Corporate extinction
Adapt to survive: the changing US model

Solar battery challenge
Rugged endurance trials in Australia’s outback

Smelting’s death knell
Aqua Metals’ technology offers viable alternative

Dreamweaver test result
How separators can beat nail penetration test

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Lead — as we’ve been told countless times — is the most recycled metal on the planet. But reclaiming it by smelting is energy-intensive, expensive and, worldwide, frequently a dirty and polluting process. An alternative may soon be on offer. *Batteries International* reports.

### The next step forward for lead recycling

Lead smelting recycling without the smelting? That’s what Aqua Metals, a Californian start-up, promises to provide later this year. If achievable then there could be huge benefits — and a huge shake-up — for the entire lead battery industry.

The idea, as such, isn’t new; this hydrometallurgical chemistry dates back to the very earliest days of electrolysis. But so far this has never been commercially practical. And many have believed that this will always be the case.

However, independent analysts and equally cynical investors, now say the opposite is true. Maggie Teliska, the head of independent testing firm, RyanTel, conducted a review for the US Department of Agriculture — one of the guarantors of a project loan from Green Bank to build the first commercial plant — and confirmed the validity of Aqua Metals’ technology and business.

"Initially I was not just sceptical but deeply sceptical about this," she told *Batteries International*. "But as I conducted the technical feasibility study and saw the hydrometallurgical process in action, I saw that this was not just viable but real and even had a genius quality to it."

Teliska whose doctorate in physical chemistry is particularly relevant, has signed a non-disclosure document barring her from revealing the details. That said she told the magazine that she had gone through the paperwork to validate areas such as the finances and the business and distribution model. "I can vouch that the costs, sales and revenue projections add up," she said.

Perhaps, most importantly she validated the fact that the refining process was scalable. On the basis of her recommendations, the USDA Rural Development Agency is guaranteeing 90% of a $10 million commercial loan from Green Bank.

Aqua Metals has also been endorsed by investors who though hampered by the NDA could legitimise to *Batteries International* what Aqua Metals is doing.

“We’ve seen the technology in ac-
“Aqua Metals’ technology has the capability to change the global lead acid battery recycling industry. It offers a lower operating cost structure, and a lower recycling volume requirement, allowing all battery manufacturers to control the availability and cost of their lead. Every battery manufacturer should consider the Aqua Metals technology in their long term strategy”

by Stephen Clarke, Selwyn Mould and Thomas Murphy, three figures who had already been working together for many years on energy storage technology including flow and bipolar lead acid batteries.

Aqua Metals was formed in 2013 by Michael Cahill, founder of investment firm Crispin Capital Management, which has taken a long position in Aqua Metals.

“Aqua Metals may currently have a small market capitalization [the total value of all the issued shares], but we could see it being worth $1 billion in the not-so-distant future.

“The ability to build an Aqua Metals modular facility next to a battery collection centre — removing the need for moving spent batteries to distant, expensive smelting centres — is a compelling business case that could witness mass adoption worldwide. Given the large market opportunity, it also would not surprise me to see a larger battery company try to acquire Aqua Metals”

Engitec, looked at a similar process and announced in 2010 with great fanfare that it was going to be a game-changer for the industry. Doe Run’s technology used a wet chemical process to selectively dissolve lead concentrates into solution, then it extracted lead from the solution using an electric current. (The electrowinning process is similar to the technology used to extract zinc from concentrates, but had never been used in primary lead production.) As a self-contained process, the activating solution is recycled back into the process indefinitely.

However, Doe Run’s plans never materialized — the $150 million the company initially sought to take the process commercial was never raised. The figure was dropped to $100 million but the firm decided in 2012 that the investment was too risky.

At the end of 2013 Doe Run shut its main smelting operation in Hercules, in the US state of Missouri following pressure from the Environmental Protection Agency and a $65 million fine for previous violations.

If Aqua Metals’ product is commercially viable, then AquaRefining has the potential to be a game changer for the industry. Aqua Metals will not release further details of its intellectual property — see box — but it is more than likely that a pulp of crushed batteries would be introduced into the electrolytic process. This might be in sponge form to provide the surface area need for precipitation of lead to occur.

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A bank consists of 3 electrolyzer units and is a skid mountable, shippable building block — two banks = one module
The firm said it believes too much detail would give commercial advantage to potential competitors (see boxed item below).

