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Recycling of aluminium waste sourced from mixed household waste

A methodology for assessing the recyclability of aluminium scrap and waste.

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This master thesis is dedicated to my nieces Erika, Solveig, Hanna, Ingrid, Elin, my soon to be nephew, and the rest to come. Watching you grow has changed me. My motivation is leaving the earth a better place than I entered it, so you, and hopefully your children, can grow up in a world as affluent as I have.

Summary

Aluminium is used in packaging for food items and beverages due to it's density, strength and preservative capabilities. In this project a methodology to asses the recyclability of aluminium waste was created, where parameters of scrap type, pre-treatment and remelting conditions are alternated in successive submersion remelting experiments with measurements of yield and analysis of metal quality.

The methodology was tested on an aluminium waste named *Cans*, which was remelted with decoating temperature as varied parameter. The sample material was decoated at $300\,°\mathrm{C}$ and $550\,°\mathrm{C}$. The decoated sample material was then remelted, 4 additions of ~ 2 kg per experiment, by submersion in a molten aluminium melt. The porosity, inclusion content and alloy composition of the metal was analysed to determine metal quality.

The experiments produced showed with statistical significance that yield of remelting, dross formation relative to melted sample material, and the chemical composition of the remelted metal depend on decoating temperature, collecting a lot of data from each experiment. $43\,\%$ of the sample material was lost during decoating, mostly due to combustion and evaporation of organics. Cans decoated at $550\,°\mathrm{C}$ was remelted with a yield of $65\,\%$ and a relative dross formation of $35\,\%$, and Cans decoated at $300\,°\mathrm{C}$ was remelted with a yield of $54\,\%$ and a relative dross formation of $46\,\%$.

Using cold PoDFA the content of inclusions in remelted Cans 300 and Cans 550 was measured as under $0.025\,\mathrm{mm}^2/\mathrm{kg}$, which is categorised as a very light inclusion content. The alloy produced contained several major alloying elements, contents reducing in order of: iron, manganese, magnesium, silicon, zinc and copper. The Cans sample material was deemed recyclable, and the methodology deemed very useful for evaluating the effects variation of different process parameters has on recycled metal.

In conclusion the Cans sample material was deemed recyclable, and the methodology deemed very useful for evaluating the effects variation of different process parameters has on recycled metal.

Preface

This work aims to be pioneering in nature rather than fundamental. A very necessary movement for material recycling has risen in Norway and in the EU, and ultimately this thesis is a contribution. By showing that aluminium packaging can be recycled with comparable yield and in some situations superior quality to used beverage ans, we hope to inspire sorting and recycling, to the benefit of waste handlers, recyclers and society as a whole. It has been a challenging project, and while I am an idealistic environmentalist at heart, the work has been performed objectively to be academically sound, with guidance from academics at the Department of Materials Science and Engineering of the Norwegian University of Science and Technology, and the researchers and engineers at SINTEF Industri.

Thank you to the stakeholders of this project: SFI Metal Production, SINTEF Industri, The Institute for Material technology at NTNU, Hydro Aluminium Rolled Products AS, IVAR IKS, Avfall Norge, Enval. This project was made possible by your contributions, your time, your advice and your materials. This project sought to illuminate the possibilities of recycling in our society, and I urge you to use the results to the benefit of your organisations and society as a whole.

First and foremost I thank my supervisor Anne Kvithyld. You are an endless source of guidance, motivation and support. You have been both a mentor and a friend to me, and I look forward to our continued collaboration.

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Last and perhaps most importantly, thank you to my father Per-Odd Eggen, for urging me to finish my degree. I often ask your advice, you always help me find my own answer.

Yours sincerely,

Sigvart Hansen Eggen Trondheim, 1. of July, 2019

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Chapter 1

Introduction

1.1 Experimental methodology

In this project a methodology to assess the recyclability of the aluminium in a material stream containing large amounts of aluminium. By this methodology large samples of the waste is pre-treated and remelted by batch whilst measuring/analysing:

- The content of volatiles (organic matter) in your sample material;
- the total yield of pre-treatment;
- the yield of pre-treated sample material during remelting;
- the analysis of metal quality of remelted metal by reduced pressure testing and PoDFA.

Whilst performing the pre-treatment one has ample opportunity to observe how each pre-treatment affects the sample material, take samples and adjust pre-treatment. Finally the effects of different pre-treatments of the sample material on the yield and quality of remelted metal may be evaluated. Additionally the pre-treatments and remelting produce several byproducts, such as oxygenated oils, ash, alumina, top dross and bottom dross, that could be characterised. The experiments are done at the biggest laboratory scale practically possible, using the same equipment, chemical (none) and methods as those used industrially. Considering this, the methodology should be useful to those who wish to asses the recyclability of a metal, with a real waste stream and a real industrial process in mind. The methodology will be completely explained in Section 4 - Methods and materials. In a swift background to motivate recycling of aluminium, and relevant theory for remelting of aluminium waste will be presented. Lastly the results from experiments following (but not completing)) the methodology will be presented and discussed.

1.2 Experiments

The original ambitious plan for the project was a factorial design with two parameters at second level and one parameter at third level with the aim to measure the qualitative effect on metal quality and yield when these following parameters were varied:

- Sample material: *Cans*, Foils *or* UBC
- Heat treatment: at 550 °C *or* at 300 °C
- compaction: at 35 MPa *or* at 300 MPa

This produced 12 combinations of parameters for remelting experiments. Thanks to some unforseen challenges, described in section 4.2, this scope was narrowed down: Only the effects of variation of decoating temperature on one sample material, *Cans* sourced from a central sorting plant for household waste would be examined. Cans and the other sample materials is described in section **??** In the process the methodology would be further developed and the recyclability of said sample material would be evaluated based on the findings. From the originally planned 12 to only 2 combinations. However the workload of these 2 combinations turned out to be more than enough for a project. Due to the success of the methodology more than enough results to discuss were produced.

There will always be a discrepancy between practices at research institutions and practices in the industry. In this project we tried to reduce this discrepancy as much as possible. The experiments were designed to be performed at the biggest lab scale possible. Instead of working with 2 g we worked with 2 kg. More than 130 kg of sample material was received and processed manually. We also chose to use pre-treatment which was feasible for industrial scenarios. We used no expensive reagents or complex machinery. Arguably the results from this project should translate well into an industrial scenario.

It quickly became apparent that recycling isn't one singular activity. We decided not to use salt flux in the project, as it produces hazardous operational conditions for equipment and operators in the laboratory alike. When recycling aluminium packaging without salt flux the following activities are necessary:

- Sorting of sample material;
- heat treatment of sample material;
- de-ashing of sample material;
- compaction of sample material;
- sawing of sample material;
- preparation of laboratory for remelting;
- remelting of sample material; and finally
- analysis of remelted material.

The activities of sorting, heat treatment, de-ashing, compaction and sawing are hereafter collectively referred to as pre-treatment.

Each activity added a dimension of complexity to the project:

- Each activity required planning, HSE-evaluations and execution;
- most of the activities rely on collaboration and/or coordination with a third parties;
- most of the activities above must be performed in order, relying on the completion of the previous activity in order to begin;

Any delay would ripple through the project, and plenty of delay was seen. The *UBC* (used beverage cans) sample material was delayed due to cyber attacks, whilst *EnvalFoils* never arrived. All the sample materials, and all the methods used in the project is described in chapter 4.

1.3 Questions answered by project

Going into this project there were several questions to be answered. The key questions were on how heat treatment affects metal yield and metal quality. The terms of yield, dross formation, and quality will be further discussed in section 3. Does decoating temperature affect the:

Q1.1 Metal yield when recycling waste aluminium waste?

Q1.2 Dross formation when recycling aluminium w?

Q1.3 Alloy content of magnesium when recycling?

Q1.4 Alloy content of lead when recycling?

Q1.5 Inclusion count of recycled aluminium waste?

Q1.6 Porosity of recycled aluminium waste?

Q1.5 and Q1.6 will be evaluated, whilst we will try to give statistical answers to questions Q1.1 - Q1.4, through the use of following hypotheses (which measure the conditions of remelted aluminium waste) in chapter 6 :

$$\begin{aligned} \text{Null hypothesis 1:} \quad & \text{Yield Cans 550} = \text{Yield Cans 300} \quad (1.1)\\ \text{Alt. Hypothesis 1:} \quad & \text{Yield Cans 550} \neq \text{Yield Cans 300} \end{aligned}$$

$$\begin{aligned} \text{Null hypothesis 2:} \quad & \text{Dross formed Cans 550} = \text{Dross formed 300} \quad (1.2)\\ \text{Alt. Hypothesis 2:} \quad & \text{Dross formed Cans 550} \neq \text{Dross formed Cans 300} \end{aligned}$$

$$\begin{aligned} \text{Null Hypothesis 3:} \quad & \text{Mg in Cans 550} = \text{Mg in Cans 300} \quad (1.3)\\ \text{Alt 3:} \quad & \text{Mg in Cans 550} \neq \text{Mg in Cans 300} \end{aligned}$$

$$\begin{aligned} \text{Null Hypothesis 4:} \quad & \text{Pb in 550} = \text{Pb in Cans 300} \quad (1.4)\\ \text{Alt Hypothesis 4:} \quad & \text{Pb in Cans 550} \neq \text{Pb in Cans 300} \end{aligned}$$

(1.5)

Chapter 2

Background

2.1 Motivation - Reducing environmental impact

2.1.1 The impact of environmental impact of primary production

Aluminium, as many other important construction materials in our day and age, is an enabler for the society we live in through the items we construct out of it. Whether these are structural components of vehicles, heat exchangers, or beverage packaging, aluminium enables technology which improves the life quality of the population. The demand for aluminium is only expected to increase on a global scale, as previously poor countries begin to industrialize and rapidly expand their infrastructure.

However the production of aluminium creates a large strain on our environment: For every metric tonne of aluminium produced 700 kg of fossile carbon in burned in the process. The real consumption of fossile fuel increases by a factor of approximately 10 when electrical power is supplied through fossile fuel power plants. Of the 863 334 GWhs of electrical energy used to produce aluminium in 2017, 70.6 % came from fossile fuels.[1] Furthermore the production of alumina through the Bayer process is itself energy intensive and results in massive quantities of hazardous byproduct called red mud. This muddy, alkaline residue isn't disposed of, rather it's stored in large open air pools that due to their sheer size has been accused of displacing indigenous populations in South American rainforests. The cost of aluminium production will be paid for decades to come, and it will be paid by us all, including those who receive no benefit from it's production.

There is therefore a strong ethical motivation to recycle aluminium when possible. If we could substitute primary aluminium, meaning aluminium produced from raw minerals, with recycled aluminium produced by recycling aluminium scrap, then the laws of economics assert that the demand and therefore the production of primary aluminium will go down, compared to a scenario where no aluminium is recycled at all.

2.1.2 The European Councils definition of recycling

Recycling is defined in many ways be different institutions. Norway and her institutions are held accountable to the European Councils directive 2008/98/EC on waste. Article 4 establishes a waste hierarchy as a priority order in waste prevention and management legislation:

(a) prevention;

(b) preparing for re-use;

(c) recycling;

(d) other recovery, e.g. energy recovery; and

(e) disposal.

Article 3 defines the terms used in article 4:

12. ’prevention’ means measures taken before a substance, material or product has become waste, that reduce:
    (a) the quantity of waste, including the through the re-use of products or the extension of the life span of products;
    (b) the adverse impacts of the generated waste on the environment and human health; or
    (c) the content of harmful substances in materials and products;
13. ’re-use’ means any operation by which products or components that are not waste are used again for the same purpose for which they were conceived;
15. ’recovery’ means any operation the principal result of which is waste serving a useful purpose by replacing other materials which would otherwise have been used to fulfil a particular function, or waste being prepared to fulfil that function, in the plant or in the wider economy. Annex II sets out a non-exhaustive list of recovery operations;
16. ’preparing for re-use’ means checking, cleaning or repairing recovery operations, by which products or components of products that have become waste are prepared so that they can be re-used without any other pre-processing;
17. ’recycling’ means any recovery operation by which waste materials are reprocessed into products, materials or substances whether for the original or other purposes. It includes the reprocessing of organic material but does not include energy recovery and the reprocessing into materials that are to be used as fuels or for backfilling operations;

Article 11 paragraph 2 sets a minimum requirement of $50\,\%$ preparing for re-use or recycling, by weight, of waste materials such as at least paper, metal, plastic and glass from household waste and similar waste streams by 2020. This paragraph was amended by directive 2018/851 to include increasing requirements of 55 %, 60 % and 65 % preparing for re-use and recycling of municipal waste by weight by 2025, 2030 and 2035 respectively.

2.2 Aluminium in packaging

Aluminium is used in packaging due to it's plethora of applicable properties allowing it to fill many roles. Examples of roles in packaging: Air barrier, light barrier, texture altering component, mechanical strength component, functional components. Most if not all roles may be covered by a substitute material, but packaging solutions with aluminium are typically effective and economically sound and therefore widely utilized.

Important properties of aluminium:

1. Very malleable, and easily shaped into trays boxes or the thinnest foils with ease, and low costs.
2. Readily available in high quantities, at acceptable costs.
3. A complete barrier against UV-radiation.
4. Very strong barrier capability against air, water, carbon dioxide, etc.
5. It has high mechanical strengthø
6. It has a low density.
7. Chemically stable at temperatures over500 °C.
8. Easily sterilized.

Aluminium is also valued by designers as a part of functional packaging: As a tray it can be placed directly in the oven. Beverage cans may be opened using an attached aluminium lever. Pills are sealed into pill cards with easily inspectable aluminium foils that breaks with a satisfying pop and ensures pristine and untampered pills. The cold feeling of aluminium tubes used to package spreadable cheeses is affiliated with fresh, refrigerated, well kept food by consumers. Aluminium packaging add value and a feeling of luxury to products, a feature highly regarded food consumers.

Aluminium in packaging can be divided into three categories of descending recyclability:

Category 1: Packaging like cans for beverages and sealed foods, trays for fish and chicken, aluminium tubes for cosmetics and food spreads and coffee pods consists of larger pieces of pure aluminium with smaller amounts of plastics or paper attached.

Category 2: Smaller pieces of aluminium are attached to larger pieces of other packaging materials, such as aluminium seals used for anything from toothpaste tubes, pill cards and wine bottles.

Category 3: Aluminium rolled into thin foil used in laminate with organics such as plastic, lacquer (which could also contain metal oxides), paper and glue. The aluminium in such foil is usually very thin between

In section 4.2 we will describe three sample materials used for our experiments: UBC consisting of category 1. Cans consisting of category 1 and 2, and Foils consisting of category 3.

2.2.1 Collection of used packaging in Norway

The collection and recycling of packaging in Norway is financed through extended producer responsibility, held by either the packaging producer or the imported of packaged goods. In Norway this responsibility is transferred from producers or importers to Grønt Punkt AS in exchange for a fee on each packaged product they release on the Norwegian market. The collection of the packaging materials are handled by material companies:

- Norsk Metallgjenvinning AS - responsible for collection of metal packaging;
- Sirkel Glass AS - responsible for collection of glass packaging;
- Norsk Resy AS - responsible for collection of corrugated cardboard packaging;
- Plastretur AS - responsible for collection of plastic packaging; and
- Norsk Returkartong AS - responsible for collection of carton packaging.

The fee system is quite simple in practice: A material is categorised as plastic, metal, glass, carton, or corrugated cardboard based on which material makes up the largest percentage of weight of the packaging. The producer then pays a fee per weight of packaging of said type they produce to Grønt Punkt, which then redirects the fees collected from each packaging category to the material company that collects the material. Upon collecting the packaging material, the material company now owns the material, and sell them on the open market. The material company freely controls the fee, and aims for a fee which equals the cost of collection of materials minus the revenue from sale of materials.

As an example: A producer chooses to package cream cheese in a tube consisting of mostly metallic aluminium by weight. Norsk Metalgjenvinning pays on average 0.100 to collect the tube, and earns 0.001 NOK on selling it to a recycler. The producer therefore pays a fee of 0.090 NOK per tube to Grønt Punkt, which redirects this fee to Norsk Metallgjenvinning in order to cover the deficit Norsk Metallgjenvinning incur while collecting the tube. [2]

Furthermore, the Norwegian government levies a tax on beverage packaging. A base tax per unit and an additional tax per unit based on packaging materials. The material based tax is reduced in proportion to collection rates of said beverage packaging category.

The material companies and Grønt Punkt are non-profit companies owned by the Norwegian packaging and food trade industries. These companies have organised several large regional or national collection systems to collect the materials. For example:

Table 2.1: Environmental taxes (in NOK) levied on beverage packaging per unit in Norway 2019. [**bevtax**]

Packaging material	Material tax	Collection rate	Tax paid
Metal	5.88	87 %	0.7644
Glass	5.88	93 %	0.4116
Plastic	3.55	89 %	0.3905
Carton	1.45	93 %	0.1015

Environmental base tax per packaged item: 1.21

- Beverage cans and bottles are collected through a pledge system. Consumers pay the pledge upon purchasing the item, and retrieve the pledge upon returning the item at the collection station available at most grocery stores in Norway.
- Paper and plastics is collected at individual household by municipal waste collection companies.
- Metal and glass is collected together at large collection bins positioned close to grocery stores.
- Some renovation companies sort recyclable packaging materials out of mixed household waste in central sorting plants.

Despite the efforts and direct economic incentives to collect the packaging materials separately (or sort them out in central sorting plants) some of these materials meant to be recycled end up in mixed household waste. Mixed household waste is a mixture of all kinds of garbage, like paper, foodstuff, diapers. Whether it be convenience, lack material sorting bins, lack of knowledge, shoddy practices in restaurants and cafes, or a plethora of other possible reasons, a lot of the packaging materials will continue to end up in the mixed household waste or it's equivalent in the foreseeable future.

2.2.2 Aluminium packaging in mixed household waste

Mepex Consult AS has granted access to picking analyses performed to assess the composition of mixed household waste in different regions of Norway. These picking analyses, made with hundreds of kilograms of mixed household waste estimated a metal waste content (described as mostly aluminium) of approximately 2 % by weight. Studies from Politecnico di Milano reported aluminium waste contents of 0.73 % and 1.63 % by weight.[3] Internal picking analyses at IVARs central sorting plant in Forus gave aluminium contents of 1.3 %.[4] [5]

It's therefore a reasonable assumption that the content of aluminium waste in mixed household waste will generally be close to 1.5 %. Content aluminium waste cannot be mistaken to equal content of aluminium. All aluminium packaging carry some non-aluminium with it. Beverage cans are coated and lacquered, whilst canned food cans often sometimes several. Furthermore most of the category 3 packaging (laminates with thin layers of aluminium) will not be categorised as aluminium. The aluminium content of mixed household waste is probably lower than the aluminium waste content, but the ratio between them changes by the day, and incredibly tedious to measure.

In studies performed at Politecnico di Milano they estimated effective yields of 92 %, 52 and 22 % when recycling used beverage cans, aluminium food trays and aluminium foils respectivly from the bottom ash of an incineration plant.[3] They estimated this by enriching the incinerator with the materials in question and measuring how the amount of aluminium in the bottom ash differed.

One should however be very careful to translate these results to Norwegian incinerators, which are run in very different conditions compared to italian incineratios.

2.3 Excursions and visits to stakeholders

Several excursions were made to visit and involve potential stakeholders. These visits were not planned in advance of the project. Fortunately the involved parties found the time required to involve themselves in the project, either through advice and insight or through sample material and even analysis.

29.01.2019 Avfall Norges Workshop on green raw materials in Oslo

Arranged by Mepex AS and Avfall Norge at Ingeniørenes Hus in Oslo, the topic on the workshop was evaluation of the value chain of the different material flows that are handled by the Norwegian renovation industry. Other discussed topics at the workshop were developments in waste collection systems to collection of metal and glass waste from households. Some municipalities had started collecting metal and glass waste at each individual household, whilst other municipalities had built central sorting plants for household waste. Both these implementations were new and thus lacking data regarding long term efficiency. Both implementations were considered positive steps for recycling. Lastly, and perhaps most importantly, it was agreed that education of the population is the most efficient way to increase collection and improve sorting of all material flows. Such education is stalling due to the lack of an universal system in Norway. Different municipalities have different collection systems requiring individual education of each municipality, which effectively halts country wide information campaigns. The workshop was attended by renovation companies from large Norwegian cities - ROAF from Oslo, BIR from Bergen, IVAR from Stavanger (whom later became an important stakeholder in this project), National Recyclers - NMG and Sirkel and others from the renovation industry.

30.01.2019 Hydro Rolled Aluminium Products in Holmestrand

Hydro Rolled Aluminium Products in Holmestrand produces rolled products for use in both packaging and structural applications. The plant produces high quality rolled sheets of aluminium, such as those used to produce aluminium packaging, by recycling aluminium being the only plant in Norway to do so. The plant is pioneering in it's use of secondary aluminium, combining aluminium scrap from several different sources to produce their alloys desired composition quality. They recycle both pre-consumer scrap sourced from machining and manufacturing industries, as well as post-consumer scrap in the form of compacted UBC. Hydro Rolled Aluminium Products thus acted as an important source of insight to the project.

19.02.2019 Visit to IVAR IKSs Central Waste Sorting Plant at Forus

Ivars central waste sorting plant at Forus, just outside Stavanger, handles household waste and similar wastes from the service industry in the Stavanger area. The waste goes through numerous sorting steps separating it into 10 categories. These categories are 5 different kinds of plastics, which are washed, refined and sold on site. The plant also recycles paper, magnetic metals and non-magnetic metals. The rest of the garbage, approximately 70 % is incinerated at the incineration plant next door. At the time of measurement, the non-ferrous metallic fraction, which consists mostly of aluminium waste, accounted for 1.3 % of the mass entering the sorting plant.

The aluminium fraction is a mix of category 1, 2 and 3 aluminium packaging (as described in section 2.2): UBC, canned food cans, aluminium trays, empty spray cans, tea candles, aluminium laminate foils, aluminium coffee pods, aluminium tubes for cream cheese or medical ointments, and other garbage aluminium packaging items. The spray cans are possibly hazardous during both pre-treatment and remelting and must be properly shredded or sorted out before such activities.

The main revenue source for the sorting plant is the sale of the refined plastic fractions. The revenue from selling the separated metal, coupled with the savings in incineration expenses from incinerating less mass makes separation of metals economically sensible. At current market prices for aluminium it doesn't make sense to separate foils, cans, and UBC, but it would be possible given economic or legislative driving force.

IVAR agreed to supply the project with category 1 and 2 aluminium packaging sorted out from their aluminium fraction by hand.

11.03.2019 Visit to Envals pyrolysis plant in Cambridge

At Envals pyrolysis plant in Cambridge organics in aluminium laminate foils are pyrolyzed by heating a carbonaceous media under a nitrogen atmosphere. The process is further described in section 3.8. The process produces a aluminium rich product polluted with carbonaceous residue, as well as a mix of aromatic and aliphatic oils. Enval is currently investigating how to remelt their aluminium product with an acceptable yield, a challenging task given the materials low yield and high content of carbonaceous pollution. Enval has agreed to supply the project with their aluminium product as a sample material in compacted briquettes.

25.04.2019 Annual meeting with Avfallsforsk with subject: Digitalization in the bio-economy in Oslo

Avfallsforsk is a research organization with members from Norwegian renovation industry. At the annual meeting a seminar on digitalization on the bio-economy was held after administrative proceedings were completed. I presented the findings of my project at the seminar, which at that time was mainly the heat treatment of the Cans sample material.

15. - 16.05.2019 International Dross and packagingkshop in Trondheim

At the international dross and packaging recycling workshop an impressive host of engineers, leaders and interest group repreesentatives from the aluminium industries of and North America, gathered to discuss and share the latest know-how on dross handling and packaging recycling. Educational reviews on dross recycling and handling of dross recycling by-products, aluminium packaging design, recycling furnace technology, the political landscape in the EU and in the US were followed by group discussions on the same topics. A highly educational workshop for a constantly changing industry.

Chapter 3

Theory

3.1 Measuring yield

Yield of remelting mass addition i should be understand as: What fraction of the metal added to the crucible that stays in the crucible as metal, after we have removed non-metallics through skimming. Yield is generally measured in percentage, and when yield is measured in kg this will be made obvious. For pure metals this ratio of amounts equals the ratio of weight:

$$\text{YIELD}^i = \frac{n^i_{Al,remains}}{n^i_{Al,added}} = \frac{m^i_{Al,remains}}{m^i_{Al,added}} \tag{3.1}$$

Where $n^i_{Al,remains}$ is the amount of metal remaining in the crucible after skimming, and $n^i_{Al,added}$ is the amount of aluminium added to the crucible If we assume our samples are pure metal, $m^i_{Al,remains}$ is the mass of aluminium remaining in the crucible after skimming, and $m^i_{Al,added}$ is the mass of aluminium added to the crucible. To proceed simplifications are necessary. The sample material added to the crucible is assumed to be pure metal, and the contents of the crucible are assumed to be pure metal. In this case:

$$\text{YIELD}^i = \frac{n^i_{Al,remains}}{n^i_{Al,added}} = \frac{m^i_{Al,remains}}{m^i_{Al,added}} \simeq \frac{m^i_{remains}}{m^i_{Sample}} \tag{3.2}$$

Where $m^i_{remains}$ is the mass of the added sample material remaining in the crucible after skimming, and Where m^i_{sample} is the mass of the sample material added to the crucible, which is measureable. In this project the mass of the sample is generally measured by subtracting the mass of the crucible before addition $m^i_{cru,before}$ from the mass of the crucible after addition $m^i_{cru,after}$.

