Science 4 Technology – Poster Exhibition 2024

Department Metallurgy

Chair of Nonferrous Metallurgy

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Poster Exhibition 2024

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Effect of low-temperature long-term aging on the mechanical properties of age-hardenable aluminum alloys

Introduction

Atomic clusters, characterized by a local accumulation of alloying atoms without a recognizable structure, mark the initial stage in the precipitation sequence and depict the first hardening increment [1-2]. The focus on cluster hardening instead of commonly found metastable phases is a promising approach for strengthening aluminum alloys with minimal loss of ductility [3].

We demonstrate that long-term low-temperature aging generates clusters that enhance both strength and elongation. In tensile tests, cluster-hardened alloys display pronounced strain-hardening properties, which we evaluate by structural analysis using atom probe tomography (APT).

Experimental

Sheets of Al-Mg-Si-Cu (6xxx) and novel Al-Mg-Zn-Cu (5/7-Crossover) alloys with the chemical composition given in Table 1 were produced by a combination of hot and cold rolling. Heat treatments according to Table 2 include long-term-aging (LTA) for 42 days at 100 °C and 60 °C after previous solution heat treatment. The formation of clusters and precipitates during LTA was analyzed via APT using a LEAP 3000 X HR (6xxx) and a LEAP 5000 XR (5/7-Crossover).

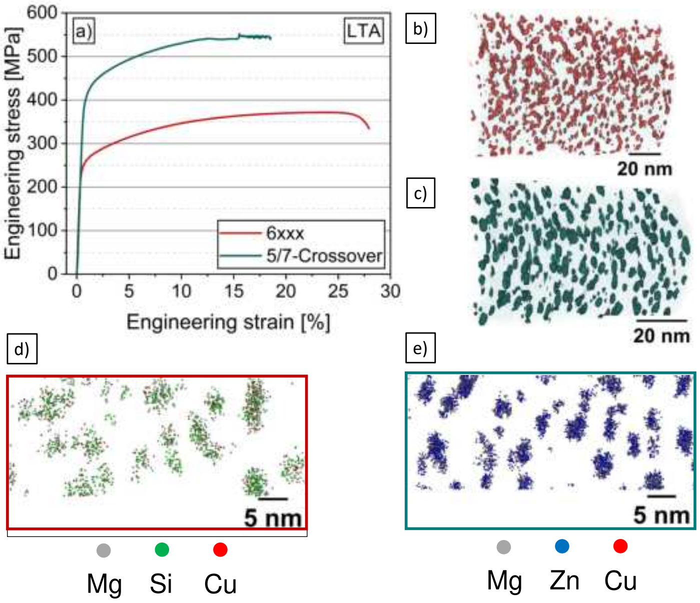

Figure 1: Engineering stress-strain curves resulting from LTA (a). Corresponding 3D-elemental maps of detected clusters for 6xxx (b) and 5/7-Crossover alloy (c). Region of Interests (ROI) for 6xxx (d) and 5/7-Crossover alloy (e) with a dimension of 20 x 10 x 50 nm^3 were chosen for a detailed representation of the cluster structure.

Table 1: Chemical composition of 6xxx and 5/7-Crossover alloy

Alloy	Mg	Si	Zn	Cu	Fe	Mn
6xxx	0.4	0.9	0.0	1.1	0.1	0.1
5/7-Crossver	5.0	0.1	3.8	0.8	0.2	0.4

Table 2: Processing of 6xxx and 5/7-Crossover alloys

Alloy	Solution annealing (SA)		Aging	
	T_{SA} [°C]	t_{SA} [min]	T_{Aging} [°C]	t_{Aging} [days]
6xxx	540	20	100	42
5/7-Crossver	465	35	60	42

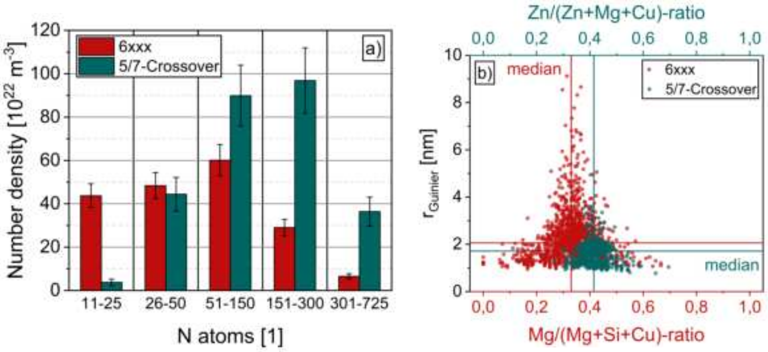

Figure 2: Number density as a function of the containing atoms (a) and sizes of clusters (Guinier radius) in relation to their chemical ratio (b).

Results & Discussion

LTA increase both yield strength and strain hardening capacity due to the presence of fine, uniformly distributed clusters/precipitates (see Figure 1), resulting in excellent ductility. As illustrated in Figure 2, both alloys exhibit dominant clusters with 51-300 atoms, alongside smaller clusters (11-25 atoms) and some larger clusters/precipitates. The median cluster size (Guinier radius) is roughly 2 nm for the 6xxx alloy and around 1.7 nm for the 5/7 crossover alloy.

Solute atoms and clusters play a dual role in improving strength and strain hardening. By hindering dislocation slip and reducing the dynamic recovery rate, they act as obstacles and increase dislocation density. When dislocations cut through clusters, local stress concentrations are relieved and dynamic recovery during deformation is suppressed. This promotes the accumulation of dislocations, which ultimately increases strain hardening [4].

References

[1] Aster P. et al.: Strain-induced clustering in Al alloys. Materialia, 32 (2023), 101964

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[3] Takata K. et al.: Improvement of Strength–Elongation Balance of Al–Mg–Si Sheet Alloy by Utilising Mg–Si Cluster and Its Proposed Mechanism. MATERIALS TRANSACTIONS, 58 (2017), 728–733.

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My research revolves around the influence of cluster formation on strength and forming behaviour of various aluminium alloys.

AMAG ROLLING

Christian Doppler Forschungsgesellschaft

Strain-induced clustering in Al-alloys

Introduction

Solute clusters are an age-hardening reaction seen in all age-hardenable aluminium alloys and can greatly influence material properties. An important aspect of clustering is the advantageous combination of strength and ductility found under certain conditions where clusters are present [1].

Here, we investigate the evolution of clusters upon deformation in a pre-aged novel AlMgZnCu crossover alloy, which shows pronounced strain hardening. Atom probe tomography (APT) measurements and a simple modeling approach revealed that significant cluster formation occurs simultaneously with plastic deformation [1].

Experimental

Sheets of an AlMgZnCu (Crossover – Alloy, CA) alloy with the chemical composition given in Table 1 were produced by a combination of hot and cold rolling. Pre-Aging (PA) at 60°C/5h was examined immediate after quenching to RT. The as-quenched (AQ) condition provides reference data on the mechanical properties [1].

Table 1: Chemical composition of the Crossover alloy

Alloy	Mg	Zn	Cu	Si	Fe	Mn
CA	5.0	3.8	0.8	0.1	0.2	0.4

The formation of clusters during PA was analyzed via APT using a LEAP 5000 XR [1].

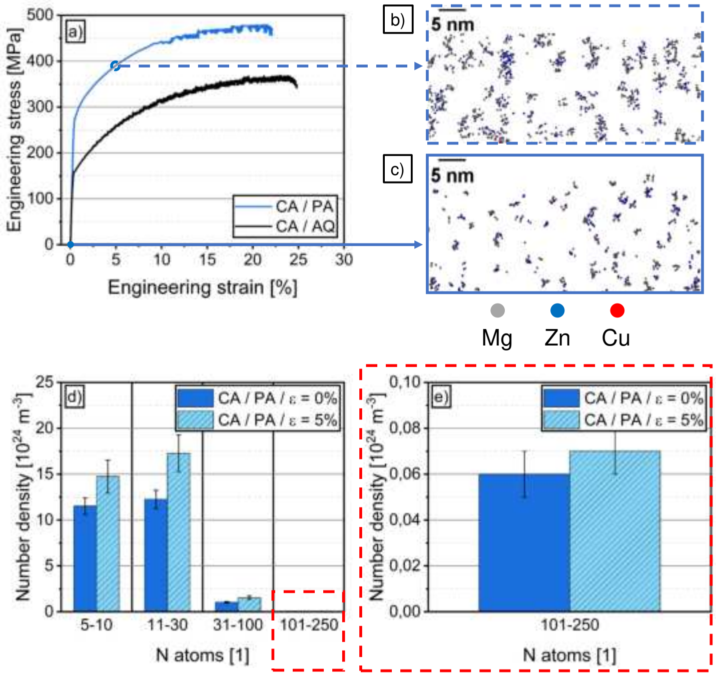

Figure 1: Engineering stress-strain curves resulting from PA and AQ (a); 3D elemental maps (20 x 1 x 50 nm³) for clusters in deformed (b) and undeformed (c) states; Number density as a function of the atoms of clusters in deformed (striped blue bar) and undeformed (solid blue bar) state (d); magnification view at larger numbers of atoms (e).

Table 2: APT analysis of Cluster-Volume fraction in PA condition

Alloy	Strain [%]	Cluster – Volume fraction [%]
CA	0	1.66
	5	2.01

Excess vacancies are decisive for diffusion and enable phase transformations. The evolution of the strain-induced excess vacancy concentration c_{ex} during deformation can be estimated with the following equation [1]:

$$\frac{dC_{ex}}{dt} = \chi \frac{\sigma \Omega_0}{H_V^f} \varepsilon - \frac{D_V \rho}{\kappa^2} c_{ex}$$

Since Mg contributes significantly to cluster formation, this alloying element was chosen to demonstrate the effect of strain-induced vacancies on the diffusion length. The diffusion length x_{Mg} after time t can be expressed by [1]:

$$x_{Mg}^2 = \int 4 \cdot f \cdot D_{Mg} \cdot dt$$

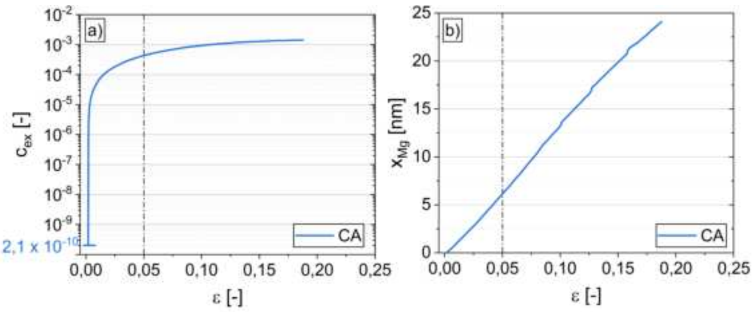

Figure 2: Evolution of the excess vacancies (a) and diffusion length (b) during deformation. The black dashed lines mark the results at 5 % strain.

Results & Discussion

Figure 1a shows tensile test results, highlighting increased clustering after 5% strain, evident in 3D element maps (Figure 1b-c) and number density assessments (Figure 1d-e). Of specific interest is the enhancement in cluster volume fraction during deformation (Table 2), indicating cluster generation [1].

The simulation results (Figure 2) illustrate the excess vacancy concentration (Figure 2a) and the mean diffusion length of Mg (Figure 2b) plotted against true strain. Vacancies, which are required by solutes to migrate, are produced in excess during deformation and support the formation of new clusters. As shown in the model above, their concentration is several orders of magnitude higher than the initial vacancy concentration after PA treatment, which is sufficient to achieve the required diffusion distances even at RT [1].

The mechanism of strain-induced clustering in this study shows potential in the future optimization of the strength/formability tradeoff [1].

References

[1] Aster P. et al.: Strain-induced clustering in Al-alloys. Materialia, 32 (2023), 101964

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Laboratory direct chill casting for alloy design

Introduction

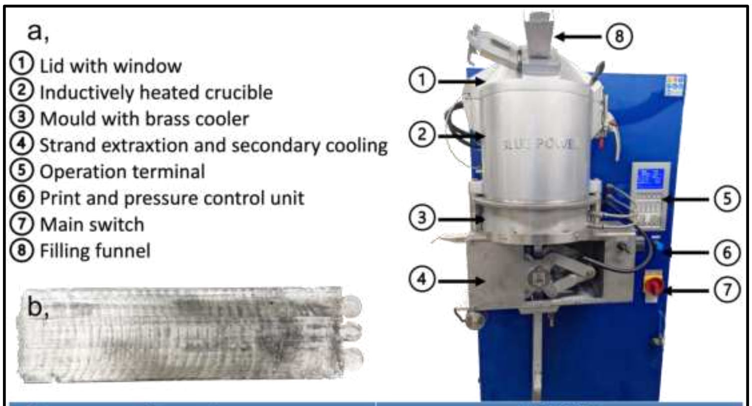
In this study, a lab-scale continuous casting plant is optimized for casting diverse aluminium alloys, focusing on controlling solidification conditions. The aim is to mimic industrial production flexibility for upcoming alloy designs and boosting sample production for enhanced productivity. Key factors influencing non-ferrous metal casting include: [1-3]

- casting temperature and overheating,
- casting speed, mould thermal conditions,
- alloy composition,
- the use of vacuum/inert gas systems (Ar, N_2).

Experimental

Figure 1 shows the VCC3000 vertical continuous caster with an inductively heated crucible and a graphite sliding mould, representing a novel laboratory-scale sample production setup. Initial tests revealed limitations in casting several aluminium alloys to the desired quality, necessitating adaptations. The issue of cooling capacity proved to be critical, with existing research highlighting cooling deficiencies. [4, 5]

Two conversion steps were taken: first, to adjust the heat transfer between the mould and the brass cooler (Figure 2a); second, to increase the overall cooler surface area (Figure 2b), resulting in a shift of the liquid-solid transition ("mushy zone") towards the end of the mould.



c, Parameters	VCC3000
Heating power max.	30 kW
Temperature max.	1500 °C
Crucible volume	3400 cm³
Cross section max.	110 x 30 cm

Figure 1: Laboratory continuous casting plant VCC3000 (a) with system parameters (c) and a cast strand (b).

