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OPTIMIZATION OF RECYCLING PROCESS OF DIE CAST ALUMINUM A380 MACHINING CHIPS

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by

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DECLARATION OF CO-AUTHORSHIP/PREVIOUS PUBLICATION

I. Co- Authorship Declaration

I hereby declare that this thesis incorporate material that is result of joint research, as follows: The machining chips A380 and the chemical analysis included in Chapter 2 were provided by Chi Liu, this thesis also incorporates the outcome of a joint research undertaken in collaboration with Xuezhi Zhang and Li Fang under the supervision of Henry Hu. The collaboration is the implementation of experiments that covered in Chapter 2 to Chapter 5 of the thesis. In all cases, the key ideas, primary contributions, experimental designs, data analysis and interpretation, were performed by the author.

I am aware of the University of Windsor Senate Policy on Authorship and I certify that I have properly acknowledged the contribution of other researchers to my thesis, and have obtained written permission form my co-authors to include the above material in my thesis

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II. Declaration of Previous Publication

This thesis includes 2 original papers that have been previously published/submitted for publication in peer reviewed conference/journals proceedings, as follows:

Thesis Chapter	Publication title/full citation	Publication status
Chapter II	**Bojun Xiong**, Xuezhi Zhang, Fang Li, Henry Hu, Chi Liu, Recycling Of Aluminum A380 Machining Chips. Light Metals 2015: Cast Shop For Aluminum Production, 1011-1015, 2015, March 15-19, 2015, Orlando, Florida, USA, TMS, Wiley, TMS 2015 144th Annual meeting and Exhibition.	Published
Chapter III	**Bojun Xiong**, Xuezhi Zhang, Fang Li, Henry Hu, Chi Liu, Process Optimization For Recycling Of Machining Chips Of Die Cast Aluminum Alloy A380. CIRP Journal Of Manufacturing Science And Technology. March, 31, 2015.	Submitted

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ABSTRACT

Aluminum alloys have been increasingly utilized in automotive industry in recent years. Along with components manufactured, considerable amount of aluminum waste in the form of scrap, dross, and machining chips is produced as by-products. In this study, aluminum chips were collected from computer numerical control (CNC) machines. The cleaning method and refining parameters were investigated. Recovery rate reached as high as 90.3% with a remarkable chemical component and microstructure compared to die-cast aluminum alloy A380 referred. Then, to optimize the recycling process, Design of Experiment (DOE) was employed. Flux types, chips/flux ratios, holding times and holding temperatures were selected as four factors and for each factor, three corresponding levels were also chosen to create Taguchi orthogonal array. Signal-to-noise (S/N) ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data. Optimum combinations of factors were analyzed and concluded for individual response and multi-response.

DEDICATION

I would like to express my deepest thanks to my parents for their unconditional love and encouragement.

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1. CHAPTER I

INTRODUCTION

Since 1980's, aluminum alloys as a light weight material have been increasingly used in the automotive industry. Worldwide average aluminum content was 7.8% of the average worldwide light vehicles curb weight of 3,183 pounds in 2009. North America (NA) has the highest aluminum penetration at 8.6% of N.A. curb weight in the world. The usage of aluminum in NA automobiles has gone from 45kg (101 lbs) in the 1970s to 150 kg (326 lbs) in 2009, and will top 170 kg (376 lbs) per vehicle by 2020. Among the 150 kg aluminum usage in each vehicle, almost 35% of automotive aluminum components were manufactured by conventional high pressure die-casting (C-HPDC) processes [1]. When C-HPDC components are manufactured, considerable amount of aluminum waste in the form of scrap, dross, and machining chips is produced as byproducts. The casting scrap is easily returned to melting, whereby most of the metal is recovered and re-utilized in production processes [2-4]. The recovery of aluminum from dross can be achieved at a recovery rate of over 80% by mixing the dross with certain types of flux [5]. During the recycling of dross and chips, however, a lot of metal is lost as a result of oxidation, and the costs of labor and energy as well as the expenditure on environmental protection increase the general cost of process.

1.1. Motivation

For most aluminum foundries, reusing aluminum chips as raw material for melting stocks is perhaps the best option as waste management policy in what concerns to economical and technical aspects. In-house recycling of aluminum machining chips presents some significant benefits over other recycling solutions, such as reduction on buying costs of raw material, elimination of chips transport costs; simplified waste management system; high cost/benefit ratio. Aluminum chips is a low density product (0.25 kg/dm^3) which makes them inconvenient for handling and transportation, and their surface area is relatively large to the volume, and their surfaces are usually covered with oxides, oil emulsion and machining fluid, which is not good for recycling by re-melting approach. Also, aluminum and aluminum-alloy chips are fouled chiefly with the coolants and lubricants used in machining, usually with oil emulsion. Directing melting such a product without suitable previous preparation would lead to several problems of different nature: (1) Economical aspects: very low metal recovery rate and high energy consumption; (2) Environmental aspects: high smoke and gases generation; (3) Quality aspects: low quality of the final product (non-metallic inclusions, gas porosities, poor mechanical properties) [6-8]

1.2. Objective and Tasks

The objective of this project is to develop an effective recycling process for aluminum A380 machining chips with good metal quality and yield. The major tasks of the present study are

- To perform a literature review on the applications and potentials of various recycling processes for aluminum chips, which should include not only the conventional method, direct melting, currently used in the industry but also the emerging technologies such as the direct conversion method, solid state transformation;
- To select a chemical solution to remove oil emulsion present on the surface of aluminum machining chips, or optimized method for chips cleaning;
- To select a suitable flux for melting operation;
- To recover chips with the selected flux in a certain amount via refining;
- To optimize the recycling process via the Design of Experiments;
- To determine the recovery rate based on the measurement of the recovered metal and the input chips weight;
- To evaluate mechanical properties of the recycled alloy including ultimate tensile strength (UTS), yield strength (YS), Elongation (E_f), and porosity content and corrosion resistance for quality assessment.

1.3. Literature Survey

1.3.1. Introduction to aluminum recycling

Aluminum is becoming popular in all kinds of fields [9] and is suitable for use in a wide variety of products for the consumer and capital goods markets. The largest markets are transportation, packaging, construction, electrical, consumer durables, machinery and equipment. Among them, transportation sector, which is one of the largest single markets for aluminum worldwide, includes the manufacture of automotive, buses, trailers, ships, railroad and subway cars, as well as aerospace applications and mobile homes. Aluminum and its alloys have outstanding corrosion resistance with good strength and low density as mentioned. For these advantages, aluminum saves more energy when used in mobile applications, and consequently gives a significant reduction in the greenhouse gas emissions over lifetime. Besides, its lightweight and recyclability have provided the impetus for the increased use of aluminum to help meet new and more stringent corporate average fuel efficiency standards.

However, the production of primary aluminum is an energy costly process [10], involving bauxite mining, purification of alumina by a Bayer process, and a molten salt electrolyte based on cryolite. With the climate change being of concern, the secondary aluminum stream is becoming an even more important component of aluminum production and is attractive because of its economic and environmental benefits. Increasing demand for aluminum-based products and further globalization of the aluminum industry have contributed significantly to the higher consumption of aluminum scrap for re-production of aluminum alloys. At the same time, tons of wastes are created during daily aluminum production. Those wastes, including slag, chips and scraps, covered with coolant are difficult to be recycled. With more and more attention drawn to the recycling industry, advanced techniques need to be developed to improve recycling process.

There are several advantages to society when aluminum is produced by recycling rather than by primary products from bauxite ores. Firstly, it is believed that the re-melting of recycled aluminum saves almost 95% of the energy required manufacturing pure aluminum from bauxite ore. Secondly, European estimates suggest that the mass of solid waste generated per ton of recycled aluminum is 95% lower than that for primary metal. Thirdly, primary aluminum productions generate both hazardous and non-hazardous emissions. Currently, a large amount of the aluminum going into products is coming from recycled products [1]. In the work of Shinzato et al [11], in addition to the recovery of metallic aluminum, salt flux and magnesium chloride, the process generates a waste, known as non-metallic (NMP), which is usually disposed in landfills. And fine grains (less than 150 um) of the NMP can also be used as raw material in cellular concrete. The aluminum content in this fraction reacts with water during the production of the concrete while releasing hydrogen. This reaction promotes the formation of pores that reduce concrete density without affecting its strength.

1.3.2. Conventional recycling processes

High metallic aluminum content (about 80% (wt.)) dross produced by the primary aluminum industry is known as white dross. Low-quality dross produced by the secondary aluminum industry containing around 5%–20% aluminum. The first submitted methodology for aluminum dross recovery was patented in the 1970s in United States by Papafingos and Lance [12]. This patent features equipment for cooling and disaggregating aluminum dross with water in order to dissolve the salts. During the aluminum dross digestion, several undesirable and potentially toxic chemical reactions end up producing hydrogen, methane and ammonia gases.

Aluminum machining chips are often collected from dealers or directly from machine shops, and always the scraps are covered with coolant, emulsion and even coating. It is very dangerous to heat the chips without cleaning because of thermal-chemical reactions of the coolant or emulsion. Usually, there are various methods for cleaning aluminum chips. When it comes to chemical methods, ultrasonic bath using acetone solution and dried in furnace at 60^{o}C is preferred. Then, according to the study of Gronostajski et al [13], compacting chips before re-melting has many advantages. Firstly, the compacted aluminum melts much better than the chips. Secondly, the coolant or moist are more likely to be drained out, while the liquid can then either be recycled for additional revenue. Thirdly, the loose chips take much more storage space and is not easy to be stacked or contained neatly, which suggests the necessity of compacting. As shown in Fig.1-1 [14], conventional recycling process is carried out with a melting phase as a fundamental step. Large amount of metal loss occurs at the phases such as re-melting, casting and sawing. Though the conventional recycling process has the combustion of coolant or emulsion, pollution emission is much lower than the primary aluminum production process. The re-melting process costs less energy. Recycling aluminum should be the essential process in the aluminum industry.

In the past, industrial practices were dominated to a large extent by the objective of producing commercial goods as cheaply as possible according to the consumers' wishes. However, whether by market-pull or legislative action, ecological factors will in the future be an equally significant driving force for new product and manufacturing process development. Today, manufacturers have to consider ecological aspects [15] such as:

(1) Choice of ecologically sound materials.

(2) Environmentally acceptable production methods.

(3) Materials recovery and effective recycling programs.

(4) Ecologically sound waste management.

As the conventional recycling processes (CRP) has been utilized for over 50 years, much improvement was made in this industry while some inevitable issues were found and concluded such as the recovery rate, emission control, melt energy cost and the quality of recycled metal.

Firstly, in the process of melting aluminum and aluminum alloy chips, on the average about 10% of the metal is burnt and about 10% of it is lost because aluminum mixes with the slag removed from the surface on the ladle. The losses are irreversible and can reach about 35% if smelting takes place in gas- or oil-fired furnaces instead of induction furnaces. The main cause of the substantial losses of aluminum and aluminum alloy waste during conventional recycling is its low density due to which it stays rather long on the surface of the molten metal and oxidizes intensively. There are further losses during casting, in the form of various discards such as risers, shrink holes and so on, which reach about 8%. Later, during the processing of aluminum ingots, there are losses amounting to about 18%. As shown in Fig.1-2, the conventional process can recycle less than 55% of aluminum scrap. Secondly, the conventional recycling process is characterized by high energy consumption, high operating costs and a large number of operations. Additional new scrap is generated after melting due to casting, cutting and rolling or extrusion processes. At last but the most important, a strategy to increase the demand of recycled aluminum materials is to increase the quality of the recycled materials. Those three issues mentioned above are the most urgent challenges for the recycling industry and accordingly, some new methods are developed focusing on the problems.

