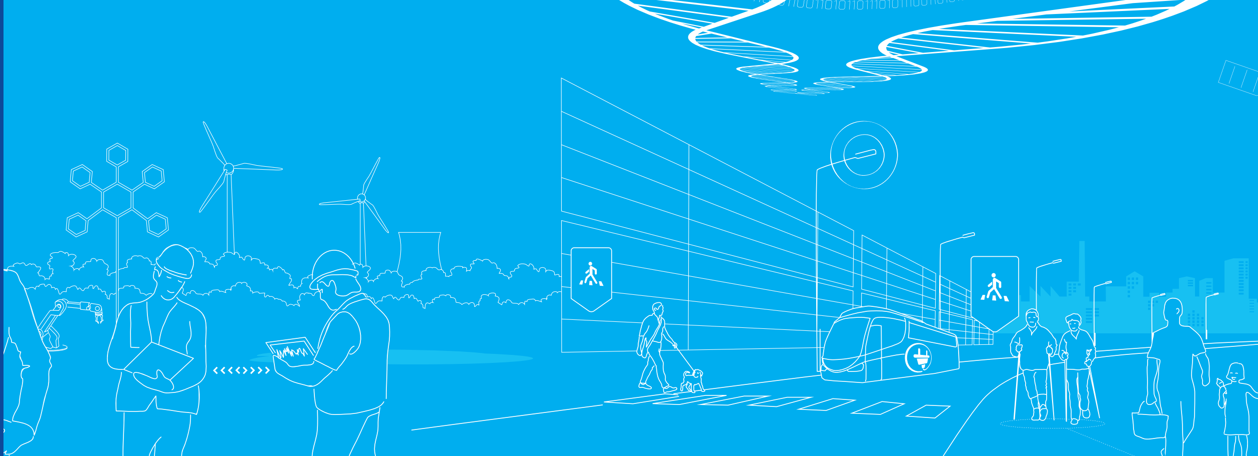
VTT RESEARCH HIGHLIGHTS 13



RESEARCH HIGHLIGHTS
13
TECHNOLOGY • SCIENCE • VISIONS

Added value from responsible use of raw materials

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VTT

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Forewords

Raw materials are an essential component of our quality of life, economy, and the well-being of modern society. In coming decades, the need for raw materials, water, food and energy will double due to the increasing population. Furthermore, the global distribution of mineral raw material resources is uneven, which leads to heavy dependence of both companies and nations on reliable access to raw materials.

In the European Union, secure and reliable access to raw materials is considered to be one of the most critical issues in the future. Raw materials are linked directly or indirectly to almost all industrial sectors, sustaining around 30 million European jobs. In order to ensure sustainable and reliable access to raw materials, the European Commission has addressed this challenge by launching several actions, e.g. the Europe 2020 strategy flagship initiative for a resource efficient Europe: Closing the loop – An EU action plan for the Circular Economy and resource efficiency. In addition, the European Commission has created a list of those critical raw materials and rare earth elements that carry economic importance and availability risk.

Finland's national wealth is based on natural resources, in which mineral raw materials have an essential role. The role of minerals is strong throughout the value chain from R&D and expertise in industrial product development to processing and services. Altogether, the annual turnover of the Finnish mineral, metal and machinery sector represents more than 35 billion € and a 15% share of all Finnish exports. However, currently 80% of the raw materials needed for industrial purposes are imported. Thus, future emphasis should be on the better exploitation of our own primary and secondary mineral resources, by generation of added value products and service innovations, and by creation of techno-economically feasible recycling processes and substitution concepts. In other words, Finland should turn the challenge of raw material dependence into a competitive advantage.

VTT's spearhead programme Mineral Economy targets to a profitable circular economy by introducing technology based solutions. The aim is to enable the generation of innovations leading to economic growth, jobs and societal well-being in Finland and in Europe.

VTT's Mineral Economy program supports circular thinking, aiming for circular economy concepts and sustainable solutions with smart raw material solutions. We have actively developed multi-technological competences to enable new innovations "outside the box", e.g. in hydrometallurgy and powder metallurgy, in our spearhead program. In order to target secure and sustainable access to resources, we promote sustainable design for closing the loop with material-efficient and low energy solutions (access to a secondary loop).

Our topics include valuable element recovery, current residue utilization, the mind-set of seeing waste as a resource, critical raw materials substitution, 3D manufacturing for narrowing the loop, remanufacturing, reuse for slowing the loop and recycling for closing the loop. Our ambitious vision is to develop the digital circular economy Modelling Factory platform for a metals ecosystem covering multidisciplinary expertise.

This publication introduces selected research highlights from VTT's Mineral Economy spearhead programme in order to disseminate our research results and to introduce new viewpoints and opportunities for the stakeholders in Finland and in Europe. Our experts cooperate hands-on in the raw material field in Finland and in Europe, for example in the EIP Raw Materials, PROMETIA (Mineral processing and extractive metallurgy for mining and recycling innovation association), and the European Union EIT Raw Materials knowledge and innovation community networks.

Anne-Christine Ritschkoff
Executive Vice President, Strategic Research

Päivi Kivikytö-Reponen
Program Manager

RESPONSIBLE AND SMART PRIMARY PRODUCTION

Solutions for responsible and intelligent mining

On a global level, the total consumption of mineral materials is expected to increase due to population growth, improved living standards, and increased urbanisation. This may lead to limited availability of certain raw materials, even if the transfer to circular economy and reduced per capita materials consumption is successful. However, due to the volatility and cyclic development typical for the minerals commodity market, it is difficult to predict future demand for mineral materials.

VTT's research on mining has been focused on selected key challenges and development needs of the sector. One of these is to reduce raw material availability risks by developing technologies for currently non-exploited mineral resources, such as lower grade and deep deposits, old waste areas and deposits in demanding environments. Potential solutions include innovative technologies for recovery of metals, but also improvement of the efficiency of mining activities throughout the chain by automation and optimisation. Automatic or semi-automatic transport systems and machines, simulation-based design and optimisation of underground mining can be cited as examples

Another important challenge relates to environmentally and socially acceptable mining. Technologies and tools are needed for efficient management of water, water purification and reduction of water use by increased recycling and reuse of mine waters. VTT has focused especially on the development of technological solutions for the removal of sulphates and management of acid mine drainage, as well as on the development of management tools, such as water footprint and process modelling. One important aspect is also the prevention of deterioration of water quality by safe disposal of tailings and waste rocks.

The development of technical solutions, tools and concepts for sustainable mining in the Mineral Economy program has created a good basis for future work. Our target is to develop solutions for sustainable and intelligent mining, meeting the needs of the changing operational environment. The specific focus areas are efficient recovery of metals from low-grade materials, mining in challenging environments, improvement of efficiency and safety of mining by automation, smart control technologies and simulation, mine water purification and reuse as well as safe utilisation of mining waste.

Water recycling in mining operations

Mona Arnold & Hanna Kyllönen

Introduction

An average mine uses 0.4 to 1.0 m^3 of water for every ton of ore processed (Gunson et al. 2012). The water volumes discharged from the process represent a significant environmental and economic burden for the mines. Given the limited availability of water in many countries, water reuse concepts are increasingly interesting strategies. However, good quality water is needed in most hydrometallurgical processes, such as in flotation (Muzenda 2010). Several physicochemical and biochemical technologies are available for purifying mine water to a sufficient level for reuse, but the challenge remains to develop economic technologies with minimal generation of rejects or sludge.

Flotation is generally one of the biggest water consuming unit operations in the mining and mineral processing chain. Recycling of water within the flotation can thus be advantageous with regard to the overall water balance, supporting reduced fresh water input to the process and decreased discharge and reagent consumption.

Concepts for water recirculation

Together with Finnish technology providers, VTT developed concepts for recirculating flotation water back to the same process using membrane filtration as the core treatment method. The reuse of water involves treating water from the process to a quality acceptable for the intended reuse while incurring the lowest possible risk to process continuity and avoiding "over-purification", leading to surplus costs. In many cases, water reuse requires a quality much lower than that of drinking water, while remaining completely safe and adequate for the process.

Reverse osmosis (RO) is typically the lowest cost treatment method for water recycling and reuse. However, due to the high scaling potential of many mine waters, the performance of this

Table 1. Elementary composition of the feed water studied.

S mg/l	Ca mg/l	Mg mg/l	N mg/l	K mg/l	Cu mg/l	Mn mg/l	Zn mg/l	Fe mg/l	Al mg/l	Ni mg/l	Cr mg/l	Si mg/l	pH	C ms/cm
2200	460	710	200	35	29	25	350	350	300	0.7	<0.1	31	2.8	7.9

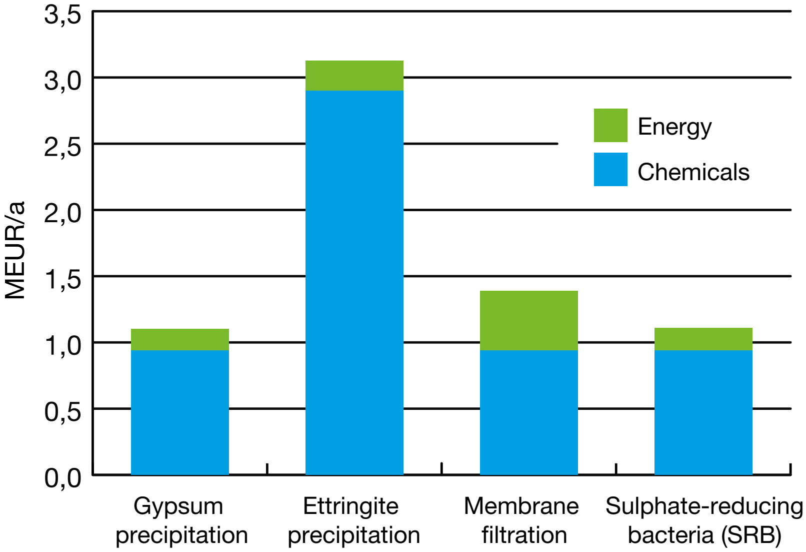

Figure 1. Flux vs water recovery for nanofiltration (NF), reverse osmosis (RO) and forward osmosis (FO) of AMD.

technology is often limited. Scaling occurs on a membrane surface when sparingly soluble salts of multivalent cations and anions, such as $CaSO_4$, $CaCO_3$, and $BaSO_4$, are concentrated beyond their solubility limit (Rahardianto et al. 2006). It is especially problematic in waters with high sulphur contents, when the sulphate level is first reduced by lime addition. Scaling can lead to significant flux reduction and salt rejection impairment, i.e. shortening of membrane life. Frequent, costly, and time-consuming cleanings are required when operating with scaling waters.

We compared innovative forward osmosis technology with low pressure nanofiltration (NF) and reverse osmosis (RO) for treating Acid Mine Drainage (AMD) (Table 1) for further reuse.

In Forward Osmosis (FO), the osmotic pressure itself is the driving force for mass transport. It is driven by a difference in solute concentrations across the membrane which allows water passage, but rejects most solute molecules or ions. The solvent will move across the membrane from the lower-concentration solute side to the higher-concentration solute side, the draw solution side. High rejection of a wide range of contaminants in FO enables the production of high quality water from wastewater. Compared to RO, less pre-treatment is required because of the lower fouling tendency due to the low hydraulic pressure applied. FO itself does not require significant energy input other than low energy pumping of the process streams. However, the “low energy and low cost” feature of FO remains debatable because a significant amount of energy may still be required to regenerate the draw solution (Akther et al. 2015). The best case would be if diluted draw solution could be used as such on-site without regeneration.

Promising results

The permeate flux in forward osmosis of AMD was good and stable (Figure 1). Permeate flux of nanofiltration at a pressure of 5.5 bar decreased dramatically, with only 20% water recovery.

The flux was more stable with a lower pressure of 2.7 bar, but this resulted in a low flux. RO performed better than NF, although water recovery was still only 40%. The water recovery of FO was significantly higher (60–80%). All applications removed sulphate and metals efficiently. However, the pH of the permeates was as low as 2.6. This factor needs to be taken into account when considering further reuse or discharge.

The best membrane performance in this case was obtained with FO, using NaCl as a draw solution. However, the cost of using synthetic 1 M NaCl as a draw solution is considerable. Sea water brine can create similar osmotic pressure to that of 1 M NaCl, and thus the use of sea water as draw solution is an interesting option. When situated at close proximity to the sea, the calculated pumping cost can be as low as 0.02 EUR/h. However, the real cost depends on the distance of the site from the sea. The efficiency of the concept should be evaluated using real sea water or sea water brine as a draw solution. Another factor needing closer assessment is long term scaling. The low pH of AMD is beneficial with regard to the risk of scaling, but it does not fully eliminate the problem.

We conclude that membrane filtration is a very promising approach for mine water reuse, although more detailed studies are needed to evaluate the sustainability of the full concepts. Such concepts must include the full life cycle, with sustainable management of the water treatment residues as well. Sustainable concentrate management, treatment and disposal, is one of the most important research items in future.

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Mine water management with advanced solution models

Peter Blomberg, Risto Pajarre, Petteri Kangas & Pertti Koukkari

Introduction

The management of large amounts of water in mining and mineral processing sites remains a concern in both actively operating and closed mining areas. When the mining site with its metal- or concentrate-producing units is operational, the challenge is to find means for economical processing with maximum yields, while minimizing the environmental impact of the water usage and waste salt treatments. Mining of sulphide ores such as pyrite and chalcopyrite provides an example of activities causing great concern. When these minerals and the gangue thereof are exposed to oxidative conditions in aqueous liquids, the oxidation sequence will lead to acidic mine drainage which will potentially leach metals from the surroundings and contaminate the groundwater and soil. For safe closure of the site, the environmental control of possible drainage will be needed.

The mining industry therefore faces increasing challenges to find sustainable practices for their process and wash waters and to deal with acid drainage. Water treatment methods and technologies are becoming an essential part of any mining activity. Current multiphase process simulation tools can be used to provide improved accuracy and better economy in controlling the

Table 1. Species currently included in the mine water activity database. The term "major species" refers to ionic species with more thoroughly evaluated solubility equilibria, also suitable for simulations at higher temperature industrial processes. The minor species are the species with more tentative and limited activity data. REE refers to Rare Earth Elements.

	Cation	Anion
Major species	Na^+, K^+, Ca^{2+}, Mg^{2+}, H^+	Cl^-, SO_4^{2-}, OH^-, CO_3^{2-}
Minor species	Al^{3+} ,Fe^{3+}, Cu^{2+}, Mn^{2+}, REE^{3+}	SiO_4^{4-}

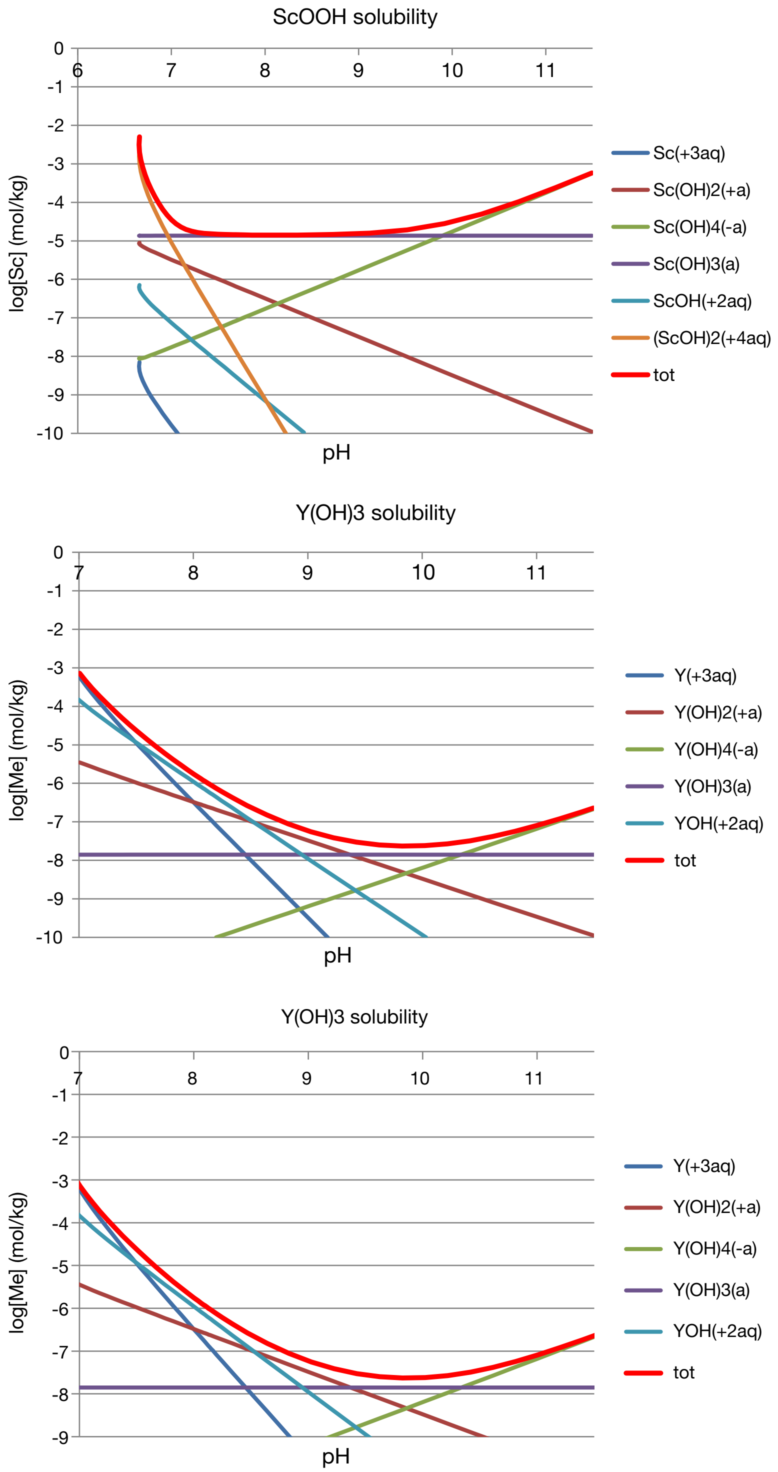

Figure 1. Calculated solubility and speciation diagrams for scandium and yttrium oxides and hydroxides.

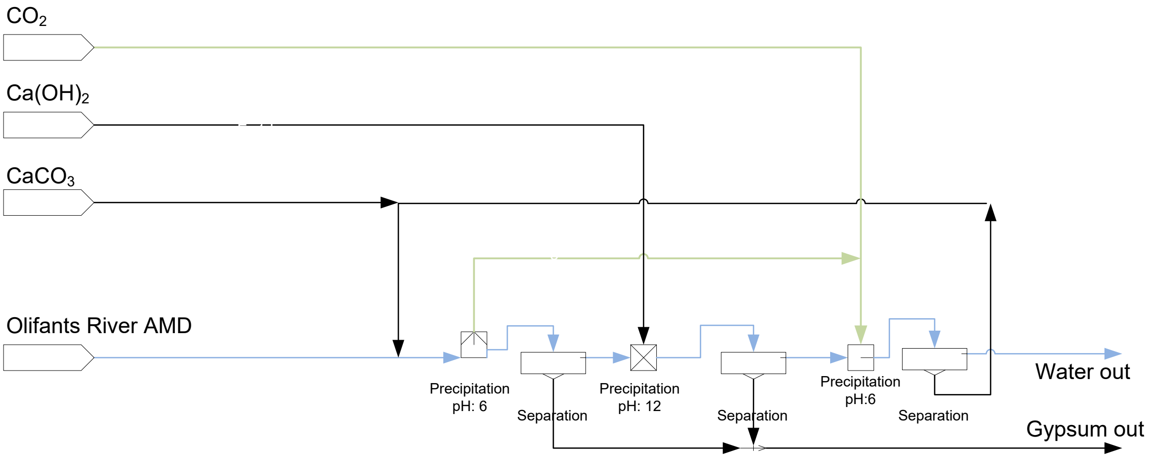

Figure 2. BALAS-ChemSheet simulation flowsheet of a simple Acid mine drainage process. For published data see Geldenhuys et al. (2003).

smooth and environmentally sound operation of the plant.