Aqua Metals’ chief commercial officer, Steve Cotton, told Batteries International that in all there are a host of related patents pending. However, it is certain that the solvent is different from the tetrafluoroboric acid used by Doe Run. Fluoroboric acid is as corrosive as nitric acid.

Cotton says AquaRefining uses harmless chemicals in the refining process.

There are four immediate business positives to Aqua Metals’ refining process.

The first is that AquaRefining appears to be far more efficient than smelting. The amount of energy needed to be input into the system is smaller –

### RECYCLING LEAD BATTERIES: A SMELTING OVERVIEW

Smelting is the only commercial process for recycling lead. It is an old and inefficient thermal reduction process technically difficult and expensive to bring into compliance with increasingly stringent environmental standards.

Smelters produce virtually all the world's mined and recycled lead. Smelting is an inefficient, energy intensive and often highly polluting process.

At its core, smelting is a high temperature (typically above 750°C/1400°F) chemical reduction process where lead compounds are heated and then reacted with reducing agents to remove the oxygen and sulfur, leaving behind lead.

The chemical reactions are endothermic, which means that heat must be continually supplied to replace the energy consumed by the reduction processes. In smelting, 5% to 15% of the lead is lost as slag and the lead produced typically contains 2% or more of impurities.

Smelting is only cost effective at large scale, typically for more than 200 tonnes of lead per day.

In addition to the high costs and inefficiencies associated with smelting, it generates large volumes of toxic solid, liquid, particulate and gaseous waste. In developed countries, there is both increased environmental regulation and enforcement of such, including monitoring of permissible blood lead levels in employees and local populations.

In the US, in particular, many smelters have been forced to close because of environmental compliance. While some modern smelters seek to comply with environmental and safety standards, they face elevated capital and operating costs as a result.

Meanwhile there has been a drift of recycling capacity and operations into countries and regions with lower environmental and labour standards and weaker levels of enforcement.

Lead smelting is consistently ranked as the third highest polluting industry in the world.

Historically, lead acid battery recycling required:
- Breaking and separation equipment
- Effluent treatment systems
- Bunkers with loose lead paste and materials dried before charging the furnaces
- Rotary or blast furnaces for smelting
- Lead refining and ingoting equipment
- Air filtration systems, and
- Paste desulfurization systems.

As a brief overview of the recycling process, a battery is broken and separated into four product streams (lead, lead oxide paste, plastics, and electrolyte) within the breaking and separation system.

Historically lead oxide paste is charged in a rotary furnace, with a number of other materials and additives, which produces toxic off gas emissions, dross, and a number of other waste products.

Because of these negative byproducts of smelting, the industry is highly environmentally regulated. Environmental regulations and labour safety standards are going to get tougher in developed nations and will be introduced in developing countries as well.

These changes will favour clean technologies, such as electrorefining, in preference to smelters and other technologies that produce airborne emissions.

High temperature rotary or blast furnaces are the main piece of equipment that has not been advanced by technology over the years. The principle goes back hundreds of years, and this is the dirtiest, and worst part of a recycling operation.

There are various shortcomings of rotary furnaces most obviously in that they can produce negative emissions and burn at over 1800°F. Strict environment regulations make it nearly impossible to build a new smelter-base recycling centre in the US.

They are also energy intensive and have to stay heated up, even when not smelting as well as requiring a large capital investment along with
making it cheaper by around a third — and because the process is modular it can be tuned to demand. (As opposed to smelters which typically have to be operated with a high-output to make economic sense.) Lead is also not lost in the slag of smelting, improving the economics as well as vastly reducing the environmental impact of hazardous disposal of the slag.

Second, AquaRefining is modular — making this a scalable product. The basic unit (see pictures of the 2013 version and its upgrade in 2014) means that the business model offers a different approach to market.

Typically smelting requires the smelter to be built on site and in large size. However, AquaRefining can be located at the hub of any distribution network or battery manufacturing location at a scale that suits the amount of lead battery recycling facilities have been getting shut down, restricted, and denied permitting to build new or expand operations due to strict environmental regulations, and it will continue to get more strict in the future.

The transport of hazardous waste is also an element in the fluctuating market.