Results & Discussion

The improved cooling rates and reduced variation in dendrite arm spacing demonstrate significant improvements from the plant adaptations. Figure 2 illustrates these improvements in semi-finished samples produced during the trials. For zinc, a roof-shaped concentration distribution is observed, indicating an enrichment of alloying elements in the strand center due to segregation processes during solidification.

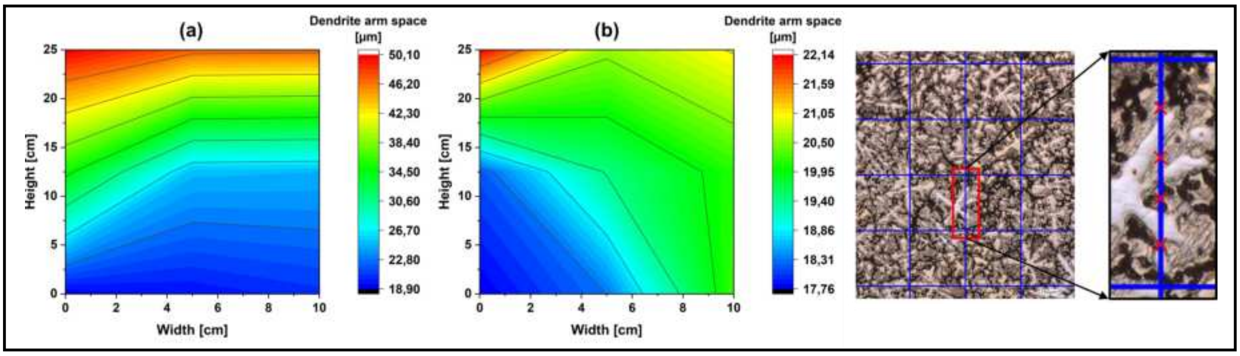


Figure 2: Comparison of the dendrite arm space after casting for the semi-finished products after adaption 1 (a) and adaption 2 (b) with higher cooling rate. The methodology of measuring the dendrite arm spacing is shown on the right.

Conclusion and outlook

In summary, the adjustments significantly reduce the variation in secondary dendrite arm spacing, thereby improving process stability. Figure 2b shows consistent heat transfer to the coolant. Tailored casting programs for different alloys require further testing. Integrating virtual simulation with real trials improves efficiency and results.

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[3] Kammer C.: Aluminium-Taschenbuch. Aluminium-Verl., Düsseldorf (2002).; [4] Kazup A., Karpati V., Hegedus B. und Gacsi Z.: Semi-continuous casting and microstructure investigation of the AlSi12 alloy. Materials Science and Engineering, (2020).;
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Direct chill casting of aluminium alloys with focus on the castability of various Al-alloys with increased scrap content on a laboratory scale.

AMAG AUSTRIA METALL

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Microstructure evolution in Al-Mg-Si alloys controlled by Fe-rich intermetallic primary phases

Introduction

Recycling aluminium alloys presents a significant challenge due to the diverse composition of aluminium scrap. Iron is problematic as it accumulates during the recycling of secondary aluminium and is challenging to remove through metallurgical methods. Iron in aluminium alloys tends to form coarse primary intermetallic phases (IMPs), which negatively impact formability and can reduce overall ductility. However, by utilizing sophisticated alloy design and processing, these otherwise detrimental primary phases can be used to control the microstructure evolution. These microstructural improvements result in unique mechanical properties such as high total elongation despite the presence of large amounts of brittle intermetallic phases, higher strength and, most notably, much higher work hardening, indicating good formability.

Modifying the morphology of harmful Fe-rich primary phases

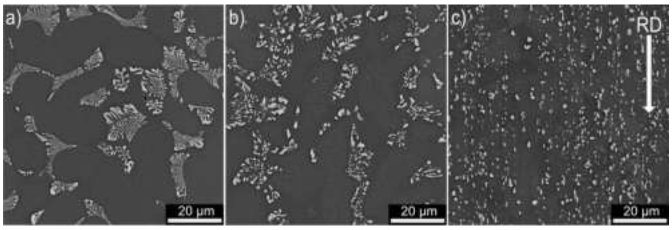

Figure 1: BSE-micrographs of the experimental alloy 6016+IMPs in the conditions a) as cast, b) homogenized and c) solution annealed (T4)

- Fast solidification results in a fine branched morphology of Fe-rich intermetallics in as-cast condition.
- Homogenization induces initial fragmentation into individual particles.
- Further thermomechanical processing, i.e. hot and cold rolling, produces finely fragmented particles with diameters of ~1 µm.

Microstructure control for enhanced mechanical properties

The first and most obvious effect of the high number of fine IMP particles is grain refinement by particle-stimulated nucleation. Near the IMPs, an increased dislocation density is formed during rolling, creating favourable sites for the formation of recrystallization nuclei. Furthermore, if the reference alloy 6016 with the particle-enriched version 6016+IMPs in the T4 state is compared, a higher yield and tensile strength, a much-improved work hardening rate, and a surprisingly high total elongation is obtained, considering an amount of ~10% brittle intermetallic primary phases. The higher strength of 6016+IMPs can be attributed to increased grain boundary hardening and the formation of geometrically necessary dislocations (GNDs). Work hardening is much more pronounced in 6016+IMPs due to the formation of additional GNDs during deformation. The good ductility can be assigned to the small size and spherical morphology of IMPs, as well as the small matrix grain size and the GND-forest surrounding the particles.

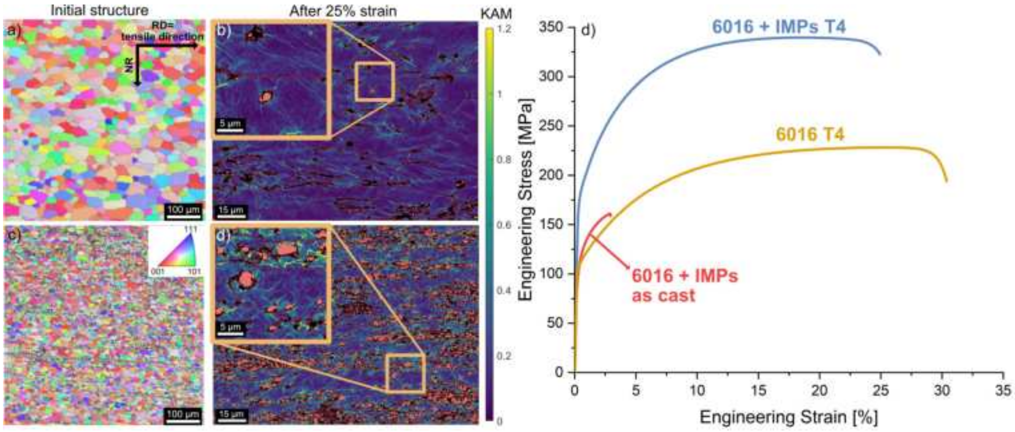

Figure 2: IPF maps of sheet normal direction (ND) of the solution treated alloy for (a) alloy 6016 and (c) alloy 6016+IMPs. EBSD kernel average misorientation (KAM), grain boundaries >10° (red) and IMPs (red) of (b) alloy 6016 and (d) alloy 6016+IMPs after applying 25% strain. The insert shows a magnified section of the micrograph.
d) Engineering stress-strain curves for alloy 6016+IMPs in T4 and as cast state and reference alloy 6016 in T4 condition.

Conclusions and Outlook

The mechanisms of IMP-fragmentation and the refinement of microstructure via Fe-rich particles has already been confirmed on an industrial scale. Substantially larger primary phases, resulting from industrial solidification rates can be effectively modified into fine particles of 1-2 µm. This offers significant potential for enlarging the property profile of Al-Mg-Si alloys to include greater strain hardening capability and prospects for recycling.

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Alloy design of aluminium alloys with special emphasis on high impurity content in the Al-Mg-Si system.

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Methane pyrolysis for hydrogen production in a liquid metal bubble column reactor

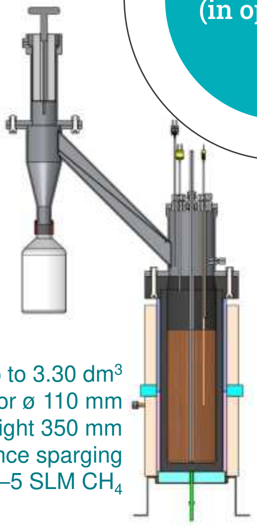
Scale-up steps for process technological development

The pyrolysis of methane (CH_4) represents a new sustainable path to produce hydrogen from natural gas. Compared to established production methods, significantly less CO_2 is generated due to conversion of methane into hydrogen and solid carbon. Among other reactor types, the liquid metal bubble column reactor (LMBCR) represents a promising process for methane pyrolysis. Currently, three different experimental LMBCR-setups are in use at the Chair of Nonferrous Metallurgy. Each setup features an inductively heated graphite crucible, to which a steel structure is attached for gas guidance and carbon output. Setup 1.1 is dedicated to alloy development, as its relatively small melt volume of 0.25 dm^3 provides high flexibility and enables the investigation of high-quality alloying elements. For Scale-up 2.2, dimensions have been increased, the discharge system for carbon and hydrogen products has been adapted, and monitoring for temperature and pressure has been further developed, along with the introduction of a more comprehensive analysis of the product gas. Scale-up 3.1 will be operational starting from July 2024. This setup offers a wide range of new possibilities with an optional gas preheating device, gas injection via bottom sparger, and a mechanical discharge system for carbon. Finally, Scale-up 4.1 embodies a significant step towards the industrial implementation of CH_4 pyrolysis in an LMBCR. This stage is characterized by a notable increase in reactor size and the capability for higher operational pressures. Additionally, it introduces integrated methods for the processing and purification of products, laying the groundwork for an efficient and sustainable, closed-loop production of hydrogen and carbon.

Setup 1.1
(in operation)

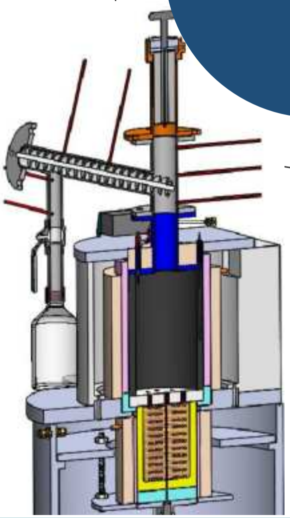
Up to 0.25 dm^3
Reactor ø 65 mm
Bath height 70 mm
Lance sparging
0,1–0,5 SLM CH_4

Scale-up 2.2
(in operation)



Up to 3.30 dm^3
Reactor ø 110 mm
Bath height 350 mm
Lance sparging
1–5 SLM CH_4

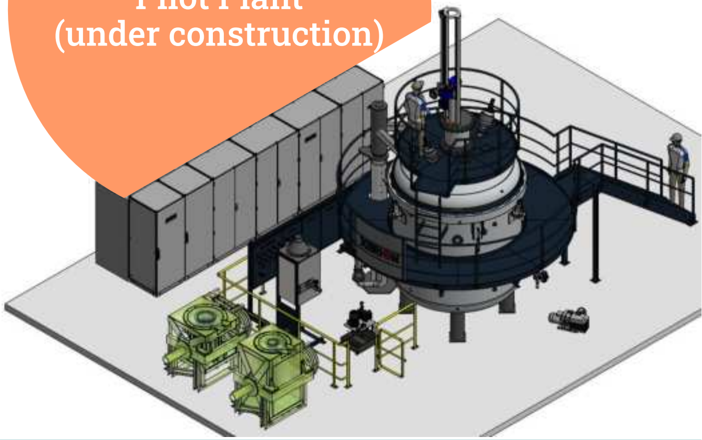
Scale-up 3.1
(under construction)



Up to 4.40 dm^3
Reactor ø 150 mm
Bath height 250 mm
Bottom sparger
Gas preheating
up to 30 SLM CH_4

Scale-up 4.1
Pilot Plant
(under construction)

10–100 dm^3
Reactor ø 150–600 mm
Bath height up to 1000 mm
Bottom sparger
Pressure 1–10 bar
5–400 SLM CH_4



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Research focus

- Methane pyrolysis in liquid metal bubble column reactors

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Radiation-resistant aluminium alloys for space application

Introduction

Humanity is advancing its space exploration efforts through innovations in metallurgy and materials science. Applications such as the Parker Solar Probe or the James Webb Telescope are essential for understanding the genesis of the cosmos and human existence. To endure the extreme conditions in space, materials must be designed from macroscopic features all the way down to the atomic level. This research sheds light on the development of an Al-based crossover alloy that is radiation-resistant.

Theoretical background

- Al-based alloys inherit low density and high strength and thus making them an interesting candidate for applications in space.[1]
- Hardening phases are prone to dissolution upon irradiation due to ballistic mixing. A cascade of point defects is generated and thus dislocation loops are formed that embrittle the material.[2]
- Typical hardening phases dissolve around 0.1 dpa, however, the T-phase ($Mg_{32}(Zn,Al)_{49}$)) exhibits a high Radiation Survivability Level of 24 dpa.
- Ultrafine-grained (UFG) microstructure to decrease radiation-induced dislocation formation. [3]

Experimental

- A merge between 5xxx and 7xxx series Al-alloys was used – the so-called crossover alloy – with 4.8Mg, 3.7Zn, 0.6Cu and 0.17Ag (wt.-%).
- Samples were fabricated via Severe Plastic Deformation using High-pressure Torsion (HPT), see Fig. 1 and Fig. 2.

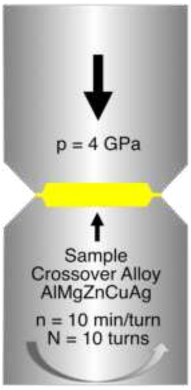

Figure 1: HPT application

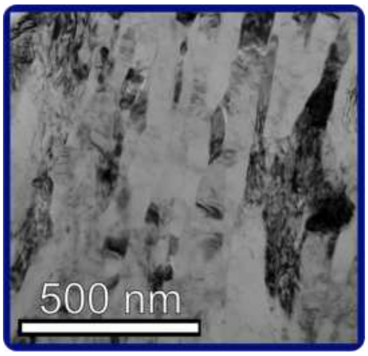

Figure 2: BF-TEM image of the HPT processed crossover alloy.