The mass of the added sample material remaining after skimming can be measured as the difference of the weight of the crucible before the addition of sample and the weight of the crucible after skimming after said addition $m^i_{cru,skimmed}$.

Thus the yield per mass addition i is computed via the three measurements $m^i_{cru_b efore}$, $m^i_{cru,after}$ and $m^i_{cru,skimmed}$ with the following equation:

$$\text{YIELD}^i = \frac{m^i_{cru,skimmed} - m^i_{cru,before}}{m^i_{cru,after} - m^i_{cru,before}} \tag{3.3}$$

Equation 3.3 circumvents burn-off: if any part of the sample material evaporates, burns or leaves the crucible during addition it won't be included in the measurements of yield.

The difference between the m^i_{sample} and $m^i_{cru,after} - m^i_{cru,before}$ may be understood as a measure of burn-off.

It's important to realise that this measurement is not completely accurate. The crucible might contain different amounts of non-metallics before and after skimming. The skimming might extract some metal, in fact the skimming will probably extract more metal than non-metallics. This is tolerable as it reflects skimming in an industrial scenario. Furthermore the sample material added to the crucible is likely not pure metal. Most metals are covered in oxides, during pre-treatment a lot of ash is generated which is enclosed into the sample material during compaction, and then there's the possibility of finding other materials hidden within your sample material.

3.2 Metal quality

Metal quality is a collective term of several different properties, values, contents and conditions of a metal. In this project the following conditions are used to evaluate the quality of recycled aluminium, and metal quality should hereafter be understood to refer to:

- Inclusion, bifilm and disperoid count or area per volume;
- elemental composition; and
- hydrogen content and porosity.

Dispersoids: Small particles, in the size interval 0.10 nm- 100 nm.[6] Titanium boride is added to aluminium melts and precipitates as disperoids during heat treatment. [7]. Dispersoids limit grain growth which is beneficial for mechanical properties of the metal.

Inclusions: Larger particles, in the size interval 1 µm - 1000 µm. Inclusions are strictly detrimental to both chemical and mechanical propertiees of a metal. Inclusions in aluminium are typically made out of aluminium oxide.

Bifilms: Small films folded to produce a double layer (bifilm). Formed when sheets of aluminium oxide are forced into the due to disturbances on the melt surfaces, as shown in fig. 3.1.The films can act like cracks in the material reducing the yield strength and ductility of the metal [8].

In this project inclusion are per kg is measured with cold PoDFA.

The elemental composition of a metal significantly alters its mechanical, chemical (corrosiobn) and conductive properties, which why most metals are utilized as alloys. Typically one aims to keep all elements within certain boundary concenctrations. In general contents of alloying elements are kept as close as possible to certain values, and contents of other elements kept as low as possible.

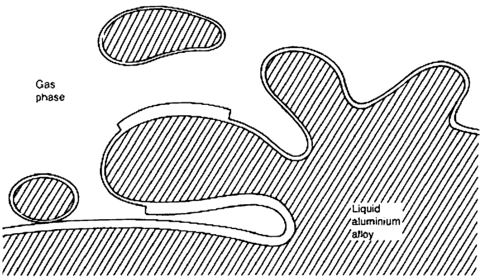

Figure 3.1: Formation of bifilm due to turbulent melt surface. [**Dispinar2004**]

Melts of metal have a capacity to dissolve lighter elements such as hydrogen, nitrogen and oxygen at certain levels. Upon cooling and solidification of the metal, these elements diffuse into microscopic pores in the metal. Quality decreases with increasing porosity, as porosity is detrimental to the mechanical, chemical and conductive properties of the metal. As with inclusions and dispersoids, some porosity is unavoidable. Porosity is typically measured using*Alscan/ALSPEK* or LECO. In this project porosity is assessed with RPT testing.

Each aspect of metal quality may be remedied through metal refining, and most metal producers employ complex refining techniques such as filtration, gas bubbling and flux refining to keep metal quality as high as possible. All agree that strict control of process parameters and raw materials is the most economical way to improve metal quality, and aluminium recyclers will wish to sell their metal without significant refining. Instead they will rely on dilution the contents of porosity and inclusions down to acceptable level by adding pure aluminium to their remelting process.

3.3 Analysis

3.3.1 Reduced Pressure Testing

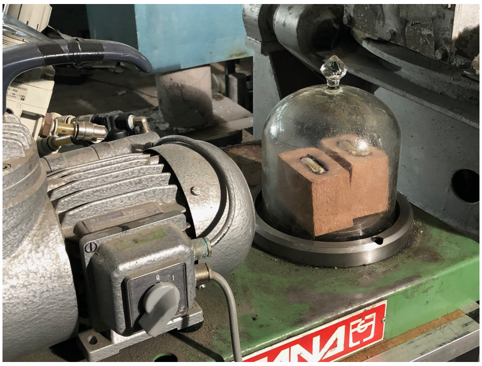

Figure 3.2: Preparation of RPT samples during remelting.

Reduced pressure tests (RPT) are performed to analyse the porosity of a liquid aluminium sample. The molten sample is poured into a sand mould and placed in a vacuum chamber which is evacuated to a reduced pressure (usually between -50 and -100 mmHg gauge pressure). As the sample solidifies, the inherent porosity grows compared to a sample solidified at gauge pressure. The top of the sample may be judged by it's smoothness or concavity, or the sample may be sectioned and polished to create a porosity profile which is then analysed visually. RPT has been popular in aluminium foundries over the years. The porosity of the sample depends inclusion content, the gauge pressure of the vacuum chamber, and the content of hydrogen. RPT cannot be used to assess hydrogen content, but it can be used to compare hydrogen contents between samples with similar inclusion counts, solidified at the same gauge pressure. [9][8].

3.3.2 PoDFA

PoDFA is a frequently used technique for determining the quantity of inclusions in an aluminium melt. Approximately 2 kg of the melt is poured into a preheated PoDFA cruciblee with a filter at the bottom. The crucible is placed over a chamber which evacuates, drawing the liquid metal through the filter, as seen in the left part of figure 3.3. After most of the metal has passed through, the crucible is left to cool. After cooling, the filter is retrieved from the crucible and cut along the centre line in the flow direction. The cut filter is then polished and examined optically by SEM and micro probe. This allows characterisation of different inclusion types and the area of those inclusions per kg metal. [**podfa**] PoDFA analysis might be performed in situ (hot-PoDFA), or samples can be decanted and melted to perform PoDFA sampling at a later time. This so-called cold podfa has been shown to produce lower readings of aluminium carbide compared to hot-PoDFA analysis [10]. Melt cleanliness is classified industrially using the following classification in table 3.1

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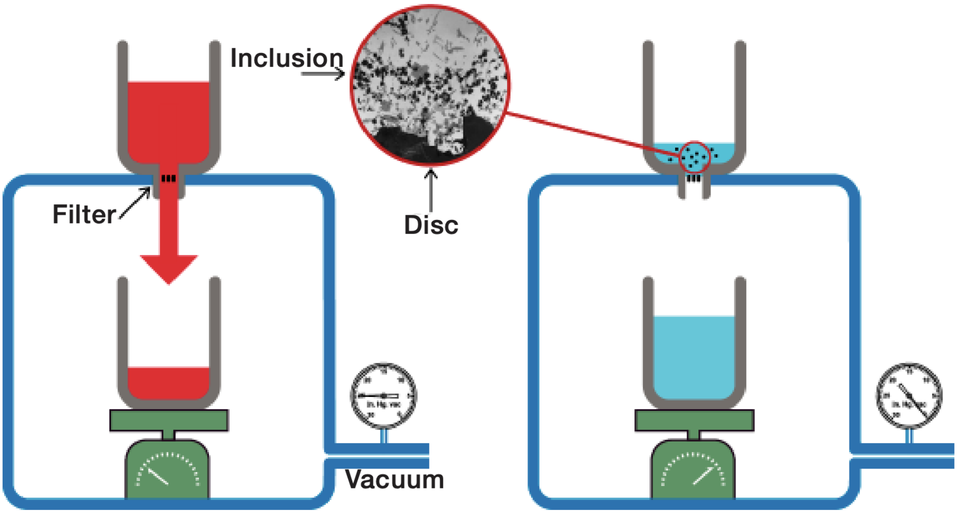

Figure 3.3: Overview of the PoDFA technology.

Table 3.1: Aluminium melt cleanliness performance evaluation using PoDFA. The evaluation assumes the use of hot-PoDFA. [11]

Classification	**Inclusion content** mm^2/kg
Very Light (1)	0.0 - 0.05
Light (2)	0.05 - 0.1
Moderate (3)	0.1 - 0.4
Heavy (4)	0.4 - 1.2
Excessive (5)	$\geq$ 1.2

3.4 Melting point and vapor pressures of select elements

Table 3.2: Melting point, and vapor pressures in pascal at select temperatures, of select elements. [12]

Element	**Melting point**	**Vapor pressure** [Pa]	
		At 327 °C (600 K)	At 527 °C (800K)
Al	660 °C (933 K)	-	$3.06 * 10^{-10}$
Cr	1907 °C (2180 K)	-	-
Cu	1085 °C (1358 K)	-	$6.60 * 10^{-11}$
Fe	1538 °C (1811 K)	-	-
Mg	650 °C (923 K)	0.0152	21.5
Mn	1246 °C (1519 K)	-	-
Ni	1455 °C (1728 K)	-	-
Pb	327 °C (600 K)	$5.54 * 10^{-7}$	0.00618
Sn	232 °C (505 K)	-	$1.26 * 10^{-9}$
Ti	1670 °C (1943 K)	-	-
Zn	420 °C (693 K)	0.653	-

3.5 The reactivity of aluminium

Gibbs free energy is a value describing the energy of a compound in a system based, derived from the first two laws of thermodynamics: Energy is conserved, and entropy increases.The Gibbs free energy is measured in joule per mole of atoms/molecules. The more negative the Gibbs free energy of a reaction, the more energy is released as the reaction occurs, and the same amount of energy would be required to inverse the reaction. The Ellingham diagram in figure 3.4 depicts the Gibbs free energy of formation of different oxides as a function of temperatures. The further down an oxide lies in the diagram, the more negative the Gibbs free energy, and the more chemically stable the oxide.The diagram only shows two metals with more stable oxides than aluminium: Magnesium and calcium. If the diagram had included the alkaline metals these would be futher below calcium. From the diagram we can observe that metallic aluminium will react with covalent compounds of other metals, reducing these metals into their elementary state and oxidising itself in the process. These so-called thermite reactions are exothermic and seldom lead to production of gaseous compounds, resulting in local temperature increases to well above 2000 °C.

$$3MeO_2(s) + 2Al(l) = Me(l) + Al_2O_3(s) \qquad \text{typically highly exothermic} \tag{3.4}$$

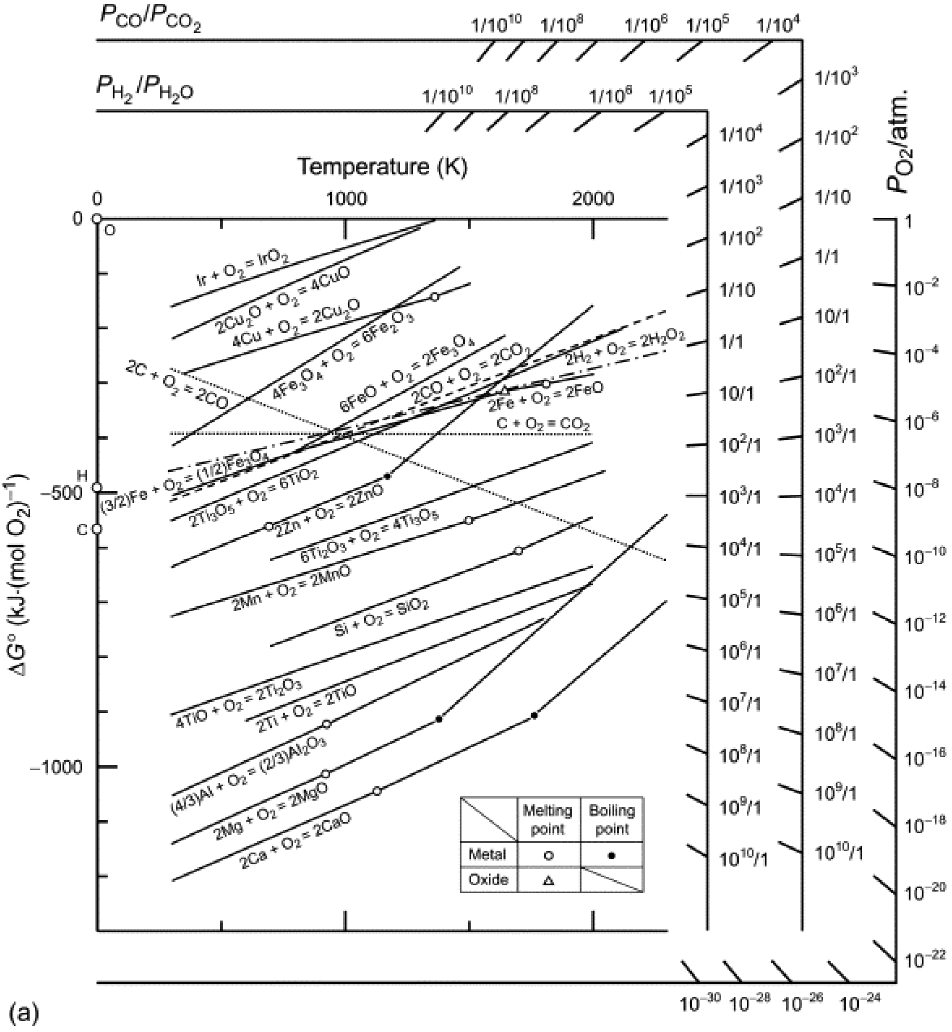

Figure 3.4: Ellingham diagram for carbon oxides and metal oxide [13]

Aluminium also forms nitrides, phosphides, hydroxides and salts in exothermic reactions. These covalent compounds of aluminium react with the moisture in the air to produce alumina and hydrides such as ammonia, phosphine and methane. Covalent compounds of aluminium are therefore often regarded as hazardous due to it's potential to produce toxic and explosive gasses such as phosphine and ammonia.

Table 3.3: Formation entalphies of several aluminium compounds. Unmarked data from [14]. * from [15], ** from [16]

Name	**Mol. Form.**	$\Delta_f H^\circ(s)$ $[kJ\,mol^{-1}]$
Aluminium	Al	0.0
Aluminium bromide	$AlBr_3$	-527.2
Aluminium carbide *	Al_4C_3	-18.3
Aluminium chloride	$AlCl_3$	-704.2
Aluminium fluoride	AlF_3	-1510.4
Aluminium hydride	AlH_3	-46.0
Aluminium hydroxide **	$Al(OH)_3$	-1293.2
Aluminium nitride	AlN	-318.0
Aluminium oxide (α)	Al_2O_3	-1675.7
Aluminium phosphate	AlO_4P	-1733.8
Aluminium phosphide	AlP	-166.5

In practice aluminium is easily handled. It's often stored outside without noticeable loss in quality. We slap it onto food that we want to consume with no second tought. It's submerged in water with practically no gas evolution, and industrially it's machined, cut, melted and rolled without much concern for it's theoretical potential to violently explode.

3.5.1 Passivation of aluminium

Sections 3.5.1, 3.5.2 and 3.6.1 are based on *Methods of Oxidation Inhibition for Al-Mg Alloys*, the doctoral theses of Dr. Nicholas Smith.[17]

The 1 - 3 nm thick aluminium oxide (hereinafter referred to as alumina) layer forms in less than a nano second. The thin layer is mechanically strong, inert, and limits further transport of oxygen to the point of being neglible.[18] The alumina layer also effectively inhibits reactions with other chemicals, such as those described in table 3.3.

The Pilling-Bedworth ratio describes thee volume of a metal oxide relative to the metal in elementary form.

$$R_{P-B} = \frac{V_{oxide}}{V_{metal}} \qquad \text{PILLING-BEDWORTH RATIO} \qquad (3.5)$$

Metals with Pilling-Bedworth ratios between 1 and 2 tend to form compact oxide layers on the metal surface that limit mass transfer thereby inhibiting further reaction of the metal.

The surface automatically becomes passive. Metal oxide typically has several different stoichiometric compositions, and crystalline metal oxides may exhibit several meta-stable structures.

Table 3.4: Pilling-Bedworth ratios of some common metal oxides.[**bradford1993**]

Oxide	**Pilling-Bedworth Ratio**
$\alpha - Al_2O_3$	1.28
$\gamma - Al_2O_3$	1.31
Cr_2O_3	2.02
CuO	1.72
Fe_2O_3	2.15
MgO	0.80
Mn_2O_3	2.40
PbO	1.28
TiO_2	1.76
SiO_2	2.15

Due to the dense oxide layer aluminium will not corrode at a meaningful pace when exposed to water and air, and is often stored outside prior to shipping with no noticeable loss in quality.

3.5.2 Continuous oxidation of aluminium at higher temperatures

When heated to and held at temperatures above at least 500 °C aluminium starts to display signs of continuous oxidation. This is due to several phase transformations which the aluminium goes through.

The exact temperatures of the phase transformations of alumina are difficult to measure, as the transformations typically occur over a temperature interval, and are heavily dependent on residence time and impurities in the oxide. Note in the borrowed figures 3.7 and 3.6 no exact temperatures are written down. Alumina first exhibits an amorphous structure if formed below formed below 377 -427 °C. If formed at higher temperatures the aluminium oxide is formed as γ-alumina. This meta stable structure eventually transforms into α-alumina at temperatures above 1000 °C, see figures 3.5 and 3.6

Each phase transformation results in an increase in density which leads to crack formation in the alumina layer, exposing the metal below to the atmosphere which leads to the formation of new alumina, which will in turn go through the phase transformations over time. In practice this leads to the oxide layer growing in thickness over time as temperatures increases. In general the loss of aluminium to such oxidation is negligible in itself, but the ticker oxide layers might have implications for dross formation, as will be discussed below. Furthermore the figures above describe alumina growth on solid aluminium. Alumina growing on liquid aluminium will act and crack differently due to the lack of a solid aluminium structure anchoring it below, however the mechanisms leading to oxidation are very similar. [17]

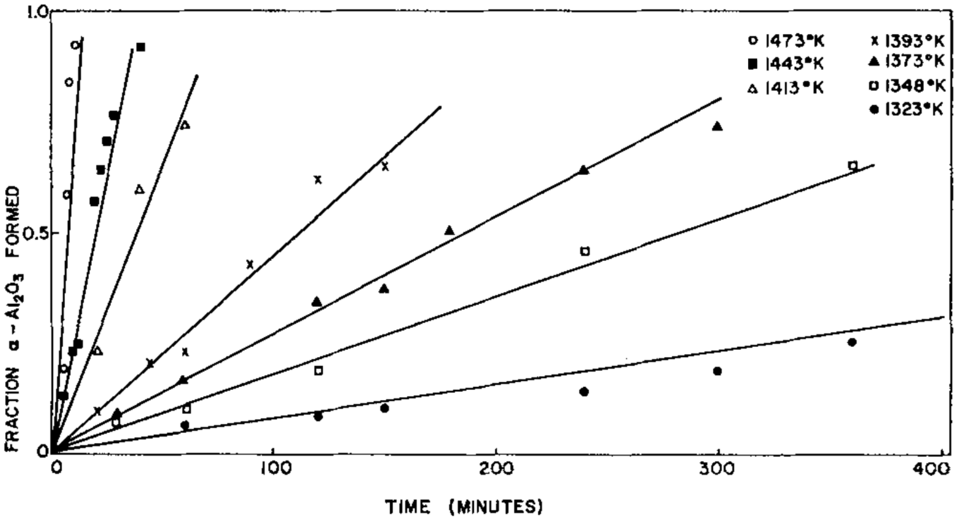

Figure 3.5: Kinetics of the phase transformation from γ- to α-alumina at different temperatures. [19]

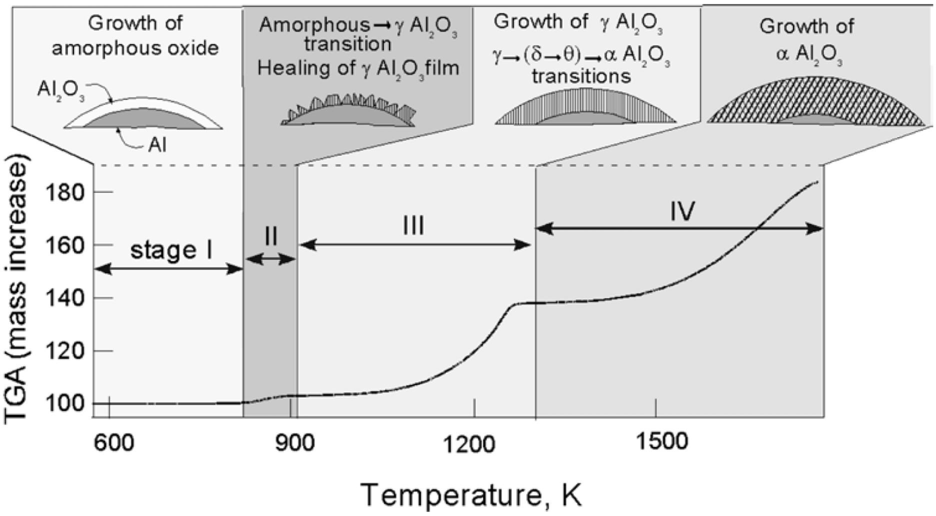

Figure 3.6: Temperature intervals of the phase transformations of alumina [20]

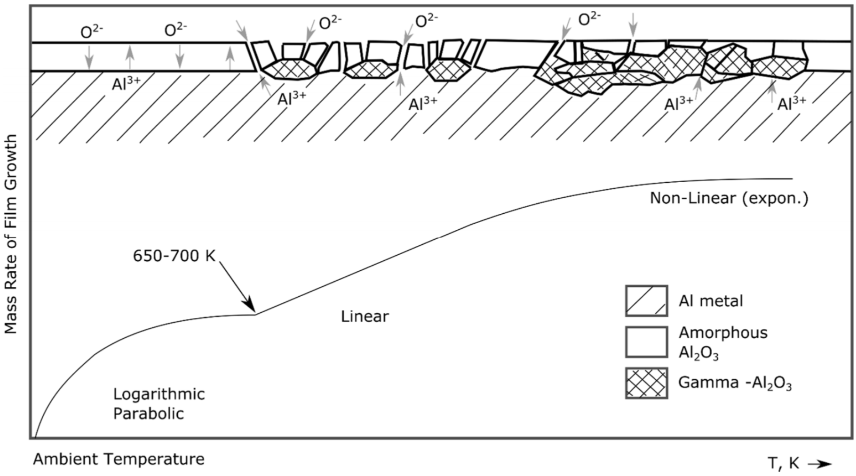

Figure 3.7: Theory: Growth of aluminium oxide layer due to phase transformation induced cracking. Redrawn from Bahk et al. [21]

3.6 Conditions leading to dross formation

Dross as a term describes a mass of oxide which has a significant amount of metal trapped within it. Due to melting at much higher temperatures the aluminium oxide formed when handling liquid aluminium will never melt. Furthermore whilst aluminium oxide is denser than aluminium, the slag oxide formed whilst handling aluminium is often very porous. It will either get trapped in the crucible lining - liquid metal - interface as bottom dross, or gather on top of the liquid aluminium as top dross, most commonly referred to as dross. The aluminium content in this dross varies between 15 - 80 $\%$ metal by mass. The production of dross during melt handling is a widely studied subject, as dross production is the no. 1 factor influencing operational yield, furthermore dross handling relies on salt fluxes with black dross, a hazardous and expensive waste to process as guaranteed byproduct.

3.6.1 Magnesium content in melt

In contrast to the slow oxidation of pure aluminium melts, melts containing magnesium will, under the same conditions as thee pure aluminium melt oxidize very differently. The Melt act normally at first, still protected by a layer of amorphous oxide containing gradually increasing amounts of magnesium oxide - MgO.

The break away time is the time this oxide transforms first into crystalline magnesium oxide or magnesium aluminate $MgAl_2O_4$ seeds. The seeds become *hot spots* where oxidation of magnesium rapidly increases, depleting the magnesium content of the melt relatively quickly. Upon depletion of the magnesium content down to a concentration translating into an activity of 0.0023, the oxidation halts. The magnesium oxide might then absorb some aluminium transforming into magnesium aluminate. Conditions and practices to increase/decrease the time before breakaway are well detailed by Cochran, Belitskus and Kinosz in figure 3.8.