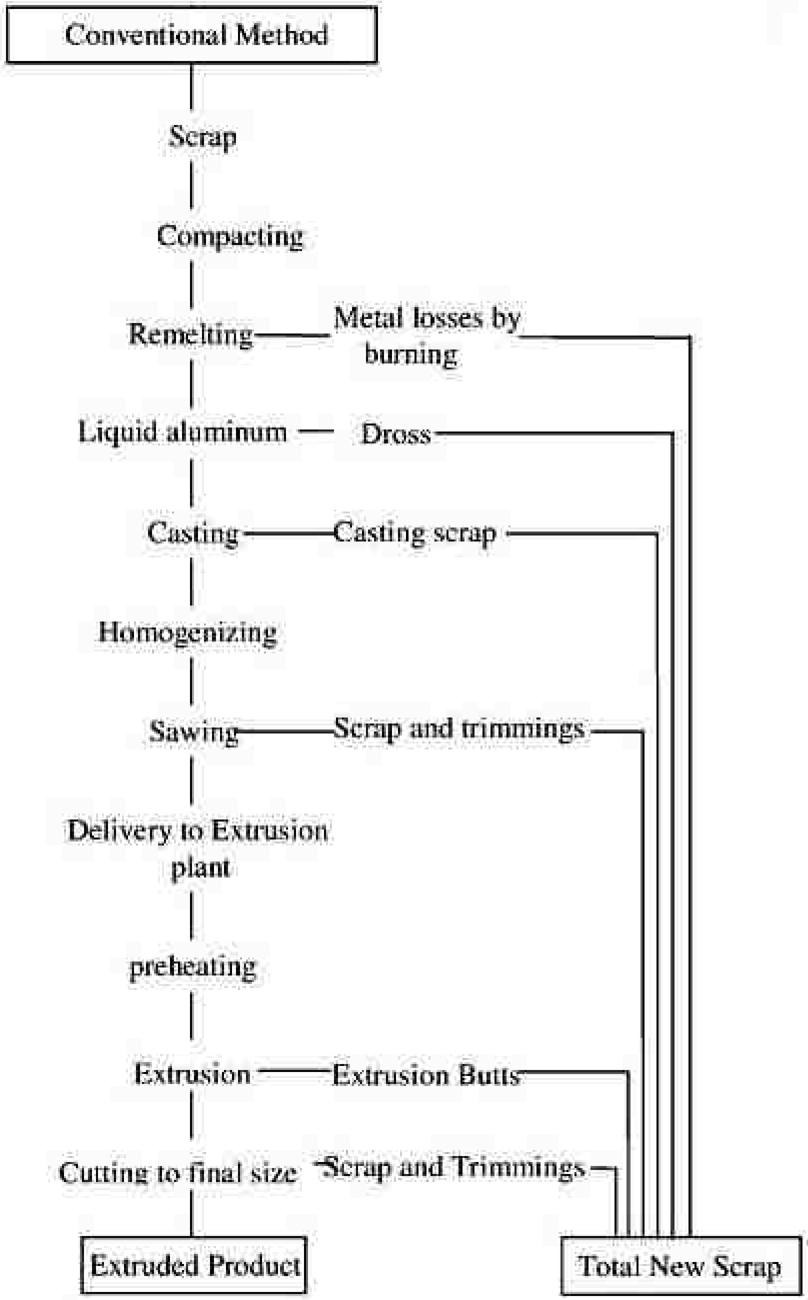

Figure 1.3-1 Flowchart of conventional recycling processes [14].

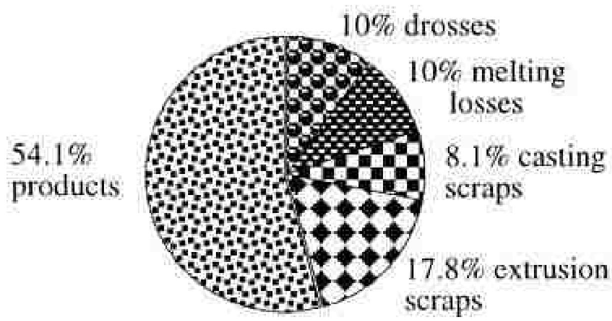

Figure 1.3-2 Metal losses during conventional recycling process [16]

During re-melting, refining, and casting process of aluminum alloys and scraps, aluminum dross, primarily oxides and nitrides of aluminum and entrapped metallic aluminum, is generated at the surface of the molten metal resulting from its uncontrolled reaction with the furnace atmosphere at elevated temperatures [14]. Recycling of aluminum dross is one of the most challenging tasks in die casting processes since it is difficult to separate the oxides from metallic aluminum even at a high temperature. In a typical recovery process, the dross is normally melted at high temperatures in a furnace. However, at elevated temperatures, free metallic aluminum in the dross is easily susceptible to oxidation and, moreover, commonly tends to ignite and burn in the presence of air to emit toxic gases. The burning of the aluminum can decrease substantially the amount of aluminum recovered [3, 17].

In of work of Hu [5] et al, dross samples of aluminum alloy A380 were collected from the reverberatory furnaces and transferring ladles at Roybi Die Casting. Wedron Flux (WF132) was selected and mixed with the Al dross with a certain weight ratio of dross to flux equal to 5:2. Melting of the dross and flux mixture was carried out in a 2.6 kw, 50/60 HZ electrical furnace with a maximum temperature of 1200 ^{0}C. The furnace temperature was set at 1000 ^{0}C when running the experiment to ensure the temperature of the inside crucible (melt) is kept around 900 ^{0}C. The melt was held at 900 ^{0}C for one hour, stirred for 15 minutes, and then the recovered aluminum alloy A380 was cast at 900 ^{0}C into a steel ingot mold to produce plates (60×80×20 mm). The chemistry of cast plates was analyzed with optical emission spectroscopy.

It was found the recovery rates of the dross from the reverberatory furnaces and transferring ladle could reach around 55% and 83% on average, respectively. The recovered alloy is free from porosity and oxides although the microstructure such as silicon phase is relatively coarse. It was observed that the recovered metal is clean, and could be used for casting production. When it comes to tensile properties, the UTS, YS and E_f , of the recovered aluminum are around 170 MPa, 120 MPa and 1% on average, which are slightly slower than those of the die cast A380. However, the tensile properties for both the recovered aluminum and the die cast A380 are at a comparable level. The coarse microstructure should be responsible for the relatively low properties of the recovered aluminum alloys.

It could be concluded that the chemistry of the recovered metallic aluminum from dross collected from both the reverberatory furnaces and transferring ladles both is compliant with the specification of aluminum alloy A380 with a satisfying tensile properties. Though this process could not achieve as high recovery rate as new recycling methods, it is much more economical and convenient for industry than purchasing new equipment.

1.3.3. New recycling methods

Puga et al [18] studied the influence of the melting furnace on the aluminum recovery rate and dross formation. The melting was performed in a 1500Hz, 50kW, 101 induction furnace using SiC crucible as lining. And another melting was performed in a 15kW electric resistance furnace equipped with a SiC crucible of the same capacity for comparison.

Their results showed that resistance furnace melting was not efficient for swarf recycling. Due to the static behavior of the molten pool inside the crucible, molten aluminum cannot break the aluminum oxide envelope that surrounds it, leading to low recovery rates (less than 60%) and high aluminum dross generation (around 30%). On the other hand, melting rate was low in resistance furnace, leading to melting times of almost 2h. However, recovery rates were higher in induction melting (around 85%) In this case, once the molten state was achieved, the interaction of current in the melt with the electromagnetic field produced a stirring motion that led to the destruction of the oxide films where liquid aluminum is entrapped, thus increasing the volume of molten aluminum that was recovered.

In the work by Gronostajski et al. [13] sintered products with predetermined properties. It has been demonstrated that such products could be manufactured from waste such as aluminum and aluminum alloy chips. The method is the conversion of the chips directly into a finished product as shown in Fig. 1-3 [14]. Chips, especially the strip chips, were comminuted by cutting them up to particles of no more than several millimeters length in a cutting mill. After cleaning and drying, chips were granulated with reinforcing phase in an atritor-type ball mill, and made them meet the sintering requirement. The mixture of the granulate product and the reinforcing phase produced in the ball mill were subjected to compacting, sintering and extrusion. The mixtures were pre-compacted by cold pressing in a device with a floating die under a constant pressure of 210±400 MPa. Hot extrusion was carried on in the temperature ranging from 500 to 550°C after cold pressing.

The most unique step was the aluminum and aluminum alloys compressed by extrusion without a melting phase. Thus, the waste was the part of the chips from which impurities could not be removed (2%) and the extrusion waste was up to 3%, and ultimately 95% aluminum or aluminum alloy were recovered, while this method saves 40% material, 26-31% energy and 16-60% labor. It was pointed out that this new technique had very low air pollution emission as compared with conventional recycling process.

Figure 1.3-3 Flow chart of direct conversion method [14]

It is studied by Cui et al [19], the aluminum chips were collected from a machine workshop and the average dimension was 1mm×0.8mm×0.3mm. The lubricant consisted of naphthenic mineral oil, fatty acid alkali-amide boric acid compound with 2-Aminothanol. The CEC processing procedures includes: 1) dried at room temperature for 24h, then thermally treated in a muffle furnace at 460°C for 30 min; 2) cleaned with acetone.

The chips were compacted at room temperature using a conventional universal testing machine. In the cold compaction process, 80g chips were charged into a cylindrical container with a diameter of 29.5mm in 2 subsequent steps and the pressure used was 400 MPa. At last, the CEC was conducted by pushing a specimen from one cylindrical chamber with a diameter, into the second chamber with the same dimensions, through a die with a smaller diameter. For the final extrusion, the opposite ram was removed.

The cyclic extrusion compression (CEC) is a kind of direct conversion method. It owns almost all the advantages of direct conversion method. Meanwhile, its procedures are simpler than common direct conversion method. Also, CEC, one of the promising semi-continuous Severe Plastic Deformation (SPD) techniques have been applied for consolidating nano particles into fully dense materials with good mechanical properties. However, solid state recycling of chips by SPD it is in infancy.

1.3.4. Summary

Compared conventional recycling process and some new kinds of recycling process, it was concluded as,

- Conventional recycling process has an average recovery rate of 55%, which is much lower than new methods.
- Conventional recycling process could reach high mechanical properties with satisfying microstructure and chemical composition. Thus, increasing recovery rate is the key point to improving conventional methods

- New recycling methods are focusing on direct conversion from chips to products, which is energy saving without melting process. The upfront capital investment for new equipment is required for the process.

1.4. Thesis Organization

This thesis combines the results of four independent manuscripts. In the first study, the chips collected directly from CNC machines were recycled with flux. The recovery rate of the recycled metal was determined based on weight measurements. To ensure the quality of the recycled aluminum, the chemistry of the recovered aluminum was analyzed. The cleanliness of the recycled metal was assessed based on microstructural analysis. The tensile properties of the recovered aluminum cast in an ingot mold were evaluated.

In the research of the second manuscript, two sets of recycling experiments were designed via Design of Experiment to optimize the recovery rate and porosity content. Flux type, chips/flux ratio, holding time and holding temperature during refining process were selected as four influencing factors, 3 levels for corresponding factors were also designed based on the results concluded in the first manuscript. S/N ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data for optimization. Maximum recovery rate was chosen as an objective and the combination of recovery rate and porosity content was also chosen as a multi-response. The results gave the optimum combinations of factors to each the objectives and the analysis of the conformation run verified the conclusion.

Studies for the third manuscript attempt to optimize the recycling process based on the recovery rate and tensile properties. In this manuscript, Flux type, chips/flux ratio, holding time and holding temperature during refining process were selected as four influencing factors with 3 corresponding levels for each factors. S/N ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data for optimization. Tensile testing was performed at room temperature on a MTS criterion Tensile Test Machine (Model 43) equipped with a data acquisition system. Recovery rate, yield strength, elongation and tensile strength were investigated as four individual responses, the rank of effectiveness for each factor and the optimum combinations were determined. Also, the multi-response objective including recovery rate, yield strength, elongation and tensile strength with weighing factors was analyzed to achieve the greatest effectiveness combination of factors. Examination of microstructure by scanning electron microscopy confirmed the consistency between the recycled alloy and the die-cast counterpart.