From geochemical analysis to hydrometallurgical applications

Solubility data and titration curves provide the basic tool for hydrometallurgical practice. They serve as an inexpensive and valuable method for e.g. determining the Base Neutralisation Capacity of acid mine waters and equally are used for pH-dependent precipitation-clarification processes. As a rule, the data is interpreted in terms of solubility of stoichiometric salts as well as in the form of pH - Eh dependent stability diagrams for stoichiometric compounds.

Although such predictions provide a practical tool for experimental and laboratory work, their accuracy is often insufficient for such multicomponent solutions as those encountered in mining and hydrometallurgical practice. For complex industrial solutions appearing in a variety of pH ranges and in changing temperatures, it is important to take into account the non-ideality of the solutions in order to obtain reliable results with engineering calculations (Koukkari et al. 1994). VTT has collected ionic interaction (activity) data for its aqueous databases for over 15 years (see Table 1) (Koukkari et al. 2001).

The database allows for reliable predictive simulations of the metal cations commonly occurring in aqueous sulphate, carbonate and chloride systems. Examples are presented in Figures 1 and 2. The database can also incorporate solubility data of complex salts and mineral compounds from major international geochemical databases such as CHESS, WATEQ4F, SUPCRT92, Geochemists Workbench, HSC and many others, including data for less commonly occurring cationic species.

Whereas most international research interest has focused on geochemical processes often related to environmental nuclear waste repository problems, VTT has long-term experience in developing pioneering simulation routines such as ChemSheet and BALAS to provide a quantitative tool for solving hydrometallurgical engineering problems (Salminen et al. 2015, Koukkari 2009, VTT 2007). The multicomponent reactors are connected to modular process modelling software typically used in chemical and process engineering. Thus, e.g. the use of alternative pH- and solubility-controlling chemicals as well as optional connections in a multi-stage hydrometallurgical plant or within a set of water treatment units can be simulated with fair accuracy (Kangas et al. in press).

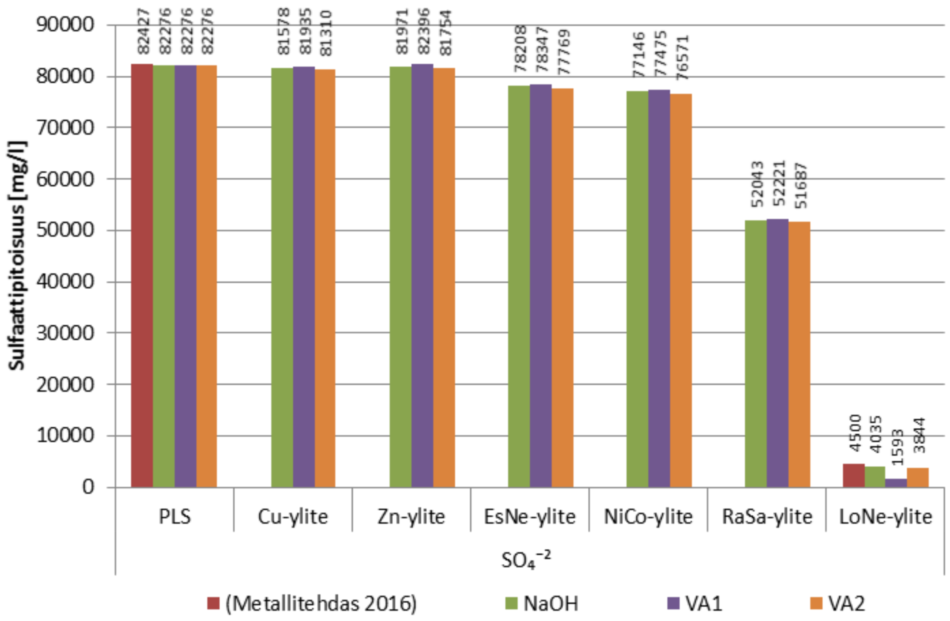

Figure 3. BALAS-ChemSheet simulation of sulphate contents in the Talvivaara mine.

From virtual laboratory to industrial practice

The multicomponent theory provides a rigorous basis for the model-based approach. All simulation results are connected either to laboratory or industrial practice. In all cases, the data used in the simulation must be developed until predictive accuracy is reached in order to ensure efficient usage of the software. The models then provide a rapid and inexpensive tool for both troubleshooting and problem solving as well as for developing new economical and environmentally benign approaches for mine water management.

Figure 3 presents results of BALAS-ChemSheet simulation of sulphate contents in the Talvivaara mine. The alternatives for base neutralisation have been simulated for three optional chemical feeds and compared with measured process data. The use of caustic (NaOH) represents the prevailing practice, whereas two alternative strategies have been assessed in terms of soluble sulphate concentration at different stages of the process.

Spreadsheet interface for engineering calculations

The complex calculation is made easy in VTT's ChemSheet software, which can be used with a spreadsheet interface embedded in Microsoft Excel™. Thus, the rigorous simulation routine can be implemented into a laptop computer and used on-site. Excel allows for build-up of a graphic interface with necessary illustrations for any new user, for whom the complex model appears as just another new Excel file. More advanced Excel users may benefit from the extensive features for handling numeric data that are available in MS Office tools.

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Choosing technologies for sulphate removal from mining effluents

Mona Arnold

Introduction

The treatment of mine water discharge has generally focused on the removal of (heavy) metals. However, regulatory agencies are becoming increasingly concerned over elevated sulphate concentrations in effluents, with discharge limits sometimes as low as 10 mg/l, but typically between 250 and 1 000 mg/l. The rationale for imposing sulphate discharge limits ranges from more generally accepted aspects of salinity contributions from high concentrations of sulphate to more controversial aspects such as proposed chronic aquatic toxicity (Arnold et al. 2016).

Alternative treatment processes for removal of sulphate from mining effluents include chemical treatment, membranes, ion exchange and biological sulphate removal. Mine location, climate, water characteristics, available utilities, footprint, and disposal areas all preclude a “one size fits all” solution. Currently the most widely used method for mine water treatment is lime precipitation, in which acidity is neutralized and metals are precipitated as hydroxides by adding lime or limestone. Although this process successfully removes the metals and increases the pH to neutral levels, the resulting effluents contain high levels of sulphate (1 500–2 000 mg/l), well above many permittable discharge levels or criteria for water reuse.

Approach

VTT compared the technical and economic feasibility of different technologies for the treatment of sulphate-containing mine waters from a mine in northern Finland. Sulphate precipitation as gypsum was chosen as the first step, followed by ettringite precipitation, biological sulphate reduction and membrane treatment as alternatives for sulphate and halide removal. The discharge from the tailings pond contained on average 8–9 g/l sulphate but negligible amounts of metals. The aim was to significantly reduce the sulphate concentration for compliance to stricter discharge limits in an economically feasible way, and to assess options for recycling the water back to the process.

In addition to practical tests, process models for each technique were created using HSC-Sim process modelling software. The calculations were made for 200 m^3/h tailings pond water feed. The reagent and energy consumption values were calculated using information from the process model.

Results

Gypsum precipitation decreased the sulphate concentration from 8–10 g/l to ca 1.4 g/l. Sodium and potassium have a significant effect on sulphate solubility, and an increase in these elements can lead to exceeding the often imposed 2 000 mg/l sulphate discharge limit. With ettringite precipitation, 700 mg/l SO_4^{2-} was achieved. However, this technology is less mature and the lack of full scale references induces uncertainty in the evaluation of its robustness and long term performance. The lowest sulphate concentration obtained with the biological sulphate treatment after gypsum precipitation pre-treatment (input 1 400 mg/l) was on average 350 mg/l. However, the sulphate concentration in the effluent varied significantly during the 47–70 d reactor experiments. Chloride or other halides were not removed with any of the above-mentioned technologies, and thus the treated water is not suitable for recycling back to the hydrometallurgical processes at the mine site. By contrast, a very pure solution with only 20 mg/l sulphate concentration in the permeate was produced using reverse osmosis after the gypsum precipitation pretreatment. In addition, the chloride concentration was around 2 mg/l and the concentrations of other halides and nitrogen were also very low in the permeate. Such a solution is suitable for water discharge, and it can also be recycled back to processes in the plant.

The water recovery of the RO process was approximately 60% and flux approximately 30 l/m2h, which are generally relatively good values for RO operations. The disadvantage of the RO treatment is the production of a concentrate containing high levels of sulphate, chloride, sodium, magnesium and nitrogen. Consequently, RO must be complemented with concentrate treatment. The concentrate could be potentially led to the gypsum or ettringite precipitation, recycled back to the existing neutralization process or used in the pasta cementation.

Sulphate removal to below 1 000 mg/l was successful with the addition of sodium aluminate to the lime-treated water. When the sulphate concentration of feed solution is high, as was the case in this study, gypsum precipitation should be carried out before ettringite precipitation in order to minimise the operational costs of the process. Operational costs of ettringite precipitation were relatively high compared to the costs of the other studied treatment alternatives because of aluminium consumption (Figure 1).

Biological sulphate reduction rates of 1 250 g/m^{3}d and 1 450 g/m^{3}d achieved in two fluidized-bed reactors were at a similar level as in several previous biological sulphate reduction studies, although not reaching as high sulphate reduction rates as those reported by Kaksonen (2004) and Liamleam and Annachhatre (2007), who performed their experiments at higher temperature. Although ethanol is considered to be a practical electron donor for commercial Sulphate-reducing bacteria (SRB) applications, a drawback is the rather low growth rate of SRB on ethanol.

Discussion

It should be noted that temperature has a high impact on various sulphate treatment technologies, and all experiments in this study were performed at room temperature. Temperature is a significant factor to take into account when sulphate removal technologies are to be operated in arctic conditions. In the studies of Isaksen and Jorgensen (1996), the biological sulphate reduction activity of permanently cold sediment samples was only 4–10% of maximal activity at 0 °C, and 10–29% of maximal activity at 5 °C. Temperature also has an effect on the membrane operation, as decreasing temperature increases the viscosity of the feed solution. Thus, the increase of temperature increases reverse osmosis efficiency and decreases pumping costs. Finally, temperature also affects gypsum solubility, which increases with increasing temperature until 40 °C and decreases thereafter. If enough excess heat is available, one alternative for the optimization of sulphate removal technologies is heating the waste water prior to the treatment.

Chemical, physical and biological processes can be successfully utilized for sulphate removal from mine waste waters. Sulphate concentrations

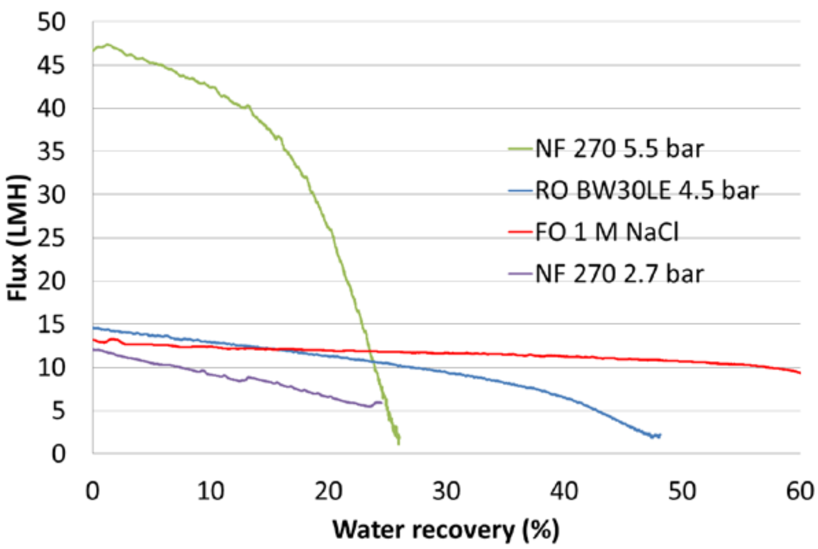

Figure 1. The consumption of reagents and energy in selected process alternatives for 200 m³/h feed.

of approximately 1 400 mg/l, 700 mg/l, 350 mg/l and 20 mg/l were obtained using gypsum precipitation, ettringite precipitation, biological sulphate treatment and reverse osmosis after gypsum pretreatment for the treatment of sulphate-rich (approximately 8 g/l SO_4^{2-}) mine waste water, respectively.

Each removal technology has its advantages, challenges and limitations related to e.g. the resulting sulphate concentrations and removal efficiencies, halide removal, retention time, operating costs and generated waste. When low sulphate levels need to be reached, gypsum precipitation can be used as the pretreatment method in combination with other sulphate removal technologies. The possibility to utilize cheaper electron donors in biological processes and aluminium from secondary sources in ettringite precipitation would significantly affect the costs of these technologies. In addition, the waste disposal costs or alternatively the possibility to produce a sellable end product have a significant effect on the overall costs of the selected process.

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Water footprint as an environmental communication tool for mines

Helena Wessman-Jääskeläinen & Elina Saarivuori

Introduction

In terms of environmental impact, water is critical for any mining project. Changes in the quality of water not only have an impact on wellbeing, health and the quality of life, but they also have ecological, cultural and economic consequences. For many reasons, Finland is particularly vulnerable in terms of water use and quality. The heavy rainfall and melting of snow in the spring lead to excess water at the mining sites, making water management especially challenging. Moreover, the winter ice cover in the Nordic conditions can cause oxygen depletion in aquatic systems, inhibiting degradation of pollutants. Therefore, water management must be individually considered at each individual mining location.

Water footprint concept

Water footprint is an environmental indicator and communication tool for the industry to reveal the local/regional environmental impacts of both water use and water quality change. It is a standardized (ISO 14046) indicator based on LCA thinking and takes into account the whole product supply chain. Water footprint can be calculated for a product, process or organisation and is globally used in different types of water intensive industries, such as the forest, energy and food industries.

Application of the concept for mining

VTT developed the concept of water footprint assessment for the Finnish mining industry in the national Green Mining project "Sustainable Acceptable Mining" (SAM, 2013–2015), which had a broad approach to sustainability by taking environmental, social and economic issues into account. The water footprint concept was based on ISO 14046 and the existing impact assessment methodologies. Data collection and calculation procedures were developed for two different mines, namely an underground copper mine (Pyhäsalmi Mine, Figure 1) and an open-pit gold mine (Kittilä Mine) and their respective supply chains. Functional units were Cu anode and Au oz, respectively.

Water footprint was found to be an applicable tool for mines and mine products. The readiness of mines to provide the needed data was good. Secondary data was needed for most of the supply chain. Water footprint highlights the environmental hotspots and indicates the impacts of different actors in the value chain from raw material to the product. In addition, water footprint provides a scientific basis and a framework to assess water efficiency, which describes the ratio between the economic value and the environmental impact of specific actions. The concept of water footprint aims to manage sustainable water utilization and its impacts, but it is also useful in benchmarking new technologies and developing water friendly branding.

Useful tool for water intensive companies

Water footprint can be a useful tool for water intensive companies in general, since it improves understanding of water flows and water use impacts of the product chains, as well as identifying opportunities for improvements. Water footprint reveals the reliance on indirect water. It may strengthen the sustainability strategy of a company by building resilience to water risks and by increasing understanding of the risk factors that arise from operating in different regions. Water footprint can be used as a decision making tool in prioritising water efficiency and guiding infrastructure investments. It is a standardised tool, which means that it can be used as such for environmental communication in company reporting.

Further reading

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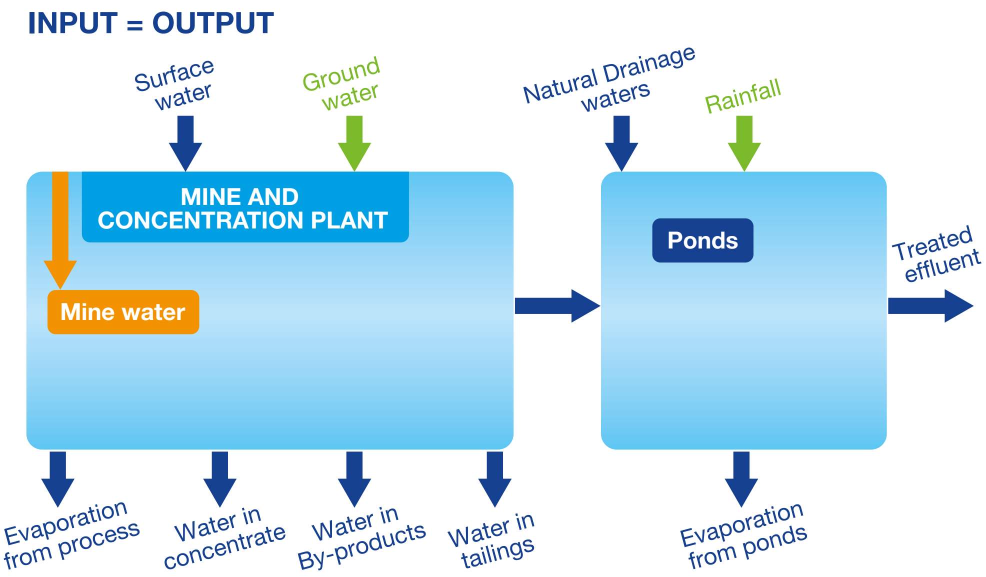

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Figure 2. The role of water footprint in environmental communication.

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On-line measurements in the mining industry

Janne Paaso & Katariina Rahkamaa-Tolonen

In the mining industry, the quality and extent of an ore body is determined on the basis of routine assays conducted on drill core and chip samples, combined with geophysical observations. Both the chemical and mineralogical compositions are important in the characterization of an ore body for commercial use. Ideally, if these data are available at the start of the mining process, they may be used for mine optimization and product control.

Mostly the analytical data are produced off-line, with a significant time lag behind production. However, the mineralogical control of valuable elements is essential in order to optimize mining and mineral processing and to avoid losses. The need for quantitative analysis is increasing, since the ores are becoming poorer and more complex in their mineralogy and content of valuables. Real time in situ analysis remains a largely unrealised opportunity in the industry (Death et al. 2009).

Since different minerals have certain chemical compositions and crystal structures, they also have characteristic spectral features which are influenced by these parameters. Determining the reflection, transmission, absorption and emission features of electromagnetic waves in minerals produces spectral images which can be translated to mineral mapping (White 2006, Zhang & Liu 2012).

Diagnostic spectral features of minerals and rocks over the electromagnetic spectrum can thus help identify the mineral composition of the ore body and host rocks. Hyperspectroscopy techniques may even be used for spatial, grain size and textural mapping of minerals, which is essential information in exploration, mine planning, etc. Many of the critical minerals have specific visible-infrared range features, which make hyperspectral imaging suitable for their rapid identification and mapping, and thus applicable for the above mentioned objectives.

