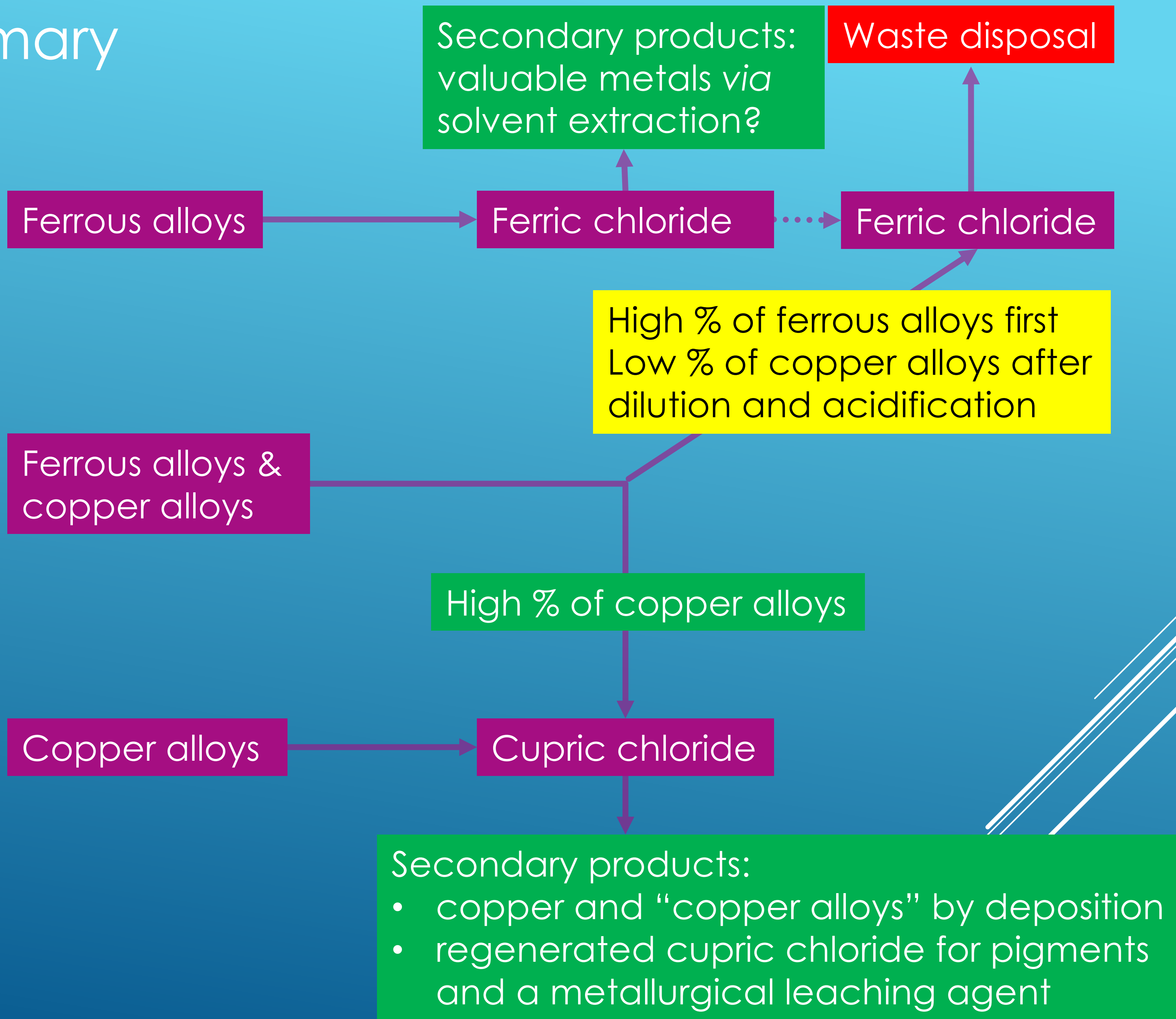
Anion Exchange Membrane: AMI-7001, (Membranes International Inc.)



Catholyte
↓
Anolyte

Y Chang *et al,* Closed-Loop electrochemical recycling of spent copper(II) from etchant wastewater using a carbon nanotube modified graphite felt anode, Environ. Sci. Tech., *52*, 5940-8, 2018.

Summary


Secondary products:
valuable metals *via*
solvent extraction?
Waste disposal
Ferrous alloys
Ferric chloride
Ferric chloride
High % of ferrous alloys first
Low % of copper alloys after
dilution and acidification
Ferrous alloys &
copper alloys
High % of copper alloys
Copper alloys
Cupric chloride
Secondary products:
• copper and "copper alloys" by deposition
• regenerated cupric chloride for pigments and a metallurgical leaching agent

How to acquire secondary products from cupric chloride etchant regeneration used to etch copper

Secondary product	Regeneration technique	Consumables	Cost
Copper (Sallo/Ricks)	Electrolytic	Electrical power	
Copper (Cemco-FSL)	Electrolytic	Electrical power	
Copper (Yang *et al*)	Electrolytic	Electrical power	
Cupric chloride excess	Sodium chlorate/HCl	$NaClO_3$ /HCl	Salt
Cupric chloride excess	Hydrogen peroxide/HCl	H_2O_2 /HCl	
Cupric chloride excess	Oxygen or air	Oxygen	

Q. What is the situation when etching copper alloys such as brass?
A. Surprisingly, the Cemco-FSL system has never been tested on brass!
Q. Is electrodialysis the technique to extract the zinc from the spent etchant?

Suggestions for reducing costs of PCM for copper and its alloys

- Reduce the complexity of a byproducts mixture by etching with cupric chloride rather than ferric chloride.
- Avoid unnecessary byproduct formation. In the regeneration process aim to use hydrogen peroxide or oxygen/air to avoid the production of salt (NaCl) as a byproduct obtained when regenerating with sodium chlorate.
- The production of valuable metallic secondary products is encouraged to offset costs of regeneration chemistry and plant.
- Electrolytic closed-loop regeneration is profitable when secondary product copper metal can be extracted and sold.
- The production of mixed metal secondary product can generate an income but is expected to be less than that for a single pure metal secondary product.

Acknowledgements

I wish to thank the following for their help in preparing this presentation:

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- Hans-Jurgen Schmitz (KSD Innovations GmbH, Hattingen, Germany) and
- Peter Lymn (Cemco-FSL, Waterlooville, Hants, UK)

Thank you for your kind attention.

Do you have any questions?

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