The Basel Convention on the Control of Trans-boundary Movements of Hazardous Wastes and their Disposal came into force in 1992 with 172 countries signing on to it. It is the most comprehensive global environmental agreement on hazardous and other wastes that strongly influences the market for lead, lead acid batteries and used lead acid batteries.

The Basel Convention also establishes limitations on which countries may be recipients of hazardous materials such as used lead acid batteries. However, trade in battery paste is unrestricted and is considered a so-called “product of commerce” undifferentiated from other products.

To add to the confusion, while the

EU and many developing nations have ratified it, the US has not, choosing to regulate lead acid batteries through similar but different EPA regulations. Furthermore some countries that have ratified it don’t enforce it in any meaningful way.

One consequence of this situation has been a significant distortion and under-reporting of the official international trade in used lead acid batteries and battery paste.

As a result, thriving, unregulated and under-reported secondary markets have developed in which pricing can be much higher than the London Metal Exchange’s price. For example, as of August 2014, the unofficial price in Pakistan was $3,400/tonne for secondary lead and $3,800/tonne for primary lead. India has had similar prices in its unregulated markets.

This situation has created a thriving sub-set of the used lead acid battery recycling industry where the batteries are broken down and the paste is shipped overseas for smelting, often in unregulated and highly polluting facilities.

This complex situation compounds pollution issues and hurts the lead battery industry which lobbies hard to reinforce for its leadership in recycling and efficient resource re-use.
PROFILE: AQUA METALS

Big is beautiful too: aerial view of the new plant being built in Tahoe. For a sense of scale the green dot in the middle of the concrete is Steve Cotton, chief commercial officer. In the background to the left is the warehouse of Battery Systems which (also inset) the partner, supplier of the used lead acid batteries to the future plant.

AquaRefining is environmentally friendly and sustainable. — an important factor given present legislative climate in Europe and the US is one where emissions and environmental regulations are becoming ever tighter.
INTELLECTUAL PROPERTY: THE KEY TO AQUA METALS

Scepticism. For most in the battery industry that’s the default position when new technology claims to be disruptive or game-changing. And all the more so if the technology on offer isn’t completely transparent.

Although Aqua Metals has filed various patents — details below — the value of the patent very rapidly becomes worthless in third world countries where intellectual property is disrespected. And all the more so when a published patent can be reverse-engineered to produce an equivalent product or process or where the secret can be easily duplicated.

Aqua Metals has been notoriously reluctant to discuss the actual process but it has allowed prospective investors to see demonstrations of the commercial pod in action.

Rob Romero, the founder of investment firm, Connective Capital described his visit saying he was sceptical of breakthrough new processing technologies, “especially when it comes to a chemical process that is over 100 years old.

He later related: “So I hired the best independent electrochemical expert I could find: Ralph Brodd, who has served in technical committees for the Department of Energy, NASA, and Lawrence Berkeley National Lab and is past-president of the Electrochemical Society.

Needless to say, he was sceptical too, having seen lots of inventions come and go, trying to purify lead without smelting.

“To allow us to see the commercial-scale production pod, the company required both of us to sign a Non-disclosure Agreement (NDA), so I’m limited on what I can say. We went to Oakland, and got a demonstration of the commercial-size pod operating at full tilt.

“We were surprised,” says Romero. “One look at Ralph told me what we needed to know. We were not only impressed by the ease by which the machine pulled out highly purified lead from the aqueous solution, also how knowledgeable and forthright the CEO Steve Clarke was with us in explaining details about their process.”

Romero said that typically he looked at three criteria when assessing new technology start-ups as potential investments.

“First,” he told Batteries International, “is the simple one of looking at the technology — does it work and can that be demonstrated as such? Then of course is it scalable? Technology that works in the lab or in a batch process doesn’t necessarily translate into something that will work on the production line. So you look at the product engineering. As part of this you look at the financial side of things — what are the gross margins on the technology, for example?

“Second, you look at the business model. What are the market opportunities out there? Where will the supply channels come from and where will the products be sold — and how.

“Last is the more intangible; what’s the market sentiment for the product. Even if it works in terms of the technology and business model, if it doesn’t fit the mood of investors it may well not fly. And sometimes, of course, even when the technology and the business model aren’t up to scratch investors will nevertheless support them.”

Issues of intellectual property are working their way through the system.