The hyperspectral imaging method is widely used in geological applications in the visible–near infrared (VNIR) and shortwave infrared (SWIR) spectral ranges. Quite recently, convenient hyperspectral cameras in the midwave infrared (MWIR) and longwave infrared (LWIR) spectral ranges have become available (Holma et al. 2011). This opens up new possibilities in mineral applications, since many minerals have useful absorption bands only in the MWIR or LWIR spectral ranges.

Raman spectroscopy of minerals is well established and can be used for identifying and classifying minerals, but also for studying their crystal structure. However, "fluorescence is the enemy of Raman", referring to the fact that fluorescence induced during Raman spectroscopy

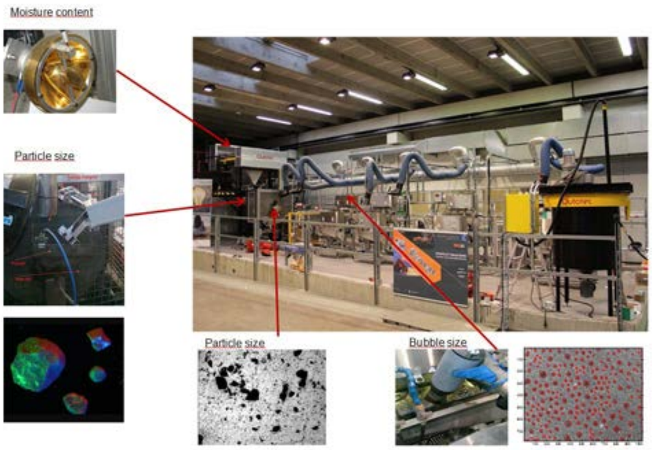

Figure 1. Oulu Mining School mini-pilot line and developed on-line measurement techniques.

inhibits or totally rules out the detection of Raman signals in some samples. An interfering feature of the fluorescence signal is the fact that the probability of Raman scattering (cross-section) is much lower than that of fluorescence (Hanlon et al. 2000, Jestel 2010). Attempts have been made by several research groups to solve this issue for decades, using different approaches. However, no widely applicable, affordable and effective solution has hitherto been found.

Raman scattering has a lifetime of much less than a picosecond, whereas fluorescence lifetimes are typically in the range of several thousand picoseconds or even nanoseconds. Thus, it is possible to suppress the fluorescence background to a great extent if short, intensive laser pulses are used to illuminate the sample rather than CW radiation, and the sample response is recorded only during these short pulses.

A novel, robust and affordable time-resolved Raman spectrometer based on a time-gated CMOS single photon avalanche detector (SPAD) and microchip-pulsed laser source was recently developed (Tenhunen & Kostamovaara 2011). In this spectrometer solution, the width of the time gate can be less than 100ps, which results in a very efficient reduction in the fluorescence background.

In order to improve the control of beneficiation processes, novel optical measurements are needed to detect process status and disturbances. The four optical measurement techniques used in the feasibility studies performed in the Oulu Mining School (OMS) mini-pilot beneficiation plant (Figure 1) include:

- on-line bubble size measurement in flotation processes,
- on-line moisture content measurement of crushed ore,
- on-line particle size measurement of crushed ore (dry) and
- off-line particle size measurement of grinded ore (wet).

State-of-the-art methods measure the bubble size of the froth on the top of the floatation cell. In this study, a technique to measure the bubble size inside the floatation cell is presented, motivated by a clear industrial need for this kind of measurement device. The applied modular imaging probe consists of an illumination unit, a viewing unit and an electronic unit.

On-line bubble size measurement inside froth in flotation processes gives a faster response to changes in the process, enabling better process control and yield. In addition, it provides better understanding of bubble behaviour in different depths of flotation cells. The performance of the technique was verified in a laboratory batch reactor and in a flotation cell at the Oulu Mining School mini-pilot line.

In the mining industry, moisture measurement is very important during mineral processing from mining to the end product. Too dry a product can result in excess dust in crushing and in handling of the final concentrates. On the other hand, too high moisture content increases transporting and handling costs, which represent a significant portion of the overall treatment costs.

The aim of NIR measurement of crushed ore studied in this project was to develop a rapid, reliable on-line moisture measurement for mineral beneficiation control. The laboratory moisture measuring set-up consists of SPECIM SWIR spectral camera and an in-house built fibre-optic probe. The NIR spectra were acquired from falling mineral crush with different moisture contents. It was observed that the water band area correlated linearly with the gravimetric method used as a reference

VTT has developed a particle size analyser for pharmaceutical processes (EyeconTM 3D particle characterizer, Innopharmalabs) for particle sizes of 50–300 µm. It uses RGB LEDs with a 1–30 µs flash pulse for imaging. The same technology was used for measuring larger particles at mineral processing plants in this study.

The particle sizes of dry, crushed ore as well as of wet, grinded ore were measured off-line and the dry ones also on-line. The wet samples were imaged with a white LED backlight in a transparent cuvette and the camera was in the opposite position. The sample was diluted with water so that the particles were distinguished from each other. The results for dry samples indicated that the particles can be recognized using a machine vision algorithm. The method for wet samples also appears to have potential as a cheaper alternative to particle sizers based on laser diffraction.

The control of a mineral beneficiation plant is difficult due to variation of the raw material and lack of on-line measurements. The proposed measurement techniques could be used to improve flotation control, reduce handling and transportation costs and to increase process knowledge.

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Why to measure

Petri Koponen

MIKES Metrology is part of VTT Technical Research Centre of Finland Ltd. VTT MIKES Metrology is specialised in measurement science and technology. As the National Metrology Institute of Finland, MIKES is responsible for the implementation and development of the national measurement standards system and for realisation of the adoption of SI units in Finland. MIKES designates other National Standards Laboratories in Finland.

Concerning different developed measurement technologies and devices etc., VTT MIKES Metrology can perform estimations and measurement uncertainty calculations. All measurements are subject to uncertainty, and a measured value is only complete if it is accompanied by a statement of the associated uncertainty. Measurement uncertainty calculations help us understand and identify the sources of uncertainty in the measurement. Furthermore, the uncertainty of a measurement reflects its quality.

In metrology, measurement uncertainty is a non-negative parameter characterizing the dispersion of the values attributed to a measured quantity. It is a quantitative indication of the quality of the result. It provides an answer to the question, how well does the result represent the value of the quantity being measured? It allows users of the result to assess its reliability, for example for the purposes of comparison of results from different sources or with reference values. Confidence in the comparability of results will help to reduce barriers to trade.

Uncertainty is a consequence of the unknown sign of random effects (such as short-term fluctuations in temperature, humidity and air pressure, or variability in the performance of the measurement device) and of the limits to corrections for systematic effects, and is therefore expressed as a quantity. It is evaluated by combining a number of different uncertainty components. The components are quantified either by evaluation of the results of several repeated measurements or by estimation based on data from records, previous measurements, specification of the equipment and experience of the measurement.

Measurement is essential to science. Without measurements, scientific models and theories cannot be rigorously tested or challenged. All measurements are subject to uncertainty, and a measured value is only complete if it is accompanied by a statement of the associated uncertainty. Without adequate attention to the implications of uncertainty, there is a risk of compromising the credibility of the whole research. When the developed model is used as a tool in decision making by the authorities involved, uncertainty estimation is the key factor defining how reliable the produced data is.

FROM SIDE STREAMS AND WASTE

TO ADDED VALUE MATERIALS

Materials processing from waste with lower energy consumption

Marjaana Karhu
Contributors: Päivi Kivikytö-Reponen, Pertti Lintunen, Juha Lagerbom & Tomi Lindroos

Industrial side streams and wastes can also be seen as unused resources. Large amounts of mining overburden, tailings and other by-products are piling up; the amount of total mineral extraction in Finland has doubled between the years 2008 and 2013 from 38.8 to 79.4 million tons (Liikamaa 2014). This is around half of the overall waste generation in Finland. The European steel industry generated about 21.8 million tons of slag in 2010 (EUROSLAG).

About 76% of the produced steel slag was recycled in several applications such as aggregates for construction or road materials, but these sectors are unable to absorb the total amount of slag generated, so the remaining 24% was still landfilled (2.8 Mtons) or self-stored (2.2 Mtons). The utilization of mining waste as a secondary raw material source has been acknowledged as an essential objective by the European Commission (2013). Therefore, any improvements in this sector will have a very high positive economic and environmental impact. Steel industry slag landfills release heavy metals to the environment (soil and water) for very long time periods, which represents a severe environmental impact.

From the environmental point of view, the assignment of waste to landfill is a significant source of pollution of air, water and soil, and further adversely affects both human health and the growth of vegetation. From the circular economy point of view the source of currently unused raw material resources may also be compromised. There is a growing need to develop solutions to generate viable technologies minimizing the impacts of the disposal of these waste streams in the environment. From the economic point of view the waste streams may also represent potential raw materials. With the help of current and novel processing technologies, it can be possible to turn side streams and wastes into secondary raw material sources and other valuable products.

Conventional processing routes for ceramic materials consume a large amount of energy and have long processing times, which increases product costs and generates a high environmental burden. Therefore it is essential to develop low energy intensity processing techniques that are less sensitive to raw material purity in order to utilization of waste as a secondary raw material source. One technological opportunity to decrease processing energy consumption is to utilize reaction heat released from exothermic (heat releasing) reactions. The lower energy consumption can then be achieved by producing heat partially in the process and utilizing the extra heat in ceramic material processing.

Development of low energy intensity processing techniques in order to exploit mine tailings as raw materials for ceramics is ongoing in VTT

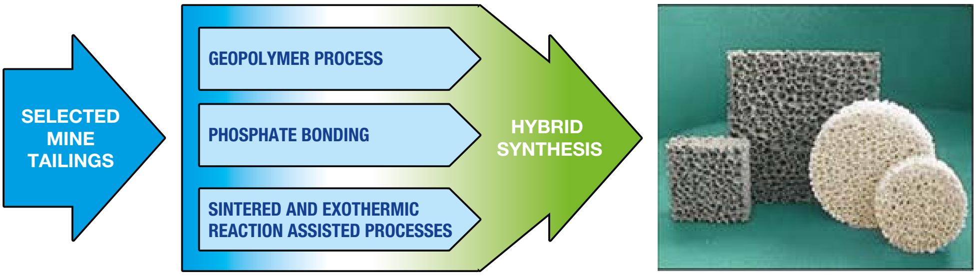

Figure 1. CeraTail project objectives.

in a project funded by the Academy of Finland, CeraTAIL (Novel synthesis methods for porous ceramics from mine tailings, 1.9.2015–31.8.2019, see Figure 1). The project has a unique cross-disciplinary consortium and research is carried out in close cooperation between the consortium partners University of Oulu, Tampere University of Technology and Geological Survey of Finland.

Chemically bonded ceramics (CBC) offers another potential low-energy intensity alternative ceramic processing route to be exploited for upgrading mine tailings into potential raw materials for ceramics. Two CBC processing methods are studied in the project: geopolymerization/alkali activation in University of Oulu and chemically bonded phosphate ceramics (CBPC) in Tampere University of Technology.

Steel industry slags have the relevant property of withstanding high temperatures, which makes them suitable raw material candidates as aggregates in the manufacturing of novel refractory materials (materials that retain their strength at high temperatures above 1000 °C). Development of a processing technology that can turn waste from the steel industry into useful feedstock for raw material for refractory materials is ongoing in VTT in the EU H2020 programme RESLAG project; see http://www.reslag.eu (Turning waste from the steel industry into valuable low cost feedstock for energy intensive industry 1.9.2015–28.2.2019, see Figure 2). The approach in the project is to maximize the amount of secondary raw materials for castable insulating refractories. Some self-funded VTT projects, e.g. Procircular (slags to

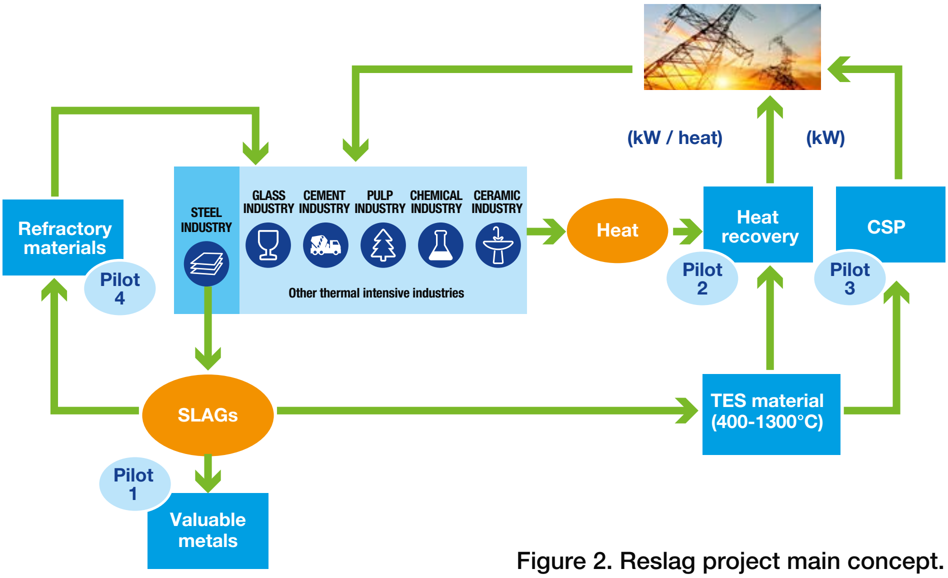

Figure 2. Reslag project main concept.

materials), HYDRO Powder (aluminium side stream utilization), and several projects funded by EIT Raw Materials have also concentrated on novel innovations based on secondary raw materials.

Industrial high added value materials are increasingly needed; in particular, porous ceramic materials for high-temperature applications are currently of great interest in the plethora of applications including absorbents, filters, light structures and supports. Castable refractory materials are needed in all thermal energy intensive industries for construction of the furnaces, kilns, incinerators or reactors, and they are also very useful for many other applications. All these materials have potential applications as secondary raw materials to substitute raw materials from primary sources.

From the points of view of unused resources and environmental considerations, exploitation of secondary raw materials would increase the material efficiency considerably and avoid the environmental footprint resulting from quarrying, thus conferring an important environmental benefit. Using low energy intensity processing routes, more economic and environmentally friendly processing towards a circular economy could be achieved.

Energy efficiency of the material processing has a significant effect on the cost structure and therefore on business models of secondary raw material utilization. By utilizing the reaction heat released from exothermic (heat releasing) reactions, processing costs can be decreased by approximately 1.3 MWh/ton compared to conventional furnace energy consumption, which decreases CO_2 emissions by as much as 40%.

In VTT, PhD thesis work is ongoing as a part of the CeraTAIL project related to the development of sustainable materials from waste and of novel sustainable processing and synthesis routes for enabling the use of secondary raw materials. This purely academic work relates to early stage technology development. First results of the potential of utilization of the heat released from exothermic reactions for external energy savings in ceramic synthesis and sintering were recently published (Karhu et al. 2016). These preliminary studies prove the potential of utilization of the heat from the reactions, but further research is needed for its full exploitation. A review article (Kinnunen et al. submitted) was also recently submitted by CeraTAIL project members concerning the utilization of mine tailings as a raw material for chemically bonded ceramics.

The technology development in RESLAG project aims towards industrial scale alternatives. Future work will relate to the demonstration of castable refractory materials from slags in pilot scale production, with the aim of enlarging the number of by-products in raw material mixtures for castable insulating refractories. Two key technological drivers are taken into account: the thermal properties, namely the thermal insulation performance that must be reached, and the mechanical strength at ambient and elevated temperatures. The work is targeting the utilisation of by-products in combustion chamber linings as well as secondary refractory layers in linings, whereas the outer layer mainly consists of primary refractories.

A potential can be seen to establish and develop the secondary raw material ecosystem alongside the current primary raw material producers, and even to establish new players and businesses within the ecosystem.

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From waste to value

Antti Pasanen

Aluminium is a widely used in modern life, and it is actually the most common metal found on Earth, totally 8% of the planet's crust. The metal is incorporated into many products, from cans, cell phones and window frames to aeroplanes. Bauxite ore is the world's primary source of aluminium, and more than 160 million metric tons of bauxite are mined each year. The steps leading from ore to metal include alumina processing and smelting. [1]

The aluminium industry produces side streams and wastes, which currently still leads to landfilling or self-storing. For every 4 kg of bauxite, 2 kg of alumina can be produced. [2] From every 2 kg of alumina, 1 kg of aluminium is produced. There have been efforts to find solutions to this loss of material starting from ore, but hitherto they have not been economically feasible. However, further processing of side streams to various materials typically uses less energy, reduces landfill waste and contributes to lower greenhouse gas emissions than starting from ore.

Waste to value demonstration

It has already been established that there are multiple possibilities for utilizing industrial aluminum and side streams e.g. for aluminium salt slag [3]. Some of the solutions have potential as primary alumina substitutes. Alumina is one of the most common ceramic materials. It has many industrial uses, especially in demanding applications, for example in abrasive, high temperature and corrosive environments. One potential solution in

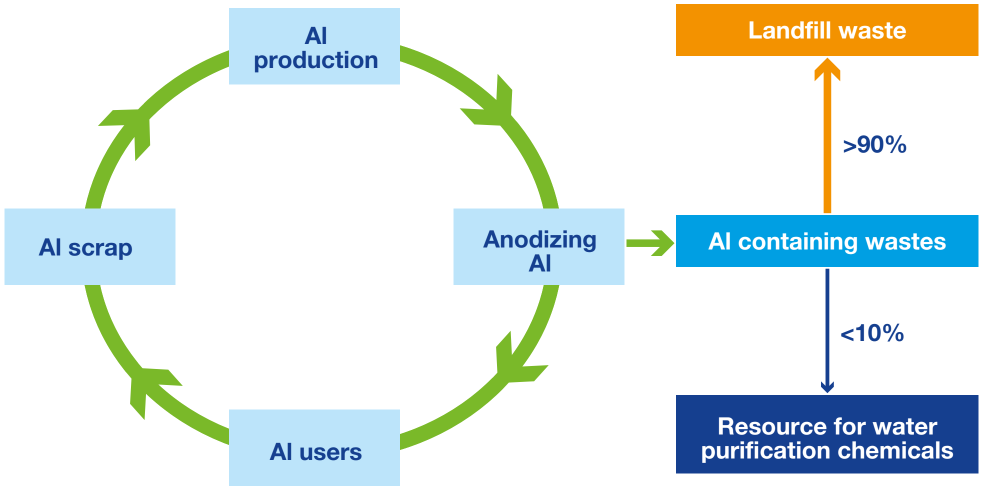

Figure 1. Aluminium anodizing side stream utilization.

our studies is substituting primary alumina (Al_2O_3) in metal-ceramic or metal-matrix composites with chemically and thermally treated alumina processed from aluminium industry wastes. The microstructure of one alumina-based composite is shown in Figure 2.

One of the aims of this research was to investigate interface modifications of alumina in order to make it more compatible with matrix material. Waste material was treated to powder form, mixed with nickel metal powder and consolidated by using hot isostatic pressing (HIP). The same method is also used for densifying components manufactured by other powder metallurgy (i.e. 3D-printed metals) or casting routes.