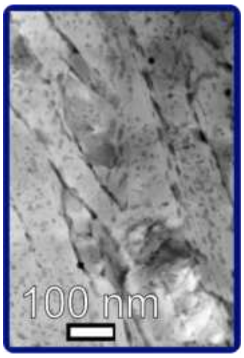

Figure 3: BF-STEM image after heat-treatment.

Results

Fig. 3 shows the nano T-phase precipitates on grain boundaries and within grains. STEM-EDX mappings in Fig. 4 revealed the stability of the T-phase before and after irradiation.

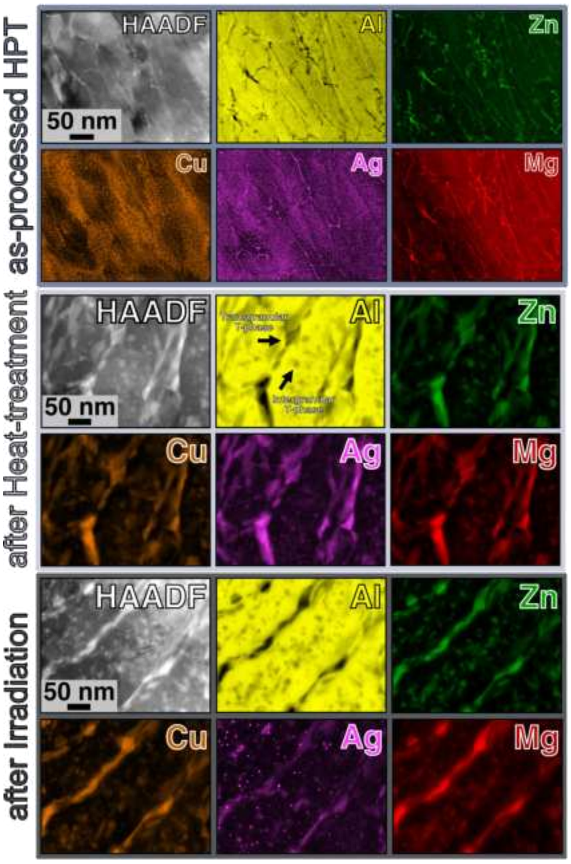

Figure 4: STEM-EDX elemental mapping of the crossover alloy.

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- Investigation on the irradiation resistance of aluminium alloys
- Development of nanocrystalline alloys

European Research Council
Established by the European Commission

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Advancing Sustainability: Organic acid-based lithium-ion battery recycling process

In the quest for sustainable technology, the recycling of lithium-ion batteries is a critical challenge. These batteries, found in devices ranging from smartphones to electric vehicles, contain valuable materials such as lithium, cobalt and nickel. However, traditional recycling methods often involve energy-intensive processes and potentially harmful chemicals, posing significant environmental risks.

The innovative use of organic acids is a breakthrough in environmentally responsible recycling. This approach uses the mild yet effective extraction capabilities of organic acids to recover precious metals without the severe environmental impact associated with conventional methods. By using compounds such as citric acid, which is naturally found in citrus fruits, we can provide a more sustainable way of recycling batteries.


Organic acidic leaching

C, Cu

Removal of contaminants

Mn, Al

Precipitation with oxalic acid

Li

Precipitation with sodium phosphate

Co, Ni

Figure 1: Workflow according to the developed SeLiReco 2.0 process

Experimental

The innovative study, "SeLiReco 2.0", explores the application of citric acid as an eco-friendly leaching agent in the recycling of lithium-ion batteries, targeting a sustainable alternative to the traditional sulfuric acid-based processes. The experimental framework was designed to assess the efficacy of organic acids, with a particular focus on citric acid, in leaching valuable metals such as lithium, cobalt and nickel from spent batteries.


Figure 2: Different types of spent lithium-ion batteries (input material)

Results

The results were promising, with cobalt and nickel leaching efficiencies comparable to inorganic acids and lithium leaching efficiencies slightly superior to inorganic acids. This highlights the potential of citric acid as a viable replacement for sulphuric acid in battery recycling processes. Subsequent stages of the SeLiReco process, including selective precipitation, have been adapted to the organic leaching environment. The quality of the recovered products, such as mixed cobalt-nickel oxalates and lithium phosphate, was rigorously assessed and found to be high, ensuring that the switch to an organic acid does not compromise the overall efficiency of the process or the purity of the recycled materials.

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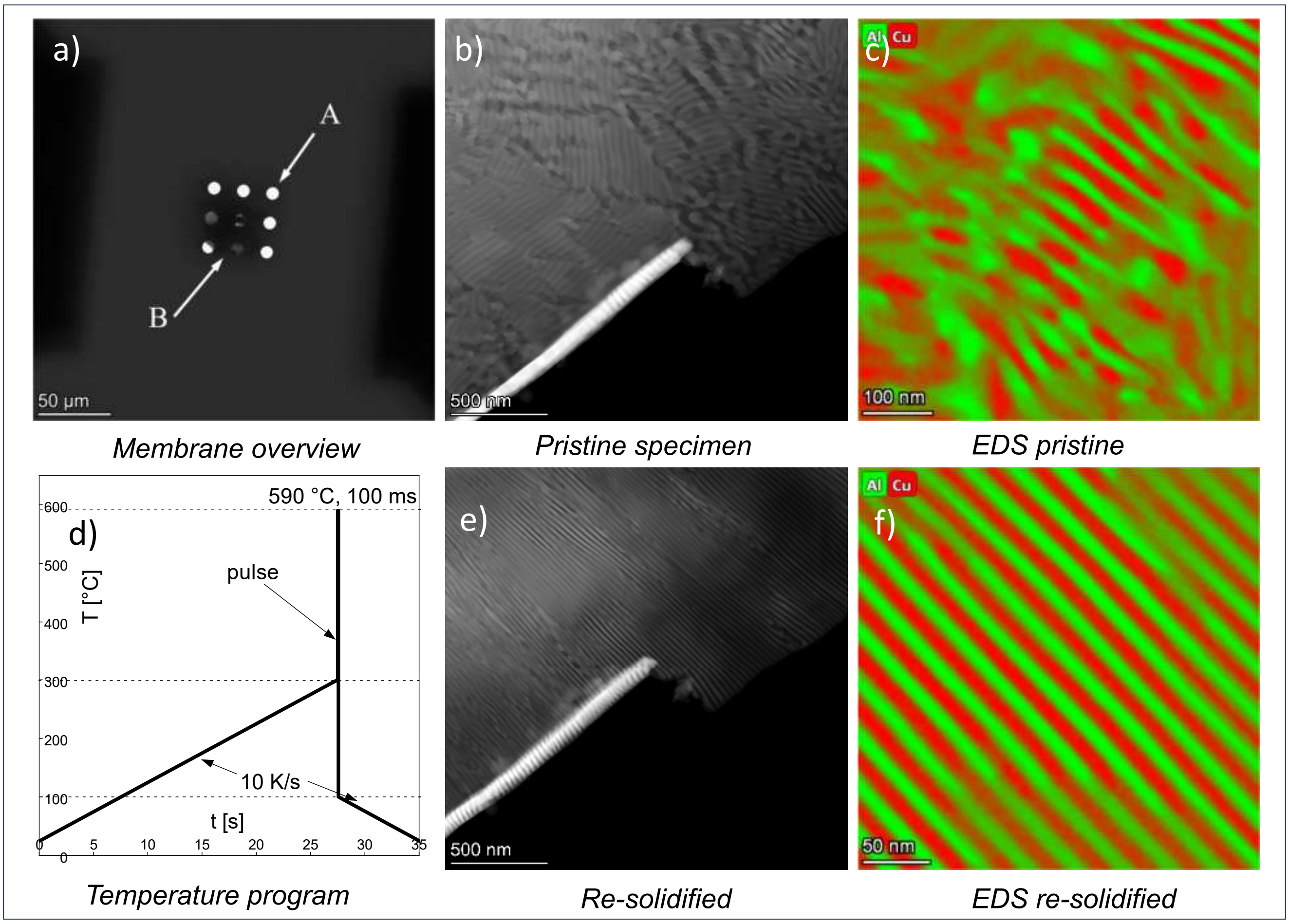
Research focus

- Recycling of lithium-ion batteries
- Recycling of cemented carbides

MEMS-based in situ electron-microscopy investigation of rapid solidification



Membrane overview *Pristine specimen* *EDS pristine*

Temperature program *Re-solidified* *EDS re-solidified*

The solidification behavior of a eutectic AlCu specimen is investigated by in situ scanning transmission electron microscopy (STEM) experiments. Solidification conditions are varied by **imposing various cooling conditions d)** via a **micro-electro-mechanical system (MEMS) based membrane a)** [1]. The methodology allows the use of material processed by a melting and casting route close to industrial metallurgically fabricated material for in situ STEM solidification studies. **Different rapid solidification morphologies b), c), e), f)** could be obtained solely on a single specimen by the demonstrated strategy. Additional post-solidification heat treatments are investigated to observe the spheroidization of lamellas during annealing at elevated temperatures. [2]

Bright areas in high angle annular dark field (HAADF) STEM **b), e)** show the **θ-Al$_2$Cu** and dark areas are **α-Al** phase as identified by EDS **c)** and **f)**. [2]

After *in-situ* re-solidification, a newly formed nanostructured hierarchy consisting of α-Al and θ-Al$_2$Cu unidirectional lamellas is obtained. In **b)** and **c)** lamellar colonies with varying orientation are obtained while after re-solidification **e), f)** unidirectional lamellas are formed. [2]

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Research focus

- Atom probe tomography
- Fast scanning calorimetry / Differential Scanning Calorimetry
- In-situ transmission electron microscopy

[1] Tunes et al., Materials 2021
[2] Dumitraschkewitz et al., Acta Materialia 2022

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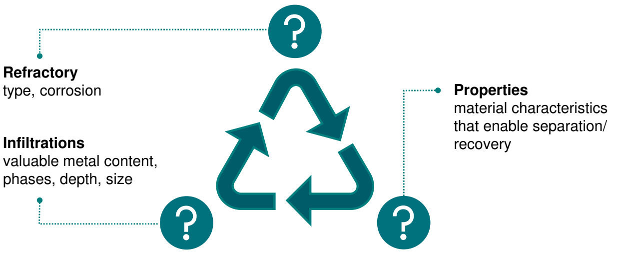
Evaluation of the recycling potential of spent refractories from nonferrous metallurgy

Introduction

Refractories are ceramic materials that are designed to be resistant to thermal stress and to withstand physical wear and corrosion caused by chemical agents. Subsequently, they are indispensable for high temperature processes, playing a key role in the supply of metals, energy, cement, and glass. At the end of their service life, the remaining material arises as spent refractory waste.

Every year, several million tons of this residue are landfilled. This is particularly adverse in nonferrous metallurgy, not only in terms of missing circularity in refractory production, but also because significant amounts of infiltrated valuable metals like Ag, Bi, Cu, Ni, Pb, Sb, or Zn are lost for recovery. To evaluate the recycling potential for both fractions, the refractory components and the infiltrated metals, knowledge regarding the following issues needs to be acquired:



Refractory
type, corrosion

Infiltrations
valuable metal content,
phases, depth, size

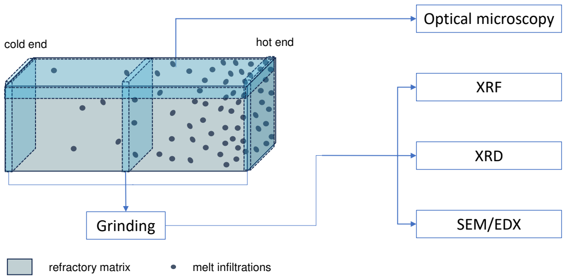
Properties
material characteristics
that enable separation/
recovery

Materials and methods

The investigated spent refractory bricks from various copper and nickel producing furnaces were provided by the industry partner RHI Magnesita.

Metal	Aggregate	Refractory type
Nickel	flash smelter	magnesia-chrome
Copper	shaft furnace, Peirce Smith converter, anode furnace	magnesia-chrome, alumina-chrome

Origin and type of refractories that were investigated in the course of this research

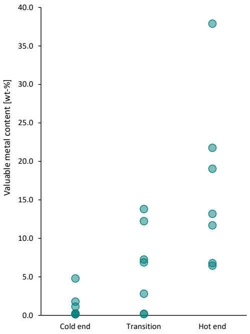


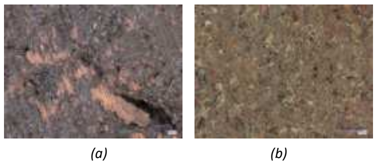
Methodology for the characterization of spent refractory bricks

Characterization methods included optical microscopy (VHX-7000), chemical analysis by X-ray fluorescence (XRF), phase analysis by X-ray diffraction (XRD), and complementary analysis by scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM/EDX, JEOL JSM-IT300).

Results

Chemical analyses revealed that the spent refractory materials contained significant amounts of valuable metals. Nonetheless, they show a broad range of the infiltration characteristics like particle size or phases.





(a) (b)

Form	fine particles … elongated strips
Size	µm … mm … cm
Depth	few cm … whole brick length
Content	0 … 40 %
Phases	metallic – oxidic – sulfidic
Grade of adhesion	free … intergrown

Left: Valuable metal content at different brick positions; top right: Optical micrograph of copper (a) and nickel sulfide (b) infiltrations; bottom right: range of different infiltration characteristics

Conclusion and outlook

Spent refractory materials from nonferrous metallurgy contain valuable metals as well as reusable refractory components to varying degrees. In a next step, holistic recycling approaches considering the complexity and heterogeneity of this residue are developed to recover the fractions in the most sustainable way.