The last of the presented manuscripts detailed the optimization for both recovery rate and corrosion resistance. DOE and ANOVA was utilized for experimental design and data analysis. In this manuscript, The calculation of the corrosion resistance of samples is based on the corrosion potential, the corrosion current density, and the anodic/cathodic Tafel slopes (β_a and β_c) which were derived from the measured polarization curves. Two multi-response objectives were selected based on different requirement for metal production, and they were investigated to make the greatest effectiveness for each case.

A table highlighting the original publication information for each of the manuscripts can be found in Table 1-1

Table 1-1 Publication information for presented manuscripts

Chapter	Manuscript Title	status
II	Recycling of Aluminum A380 Machining Chips	Published
III	Process Optimization for Recycling of Machining Chips of Die Cast Aluminum Alloy A380	Submitted
IV	Optimization of Aluminum Chips Recycling Process for Recovery Rates and Tensile Properties of Aluminum Alloy A380	Unpublished
V	Optimization of Aluminum Chips Recycling Process for Corrosion Resistance of Aluminum Alloy A380	Unpublished

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2. CHAPTER II

RECYCLING OF ALUMINUM A380 MACHINING CHIPS

Aluminum and its alloys have experienced significant increases in their usage in the automotive industry for the past few decades. Large quantity of aluminum is being produced everyday with huge waste such as dross and chips. Due to environmental and cost issues, production of aluminum via recycling is increasingly becoming a must for further expansion. However, technologies for aluminum recycling are far from perfection, in particular for machining chips. In this work, machining chips of aluminum alloy A380 were collected from computer numerical control (CNC) machines and then cleaned. The cleaned chips were thermally recovered with two fluoride-containing fluxes and one fluoride-free flux. The recovery rate of the recycled metal was determined based on weight measurements. The results of tensile testing, microstructure analysis and chemical composition evaluation indicate that the quality of the recovered metal is comparable to die-cast A380.

2.1. Introduction

In the past two decades, aluminum alloys as a light weight material have been increasingly used in the automotive industry. Worldwide average aluminum content was 7.8% of the average worldwide light vehicles curb weight of 3,183 pounds in 2009. North America (NA) has the highest aluminum penetration at 8.6% of North

America curb weight in the world. The usage of aluminum in North American automobiles has gone from 45kg (101 lbs.) in the 1970s to 150 kg (326 lbs.) in 2009, and will top 170 kg (376 lbs.) per vehicle by 2020. Among the 150 kg aluminum usage in each vehicle, almost 35% of automotive aluminum components were manufactured by conventional high pressure die-casting (C-HPDC) processes [1]. When C-HPDC components are manufactured, considerable amount of aluminum waste in the form of scrap, dross, and machining chips is produced as byproducts. The casting scrap is easily returned to melting, whereby most of the metal is recovered and re-utilized in production processes. The recovery of aluminum from dross can be achieved at a recovery rate of around 80% by mixing dross and chips with certain types of flux [2-6]. During the recycling of chips and dross, however, a lot of metal is lost as a result of oxidation, and the costs of labor and energy as well as the expenditure on environmental protection increase the general cost of the process. The chips as a by-product not only bring huge waste, but also could produce pollution to the environment. Also, due to high market demand for cost saving on die castings, the recovery of Al chips becomes critical for die casters. However, recovery rates of the chips are often unknown to die casting shops since most chips are presently recycled externally and aluminum content in the chips depends on the practice of molten metal processing.

According to Gronostajski J.[7], in the process of melting aluminum and aluminum alloy chips, on average 10% of the metal is burnt and about 10% is lost because dross formed by mixing molten aluminum and slag were removed from the surface on the ladle. And add by 8% loss of casting scraps, 72% aluminum would be recycled after casting. Thus the anticipated recovery rate is around 72%.

In this study, the chips collected directly from CNC machines were recycled with flux. The recovery rate of the recycled metal was determined based on weight measurements. To ensure the quality of the recycled aluminum, the chemistry of the recovered aluminum was analyzed. The cleanliness of the recycled metal was assessed based on microstructural analysis. The tensile properties of the recovered aluminum cast in an ingot mold were evaluated.

2.2. Experimental Procedures

2.2.1. Materials

Machining chips of aluminum alloy 380 shown in Fig.2- 1 were the raw material to be recycled, of which chemical composition is listed in Table 2-1.

Figure 2.2-1 Machining chips of aluminum alloy A380.

Table 2-1 Chemical Compositions of Aluminum Alloy A380 [8]

Alloy	Element (in wt. %)			
A380	Si	Cu	Zn	Fe
	7.5-9.5	3.0-4.0	3.0 max	1.3 max
	Ni	Mg	Sn	Mn
	0.5 max	0.1 max	0.35 max	0.5 max

2.2.2. Cleaning

For safety and health considerations, wet machining chips should be cleaned before thermal recycling. There are several cleaning methods such as cleaning with solvent, thermal method and hot press. In this study, cleaning with water and solvent was applied, and their cleaning effect was concluded by the observation of the reduction in smoke emission during the heating stage of the thermal recycling process. The clean processes included rinsing wet chips with water at room temperature, and soaking them in plastic buckets with acetone for 6 hours, ladling them onto aluminum foils, and dry them in a fume hood for 12 hours.

2.2.3. Refining

300g of cleaned chips were loaded into a clay-graphite crucible inside an electric resistance furnace, the crucible was heated to 500^{o}C for 20 minutes of preheating to remove moisture, and then refining flux was added into the crucible to cover the chips. Three different kinds of fluxes made by Basic Resources Inc. were selected for the purpose of comparison. They were Al-clean 101 [9], Al-clean 113 [10] and Al-clean 116 [11]. Two of them, Al-clean 101 and Al-clean 116 were fluoride-containing flux, and Al-clean 113 was fluoride-free flux. 1:1 of chips/flux weight ratio was employed. The crucible with chips and flux was held at 500°C for 20 minutes.

2.2.4. Melting and casting

After chips and flux were preheated, the temperature of the furnace was increased to 800°C for 60 minutes to 90 minutes. The slag floating on top of liquid aluminum was scooped out. After cleaning the slag, the recovered liquid aluminum alloy was poured at 720°C into an ingot mold and cast as a plate. The solidified aluminum plate was quenched in tap water. Three typical experiments on cleaned chips were conducted following Table 2-2. Here, each trial of 22 experiments was carried out based on previous trial. Here, No.11, 16 and 18 were selected for their high recovery rate. And to reach the ideal recovery rate, flux types, chips/flux ratio and holding time were selected as the influencing factors during these experiments.

Table 2-2 Experimental records of refining and melting processes

No.	Chips (g)	Flux type	Chips/flux ratio	Hold time (min)	
				Heating	Melting
11	300	116	1 : 1	15	60
16	300	113	1 : 1	20	75
18	300	101	1 : 1	20	60

2.2.5. Determination of recovery rate

Chips were weighed prior to refining experiments, and the recovered aluminum alloy was weighed after the experiments. The recovery rate of the chips was determined based on the following expression:

$$\text{Recovery Rate (\%)} = (\text{Weight of recovered Al}) / (\text{Chips Weight}) \qquad (2—1)$$

2.2.6. Quality Assessment of Recycled Aluminum Alloy

2.2.6.1. Density measurement

Following the measurement of specimen weight in the air and distilled water, the actual density (D_a) of each sample with the dimensions of 10x10x10 mm was determined using the Archimedes' principle [12].

$$D_a = W_a D_w / (W_a - W_w) \qquad (2—2)$$

Where W_a and W_w are the weight of the specimen in the air and in the water, respectively, and D_w is the density of water.

2.2.6.2. Tensile testing

The mechanical properties of the recycled aluminum were evaluated by tensile testing, which was performed at room temperature on a MTS criterion Tensile Test Machine (Model 43) equipped with a data acquisition system. Following ASTM B557 -14[13], 3 chosen flat tensile specimens (25 mm in gage length, 6 mm in width, and 3 mm in thickness) were machined from each recycled aluminum plate and 3 tensile tests were carried out for each flux type.. The tensile properties, including ultimate tensile strength (UTS), 0.2% yield strength (YS), and elongation to failure (E_f) were recorded during the tests.

2.2.6.3. Microstructure analysis

The microstructure of the recycled alloy characterized by a Buehler optical image analyzer 2002 system was used to determine the primary characteristics of the specimens prepared by the standard metallographic procedure. The detailed features of the microstructure were analyzed using a FEI Quanta 200 FEG scanning electron microscope (SEM).

2.2.6.4. Chemical analysis

The chemical composition of the recovered alloy was analyzed by an optical emission spectrometer (ARL 4460 metal analyzer).

2.3. Results and Discussion

2.3.1. Recovery Rate

Table 2-3 lists the recovery rates of three typical Al chips samples from the CNC machines and cleaned with acetone. For the purpose of comparison, the data listed in Table 2-3 were also plotted in Fig.2-2. It can be seen that the recovery rates of the chips are around 90%, three kinds of fluxes have similar effect on aluminum recovery rate.

Table 2-3 Recovery rates of recycled aluminum chips

No.	Cleaned chips weight (g)	Weight of recovered Al (g)	Recovery rate: (Wt. %)
11	300	268	89.3%
16	300	265	88.3%
18	300	271	90.3%

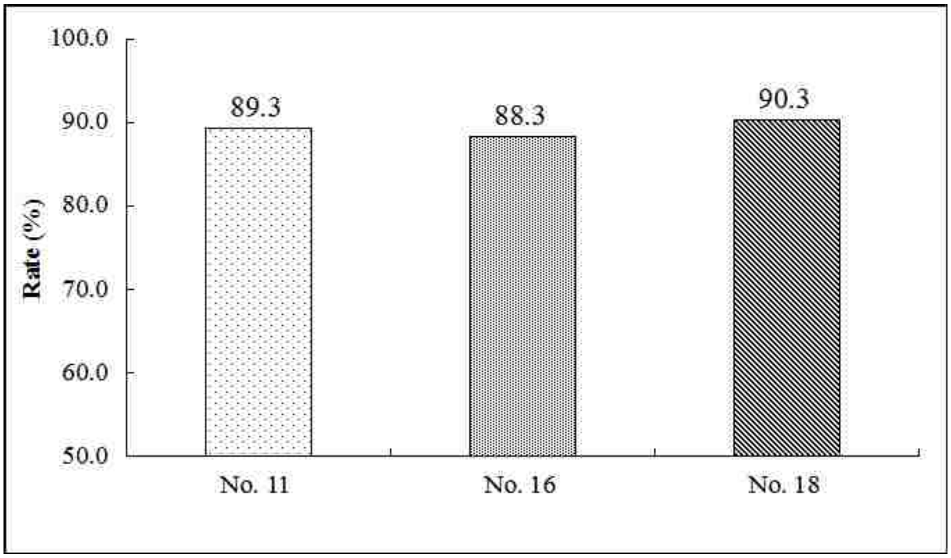

Figure 2.3-1 Recovery rates of twenty-two recycling experiments

2.3.2. Density Measurement

The density measurements of the recovered aluminum are given in Table 2-4. Those recycled aluminum have an average density of 2.7567 g/cm^3, which was slightly lower than that of the die-cast A380 alloy [14].

Generally, the relatively low density is due possibly to the fact that the recovered aluminum which was cast in an ingot mold under open atmosphere may be less dense than die cast alloy 380 under an applied pressure with low porosity. Compared to No.16, which had a holding time of 75minutes during melting process, the density of No.11 and No.18 were slightly lower because of the entrapment of porosity and impurities. It suggested that the extended holding time during refining enhanced the elimination of oxides and impurities from chips by the reaction between the flux and chips.