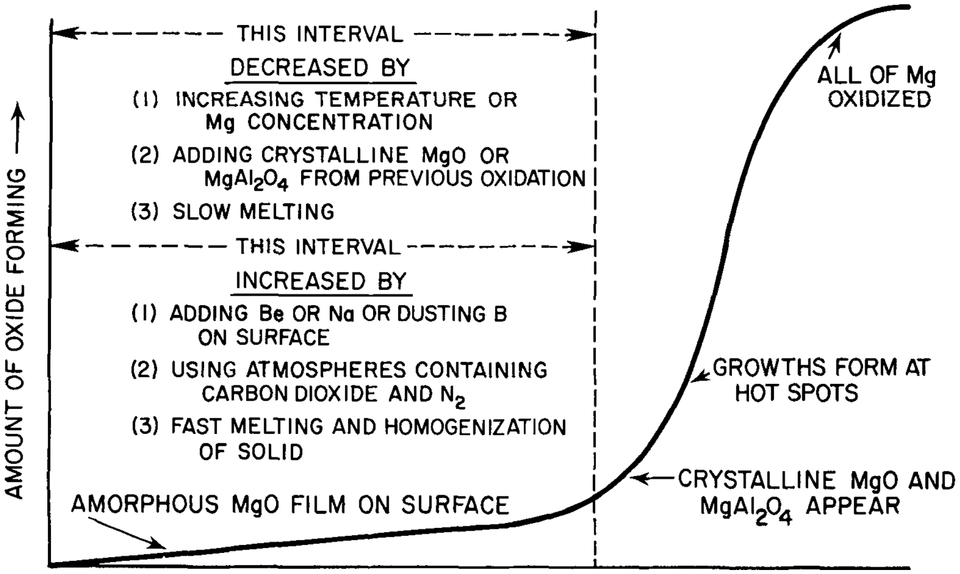

Figure 3.8: The general oxidation behavior of Al-Mg melts. [18]

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3.6.2 Carbonaceous gas from pyrolysis of hydrocarbones

Skriv om Al4C3!

CO_2, CO, CH_4, C_4H_{10}, typical products from pyrolysis of hydrocarbones and combustion of carbon, was shown to increase dross formation on an aluminium melt exposed to 95 % argon + 5 % gas in a series of experiments by Steglich. The impact of the gas increased in the order of C_4H10, CH_4, CO_2 and CO. However, the metal content in the dross formed by CO_2 was thrice that of metal content in dross formed by CO. [22]

The following reaction mechanism has been suggested for the reduction of CO_2 by a liquid aluminium melt.

$$\begin{aligned} CO_2(g) &\rightleftharpoons CO_2(ads) \\ CO_2(ads) &\rightleftharpoons CO(ads) + O(soln) \\ CO(ads) &\rightleftharpoons CO(g) \end{aligned} \tag{3.6}$$

In addition the following reaction mechanism was suggested for the reduction of CO by a liquid melt:

$$\begin{aligned} CO(g) &\rightleftharpoons CO(ads) \\ CO(ads) &\rightleftharpoons C(s) + O(soln) \end{aligned} \tag{3.7}$$

These reduction mechanisms are dependent on the partial pressures of CO_2 and CO in the system. The reduction of CO_2 seems to generate insufficient partial pressure for CO reduction, rather a source of CO is necessary for the complete reduction of carbon dioxide. This could be achieved by rising temperatures as the Boudouard reactions turns CO_2, but this again relies on the presence of solid carbon.

$$CO_2(g) + C(s) \rightleftharpoons 2CO(g) \qquad \text{The Boudouard Reaction} \tag{3.8}$$

Steglich further notes that formation of aluminium carbide is unlikely according to thermodynamics, as aluminium carbide could react exothermically with both oxygen and carbon dioxide, both reactions producing alumina either solid carbon or carbon dioxide.

3.6.3 Coating

3.7 Salt fluxes in aluminium recycling

Fluxes are made by adding solid salts, typically a mixture of $50\,\%$ - $70\,\%$sodium chloride and $30\,\%$ -$50\,\%$ potassium chloride with a few $\%ts$ of fluoride salt, like sodium fluoride or calcium fluoride,to a furnace. The mixture of salts have a low melting point and viscosity, significantly improving metal yield by improving coalescence of metal droplets trapped in the dross, due to the aluminium oxide breaking properties of fluoride. The flux also serves as a barrier against the atmosphere, and can absorb a certain amounts of inclusions from thee melt.

When flux is added to a process the result is a significantly reduced dross formation, which often leads to significantly improved yield. The ability to extract aluminium from dross is so efficient that the industry standard has become to any dross produced with flux to extract aluminium. Flux is also very cheap. There are substantial drawbacks to using flux:

- Used flux becomes to black dross, a hazardous waste material which is difficult to recycle and typically deposited in landfills.
- Fluxes rely on fluorides, which represents a significant operational hazard to workers, and requires additional HSE steps during operation.
- Fluxes may produce, fluoride, chloride or sodium gases, which are hazardous to operators and detrimental to equipment.

These negative aspects of fluxing are strong driving forces to limit the use of flux for remelting.

3.8 Decoating

Organic molecules such as the proteins, sugars, fats, cells, starches that together with water make up food stuff consist mainly or completely of hydrocarbones. Coatings, resins, lacquer, plastics and paper are also composed mostly of hydrocarbones. Most of the matter that contaminate aluminium packaging waste is inherently a mixture of hydrocarbones.

These hydrocarbones are a mixture of numerous molecules which chemical stochimetry is generally written as $C_xH_yO_z$. In the process called Pyrolysis () larger hydrocarbon molecules are broken into smaller hydrocarbon molecules (a phenomenon called scission) by heating in a low oxygen atmosphere. These smaller molecules tend to evaporate into gas phases like methane CH_4, water - H_2O and carbon dioxide CO_2, or larger molecules condense into a tar phase. Much of the carbon stays behind forming a solid char. The composition of these pyrolysis products depend on the pressure, temperature and oxygen content during pyrolysis. Decoating is quite similar to pyrolysis.

The decoating we describe in this project consists of heating scrap up to certain temperatures in air, in order to break down (via scission) and combust (via reaction with the oxygen in air) hydrocarbones on a metallic surface. Thus Decoating should be understood as the combination of two processes:

1. Scission of hydrocarbones;
2. Combustion of pyrolysis products.

Scission reeactions are endothermic, meaning they require heat to procee cooling doactice cool down their surroundings as they occur. The rate of both endothermic reactions and exothermic reactions both generally increase significantly as you increase the temperature in which they occur. The transport of reactants to the reactions, or products away from reactions limit the reaction rates once they reach a certain rates.

The combustion should not be taken for granted. At lower temperatures (below 500 °C) or at low oxygen concentrations combustion might not occur at all. Scisssion reactions are generally endothermic, cooling down the chamber. Furthermore the chamber filled with combustible gasses could easily become starved of oxygen

Aluminium scrap with an organic content higher than 5 % is decoated in order to improve yield and metal quality from remelting, as well as improving operational stability and limiting emission of hazardous and toxic gases while remelting. Historically the combustion of the organic fraction has been inevitable, but new technology allows extraction of the organic without combustion, technology to be discussed below.

In his experiments on aluminium coalescence Capuzzi showed that coating had a negative impact on coalescence during remelting. Coalescence is the process in which 2 or more droplets of liquid metal merge together. The lower the ability of the metal to coalesce, the higher the dross formation. Further it was shown that decoating the aluminium at 400 °C had no effect on the coalescence, whilst coated aluminium decoated at 600 °C coalesced equally well as uncoated aluminium.[24] [25]

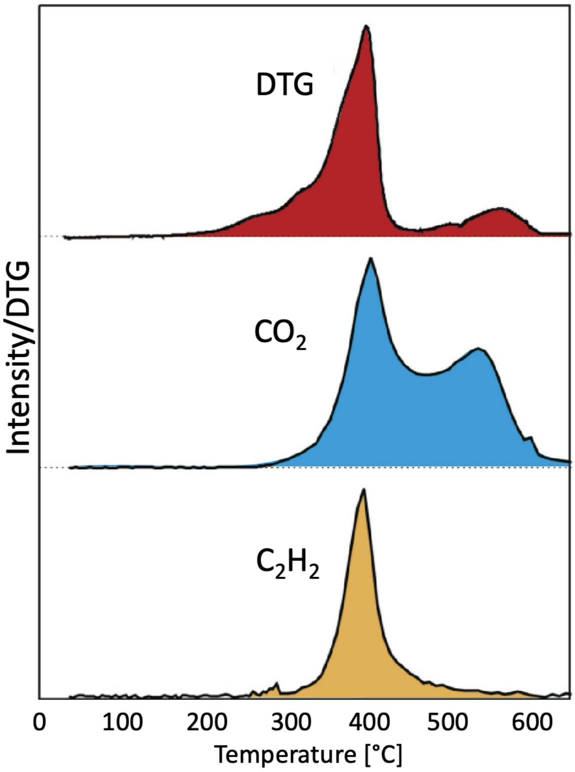


Figure 3.9: Emission of scission products (meeasured via C_2H_2) and combustion products (measured via CO_2 from a coated metal substrate. [23]

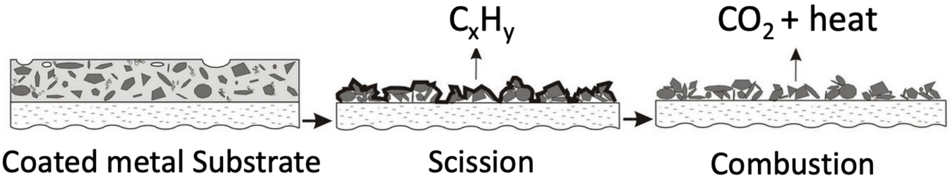


Figure 3.10: Illustration of decoating of a coated metal substrate. First the hydrocarbones break down via scission, then residual char combusts with increasing temperatures. [23]

Decoating processes are full scale operations in of themselves, requiring shredding or other sizing techniques, grading and separation for efficient operation. Key to operation is proper afterburning of decoating gas which limits emissions, typically to required levels.

The three decoating solutions described below are each heated through the combustion of both the organic contents of the feedstock and natural gas when the feedstocks organic content is too low, or moisture too high.

In Bed-type decoaters the feedstock is transported through ovens with decoating gas by a belt either continosly or by batch. This technology is being phased out in t

The rotating kiln furnace is most commonly used for decoating, in which the material is continuously fed into a tilted rotating kiln against a flow of decoating gas. The kiln allows mixing of the scrap, good metal-gas contact ensuring thorough and homogenous decoating, and residence time is adjusted through the tilting angle of the kiln.

The fluidized bed decoater offers the best potential for temperature control of the decoating solutions, it was developed by Alcan in the 1990s to decoat scrap materials with high organic contents (up to $50\,\%$ In a fluidized bed particulate matter of low density is maintained in suspension in an upwards gas flow. The matter and gas will collectively act as a boiling liquid. Scrap fed into the fluidized bed will sink towards the bottom where it is extracted. The fluidized bed has a higher heat capacity and a much higher heat conductivity than the gas in simpler decoaters, which allows complete decomposition and very precise temperature control. This allowed optimum metal recovery. Notably, the operation of fluidized bed decoater is more complex compared to a kiln, and those ran by Alcan struggled with the losing the particulate matter over time, an issue brought on by the low density and windy topology of scrap aluminium. Ultimately the cost of operation is expected to be greater for a fluidized bed decoater than for a kiln decoater.[26] [27]

3.8.1 The Enval Process

In the enval process microwaves are used to induce pyrolysis of in a nitrogen atmosphere to separate organics out of aluminium laminates, producing an organic micture of aromatic and alphatic oils as well as a very low density aluminium material contaminated with significant amounts of carbon. Their feed stock is currently very sorted packaging. They will get bags of identical, pre-consumer packaging that they recycle. A varied and challenging feed stock from MSW might be difficult to process with the Enval process, however at the same time the process should be possible to optimize towards treating different kinds of feed stock. Enval is currently investigating how to remelt the aluminium with a good yield, a challenging task given the low density and high amount of carbon contamination in their feed stock.

3.8.2 The behavior of gas in a closed system

Gas in a closed system that isn't very hot or at very high pressure is expected to act as an ideal gas following the ideal gas law:

$$PV = nRT \tag{3.9}$$

If the volume V of a system is constant, this means that the pressure P of the ideal gas is dependent on the number of gas molecules n and the temperature T of the gas.

Chapter 4

Methodology, Materials and experiments

4.1 Methodology

The methodology consists of four categories of activities:

1. Retrieve aluminium sample material(s)
2. Pre-treat the sample material(s)
3. Remelt the sample material(s)
4. Analyse the recycled sample material(s)

Examples of sample materials are: Used Beverage Cans, The aluminium fraction of a central sorting plant, pure aluminium. Pre-treatment entails any procedure to prepare the material for recycling. In this project pre-treatment comprised of decoating, ash-removal, compaction and sizing. During Remelting most of the samples for analysis are extracted.

In the methodology the effects of different variations of pre-treatment and remelting are examined through direct measurements of yield and dross formation during remelting, and through analysis thereafter. This is most efficiently done through a numerical design on the 2. (or 3.) level. In such a design you chose a number of factors that you could vary at two quite different setting when performing an experiment. For example:

A = Sample material - UBC or Cans;

B = decoating temperature - 300 °C or 550 °C;

C = compaction pressure - 35 MPa or 300 MPa.

The same remelting experiment is then performed using all variations of factors A, B and C. With the factors above this gives 8 different setups. If you increase the amount of settings to a factor by 1, this increases the amount of setups by 50 %. If you introduce a new factor with 2 settings, this increases the amount of setups by 100 %. It's tempting to just measure one more setting or one more factor, but the workload of the design increases exponentially. Replicates are ran to improve the amount of samples you

Such a design is presented in table 4.1. .

Table 4.1: Experimental structure of a factorial design at the 2. level

Randomised Experimental sequence - n = 36

A = Sample Material			+ = Cans	- = UBC	* = Foils
B = Heat treatment T			+ = 300 °C	- = 550 °C	
C = Compaction Pressure			+ = 35 MPa	- = 300 MPa	
Replicate 1	Replicate 2	Replicate 3	**A**	**B**	**C**
8	15	36	+	+	+
11	23	31	-	+	+
12	17	28	*	+	+
7	20	27	+	-	+
5	16	29	-	-	+
10	13	32	*	-	+
6	14	26	+	+	-
9	22	34	-	+	-
1	19	25	*	+	-
2	24	33	+	-	-
4	21	30	-	-	-
3	18	35	*	-	-

4.2 Sample Materials

For the project we considered recycling of 4 sample materials: UBC, Cans, Foils and EnvalFoils. Preliminary experiments were performed through weeks X - Y to assess the feasibility of recycling the first three: Small samples of Cans, Foils and UBC were pre-treated and remelted at a small sample size. The results of the preliminary experiments mentioned below and discussed in full in section 5.1. Full scale pre-treatment and remelting was only completed with Cans, with reasoning below. For description of aluminium packaging categories referenced below see section 2.2.

4.2.1 UBC

UBC consists of category 1 aluminium packaging : used beverage cans. The material also contains some pieces of plastics that are revealed upon dismantling the material. Still the material typically consists of approximately 97 % aluminium by weight. The UBC is a remarkable material in its homogeneity and it's very low contents of residual food stuff, which is removed during collection. UBC is collected through a separate pledge based collection system - *Pantesystemet* , and therefore readily available in large, homogeneous orderly quantities. The UBC was therefore planned to be included in the project to function as a standard against which the results of remelting the other materials could be compared against. UBC was delivered cut and compacted from Hydro Holmestrand. Due to a cyber attack on hydro delivery was delayed by a month, and we were unable to heat treat the material due to it arriving after the incident described in section **??**. Therefore experiments with UBC were discontinued.

4.2.2 Cans

Cans is a sample material consisting of mostly category 1 packaging with some category 2: aluminium packaging sorted out of mixed household waste, such as:

- Aluminium beverage cans,
- aluminium cans for canned foods,
- aluminium trays for fish fillets,
- partially empty tea candles,
- aluminium screw corks for bottles,
- used aluminium coffee pods,
- aluminium tubes for cream cheese,
- and other aluminium packaging items.

The material consists of approximately 50 % metal by weight, almost solely aluminium, and contains significant pollution in the form of organic matter. Around 5 % by weight is lacquering and coating, whilst the remaining 45 % of the material is highly odorous residual food stuff with significant water content. The material was sourced from the aluminium fraction produced at IVARs central sorting plant for mixed household waste and hand sorted to remove category 3 packaging as well as closed containers. Cans was pre-treated and remelted at full scale in the project.

4.2.3 Foils

Foils is a sample material consisting solely of category 3 packaging sorted out of mixed household waste, such as bags for coffee, chips, spice mixes, sauces etc. The material consists of approximately $5\,\%$ metal by weight, the rest being organic matter such as paper, plastic and coating. The material was sources from IVARs central sorting plant for preliminary experiments. During preliminary experiments it was discovered that heat treatment of foils resulted in evaporation of approximately $90\,\%$ of the sample material, further more approximately $50\,\%$ of the remaining solids would be composed of non-metallics. This in turn meant that we would require 10 times more Foils than Cans to create equal amounts of sample material for remelting. Additionally it meant that we would produce 10 times more pollution whilst pre-treating Foils. We therefore opted not to use Foils as a sample material.

4.2.4 EnvalFoils

Envalfoils is a sample material received from Envals process plant in Cambridge. EnvalFoils is produced through heat treatment of highly homogenous category 3 packaging in a fluid bed de-coater in nitrogen atmosphere below atmospheric pressure. The resulting material consists of smaller pieces of thin aluminium foil with significant contetnts of carbonaceous residue. There are also visible traces of the original lacquer visible on the material, likely the heavier components of pigments such as titanium white TiO_2. The material flaky material has an effective density of approximately $0.05\,\mathrm{gcm}^-3$ prior to compaction, afterwhich the density is increased to approximately $1\,\mathrm{gcm}^-3$, the same density as water.

EnvalFoils was planned to substitute Foils in the project, however contrary to their promises Enval failed to deliver the sample material, and 20 days before the end of the project, the material still hasn't arrived. Had it arrived earlier pre-treatment and remelting of EnvalFoils would have commenced.

4.3 Pre-Treatment

I could probably write something here. The experiments were performed in written order, heat treatment followed by de-ashing, compaction, sawing and finally remelting.

4.3.1 Heat treatment

Cans was de-coated in batches consisting of 6 containers holding 1.5 to $4\,\mathrm{kg}$ of sample material each. The stainless steel containers had an area of $500\,\mathrm{mm}$ x $300\,\mathrm{mm}$ and three different heights: $100\,\mathrm{mm}$, $150\,\mathrm{mm}$ and $200\,\mathrm{mm}$. This kind of container is usually utilized in larger professional kitchens. For preliminary heat treatment, batch size was reduced to approximately 1 kg.

While de-coating of each container in each batch were weighed before being filled with material, after being filled with material, and after completed heat treatment. Weight measurements for batch 3, 4, 5, 6 were made using XXXX, whilst weight measurements for batches 1, 2 and 7 were made using YYYY. The YYYY was unfit for these purposes, giving inaccurate weight measurements if samples weren't correctly centered on the scale, which was difficult for such large sample containers. Additionally the scale didn't measure above $4200\,\mathrm{g}$, which made weight measurements time-consuming. For all but batches 1 and 7, the furnace was preheated to $300\,^{\circ}\mathrm{C}$ before sample insertion. Batch 1 was inserted into a furnace preheated to $550\,^{\circ}\mathrm{C}$, and batch 7 was inserted into a cold furnace. For batches 2, 3, 4, the furnace set-temperature was kept at $300\,^{\circ}\mathrm{C}$ for approximately 45 minutes before the it was increased to $550\,^{\circ}\mathrm{C}$. The furnace set-temperature was kept at $300\,^{\circ}\mathrm{C}$ for the entire duration of pre-treatment of batches 5, 6 and 7. During pre-treatment at $300\,^{\circ}\mathrm{C}$, the furnace would reside at $300\,^{\circ}\mathrm{C}$ for up to an hour, at which point the temperature would rise without the furnace powering it's heat elements, as will be seen in section 5 figure 8.10. Samples were extracted from the furnace once the chimney stopped producing smoke and the temperature fell below $300\,^{\circ}\mathrm{C}$. The duration of each pretreatment was approximately $90\,\mathrm{minutes}$ for samples pre-treated at $550\,^{\circ}\mathrm{C}$, and $180\,\mathrm{minutes}$ for samples pre-treated at $300\,^{\circ}\mathrm{C}$. After extraction and weighing the samples were transferred to barrels categorised by heat treatment temperature.

4.3.2 Ash removal and compaction

De-coated sample material was tumbled to remove carbonaceous residue and ash. The sample material was fed into the tumbler in portions of 65 L. During tumbling the tumblers rotational axis was kept 30 degrees above the horizontal plane. After 10 minutes of tumbling most of the sample material was removed from the tumbler by hand, before new material was fed into the tumbler. After each 4th portion the tumbler was completely emptied and aluminium bits were manually removed from the residual carbon and ash that were extracted from the bottom of the tumbler.

Ash was removed from the heat treated sample material via tumbling in a 130 L cement mixer. The ash was removed from sample material by the barrel. The barrel content was placed into an intermediary barrel, from which it was then transferred to the cement mixer which was kept under heavy ventilation. The mixer was turned so it's rotational axis stood 30° against an horizontal axis and turned on. After a residence time of 5 minutes the sample material was extracted from the mixer into it's original barrel. Thereafter new material was added from the intermediary barrel. When the intermediary barrel was emptied, the cement mixer was halted and residue was removed from it's interior.

Preliminary samples were not de-ashed.

The pre-heated and de-ashed materials were compacted at Stena Aluminium recycling in Hommelvik. The press worked at $35\,\mathrm{MPa}$, a weight of 250 tonnes divided over $20\,\mathrm{cm}^2 \times 35\,\mathrm{cm}^2$. After compaction the material was placed into barrels based on the materials heat treatment temperature.

4.4 Remelting

Remelting was performed in crucible placed in the midst of refractory lined, water cooled, copper coils which by electrical current induced a magnetic field through the crucible to heat it's metallic content. The crucible was always kept under strong ventilation, except for during decantation. The sample material was melted by submerging it in a heel of liquid aluminium kept at 700 to 800 °C. The coils were powered to keep the temperature closer to 800 °C, however the temperature would drop sharply when cold sample material was introduced to the crucible. The sample material was weighed into portions of approximately 2 kg which were then placed onto the refractory lining in order to preheat from the strong radiation emitted by the molten crucible. The material was then lowered into the crucible, and kept partially or completely submerged by an iron rod.

For each addition (save for the initial metal heel or for experiments with pure Al), dross was transferred from the top of the melt to an iron bucket using a perforated iron ladle. Thereafter RPT samples were extracted using a refractory-coated iron ladle and poured into pre-made sand moulds which were then placed into an evacuation chamber where they were kept until the next RPT samples were ready approximately 10 minutes later. After RPT samples were removed the next portion of sample material was prepared and the process was repeated for all portions of the experiment. After extracting RPT samples from the last portion, the crucible contents were decanted into cast iron moulds. After decantation bottom dross and bottom metal was removed from the crucible and decanted into another cast iron mould. This experimental procedure was inspired by similar remelting experiments performed by Kristoffersen et. al. [28]

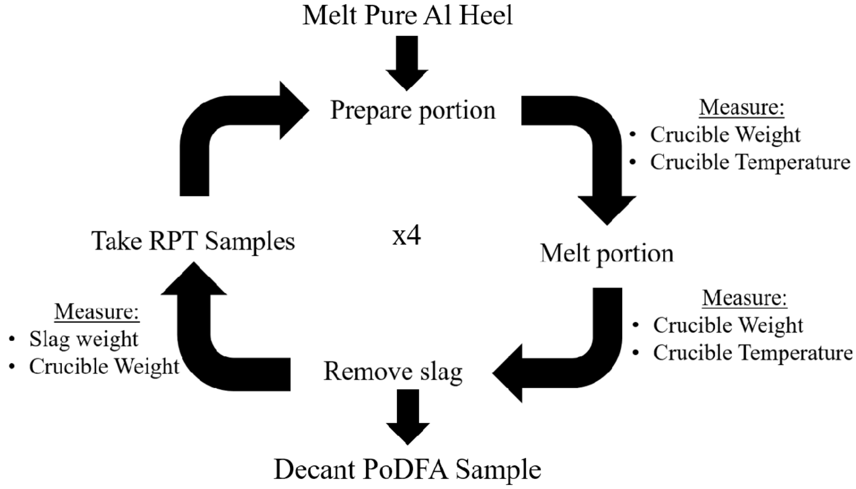


Figure 4.1: Method: Simplified flowchart of remelting.

During the experiments the following measurements were logged:

- The time of beginning the addition of each portion;

- the mass of the crucible before addition of a portion;
- the mass of the crucible after addition of a portion;
- the mass of the crucible after removal of slag from the crucible;
- the mass of the slag removed from the crucible;
- the temperature of the crucible before and after addition of a portion.

To negate weight measurements interference from the drop coil on the scale, the crucible was only weighed whilst the drop coil was powered off.

4.5 Use of software for statistical analysis and datavisualisation

An introduction into statistics would be outside the scope of this project. However it is necessary to explain what statistical models have been used to prepare our confidence intervals, confidence bands and to test our hypotheses.

The populations we are testing, like the yield of remelting Cans 550, or the dross formed on Cans 300, or the amount of Mg found in remelted pure aluminium, are assume to be normally distributed. Since we always sample less than 30 points from these populations, our points will be Student-T-distributed. Therefore confidence intervals are calculated using the Student-T distributions.

To test our hypothesis *Minitab 2018 Statistical Software [C]* (hereinafter referred to as *Minitap*), a statistical software available through the NTNU program farm, was used on the data series in question. This produced confidence intervals for the difference between the the populations the data series sampled. When this interval doesn't include 0, the 0-hypothesis has been rejected, and the alternative hypothesis is considered true. The hypotheses are listed in the introduction, and are tested in section6: Discussion.