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Christian Doppler Laboratory
for selective recovery of minor metals using
innovative process concepts

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Research focus: Recycling approaches for
spent refractories from nonferrous metallurgy

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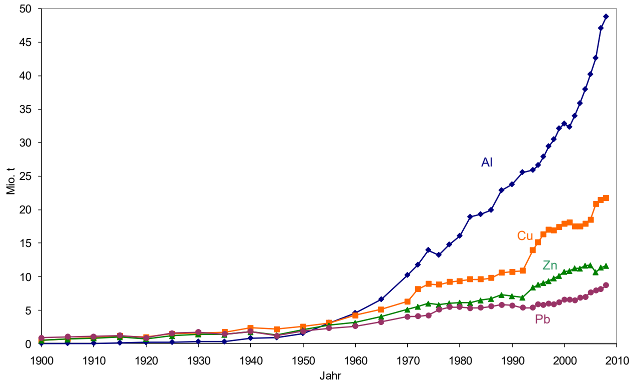
Christian Doppler Laboratory

for selective recovery of minor metals using innovative process concepts

Introduction

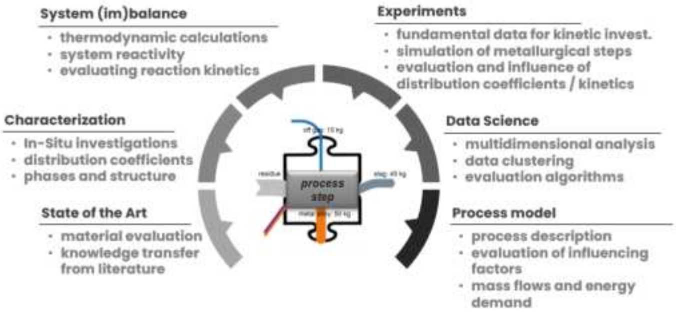
A wide variety of metals of considerable relevance to the European high-tech industry, and therefore also for our society, are supplied by the nonferrous metal industry. As the technologies became rapidly more complex in the last decades, the number and kind of metals and alloys utilized were getting more specialized and unique. With this technological innovation, the demand for minor elements increases steadily. Since their primary production, in most cases, can only be achieved economically as a by-product, it is difficult to respond to peaks in demand for minor elements. This, in turn, underlines the great need for recycling to compensate for these gaps.



In this context, together with the industry partners, methods for determining the distribution of metals in the phases and compounds that occur in industrial intermediates, by-products, and residues are developed and applied, and the possibilities of influencing the behavior in hydro- and pyrometallurgical processes are investigated. This subsequently enables the development of extraction methods for selected elements.

Methods and solution approach

As part of the Christian Doppler Laboratory, primary research is carried out directly on industrial process streams, including thermodynamic calculations and a comprehensive characterization. This builds the fundamental knowledge for the subsequent development of recycling concepts for the targeted valuable elements contained in the materials.



System (im)balance
- thermodynamic calculations
- system reactivity
- evaluating reaction kinetics

Experiments
- fundamental data for kinetic invest.
- simulation of metallurgical steps
- evaluation and influence of distribution coefficients / kinetics

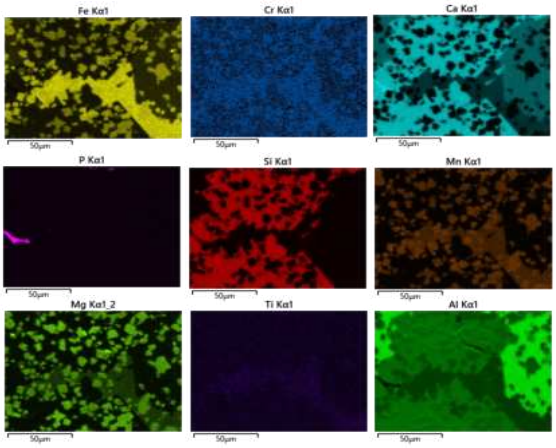
Characterization
- In-Situ investigations
- distribution coefficients
- phases and structure

Data Science
- multidimensional analysis
- data clustering
- evaluation algorithms

State of the Art
- material evaluation
- knowledge transfer from literature

Process model
- process description
- evaluation of influencing factors
- mass flows and energy demand

Since residual industrial materials can differ significantly in their behavior, various innovative processes using chemical and physical properties for separation are used. These include, for example, chlorination of valuables or targeted crystallization during the cooling of slags. The concepts, that can be based on both pyrometallurgical and hydrometallurgical app- roaches, are then evaluated experiment- tally in small-scale setups.



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Sustainable aluminium alloys from the recycling of mixed automotive scraps

Introduction

About one third of the annual global aluminium production of 100 million tons arises from recycling. The automotive sector plays an important role for the sustainability of aluminium alloys as it is the biggest consumer of secondary cast alloys, especially for the internal combustion engine. As this market is expected to shrink, a surplus of scraps that cannot be recycled in a proper way is forecasted.

This work breaks out of the classical, stare scheme of aluminium alloys and presents a way to process secondary wrought alloys from mixed automotive scraps.

Processing

Three different types of automotives are investigated, an average EU-car (EU), a pickup-truck (PU) and an electric car (EC). Three different dismantling scenarios are applied, no dismantling (ND), current dismantling (CD) and theoretical dismantling (TD).

The alloys are melted in an induction furnace and casted in a copper mould. After a homogenization they are hot and cold-rolled. The plates get solution annealed, water-quenched and paint-baked afterwards. The compositions of the alloys are shown in Table 1.

Table 1: Chemical composition of the investigated alloys

Alloy	Si [%]	Fe [%]	Cu [%]	Mn [%]	Mg [%]	Zn [%]
EU ND	5.2	0.5	0.7	0.3	0.8	0.4
EU CD	4.5	0.6	0.7	0.4	0.9	0.4
EU TD	3.8	0.5	0.3	0.4	0.9	0.3
PU ND	4.5	0.5	1.2	0.3	0.5	0.5
PU TD	0.8	0.2	0.1	0.1	0.5	0.1
EC ND	1.4	0.2	0.2	0.2	1.5	0.1
EC TD	0.6	0.2	0.2	0.2	1.6	0.1

Results

The alloys show a good combination of strength and ductility. In the paint-baked state, the tensile strengths range from 200 to 350 MPa with elongations of 19 - 27 %. This is shown in Fig. 1. Fig. 2 shows the microstructural evolution of the alloy „PU ND“. During homogenizing, a strong spheroidization of the silicon is visible, whereas iron-phases remain needle-shaped and are fractured in the finished sheet.

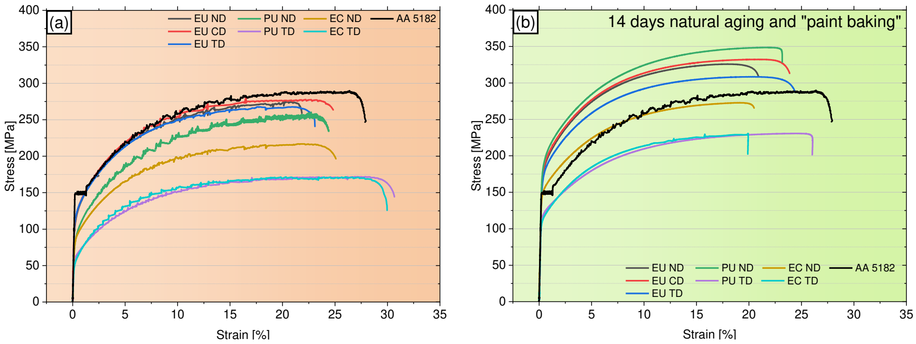

Fig. 1: Engineering stress-strain curves of the alloys investigated in (a) the solution-annealed state and (b) after a paint-bake treatment

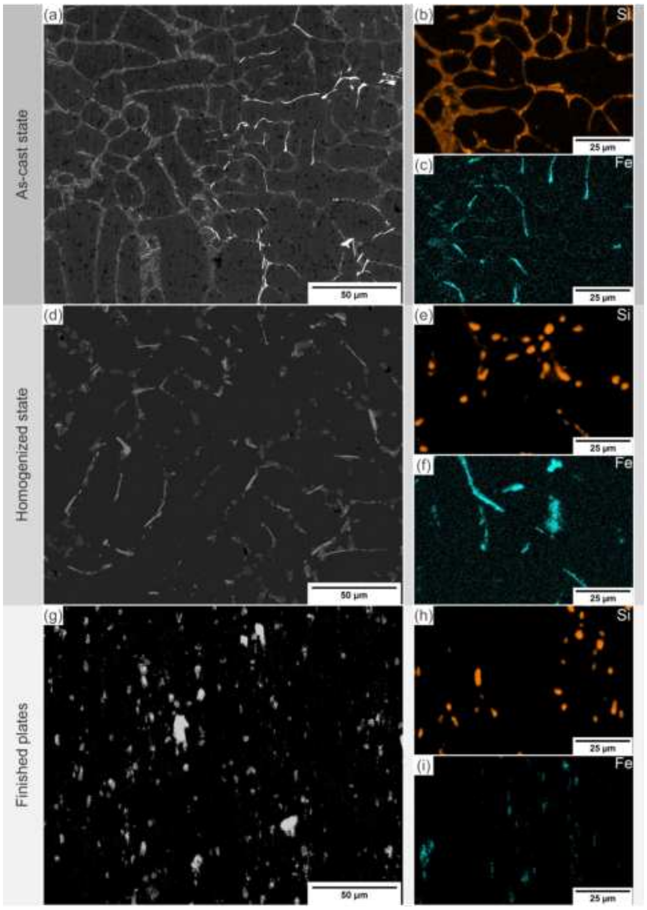

Fig. 2: BSE micrographs of alloy "EU S1" in (a) the as-cast-state, (d) the homogenized state and (g) the finished plate, with corresponding EDX mappings for silicon (b, e, h) and iron (c, f, i)

Discussion

The alloys are excellently hot and cold-rollable and their mechanical performance is much better than one might expect from their chemical composition. At first, the high cooling rates of 60 K/s in the copper mould is beneficial for the refinement of the microstructure. Silicon phases show a strong spheroidization during homogenization leading to higher ductility. The iron containing particles are fractured during rolling in smaller particles with low aspect ratio. Finely dispersed intermetallic phases are reported to limit grain size due to particle stimulated nucleation. The finer microstructure is beneficial for the mechanical properties. This work shows that under certain circumstances it is possible to process mixed automotive scraps directly to secondary wrought alloys.

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- Research focus: Development of sustainable aluminium alloys from the recycling of mixed automotive scraps

Hydrometallurgical treatment of Cr-Ni rich dust

Investigation regarding the extraction of Cr and Ni from stainless steel dust using different acids

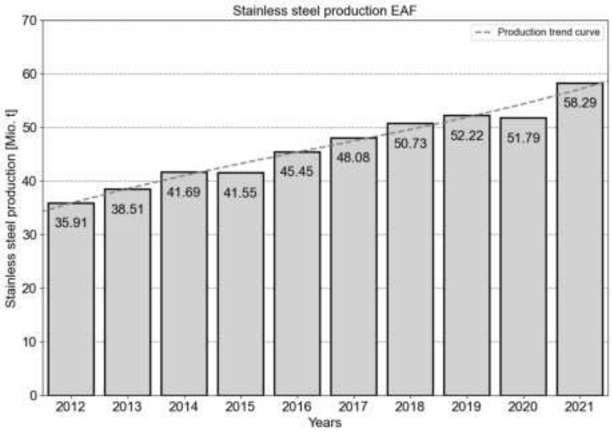

Figure 1: Stainless steel production from 2012 to 2021

In times of growing production rates of various metals and other products, the recycling of residues gains more attention. The corresponding accumulation of residues such as dust, slag and sludge increases directly with these growing production rates. For example, as can be seen in Figure 1, stainless steel production rose by around 6 %/year between 2012 and 2021. The current industrially applied processes to recover valuable metals like Cr and Ni out of stainless steel dust are commonly carbon based as well as energy intensive. Hydrometallurgical processes to recover those metals from dust via acid leaching can offer a more ecological friendly and economical solution to this specific residue. Therefore, the project "HydroStäube", a collaboration of multiple partners, focusses on this promising possibility to develop and evaluate a potential process for such residues. After thoroughly characterizing eight different dusts regarding their identifiable phases (XRD), morphology (SEM), elemental- (ICP-OES) and thermogravimetric analysis, one dust was chosen to conduct leaching experiments with.

The first experiments were conducted to evaluate five different acids, as can be seen in Figures 2 to 3, regarding their potential to leach Cr and Ni. As can be seen in Figures 2 to 3, the most auspicious candidate for relatively mild condition was hydrochloric acid. Based on that, further experiments with HCl are currently conducted under harsher conditions like higher temperatures and longer leaching time, which are already showing promising results. Furthermore, after finding the parameters for maximum extraction, precipitations experiments are being planned.