Possibilities for new ecosystems and new business

Landfilling wastes not only generate costs for companies, but also impart a negative aspect to the public image of the company. We believe that by modifying existing side streams into products, or raw material for another product, it is possible to add value and turn costs into a cash flow. Here one potential example was introduced of how to utilize waste material in components. Further investigations are needed into compatibilities with different matrices.

Finding new solutions to avoid landfilling of side streams will open potential for new ecosystems within the industry, or even create business potential for new SME companies. From a technical point of view new alumina-based products will interest end users seeking better and cheaper material solutions for demanding applications. The metal and ceramic industry could substitute their primary raw materials with waste-based solutions, thus using less energy and lowering greenhouse gas emissions while increasing overall efficiency.

[1] http://www.aluminum.org/industries
[2] http://www.aluminum.org/industries/production/primary-production
[3] http://www.ncbi.nlm.nih.gov/pubmed/22480708

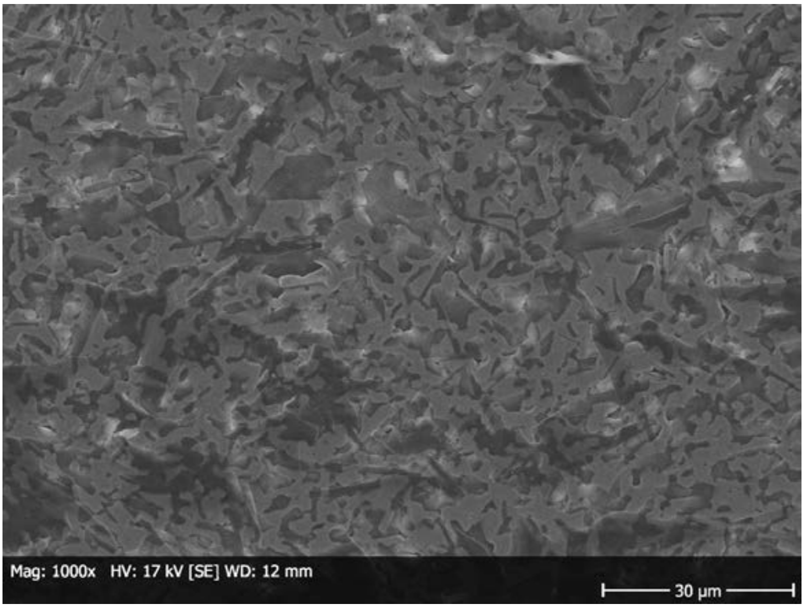

Figure 2. Scanning Electron Microscopy (SEM) image of an Al_2O_3-Ni composite, magnification 1000x.

Utilizing metallic side streams as raw material for powder-based additive manufacturing

Joni Reijonen

This study is part of an ongoing master's thesis being carried out at VTT under the Mineral Economy spearhead program for the degree of MSc in mechanical engineering from Lappeenranta University of Technology, and is expected to be ready by the end of 2016.

The goal of this thesis study is to identify the potential benefits of recycling metallic waste streams as raw material for powder-based additive manufacturing, as compared to the conventional recycling route into bulk feedstock. Experimental work is carried out to determine whether 100% scrap feedstock could be used in powder production without sacrificing the quality of parts manufactured from the powder. Gas atomization is recognized as the most potential recycling route for scrap metal, because of relatively low energy consumption and the possibility to prepare powders form various different materials and forms of solid scrap. In addition, a special case of preparing spherical powders from a specific waste stream with only mechanical milling is demonstrated. As melting is avoided, the energy consumption of such a recycling route is only a fraction of that of gas atomization.

Recently, there has been increasing concern about the sustainability of the manufacturing industry, a major consumer of energy and raw materials and therefore a major producer of greenhouse gas (GHG) emissions and waste. The manufacturing industry is estimated to account for 18.8% of GHG emissions and 11% of waste produced in the European Union. (Eurostat 2012, Eurostat 2013.)

Today, manufacturing of products from metals and alloys is typically accomplished by subtracting material from semi-finished bulk feedstock to achieve the desired shape. The manufacturing industry therefore has a significant side stream of waste material consisting of machining turnings, cuttings, stampings etc. which are referred to as new scrap.

Figure 1 shows the material flows of three different structural metals during the manufacturing phase of products. The amount of new scrap generated has been estimated to be 14.6% for steel, 13.7% for aluminium and 55% for titanium from the input material stream. Titanium is mostly used by the aerospace industry, where complex structures with low weight but high strength are needed. As the complexity of the product increases, so does the amount of scrap generated during subtractive manufacturing.

Circular economy is a concept of preserving the value of products, materials and resources

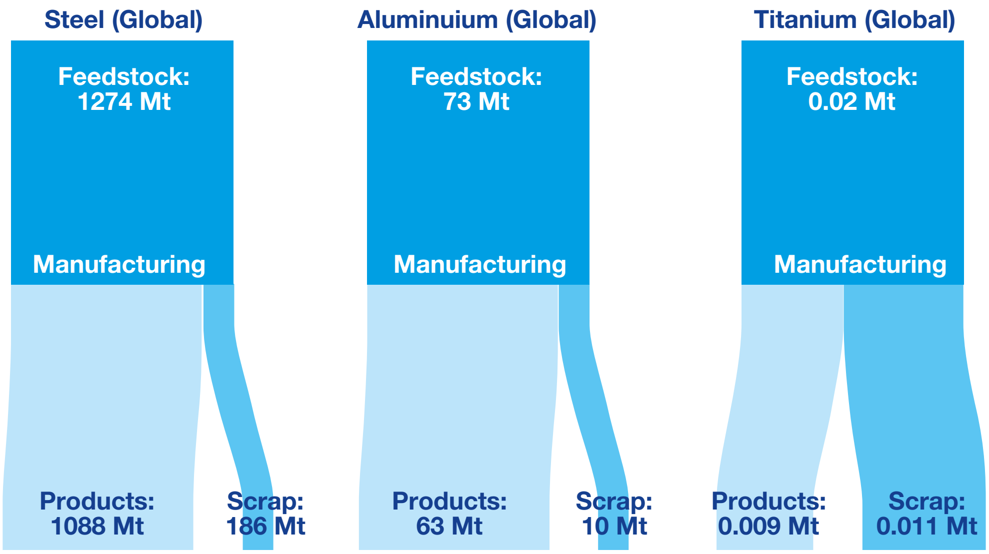

Figure 1. Material flow of steel (left), aluminium (centre) and titanium (right) in the manufacturing phase of finished metal products. Note that for steel and aluminium the data is global, whereas for titanium only national data from the U.S. was available. (Cullen et al. 2012, Stacey 2015, Goonan 2010.)

in the economy for as long as possible, thus minimizing waste generation and maximizing reuse and recycling. The primary objective in the sustainable material usage in circular economy is to prevent the formation of this new scrap. (COM(2015) 614.) Novel manufacturing technologies with less waste generation than in the conventional subtractive manufacturing are needed.

Additive manufacturing (AM) is a fundamentally different manufacturing methodology, as material is added only where it is needed, instead of removed from where it is not needed. After minimizing the scrap generation with suitable manufacturing technology choices (such as additive manufacturing), the next step is efficient recycling of the unavoidable scrap that is produced in the process. Conventional open-loop recycling via re-melting and forming into semi-finished bulky feedstock is energy intensive and results in non-functional recycling and dilution losses of alloying elements. To avoid these issues, closed-loop recycling of new scrap into powder feedstock useable in powder-based additive manufacturing is studied. Powders prepared from recycled scrap would also reduce the relatively high price of the powder feedstock used in additive manufacturing.

Powder bed fusion (PBF) is widely regarded as the most prominent additive manufacturing technology for the production of complex metallic parts. In PBF a thin layer of powder is spread on the building platform, and based on the 3DCAD-model a cross-section of the desired geometry is selectively melted by a laser or electron beam. The platform is lowered, a new layer of powder is spread and the process is repeated until a finished product emerges from the powder bed. Almost fully dense parts with mechanical properties comparable to those of wrought or cast counterparts can be produced. To achieve this, consistent spreading of dense powder layers is essential, and this requires good flowability and packing density of the powder

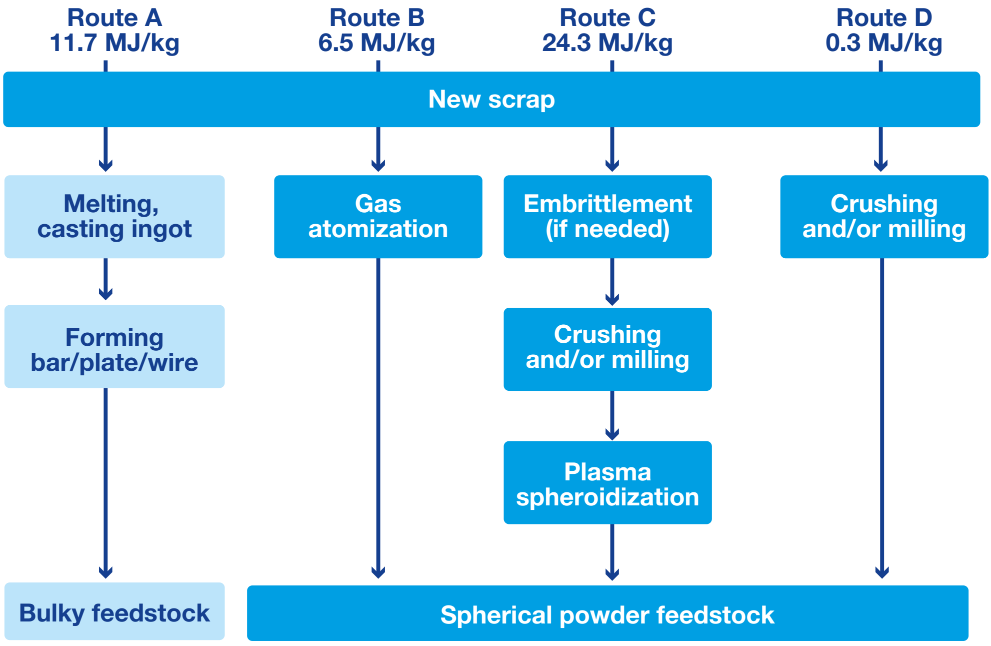

Figure 2. Processing routes for preparing spherical powder from scrap steel with the estimated specific energy consumption (SEC) values for each route. (Grimes et al. 2008, Morrow et al. 2007, Jankovic 2003, Boulous 2016.)

used as feedstock. Properties such as strict particle size distribution and spherical particle morphology promote good flow and packing density, however preparation of powder with such properties present challenges.

The dominant method of producing spherical metal powders is melt atomization. There are several different atomization techniques used to produce powders from different metals and alloys. Some require feedstock in the form of high quality bar or wire, which already have high embodied energies. Those processes that can utilize scrap metal as feedstock are limited to gas atomization and mechanical milling. Unfortunately, mechanical milling produces powder particles with irregular shapes, which are far from ideal for additive manufacturing. The milled powders can be further plasma spheroidized to enhance flow and packing properties. This technique was quite recently commercialized, but so far still suffers from rather low throughput and high energy consumption.

Figure 2 compares the different routes to produce spherical powders from scrap feedstock in terms of specific energy consumption (SEC). The conventional recycling route into bulk feedstock is also included. As can be seen, gas atomization uses less energy in recycling than the conventional route. If only mechanical milling could be used to produce spherical powders from scrap, the SEC could be significantly reduced even from that of gas atomization.

Few authors have demonstrated the production of spherical powder from scrap feedstock of a specific material following different processing routes. (Yang et al. 2013, Sun et al. 2016, Han et al. 2015.) Common for these studies is that only the powder is prepared and no actual parts or test pieces are manufactured with any powder metallurgy application of the powder. Two of the processing routes (Sun et al. 2016, Han et al. 2015) also consisted of multiple different treatments and relied on the use of chemical consumables, thus decreasing the

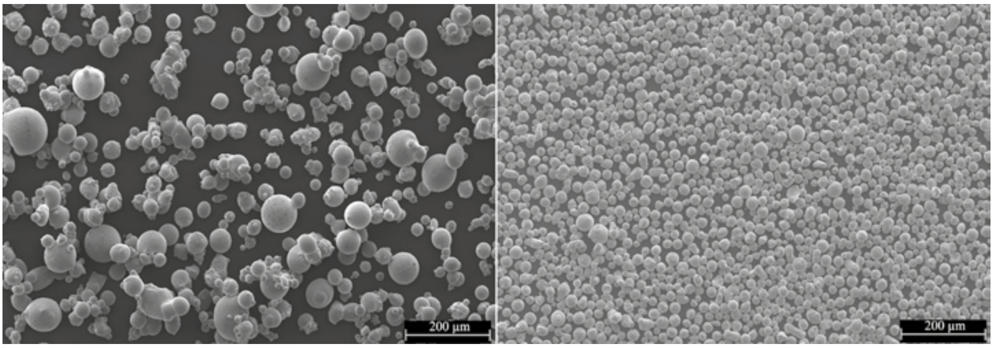

Figure 3. SEM-images of the sieve residue powder before (left) and after (right) jet milling. The material is 316L stainless steel.

feasibility of the recycling. The most promising process was the recycling of titanium machining scrap proposed by Yang et al. (2013) which is essentially the same as the proposed recycling route C in Figure 2, consisting only of hydrogen embrittlement, milling and plasma spheroidization.

The objective of the current study is to prepare spherical powder with even less processing steps than reported by previous authors and also to additively manufacture samples for mechanical testing from the powder produced. As the annual demand for AM powders is measured in hundreds of metric tonnes and the global scrap generation is in millions of metric tonnes, only those waste streams deemed most suitable for powder preparation need be selected and still the annual demand for AM powders is fully satisfied. For the ongoing experiments the waste generated in powder bed fusion in the form of support structures and agglomerated sieve residue powder was selected, as it is not subject to contamination in the manufacturing process. For the support structures routes B and C from Figure 2 are considered.

Gas atomization is a suitable recycling route for any solid form of scrap with low levels of impurities and is not limited only to support structures, which were selected here as a single representative of such forms of waste. Mechanical milling together with plasma spheroidization is mostly suited for brittle materials that can be easily milled without additional embrittlement treatments.

Currently, gas atomization appears to be the most promising way to recycle, in terms of specific energy consumption and because of the possibility to utilize various different scrap materials. Some producers of gas atomized powders already state that they use selected scrap metal along with virgin metals as raw material in their powder production. In this work, however, the attempt is to manufacture test specimens with PBF from powder prepared of 100% recycled scrap. The results are compared to parts manufactured from commercially available gas atomized powders in order to see whether there are any differences in the mechanical properties.

The agglomerated sieve residue powder is a special case of scrap as it consists of already spherical particles that are just agglomerated to each other, preventing its direct reuse in the powder bed fusion process. This waste stream accounts for around 2–5% of the initial powder material after each build. (Ford & Despeisse 2016.) Several different types of mechanical mills were tested to determine whether these agglomerates could be merely separated from each other, without destroying the spherical shape of the individual particles. Recycling the powders back to usage without melting would significantly reduce the specific energy consumption (route D in Figure 2). This also further increases the overall raw material usage efficiency of powder bed fusion close to 100% (if the parts are manufactured without support structures).

Figure 3 shows the sieve residue powder before and after air jet milling, which provided the most promising results. As can be seen, the agglomerates have effectively separated and the particles have remained almost spherical. The next steps in the ongoing experiments are to additively manufacture test specimens from the jet milled powder and to prepare gas atomized powders from 100% scrap feedstock.

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Plastic composites utilizing soap stone and gypsum

Mika Paajanen, Mikko Karttunen & Eetta Saarimäki

Various mineral side streams are produced by the mining and metal industries, many of them with limited utilization possibilities. Therefore, side streams are led to massive ponds, which is expensive because of both pond construction and monitoring costs. Moreover, these materials classified as waste may have properties that are suitable for many reuse purposes. In this example, VTT used soap stone and gypsum side streams for manufacturing plastic composites.

An example from the laboratory – Soapstone composite

Soap stone powder was mixed with polypropylene (PP random copolymer Moplen RP241H; Basell) in order to improve thermal conductivity and to achieve a good compromise with specific heat. Soap stone mass fractions of 40%, 50%, 60% and 70% were used in PP. The thermal conductivity, specific heat and tensile strength were measured and compared with the cor-

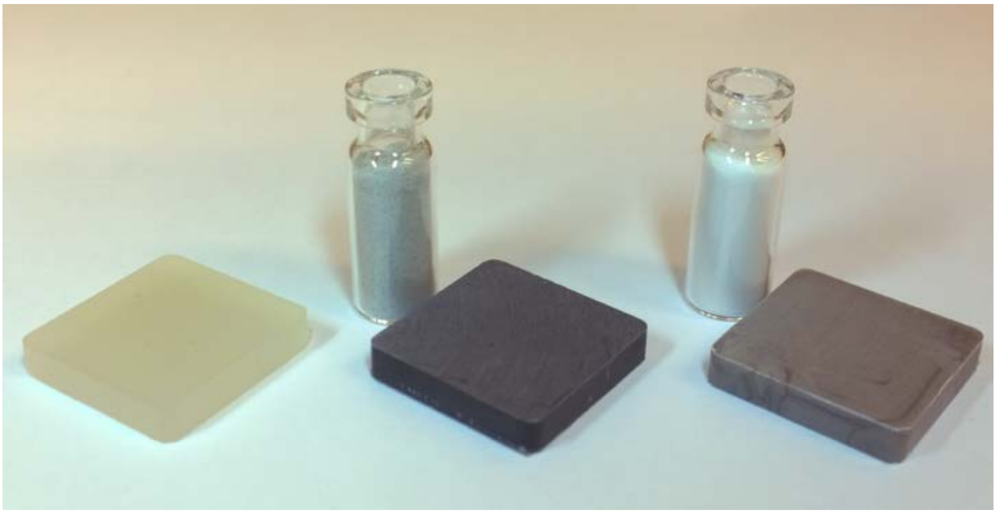


Figure 1. Polypropylene (RCPP) sample (left), 70 wt-% filled soap stone sample with soap stone powder (centre) and 70 wt-% filled talc sample with talc powder (right).

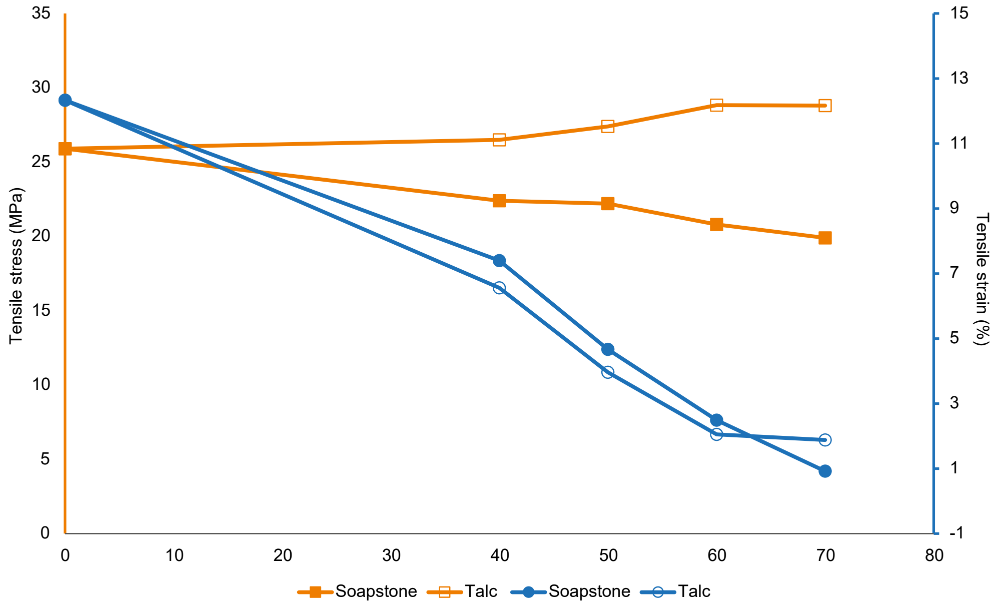


Figure 2. 70 wt-% filled soap stone and talc samples. Tensile strength and strain.