Further we generated a lot of plots using the *Seaborn* library of the open software programming language *Python.* These plots automatically contain confidence intervals or confidence bands generated through a statistical method called bootstrapping, which produces confidence intervals very similar/identical to those produced using a *Student-T* distribution.

Chapter 5

Results

5.1 Results from preliminary experiments

Cans, Foils and UBC sample material was pre-treated and remelted according to methodology. All the samples were heat treated in the same batch at 550 °C. The transformation of the samples through heat treatment and compacting is pictured in figures 5.1, 5.2 and 5.3. Weight measurements before and after heat treatments are listed in table 5.1.

Table 5.1: Condition of samples after preliminary heat treatment

Batch	Weight before	Weight after	Weight loss	Ash
Foils	1114 g	174 g	84.4 %	a lot
Cans	688 g	373 g	45.8 %	some
UBC	1221 g	1175 g	3,8 %	little

After heat treatment the sample material was visibly de-coated, with some white pigment remaining on sample surfaces. Sample surfaces were less shiny than typical aluminium foil. The aluminium was contaminated with ash after heat treatment. The amount of ash is approximated in table 5.1, but the ash contents were not quantified or chemicallly analysed. Whilst the sample material faced some handling during weight measurements and compacting, no direct measures to remove ash was made before remelting, in efforts of staying true to planned methodology. The UBC sample material fell apart, not adhering after compaction as Foils and Cans, which acted more like a solid piece of material after compaction.

Figure 5.1: Samples of Cans (left) and Foils (right) before preliminary heat treatment.

Figure 5.2: Preliminary: UBC (left), Cans (middle) and Foils (right) after heat treatment

Figure 5.3: Preliminary: Cans (left) and foil (right) after compaction

The results from preliminary remelting of Cans, Foils and UBC are listed in table 5.2.

Table 5.2: Yield from preliminary remelting.

Batch	Heel	Sample	Dross	Decanted	Yield
Foils	551.0 g	176.0 g	184.5 g	522.4 g	−16.3 %
Comment: A lot of dross with high metal content. We suspect a exothermic reaction occurred between the ash and the liquid aluminium.					
Cans	563.8 g	213.3 g	48.4 g	686.7 g	58.1 %
corrected				721.7 g	74.0 %
Comment: Corrected with +35 g to decanted, the metal we assume could have been removed from crucible bottom, as done with UBC batch.					
UBC	648.8 g	218.2 g	51.3 g	807.3 g	72.6 %
Comment: Removed 37 g metal from bottom of crucible.					

The preliminary remelting was performed using the same remelting procedure as the main remelting experiments described in section 4.4 with two main differences: There was only 1 addition of sample material per experiment (coupled with 1 addition of pure AL), and there was no cleaning of the crucible between experiments. After remelting of the UBC sample, the crucible was cleaned out using pressurized air and the iron rod. Some bottom dross and a metal sheet of significant size (37 g) was extracted, this is the basis of the correction made in table 5.2.

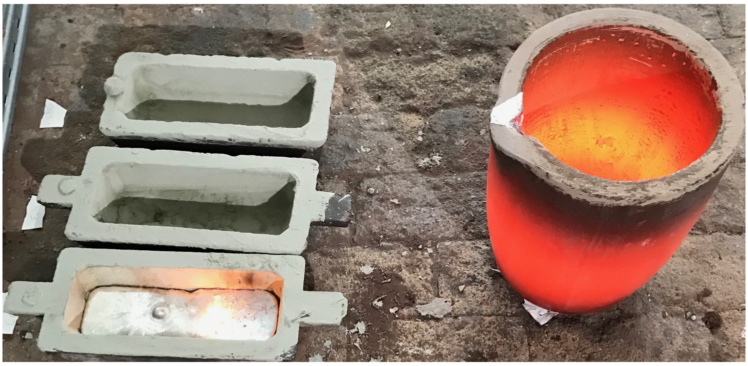


Figure 5.4: Sample of remelted cans next to still molten crucible.

The temperature in the crucible was measured with a thermocouples before and after addition, and assessed visually through the color of the melt. Upon reaching temperatures of 800 °C the material starts glowing red. The temperature was 700 °C before adding Cans and UBC, and 800 °C before adding Foils. After addition of foils white light was emitted from the crucible. The metallurgical glove used to hold the iron pike caught fire, and temperature measurements read 1300 °C. These temperature measurements were made after the furnace power had been shut off, and higher temperatures were likely reached. This is much hotter than the temperature reached in any experiment performed in this project, which rarely superseded 850 °C.

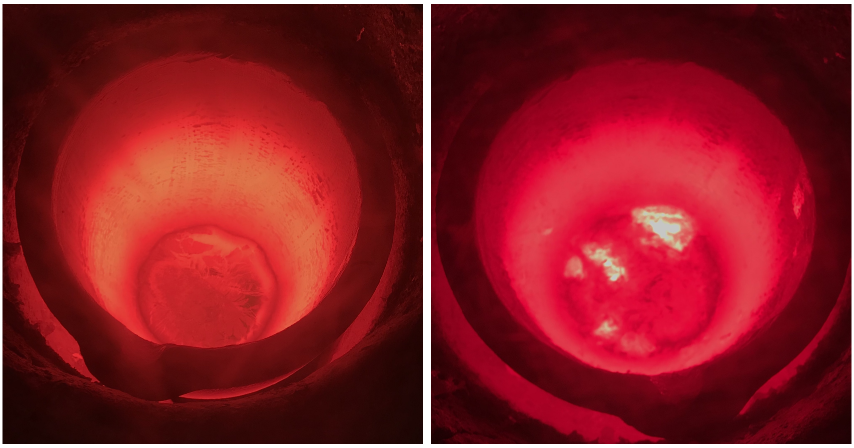


Figure 5.5: Crucible with liquid aluminium from pure aluminium and added samples of Cans (left) and Foils (right).

Figure 5.6: Empty crucibles after remelting Cans (left) and Foils (right). The left crucible contains a sheet of metal whilst the right crucible contains bottom dross.

5.2 Decoating

Table 5.3 lists the weight measurements and calculated mass losses from heat treatment of the Cans sample material. The sample material was decoated over three days, and culminated in an unexpected incident at the end of the last batch in which the furnace door, discussed below in section 5.2.3. Heat treatment was by far the most challenging and the most hazardous operation of the project. The sample material would lose on on average 42 % of it's mass during the treatment. This mass evaporated leaving the chimney as a heavy smoke that often fell towards the ground after leaving the chimney. Due to complaints of an odour similar to burnt food by the floors above the laboratory, the heat treatments were performed after working hours.

Table 5.3: Weight measurements from heat treatment with calculated mass loss, per batch and per sample material, by average and in total.

By batch:

Batch: –	Temp. [°C]	Size before [kg]	Size after [kg]	Mass loss [kg]	Mass loss [%]
1	550	14.2	8.4	5.8	41.0
2	550	14.8	8.2	6.6	43.8
3	550	15.4	8.5	6.9	44.6
4	550	21.4	12.3	9.1	42.2
5	300	16.8	9.5	7.3	42.1
6	300	23.8	13.6	10.2	42.7
7	300	19.5	12.1	7.4	37.4

Averages weight measurements per portion by heat treatment temperature:

Temp. [°C]	Portions #	Size before [g]	Size after [g]	Mass loss [g]	Mass loss [%]	St.Dev. [%]
550	24	2743	1555	1887	42.9	6.0
300	13	3388	1929	1458	42.0	5.5
Both	37	2958	1680	1278	42.6	5.9

Total mass heat treated:

Temp. [°C]	Size before [kg]	Size After [kg]	Mass loss [kg]	Mass loss [%]
550	65.8	37.3	28.5	43.3
300	60.2	35.3	24.9	42.60
Both	126.0	72.6	53.4	42.4

5.2.1 Decoating of Cans at 550 °C

Cans decoated at 550 °C had:

- Shrank noticeable in volume;
- lost on average 43 % of it's mass;
- it's surfaces had lost all color and turned metallic grey matt (non-glossy) or white;
- evidence of melting (disintegrated pieces of sample material coupled with metal droplets);
- residual grey ash on sample material or in bottom of troughs;
- very little odour.

The decoating lasted for approximately 90 minutes and was considered ended when the process stopped generating smoke. At the later stages of decoating the furnace chamber regularly measured 600 °C, due to combustion of pyrolysis products. As will be shown in 5.2.3, the temperature in the centre of the sample portions is assumed to have reached much higher temperatures. This was also evident from melted material. The surface material turning metallic grey matt was an indicator that the material was successfully decoated. The observations which indicated melting, namely reduced volume, droplets and disintegrated material was more evident on samples decoated in taller troughs. Some surfaces were covered white, probably due to remaining titanium oxide from, a widely used white pigment. The grey ash was partly removed from the sample material during de-ashing. Some as fly-ash, whilst some ash was recovered from the bottom of the cement mixer used for ash removal.

Figure 5.7: Examples of sample pieces from Cans 550. A partly opened canned food can filled with black char (left). A partly disintegrated aluminium tray (right).

5.2.2 Decoating of Cans at 300 °C

Cans decoated at 300 °C had:

- Lost on average 42 % of it's mass;
- its surfaces turn black;
- no evidence of melting or reduction in size;
- residual black ash on sample material or in bottom of troughs;
- buildup of tar in bottom of troughs;
- an odour similar to burnt food.

The decoating lasted for approximately 180 minutes and was considered ended when the process stopped generating smoke. At the later stages of decoating the furnace chamber regularly measured over 350 °C, due to combustion of pyrolysis products. As will be shown in 5.2.3, the temperature in the centre of the sample portions is assumed to have reached much higher temperatures. The black surface of the material was likely a carbon based residue from organic coating. In contrast to Cans 550, the material showed no indications that melting had occured. The black ash was partly removed from the sample material during de-ashing. Compared to de-ashing of Cans 550, less fly-ash was observed.

Figure 5.8: Observations from decoating of Cans 300. A trough filled with pyrolysis products. Pieces of aluminium adher to thee trough due to the glue-like properties of the pyrolysis products (left). A pieces of aluminium glued together by partly melted cheese and wax from prematurily ended decoating of batch 7 (right).

5.2.3 Temperature measurements and incident of batch 7

Temperature measurements were made using thermocouples during decoating of batch 7 (at 300 °C). Thermocouples 1, 2, 4, 5 and 6 were positioned were squeezed inside the troughs at the sides, thermocouple 3 was positioned the middle of the samplematerial in a trough, whilst thermocouple 7 was placed in the middle of the furnace chamber. The temperature measurements are presented in figure 8.10.

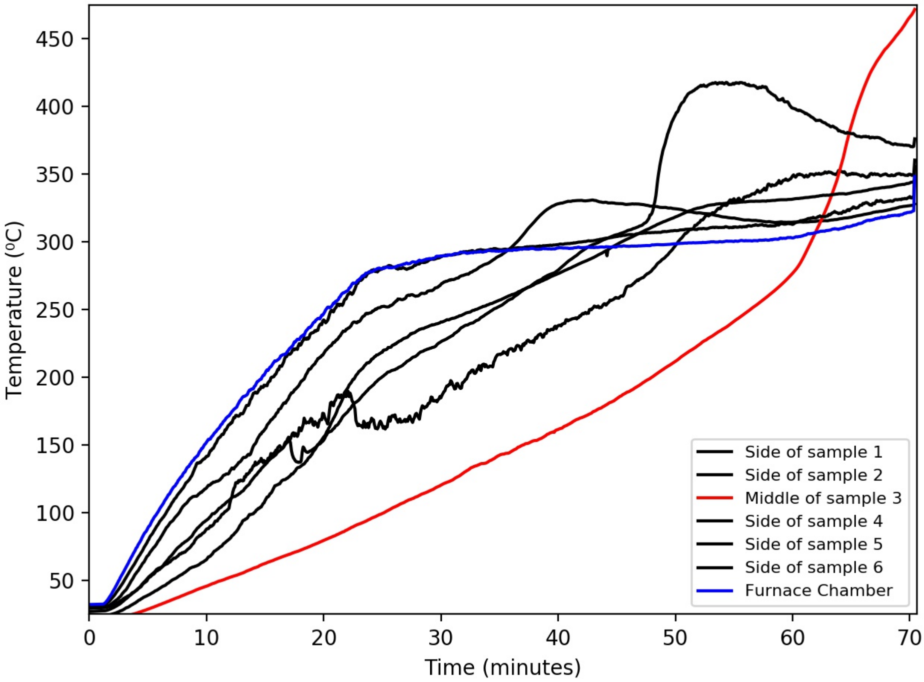


Figure 5.9: Results: Temperature measurements during de-coating of batch 7.

During dec-oating the temperature of the furnace chamber was always measured to be within 1 to 2 °C of the temperature displayed on the furnace control panel, affirming the accuracy of the furnace display. The temperature is the middle of a sample lagged greatly behind the temperature in the furnace chamber, 200 °C behind 30 minutes in. The temperatures of the samples all increased rapidly after having reached about 300 °C.

Shortly after the 70 minute mark we can see the temperature measurements increasing sharply. At this point in time the furnace door was forcecibly opened, bending it's locking mechanisms, by pressure building in the furnace chamber. The furnace door swung open colliding into the barrel placed infront of it. The door didn't swing violently, evident by the only minor displacement of the barrel. As the door opened the contents of the furnace caught fire, producing smoke which started the fire alarm. The furnace contents were extinguished, and the windows were opened to air out the furnace hall.

Due to construction acitivities in the furnace hall (which is designed to be smoke- and fire proof, smokedetectors below the furnace hall were activated, and the emergency serevices secured the area shortly after. The laboratory workers present were examined for smoke inhalation and decleared unharmed by medical personel. The incident was reported to NTNU Emeregency, and thereafter HSE reports were filed to NTNU and SINTEF through the proper channels. The incident, it's cause and what should be learned will be discussed in chapter 6

5.2.4 Images and Observations from decoating

- The cans sample material consisted mostly of empty beverage cans and empty canned foods cans.
- Canned food cans were seldom properly opened. Often they were barely opened with significant amounts of food stuff left inside.
- Used aluminium coffee pods always had residual coffee left in them.
- Aluminium trays, such as those used to cook poultry in professional kitchens, were always curled up and filled with fat. Post heat treatment these were often significantly melted.
- Aluminium tubes that carried cream cheese spreads, make-up and medicinal ointments were generally intact upon arrival, and filled with solid masses post heat treatment. Some of this solid mass seemed to be carbonaceous in nature, whilst others were rich in color, probably from pigments.
- The primary pollution in the compacted, heat treated material were aluminium-clad glass necks from wine bottles.
- Some iron scrap got mixed into the sample material. This metal did not dissolve upon melting.
- Only one thin strand of copper was observed in the sample material.
- Decoating in taller containers led to more melting of sample material and more oxidation, possibly due to larger portions and a more protected portion centre.
- Decoating at 550 °C

Figure 5.10: Cans sample material before decoating.

5.2.5 Further pre-treatment

Ash was removed from Cans 300 and Cans 550 prior to compaction using a cement mixer. The sample materials were then compacted using a 35 MPa press. The compaction reduced the volume of the samples to less than $\frac{1}{6}$th of the original volume. After compaction the samples adhered together in looking like A4-page size briquettes of varying heights. These bricks were too large to be practically added to the crucible in 2 kg additions. They were therefore cut into pieces using a metal band saw. This introduced some lubricant into the samples. Cut samples were therefore preheated to $200\,^{\circ}\mathrm{C}$ with the same furnace used for decoating. Figure **??** depicts the sample material before and after pretreatment. Through the course of pre-treatment the sample material had lost approximately $47\,\%$ of it mass. Before compaction 6 barrels of volume $250\,\mathrm{L}$ for a total volume of $1.5\,\mathrm{m}^3$ were required to hold the sample mass. After compaction the sample mass all fit in one barrel.

Figure 5.11: Cans sample material decoated at 300 °C - *Cans 300*

Figure 5.12: Cans sample material decoated at 550 °C - *Cans 550*. In thee middle we see a white porous material deposited on the side of the trough surrounding sample material.

5.3 Results from remelting

10 remelting experiments were performed over a duration of 3 days: 4 with Cans 550, 4 with Cans 300 and 2 with pure aluminium. Each addition consisted of 3-4 pieces which weight ranged from 200 g to 1100 g. These pieces would be combined so that the total weight of the addition would be as close as possible to 2 kg. The pure aluminium was prepared by cutting aluminium ingot into 2 kg pieces. All identical activities in each experiment were performed by the same laborant(s).

From the remelting experiments we logged a data set consisting of the following data series:

1. Mass of the crucible before addition of sample material, recorded with the digital scale carrying the crucible;
2. mass of the crucible after addition of sample material, recorded with the digital scale carrying the crucible;
3. mass of the crucible after slag had been extracted, recorded with the digital scale carrying the crucible;
4. mass of the sample material added to the crucible:
    - 4a Measured by a separate digital scale
    - 4b obtained by subtracting series 1 from series 2.
5. mass of the dross skimmed from the crucible:
    - 5a Measured by a separate digital scale
    - 5b recorded with the digital scale, obtained by subtracting series 3 from series 2.
6. the yield in kg: mass of added sample material remaining in the crucible after skimming, computed by subtracting series 1 from series 3.
7. the weight of the bottom dross;
8. the weight of the metal decanted from the crucible;
9. the weight of the bottom metal removed from the crucible.
10. the temperature of the crucible before addition of sample material; *
11. the temperature of the crucible after addition of sample material; *
12. the time at which each addition was begun; *

Recording the data series (marked with *) wasn't deemed a priority during experiments due to time constraints, and at times omitted.

5.3.1 Interpretation of weight measurements

Since we have paralell sets of values for data series 4 and 5 we must chose which data to use. This is important since data series 4 is used to calculate yield. Data series 4a and 4b, and 5a and 5b differed significantly at first. This was due to an inherent weakness of the measuring method for data series 1 - 3, which translated into errors in data series 4b and 5b.

The inherent weakness is described as following: During experiments the crucible would be subjected to a lot of stirring, both during addition of sample material and skimming of dross. The diameter of the crucible was smaller than the diameter of the refractory larger, but not to a sufficient degree. During evaluation of the data set it became clear that the crucible had come in slight contact with the refractory wall, reducing the recorded weight somewhat.

Data series 4a and 5a are considered more reliable because they were made on a separate scale, where errors were easier to avoid. Therefore data series 1. - 3. were revised based on the differences between series 4a. vs 4b. and 5a. vs 5b, to produce a new, revised data set. The revised data set has much lower variance than the original. Furthermore the difference between series 4a and 4b, 5a and 5b are now tolerable. The original, revised and difference data sets are too large to print in this thesis, and therefore come attached.

Figures 5.13 and 5.14 present the data series 4 - average weights metal added and data series 5 - dross skimmed per addition. Parallels 4a and 5a are hereinafter denoted *Measured* whilst parallels are hereinafter denoted *Recorded.*

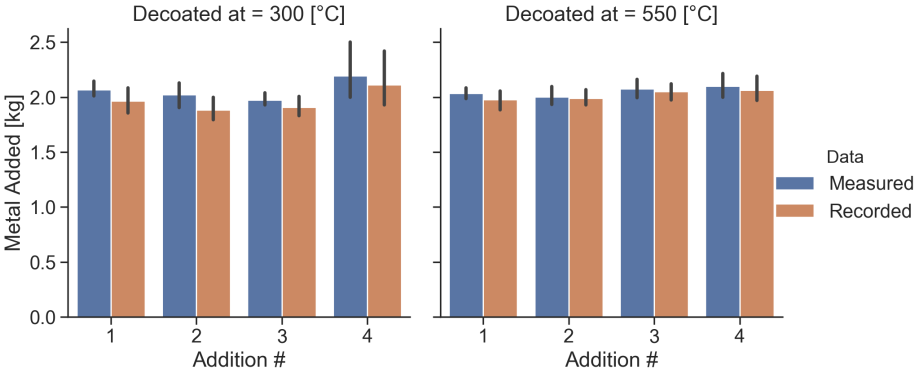


Figure 5.13: Sample material added to the crucible on average, per addition, for Cans 550 and Cans 300.

Note that the difference between recorded and measured metal added in figure 5.13 is larger for Cans 300 than for Cans 550. This is due to the burn-off of the carbonaceous residue on Cans 300. The matter which will burn during addition is included in the *measured* sample material weight, and omitted in the *recorded*sample material weight. Since there is next to no burn-off with the Cans 550 sample material comparisons between

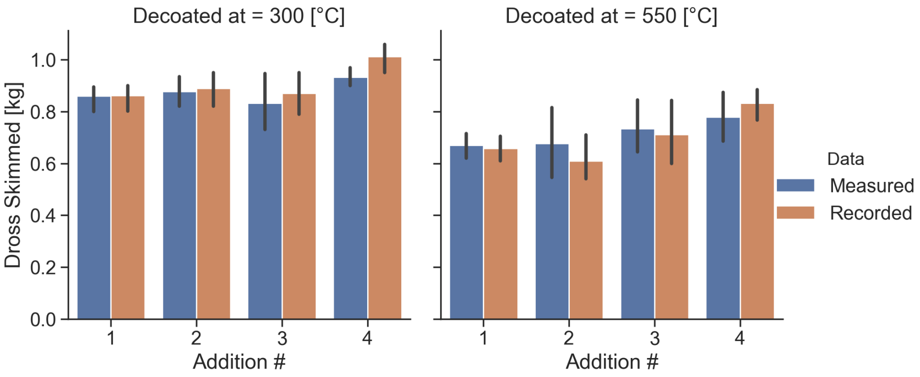


Figure 5.14: Dross skimmed from the crucible on average, per addition, for Cans 550 and Cans 300.

yields computed using *recorded* weights are more valid than yields computed using *measured* weight. Because of the considerations above yield is hereafter computed using the *recorded* sample material added - data series 4b, a data series computed based on data series 1 - 3, which were revised based on data series 4a and 5a.

Data from series 4b, 5b and 6 - sample material added, dross skimmed, and yield in kg, for each addition, for two typical experiments, one with Cans 550 (experiment 6) and one with Cans 300 (experiment 8), is presented in figure 5.15.

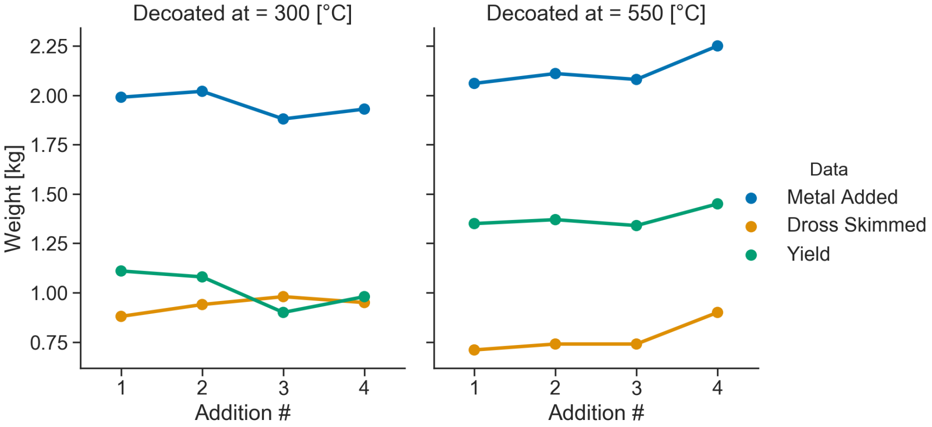


Figure 5.15: Typical Metal added, dross skimmed, and yield per addition of remelting experiments 6 - Cans 550 and 8 - Cans 300

5.3.2 Yield

An overview of the number of additions, date, sample material and average yield of each experiment is presented in table 5.4. To calculate yield the following equation is retrieved from chapter 3

$$\text{YIELD}^i = \frac{m^i_{cru,skimmed} - m^i_{cru,before}}{m^i_{cru,after} - m^i_{cru,before}} \tag{3.3}$$

$m^i_{cru,skimmed}$ translates to data series 3, $m^i_{cru,before}$ translates to data series 1, $m^i_{cru,after}$ translates to data series 2.

$$\text{YIELD}^i = \frac{\text{DATA } 3^i - \text{DATA } 1^i}{\text{DATA } 2^i - \text{DATA } 1^i} = \frac{\text{DATA } 6^i}{\text{DATA } 4\text{B}^i} \tag{5.1}$$

Date	Experiment #	# of additions	Sample Material	Average Yield
29.05.2019	1	4	Cans 550	71.0 %
30.05.2019	2	4	Cans 300	67.0 %
30.05.2019	3	4	Pure Al	100.0 %
30.05.2019	4	4	Cans 550	73.2 %
30.05.2019	5	4	Cans 300	60.4 %
30.05.2019	6	4	Cans 550	71.0 %
31.05.2019	7	3	Pure Al	100.0 %
31.05.2019	8	4	Cans 300	61.6 %
31.05.2019	9	3	Cans 300	65.5 %
31.05.2019	10	4	Cans 550	74.0 %
Average Yield of Cans 550: 65.3 %, with standard deviation 4.9 %. Average Yield of Cans 300: 53.8 %, with standard deviation 4.5 %.				

Table 5.4: Average yield, sample material, number of additions and date of each remelting experiment.

5.3.3 Data recorded per addition

The yield, relative dross formed, time lapsed, mean temperature and relative addition, per addition for each experiment (except experiments 3 and 7, with pure aluminium) is presented in figures 5.16 - 5.20. These results per addition are used to look for correlations between yield, relative addition, time and temperature in chapter 6 - Discussions. An overview of the sample material used in each experiment is found in table 5.4.