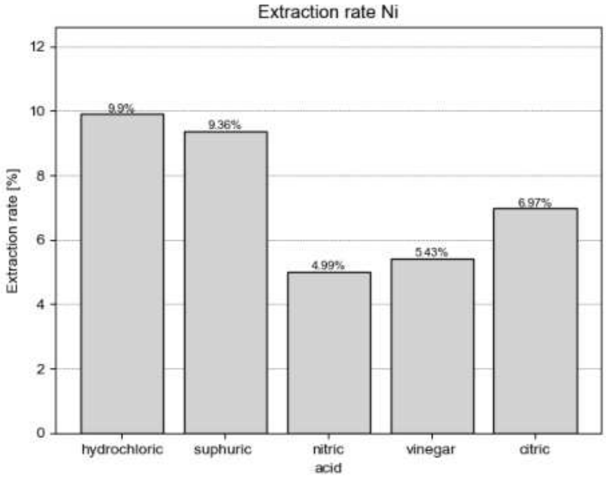

Figure 2: Extraction rate of Ni for different acids

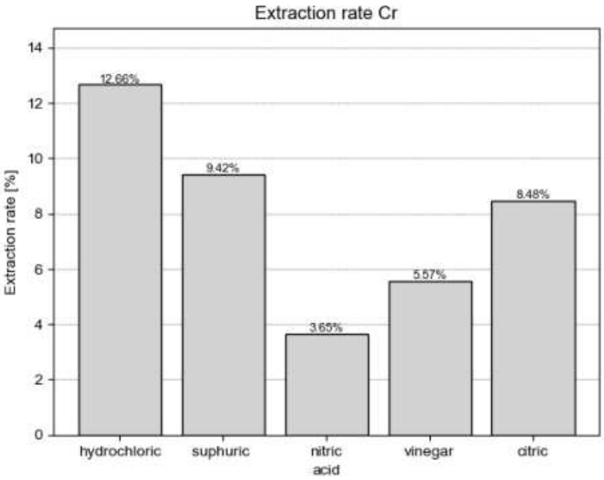

Figure 3: Extraction rate of Cr for different acids

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- Hydrometallurgical extraction of valuable metals from dust
- Precipitation of valuable metals from aqueous solutions

HydroStäube
Das Land Steiermark
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K1 MET
metallurgical competence center
ZUKUNFTSFONDS STEIERMARK

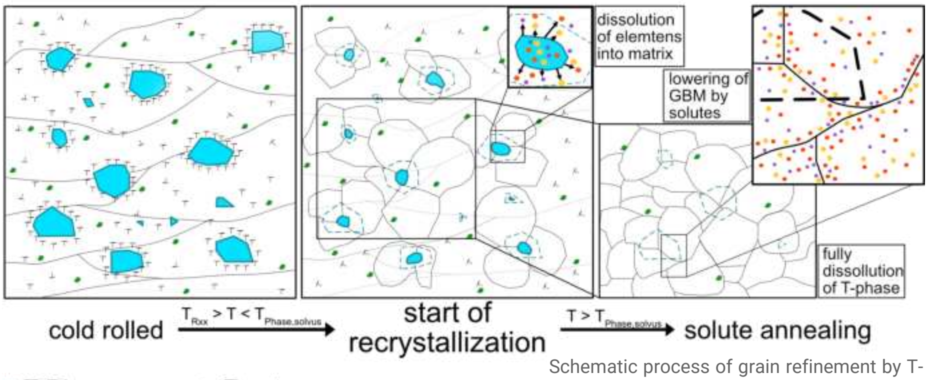
Al-Mg-Zn-(Cu) Crossover alloys

New pathways for aluminium alloys

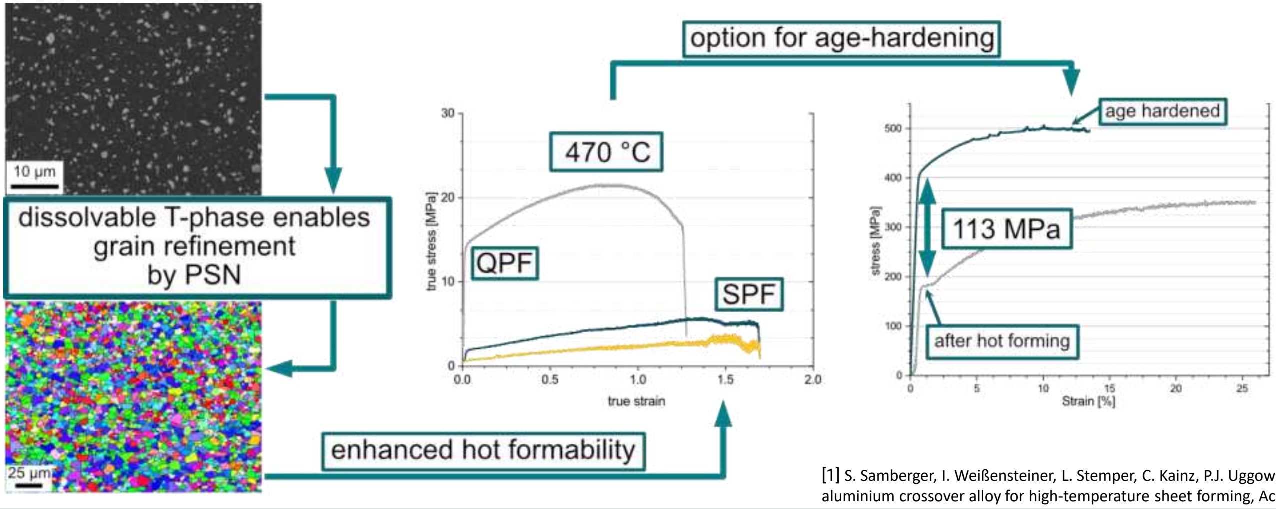
The use of conventional wrought aluminium alloys, especially in the automotive industry, always requires a compromise between the required strength and sufficient formability. The use of aluminium crossover alloys (a combination of 5xxx and 7xxx series alloys) avoids this compromise, as these alloys offer both high strength and excellent plastic formability and are therefore more than capable of meeting the industrial requirements of future lightweight construction applications.

The aim of our work is to improve experimental crossover alloys, which already offer high strength and good plastic or superplastic formability, to meet the extensive requirements of industrial applications. Of particular interest are the challenges of optimum processing and recyclability. For the latter, the influence of tramp elements introduced by recycling processes and their effect on the property profile are of great importance and require intensive research.

In the novel 5/7 crossover alloys, hardening is achieved by precipitation of small T-phase $Mg_{32}(Al,Zn)_{49}$ particles. However, coarse (µm size) T-phase particles can be used after rolling for grain refinement during heat treatment. This fine-grained microstructure gives the alloy superplastic forming properties and allows age-hardening by re-precipitation of small (nm size) particles after forming.



Schematic process of grain refinement by T-phase induced particle stimulated nucleation [1]



T-phase giving the option grain refinement and age hardening after a hot forming process [1]

[1] S. Samberger, I. Weißensteiner, L. Stemper, C. Kainz, P.J. Uggowitzer, S. Pogatscher, Fine-grained aluminium crossover alloy for high-temperature sheet forming, Acta Materialia 253 (2023) 118952.

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Focus:

- Optimization of new aluminium crossover alloys
- Recyclability of aluminium crossover alloys

Metallurgy in Extreme Environments

Space Exploration, Thermonuclear Fusion, Corrosion and Hydrogen

In our journey, we have discovered the pivotal role multidisciplinary science plays in both space exploration and sustainable energy endeavors. We have found ourselves immersed in the world of non-ferrous materials; particularly advanced metallic alloys capable of resisting the deleterious driving forces existing in harsh environments. This summary reflects on our involvement in exploring novel alloy design principles, tackling high-temperature challenges, enhancing corrosion resistance, engineering materials with superior irradiation resistance and pioneering hydrogen-resistant energy materials.

Advanced metallurgy for space exploration and settlement

Space exploration require materials that can endure the harshest conditions mainly posed by highly-energetic proton irradiation from the Sun and its thermonuclear coronal instabilities. We are continuously developing advanced lightweight metal alloy design principles tailored for such demands. Investigating radiation effects in extraterrestrial environments and understanding materials' behavior under non-equilibrium thermodynamics conditions have been integral parts of our research. We are passionate about unraveling the mysteries of space materials' degradation and synergistic effects within the space environment surround Earth, thus contributing to further advancements in the human-based exploration of the solar system.

New materials for thermonuclear fusion and hypersonic flight

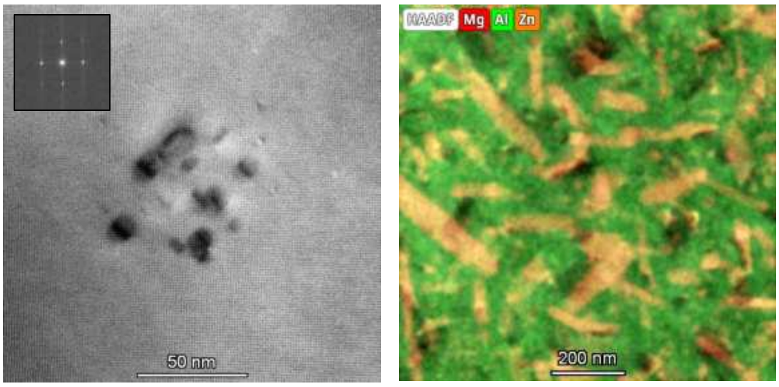
We are developing ultra-high-temperature new materials for fusion energy and hypersonic systems. Recently, we introduced the ZIP phases: a new class of alloys with exceptional thermal stability. These are chemically and structurally complex alloys with distinct atomic-ordering arrangements. Additionally, we explore novel refractory amorphous and crystalline alloys' responses to irradiation conditions simulating future fusion reactors.

Enhancing corrosion resistance in non-ferrous alloys

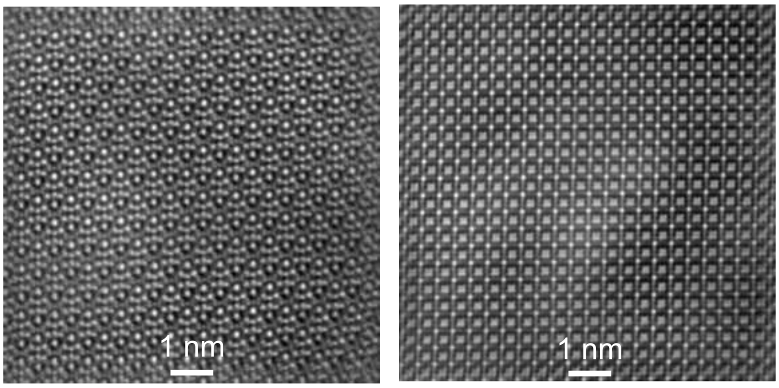
We are advancing anti-corrosion techniques for non-ferrous alloys, especially in aerospace aluminium alloys. Our work with nanomaterials and anodization has shown significant potential in improving corrosion resistance. We've also innovated self-healing materials for corrosion control in various environments at the microstructural level.

Advancing hydrogen-materials research for energy transition

To support the shift towards renewable energy, we are researching how materials interact with hydrogen, focusing on mitigating materials degradation from monoatomic hydrogen. Our projects, HYDROINGENIUS and HYDRONVISION, seek funding to develop new methods for understanding and combating hydrogen embrittlement at the atomic level. Additionally, we aim to create cost-effective, corrosion-resistant materials for proton-exchange membrane fuel cells.

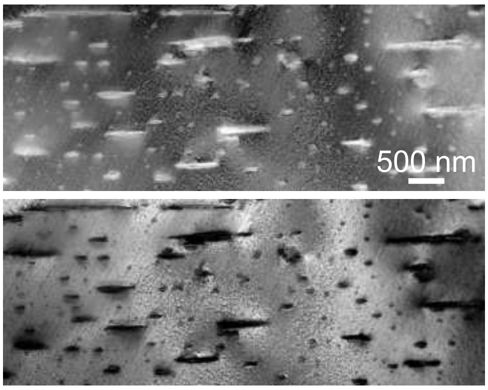


Aluminium is a strategic material for the exploration of the solar system. Space radiation effects in (left) pure aluminium and (right) aluminium alloys is a major focus of our group.



The atomic structure of the Nb3SiNi2: the first Zigzag Intermetallic Phases (ZIP Phases).

Anodization against corrosion.



Hydriding effect on hydrogen-charged zirconium.

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Project partners:

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UNIVERSITY OF BELGRADE
Massachusetts Institute of Technology
Berkeley UNIVERSITY OF CALIFORNIA
AIT AUSTRIAN INSTITUTE OF TECHNOLOGY
University of HUDDERSFIELD
ETH zürich
USP UNIVERSIDADE DE SÃO PAULO

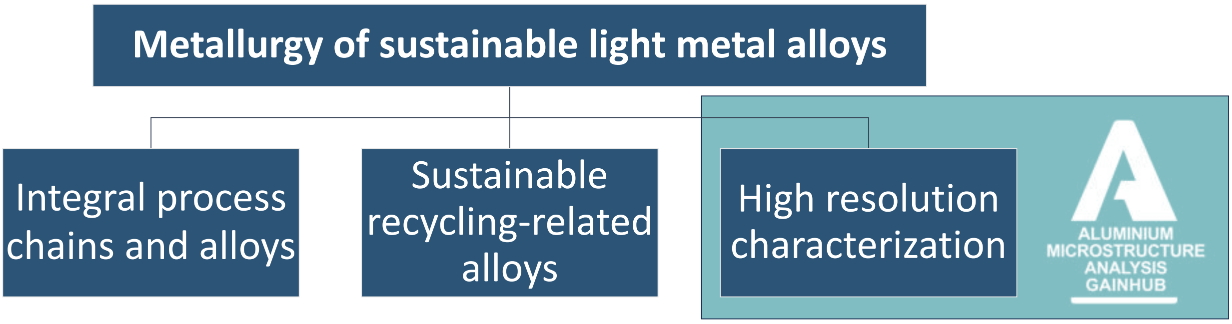
Light Alloys

at the Chair of Nonferrous Metallurgy

Team & Projects
An international team of professors, postdocs, technicians, assistants and doctoral students (currently 11) is working on research questions in the following main projects:

- Christian Doppler Laboratory for Advanced Aluminium Alloys (TripleA)
- Intermetallic Phase Heterostructured Circular Aluminium Alloys (Heterocircal)
- Aluminium Microstructure Analysis Gainhub (AMAGh)

Scientific cornerstones
of the coverd field of metallurgy of sustainable light metal alloys are i) an integral consideration of process chains and alloys of today's production under aspects of sustainability, ii) a new way of thinking about sustainable recycling-related alloys and iii) high-resolution characterisation. Point iii) plays an important role for both approaches i) and ii), but i) and ii) are also closely interconnected, for example by means of the integral consideration of production routes.



Metallurgy of sustainable light metal alloys

Integral process chains and alloys

Sustainable recycling-related alloys

High resolution characterization

ALUMINIUM MICROSTRUCTURE ANALYSIS GAINHUB

Methods
ranging from simulations, melting, casting, heat treatment and rolling to mechanical testing (tensile, hardness and bending test) and microscopic characterisation (SEM-EBSD, S/TEM, APT; all coordinated within AMAGh) are applied. We examine scale-bridging metallurgy of sustainable light metal alloys.