Table 2-4 Density measurement of recycled aluminum

No.	11	16	18	Alloy A380 (Die-cast)[14]
Density (g/cm3)	2.7572	2.7917	2.7213	2.7981

2.3.3. Tensile properties

According to Table 2-5, die casting aluminum-alloy A380 has the tensile strength of 182.18 MPa; here the recycled ones reached 202.71 MPa, and also the other two have UTS values higher than the die cast one. It may be because the water quenching right after casting the recycled alloy led to an increase in the UTS.

The yield strength of the die cast A380 is 136.02 MPa. But, the yield strength of the recycled alloys was lower than 100 MPa. The reduction in yield strength should be likely attributed to the fact that the recovered alloys entrapped porosity and impurities due to incomplete refinement. This observation suggested that the secondary refining needs to be applied the recycled alloys for further cleaning. .

Table 2-5 Tensile properties of the recovered alloy and A380

No.	11	16	18	Alloy A380 (Die-cast)[14]
UTS (MPa)	187.23	202.71	192.87	182.18
YS (MPa)	92.3	91.12	91.36	136.02
Ef(%)	2.79	4.37	3.10	1.11

The yield strength of the die cast A380 is 136.02 MPa. But, the yield strength of the recycled alloys was lower than 100 MPa. The reduction in yield strength should be likely attributed to the fact that the recovered alloys entrapped porosity and impurities due to incomplete refinement. This observation suggested that the secondary refining needs to be applied to the recycled alloys for further cleaning.

2.3.4. Microstructure analysis

Fig. 2-3 to Fig. 2-5 show the microstructures of the aluminum alloys recovered from chips. It is seen from Fig.2-3 that the recovered alloy had some black dots, which were porosity and oxides. But the content of porosity and oxide inclusions ratio was relatively low. It is evident that the microstructure of the ingot mold-cast recovered aluminum alloy contained α ($Al_{15}Fe_3Si_2$) phase and β (Al_5FeSi) phase, Si phase, $CuAl_2$. The phase observation indicates that the recovered aluminum alloy possessed the same types of phases as those present in the die cast A380 given in reference 14.

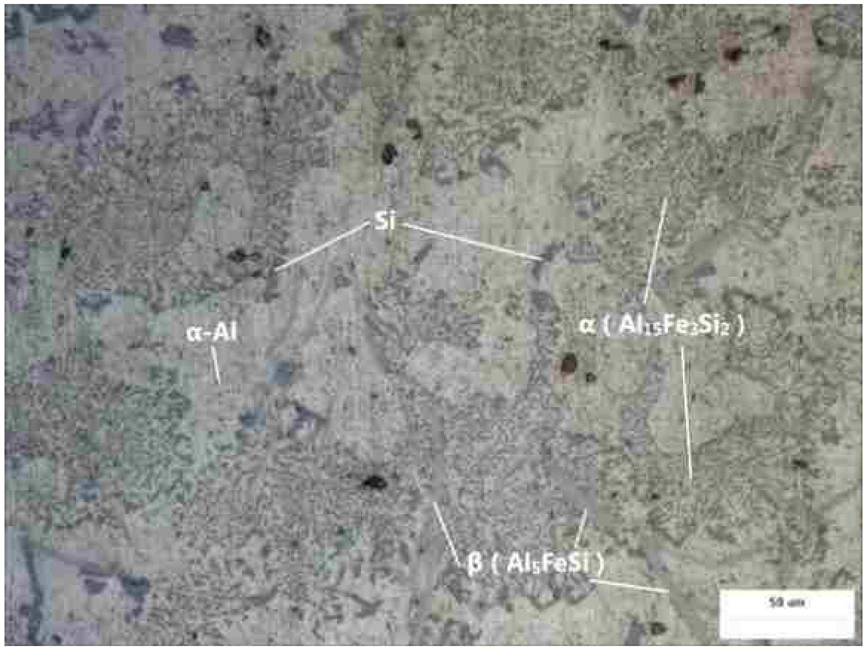

Figure 2.3-2 Optical micrograph showing microstructure of the recycled alloy

Figure 2.3-3 Optical micrograph showing microstructure of the recycled alloy

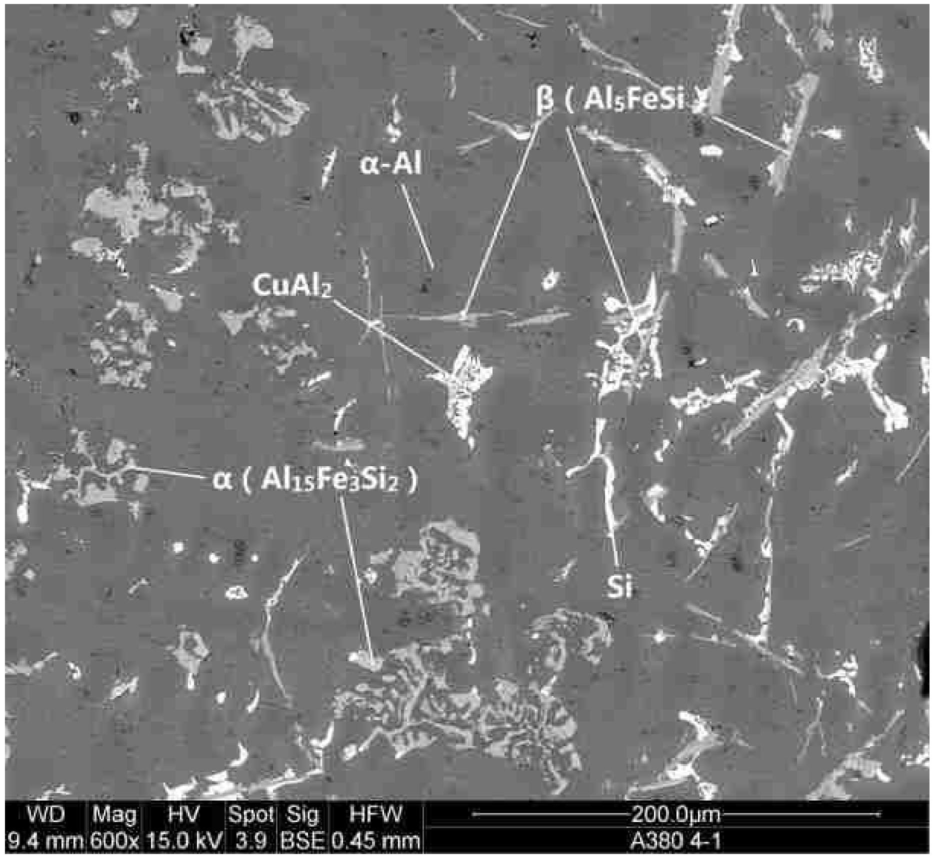

Figure 2.3-4 SEM micrograph showing microstructure of the recycled alloy

2.3.5. Chemical analysis

Though chemical analysis was carried on 3 pieces of recycled plates from experiment No. 11, 16, 18, they exhibited similar results. Table 2-6 exhibits the chemical composition of recovered aluminum plates. It is seen the chemical composition for most elements of recovered metal was similar to die-cast A380 aluminum referring to Table 2-1 mentioned above.

Compared to Fig.2- 1, concentration of each element were still within the range. However, silicon concentration was in a high level within the range 7.5-9.5 while magnesium had a relative low concentration. This is possibly because silicon is more stable in elevated temperature compared to magnesium; magnesium would be more likely oxidized than silicon at the temperature.

Table 2-6 Chemical composition of the recovered aluminum alloys

No.	Element (in wt. %)			
	Si	Cu	Zn	Fe
11	9.353	3.315	2.043	1.016
16	9.400	3.454	2.054	0.976
18	9.414	3.332	2.298	1.000
	Ni	Mg	Sn	Mn
11	0.077	0.006	0.019	0.219
16	0.081	0.009	0.020	0.208
18	0.077	0.006	0.020	0.220

2.4. Conclusions

Conclusions were drawn based on recycling experiments in this study:

Acetone is a good agent to remove oil emulsion presented on the surface of aluminum machining chip.

Cleaning fluxes 101, 113 and 116 had similar influence on recovery rates, but considering environmental concerns, Al-clean 113, a fluoride-free flux, was suggested.

The recovery rate of aluminum alloy chips A380 reached as high as 90.3%.

The mechanical properties of the recovered aluminum were as good as those of the die-cast A380 aluminum alloy. The microstructure of the recovered aluminum alloys also contained the primary α-Al, Si phase, $CuAl_2$, Fe containing inter-metallic phases, which are almost the same as those present in the die cast A380. Despite of high concentration of silicon and low concentration of magnesium, the recovered aluminum alloy had an acceptable chemical composition.

Acknowledgements

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3. CHAPTER III

PROCESS OPTIMIZATION FOR RECYCLING OF MACHINING CHIPS OF DIE CAST ALUMINUM ALLOY A380

Due to environmental and cost issues, production of aluminum via recycling is increasingly becoming essential for further expansion. However, technologies for aluminum recycling are far from perfection, in particular for machining chips. In this work, machining chips of high pressure die cast aluminum alloy A380 were collected from computer numerical control (CNC) machines and recycled under a series of designed experiments using Taguchi Method. To optimize recycling process, flux types, chips/flux ratio, holding times and holding temperatures were selected as four factors. For each factor, three corresponding levels were also chosen to create Taguchi orthogonal array. Recovery rate and porosity content were selected as two individual responses to evaluate the effectiveness of the recycling process and the quality of the recycled alloy. Also, S/N ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data for optimization. The optimum combinations led to the highest recovery rate of 92.03% by using Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, 60 minutes as the holding time and 760℃ as the holding temperature, while the combination using Al-clean 113 as the refining flux, 10:4 as the chips/flux ratio, 60 minutes as the holding time and 800℃ as the holding temperature made the recycling process effective considering both the recovery rate and porosity content as objective functions. Examination of microstructure by scanning electron microscopy confirmed the consistency between the recycled alloy and the die-cast counterpart.

3.1. Introduction

In the past two decades, aluminum (Al) alloys as a light weight material have been increasingly used in the automotive industry. North America (NA) has the highest aluminum penetration at 8.6% of North American curb weight in the world. The usage of aluminum in North American automobiles has gone from 45 kg (101 lbs.) in the 1970s to 150 kg (326 lbs.) in 2009, and will top 170 kg (376 lbs.) per vehicle by 2020. Among the 150 kg aluminum usage in each vehicle, almost 35% of automotive aluminum components were manufactured by conventional high pressure die-casting (C-HPDC) processes [1]. When C-HPDC components are manufactured, considerable amount of aluminum waste in the forms of scrap, dross, and machining chips are produced as byproducts. The casting scrap is easily returned to melting; where by most of the metal is recovered and re-utilized in production processes. The study by Gronostajski and Matuszak [2] showed that, in the process of melting aluminum and aluminum alloy chips, on average, 10% of the metal was burnt and about 10% was lost because dross formed by mixing molten aluminum and slag were removed from the surface of liquid aluminum in the ladle. Also considering 8% loss of casting scraps, 72% aluminum would be recycled after casting. Thus the anticipated recovery rate of conventional recycling processes was around 72%. The recovery of aluminum from dross can be achieved at a recovery rate of around 80% by mixing dross [3] and chips [4] with certain types of fluxes. During the recycling of machining chips and melt dross, however, large amount of metal is lost as a result of oxidation, and the costs of labor and energy as well as the expenditure on environmental protection increase the general cost of the process. The chips as a by-product not only bring huge waste, but also could produce pollution to the environment. Also, due to high market demand for cost saving on die castings, the recovery of Al chips becomes critical for die casters.