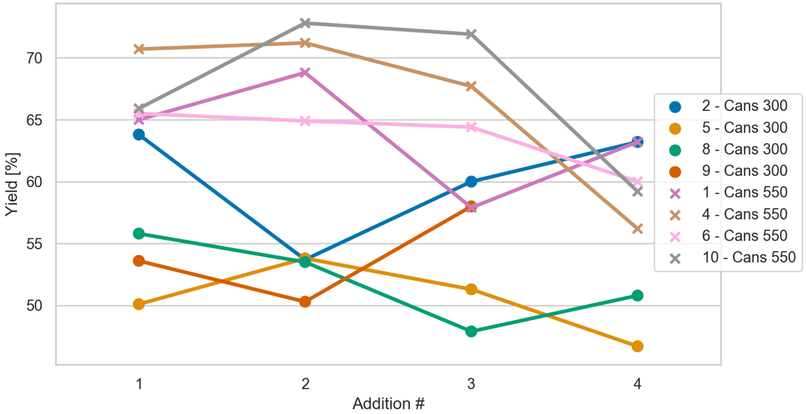


Figure 5.16: Yield per addition for experiments with Cans 550 and Cans 300.

5.3.4 Observations from remelting experiments

- When dipped into the molten melt, Cans 300 released large amounts of flammable gas, as shown in figure 5.21. The gas would visibly bubble through the melt if the sample was completely submerged. The flammable gas invariably started burning. The flames would stand out of either the sample material, or even cover the entire crucible, depending on the experiment, and on addition.
- Significant amounts of dross were formed when remelting both Cans 300 and Cans 550.
- The melt would be cooled down to 660 °C several times each addition, each time a new piece of sample material was placed into the crucible.

5.3.5 Results from OES analysis

A sample from each experiment was analysed with Optical Emission Spectroscopy, wherein the characteristic electromagnetic radiation emitted from a dischange of plasma generated from the sample material using electrical charge. The contents of the major alloying elements, Si, Fe, Cu, Mn, Mg and Zn, are shown in figure 5.22, whilst the contents of the minor alloying elements, Ti, Cr, Ni, Pb and Sn, are shown in figure 5.23.

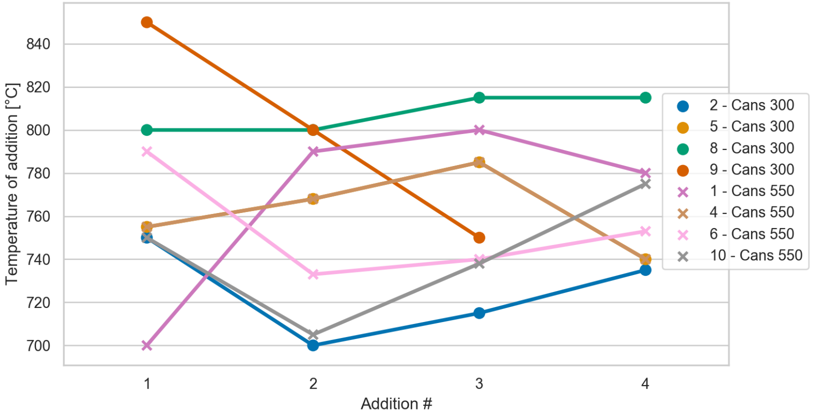


Figure 5.17: Mean temperature per addition for experiments with Cans 550 and Cans 300.

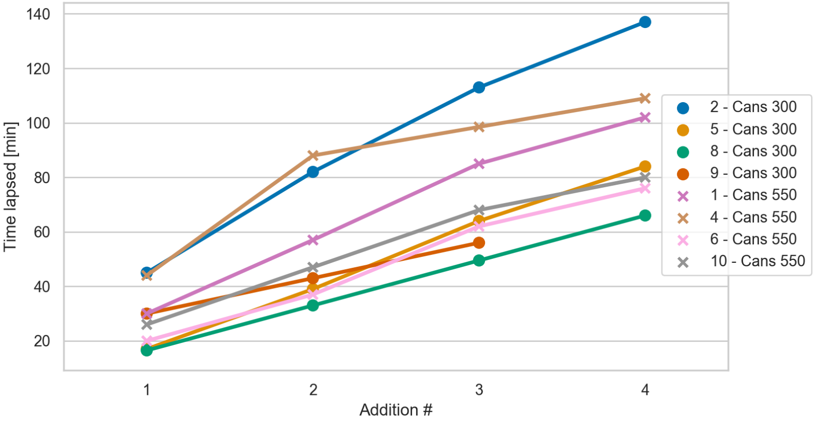


Figure 5.18: Time lapsed per addition for experiments with Cans 550 and Cans 300. Many of the points presented are estimated through linearization.

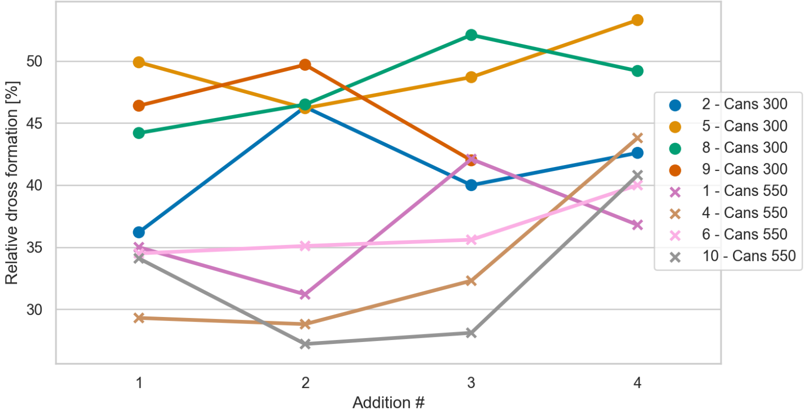


Figure 5.19: Relative dross formed per addition for experiments with Cans 550 and Cans 300.

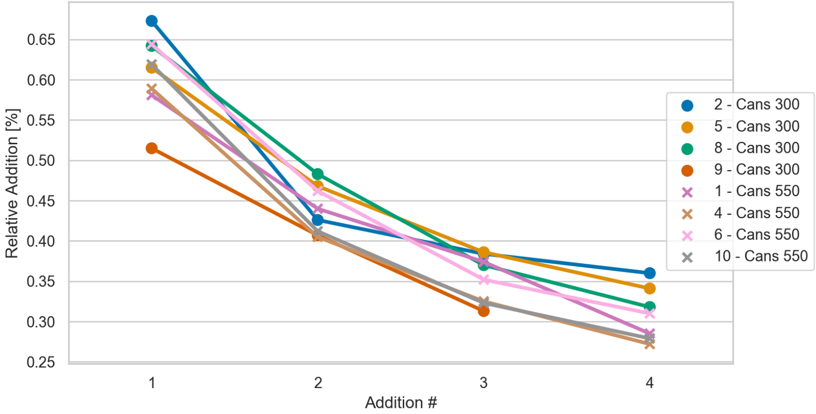


Figure 5.20: Relative addition per addition for experiments with Cans 550 and Cans 300.

Figure 5.21: Combustion of flammable gases released from Cans 300 during remelting.

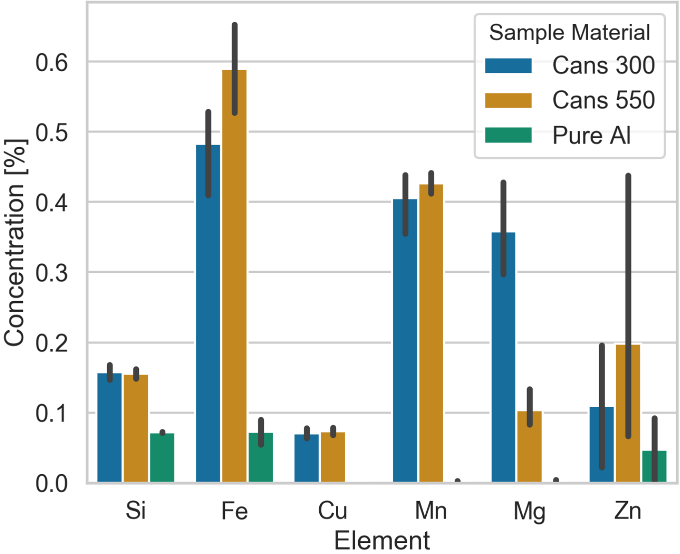


Figure 5.22: Content of major alloying elements of each sample material with 95 % confidence intervals.

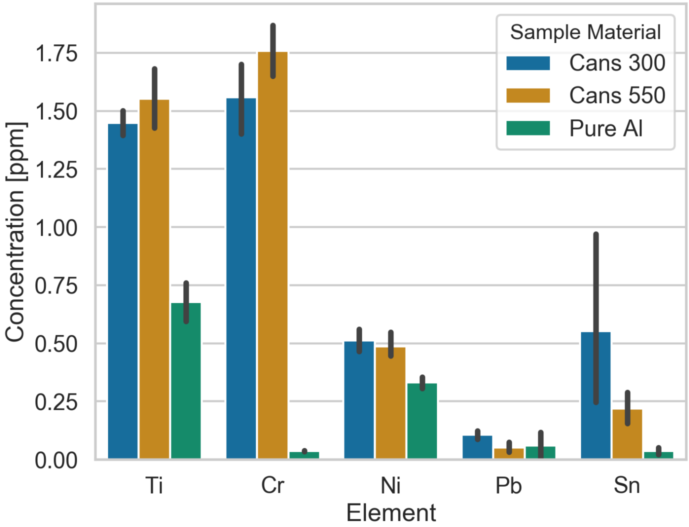


Figure 5.23: Content of minor alloying elements of each sample material with $95\,\%$ confidence intervals.

5.3.6 Results from PoDFA analysis

he results from PoDFA (Porous Disk Filtration Apparatus) analysis of each experiment are displayed in figure 5.24. Experiment 1 had a large quantity of unknown inclusion particles designated unknown that weren't observed in any other experiments in addition to a very high content of carbides. The carbides contents of experiments 1, 8, 9 and 10 were approximately 500 times those of experiments 2-7.

The Ti-rich particles could stem from titanium boride TiB_2 which is added to aluminium as a grain growth inhibitor.

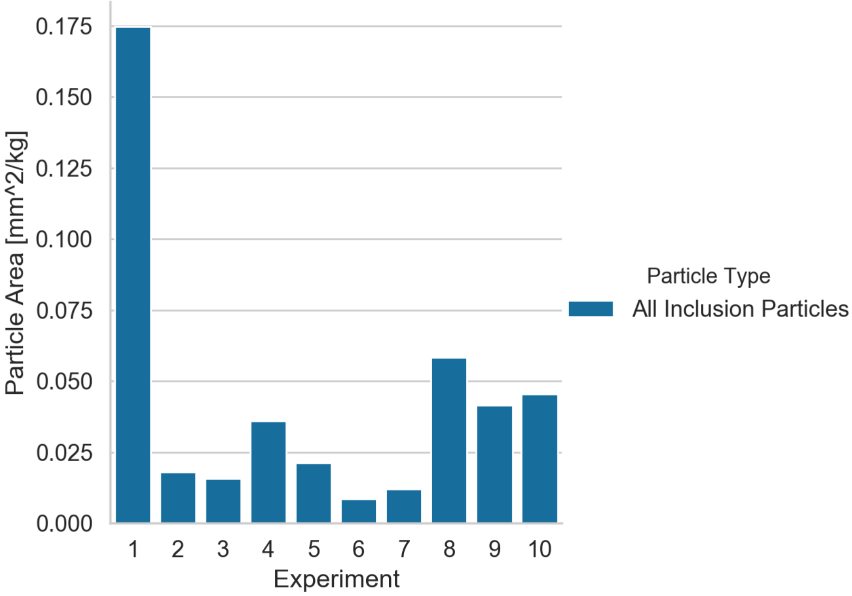


Figure 5.24: Total inclusion content in mm^2/kg for each experiment, obtained through PoDFA analysis. The large content of experiment 1 is classified almost entirely as *unknown*. Experiments 1,4,6,10 from Cans 550, 2,5,8,9 from Cans 300 and 3,7 from pure aluminium

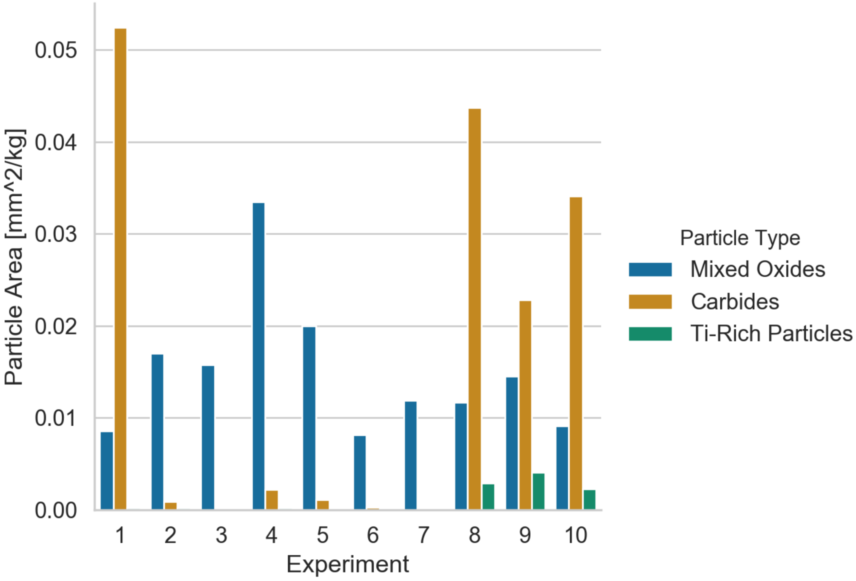


Figure 5.25: Inclusion content in mm^2/kg for different inclusion types, for each experiment, obtained through PoDFA analysis. Experiments 1,4,6,10 from Cans 550, 2,5,8,9 from Cans 300 and 3,7 from pure aluminium

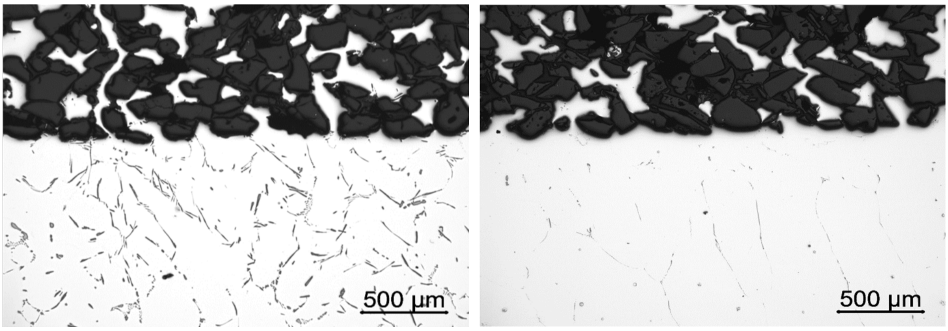


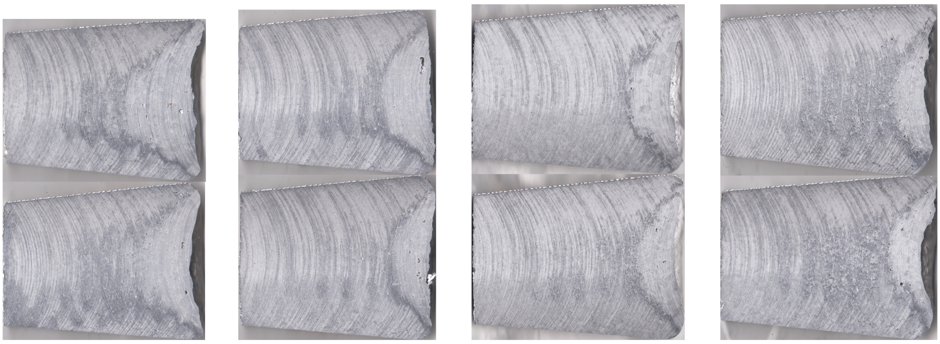
Figure 5.26: Scanning Electron images of PoDFA analysis filters, experiment 6 - Cans 550 (left) and experiment 7 - Pure Al (right)

5.3.7 Results from Reduced pressure testing (RPT)

Selected RPT samples are displayed in figures 5.27 - 5.30. The cut and polished samples from RPT were received less than a week before the due date of the project. Quantifying the porosity by microscope was not possible in the available time frame. Through visual assessment porosity seems to be increasing in the order of: Pure aluminium $<$ Cans 550 $<$ Cans 300 Exp. 5 $<$ Cans 30 Exp. 8. The pictures are found in a larger format in the appendix section 8.7

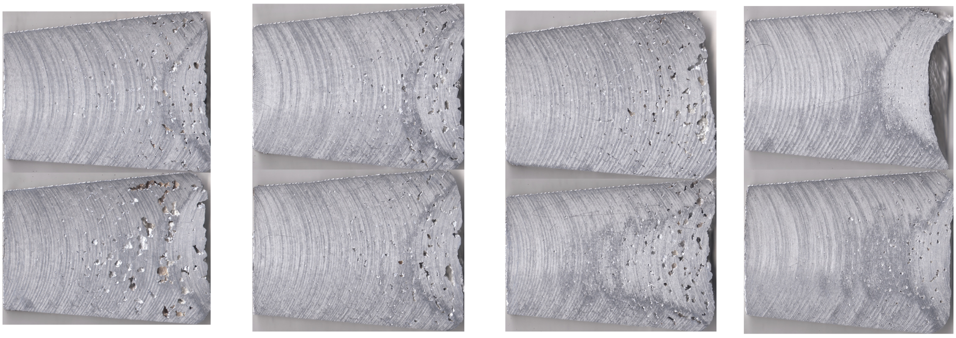
3.1 3.2 3.3 3.4

Figure 5.27: Polished RPT samples from experiment 3 - Pure Al.



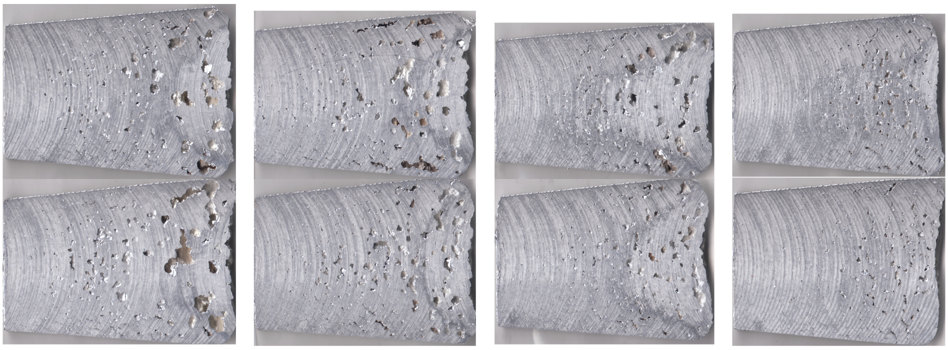
6.1 6.2 6.3 6.4

Figure 5.28: Polished RPT samples from experiment 6 - Cans 550.



5.1 5.2 5.3 5.4

Figure 5.29: Polished RPT samples from experiment 5 - Cans 300, with low inclusions content



8.1 8.2 8.3 8.4

Figure 5.30: Polished RPT samples from experiment 8 - Cans 300, with high inclusion content.

Chapter 6

Discussion

6.1 Preliminary experiments

The main objective of the preliminary experiments were assessing the viability of the planned experimental methodology for use in this project using the Cans, Foils and UBC sample materials.

The weight losses found during decoating showed that to produce 50 kg of decoated sample material, 100 kg of untreated cans would be required, and 500 - 1000kg of untreated foils, due to the high ash contents of foils which was assumed to make up between $1/3$ to $2/3$ of the decoated sample material. This observation was the reasoning behind the reluctant decision to omit Foils in the main experiments. The decoated carbons either end up in the ventilation system or in the campus air, and a metric tonne of pollution was deemed too much.

Qualitatively we observed that the yields of remelting Cans and UBC are similar. There is no basis for a statistical conclusion due to only having one data point of each yield, but this reinforced the choice of both Cans and UBC as a well fitting sample material for the main series of experiments.

The remelting of Foils was very interesting: When added to the melt of molten aluminium, Foils produced more dross than the total sample weight. The white light indicated a strongly exothermic reaction, which is supported by the low yield seen in table 5.2. The nature of the reaction itself is uncertain. The most plausible explanation is that air, ash or both trapped between the foil layers reacted with liquid aluminium. This is suspected to relate to the exothermic reaction indicated by the strong white light seen in figure5.5 and the unparalleled rise in melt temperature. The nature of the reaction itself is uncertain. The most plausible explanation is that air, ash or both trapped between the foil layers reacted with liquid aluminium. Therefore it was decided to remove ash from the decoated sample material in the main series of experments. Foils would have been a very interesting sample material to study, but would require more sophisticated decoating equipment than found at NTNU.

Such a decoating equipment was found at Enval in Cambridge, but the sample material dubbed EnvalFoils did not arrive in time for pre-treatment and experiments to commence. Upon completion of the project the sample material has yet not arrived.

6.2 Decoating and other pretreatment

The transformation of the Cans samplematerial from arrival through decoating, ashremoval, compaction and sizing is shown in figure6.1.

Figure 6.1: The Cans sample material at the before (left) and after (right) pretreatment.

Whilst decoating was essentially successful it can be argued that is was not optimised. The decoating of Cans at 300 °C and 550 °C produced quite similar mass losses. Judging by it's appearance Cans 300 is covered in residual carbonaceous char due to an unfinished combustion of pyrolysis products: A lower loss of mass is expected for Cans 300. Therefore the similar mass loss could indicates mass fain for Cans 550, from oxidation during decoating. This is supported by numerous observations of oxidised Cans 550 during experiments and sample handling. Figure **??** shows the contents of a trough flipped upsides down: The materials are very light in colour or white, signs of oxidation.

Moreover we observed differences in how deformed and oxidised Cans 550 appeared depending the height of the troughs we used to hold sample material during decoating. Taller, larger troughs produced more oxidised and more deformed products. The taller the trough the more isolated the contents are from the surrounding furnace chamber, and the higher the temperature is reached in the sample mass during decoating. Higher temperatures again lead to more melting of sample material and more oxidation.

Figure 6.2: The contents of a large trough of Cans decated at 550 °C flipped over. The material seems to be covered in metal oxides.

It is therefore unsurprising as there are no trough based decoating systems available. Perhaps perforated troughs would have produced a more evenly decoated material.

At 300 °C decoating wasn't completed, whilst at 550 °C decoating was completed, but the samples were allowed to oxidise. In the optimal process, decoating is completed with minimal oxidation, which could be achieved through proper temperature control an with little variance in temperature and contents of both oxygen and pyrolysis products in the decoating chamber. The temperature measurements shown in figure 8.10 indicate this wasn't the case during decoating in the process.

The kiln decoater and the fluid bed decoater described in section 3.8 could provide the desired process control. The optimal decoating process would be characterised by highest mass loss possible, which is achieved through completed decoating combined minimal oxidation.

6.2.1 Batch 7 - Temperature measurements and incident

Temperature measurements of batch 7 shows that the temperature in the middle of a batch differs strongly from the temperature in the furnace chamber. First the temperature lag behind as evaporation of moisture and scission reactions dominate, then the temperature pulls ahead as combustion beings and the reactions turn more exothermic.

Cause of incident

The incident was caused by a pressure buildup in the furnace. So much is clear from viewing the bent bolt which held the door shut. It is also believed that the bending of the bolt absorbed most of the energy, meaning that the door didn't built up any significant torque while opening. This is assumed as the door didn't have enough torque to push away the empty barrel positioned in front of it.

As described in section 3.8.2, for an ideal gas in a constant volume, the pressure depends on the temperature of the gas, and the number of gas molecules present. If you double either the number of gas molecules, or the temperature, the pressure doubles as well.

The sample material decoated in batch 7 came from the very bottom of the container holding the raw Cans sample material, where the densest material gathered. The batch wasn't specifically large, but the material was split in 4 large troughs rather than 6, and it was particulary dense.

As shown in figure 8.10 the temperature in the middle of the samples exceeded the temperature of the furnace chamber around the 70 minute mark, which was near the end of the decoating process. In the dense sample, a lot of hydrocarbones were concentrated on a realtively small volume. At some point the reactions in the middle of the sample became increasingly exothermic. This led to a sharper than usual temperature increase in the furnace chamber. As evident of picture**??** there was still hydrocarbones in the furnace that would need to be pyrolised. The increase in temperature drastically increased reaction speed of the scission reactions, leading to the significant production of gas molecules which resulting pressure increase opened the furnace door.

It is assumed that if lower quantities of sample materials had been decoated, this likely wouldn't have occured.

It is therefore advised (and genereally agreed upon) that future decoating experiments must be performed with specialised decoating equipment, including proper off-gas filtration systems.

6.3 Yield and dross formation

6.3.1 Decoating temperature

The statistical reasoning and testing of hypotheses is applied for all hypothesis below. The reasoning and testing is explained in full for hypothesis 1, and for hypotheses 2 - 4 only the summaries are presented. The statistical reasoning in full for each hypothesis is found in section 8.8 in the appendix.

The yields of remelting Cans 300 and Cans 550 (illustrated in figure 8.12 were hypothesised to be equal in null hypothesis 1 (In introduction). This hypothesis was tested using Minitab 2018.