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AMAG ROLLING

Christian Doppler Forschungsgesellschaft

FFG Promoting Innovation.

erc

Hydrogen from Methane Pyrolysis in Liquid Metals

A promising alternative for a sustainable energy transition

Hydrogen is considered a pivotal vector for diversifying and decarbonizing today's energy landscape. However, exploiting this advantage requires an economically feasible, low-emission production route. In this context, methane pyrolysis appears as a promising alternative to prevalent CO_2-intense processes (see figure 1). [1]

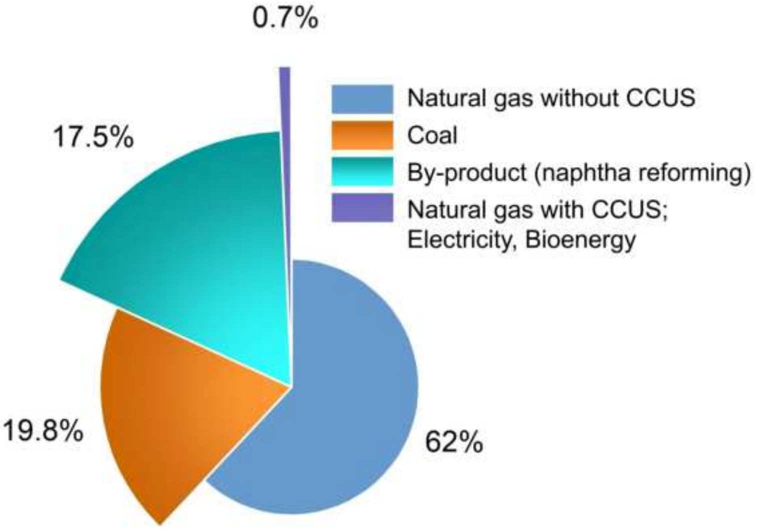

Figure 1: Global hydrogen production in 2022 by technology [2]

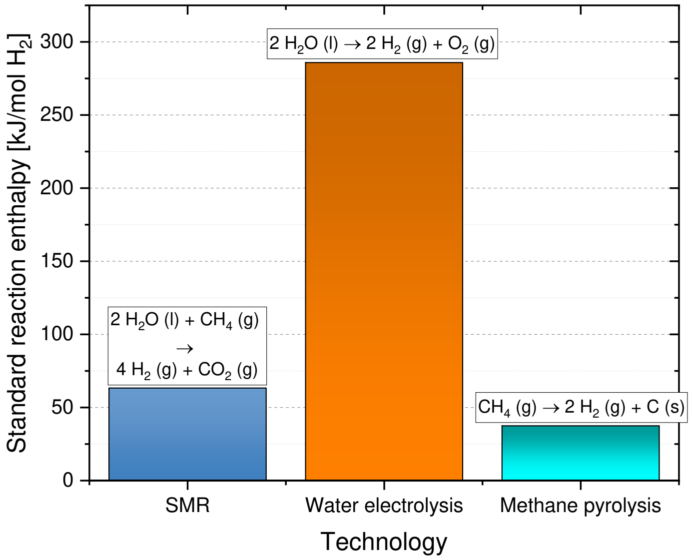

Figure 2: Standard reaction enthalpy of steam reforming of methane, water electrolysis, and methane pyrolysis for hydrogen production (calculated with FactSage™ 8.3)

Methane pyrolysis describes the dissociation of CH_4 at elevated temperatures in an oxygen-free atmosphere. The base reaction generates no CO_2 but solid carbon (see equation (1)), representing a second valuable product besides hydrogen. The standard reaction enthalpy for methane pyrolysis and the values for the benchmark processes, steam reforming of methane, and water electrolysis are depicted in figure 2. The comparison shows the relatively low standard reaction enthalpy of methane pyrolysis, thus underlining its vast potential. [3]

$$CH_4 \rightarrow 2\,H_2 + C \qquad (1)$$

The research conducted by the Chair of Nonferrous Metallurgy focuses on methane pyrolysis in liquid metallic catalysts, which offer essential benefits, e.g., the prevention of deactivation due to the accumulation of pyrolysis carbon. Experiments show that the choice of the melt can influence product quantity and quality (see figure 3).

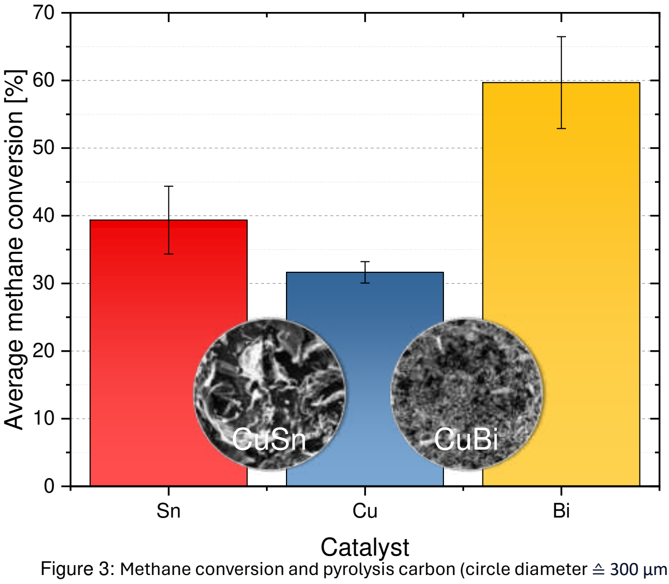

Figure 3: Methane conversion and pyrolysis carbon (circle diameter ≙ 300 µm) resulting from experiments with different catalysts

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Sources:
[1] IEA. *Global Hydrogen Review 2023*IEA: Paris, 2023
[2] IEA. *Global hydrogen production by technology in the Net Zero Scenario, 2019-2030*IEA: Paris, 26.10.2022
[3] Scheiblehner, D., Antrekowitsch, H., Neuschitzer, D., Wibner, S. and Sprung, A. *Hydrogen Production by Methane Pyrolysis in Molten Cu-Ni-Sn Alloys. Metals* 2023, *13, 7*, pp. 1310. doi:10.3390/met13071310

Cemented Carbide Recycling

The zinc process in the gaseous state

- Cemented carbides (CC), a composite material, consist of a hard phase (tungsten carbide) embedded in a ductile metallic binder (cobalt) (Fig. 1).
- Recycling of CC is an indispensable aspect of conserving critical raw materials, highlighting its profound environmental, economic and strategic importance.
- The zinc process for recycling chunky hard metal scrap is considered the best possible method for recycling cemented carbides due to the direct recovery of the input materials (Fig. 2).
- Zinc is loaded into a sealed graphite crucible with scrap and heated up to around 1000 °C. It then diffuses into the CC, to react with the binder metal.
- The formation of intermetallic Co-Zn phases during the disintegration leads to the spalling of carbide lamellae due to the higher volume of these phases compared to pure cobalt (Fig. 3).

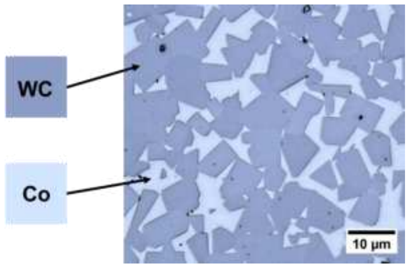


Fig. 1: A schematic display of a CC microstructure

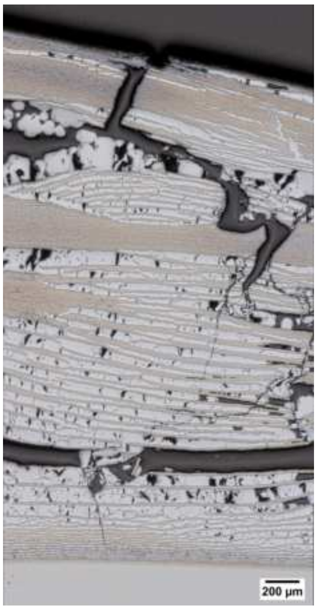
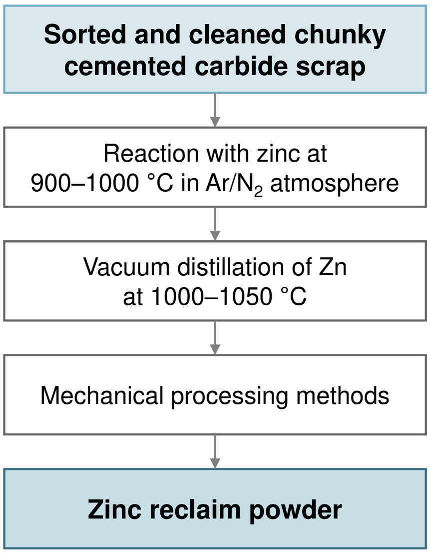


Fig. 3: CC sample before and after disintegration with zinc (left) and LOM image of the disintegration layer with the alternating layers of the Co-Zn phases and the carbide lamellae (right)



Sorted and cleaned chunky cemented carbide scrap

Reaction with zinc at 900–1000 °C in Ar/N_2 atmosphere

Vacuum distillation of Zn at 1000–1050 °C

Mechanical processing methods

Zinc reclaim powder

Fig. 2: A simplified process flowsheet of the zinc process for recycling chunky CC scrap

- After removing zinc using vacuum distillation, the remaining porous sponge can be converted into a powder through mechanical.
- The so-called zinc reclaim powder shares the same chemical composition as the feed material and is directly used in the production of new CC.
- Due to the high temperature during disintegration, zinc is present as a mixture of liquid and gas. Since this step is a diffusion-controlled process, utilizing the kinetics is very advantageous.
- Through adaption of the experimental set-up, it is possible to contact cemented carbides with gaseous zinc only.
- Using zinc vapor, better diffusion kinetics allow for higher disintegration rates. Liquid zinc infiltrates CC at 1 mm/h compared to approximately 3 mm/h when gaseous Zn is used.
- The change of the aggregate state of zinc during the disintegration allows a shorter duration of the process, resulting in considerable energy savings during the recycling of cemented carbides.

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Poster Exhibition 2024

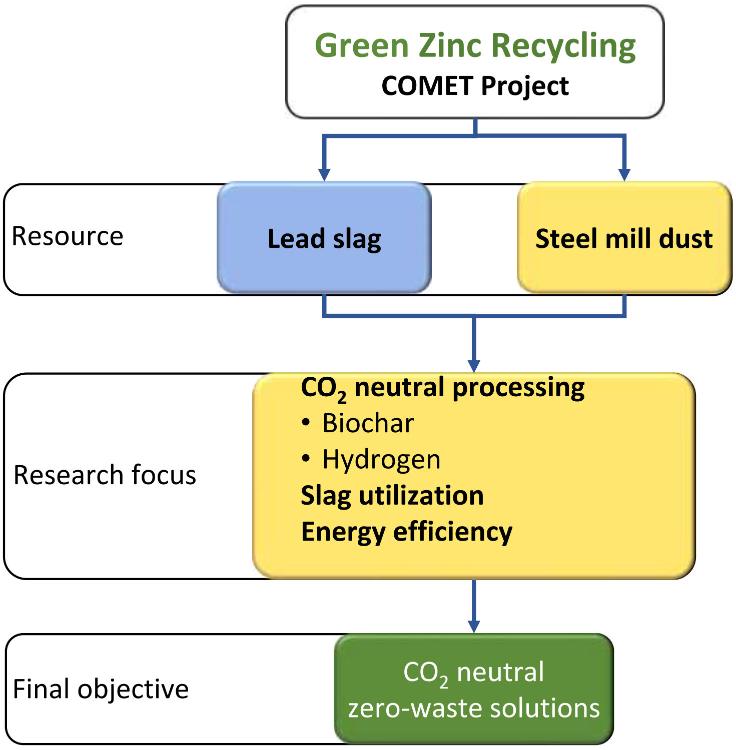
MONTAN UNIVERSITÄT LEOBEN

Science 4 Technology @MUL

CO_2 Neutral Zero Waste Solutions for Zinc Recycling

Introduction

Zinc, a vital metal in society, is extensively used for corrosion protection for steel. Beyond corrosion protection, zinc finds application in industries such as rubber, ceramics, fertilisers, casting products, pharmaceuticals, and nutritional supplements. The European zinc demand ranges from 2.4 to 2.7 Mio. tons/a. Unfortunately, Europe is weak in primary zinc resources. However, it is very often present in by-products from metallurgical industry. Due to its volatile character, zinc is frequently found as oxide in the dust of steel recycling facilities. Furthermore, slags from the lead industry also contain interesting amounts of zinc. Such by-products have the potential to contribute essentially to the European demand.



Green Zinc Recycling
COMET Project

Resource

Lead slag

Steel mill dust

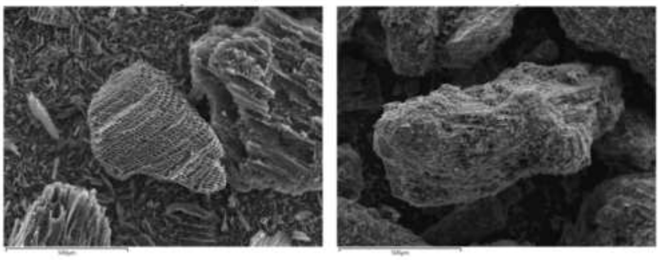
Research focus

CO_2 neutral processing
- Biochar
- Hydrogen

Slag utilization
Energy efficiency

Final objective

CO_2 neutral
zero-waste solutions



SEM images of two biocoke samples. The left image shows an original biocoke, the right image a sample with modified surface.