However, recovery rates of the chips are often unknown to die casting shops since most chips are presently recycled externally and aluminum content in the chips depends on the practice of molten metal processing. Reducing the aluminum loss is the key to optimize the conventional recycling process. There are several influencing factors during the processes, such as flux types, amount of flux, stirring time, protective gas, holding time and holding temperature during melting, pouring temperature, etc., and for each factor, there are quantities alternative levels. To find the optimum process, many combinations of influencing factors and levels need to be experimented.

The Taguchi method uses a special design of orthogonal arrays to study all the designed factors with a minimum of experiments at a relatively low cost. Orthogonality means that factors can be evaluated independently of one another; the effect of one factor does not interfere with the estimation of the influence of another factor [5].

In this study, the Taguchi method for design of experiment (DOE) was used for the optimization of the recycling process for machining chips of high pressure die cast aluminum alloy A380. Since the preliminary results [6] indicates that the recovery rate was primarily determined by several key process parameters such as flux type, chips/flux ratio, holding time and holding temperature during melting, the present design of experiment took into account the influencing extent of each individual process parameter. This consideration led to the selection of those four influencing factors with three different levels. The results of the factor response analysis were used to derive the optimal level combinations. The contribution of each factor was determined by an analysis of variance. The chips collected directly from CNC machines were recycled with refining flux. The recovery rate of the recycled metal was determined based on weight measurements. To ensure the quality of the recycled aluminum, the porosity content and microstructure of the recovered aluminum alloy was analyzed.

3.2. Experimental Procedures

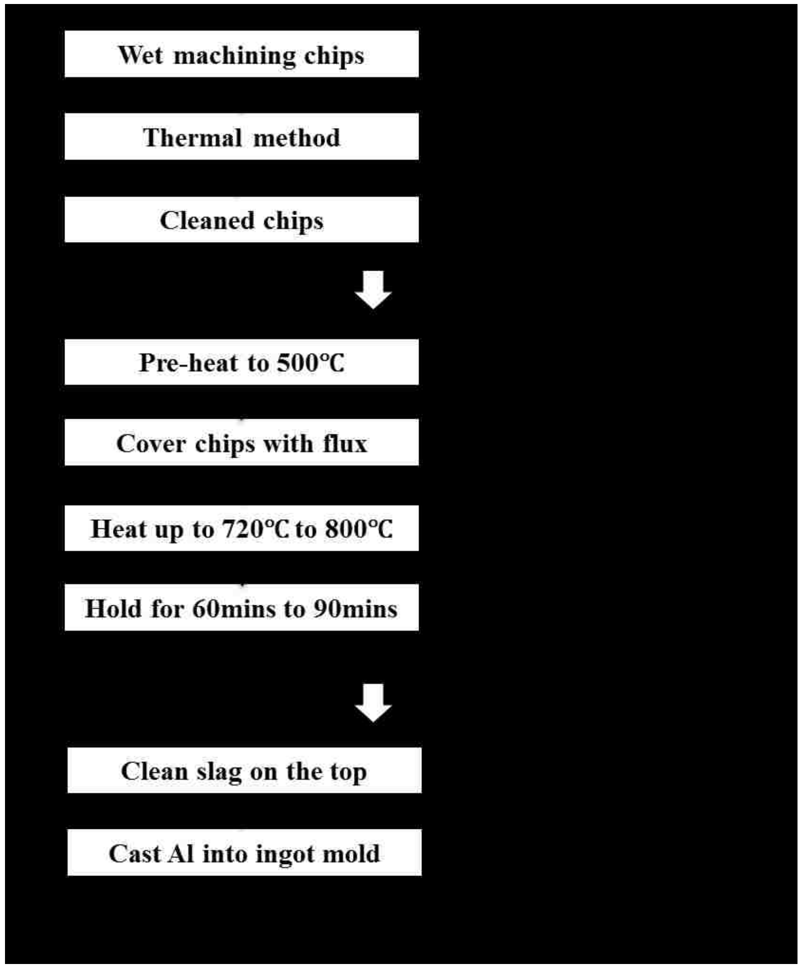


Figure 3.2-1 Flowchart of the recycling process

Fig.3-1 shows the flowchart of the recycling process used in this study. After cleaning, chips were loaded into a crucible and pre-heated to 500℃. Flux types and chips/flux weight ratio were selected as factors A and B in the DOE, respectively. The holding time and holding temperature were chosen as factors C and D.

3.2.1. Materials

Machining chips of high pressure die-cast aluminum alloy 380 shown in Fig.3-2(a) were the raw material to be recycled. The chips were wet and covered with coolants when collected from the CNC machines. Fig.3-2(b) shows one of the recycled aluminum plate.

(a) (b)

Figure 3.2-2 (a) machining chips of aluminum alloy 380, and (b) a cast plate of the recycled alloy.

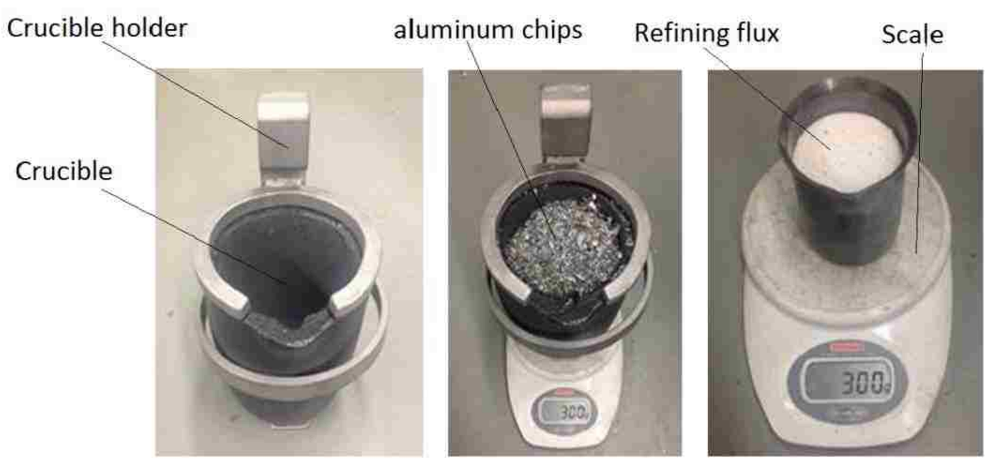
3.2.2. Cleaning

For safety and health considerations, wet machining chips were cleaned before refining process. Thermal method was employed in this study. Wet machining chips were loaded into a crucible and then, the crucible was heated up to the temperature of 400℃ for 45mins to 60mins in a furnace. With this kind of cleaning method, emulsions and coolant were easily burnt out. Then place those cleaned aluminum chips in a fume hood. Fig.3-3(a) showed a clay graphite crucible and a crucible holder used during cleaning and refining process.

3.2.3. Refining

300 grams of cleaned and dried chips were loaded into a clay-graphite crucible inside an electric resistance furnace. The chips inside the crucible was heated to 500℃ for 20 minutes of preheating to remove any entrapped moisture, and then refining flux was added into the crucible to cover the chips. Three different kinds of fluxes made by Basic Resources Inc. were selected for the purpose of comparison. They were Al-clean 101 [7], Al-clean 113 [8] and Al-clean 116 [9]. Two of them, Al-clean 101 and Al-clean 116 were fluoride-containing flux, and Al-clean 113 was fluoride-free flux. The chips/flux ratio was selected based on DOE. The crucible with chips and flux was held at 500℃ for 20 minutes.

After chips and flux were preheated, the temperature of the furnace was increased to a desired temperature for holding a fixed period of time given by the DOE.



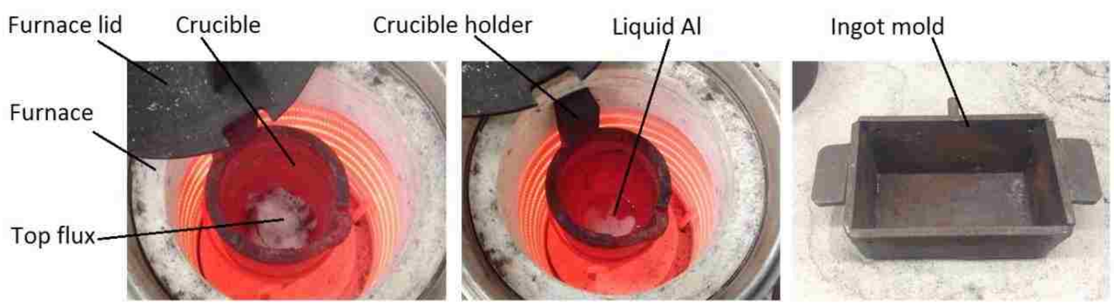
(a) (b) (c)

Figure 3.2-3 (a) crucible and its holder used in cleaning and refining process; (b) aluminum chips loaded into crucible; (c) refining flux.

3.2.4. Melting and casting

The slag floating on top of liquid aluminum was scooped out after the holding process. After removing the slag, the recovered liquid aluminum alloy was poured into an ingot mold and cast as a plate (Fig.3-1(b)). The solidified aluminum plates were quenched in water for analysis.

Fig.3-4(a) showed the melt mixture of the flux and chips in the crucible as the holding temperature reached 800℃, while Fig.3-4(b) depicted the recovered aluminum alloy after slag removal and before casting the alloy into the ingot mold (Fig.3-4(c)).



(a) (b) (c)

Figure 3.2-4 (a) melt mixture of the flux and chips; (b) recovered Al in the crucible; (c) ingot mold.

3.2.5. Recovery rate and porosity content

Chips were weighed after cleaning and prior to refining experiments, while the recovered aluminum alloy in the form of the cast plate was weighed after refining experiments. The recovery rate of the chips was determined based on the following expression:

$$\text{Recovery rate } (\%) = \frac{\text{weight of recovered Al}}{\text{chips weight}} \times 100 \qquad (3\text{—}1)$$

here the weight of the cleaned and dried aluminum chips was 300 grams for each test of all the nine designed recycling experiments. The plan for the DOE and the weight of the recycled aluminum plates are given in Table 2-2.

To determine the porosity content of the recycled alloy, density measurements were performed. The weight of specimens cut from the recycled plates with the dimensions of 10×10×10 mm was measured in the air and distilled water. The actual density (D_r) of each sample was determined using the Archimedes' principle [10]. Then, comparing to the density of the die-cast aluminum alloy A380 [11] the density and porosity content were calculated by using the equations below:

$$D_r = \frac{W_a \times W_w}{W_a - W_w} \qquad (3\text{—}2)$$

$$\text{Porosity } (\%) = 1 - \left(\frac{D_r}{D_a}\right) \times 100 \qquad (3\text{—}3)$$

where W_a and W_w are the weight of the specimen in the air and in the distill water, respectively, and D_w is the density of water; Dr is the density of recovered aluminum plates and D_a is the theoretical density of die-cast aluminum alloy A380, 2.7981 g/cm3.

3.2.6. Microstructure Analysis

Specimens for microstructural analyses were cut from the interior of the components and prepared following the standard metallographic procedures. After proper polishing and etching (0.5% HF acid solution), microstructural changes were examined on the surface of metallographic specimens obtained from as-cast samples using scanning electron microscopy (SEM).

3.3. Taguchi design of experiment

3.3.1. Design of orthogonal array

Concluded from the experimental procedures, Table 3-1 gave the parameters selected for specific experimental parameters. Here four factors (flux type, chips/flux ratio, holding temperature and holding time during melting) with three levels were selected shown in Table 3-2. The factors and levels were used to design an orthogonal array L_9 (3^4) for experimentation (Table 3-3). Since each experiment was repeated once for verification, in total, the eighteen (18) tests were conducted base on the DOE given in Table 3-1 with four factors and three levels.