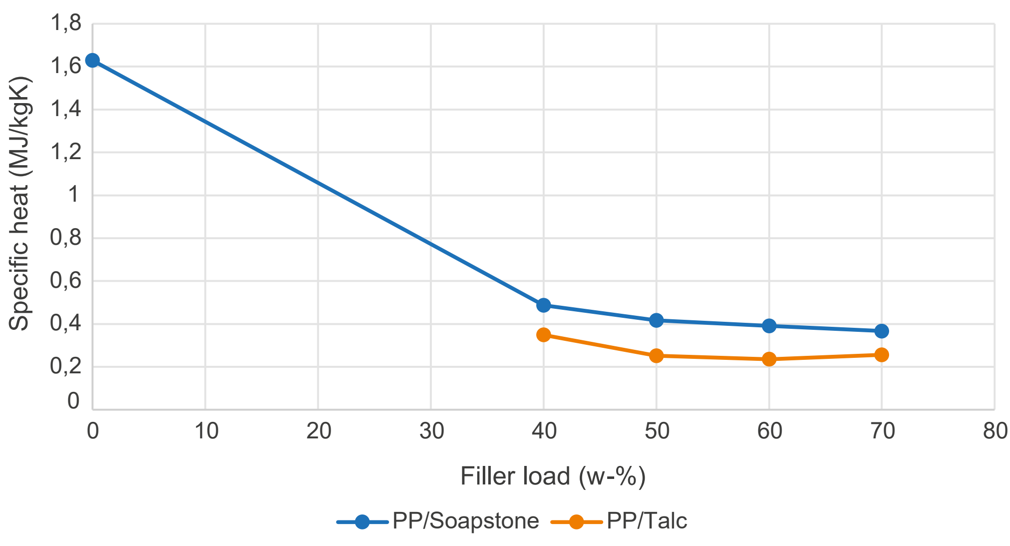


Figure 3. Specific heat as a function of filler load for soap stone and talc-filled PP samples.

responding properties of similar samples filled with talc, which is an inexpensive commercial filler. A photo of polypropylene (PP) compared to PP / soap stone and PP / talc samples is shown in Figure 1.

Tensile strain was rather similar with talc- and soap stone-filled compounds, as is shown in Figure 2. The initial results show that thermal conductivity of talc-filled PP was about 20% better at 70% filler loading, but that the specific

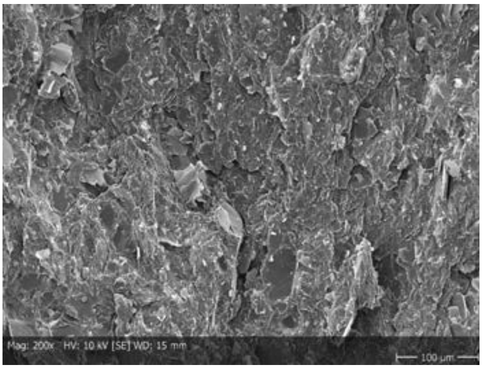


Figure 4. SEM image of RCPP sample filled with soap stone, with a 70 wt-% filler ratio.

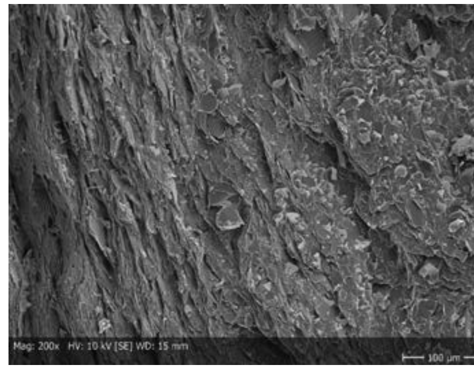


Figure 5. SEM image of RCPP sample filled with talc, with a 70 wt-% filler ration.

heat was about 40% higher with soap stone filler, as is shown in Figure 3.

As the mechanical tests show, both talc and soap stone were successfully mixed in random copolymer polypropylene (RCPP) with up to 70 wt-% filler ratio. The SEM images of the 70 wt-% filled composites are shown in Figure 4 and Figure 5.

An example from the laboratory – Gypsum composite

Gypsum releases water between 100 and 170 °C (Figure 6). The released water amounts to about 20% of the total mass. Gypsum powder was mixed with polycaprolactone (PCL), which has a low processing temperature allowing compounding and injection moulding without

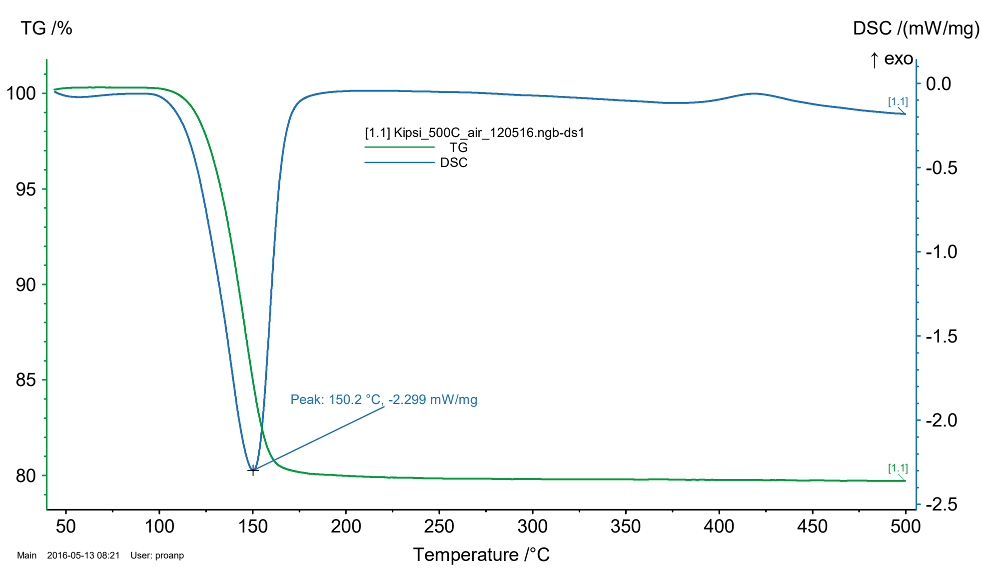


Figure 6. TGA graph of gypsum powder.

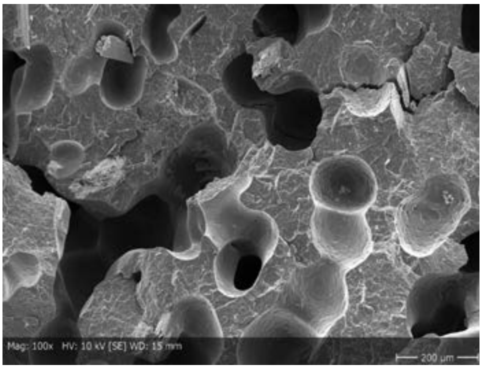


Figure 7. SEM images of gypsum-filled (10 wt-%) PP samples. Spherical shapes are part of the foam structure

releasing the water from gypsum. The gypsum-PCL composites were also produced up to 70 wt-% filler content. Foaming was achieved only with subsequent heating at 130 °C in an oven, or in a microwave oven. However, the foaming effect was weak, as the foam tended to collapse due to the soft nature of the PCL plastic at high temperature. Gypsum (10 wt-%) PP compounds were also produced, which showed expansion at the extrusion processing phase (see Figure 7). More extensive tests are recommended in the future, with polymers having suitable softening/melting temperatures for permanent foaming matching with the water release temperature of gypsum.

Conclusions

The driving force for this project was the prevailing position of Finnish companies, who produce both soap stone powder (fireplace manufacturers) and gypsum (phosphoric acid manufacturers) in considerable quantities. Utilization of these raw materials for plastic composite products offers business potential by turning waste-classified materials, which currently only generate expenses, into added-value products. Soap stone plastic composite could be used as a thermal energy storage, e.g. as heat storing/providing water pipes. Gypsum, could be used as a foaming agent for selected polymers. The work is continued to connect side stream producers with potential utilizing industries.

SUBSTITUTION AS A SOLUTION

Importance of CRM-containing applications for the European economy – Analysis of ICT and electronics, energy and transport sectors

Ulla-Maija Mroueh

Introduction

Substitution, mining, reuse, recycling and increasingly also dematerialisation can be seen as complementary actions for preventing raw material shortages. Substitution is generally a long term strategy, and as such it is mainly focused on applications which are estimated to be under threat but at the same time relevant to the industry and to society in the long term. It has also been found that the first reaction to increasing material costs due to potential supply risks is to try to minimise the content of expensive materials, and to consider other measures only if this does not solve the problem (CRM_InnoNet 2015).

The European Commission has created a list of raw materials which are important to European economy but at the same time associated with supply risks (European Commission 2014). An important point is that criticality is not only dependent on scarcity of a resource. From the industry's point of view a resource is critical if it provides unique properties and functions and therefore has few alternatives (CRM_InnoNet 2014). Therefore, criticality may also depend on local conditions and user sector. In addition, it is a dynamic concept, which is one of the challenges of substitution.

This article presents the key results of a study aiming towards identification of the relevance for the European economy of applications containing CRM (Critical Raw Materials). The data was further used for prioritisation of the applications for a substitution roadmap. The study was part of the EU FP7 project Critical Raw Materials Innovation Network (CRM_InnoNet).

Approach

The analysis was performed by examination of the CRM-related supply chains of the ICT and Electronics, Energy and Transport sectors. The focus was on studying where in the supply chains the value for Europe is produced, identifying potential CRM-related bottlenecks and providing indications of which applications could be under threat. The key analysis criteria were critical raw material (CRM) dependencies and economic and strategic relevance for Europe.

In the subsequent screening stage, 12 applications were selected for further analysis based on the use of CRMs in the application and the share of EU production of the value consumed or used in Europe:

- Energy sector: Photovoltaics (Copper-Indium-Gallium-di-Selenide [CIGS] technology), wind turbines and energy storage (Li-ion and Nickel Metal Hydride [NiMH] batteries)
- ICT and electronics sector: LED lighting, displays and screens, optical fibres, magnetic resonance imaging (MRI), washing machines; assembled printed circuit boards (PCB) and electronic components
- Transport sector: Automobiles, heavy vehicles and commercial aeroplanes.

The second stage, the actual supply chain analysis, consisted of:

- Statistical analysis of European production, import, export and jobs over each stage in the production of the chosen application.
- Analysis of criticality, strategic relevance and development of future vulnerability, based on technical and market reports as well as interviews with experts.
- Interviews of selected European companies on the current risks and risk provision strategies related to the CRMs.

European production dependent on components from outside Europe

The key results of the analysis of economic importance are summarised in Figure 1. The figure shows the structural components and materials containing CRMs in the target applications, and Europe's position in the supply chains of the applications based on production values from the Eurostat PRODCOM database. The box colours in Table 1 represent production for the whole industry, not only the share which is used in the production of a specific application. For example, for electronic circuits, each box represents the total production of electronic circuits in Europe.

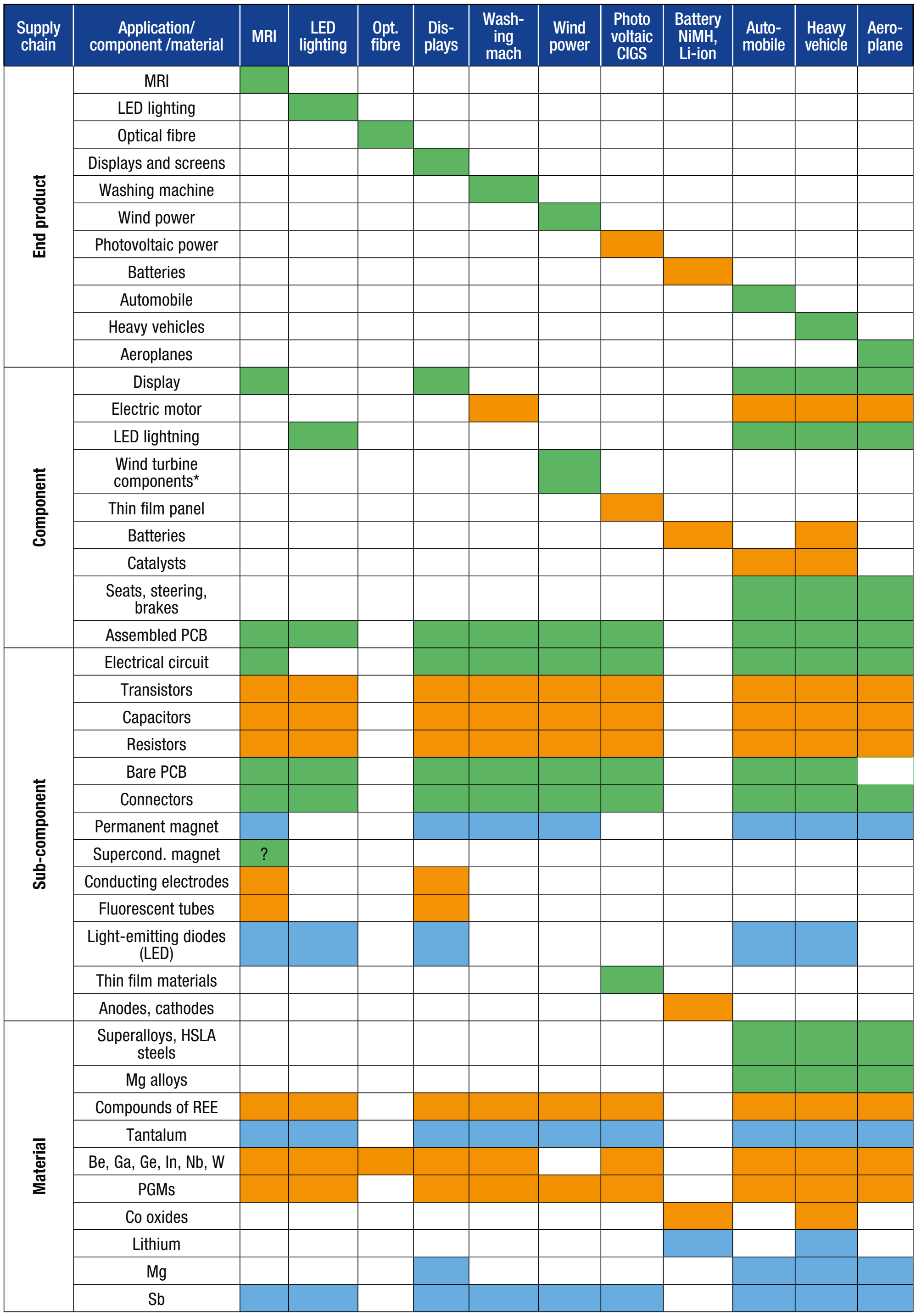
As can be seen from the figure, the EU occupies a good position in global production of most of the target applications, with one or more EU companies in the global top ten and a significant number of jobs in Europe linked to end product manufacturing. There are good possibilities for future growth, because market reports predict that the demand for almost all of the applications will increase significantly by the year 2020.

For some end products, significant component production can also be found in Europe. These products include automobiles, heavy vehicles, aeroplanes, wind energy, MRI industry and a part of electronic components of printed circuit boards. Although the presence of European companies in the component production is characteristic to the sectors where Europe is strong in the end products, most of the applications are dependent on some essential CRM-containing component(s), which are mainly produced by non-European companies. For this reason, several applications are exposed to significant CRM issues.

To mention some examples, nearly all the products are exposed to CRM issues through the use of electronic components. Dependence on permanent magnets, which are mainly produced in China and the U.S., is a potential risk for the EU wind power industry, automobile and other transport applications and to some extent also for large domestic applications. Catalytic converters and magnesium alloys manufactured outside Europe are essential for transport applications. The LED lighting sector is dependent on semiconductor dies produced abroad, and germanium scarcity may result in a bottleneck in optical fibre production. For all of these components, the substitution solutions are either in a very early phase of development or they suffer from performance problems. In mining and refining of many of the critical raw materials, Europe is totally or almost totally dependent on the outside world.

Substitution – an opportunity to Europe

The analysis showed that Europe is strong on production of the selected applications. The challenge is, however, that many of the end products are dependent on components manufactured outside Europe. On the basis of interviews and reviews made, it can be estimated that the substitution of CRMs, especially new kinds of



- Production in the EU and rather good position in global production/jobs in the EU (the sector to which the application/component/material manufacturer belongs)
- Some production in the EU, although main production outside the EU/jobs in the EU (the sector to which the application/component/material manufacturer belongs)
- Not much production in the EU /jobs in the EU (the sector to which the application/ component/material manufacturer belongs

Figure 1. A summary of Europe's position in the supply chains of the applications.

substitute solutions offering new functionalities, can in many cases be seen as an opportunity for Europe. There is active research on these sectors within Europe, although the main research focus has hitherto not been on substitute solutions. The companies involved emphasized that solutions to substitution must be looked at from a functional perspective. Instead of substituting individual materials, the approach may be to replace the application or a component containing CRMs. In most cases the whole process or system needs to be reconfigured, which makes substitution a demanding task.

As a result of this analysis together with the additional data produced in a materials-orientated analysis, it was decided to choose a component specific approach for development of substitution roadmaps. The roadmaps (CRM_InnoNet 2015) focused on the potential substitution strategies for the following component/product types:

- Printed Circuit Boards and electronic components,
- Permanent magnet-based applications such as electric drives and motors,
- Batteries and accumulators,
- High-value alloys,
- Photonics, including high-end optics.

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Replacement of antimony trioxide in flame retardant plastic composites

Outi Härkki & Eetta Saarimäki

Motivations for antimony trioxide replacement

Antimony trioxide (ATO), Sb_2O_3, is a widely used and effective synergist in PVC flame retardant (FR) systems. It is also used in thermoplastic elastomers. ATO increases the effectiveness of the flame retardant and has a smoke suppression effect. However, there are a number of reasons to look for alternatives to ATO. It is expensive (> 10 000 euro/ton), and the price is expected to rise in the future. Also there are challenges with ATO availability with China dominating the market. Moreover, in the EU, antimony is listed in the critical raw materials list, and antimony trioxide is classified as a harmful chemical, labelled by risk phrase R40 (limited evidence of carcinogenic effect) and the hazard statement H351 (suspected of causing cancer by inhalation). The use of ATO is not yet banned, but voluntary eco labels are gaining popularity for retardants containing less than 0.1% by weight of substances that are assigned an R40 phrase. Companies report that the current situation is a balancing act between costs, performance and environmental health and safety.

Figure 1. ATO-free PVC coating in a lab scale testing phase: sample preparation in a two-roll mill.