To counteract CO_2 emissions, the use of hydrogen is proposed. However, this is far from getting operational within the next decade, due to the lack of corresponding infrastructure, hydrogen production and hydrogen based metallurgical processes. A faster realisable alternative is coke made by biomass pyrolysis, exhibiting similar behaviour compared to fossil carbon carriers. The project aims to modify bio-coke to meet requirements for the implementation in metallurgical processes, and to modify treatment procedures for slag conditioning for the utilisation in the construction industry, promoting a zero-waste approach. The research explores short-to-mid-term and long-term solutions for CO_2 neutrality and a zero-waste concept, involving eight partners from industry to guarantee applied research.

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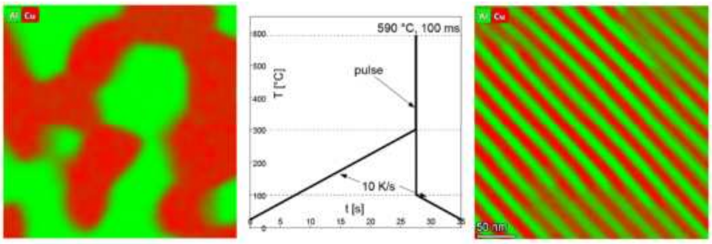
In situ electron microscopy

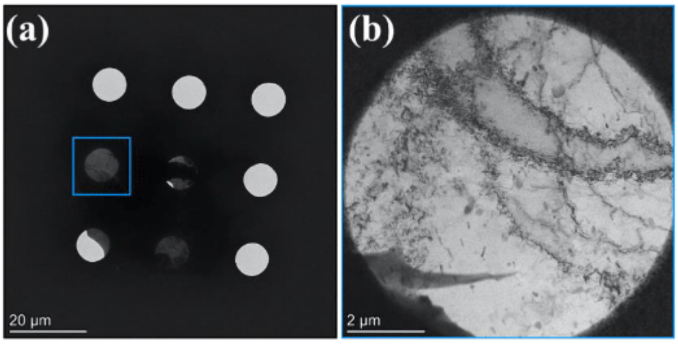
A laboratory on a chip

Transmission electron microscopy (TEM) is an advanced technique, which enables imaging, crystallography and chemical analysis localized at the nano-atomic scale. Since the newest generations of spherical aberration correctors, incredibly high resolution imaging (~0.063 nm) is routinely feasible. Due to this fact, the focus shifted from just increasing resolution further to other topics: resolution improvements at lower acceleration voltages; high-speed imaging; live observation of processes in materials during heating, electrical biasing, fluid and gas exposure (*in situ* experiments).



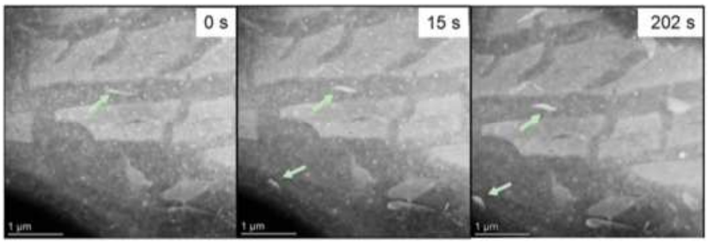
Re-solidification of an Al-Cu alloy with extremely high cooling rate left to right: coarse initial microstructure; spike heating temperature program; fine lamellar eutectic structure

Re-arranged from: Dumitraschkewitz, Tunes et al. MEMS-Based in situ electron microscopy investigation of rapid solidification and heat treatment on eutectic Al-Cu, Acta Materialia 239 (2022), 118225



a) Sample on the surface of a heating chip (with electron transparent holes)
b) TEM bright field image of the sample microstructure

Tunes, Quick et al. A Fast implementation-Free Sample production method for Large Scale Electron-Transparent Metallic Samples destined for MEMS-Based in Situ S/TEM Experiments, Materials 14 (2021), 1085, p. 1-9



Annealing of the Al-Au system at 460 °C after melting of Au nanoparticles on an Al sample
Image series: Dissolution of smaller Al-Au precipitates and growth of larger ones

Re-arranged from: Coradini, Tunes et al. In situ transimission electron microscopy as a toolbox for the emerging science of nanometallurgy, Lab on Chip, 14 (2023), 3186-3193

Especially live observation of material processes in changing environments is hot topic in recent years. Active research and development of these *in situ* methods decreased the instability and drift during experiments which facilitated data acquisition and evaluation. This resulted both in increased demand and availability of so called chip-based systems, where heating, electrical biasing or fluid exposure can be integrated on chips much smaller than a fingernail.

Conclusion

In situ experiments on small chip based systems have wide applications in materials science, including mechanical properties, corrosion, chemical reactions during catalysis and local measurement of electrical parameters. Live observation within the microscope permits the exact tracking of the onset of microstructural changes and the focus on specific regions of interest in detail. This leads to insights into microstructural changes and their relation to environmental or processing parameters previously not possible.

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- Electron Microscopy (SEM, TEM)
- *In situ* TEM (heating, electrical biasing)
- Advanced material characterization
- Alloy development (Al, Mg)

Additional information:

www.3dnanoanalytics.at

Demonstration plant for hydrogen and carbon production via methane pyrolysis

At the Chair of Nonferrous Metallurgy, methane pyrolysis is investigated in molten metal reactors, where the used metal or alloy has a catalytic effect on CH_4 decomposition. Technically mature laboratory plants already exist for this purpose. Thereby, research focuses both on optimising the composition of the catalytically active metal alloys and on upscaling the corresponding laboratory plants. Based on this preliminary work, a Hydrogen Research Centre (HRC, Figure 1) will be set up in which various pyrolysis technologies (liquid metal bath, plasma) and complete product gas processing for the production of carbon and hydrogen of varying purity can be investigated experimentally in collaboration with other departments. The demonstration plant (Figure 2) planned at the HRC consists of different units that are necessary for a comprehensive investigation of various process parameters that affect methane pyrolysis. The central metallurgical unit is an induction melting plant (Figure 3), which can be operated at pressures of up to 10 bar. A plasma reactor can also be operated instead (Chair of Ferrous Metallurgy). The hot gas filter with heat exchanger is planned for the subsequent product stream treatment to separate the solid carbon produced during methane decomposition. The particle-free gas stream, which essentially consists of hydrogen and unreacted CH_4, can be separated using a membrane process in order to enrich H_2. Furthermore, it is possible to completely neutralise all products generated during research with the help of a thermal post-combustion chamber. Comprehensive exhaust gas analysis with appropriate measurement technology with regard to the quantity, temperature and composition of the product gas forms the basis for the calculation of gas conversion rates and the determination of mass balances, among other things. This allows a well-founded evaluation and interpretation of the test results as well as the targeted control of the processes.

Figure 1: Hydrogen Research Centre

Figure 3: Induction melting plant

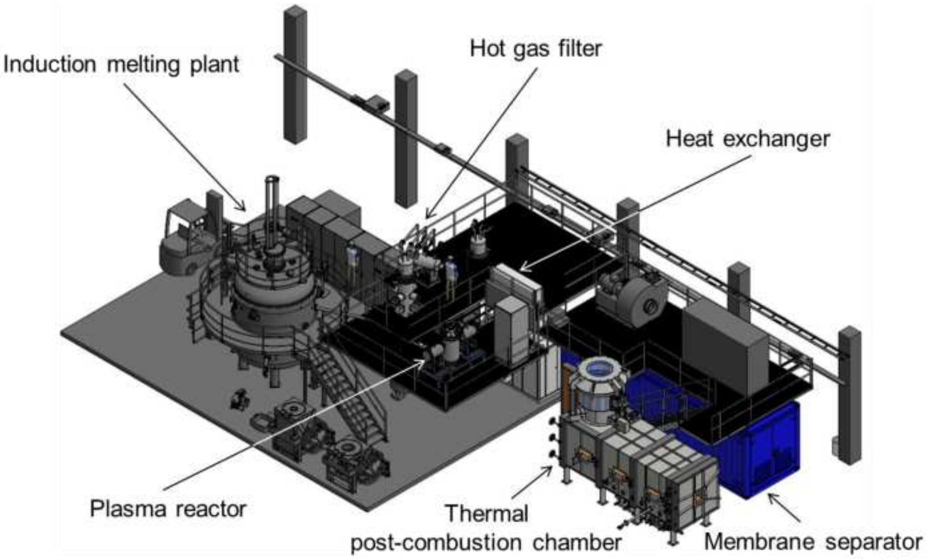

Figure 2: Demonstration plant for methane pyrolysis

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Recycling of red mud from the aluminium industry

Reduction of the iron oxide contained in the red mud under adjustment of a desired slag composition

Primary aluminium production is divided into two process stages, with high-purity aluminium oxide being produced in the first one. In the Bayer process, bauxites are digested with aqueous caustic soda at an elevated temperature and pressure. The second step involves the fused-salt electrolysis of the oxide, which produces aluminium with a purity of 99.7–99.9%. The Bayer process generates a residual material, the so-called red mud, in quantities of 0.4–1.4 t/t Al_2O_3, with an average composition of 20–50% Fe_2O_3, 20–30% Al_2O_3, 5–9% Na_2O, 5–20% SiO_2 and 3–12% TiO_2.

The focus of the experiments carried out as part of this research project is on the production of a saleable slag for utilisation in the cement industry with simultaneous reduction of the iron oxide contained in the red mud to metallic iron. The latter can be reduced by adding graphite and a slag adapted to the desired composition (similar to slag sand) is produced by addition of lime and other slag formers. Furthermore, the slag should have a glassy character due to rapid cooling.

Experimental

In order to be able to carry out the corresponding reduction tests, the red mud used is first crushed manually. The corresponding materials are shown in Figure 1.

Figure 1: Red mud used, before and after crushing

The quantities of quartz sand SiO_2, quicklime CaO, magnesia MgO and graphite C required to achieve the slag composition and the reduction reaction are then determined and the input materials are mixed together. The melting process of this feed takes place in an induction furnace at 1500 °C (graphite crucible), with a reaction time of 30 minutes after reaching this temperature. The melt is then poured into a water bath in order to ensure a fast solidification and thus adjust the amorphous structure of the slag. Figure 2 shows the feed mixture for the melting experiments and the following quenching process.

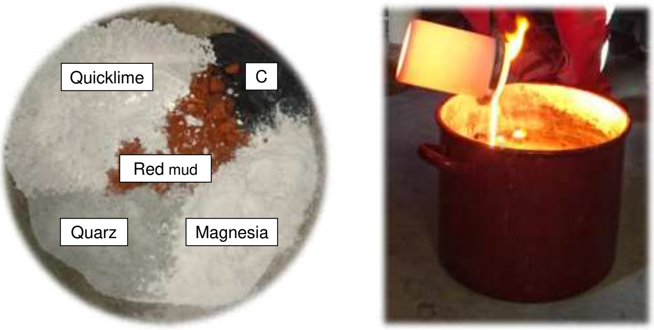

Figure 2: Feed materials for the melting experiments and quenching the melt in a water bath

Results

The mass flow diagram for one of the successful melting experiments is shown in Figure 3.

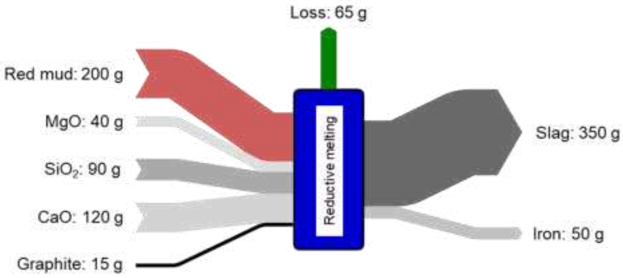

Figure 3: Mass flow diagram for a reductive melting experiment

The experiments show that red mud can be recycled by adding graphite and slag formers. The products are metallic iron and a slag that has the same properties as granulated blast furnace slag and can therefore be used in the cement or brick sector.

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Research focus:

- Recycling of aluminium containing materials
- Methane pyrolysis in molten metal reactors

Process optimisation of aluminium recycling

Influence of the furnace atmosphere on the pyrolysis of aluminium scrap

Recycling of aluminum scrap mainly takes place in chamber furnaces or tilting rotary drum furnaces, which are operated with natural gas. Organic material adhering to the scrap is utilised as an energy source in these furnaces to substitute fuel. As a result, natural gas can be saved and the process can be run almost autothermally. Nevertheless, fuel-based CO_2 in considerable figures is emitted. Furthermore, what if the availability of natural gas diminishes due to geopolitical reasons or depleting resources?

An alternative energy source for smelting is necessary – and it could be electricity, as it is a clean form of heating. However, there are currently no technological concepts to heat industrial melting furnaces exclusively with electrical energy. Another feasible option is hydrogen, which emits only water and no fuel-based CO_2. But there is a lack of research on the influence of H_2O in the pyrolysis process of contaminated aluminium scrap, especially when combined with parameters like temperature, duration or complex atmosphere composition.

Lab-scale tests by thermogravimetric analysis (TGA) show the effect of temperature as basis for further investigations. Specimen of used beverage cans (UBC) with a weight of 0.32 g/test are used, as shown in Fig. 1.

Fig. 1: Pictures of the aluminium platelets cut out from UBCs after pyrolysis; from left to right: original material, after 400 °C, after 450 °C, after 500 °C, after 550 °C

Upscaling from laboratory scale to a technical scale leads to a sample mass of 25 kg per test.

On the technical scale, industrial scrap is pyrolysed in a resistance-heated furnace up to 600 °C. The scrap before and after the process is shown in Fig. 2.

Fig. 2: Industrial scrap before (left) and after (right) pyrolysis at 550 °C for 6 h

The main objective is to analyse the formed off-gas, and the mass change during the process. The off-gas composition provides astonishing information, such as time course and quantities (see Fig. 3), allowing further assessment of scrap and processes.