With a $95\,\%$ confidence interval the difference between the yield of Cans 300 and Cans 550 was shown to be between $7.97\,\%$ and $15.13\,\%$, the mean difference being $11.55\,\%$. Since this interval doesn't include zero the test necessitates the rejection of the null hypothesis above, instead accepting the alternative hypothesis: that the two yields differed.

The P-value of the test was $3.6 * 10^{-7}$, making it valid for confidence intervals below 99.9999 percent.

Summary of testing hypothesis 1 on yield when remelting Cans 300 and Cans 550: Significantly better yield, shown with a P-value of $3.6 * 10^{-7}$, and difference of $11.55\,\%$ on average, was obtained when remelting Cans 550 compared to Cans 300. On average Cans 300 was remelted with $53.78\,\%$ yield and Cans 550 was remelted with $65.33\,\%$ yield. Reasoning found in section 8.8 of the appendix.

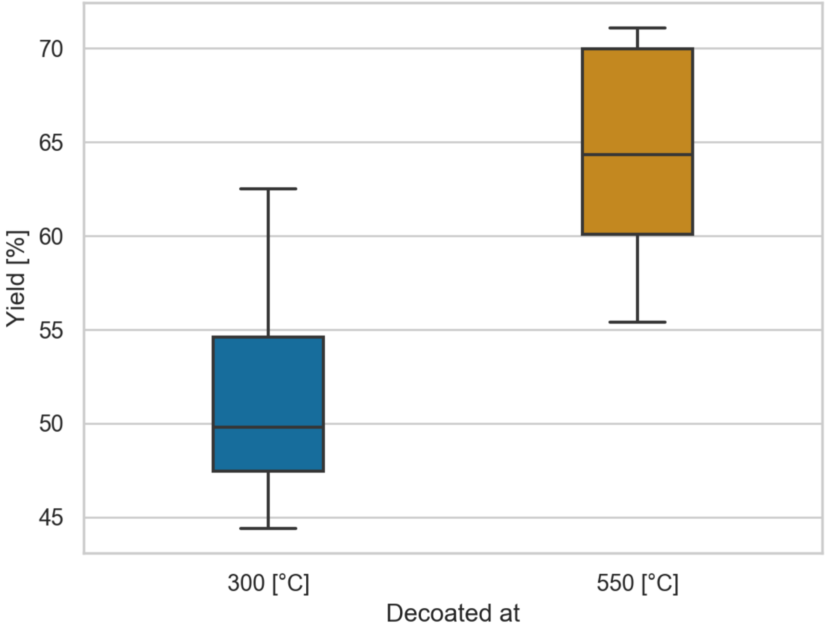


Figure 6.3: The yields of remelting of Cans 550 and Cans 300. The colored rectangles represent $95\,\%$ confidence intervals.

Summary of testing of hypothesis 2 on relative dross formation when remelting Cans 300 and Cans 550: Significantly lower relative dross formation, shown with a P-value of $3.6 * 10^{-7}$, a difference of $11.55\,\%$ on average, was obtained when remelting Cans 550 compared to Cans 300. On average Cans 300 was remelted with $46.22\,\%$ relative dross formation and Cans 550 was remelted with $34.67\,\%$ relative dross formation. Reasoning found in section 8.8 of the appendix.

The two proofs are strikingly similar down having identical intervals and P-values. Due to the way yield and relative dross formation was measured in the project they always sum to one. They are paired values and come from paired measurements. Therefore the proofs come out identical.

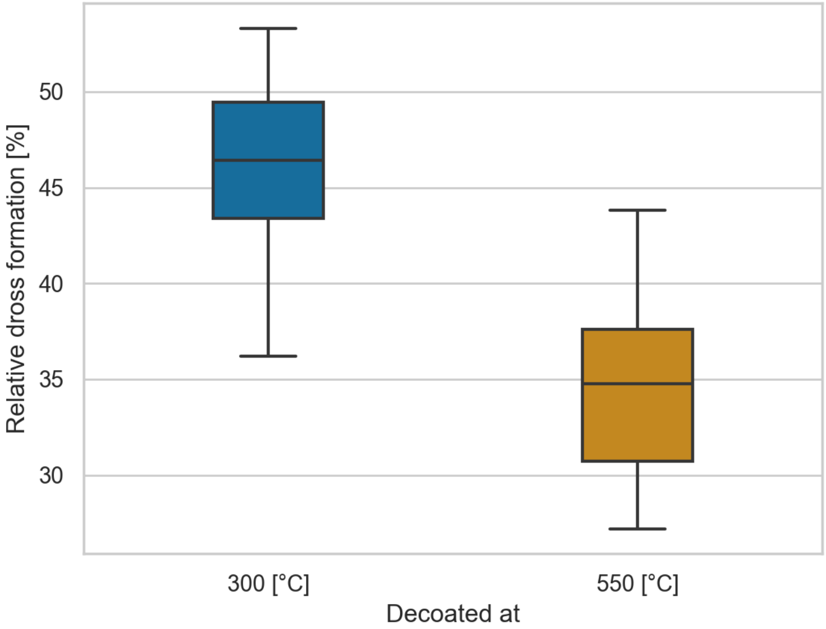


Figure 6.4: The relative dross formation from remelting of Cans 550 and Cans 300. The colored rectangles represent $95\,\%$ confidence intervals.

Why did the decoating temperature affect metal yield so profoundly, with the Cans decoated at $550\,°C$ giving a $21.4\,\%$ better yield upon remelting?

Cans 550 had formation of oxides during decoating, whilst Cans 300 was covered in char.

An aluminium melt could react with the char in three ways:

1) A direct reaction between carbon and aluminium forming aluminium carbide.
2) The carbonaceous gases released from the char as it warms up and burn leads to increased dross formation.
3) The char could prevent coalescence of metal during remelting, increasing dross formation.

1) is unlikely: Little aluminium carbide was observed in PoDFA analysis, and thermodynamics strongly favour the production of alumina over aluminium carbide. Any aluminium carbide formed could react with both CO_2 and O_2 given the opportunity. Aluminium carbide may have acted as an intermediary, but no noticeable amounts of aluminium carbide was kept.

2) is likely: During remelting of Cans 300 significant gas production and combustion

was observed. Fire indicates both the presence of CO_2 and flammable gasses like CO or CH_4. This is supported by the high concentration of magnesium in remelted Cans 300, which indicates that the magnesium oxidation didn't break away. The break away of magnesium oxidation is delayed by CO_2. Therefore we conclude the difference in yield is a direct result of the production of carbonaceous gases which increase dross formation during remelting.

3) is likely: Carbon was layered around every piece of aluminium due to the packaging method. As the aluminium melted it could become covered in carbon as described in 3.8.

It's worth repeating that the weight measurements of the Cans 300 additions used to record both yield and relative dross formation didn't include the carbonaceous residues, as they were taken after the char had finished burning. This does mean these measurements included some weight in the form of aluminium oxide. However as shown by figure **??** most of the dross formed by the presence of CO_2 consists of metallic aluminium, hence the additional mass of oxide wouldn't be too noticeable during weighing.

The Cans 550 material was visibly covered in oxide. The oxide content would lead to the formation of an initial amount of dross. Dross is often described as the leading cause of dross. If some aluminium in dross oxidises, for example due to thermite reactions, the temperature of the dross increases significantly and promotes further oxidation. Once a significant amount of dross is formed it will draw up liquid aluminium through capillary forces and oxidize it. During remelting experiments dross was skimmed right away, and this might have contributed to keeping dross formation as low as possible.

6.4 Variation in yield and relative dross formation through the experiments

For the findings of yield to be valid, it necessitates that all the experiments were performed in similar conditions. Specifically if remelting conditions were made under different conditions, which had a significant impact on yield and dross formation, it compromises the validity of our findings.

To assess how yield was affected by variations in parameters duration, mean temperature, relative addition, and experiment $\#$, the yields of each addition was plotted against these parameters in figures 6.5 - 6.8, based on these values for each addition per experiments are presented in figures 5.16 - 5.20.

For parameters duration of addition, experiment, time and temperature the trendlines show correlations with the yield. However in all cases (except for the yield of Cans 550 against temperature and time) the confidence bands surrounding these trendlines stretch out to include a horizontal line. This means we cannot prove a statistically significant correlation using a 95 $\%$ confidence interval. Further more, as noted in section 5.3, the measurement of temperature and duration was not a priority during the experiment. The measurements give a very incomplete representation of the actual duration and temperature of each addition, and we therefore dismiss these correlations. This is not a proof of no correlation, it's absence of proof of correlation based on our data. It means that in these experiments the parameters simply were not varied sufficiently to observe correlation. Notably, relative dross formation depends on yield mathematically, and the correlation between yield and relative dross formation is therefore guaranteed and unnecessary to investigate.

To summarise: We cannot reject the conclusions of differing yields between Cans 300 and Cans 550 based on variations in experimental parameters.

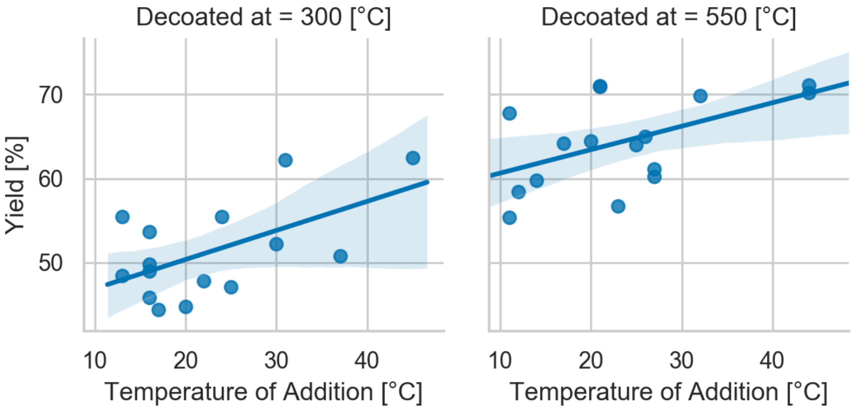


Figure 6.5: Yield per addition of Cans 300 and Cans 550 as a function of Temperature of addition, with trendlines with 95 % confidence bands.

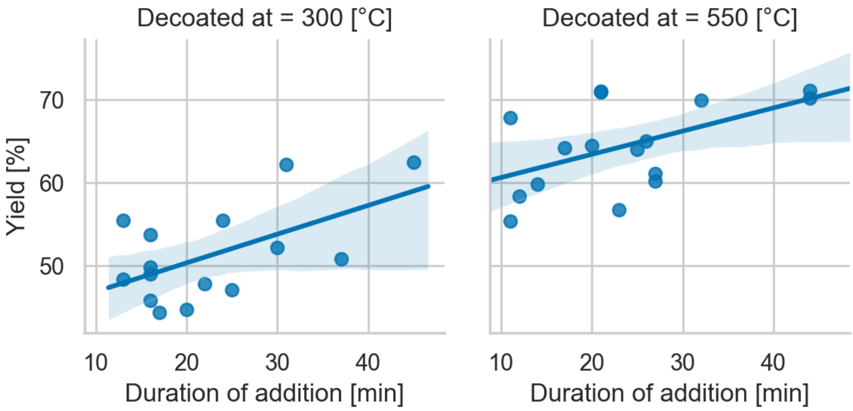


Figure 6.6: Yield per addition of Cans 300 and Cans 550 as a function of duration of addition, with a trendline and 95 % confidence band.

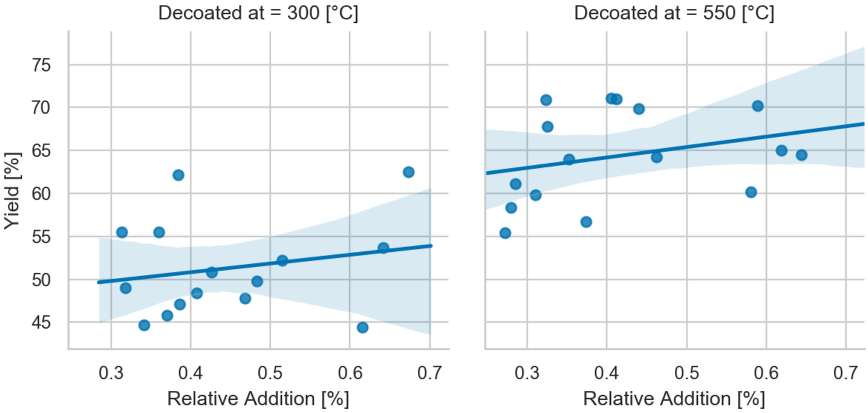


Figure 6.7: Yield per addition of Cans 300 and Cans 550 as a function of relative addition, with trendlines and 95 % confidence bands.

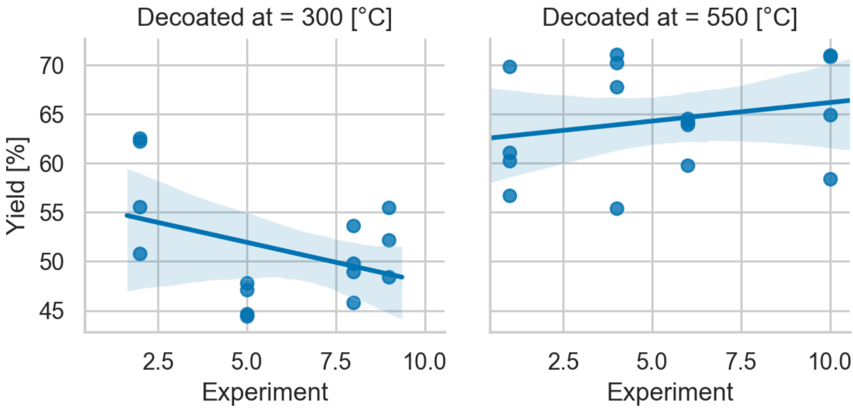


Figure 6.8: Yield per addition of Cans 300 and Cans 550 as a function of experiment, with trendlines with 95 % confidence bands.

6.5 PoDFA analysis - inclusion contents

From the PoDFA analysis we see that the inclusion area of the melt varies greatly throughout the experiments.

The difference in inclusion area between experiments 2-7 and experiment 1, 8, 9 and 10, as seen in figure **??** could be due to reapplying chamotte coating to the crucible. The chamotte coating was reapplied before experiment 2 and experiment 7. During decantation of the melt in experiment 7, a large amount of chamotte-colored bottom dross was extracted. This was found curious, but no new chamotte coating was applied and experiments continued as usual. If the chamotte-colored bottom dross was infact the improperly applied chamotte coating this would alter the crucible, and therefore the remelting conditions. This seems plausible given that the concentration of aluminium carbides in the melt increased by a factor of $\sim$500 in the following experiments. However the oxide count seems to be unaffected by this change in remelting conditions.

Figure 6.9: The chamotte-colored bottom dross cleaned out of the crucible after decantation of melt from experiment 7.

Given the explanation above is correct the inclusion count of experiments 2 - 7 are evaluated separately. In figure 6.10 shows the inclusion count for experiments 2 - 7, whilst figure 6.11 shows the calculated mean inclusion contents of each inclusions in melts from remelting Cans 300, Cans 550 and Pure Al. Cans 550 has much higher variance in oxide contents, but this isn't suspect due to the small sample size of 5.

Noticeable, the inclusion contents are practically similar in each sample material for each inclusion type, and the inclusion content is classified as very light, comparable to primary aluminium after filtration according to table 3.1.

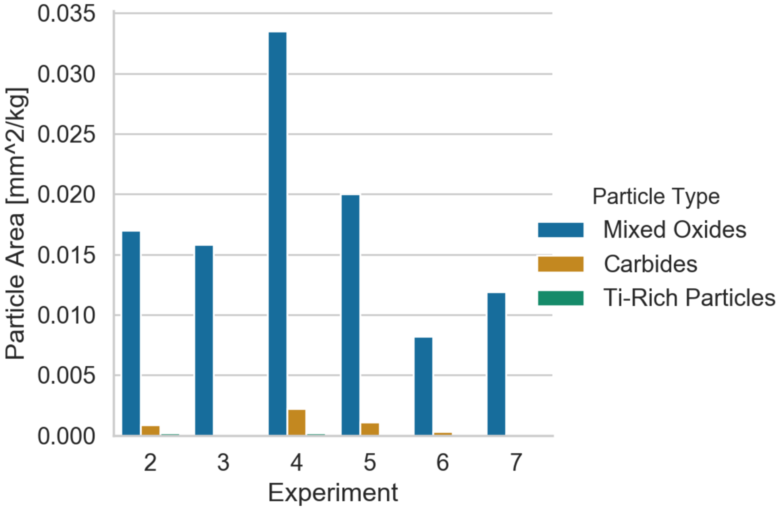


Figure 6.10: Inclusion content in mm^2/kg of different types of inclusions for experiments 2 - 7, obtained through PoDFA analysis.

However there are important weaknesses with our measurements:

- Analysis was done with cold-PoDFA not hot-PoDFA, which gives reduced measurements of inclusion contents;
- large variance of the Cans 550 inclusion contents;
- only 10 samples analysed;
- 4/10 sample measurements characterised as outliers and ommitted. However as seen in figure 5.24 experiments 2 - 10 would still be classified as very light, except experiment 8 which would be classified as light.

However, the comparable inclusion contents of Cans 300 and Cans 550 with pure aluminium in experiments 2 - 7 still indicate that remelting of Cans yields similar inclusions to remelting pure metals. In summary, the inclusion contents of Cans 300 and Cans 550 was very low and comparable to remelted pure aluminium.

6.6 Chemical analysis - alloy composition

The contents of major alloying elements in the aluminium melt, shown in figure 5.22, is much greater for Cans than for pure aluminium.

Most of the alloying elements are brought with the aluminium packaging, which is typically alloys for strength. The iron probably comes from experimental handling: the samples were decoated in steel troughs which oxidised noticeably during decoating, changing

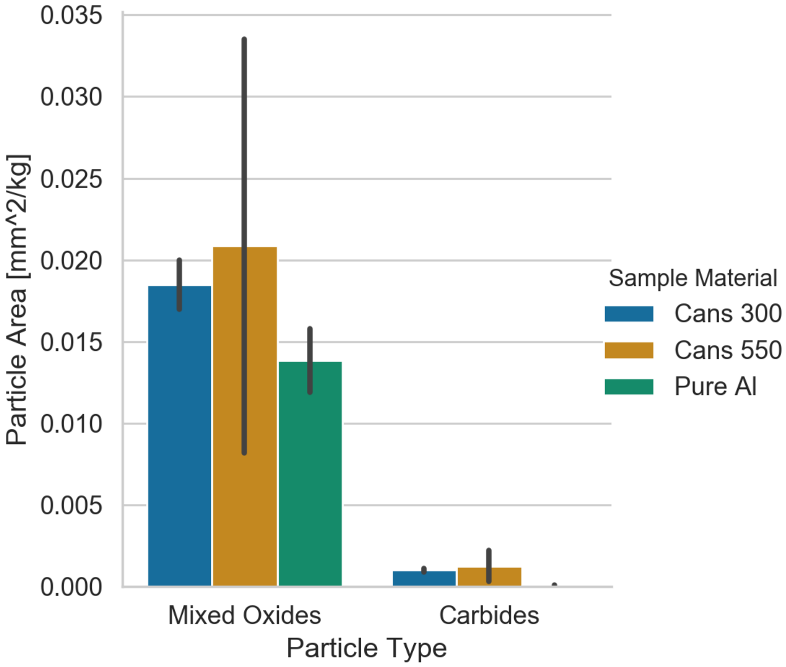


Figure 6.11: Mean inclusion content in mm^2/kg, for oxide, carbide and Ti inclusions, for remelted Cans 300, Cans 550 and pure aluminium, obtained through PoDFA analysis.

color from steel grey to rust brown. Skimming was performed with a perforated iron ladle, samples were kept submerged with an iron rod. Any rust brought with the sample material to the melt will be reduced to elemental aluminium, as shown the stability of iron oxide in figure 3.4. In an industrial scenario much of the iron content would be avoided through procedural design, reducing exposure to iron.

Manganese, magnesium, zinc, silicon and copper come from the different alloys used in aluminium packaging. These aren't easily avoided in an industrial scenario, and must be worked around through dilution with primary metal, or by using each melt for an alloy that fits it's initial composition after remelting.

By inspection figures 5.22 and 5.23 seem to reveal a higher concentration of elements Mg - magnesium, Pb - lead and Sn - tin. The same reasoning and method was utilised to reject the hypotheses, the details of the statistical argument has been left in nonetheless for those interested.

Summary of testing hypotheses 3 and 4 on magnesium and lead content in remelted Cans 300 and Cans 550: Significantly higher concentrations, with P-values of 0.01 and 0.025 for and differences of $0.255\,\%$ and $0.005\,\%$ on average for magnesium and lead respectively, was found in remelted Cans 300 compared to remelted Cans 550. Average contents of magnesium and lead were $0.358\,\%$ and $11\,\mathrm{ppm}$ respectively in Cans 300 and $0.104\,\%$ and $5\,\mathrm{ppm}$ in Cans 550. Reasoning found in section 8.8 of the appendix.

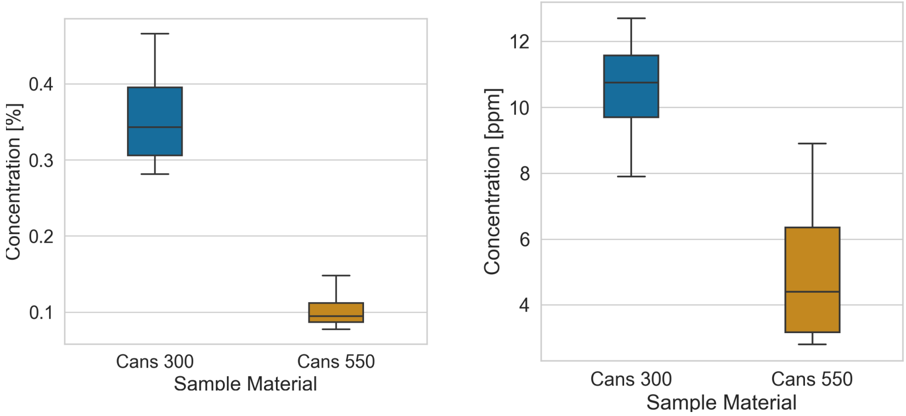


Figure 6.12: The mean concentration of magnesium (left) and lead (right) in remelted Cans 300 and Cans 550, with $95\,\%$ confidence intervals and min/max values.

Why was the concentration of magnesium and lead larger in material decoated at $300\,^{\circ}\mathrm{C}$ than at $550\,^{\circ}\mathrm{C}$?

1. Lead and/or magnesium evaporated during decoating of Cans 550.
2. Lead and/or magnesium oxidised during decoating of Cans 550.
3. Lead and/or magnesium evaporated during remelting of Cans 550, but not during Cans 550.
4. Lead and/or magnesium oxidised during remelting of Cans 3550, but not during Cans 550.

During decoating, the furnace chamber would be low in oxygen, and high in pyrolysis products which have been shown to break down the passivizing features of alumina. This would allow lead to evaporate during decoating of Cans 550. The temperatures experienced by the samples were higher than $550\,^{\circ}\mathrm{C}$, and both lead and magnesium have noticeable vapour pressures at these temperatures, as seen in table 3.2. As the evaporated species were transported to regions in the decoated with higher oxygen contents, the species would oxidise and deposit on nearby surfaces. Such deposition patterns are visible in figure **??**. The sample material is visibly oxidised as observed in figure **??**. Only magnesium and aluminium would oxidise directly from the solid metal, as they form much stabler oxides than lead, as shown bu figure3.4. It is therefore likely that lead evaporated and then oxidised during decoating at $550\,^{\circ}\mathrm{C}$ to a much larger degree than at $300\,^{\circ}\mathrm{C}$. Magnesium is likely to have both evaporated and oxidised directly from the metal when decoated at $550\,^{\circ}\mathrm{C}$.

If the lead evaporated from the solid aluminium during decoating at 550 °C it would certainly evaporate during remelting of Cans 300 at 800 °C? Only if given the chance. The evaporation process is limited by the transport of lead from the bulk phase to the metal/gas boundary. This bulk phase is much thicker in the melt than during decoating. Evaporation of lead during remelting would therefore require a lot of stirring. The melt was occasionally stirred during sample addition and skimming, however the melt was generally laid to rest. Some magnetic stirring also must have occured due to the magnetic field induced by the drop coil, but stirring was largely absent. Furthermore, most of the time both the Cans 550 and the Cans 300 sample material was covered by an oxide layer during remelting, which would effectively halt evaporation of lead.

It is therefore plausible that lead evaporated during decoating of Cans 550, but not during remelting of Cans 300 due to being limited by mass transfer and an alumina layer.

The first oxidation of magnesium on Cans 550 occured during decoating. It's implausible that none of the oxide visible on Cans 550 contains magnesium. This oxide would act as hot-spots, significantly reducing breakaway time of magnesium oxidation as per figure3.8. Break-away is described to result in all of the magnesium in the sample oxidising.

The breakaway time on Cans 300 however was significantly increased by the presence of CO_2 over the crucible during remelting, and there wasn't necessarily any magnesium oxide present on the samples. In such conditions the break away time of magnesium can be several hours [17], much longer than the resident time of the melt.

To summarise: Lead evaporated during decoating at 550 °C. Lead may also have evaporated during remelting, but to a smaller degree evident by the difference in lead concentrations. Magnesium oxidised during decoating at 550 °C and any remaining magnesium thereafter oxidised during breakaway oxidation which occured rapidly due to the presence of magnesium oxide.