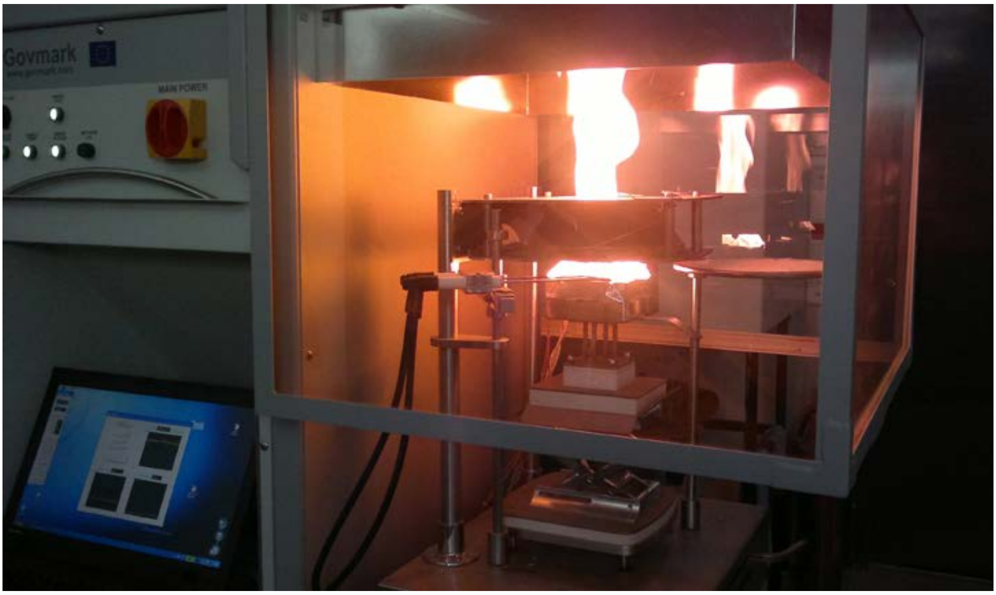

Figure 2. ATO-free PVC coating in a lab scale testing phase: cone calorimeter test.

Antimony replacement with VTT's new halogen free flame retardant

VTT has developed an ATO replacement with a selected modified mineral filler. Aluminium silicate di-hydrate (also known as kaolin), as a layered mineral filler, serves as a flame retardant synergist in PVC composites. The material and composition is safe, halogen-free and environmentally friendly. Estimated manufacturing costs are only about half those of ATO.

Aluminium silicate lowers the rate of heat release and smoke production when PVC composite material burns. Slower burning rate of the material will allow more time for people to escape from fire. A production scale trial run and a large scale fire test for building materials (SBI Test according to EN 13823:2010) have shown aluminium silicate di-hydrate to be a viable replacement for antimony trioxide in a soft PVC composite, thus giving it commercial potential in the flame retardant market. VTT has a patent application (B3099PFI, B3099PC) pending.

The option to replace an expensive and harmful flame retardant in polymer composites is interesting for PVC composite manufacturing, cables and pipes representing a major application area. The textile industry and thermoplastic elastomers used in machine parts and vehicles are also large potential application areas.

As a next step, we have also recognized the need for a halogen-free, cost competitive and effective flame retardant system in polyolefin composite manufacturing. A flame retardant formulation based on Al-silicate, a safe-to-use mineral filler, is now in development stages for the large polyolefin composite market. VTT has filed a patent application B6287PFI. VTT is looking for commercialization partners to take the technology to the market.

Novel titanium carbide based hard metal alternative for traditional WC-Co

Tomi Lindroos, Pertti Lintunen & Marjaana Karhu, VTT
Jukka Kemppainen, Exote Ltd.

Abstract

Cemented carbides are primarily used in metal cutting tools, metal forming tools (e.g. dies), construction and mining equipment where unique combination of mechanical, physical, and chemical properties are needed. The majority of cemented carbide material solution is based on tungsten carbide (WC) with cobalt (Co) binder metal. Development of novel titanium carbide (TiC) based hard metal is introduced. Properties of developed novel TiC based hard metal compositions are compared against traditional WC-Co grades. Performed tests show that mechanical properties (flexural strength, fracture toughness and hardness) of studied compositions are comparable to medium and coarse grain size WC-Co grades. The behavior of varied material compositions in crushing pin-on-disk test is also evaluated. Gained results shows that developed TiC hard metal grades are potential candidates to substitute traditional WC-Co in certain applications.

Introduction

Hard, wear-resistant materials are essential to many technological applications. Mere hardness is often not enough, but for practical purposes the materials need to reconcile partially contradictory requirements such as hardness, toughness and ductility as well as resistance to a demanding environment (corrosion, chemicals or even ionizing radiation). The material choices are further limited by cost, abundancy (e.g. critical metals), environmental regulations (e.g. carbon footprint or recyclability) and toxicity. Therefore, even within the field of wear-resistant materials, there is a need to tailor wear resistant materials for specific applications. Composite materials that consist of a super hard material (e.g. tungsten carbide, WC) incorporated in a matrix (e.g. cobalt, Co) are a popular choice.

Carbide composite materials are ubiquitous in machining tool materials, mining equipment, ballistic shielding, excavator teeth and other materials that require high resistance to wear and impact. At present, the most widely used carbide composite is WC in a Co matrix (WC-Co). Its hardness (2150 HV) stems from the inherently hard WC particles, excellent wetting properties (i.e. Co spreads over WC) and the strong adhesion of WC to the Co matrix. It has properties that are difficult to match with any other known composite.

However, the availability of raw tungsten supply and the rising price as well as environmental, energy and toxicity issues of composite matrix metals have attracted intense research into substitutive materials. Tungsten W and cobalt Co are listed as critical metals by the EU (European Commission 2014). Tungsten is considered to be the most important critical metal with a high supply risk in the EU. In addition to economic importance and the supply risk of cobalt, the International Agency for Research on Cancer (ARC) reported that cobalt and cobalt compounds may cause cancer. High hardness and ductile components and coatings are widely used in Finland and new alternatives are crucial for Finnish machinery and process industry equipment manufacturers. Europe's share of the world's primary and secondary tungsten consumption is estimated at 12 000 tonnes, 13% of the world total of 90 000 tonnes for 2011, 74% of it is imported. In 2017, tungsten production is expected to reach 115 000 t/year. 60% of tungsten is used for tungsten carbide.

One of the most promising candidates for substitution of the WC is the titanium carbide (TiC). The hardness of TiC is higher than that of WC and, by substituting WC with TiC, the CO_2 emissions could be decreased by almost 40%. The embodied energy of WC is 1342 MJ/ dm^3 and TiC 820 MJ/dm^3. The difference converts to a carbon footprint and CO_2 release of 82.8 kg/ dm3 for WC and 46.8 kg/dm^3 for TiC. Titanium is also not a critical metal; it is highly recyclable, environmentally benign and non-toxic. It is also more economic.

In this paper titanium carbide (TiC) based hard metal based on novel manufacturing route enabling formation of metastable structures leading enhanced properties is introduced. Properties of developed novel TiC based hard metal compositions are compared against traditional WC-Co grades.

Materials and testing

The studied TiC based hard metal compositions are manufactured via unique processing route developed by VTT and Exote. The process is based on reactive hot pressing enabling rapid synthesis of materials and components. The studied metal matrix composites are based on TiC hard phase in Ni matrix. The starting point for development work is Exote material named E6-55 which has nominal composition TiC-23 wt-% Ni. To enhance properties of this basic composition nanosize additives are utilized. Two new materials are introduced E6-55N and E6-53N. Material E6-55N has same nominal composition than E6-55 while material E6-53N has lower (15 wt-%) binder content.

Mechanical properties of manufactured material samples are determined with 4-point bending tests and Vickers hardness tests. Flexural strength is measured at 4-point bending test according to ASTM C1161 Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature. Fracture toughness is determined by Single Edge Notched Beam (SENB) test under 4-point loading. Vickers hardness values are measured with 10 kg load.

Abrasive wear properties of samples are studied with crushing pin-on-disc testing. Unlike in common pin-on-disc setup, in crushing pin-on-disc the pin and the disc are not in contact with each other during the test, and thus the wear is induced purely by the abrasives. In the test, the pin is pressed against the abrasive bed on the rotating disc with a force of 235 N for 5 seconds, followed by an idle time for the abrasive to replenish between the pin and the disc. The abrasive is maintained on the disc with a collar. The disc material is tool steel (690 HV). Granite is used as an abrasive. The wear is measured as mass loss, which was then converted to volume loss to enable better comparison of wear in materials with different densities. More detailed description of test arrangement is presented by Ratia et al. (2014). Microstructure of the samples is investigated by scanning electron microscopy (SEM), JEOL JSM-6400. 3D profilometer, Sensofar Plμ 2300, is used for surface investigations after abrasive wear tests.

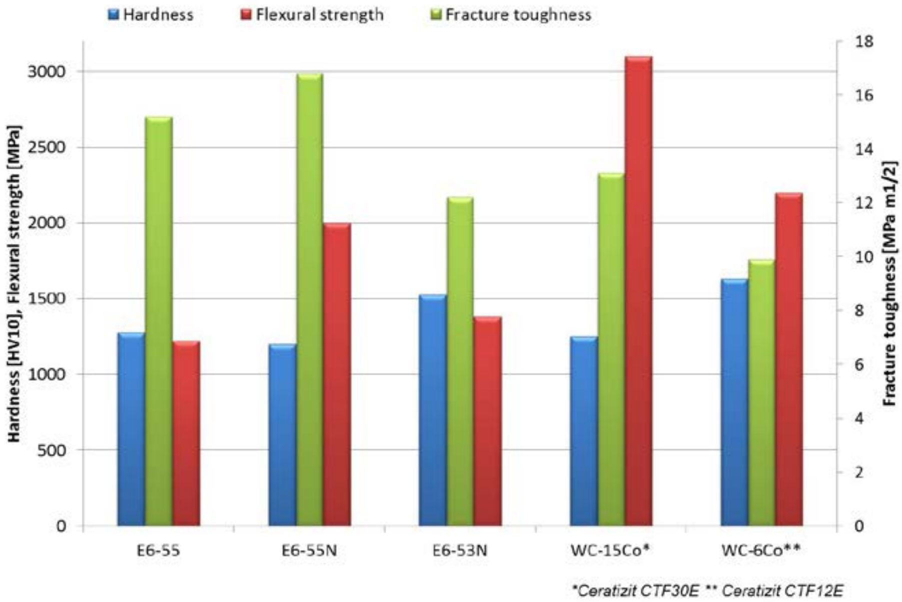

Figure 1. Mechanical properties of studied material compositions (E6-55, E6-55N and E6-53N) compared to two medium carbide size grade WC-Co composites.

Results and discussion

The results of mechanical tests are represented in Figure 1. The measured values are compared against the values presented (see Wear Parts Main Catalogue) for two commercial medium grade WC-Co materials, CTF30E and CTF12E. Properties of the basic composition E6-55 are enhanced due to nanosize additives: material composition E6-55N has 60% increase in flexural strength and 11% increase in fracture toughness values. Slight decrease of hardness from 1277 HV10 to 1201 HV10 is measured. By lowering the binder content hardness can be obviously increased. Lower binder content material, E6-53N, has 20% higher hardness and 12% higher flexural strength than basic composition E6-55. Lowering the binder content has remarkable effect on fracture toughness: 20% decrease of fracture toughness values compared to the basic composition E6-55.

The hardness values of basic composition and nano modified composition, E6-55 and E6-55N are comparable to WC-15Co grade which is a reasonable reference grade when wear resistance is evaluated. Despite of remarkable increase of flexural strength of E6-55N due to modification by nanosize additives the value is still far away from flexural strength value of WC-15Co. From this point of view it is slightly surprising that E6-55 and E6-55N both have remarkably higher fracture toughness than WC-15Co. However, it should be noted that in the case of values for reference WC-Co materials, it isn't mentioned what standards have been used in determination of properties. So there could be some deviation in values due to different measurement method. Especially the determination of fracture toughness is detected to be really challenging to achieve comparable values with even slightly different measurement methods.

When compared against WC-6Co grade the hardness value of E6-53N composition is reaching value of WC-6Co. This is due to the smaller

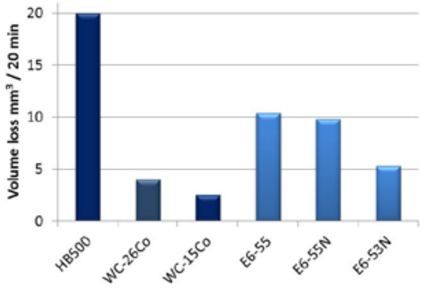

Figure 2. Volume loss of studied materials (E6-55, E6-55N and E6-53N) during 20 min test period compared to high abrasion resistant steel and medium grades WC-Co.

binder content of E6-53N compared to other developed TiC based compositions. With the same manner flexural strength value for E6-53N is lower compared to E6-55 and E6-55N having higher binder content. And respectively fracture toughness values of developed TiC compositions are higher than for WC-6Co.

The results of crushing pin-on-disc tests are presented in Figure 2 and Figure 3. In Figure 2 volume loss of sample materials are presented after 20 minutes test period. Results are compared against reference materials: martensitic quenched wear resistance 500 HB steel, medium grade WC-15Co and WC-26Co with hardness values 1260 HV10 and 870 HV10 respectively. Hardness is typically detected to dominate the abrasive wear resistance of materials which can be seen as a clear difference between volume loss values for materials E6-55N (1201 HV10) and E6-53N (1528 HV10). Despite of the fact that materials E6-55 (1277 HV10) and E6-55N have hardness values at same level than for WC-15Co, the abrasive wear is four times higher. By decreasing binder content in the case of E6-53N remarkable increase of wear resistance can be seen, but still wear rate is double compared to WC-15Co. In the case of nano alloyed E6-55N slightly higher wear resistance is detected than basic composition E6-55, although E6-55N has slightly lower hardness than E6-55. This is evidence that also flexural strength and

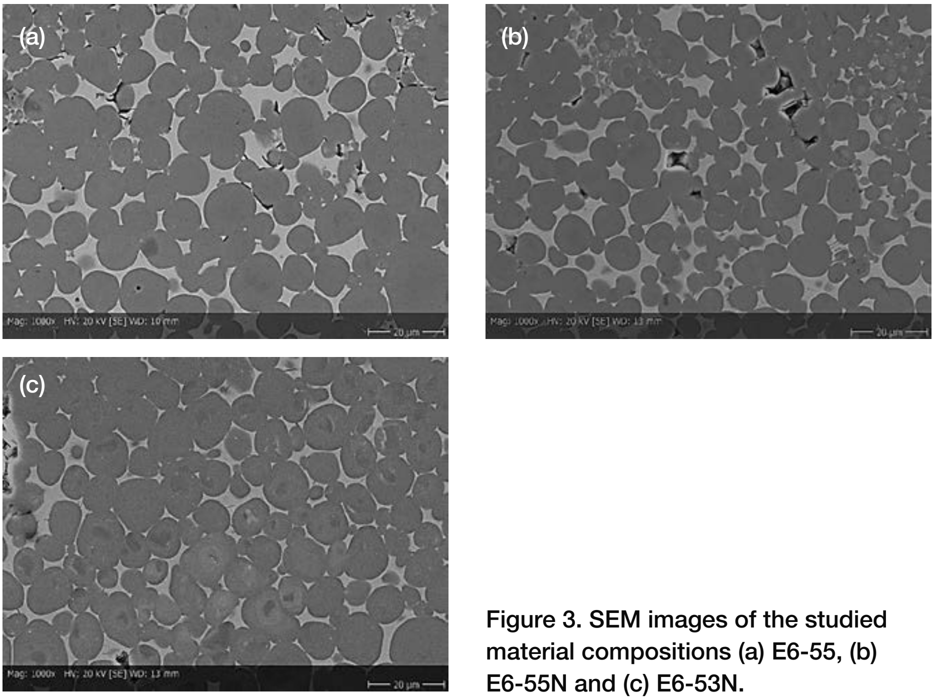

Figure 3. SEM images of the studied material compositions (a) E6-55, (b) E6-55N and (c) E6-53N.

fracture toughness have some role in abrasive wear resistance behind the dominating hardness.

Stability of abrasive wear process is studied by running crushing pin-on-disk test in 10 min period and measuring volume losses after each period. The results are clearly showing that wear process is linear and marks about suddenly increase of wear cannot be detected. This is evidence that studied materials are homogenous and unexpected spalling or similar doesn't happen.

Figure 3 shows SEM images of the studied material compositions (a) E6-55, (b) E6-55N and (c) E6-53N. Figures shows round TiC carbides with average size 10 µm (dark phase) quite well dispersed in the metallic Ni binder (whiter phase). Some porosity is detected.

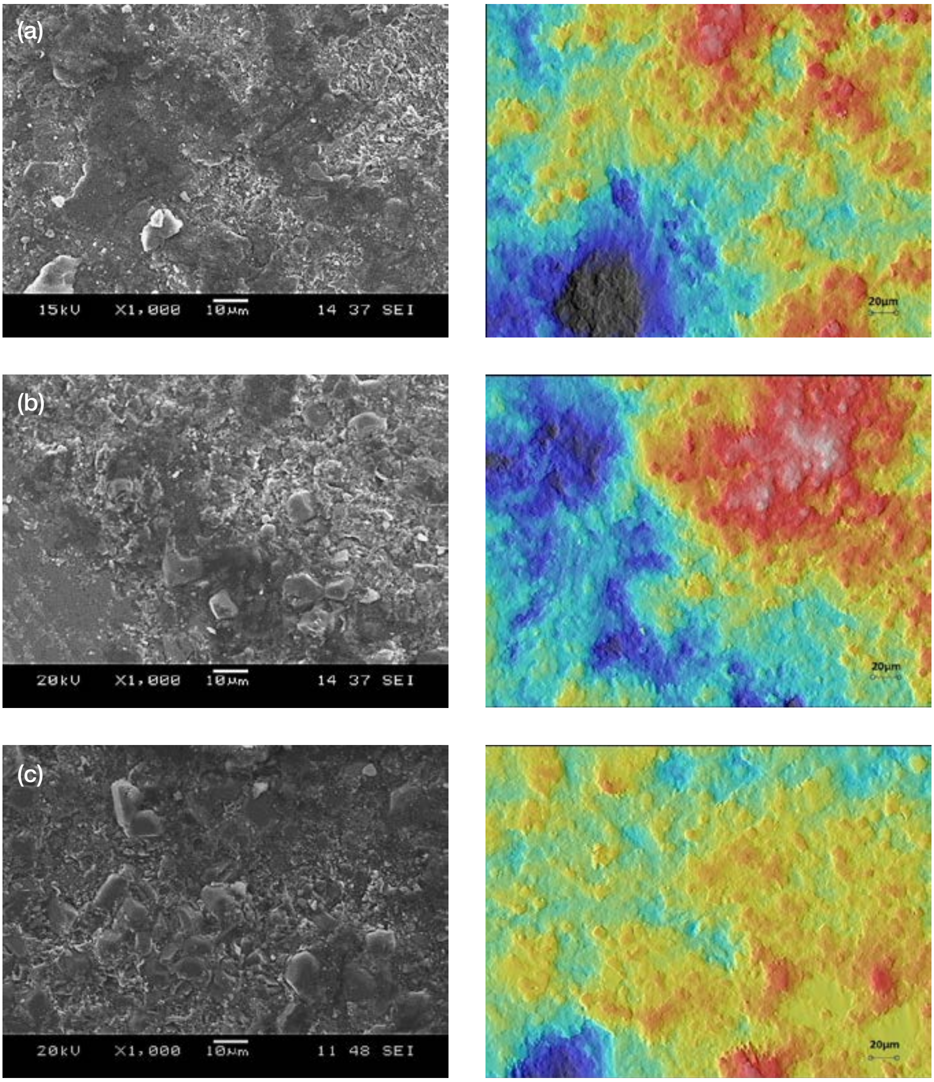

Figure 4. SEM and profilometer images of the wear surfaces after crushing pin-on-disc tests a) E6-55, (b) E6-55N and (c) E6-53N.