Table 3-1 Summary for experimental parameters

Flux type (Al-clean)	Chips/flux ratio	Heating time (mins)	Heating temperature (°C)	Holding time (mins)	Holding temperature (°C)	Stirring time (mins)
101	10:3			60	720	
113	10:4	20	500	75	760	5
116	10:5			90	800	

Table 3-2 Design factors and levels

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	101	10:3	60	800
2	113	10:4	75	760
3	116	10:5	90	720

Table 3-3 Designed experiment plans

Experiment	A Flux Type (Al-clean)	B Chips/Flux Ratio	C Holding Time (mins)	D Holding Temperature (℃)
1	(1) 101	(1) 10:3	(3) 90	(2) 760
2	(2) 113	(1) 10:3	(1) 60	(1) 800
3	(3) 116	(1) 10:3	(2) 75	(3) 720
4	(1) 101	(2) 10:4	(2) 75	(1) 800
5	(2) 113	(2) 10:4	(3) 90	(3) 720
6	(3) 116	(2) 10:4	(1) 60	(2) 760
7	(1) 101	(3) 10:5	(1) 60	(3) 720
8	(2) 113	(3) 10:5	(2) 75	(2) 760
9	(3) 116	(3) 10:5	(3) 90	(1) 800

3.3.2. Signal-to-noise analysis with multiple characteristics

In process design, it is almost impossible to eliminate all errors caused by the variation of characteristics. An increase in the variance of multiple characteristics lowers the quality reliability of the recycling process. The Taguchi method uses signal-to-noise (S/N) ratio instead of the average value to interpret the trial results data into a value for the evaluation characteristic in the optimum setting analysis. To minimize the influence of the recovery rate and porosity variation on the analysis of experimental data, the signal-to-noise(S/N) ratio was employed, which converted the trial result data into a value for the response to evaluate the recycling process in the optimal setting analysis. The S/N ratio consolidated several repetitions into one value which reflected the amount of variation present. This is because the S/N ratio can reflect both the average and the variation of the quality characteristics. There are several S/N ratios available depending on the types of characteristics [10]: lower is best (LB), nominal is best (NB), and higher is best (HB). In the present study, recovery rates were treated as a characteristic value. Since the recovery rates of the recycling process were intended to be maximized, the S/N ratio for HB characteristics was selected, which was be calculated as follows:

$$S/N_{HB} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{\eta_{pi}^{2}}\right) \qquad (\text{-3—4})$$

where n is the repetition number of each experiment under the same condition for design parameters, and$\eta_{pi}$is recovery rate of an individual measurement at the ith test.

The porosity level was treated as negative effects or defects to aluminum alloys. The response was intended to be minimized; the S/N ratio for LB characteristics was selected and was calculated as:

$$S/N_{LB} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\eta_{pi}^{2}\right) \qquad (\text{-}3\text{—}5)$$

where n is the repetition number of each experiment under the same condition for design parameters, and$\eta_{pi}$is the porosity level of an individual measurement at the ith test. After calculating and plotting the mean S/N ratios at each level for various factors, the optimal level, that was the largest S/N ratio among all levels of the factors, was determined.

The proposition for the optimization of recycling process with multiple performance characteristics (two objectives) using a weighting method is defined as the Eqs. (3-6) – (3-8):

$$Y_{SUM} = Y_P \times w \qquad (3\text{—}6)$$

where

$$Y_{SUM} = \begin{bmatrix}\eta_{1c}\\ \eta_{2c}\\ \vdots\\ \eta_{9c}\end{bmatrix};\quad Y_p = \begin{bmatrix}\eta_{11}\eta_{12}\\ \eta_{21}\eta_{22}\\ \vdots \quad \vdots\\ \eta_{91}\eta_{92}\end{bmatrix};\quad w = \begin{bmatrix}w_1\\ \\ w_2\end{bmatrix} \qquad (3\text{—}7)$$

$$\sum_{i=1}^{2} w_i = 1 \qquad (\text{-}3\text{—}8)$$

where w_1 and $w_2$are the weighting factor of recovery rate and porosity, respectively. η_{jc} is the multi S/N ratio in the jth test, η_{ji} is the ith single response S/N ratio for the jth test; w_i is the weighting factor in the ith performance characteristics.

The objective function was formulated according to the previous optimization criteria:

$$\text{Maximize } f(X) = w_1 \cdot \eta_{recovery} + w_2 \cdot \eta_{porosity} \qquad (3\text{—}9)$$

the above objective function is presented in an analytical form as function of input parameters since increased productivity and reduced porosity play the important roles during recycling of machining chips. However, in the actual manufacturing process, for different metal specifications, the two characters should be considered as different critical roles by weighting factors. When quality demand becomes critical, high weighting factors of porosity needs to be considered. For metal yield requirement, high recovery factor may require due to the consideration of cost saving. In this study, case 1 (1.0, 0), and case 2 (0.5, 0.5) with two different combinations of weighting factors were selected for demonstrating recycling requirements.

3.3.3. Analysis of variance (ANOVA)

The analysis of variance (ANOVA) on the experimental results was performed to evaluate the source of variation during the recycling process. Following the analysis, it was relatively easy to identify the effect order of factors on recovery rate and porosity level of the recycled alloys as well as the contribution of factors to corresponding characteristics. In this study, the variation due to both the four factors and the possible error was taken into consideration. The ANOVA was established based on the sum of the square (SS), the degree of freedom (D), the variance (V), and the percentage of the contribution to the total variation (P). The five parameters symbols typically used in ANOVA [12] are described below:

1. Sum of squares (SS). SS_P denotes the sum of squares of factors A, B, C, and D; SS_e denotes the error sum of squares; SS_T denotes the total sum of squares.

The total sum of square SS_T from S/N ratio was calculated as:

$$SS_T = \sum_{i=1}^{m} \eta_i^2 - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \qquad (3—10)$$

where m is the total number of the experiments, and η_i is the factor response at the ith test.

The sum of squares from the tested factors, SS_p, was calculated as:

$$SS_P = \sum_{i=1}^{m} \frac{\left(S_{\eta_{jc}}\right)^2}{t} - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \quad (3-11)$$

where m is the number of the tests (m= 9), j the level number of this specific factor p, t is the repetition of each level of the factor p, and $S_{\eta j}$ the sum of the multi-response S/N ratio involving this factor p and level j.

2. Degree of freedom (D). D denotes the number of independent variables. The degree of freedom for each factor (D_P) is the number of its levels minus one. The total degrees of freedom (D_T) are the number of total number of the result data points minus one, i.e. the total number of trials times the number of repetition minus one. And the degree of freedom for the error (De) is the number of the total degrees of freedom minus the total of degree of freedom for each factor.

3. Variance (V). Variance is defined as the sum of squares of each trial sum result involved the factor, divided by the degrees of freedom of the factor:

$$Vp\ (\%) = \frac{SS_P}{D_P} \times 100 \quad (3-12)$$

4. The corrected sum of squares (SSp). SSp is defined as the sum of squares of factors minus the error variance times the degree of freedom of each factor:

$$SS_P{}' = SS_P - D_P V_e \quad (3-13)$$

5. Percentage of the contribution to the total variation (P). Pp denotes the percentage of the total variance of each individual factor:

$$Pp\ (\%) = \frac{SS_P{}'}{SS_P} \times 100 \quad (3-14)$$

3.4. Results and Discussion

3.4.1. Multi-response of S/N ratios

The recovery rate and porosity content were selected as two original responses. Two combinations of weighting factors were selected in this study for the multi-response S/N ratio calculated from Eqs. (3-6) – (3-9) to evaluate the effectiveness and efficiency of the recycling process and the quality of the recycled plates for different requirements.

Table 3-3 gives the data of original results. The recovery rates were calculated with Eq. (3-1) using the weight of recycled aluminum. The density and porosity were calculated with Eq. (3-2) and Eq. (3-3), respectively.

Table 3-4 Data of original results								
Experiment	Recycled Aluminum(g)		Recovery rate (%)		Density (g/cm^3)		Porosity (%)	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
1	260.68	255.71	86.89	85.24	2.7356	2.7885	2.2319	0.3413
2	230.80	256.18	76.93	85.39	2.7807	2.7785	0.6210	0.6996
3	228.27	262.66	76.09	87.55	2.7453	2.7836	1.8877	0.5184
4	267.53	270.31	89.18	90.10	2.7781	2.7813	0.7152	0.6021
5	235.23	247.15	78.41	82.38	2.7658	2.7692	1.1549	1.0333
6	259.36	274.32	86.45	91.44	2.7641	2.7796	1.2134	0.6607
7	270.63	267.40	90.21	89.13	2.7683	2.7524	1.0646	1.6317
8	246.14	265.50	82.05	88.50	2.7551	2.7786	1.5370	0.6951
9	257.85	252.31	85.95	84.10	2.7554	2.7800	1.5262	0.6480

Table 3-5 S/N ratio of multi-response objectives				
Experiment	S/N ratio of Recovery rate	S/N ratio of porosity	S/N ratio of Multi-response	
			case 1 (w_1=1.0, w_2=0)	case 2 (w_1=0.5, w_2=0.5)
1	38.70	-4.06	38.70	17.32
2	38.15	3.59	38.15	20.87
3	38.19	-2.82	38.19	17.68
4	39.05	3.60	39.05	21.32
5	38.10	-0.79	38.10	18.65
6	38.97	0.20	38.97	19.59
7	39.05	-2.78	39.05	18.13
8	38.60	-1.53	38.60	18.53
9	38.59	-1.38	38.59	18.60

Since the objective, recovery rate, was intended to be maximized, the S/N ratio for HB (higher-is-better) characteristics was used; while the porosity level was intended to be minimized, the S/N ratio for LB (lower-is-better) characteristics was used. The S/N ratio of these two responses was given in Table 3-4, and the multi-responses of S/N ratio using two weighting factor combinations were also concluded in Table 3-4. The response of each factor to its individual level was calculated by averaging the S/N ratios of all experiments at each level for each factor. With three combinations of weighting factors, the factor's mean multi-response S/N ratios for each level are summarized in Table 3-5, respectively. For instance, the mean S/N ratio (38.93) for flux type and level 1 was the average value of the S/N ratios of experiment No.1 (38.70), No.4 (39.05) and No.7 (39.05).

3.4.2. Optimal recycling factors

The mean S/N ratio of the recovery rate was influenced by four factors, the flux type, chips/flux ratio, holding time and holding temperature. For each factor, the mean S/N ratios of case 1 (w_1=1.0, w_2=0) and case 2 (w_1=0.5, w_2=0.5) were plotted in Figs. 3-5 and Fig.3-6 based on the results given in Table 3-6

Table 3-6 The factor's Mean multi-response S/N ratio for each level with two weighting factors.

level	Mean S/N ratio for case 1 (w_1=1.0,w_2=0)				Mean S/N ratio for case 2 (w_1=0.5, w_2=0.5)			
	A Flux type	B Chip/flux ratio	C Holding time	D Holding tempera ture	A Flux type	B Chip/flux ratio	C Holding time	D Holding temperat ure
1	38.93	38.35	38.73	38.60	18.92	18.62	19.53	20.27
2	38.28	38.71	38.61	38.76	19.35	19.85	19.18	18.48
3	38.59	38.75	38.46	38.45	18.63	18.42	18.19	18.16

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It is shown in Fig.3-5 that mean S/N ratio of the factor flux type (factor A) reaches maximum using flux Al-clean 101 (level 1), and has the minimum using flux Al-clean 113 (level 2). As the flux Al-clean 101 has a melting temperature around 500℃ and Al-clean 113 has a melting temperature between 690℃ and 705℃. The flux Al-clean 101 is more easily softened to have larger contact area with aluminum chips to achieve higher effectiveness.

The effect of the chips/flux ratio (factor B) on the mean S/N ratio of the recovery rate also plotted in Fig.3-5. The mean S/N ratio of recovery rate grows when additional flux added. It can be seen from the ratio 10:3 (level 1) to the ratio 10:4 (level 2) that the additional flux enhances the recovery rate. This might be because sufficient flux can greatly protect the aluminum chips from being oxidized during melting process. The curve seems to reach to a plateau from the ratio 10:4(level 2) to 10:5 (level 3).This observation implied that the excessive amount of flux would result in minor effect on recovery rates. In the viewpoint of cost saving, the ratio 10:4 might be considered for recycling production. While the ratio 10:5 was employed, the recovery rate becomes the highest.