Notably we were not able to show a statistical significant difference in the contents of tin, but tin and lead is expected to behave similarly during decoating and remelting, and the considerations regarding lead below also count for tin, including the high vapour pressure during remelting.

In future experiments it would be important to develop and use a highly autonomous experimental procedure to limit variance as much as possible. The furnace should be powered with identical protocols, and the dross should be removed by the same person with the same method in each experiments. Furthermore the experiments should be performed in a randomised order.

6.7 The recyclability of Cans

The material flow of the aluminium waste through our recycling process, given decoating at 550 °C is visualised in figure 6.13. The mass loss from decoating and remelting are well documented, whilst mass loss from ash removal and sizing are rough estimates. The process yield of the total recycling process was30 %. The process yield was 57 % if mass lost during decoating isn't included. This is much lower than what an industrial process, but an industrial process would utilise salt flux, as described in section?? whilst remelting, which significantly improve the metal yield in the remelting step, and thus the yield of whole recycling process. The results from this project shows that a sample material like Cans is recyclable with good yield, an inclusion content on par with filtered primary aluminium, and acceptable alloy quality. The yield and alloy quality would be further improved in an industrial handling through proper decoating equipment and, possible remelting with salt flux and not consistently handling the material with iron equipment.

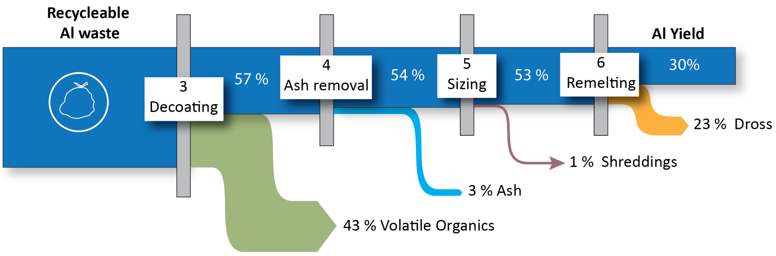


Figure 6.13: The material flow of the *Cans* sample material during this project.

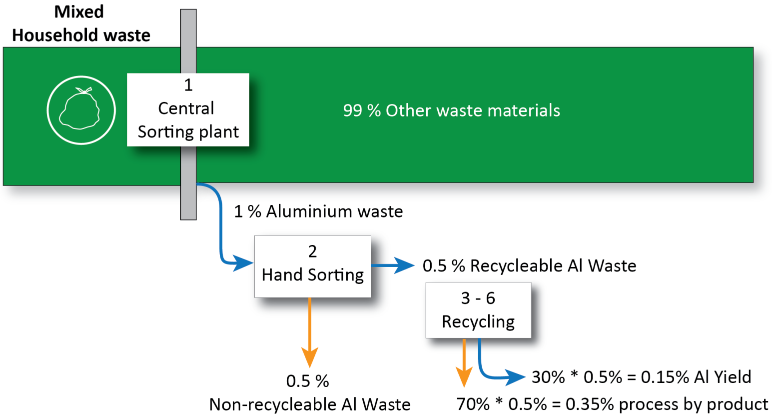


Figure 6.14: A typical flow of aluminium waste recycled from mixed household waste, with the same recycling yield as achieved in the project.

The challenge occurs with the availability of the Cans material. As seen in figure 6.14 the Cans material makes up approximately $0.5\,\%$ of mixed household waste produced in Norway today. The labour of extracting aluminium is huge, but most of this labour will be done at the central sorting plant for mixed household waste. To summarise: Cans is recycleable, and should be recycled.

Chapter 7

Conclusions

The reader is reminded that *Cans 550* = Aluminium waste from mixed household waste decoated at 550 °C and *Cans 300* = Aluminium waste from mixed household waste decoated at 300 °C.

7.1 Decoating

1. Decoating of Cans at 300 °C resulted in 42 % mass loss. Cans 300 was not completely decoated, and covered by black char after decoating. However Cans 300 did not oxidise during decoating.
2. Decoating of Cans at 550 °C resulted in 43 % mass loss. Cans 550 was completely decoated, but the samples oxidised to some degree during decoating.
3. Decoating experiments should be performed in specialised equipment.
4. Good decoating is characterised by a high mass loss of organic contents, and low oxidation of metal, which results to a large mass loss.

7.2 Yield

1. Cans 300 and Cans 550 were remelted with 54 % and 65 % yield respectively. The difference between the two was shown to be statistically significant. Carbonaceous gases promoted dross formation during remelting of Cans 300, whilst char inhibited coalescence of metal droplets further promoting dross formation.
2. Cans 300 and Cans 550 were remelted with 46 % and 35 % relative dross formation compared to sample material added. The same reasoning above explaining the

difference in yield also explains why Cans 550 can be remelted with significantly lower dross formation than similar waste Cans 300.

3. The duration, which experiment the addition was in, and relative addition size and compared to melt in the crucible and melt temperature did not significantly affect remelting yield, affirming the validity of the yield measurements.

7.3 Quality

1. The alloy contents of magnesium in remelted Cans 300 and Cans 550 were $0.36\,\%$ and $0.10\,\%$ respectively. The difference between the two was shown to be statistically significant. Magnesium oxidised during decoating due to the sample material experiencing temperatures above $600\,°\mathrm{C}$. This oxide spurred further oxidation of during remelting. Magnesium didn't oxidise during decoating of Cans 300, and the release of carbonaceous gases during remelting of Cans 300 inhibited magnesium oxidation.
2. The alloy contents of lead in remelted Cans 300 and Cans 550 were $11\,\mathrm{ppm}$ and $5\,\mathrm{ppm}$ respectively. The difference between the two was shown to be statistically significant. Lead evaporated during decoating of Cans 550 due to having noticeable vapour pressures at $600\,°\mathrm{C}$.
3. The alloy produced by remelting Cans 300 and Cans 550 contained the major alloying elements, in increasing order: copper, zinc, silicon, magnesium, manganese, iron. The contents were between $0.1\,\%$ and $0.6\,\%$. The alloying elements came with the different alloys used in aluminium packaging. The iron content was probably increased because of handling with iron tools during remelting and decoating in iron troughs.
4. The contents of silicon, iron and zinc were between $0.05\,\%$ and $0.1\,\%$ for remelted pure aluminium. This indicates that silicon, zinc and iron was added to the melt during procedures. As melts of pure aluminium weren't skimmed, this effect is expected to be higher for Cans 300 and Cans 550.
5. The porosity of remelted pure aluminium, Cans 300 (with high and low inclusion contents) and Cans 550 increases in the order: Pure aluminium < Cans 550 < Cans 300 (low inclusion content) < Cans 300 (high inclusion content). Metal quality increases with decreasing porosity.
6. The inclusion count of remelted pure aluminium, Cans 550 and Cans 300 increases in the order of: Pure aluminium < Cans 550 < Cans 300. The mean particle area of inclusions in each remelted material was below $0.025\,\mathrm{mm}^2/\mathrm{kg}$, which is categorised as a very low inclusion content. Metal quality increases with decreasing inclusion count.
7. Measurements were made with cold PoDFA, which differs from hot PoDFA that is the basis for the categorisation, but the metal particle area of Cans 300 and Cans 550

would still be comparable to filtrated primary aluminium.

7.4 Methodology

The sample material Cans has been shown to be successfully recycled with decent yield and metal quality. The success of the recycling of the Cans sample material, and the ability to statistically prove it being a success shows that the methodology developed in the project is fitting for its purpose: To assess the recyclability of the aluminium in a material stream containing large amounts of aluminium.

Chapter 8

Further Work

As described in section 1 this work has been pioneering in essence. We have developed a method that gives statistically measurements of yield and different measures of metal quality with relatively few experiments. Several improvements to the methodology are discussed in section **??**. The list of future work is divided into three sections describing which methods, materials, parameters to be varied, and analysis techniques utilised, should be considered for future work.

8.1 Methods

Future experiments should separate the raw sample materials into set batches. Experiments of large scale should use a factorial design on the second level with three parameters, whilst smaller projects should use a reduced scope with only one or two varied parameters. The sample materials should then be stored, transported, pre-treated, remelted and analysed by batch. This represents a logistical challenge, but would allow measurements of how both planned and unplanned events affect the sample material.

8.2 Materials

The methodology should be used on the Foils, EnvalFoils and UBC materials described in section **??**. Other alternatives are swarf (metal scrap from machnining), with and without oil, lacquered and unlacquered profiles used for construction, and heat exchangers, but the method can be utilised for any aluminium waste or scrap.

8.3 Analysis

In future work similar analysis methods of remelted metals alloy composition, porosity and inclusions contentshould be utilised. In addition the following analysis can be performed, based on what one wants to measure:

- The metal in the sample material should be chemically analysed before and after decoating as well as after remelting (or extrusion).
- Samples for chemical testing should be extracted per addition.
- A technique for hydrogen measurement, like Alscan/ALSPEK should be
- The time and temperature the melt should be tracked with software.
- Ashes produced from decoating and de-ashing should be chemically analysed by batch.
- Inclusion count should be measured by hot PoDFA.
- Drosses from remelting should be analysed for aluminium content and oxide chemistry.
- The density of the sample material after each step of pretreatment and remelting should be recorded.
- Analysis of sample oxide layer using scanning electron microscope.

In addition one could measure :

- The surface area of the sample material;
- the density of the sample material after each process step;
- the aluminium content in sample materials through dissolution in acid and evolution of H_2.

Appendix

8.4 Other yield measurements.

The yield and slag (and burn-off for Cans 300) of each addition in each experiment is shown for Cans 550 in figure **??** and Cans 300 in figure 8.1.

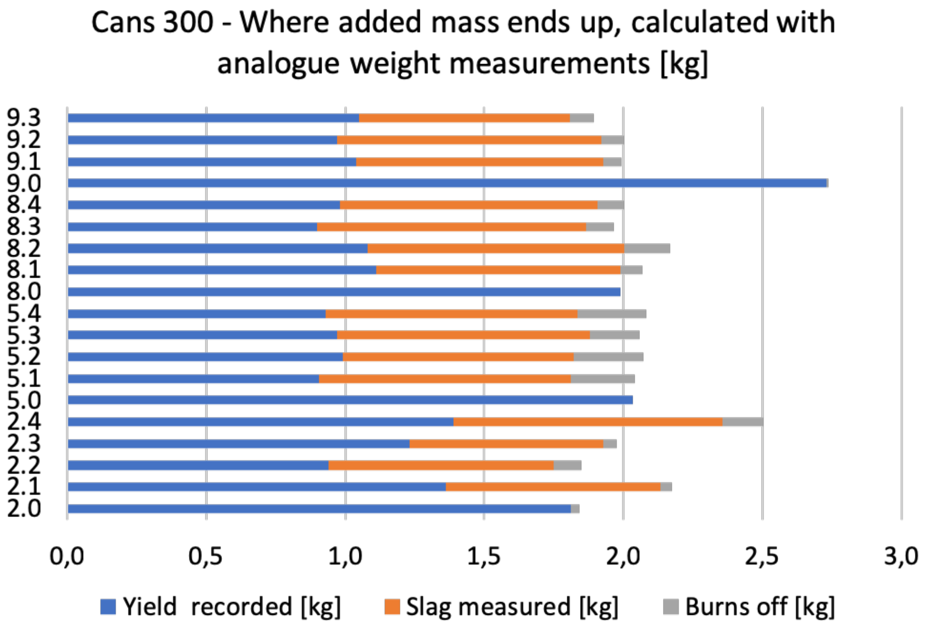


Figure 8.1

Figures 8.2 and 8.3 show the same the same figure, with percentage mass.

The yield and relative addition, with relative addition being the weight added to the crucible/weight in the crucible, is shown for each addition in each experiment in Cans 550 in figure **??** and Cans 300 in figure **??**.

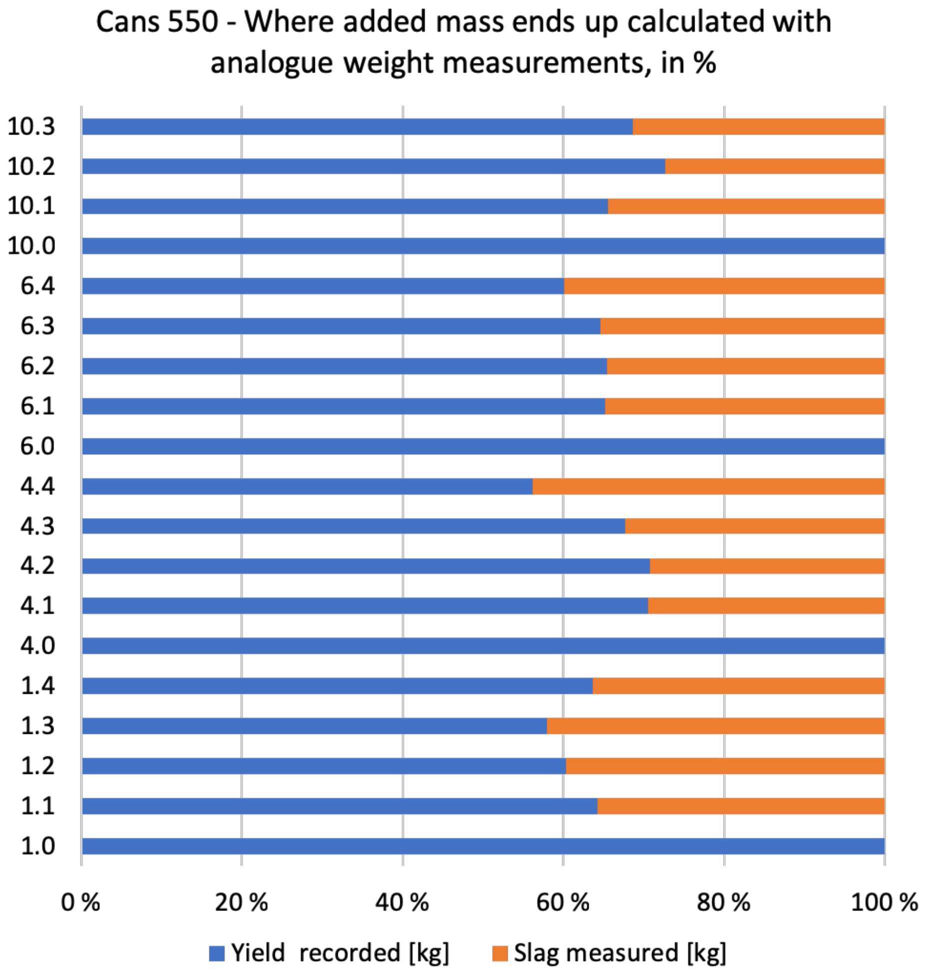

Cans 550 - Where added mass ends up calculated with analogue weight measurements, in %
10.3
10.2
10.1
10.0
6.4
6.3
6.2
6.1
6.0
4.4
4.3
4.2
4.1
4.0
1.4
1.3
1.2
1.1
1.0
0 %
20 %
40 %
60 %
80 %
100 %
Yield recorded [kg]
Slag measured [kg]

Figure 8.2

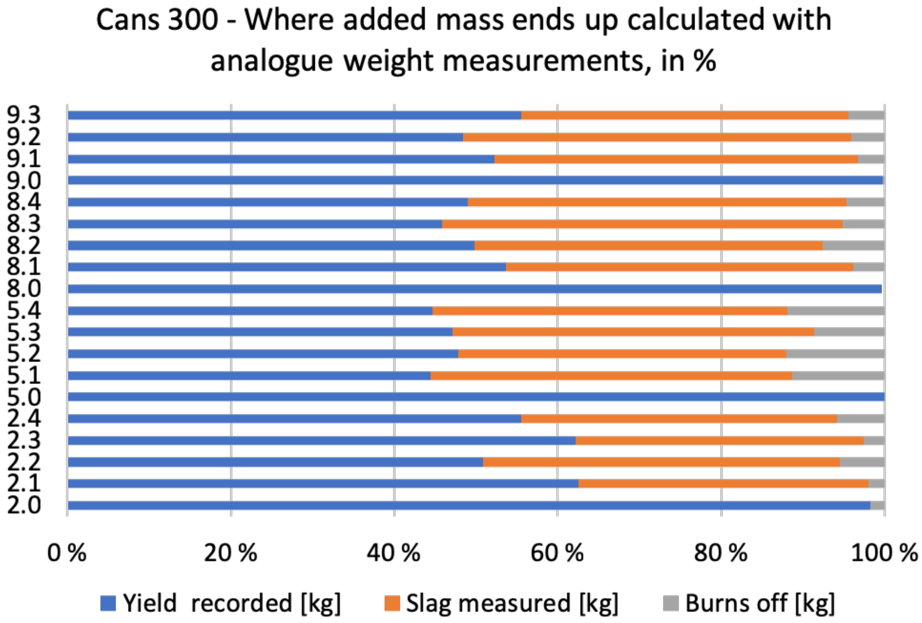


Figure 8.3

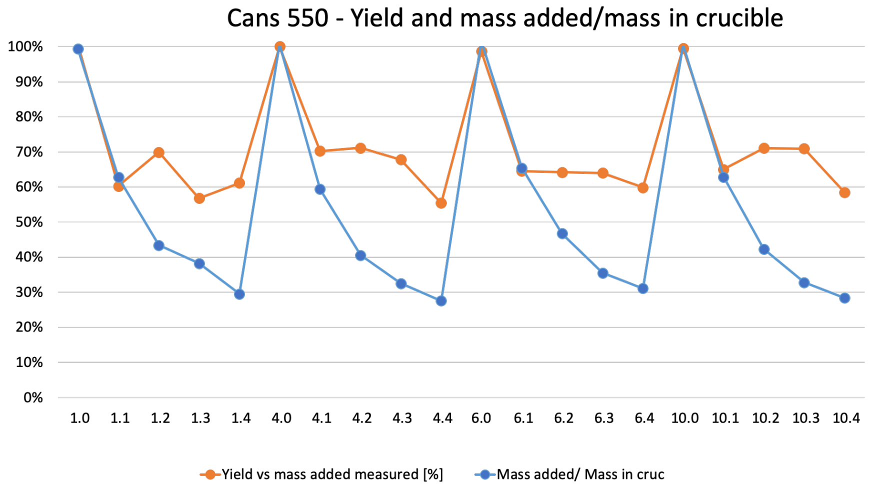


Figure 8.4

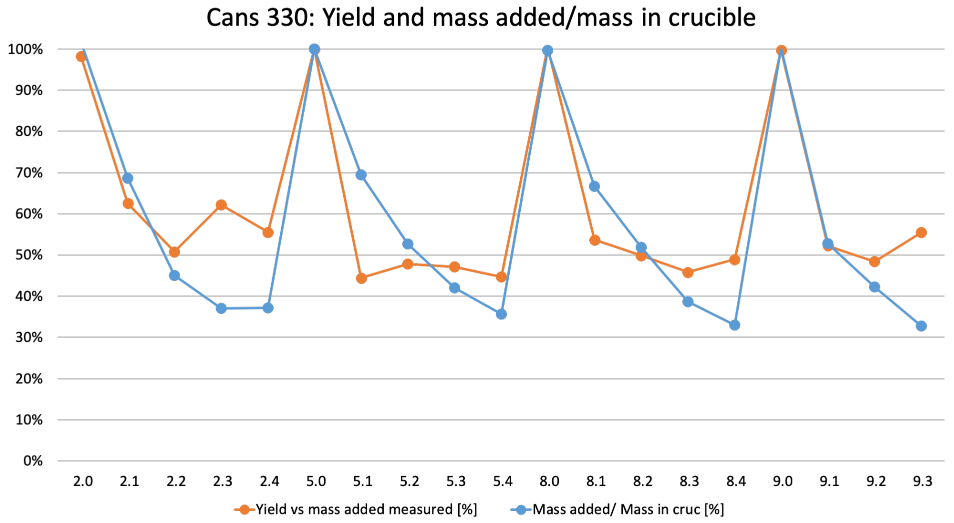

Cans 330: Yield and mass added/mass in crucible
100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
2.0
2.1
2.2
2.3
2.4
5.0
5.1
5.2
5.3
5.4
8.0
8.1
8.2
8.3
8.4
9.0
9.1
9.2
9.3
Yield vs mass added measured [%]
Mass added/ Mass in cruc [%]

Figure 8.5

8.5 other weight measurements - Metal added and slag removed

The amount of metal added to the crucible per addition, measured through both scale A and scale B, for each experiment is shown for Cans 550 in figure 8.6 and Cans 300 in figure 8.7. The amount of metal remaining in the crucible per addition after skimming, measured through both scale A and scale B, for each experiment, is shown for Cans 550 in figure 8.8 and Cans 300 in figure **??**. The amount of dross skimmed from the crucible per addition, measured through both scale A and scale B, for each experiment is shown for Cans 550 in figure **??** and Cans 300 in figure 8.11.

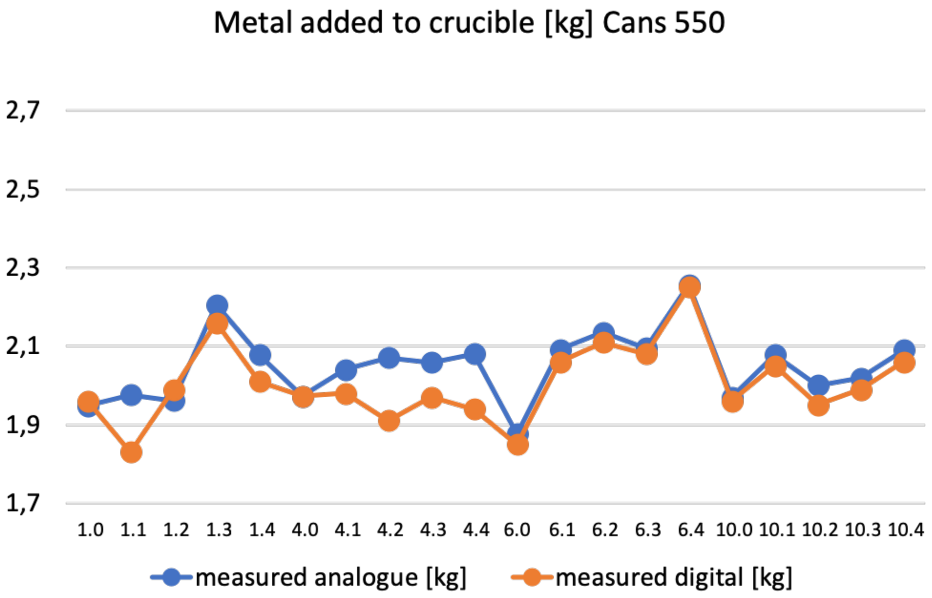


Figure 8.6

8.5.1 Time and duration of experiments

The time of addition wasn't always written down during experiments, and thus an accurate set of times of additions aren't available. However for all additions at least 2 points are available, and by through linearization we can estimate the time of each addition, and therefrom the durations.