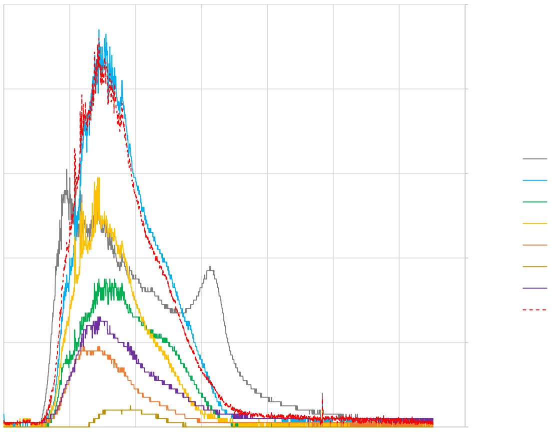

Fig. 3: Off-gas analysis by FTIR-spectroscopy showing several components and the accumulated calorific value; experiment with simulated atmosphere of a hydrogen-air-burner

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Research interests

- Recycling processes, focus on Cu and Al
- Process optimisation, towards zero waste, lower energy consumption and environmental protection

NICHTEISENMETALLURGIE · NONFERROUS METALLURGY

AMAG AUSTRIA METALL

Decarbonisation of Aluminium-Recycling

Substitution of Natural Gas (NG) with Hydrogen (H_2)

About 5 million tonnes of aluminium are recycled in the EU every year [1]. Various melting and holding processes generate approx. 0.3 t CO_2e/t Al [2]. The primary source of these emissions is the use of natural gas (NG) with methane (CH_4) as its main component for heating the furnaces. As can be seen in the following reaction, the combustion of 1 Nm³ CH_4 releases the same amount of carbon dioxide (CO_2).

$$CH_4 + 2\,O_2 \rightarrow CO_2 + 2\,H_2O$$

Hydrogen (H_2) is particularly suitable as alternative fuel to NG for decarbonisation. The combustion of green H_2 (reaction below) does not produce any CO_2 emissions. However, the content of water vapour (H_2O) in the furnace atmosphere increases by 50–85 %, depending on the burner setup.

$$H_2 + \tfrac{1}{2}\,O_2 \rightarrow H_2O$$

Due to the high reactivity of liquid aluminium, H_2O molecules react with the molten metal and are converted to H_2 and Al_2O_3 (Fig. 1). The formed hydrogen can subsequently be absorbed by the melt. Before solidification, it is imperative to reduce dissolved hydrogen [H] to minimum values with gas purging treatments. Otherwise, pores and other defects may be formed in the cast products during solidification.

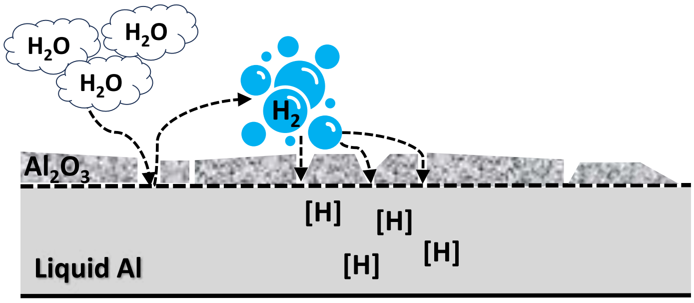

Fig. 1: Schematic reaction of liquid Al with water vapour

The project investigates the metallurgical effects of a changed furnace atmosphere due to the application of an alternative fuel (H_2 instead of CH_4).

In addition to the hydrogen content, special consideration is also given to the oxidation behaviour of the liquid metal. Even a slight increase in oxidation, referred to as dross formation, would impair the yield of industrial processes and thus their economic efficiency.

The conditions in various laboratory furnaces (Fig. 2 and 3) are modified to simulate industrial plants as close as possible.

Fig. 2: Lab-scale hearth furnace for wrought alloys

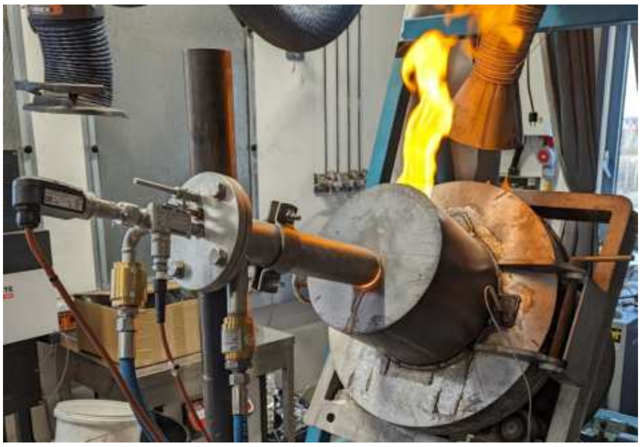

Fig. 3: Lab-scale tilting drum furnace for cast alloys

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[1] https://european-aluminium.eu/about-aluminium/aluminium-industry/ (11.04.2024)
[2] https://international-aluminium.org/statistics/greenhouse-gas-emissions-aluminium-sector/ (11.04.2024)

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Research areas

Influence of the furnace atmosphere in melting and holding processes on melt quality and oxidation of aluminium melts

AMAG
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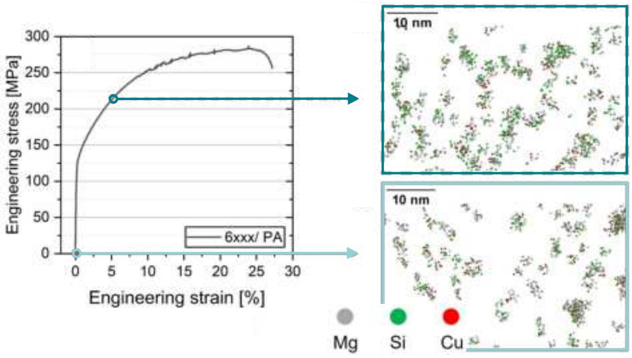
Aluminium Microstructure Analysis Gainhub - AMAGh

The world of aluminium is currently in a state of change, boundaries of the known alloys and their classes are being pushed to (i) increase performance (e.g. the cross-over concept) and (ii) realize alloys with high recycled content. The microstructure is a key factor for the physical and chemical properties of these special Al-products.

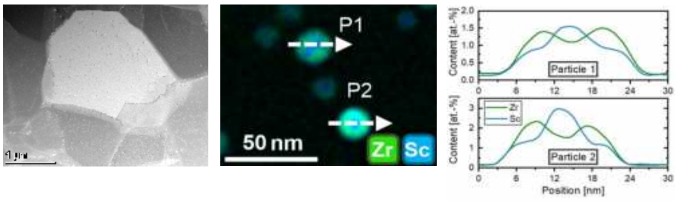
ALUMINIUM
MICROSTRUCTURE
ANALYSIS
GAINHUB

The **Aluminum Microstructure Analysis Gainhub (AMAGh)** enables highly qualified young scientists to learn, apply and develop state-of-the-art materials characterization techniques such as electron microscopy and atom probe tomography to advance the fundamental understanding of the effects of microstructural constituents and to contribute to the scientific and technological advancement of Al-alloys.



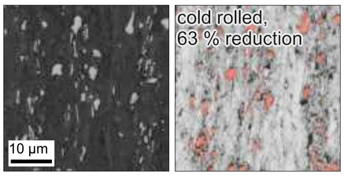
The **3D structure of finest microstructural features**, such as clusters or hardening phases can be characterized at the nanometer scale using **atom probe tomography** (**APT**, field evaporation of individual atoms). These individual signals are then reconstructed into a 3D model of the sample. In the given example Aster et al. used APT to show deformation-induced clustering in a 6xxx alloy.

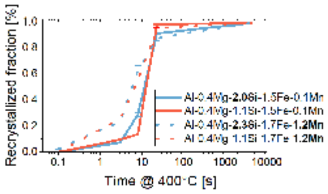
P. Aster, P. Dumitraschkewitz, P. J. Uggowitzer, F. Schmid, G. Falkinger, K. Strobel, P. Kutlesa, M. Tkadletz, and S. Pogatscher, (Materialia, 2023).

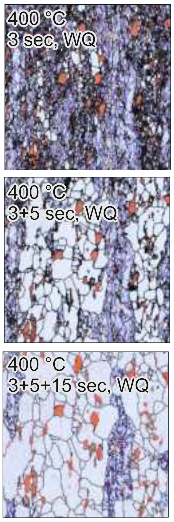


Hardening phases, dispersoids, nanocrystalline materials, single dislocations are ideally analyzed by transmission electron microscopy. In the present example of a ternary Al-Zr-Sc alloy, automated **STEM tomography** allowed to 3-dimensionally study the **core-shell morphology** of the precipitates and to establish a correlation between heterogeneous nucleation sites (e.g. grain boundaries, dispersoids) and the distribution of precipitates.

F. Schmid, D. Gehringer, T. Kremmer, L. Cattini, P. J. Uggowitzer, D. Holec, and S. Pogatscher, Materialia 21, 101321 (2022)







Exploring the recrystallization behavior of a 6xxx alloy (Al-0.4Mg-2.3Si-1.7Fe-1.2Mn), high in primary and secondary precipitates , involved **stepwise annealing** of cold-rolled samples**, accompanied by detailed EBSD/ EDX analyses**.

Particle-stimulated nucleation (PSN) was identified as the primary origin of nucleation, the efficiency of dispersoids in retarding nucleation could be shown to depend on the surrounding matrix, and the effect of microstructural constituents on the final texture (randomization due to PSN) was revealed.

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Spectrum of Methods:

- Scanninng electron microscopy
- Transmission electron microscopy
- Atom probe tomography
- Degradation and corrosion of Al- and light metal alloys

Christian Doppler Forschungsgesellschaft

AMAG ROLLING

Microstructures and Formability in Novel Aluminium Alloys

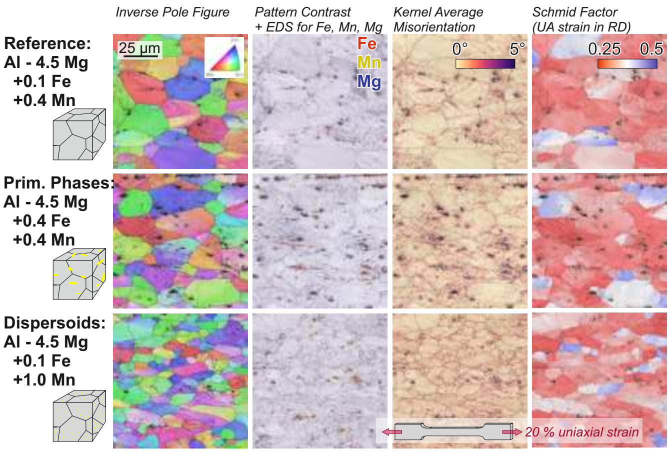
Insights from EBSD

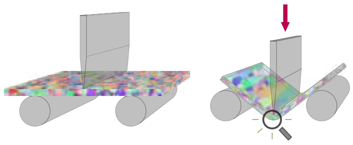
Our research is focused on advancing **high-performance secondary aluminum alloys** to reduce emissions and production costs without compromising material quality, despite the presence of tramp elements.

Development of "composition tolerant" alloys that can accept a wide range of processing conditions, requires a **thorough understanding of the effects of microstructural constituents** (rather than just alloying elements) is critical, as they can affect, dislocation behavior, recrystallization or formability of the material.

The following electron backscatter diffraction **(EBSD)** datasets show how **tramp elements affect the microstructure and** therefore the **dislocation mobility** in an Al-4.5 Mg alloy.
The microstructure has been varied by the **addition of Fe** (forms primary phases) **and Mn** (forms dispersoids), the latter significantly refining the Al grains. The initial texture is weak (due to the intermetallics) and similar for all alloys. Mechanical contrasts are strong between the intermetallics and the Al matrix and weak between differently oriented grains (Schmid factors).

Differences in plastic behavior can be seen **at 20 % uniaxial tensile loading** (shown below): Primary particles lead to a local increase in geometrically necessary **dislocations, while dispersoids homogenize the local deformation.**

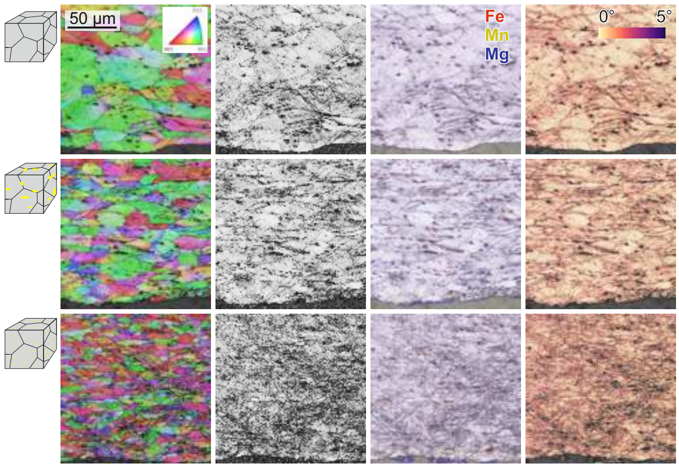


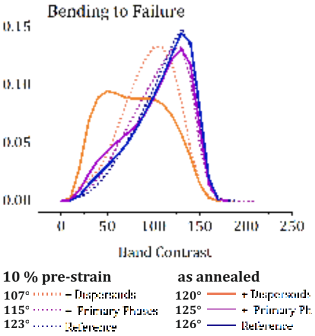


Bending causes high strains in the outer layer of a sheet, initiating surface undulations, strain localization, delamination/fracture of intermetallic particles, and shear band (SB) formation, preceding material failure.

Below, the model alloys are shown after **10% prestraining and bending to 81°**.
Significantly increased orientation gradients are present in the **dispersoid-rich alloy** (dislocation interactions and entanglement), **promoting SB formation** already at low bend angles. Conversely, lower dispersoid densities exhibit lower in-grain misorientations and only micro-SBs.

10 % uniaxial strain + bending to 81 °





The **Kikuchi band contrast** is **related to the defect density** (grain boundary regions have been excluded).
Despite achieving higher bend angles, alloys **with low dispersoid densities** show higher post-mortem BCs, suggesting **easier dynamic recovery**.

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Research focus:

- Deformation structures and formability
- Analysis and design of textures
- Microstructure-property relationships in light metals and composite materials
- Material degradation and corrosion

ALUMINIUM MICROSTRUCTURE ANALYSIS GAINHUB
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