The lines plotted from C1 to C3 are the effect of holding time (factor C) on the mean S/N ratio of the recovery rate. The curve is much smoother without sharp fluctuations comparing to other plots, which means holding time has minor effect on the recovery rate. The mean S/N ratio decreases when extended holding times are employed. However, aluminum chips are more likely to be oxidized when being kept at elevated temperatures for a prolonged period of time. Thus, 60mins (level 1) is selected for its higher S/N ratio response.

The plot points D1 to D3 shows the holding temperature (factor D) on the mean S/N ratio of recovery rate. The mean S/N ratio reaches the peak at 760℃ (level 2), and then drops to720℃ (level 3).The working temperature of the three fluxes is within the temperature range of 700℃ to 800℃. Since the energy consumption for recycling is high and chips are more likely to be oxidized at high temperatures, the medium temperature is preferred.

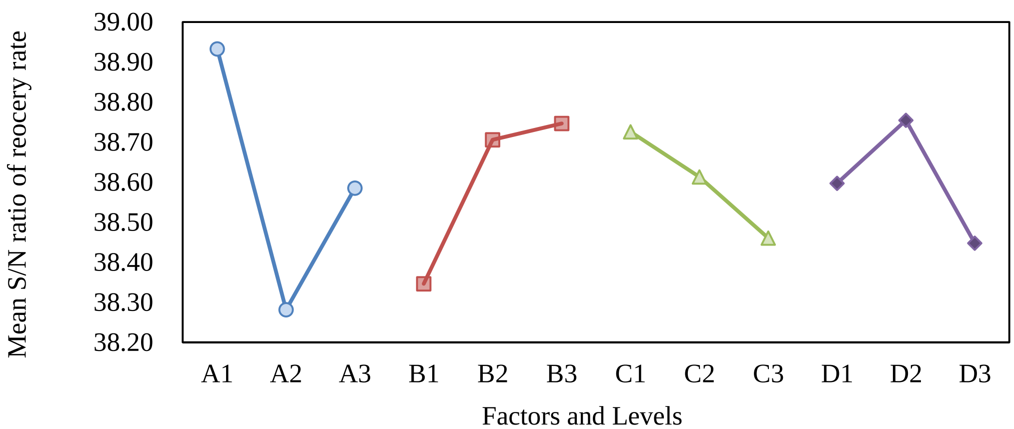


Figure 3.4-1 Multi-response signal-to-noise graph for case 1 (w_1 = 1.0, w_2=0)

By selecting the highest value of the mean S/N ratio for each factor, the optimal level can be determined. On this basis, the optimum combination of levels in terms of maximizing the recovery rate for this recycling process is A1B3C1D2; i.e. Al-clean 101 as the refining flux; 10:5 as the chips/flux ratio; 60mins as the holding time and 760℃ as the holding temperature.

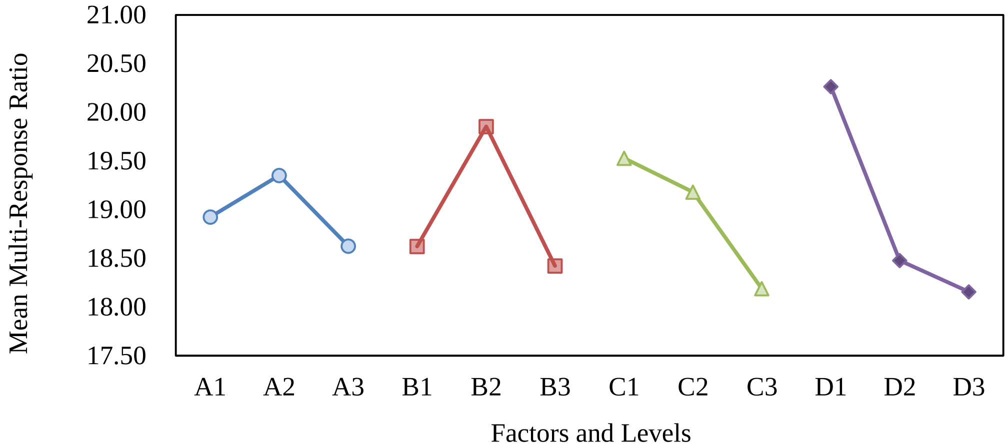


Figure 3.4-2 Multi-response signal-to-noise graph for case 2 (w1 = 0.5, w2=0.5)

Fig.3-6 shows the effect of flux type (factor A) on the mean S/N ratio of both the recovery rate and porosity content. The curve of factor A rises and then drops at level 3. It can be seen that both the flux Al-clean 101 (level 1) and Al-clean 113 (level 2) have advantages over Al-clean 116 (level 3). Both level 1 and level 2 reduced the porosity content in the recycled alloys. The flux of Al-clean 113 (level 2) performs slightly better than Al-clean 101 (level 1).

The plot of the chips/flux ratio (factor B) vs. mean S/N ratio shown in Fig.3-6 starts with a low S/N ratio at the chips/flux ratio 10:3 (level 1) and reaches its peak at 10:4 (level 2), then decreases to the ratio 10:5 (level 3). It can be concluded that suitable amount of flux has the ability to reduce impurities and inclusions trapped in molten aluminum. The chips/flux ratio 10:3 (level 1) is insufficient to eliminate impurities while chips/flux ratio 10:5 (level 3) might introduce excessive foreign particles causing impurities and inclusion issues during refining and casting.

The effect of the holding time (factor C) on the mean S/N ratio of the porosity is shown in Fig.3-6. The mean S/N ratios are low at the holding time of 90mins (level 3), and the maximum value is at 60mins (level 1). And using 75min (level 2) has similar but less effectiveness than 60mins. For 60mins holding time, the reaction between molten aluminum and flux is adequate for impurities and inclusions to float and settle and to be eliminated and separated from the liquid aluminum. When the holding time is excessive, on the other side, new impurities and inclusions could have great potential to be generated and trapped by the liquid aluminum due to oxidation, especially when the holding time is extended to90mins. Thus, 60mins as the holding time should be preferred. The effect of holding temperature (factor D) on mean S/N ratio is given in Fig.3-6. The mean S/N ratio reaches the maximum at 800℃ (level 1), and then drops at 760℃ (level 2). Then, there is a slightly decrease from 760℃ (level 2) to 720℃ (level 3). The liquid aluminum has a low viscosity and better fluidity, and becomes more competent to react with flux at elevated temperature, which helps separate inclusion from molten alloy. As such, 800℃ (level 1) should be recommended for minimizing the porosity content.

By selecting the highest value of the mean S/N ratio for each factor, the optimal level can be determined. Hence, the optimum combination of the levels in terms of minimizing the porosity content for the present recycling process is A2B2C1D1; i.e., Al-clean 113 as the refining flux; 10:4 as the chips/flux ratio; 60mins as the holding time and 800℃ as the holding temperature.

3.4.3. Factor contributions

The contribution of each factor to the recovery rate can be determined by performing analysis of variance based on Eqs. (3-3) – (3-7). The results of analysis of variance (ANOVA) for case 1 (w_1=1.0, w_2=0) and case 2 (w_1=0.5, w_2=0.5) are summarized in Table 3-7 and Table 3-8, respectively.

Table 3-7 Results of the ANOVA for case 1 (w1=1.0, w2=0)

Factors	Degree of freedom (D)	Sum of squares (SS_p)	Variance (V)	Corrected sums of squares (SS_p')	Contribution	Rank
Flux type	2	0.64	0.32	0.64	54.13%	1
Chips/flux ratio	2	0.29	0.15	0.29	24.75%	2
Holding time	2	0.11	0.06	0.11	9.04%	4
Holding temperature	2	0.14	0.07	0.14	12.08%	3
error		0.00	0.00		0	
Total		1.17			100%	

Table 3-7 shows the contribution of the four factors in case 1, i.e. the flux type, chips/flux ratio, holding time and holding temperature is 54.13%, 24.75%, 9.04% and 12.08%, respectively. Flux type has a contribution of 54.13%, which is higher than the sum of the rest three factors. It has the major influence on the recovery rate. The chips/flux ratio makes medium contribution while holding times and holding temperatures during the refining process both have minor effects on the recovery rate for their contribution percentages are around 10%. Table 3-8 gives the contribution of the four factors in case 2, i.e. the flux type, chips/flux ratio, holding time and holding temperature is 5.32%, 23.91%, 19.28% and 51.49%, respectively. The holding temperature makes a contribution of 51.59%, higher than the sum of the rest three factors, which has the major influence on the porosity content of the recycled alloy. The chips/flux ratio takes the second place with a contribution of 23.91%. The holding time has minor influence on the porosity formation while the flux type has rarely effect on porosity at the contribution of 5.32%.

Table 3-8 Results of the ANOVA for case 2 (w1=0.5, w2=0.5)

Factors	Degree of freedom (D)	Sum of squares (SSp)	Variance (V)	Corrected sums of squares (SSp')	Contribution	Rank
Flux type	2	0.80	0.40	0.80	5.32%	4
Chips/flux ratio	2	3.60	1.80	3.60	23.91%	2
Holding time	2	2.90	1.45	2.90	19.28%	3
Holding temperature	2	7.74	3.87	7.74	51.49%	1
error		0.00	0.00		0.00	
Total		15.04			100%	

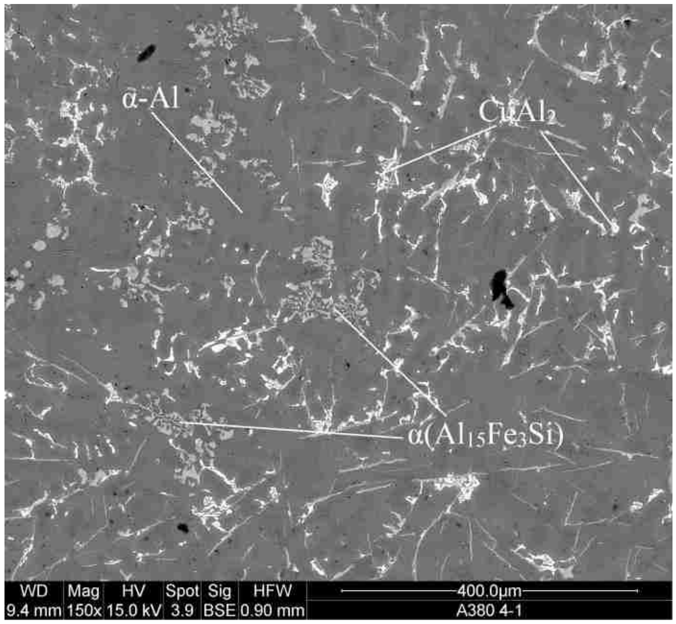
3.4.4. Confirmation run

As the last step of verifying the optimal combinations drawn from the DOE and the above discussion, two individual confirmation experiments were conducted focusing on two optimization response, the recovery rate and the porosity content.