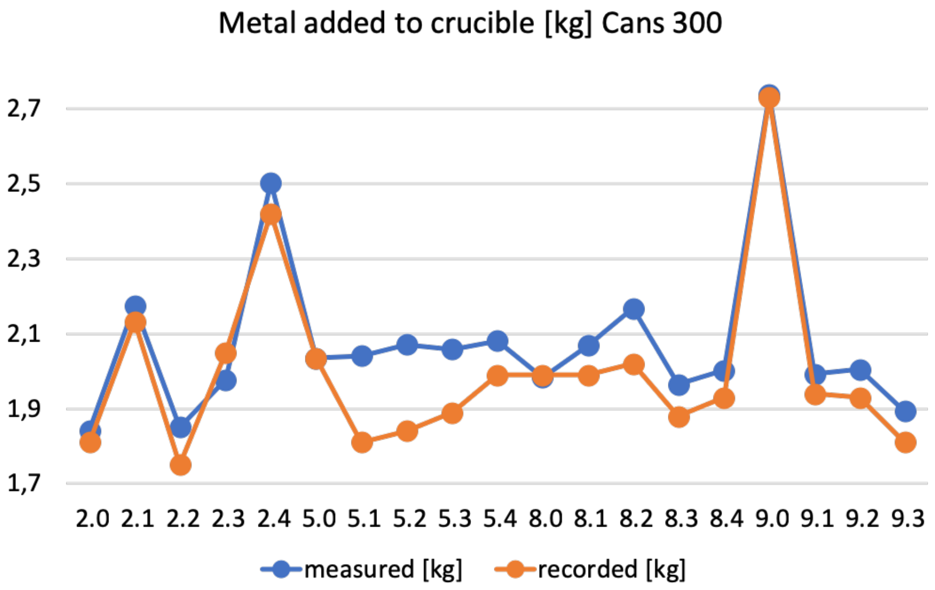


Figure 8.7: metal added 300

8.6 Testing of statistical hypotheses

8.7 Images from RPT

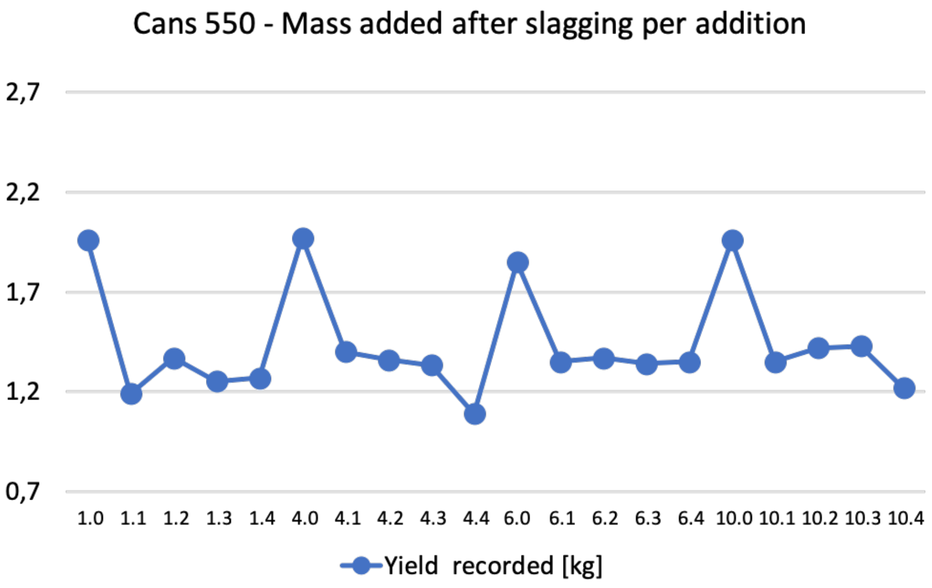


Figure 8.8

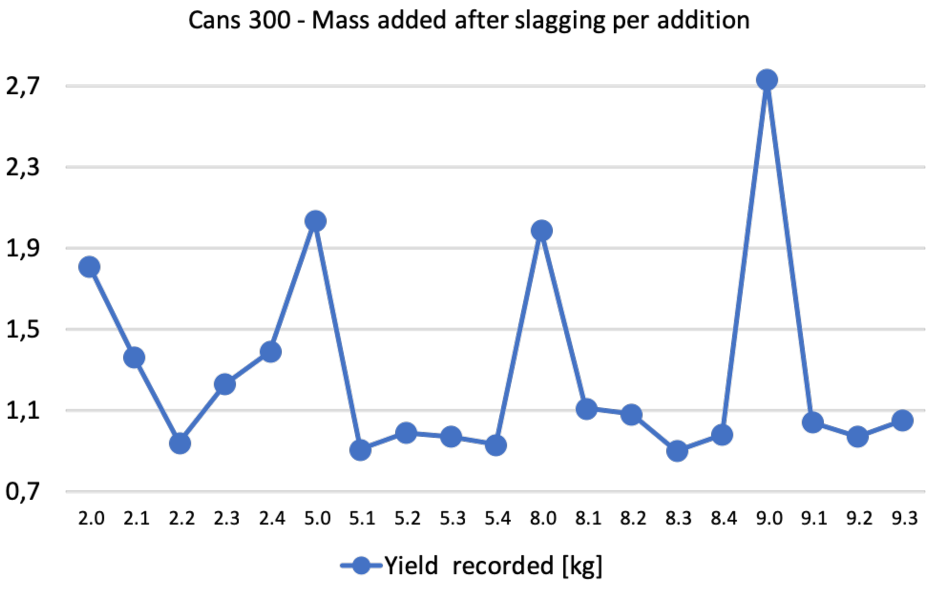


Figure 8.9

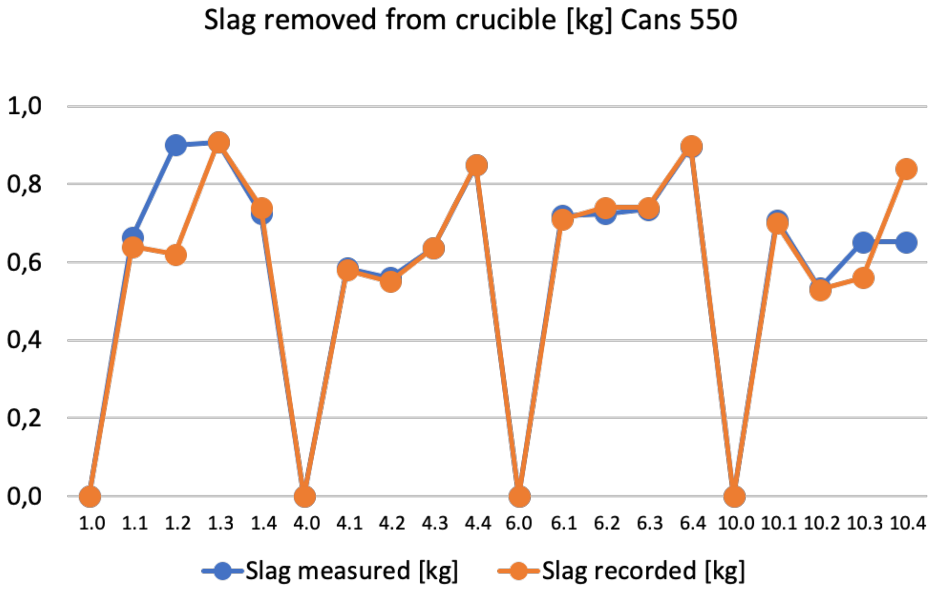


Figure 8.10

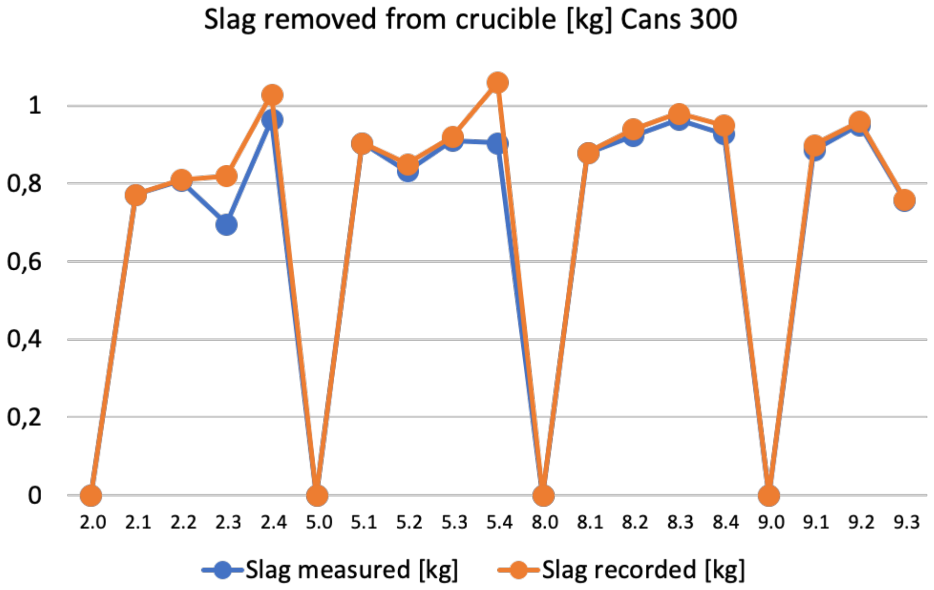


Figure 8.11: Dross skimmed per addition per experiment Cans 300

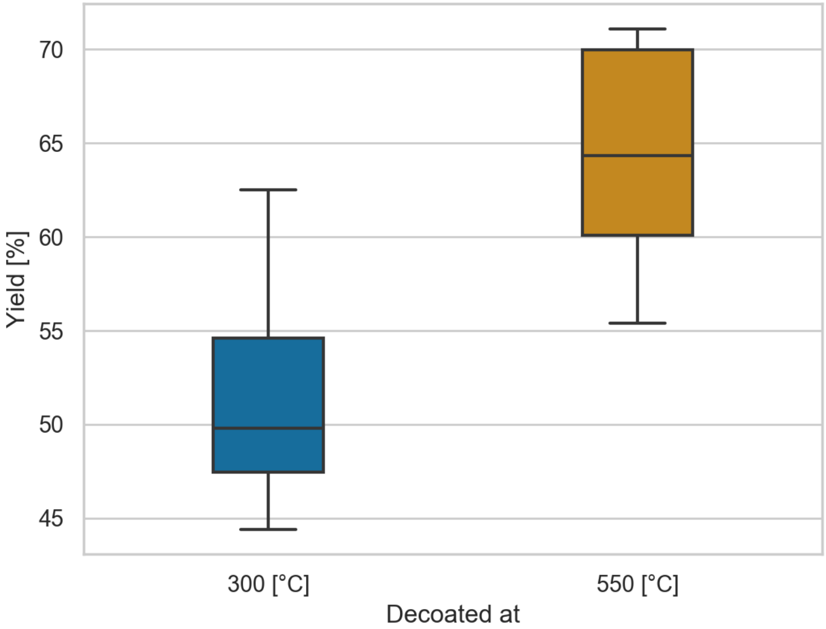


Figure 8.12: The yields of remelting of Cans 550 and Cans 300. The colored rectangles represent 95 % confidence intervals.

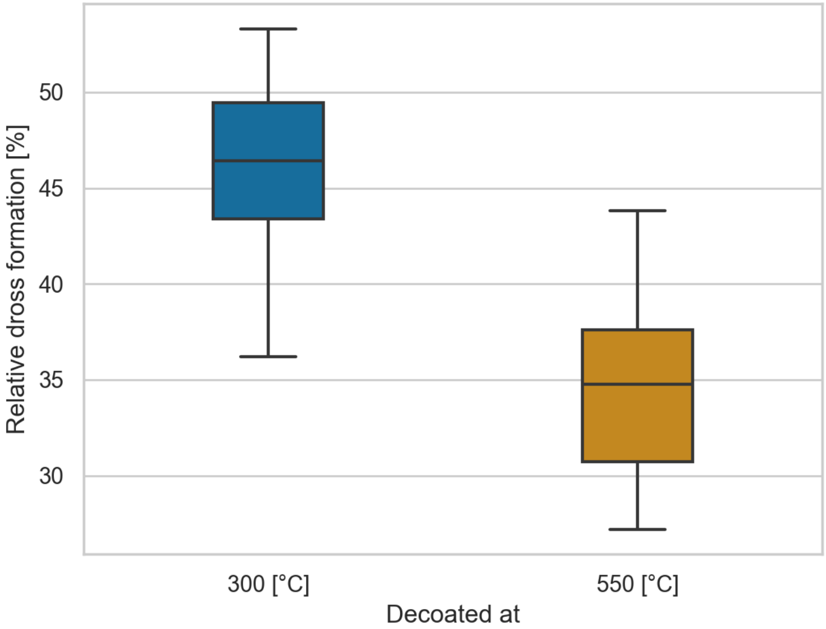


Figure 8.13: The relative dross formation from remelting of Cans 550 and Cans 300. The colored rectangles represent $95\,\%$ confidence intervals.

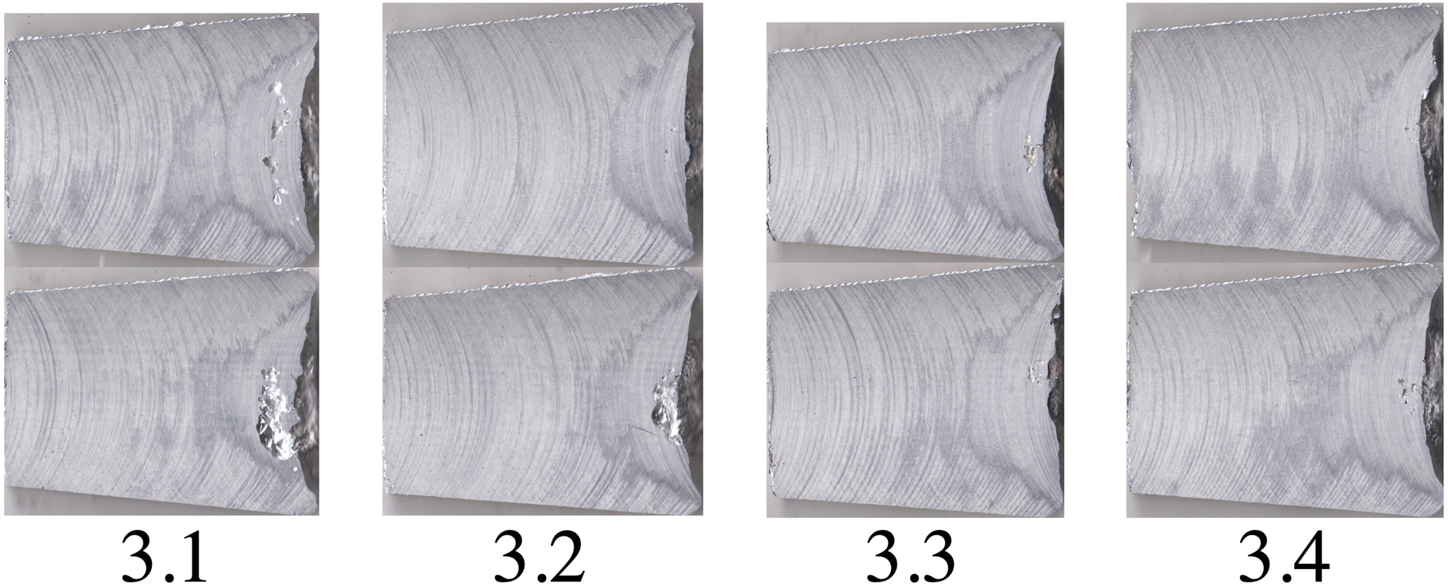


Figure 8.14: Polished RPT samples from experiment 3 - Pure Al.

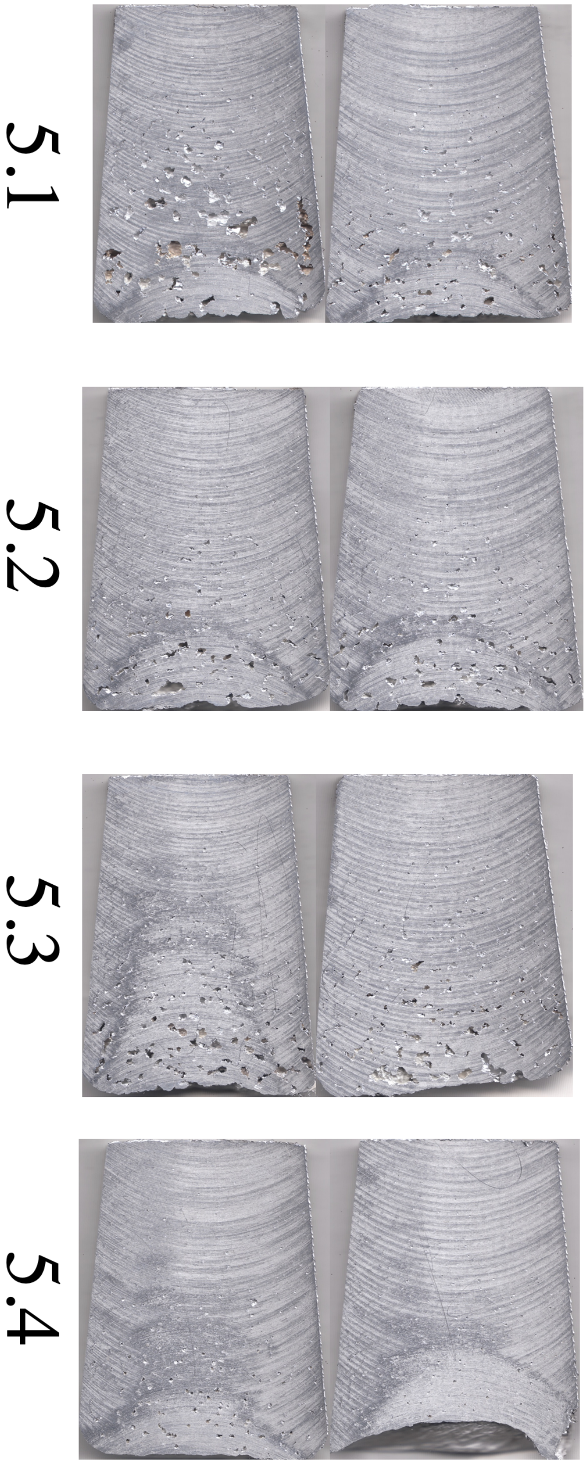


Figure 8.15: Polished RPT samples from experiment 5 - Pure Al.

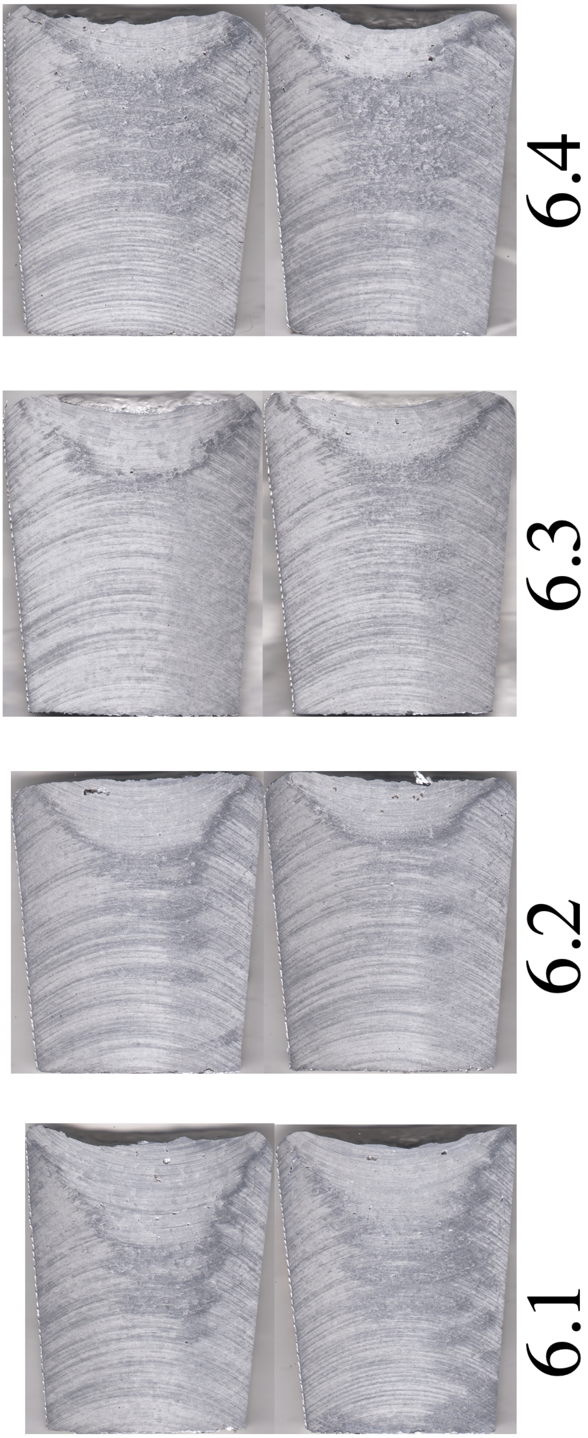


Figure 8.16: Polished RPT samples from experiment 6 - Pure Al.

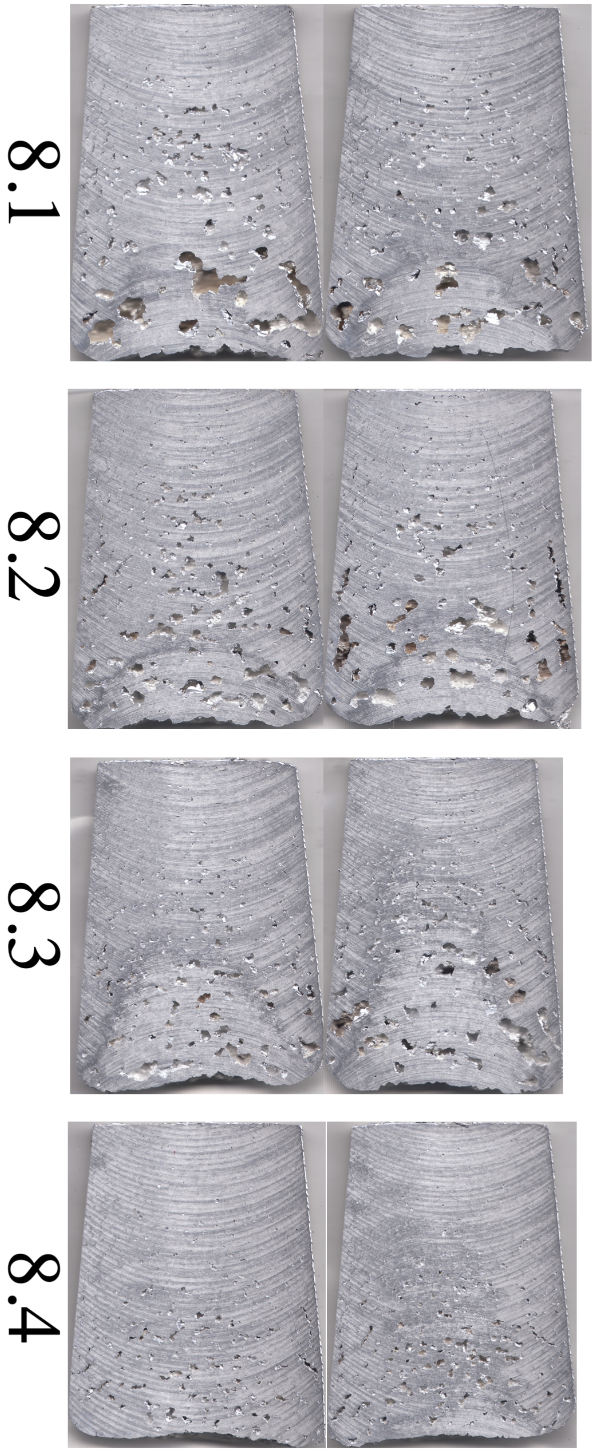


Figure 8.17: Polished RPT samples from experiment 8 - Pure Al.

8.8 Statistical Reasoning

8.8.1 Hypothesis 1

The yields of remelting Cans 300 and Cans 550 (illustrated in figure 8.12 were hypothesised to be equal in null hypothesis 1 (In introduction). This hypothesis was tested using Minitab 2018.

With a $95\,\%$ confidence interval the difference between the yield of Cans 300 and Cans 550 was shown to be between $7.97\,\%$ and $15.13\,\%$, the mean difference being $11.55\,\%$. Since this interval doesn't include zero the test necessitates the rejection of the null hypothesis above, instead accepting the alternative hypothesis: that the two yields differed. The P-value of the test was $3.6 * 10^{-7}$, making it valid for confidence intervals below 99.9999 percent.

Summary of testing hypothesis 1 on yield of Cans 300 and Cans 550: Significantly better yield, shown with a P-value of $3.6 * 10^{-7}$, and difference of $11.55\,\%$ on average, was obtained when remelting Cans 550 compared to Cans 300. On average Cans 300 was remelted with $53.78\,\%$ yield and Cans 550 was remelted with $35.33\,\%$ relative dross formation.

8.8.2 Hypothesis 2

The relative dross formation, meaning the fraction of the weight of the sample material which was skimmed out and classified as dross, of Cans 300 and Cans 550 (illustrated in **??** were hypothesised to be equal in null hypothesis 2. The hypothesis was tested using Minitap 2018.

With a $95\,\%$ confidence interval the difference between the relative dross formations of Cans 300 and Cans 550 was shown to be between $7.97\,\%$ and $15.13\,\%$, the mean difference being $11.55\,\%$. Since this interval doesn't include zero the test necessitates the rejection of the null hypothesis above, instead accepting the alternative hypothesis: that the two yields differ. The P-value of the test was $3.6 * 10^{-7}$, making the it valid for confidence intervals below 99.9999 percent.

Summary of testing of hypothesis 2 on relative dross formation of Cans 300 and Cans 550: Significantly lower relative dross formation, shown with a P-value of $3.6 * 10^{-7}$, a difference of $11.55\,\%$ on average, was obtained when remelting Cans 550 compared to Cans 300. On average Cans 300 was remelted with $46.22\,\%$ relative dross formation and Cans 550 was remelted with $34.67\,\%$ relative dross formation.

8.8.3 Hypothesis 3 and 4

The concentration of magnesium in remelted Cans 300 and Cans 550 (illustrated in figure 6.12 were hypothesised to be equal in null hypothesis 3 (In introduction). This hypothesis was tested using Minitab 2018.

With a 95 % confidence interval the difference between the concentration of Magnesium in Cans 300 and Cans 550 was shown to be between 0.117 % and 0.392 %, the mean difference being 0.255 %. Since this interval doesn't include zero the test necessitates the rejection of the null hypothesis above, instead accepting the alternative hypothesis: the two concentrations differ differed. The P-value of the test was 0.01, and making it valid for confidence intervals below 99 percent.

The concentration of lead in remelted Cans 300 and Cans 550 (illustrated in figure 6.12 were hypothesised to be equal in null hypothesis 4 (In introduction). This hypothesis was tested using Minitab 2018.

With a 95 % confidence interval the difference between the concentration of Lead in Cans 300 and Cans 550 was shown to be between 0.0001 % and 0.010 %, the mean difference being 0.005 %. Since this interval doesn't include zero the test necessitates the rejection of the null hypothesis above, instead accepting the alternative hypothesis: the two concentrations differ differed. The P-value of the test was 0.0254, and making it valid for confidence intervals below 97.6 percent.

Summary of testing hypotheses 3 and 4 on magnesium and lead content in remelted Cans 300 and Cans 550: Significantly higher concentrations, with P-values of 0.01 and 0.025 for and differences of 0.255 % and 0.005 % on average for magnesium and lead respectively, was found in remelted Cans 300 compared to remelted Cans 550. Average contents of magnesium and lead were 0.358 % and 0.0011 % respectively in Cans 300 and 0.104 % and 0.0005 % in Cans 550.

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