Figure 4 shows SEM and 3D profilometer images of (a) (a) E6-55, (b) E6-55N and (c) E6-53N after crushing pin-on-disc tests. Wear surface investigations reveal removal of soft metallic binder phase, fragmentation and crushing of carbides and removal of fragments by removing the binder. It is evident that binder matrix has worn more severely than the carbides. It seems that the matrix is first removed during crushing pin-on-disc tests and fragmentation of carbides starts only when the surrounded matrix has removed.

Conclusions

Gained mechanical test results (hardness, fracture toughness) shows that developed TiC hard metal grades are potential candidates to substitute traditional WC-Co in certain applications where high hardness and fracture toughness is needed. Crushing pin-on-disc tests reveal that wear rate for highest hardness composition E6-53N is double compared to WC-15Co. Wear surface inspection reveal removal of soft metallic binder phase which suggest that in the future development should concentrate on binder phase development. On the other hand now used crushing pin-on-disc test is very aggressive one, so in future also other abrasion tests will be conducted for wear resistance, and more detailed wear mechanism investigations.

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CIRCULAR PRODUCTS & DIGITALIZING THE LOOP

Towards circular product design

Päivi Kivikytö-Reponen

Our vision of Circular Economy is very dependent on the circular design implementation as in daily circular product design. It is worth mentioning that even 80% of product environmental impacts can be determined in design phase. Circular product design has many strategies and viewpoints; generally it looks at extending the life cycle of products, systems and components in order to preserve the value in the product life cycle, resource efficiency, creating value from waste and closing the loops.

It is very natural that every product on the market has its own business models. In circular product design business models are often highlighted when a possibility is seen for the company to transfer e.g. from product selling to service selling, or to shared products.

It appears that circular product design actually covers many aspects of eco-design, which is an approach to designing products with special consideration for the environmental impacts of the product throughout its whole lifecycle. However, an environmental and energy-oriented approach of eco-design could be seen as a core of circular product design. There is need to highlight design for circularity, design for remanufacturing, design for reuse and design for recycling. Circular product design covers design issues related to material perspectives, lifetime, performance, waste and use of secondary raw materials. Circular product design can be seen as a gateway to new businesses with new ecosystems.

In VTT's Mineral Economy Spearhead program, we have strongly focused on circular themes such as longer lifetime, resource (material and energy) efficient products and waste towards a product in our project portfolio. The accumulated expertise is described more specifically in this article in the lifecycle solutions and waste-to-value sections. An additional important and active theme in circular product design is remanufacturing, reuse and a product that recycles, for closing the loop.

For example, in the efficient manufacturing theme, a heavy duty valve block experienced dramatic weight reduction due to the new design possibilities of metal 3D printing; the mass after smoothing operations was 578.4 g, representing a 76% reduction compared to the original mass (2.5 kg). [1] Furthermore the additive manufacturing itself reduces material loss in production, i.e. system loss.

In the theme of waste as a product we have active research for high volume industrial side streams, such as tailings, steel and aluminum wastes and side streams, covering globally millions of tons of materials that can be considered as a potential resource in circular design. As an example, on average one kilogram of aluminum

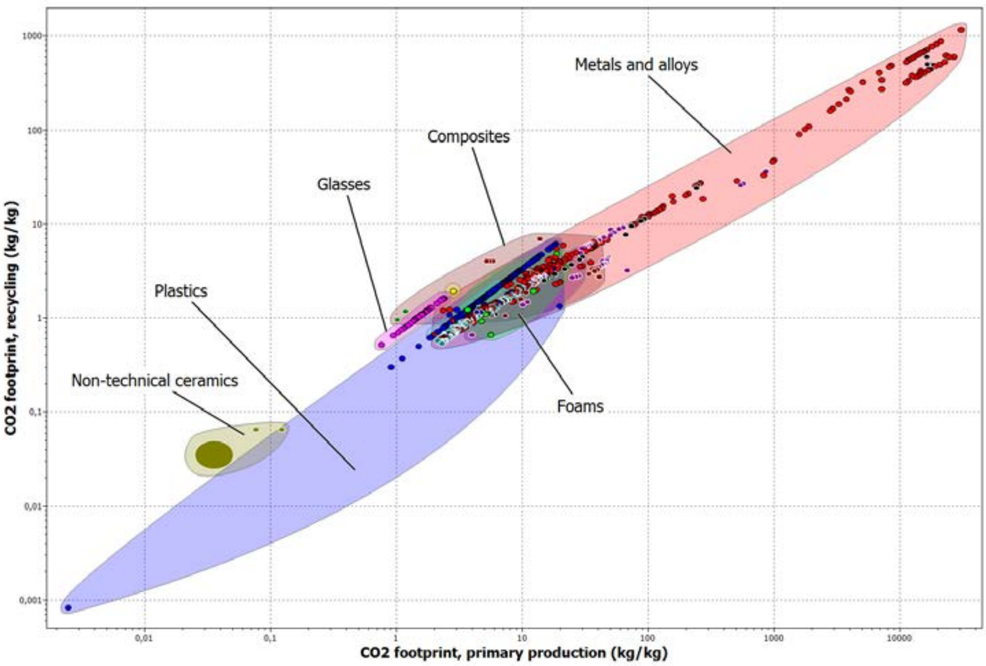

Figure 1. Comparison between carbon footprint of recycling and primary production. Source: Granta CES Selector 2015.

is processed from four kilograms of bauxite [2], and the annual world primary aluminum production total for 2015 was 57 890 thousand metric tonnes [3].

Lifecycle solutions

Longer lifecycles of products are enabled by entire life-cycle thinking and design, sustainable design and solutions, remanufacturing and reuse coupled e.g. with circular business models and recycling of the products.

Circular design in the nutshell is aiming at extending product lifecycles and closing the materials loops:

- to design and manufacture using raw materials that can be recycled
- to increase manufacturing sustainability
- to utilise additive manufacturing for decreasing waste
- to develop product modularity for dis- and reassembly
- to generate digital product data and tracking for increased circularity,
- to substitute toxic and critical components, etc.

Our enabling project portfolio for circular design includes the concept development, technology development and business models, one example being the project "From Data to wisdom – Approaches enabling circular economy" (D2W) [4]. The main goal of D2W is the systematic identification of relevant data, creation of radically new value constellations, and the conversion of this data into wisdom that is used to pilot and implement new circular operational and business models.

A sustainability target is essential in order to use planet resources wisely and responsibly and to eliminate the enormous waste generation seen today. Therefore, sustainability and responsivity are not separate subjects but cover all aspects of a product's lifecycle. For the technology industry, our activities include handprint development, sustainable value creation in manufacturing and sustainable business models. For example VTT coordinates the Carbon handprint project, in

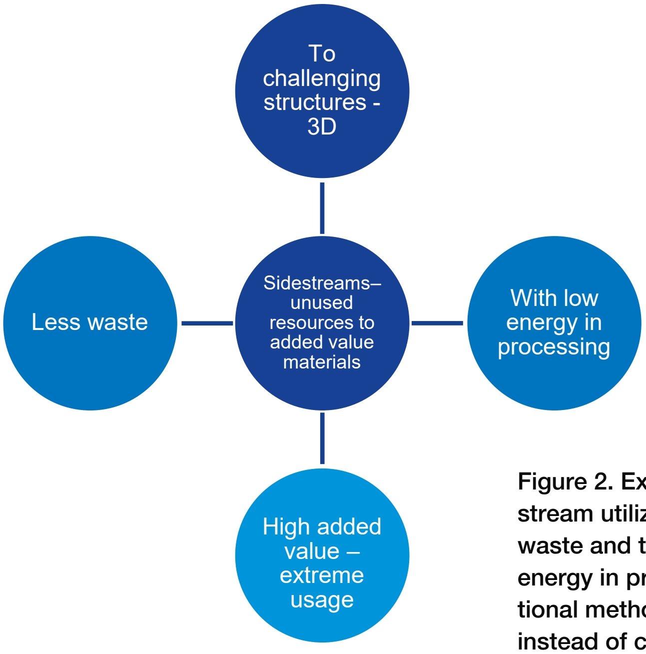

Figure 2. Examples of approaches in side stream utilization in a product minimizing waste and the utilization of material and energy in production compared to conventional methods, aiming at added value instead of conventional bulk production.

which guidelines for the assessment of positive actions and communication are developed. Handprints can be used to measure and communicate the products and activities related to the environmental benefits, as well as to direct customer activities towards competitive but sustainable choices. [5]

Remanufacturing means the return of a used product to at least its original performance with a warranty that is equivalent to or better than that of the newly manufactured product. Remanufacturing offers a potential to develop new business and is an increasingly popular area of development. Our networking in remanufacturing includes e.g. ERN Remanufacturing – Map of the Remanufacturing product design landscape. [6]

Value from side streams and waste

How can we make use of waste in a product and accept the idea in a larger scale? All secondary resources, for example end of life products, industrial side streams and wastes, are our wasted resources of today if they are not utilized and returned to production. However, products made from secondary sources can be used in various applications. The potential solution is not necessarily found in the same industrial sector that generated the waste. This area has high innovation potential, and there is an urgent need for technology development. Figure 2 shows examples of approaches in side stream utilization as a product. VTT has developed a platform for piloting new technologies for side stream and non-organic waste utilization before potential new businesses, start-ups or SMEs need even to invest in processing facilities. [7]

[1] http://www.vtt.fi/files/services/mav/ValveBlock_VTTInternetVersion.pdf
[2] http://www.aluminum.org/industries/production/primary-production
[3] http://www.world-aluminium.org/statistics/
[4] http://www.vtt.fi/sites/datatowisdom
[5] http://www.tekes.fi/nyt/uutiset-2016/hiilikadenjalki-mittaa-tuotteiden-ja-palveluiden-positiivisia-ymparistovaikutuksia/
[6] www.remanufacturing.eu
[7] http://www.vttresearch.com/services/smart-industry/factory-of-the-future-(2)/materials-and-manufacturing/material-solutions-from-powder-to-product

Targeting zero-waste processing and manufacturing digital spare parts as a case of additive manufacturing

Tarja Laitinen, Joni Reijonen & Tuomas Pinomaa

Introduction

Traditional original equipment manufacturers (OEMs) are transferring their business models strongly towards service providers. Currently full service agreements, earning models based on operational parameters and equipment leasing are typical. Future OEM business models could be based on simply providing the process availability to the end customers. Once the equipment manufacturer, maintenance provider and possibly even the operator become the same party, resource efficiency can be optimised over the whole lifecycle of an equipment.

A typical component lifecycle starts from primary material production, mining. Once the component has served its purpose, it is either reused or wasted. The resulting carbon and water footprints are large and solutions are being searched from new technology breakthroughs including new material design concepts, additive manufacturing methods and new recycling and recovery technologies. Future society will be based on sustainable development, in which zero waste and reuse are of central importance. Zero-waste manufacturing and materials reuse, including efficient utilisation of side streams as well as more efficient recovery technologies, provide the basis for the sustainable exploitation of natural resources.

Digital spare parts

Digitally designed (computer aided design, CAD) components have been part of normal engineering routines already for a long time. But when combining digital material models (integrated computational material engineering, ICME) and digital manufacturing methods (additive manufacturing, AM) into 3D CAD designs, we end up with a truly digital component. ICME transfers the traditional material development based on an experimental trial and error approach into a digital approach and enables new angles for component design through true performance-based material development. Digital design and digital manufacturing combined with digital material design masters the life cycle of a component with endless variation possibilities in extremely short time periods. The digitally developed, performance and design optimised, load-bearing components can be executed through a powder metallurgical (PM) route manufactured by AM. Adaptation of ICME in design is expected to decrease the time-to-market of new solutions and products by at least 25–50% (Gibbs 2013), resulting in return-of-investment by a factor of 3 to 9 across industry sectors (Lockheed 2013, Allison 2010), and to do so at a fraction of the cost (ICME 2013).

Spare parts are critical to ensure the operational performance of an equipment. Continuously decreasing product lifetimes, increasing demands for rapid time-to-market and strict service level agreements have led to enormous centralised warehouses with remarkable capital investments. As a rule of thumb, 80% of spare parts are slow movers, which account for less than 20% of the sales. OEMs are obliged to deliver spare parts for equipment built several decades ago. In addition, transportation of spare parts from centralized warehouses to plant sites, e.g. in 24 h upon request according to service agreements, is not energy- or cost-efficient. As an example, Sandvik Mining and Construction flies 1000 tons of spare parts annually around the globe (Laitinen et al. 2015). The everlasting cost efficiency improvement with leaner raw materials and lower-cost labour of manufacturing processes is also a growing challenge.

The process of joining materials to make objects from 3D digital model data, usually layer upon layer, as opposed to subtractive methodologies, is referred to as additive manufacturing (AM). AM is expected to result in the “Third Industrial Revolution” (The Economist 2012). Transfer from conventional machining to AM will also change business logistics and create new business models and value networks. The potential benefits endowed by AM compared to conventional manufacturing are undeniable: simpler supply chains with shorter lead times and lower inventories, no (or significantly less) need for tooling, production of small batches becomes economically feasible, product optimization for function, more economic manufacturing of custom designs (complex shapes) and significant reduction of waste material (Khajavi et al. 2014). In the current era of digitalized manufacturing, the capability of delivering additional value to customers by providing what they need when they need it has become increasingly important.

Locally, on-demand AM-produced spare parts with minimum transportation and no warehousing would be resource efficient. Minimum, short distance transportation would diminish the dependence on carbon-emitting fuels and improve energy efficiency. AM also offers possibilities for minimizing waste. The slow moving spare parts ageing and failing during the warehouse phase would not exist. AM techniques have the potential to approach 100% raw material utilisation due to the utilisation of material only in the component being processed. Local materials, possibly side streams of other manufacturing processes or secondary raw materials, could be utilised and the labour force would be locally employed. Two alternative material circulation routes for designed components are presented in Figure 1; one with primary production and conventional manufacturing methods, and the other with recycled materials and additive manufacturing methods. The most energy efficient solution would be if materials could be recycled without a melting phase, using different powder treatment procedures.

Possibilities

AM offers freedom of design. The component can be designed for function, not for manufacturing. Topological optimisation tools provide the means for determination of the optimum designs, once the design space and its limitations, as well as loads and other boundary conditions are defined. The component performance is the first priority, but when this is achieved, the novel designs enabled by additive manufacturing offer additional possibilities for weight reduction, internal structures, embedded intelligence, improved performance and cost efficiency.

Components can be made with hollow or complex lattice structures which retain structural

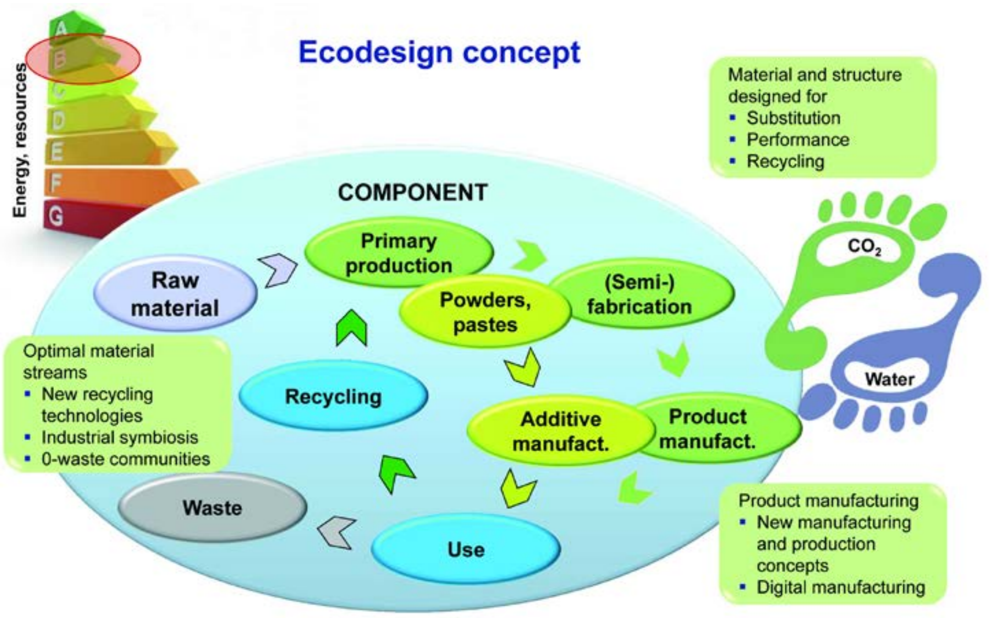

Figure 1. Two alternative material circulation routes, one with primary production and conventional manufacturing methods, the other with recycled materials and additive manufacturing methods.

strength with reduced weight. The size, position and orientation of internal channels can be freely chosen with improved surface quality when compared to surface channels obtained by subtractive manufacturing methods, thus also improving the performance of the component. Internal probes and sensors can be manufactured simultaneously with the component.

Moreover, AM provides a possibility for continuous component improvement by adjusting the design of spare parts and adding new features to it based on customer feedback and use history of the spare part. Spare parts with embedded intelligence would provide information on their condition in real time, thus providing means for condition-based maintenance (CBM). Older equipment could be retrofitted by next generation spare parts with novel properties and improved functionality. Intellectual property rights (IPR) would be merely embedded in material recipes, optimal designs and advanced manufacturing methods, no longer as published patents. The volume of pirate copies of brand products would diminish once the IPR became more difficult to duplicate. Internal, possibly structural tags could certify the spare part as an original OEM spare part.

Conclusion

Additive manufacturing / 3D manufacturing enables both solutions for narrowing (towards zero waste) and slowing (for a longer lifecycle) the material loop, exemplified with the case of smart digital spare parts. VTT has developed a platform for piloting additive manufacturing solutions before potential new businesses, either start-ups or SMEs, need even to invest in processing facilities. (VTT web site.)

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Circular economy on a platform

Sami Majaniemi

Introduction

Economy, ecosystem and platform – these along with a few other elusive concepts need to be introduced in our quest to formulate a new type of operational environment for circular economy. In plain English, this means that we need to understand what kind of design principles, interaction methods and tools help us steer the utilization of raw materials at different steps of materials manufacturing and a product's lifetime so that waste generation and various types of footprints are minimized in order to close the material loops.

The most plausible way of making sense of various types of material and information streams at a systemic level is to utilize digital platforms. Organizing knowledge generation based on heterogeneous information streams on a larger scale, e.g. for the purposes of regional or national level decision making, is a challenge that belongs to a class of so-called wicked problems. The optimal solutions to a wicked problem would benefit many stakeholders, but the authoritative power to implement a working solution may be lacking. Even defining the common objectives is usually difficult, as stake-holders have differing short term interests.