In November 2013, Aqua Metals filed with the US Patent and Trademark Office, a provisional patent covering multiple aspects of the AquaRefining process, including all aspects of its proprietary water-based solvent and its novel electrolyzer.

In November 2014, the provisional patent application was converted into a non-provisional patent application which was filed in accordance with the Patent Cooperation Treaty and contained 35 claims.

The claims seek patent protection for the entirety of the novel aspects of the Aqua Metals process, starting with the dissolution of the lead compounds recovered from a used LAB, the solvents used and the range of chemical compositions under which they are effective. The claims also extend to novel aspects of the electrochemical apparatus and the range of electrochemical parameters, such as electrical current, voltage and solution pH.

The claims seek patent protection for the type and composition of the electrodes used, the form and quality of the lead produced and methods of removing the lead from the electrodes.

In May 2015, the firm filed an additional non-provisional patent application with the USPTO in accordance with Patent Cooperation Treaty which contained 39 claims. These claims seek to provide additional, complementary and alternative aspects of the November 2014 filing.

It also filed an additional six provisional patent applications with the USPTO containing a total of 54 claims.

“These filings seek to extend our patent protection in our core process technology and seek patent coverage for areas including ancillary processes, electrolyte and water recovery, the form and uses of the lead produced and applications of our process to materials other than lead,” says Steve Clarke.

“We regard the protection of our technologies and intellectual property rights as an important element of our business operations and crucial to our success. We rely primarily on a combination of patent laws, trade secrets, confidentiality procedures and contractual provisions to protect our proprietary technology.”
expected and unexpected outcomes,” he said.

“Distribution networks around the world would reform in different fashions, controversial practices such as expensive shipping of lead to less environmentally strict countries would cease. This could also encourage indirectly a better media perception of lead — and who knows where that would fly?” He was uncertain, however, that a cheaper recycling cost would translate into cheaper prices for the metal. “Theoretically this could happen but there’s too many other variables to consider.”

Other business aspects, particularly the most basic issue of supply and demand — the supply of old batteries to be recycled and demand for the refined product — have been tackled.

Cotton says Aqua Metals has already established an agreement with Battery Systems, a distribution specialist in the western US with a 200,000 square foot battery storage facility literally next door to Aqua Metals’ new Nevada plant.

This agreement will provide up to 100% of the used acid lead acid batteries for feedstock as well as offtake of recycled product for a conversion fee with a provision to convert to a merchant model in the future.

The company says it is also actively working with a diverse supply chain of used lead acid batteries including from large enterprises who are increasingly conscious of where and how their batteries are recycled.

Aqua Metals says it has identified further potential locations across the US. In the company’s securities filing document known as S1, the firm said: “we have the potential to locate multiple smaller facilities closer to the source of used lead acid batteries. If this ‘distributed recycling’ approach proves to be possible, we believe it will further enhance the economics of AquaRefining over smelting by reducing transport costs and supply chain bottlenecks.”

Wirtz Manufacturing, an early minority shareholder in Aqua Metals, is providing all of the equipment for the Aqua Metals turnkey facility with the exception of the Aqua Metals proprietary equipment.

John O. Wirtz, president of Wirtz Manufacturing Company, said; “The Aqua Metals technology has the capability to change the global lead acid battery recycling industry. It offers a green solution for recycling lead acid batteries which is a game changer by itself.

“But it also offers a lower operating cost structure, and a lower recycling volume requirement allowing all battery manufacturers to control the availability and cost of their lead. Every battery manufacturer should consider the Aqua Metals technology in their long term strategy.”

The turnkey aspect to the product is vital to the way that Aqua Metals could expand if it decides to develop with a franchise business model.

Although the final product is modular — meaning that large recyclers of used lead batteries simply have to buy extra modules to accommodate demand — the likelihood is that smaller battery makers, especially in the developing world, will be early buyers of the system.

“Consolidation in the lead supply market will make it increasingly attractive for small and medium sized lead acid battery manufacturers to buy their own battery recycling plant to preserve their supply of lead. This will require equipment that can be provided at a small scale (20 tonnes to 40 tonnes per day) and with low to minimal environmental impact,” said one analyst.

Aqua Metals says it is also hoping to qualify the AquaRefining modules and facility for future ISO14000 environmental management standards certification.