Generation of system level understanding creates value for all users

In order to gain better insight into the role of platform-based information gathering and refining in circular economy, we can think of the types of questions to which different user groups would like to get answers. The answers do not have to be exact in many decision-making cases; trends or order of magnitude estimates suffice in many cases. For example, regulators (legislators, municipal authorities etc.) would find it beneficial if it were possible to understand what types of economic and ecological impacts could be expected from different regulatory measures. Another user group operating on systemic level questions are investors, whose interests lie in the ability to compare the cost-effectiveness of different technology solutions across lengthy production chains. More specific materials related questions will be raised by businesses interested in using for example the wastes or side streams. These companies would benefit from being able to compare which components in their product's material composition could be replaced by cheaper, more abundant or more durable material alternatives.

As the answers to the questions presented above must be put together from many cross-disciplinary sources (materials science, engineering, sustainability assessment, economics, political impact analysis etc.), the content producers of the platform must represent numerous disciplines whose interaction has not historically been very strong. Although this creates another challenge for organizing the platform-based knowledge generation, it also creates a possibility to form a cross-fertilizing market place for solution providers (e.g. software companies, modelling agencies, researchers), cross-disciplinary translators, as well as new knowledge generation service models, among other things.

Existing tools and practices in the making

On a technology level there are many tools which are helpful in increasing our shared understanding of the information streams related to circular economy. The necessary technological solutions deal with establishing suitable standards for transferring, packing and interpreting data (e.g. data models, measurement standards, interface protocols, information visualization and analysis tools). Furthermore, new technological solutions are needed for developing hierarchical material models enabling property prediction with sufficient accuracy. We also need systemic level simulation models giving us insight into how the interplay of science, engineering and policy choices affects the everyday life of a company, individual or a circular economy stakeholder group.

The platform acts as a glue, which brings the information from these seemingly different areas together and enables its users to collaboratively develop their shared view of the wicked problem and its possible solutions. The platform technology has been developed over the years in various domestic and international projects in which VTT has participated. Moreover, there are currently a number of technologies which can be used in finding answers to design questions such as how to create longer lasting products, how to utilize measurement information in production process control and how to combine existing LCA and eco-design tools with business model scenario-making, or to develop completely new tools for supporting CE decision making and e.g. circular design strategy evaluation.

Despite the fact that there is more data available than ever before, and more means to process it into an understandable form, we are still lacking the means of creating a shared understanding on a larger scale. Platform technology represents a necessary, although still insufficient ingredient in striving towards this goal. What is also required is the development of means to appreciate long term design objectives over the short-term ones together with a culture in which shared knowledge is understood as a source of wealth by all participants of the platform ecosystem. As explained in more detail below, this does not imply that every bit of information would have to be available to all members of the ecosystem. Indeed, it is possible simultaneously to respect the sensitive data sources and business secrets while producing useful macro level analysis results for all the ecosystem participants.

What next – The era of information banking

Currently, various groups at VTT are involved in several international collaborations developing circular economy design tools and practices. For example, two projects funded by the European Institute of Technology focus on creating a virtual work space for circular economy participants, and one large strategic research project is funded by the Academy of Finland. In addition to the strategic decision making for circular economy, practical circular design would benefit from new digital tools. One of the goals is to combine process simulation, LCA and financial models in mineral processing into an integrated tool enabling users to solve scale- and discipline-dependent multi-design problems. The demonstration cases also address the establishment of value-creation networks, an example of which has been depicted in Figure 1.

Here one comes across the chicken and egg problem referred to in the previous section: How is it possible to motivate potential ecosys-

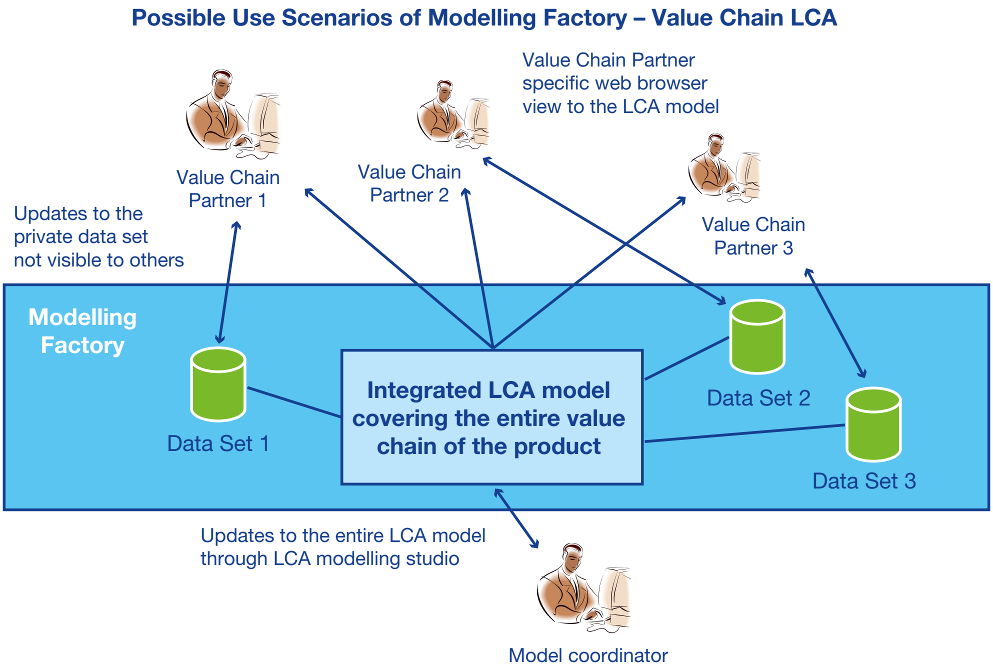

Figure 1. Value chain LCA as an example of sharing information on a circular economy design platform, enabler for model based collaborative value-network design (Modelling Factory).

tem participants to relinquish their information to a trusted operator, who exploits the power of the platform to reveal useful estimates on the performance capability of the ecosystem as a whole, while protecting the privacy of the individual businesses? The performance capability information can be used by the ecosystem members to design their own production, participation and business models in such a way that the final products of the network are more acceptable to consumers, and therefore all ecosystem members can extract more value e.g. in the form of increased sales.

In order for this type of interaction to work, a trust network needs to be developed together with the information sharing network. This is a tall order, if there is no such trust network to begin with. However, working examples do exist: The banking system can be seen as an analogous construction, where instead of information units the transactions are settled in monetary units.

Financial trust networks enable the use of sensitive (monetary) information, creation of new financial instruments (cf. aggregate databases), automated validity checks (cf. block-chain) and production of systemic level information (e.g. stock market indices), which all the participants can utilize despite the fact that the individual operations data can be very sensitive from the point of view of any individual participant. Although all this power can be misused, the banking system has demonstrated that organizing this type of information refining and sharing activity on a large scale is possible when the participants believe in the usefulness of the construction and trust its technical realization. Why should a similar success not be attainable in the case of design information refining trust networks?

RECYCLING THE PRODUCTS

Scrap recycling

John Bacher
Contributors: Jutta Laine-Ylijoki & Ulla-Maija Mroueh

Introduction

Resource scarcity is a dynamic concept, depending on many factors and varying over time. Different industries hold different views on which is the most importance resource for their business. Furthermore the concept of supply risk varies by region, and therefore various lists of critical elements or resources exist. Recently, prices of certain minerals and elements have been increasing dramatically due to global supply shortages and increasing demand. These elements are vital components of advanced technologies, such as cell-phones, wind turbines, permanent magnets and semi-conductors. This relative scarcity of certain valuable elements has prompted many companies to search for new mineral sources. In addition to new mining ventures, recycling of different wastes, such as Waste Electrical and Electronic Equipment (WEEE) and End of Life Vehicles (ELV) has been suggested as possible untapped resources.

The perceived supply risk steered the EU to identify 20 critical raw materials or metal groups, so-called Critical Raw Materials (CRMs). These are materials that are considered to be vital for development in areas such as computers, electronics and electric vehicles and for which the utilisation is expected to increase significantly in coming decades. For example, the global demand for rare earth oxides (REOs) is expected to reach 150 000 tonnes by 2020, e.g. due to increased utilisation in cleantech (Adamas Intelligence 2014). Furthermore, the known assets of several of these substances are concentrated in countries outside the EU. This means that CRMs are both of high economic importance and vulnerable to supply disruption. Their extraction also causes significant environmental impacts. For all these reasons, increasing the recovery of critical raw materials is one of the challenges that must be addressed and overcome with innovative industrial processes.

Equipment design and WEEE recycling

CRMs are often present in electronic devices. Currently, less than 1% of CRMs are recycled from End-of-Life (EoL) equipment (UNEP 2013). Therefore, a key source of CRMs could be WEEE. With an annual growth rate of 3–5%, WEEE is one of the fastest growing waste streams. In the EU alone, about 12 million tonnes of WEEE is produced annually. On a global scale WEEE amounts already to 20–50 million tonnes per annum (UNEP 2013). However, only about 30% of the WEEE generated in the EU is currently properly recycled, and hence ~70% is not recycled at all or only poorly recycled.

There are a variety of possible technologies and unit processes for recovery of CRMs from WEEE. These include thermal, hydrometallurgical and bio-hydrometallurgical technologies. However, due to the low concentrations of valuables and the general heterogeneity and complexity of electronics, it is still a challenge to combine these technologies into economically feasible recycling solutions for CRM recovery.

Figure 1. Electronics components and dust generated in the crushing process.

In addition, it can be expected that the complexity of electronics will further increase. In order to improve the efficiency of devices and to decrease material costs, the electronics producers are aiming towards miniaturization and multifunctionality of components. This leads to decreasing amounts of valuables per device and increased use of joined materials difficult to separate. The efficient recovery of CRMs from WEEE is also hampered by the fragmentation of the recycling industry, in which several players across the value chain are optimizing their own sector. This leads to losses of CRMs and poor profitability. Furthermore, in order to increase the overall recovery of valuable materials from WEEE, more in-depth information and knowledge is required concerning the behaviour of materials/elements and flows over the whole WEEE treatment chain. Issues such as distribution of elements in processes, liberation of materials and components as well as losses play a crucial role in reforming the recycling operations.

Crushing losses remain unrecovered in WEEE recycling

Complex electronic devices entering our recycling systems often generate losses in the whole treatment chain. In order to separate valuable Printed Circuit Assemblies (PCA) and other metal parts from plastics and other materials, the feed material needs to be broken. In this crushing process, the materials are disengaged from each other with various liberation distributions. Liberation describes how well the materials are disconnected from each other. Poorly liberated particles composed of several materials affect the physical properties of the particles in such a manner that the separation based on some physical property (i.e. density, colour, magnetism) weakens (UNEP 2013). Generally, the liberation of materials is increased when particle size is decreased through size reduction (Castro et al. 2005, Menad et al. 2013, Quan et al. 2012, Zhang & Forssberg 1997).

However, separation processes may have particle size limitations below which the efficiency starts to decrease. This leads to an optimization between particle size and liberation of materials. In addition, size reduction generates differently sized particles. Within this size distribution fines and dusts are also generated, even though the majority of particles are within the desired size range. The loss of valuable elements has been reported with these dusts (Bachér et al. 2015, Chancerel & Rotter 2009, van Schaik & Reuter 2014, UNEP 2013). This relation between the losses of valuable elements with dusts and the liberation of desired components in WEEE recycling becomes relevant when mechanical treatment is carried out to reach recycling targets.

Research at VTT (Bachér & Kaartinen 2016) has focused on investigating the relationship between the liberation of valuable Printed Circuit Assemblies (PCA) and dust generation in the crushing processes of two different types of mobile phone samples (regular vs. sophisticated). The results revealed that the overall PCA grade in both samples was approximately 70%, with around 3.5% dust generation. However, the liberation distribution of PCAs differed between mobile phones, resulting in better distribution for the sophisticated phones. The platform-based design connecting surrounding components to the PCA in regular phones, together with smaller initial size, resulted in poorer liberation distribution of PCA. Furthermore, the dust fractions included both noble and scarce metals, such as Neodymium from speaker magnets and motors for vibration but also contaminants that need to be taken into account when further processing is planned. A higher gold concentration was detected in dusts from regular phones, since the protective plastic casing crushed more easily, thus exposing the PCA surface to grinding.

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Recovery of metals from low-grade ores and residues

Päivi Kinnunen, Jarno Mäkinen & Inka Orko

Scarce raw material deposits drive to novel process needs

Easy-to-access mineral deposits are exhausting globally, and the remaining minerals typically have lower concentrations and more complex structures. The economy of extracting metals from such ores is generally poor with the current industrial methods.

The mass-fraction of valuable minerals may vary substantially from a few grams per ton of rock in gold mines to over 50% in iron ores. Every mine site is specific in terms of ore type and gangue material, and the production technologies need to be designed individually case-by-case in a holistic way.

In addition to low-grade primary ore deposits, significant amounts of metals are locked up in industrial process residues, for example in tailings, metallurgical sludges, slags, dusts and ashes. Mining and crushing energy costs can be avoided by utilizing already crushed secondary materials. Traditional pyro- and hydrometallurgical approaches designed for high-grade metal ores and concentrates do not suffice for these low-grade and complex resources. New unconventional and hybrid processing methods need to be developed for better separation and higher metal recovery rates in order to ensure metal recovery in an environmentally, socially and economically sustainable way.

VTT's novel concepts for extracting more value from minerals

VTT has developed new concepts to recover economically important and critical metals from lower-grade sources. We combine both novel and current industrial technologies, including mechanical, chemical, physical and microbiological processes. The unit operations include pre-treatment, metal extraction, metal recovery and (residual) matrix valorisation, as well as water treatment.

To support the work, VTT has invested in state-of-the-art research infrastructure for metal recovery experiments. Our hydrometallurgical Flexmet platform is used extensively for chemical acid leaching and precipitation for increased metal selectivity and yield. New unique research equipment such as crystallization analyser, particle view and particle tracker have been taken into use with the support of Academy of Finland FIRI funding. VTT is also the coordinator of the KIC Raw Materials hydrometallurgical infrastructure networking project, which connects European hydrometallurgical infrastructures together.

In addition to chemical processes, microbiological leaching methods have given promise in valuables recovery from low grade ores and side-streams. In addition to classical bioleaching of base metals (copper, nickel and zinc) from sulphide ores and mine site tailings, work has been intense with novel solutions such as bioleaching of phosphorus from apatite minerals, with 90% phosphorus leaching yields. Bioleaching has also proved to be an effective and selective method for removing uranium impurities from phosphate ores. In addition to leaching, microbiological methods can be utilized for valuables recovery. For example, VTT has filed a patent (FI 125550 B) on a method for recovering rare earth elements from sulphate wastes utilizing sulphate-reducing bacteria for precipitation of REE concentrate.

As an example of VTT's work with secondary raw material sources, the Jarogain project aims at extracting and refining valuable metals from jarosite waste. VTT has developed and filed a patent on a concept to extract silver, lead, zinc and iron from the mineral waste formed in zinc manufacturing. Furthermore, VTT in cooperation with Aalto University is developing a business case based on the technology. According to preliminary investment estimates, with long-term average metal prices and current process yield expectations, the payback time for an extraction facility may be 5–6 years.

Business benefits from cost competitive technologies

The new technologies will enable companies valorise their low-grade materials, gain cost savings and add new income streams, and in the larger scale, increase our resource efficiency and decrease our dependency on metal imports. For example, 500 000 tonnes of iron-rich sludges are produced annually in the EU. The metal value in the tailings of a single mine alone can be over 100 M€. Moreover, it is not just a question of the metal value locked in the wastes, but also of the costs related to waste disposal. If harmful properties can be removed from wastes, they can

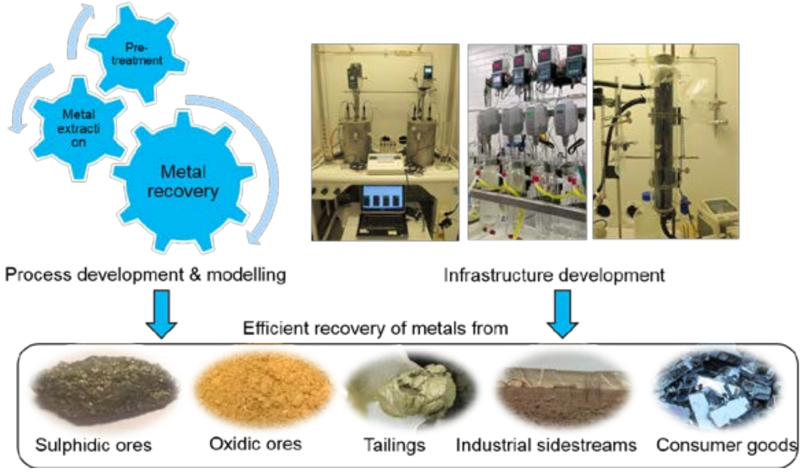

Figure 1. VTT has developed new processes and infrastructure for metals recovery from various low-grade sources.

be used as raw materials for e.g. construction materials or added-value products instead of being sent to waste disposal.

Opportunities for the mining, metals refining, chemical and energy industries

Innovative use of mechanical, physical, chemical and biological methods offers adaptability of processes for various challenges in side stream treatment in the mining, metal, chemical and energy industries. In all these industry sectors, side streams and wastes are unique, according to mineralogy, chemical composition and impurities, but by combining and optimizing the right processes, solutions can be tailored to solve the treatment challenges. Currently, VTT is working with treatment processes for e.g. metal removal and recovery from almost all industrial sectors.

Continued work towards industrial implementation

The development of new metallurgical unit operations for both primary ores and secondary industrial side streams continues e.g. in the METGROW+. METGROW+ is an EU-funded project under grant agreement No 690088 coordinated by VTT for nickel-cobalt laterites, iron-rich sludges, chromium-rich sludges and fayalitic slags with a systemic approach to couple the individual unit operations. The results will also be widely applicable to other material streams containing metals. Long-term targets of our projects are increased selectivity, higher yields and improved internal water circulation in industrial processes. VTT is also engaging in customer work to take the technologies to industrial use.

Series title and number
VTT Research Highlights 13

Title	Added value from responsible use of raw materials
Author(s)	Päivi Kivikytö-Reponen, Ulla-Maija Mroueh & Jarno Mäkinen
Abstract	Raw materials are an essential component of our quality of life, economy, and the well-being of modern society. In coming decades, the need for raw materials, water, food and energy will double due to the increasing population. Furthermore, the global distribution of mineral raw material resources is uneven, which leads to heavy dependence of both companies and nations on reliable access to raw materials. VTT's spearhead programme Mineral Economy targets to a profitable circular economy by introducing technology based solutions. The aim is to enable the generation of innovations leading to economic growth, jobs and societal well-being in Finland and in Europe. The programme has actively developed multi-technological competences to enable new innovations and sustainable access to resources, e.g. in hydrometallurgy and powder metallurgy. The topics include sustainable design, innovative use of waste and low grade minerals as a resource, substitution of critical raw materials, 3D manufacturing for narrowing the loop, remanufacturing, reuse for slowing the loop and recycling for closing the loop. Digitalization and utilizing digital platforms are enablers for responsible use of raw materials. Our target is to develop the digital circular economy Modelling Factory platform for a metals ecosystem covering multidisciplinary expertise. This publication introduces selected research highlights from VTT's Mineral Economy spearhead programme in order to disseminate our research results and to introduce new viewpoints and opportunities for the stakeholders in Finland and in Europe.
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