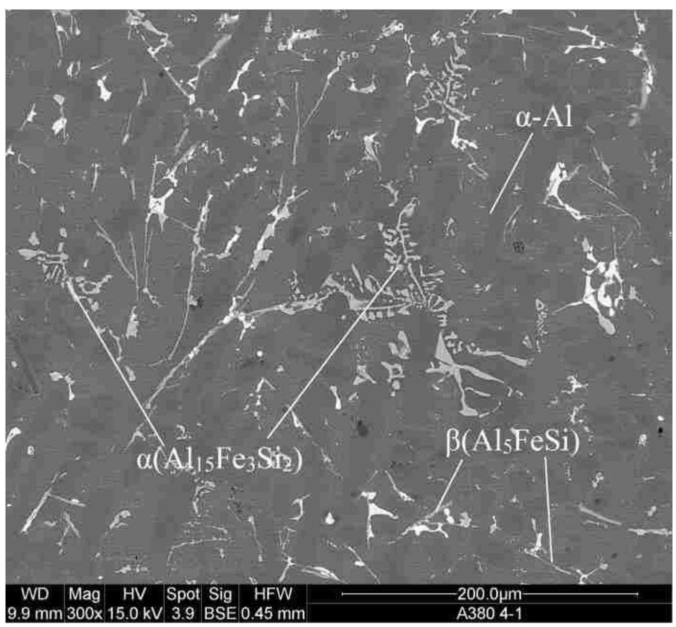
As discussed above, the designed factors A1B3C1D2 are selected as the optimal combination for case 1 (w_1=1.0, w_2=0), experimental conditions are set as: Al-clean 101 for the refining flux; 10:5 for the chips/flux ratio; 60mins for the holding time and 760℃ for the holding temperature. The results from the confirmation experiment show that 276.09 grams of aluminum alloy 380 recovered from 300 grams aluminum chips. Its recovery rate reaches as high as 92.03% with porosity content of 0.87%. The S/N ratio of multi-response of case 1 is calculated as 39.28 using Eqs. (3-4) - (3-9). Which is the highest value comparing with the S/N ratio of multi-response for case 1 in Table 3-4, it verifies the most effective combination of experimental factors and levels as predicted when the metal yield is a major concern.

For case 2 (w_1=0.5, w_2=0.5), the factors A2B2C1D1 are selected as the optimized combination. In this confirmation experiment, refining flux is Al-clean 113; chips/flux ratio is 10:4; the holding time of 60mins is selected; the holding temperature is 800℃. The results show that 241.68 grams of aluminum are recovered with a recovery rate of 80.56% and the porosity content is 0.56%. The S/N ratio of multi-response for the confirmation run is calculated as 21.59 with Eqs. (3-4) – (3-9), which is higher than the S/N ratios of multi-response for case 2 shown in Table 3-4. It verifies A2B2C1D1 is the optimal combination when both the metal yield and the quality of the recovered aluminum were both required.

Also, Fig.3-7(a) and (b) showed the microstructure of recycled aluminum of two confirmation runs. It is evident that the microstructure of the ingot mold-cast recovered aluminum alloy contained α ($Al_{15}Fe_3Si_2$) phase and β (Al_5FeSi) phase, Si phase, $CuAl_2$. The phase observation indicates that the recovered aluminum alloy possessed the same types of phases as those present in the die cast A380 given in references 3 and 11.



(a)



(b)

Figure 3.4-3 SEM micrograph showing microstructure of the recycled aluminum alloy for confirmation runs (a) case 1, (b) case 2.

3.5. Conclusions

The Taguchi method for the design of experiment has been used for optimizing the recycling process for the machining chips of high pressure die cast aluminum alloy A380. Four factors, three levels for each factor, and two objectives were considered in the DOE.

To achieve the maximum recovery rate, the signal-to-noise ratio of HB characteristics was employed to calculate the S/N ratio of recovery rate. To minimize the porosity content, the signal-to-noise ratio of LB characteristics was utilized to calculate the S/N ratio of porosity level. The optimum combinations were worked out based on the S/N ratio of each factor.

For case 1, the metal yield was the only requirement for the recycling process. The optimum combination (A1B3C1D2) was Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, and 60mins as the holding time and 760℃ as the holding temperature. The flux type made the major contribution to recovery rate with the percentage of 54.13%, which was higher than the sum of the rest three factors. The chips/flux ratio made medium contribution while both the holding time and holding temperature during refining process had minor effect on the recovery rate for their low contribution percentages.

For the objectives of case 2 (w_1=0.5, w_2=0.5), Al-clean 113 as the refining flux; 10:4 as the chips/flux ratio; 60mins as the holding time; 800℃ as the holding temperature, A2B2C1D1 was selected as the optimized process parameters. The holding temperature made the major influence with a contribution of 51.49%, and the chips/flux ratio and the holding time had the moderate effects of 23.91% and 19.28%

on the porosity, respectively, while and flux type has minor influence with the contribution of 5.32%.

By comparing case 1 and case 2, it could be seen that the recovery rate dropped significantly from 92.03% to 80.56% when the porosity content of the recovered aluminum was considered. But, the reduction in the porosity content was limited by a small amount from 0.87% to 0.56%.

Acknowledgments

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3.6. References

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4. CHAPTER IV

OPTIMIZATION OF THE ALUMINUM CHIPS RECYCLING PROCESS FOR RECOVERY RATES AND TENSILE PROPERTIES OF A380 ALLOY

In this study, recycling process of aluminum alloy A380 was conducted via Design of Experiment. Taguchi orthogonal array were designed based on flux types, chips/flux ratio, holding times and holding temperatures as four factors while for each factor, three corresponding levels were selected. Recovery rate, tensile strength, elongation at fracture and yield strength was selected as four individual responses to evaluate the effectiveness of the recycling process and the quality of the recycled alloy. Also, S/N ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data for optimization with weighing factors of corresponding responses. For the four individual responses, the rank of effectiveness of factors (factors selected in Taguchi orthogonal array) and the optimum combinations were concluded. For the multi-response with weighing factors, the combination using Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, 60 mins as the holding time and 760℃ as the holding temperature achieved the recycling process effective considering both the recovery rate and tensile properties as objective functions. Examination of microstructure by scanning electron microscopy confirmed the consistency between the recycled alloy and the die-cast counterpart.

4.1. Introduction

Aluminum alloys as a light weight material have been increasingly used in the automotive industry for the past two decades. Among the aluminum usage in each vehicle, almost 35% of automotive aluminum components were manufactured by conventional high pressure die-casting (C-HPDC) processes [1] When C-HPDC components are manufactured, considerable amount of aluminum waste in the forms of scrap, dross, and machining chips are produced as by products. The casting scrap is easily returned to melting; where by most of the metal is recovered and re-utilized in production processes. The study by Gronostajski and Matuszak [2] showed that, in the process of melting aluminum and aluminum alloy chips, on average, 72% aluminum would be recycled after casting. The recovery of aluminum from dross can be achieved at a recovery rate of around 80% by mixing dross [3] and chips [4] with certain types of fluxes. During the recycling of machining chips and melt dross, however, large amount of metal is lost as a result of oxidation, and the costs of labor and energy as well as the expenditure on environmental protection increase the general cost of the process. The chips as a by-product not only bring huge waste, but also could produce pollution to the environment. Also, due to high market demand for cost saving on die castings, the recovery of Al chips becomes critical for die casters.

However, recovery rates of the chips are often unknown to die casting shops since most chips are presently recycled externally and aluminum content in the chips depends on the practice of molten metal processing. Reducing the aluminum loss is the key to optimize the conventional recycling process. There are several influencing factors during the processes, such as flux types, amount of flux, stirring time, protective gas, holding time and holding temperature during melting, pouring temperature, etc., and for each factor, there are quantities alternative levels. To find the optimum process, many combinations of influencing factors and levels need to be experimented.

The Taguchi method uses a special design of orthogonal arrays to study all the designed factors with a minimum of experiments at a relatively low cost. Orthogonality means that factors can be evaluated independently of one another; the effect of one factor does not interfere with the estimation of the influence of another factor [5].

In this study, the Taguchi method for design of experiment (DOE) was used for the optimization of the recycling process for machining chips of high pressure die cast aluminum alloy A380. Since the preliminary results [6] indicates that the recovery rate was primarily determined by several key process parameters such as flux type, chips/flux ratio, holding time and holding temperature during melting, the present design of experiment took into account the influencing extent of each individual process parameter. This consideration led to the selection of those four influencing factors with three different levels. The results of the factor response analysis were used to derive the optimal level combinations. The contribution of each factor was determined by an analysis of variance. The chips collected directly from CNC machines were recycled with refining flux. The recovery rate of the recycled metal was determined based on weight measurements. To ensure the quality of the recycled aluminum, the mechanical properties and microstructure of the recovered aluminum alloy was analyzed.

4.2. Experimental Procedures

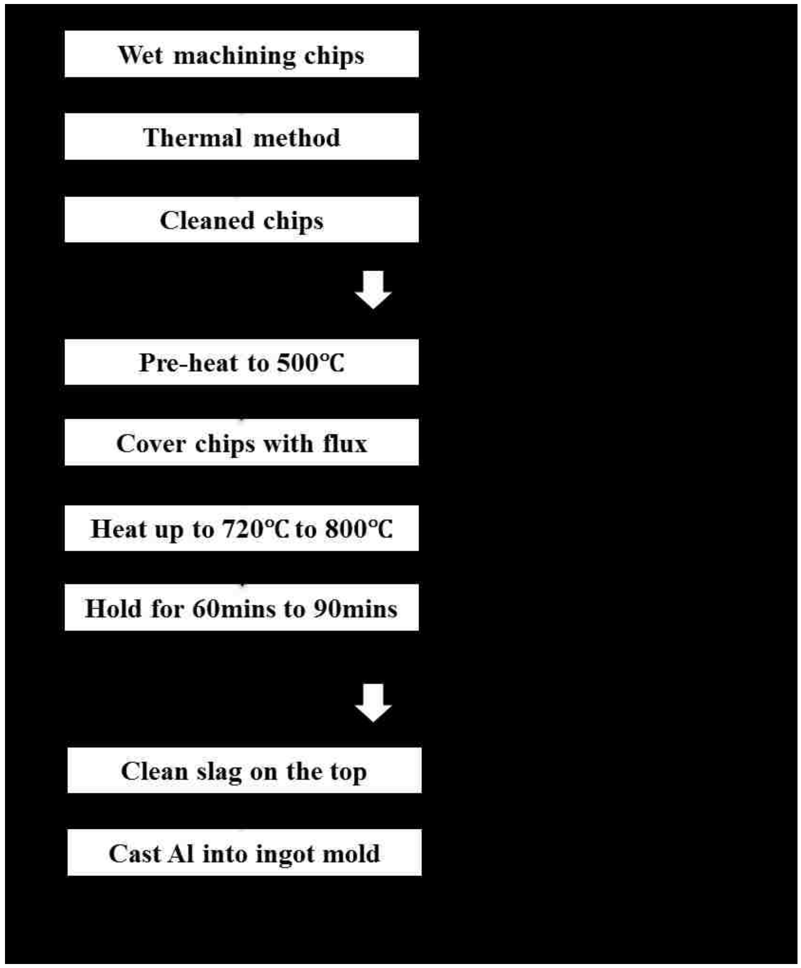
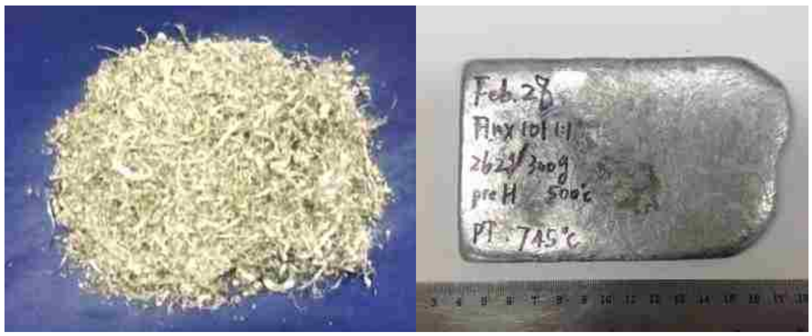


Figure 4.2-1 Flowchart of the recycling process

Fig.4-1 shows the flowchart of the recycling process used in this study. After cleaning, chips were loaded into a crucible and pre-heated to 500°C. Flux types and chips/flux weight ratio were selected as factors A and B in the DOE, respectively. The holding time and holding temperature were chosen as factors C and D.

4.2.1. Materials

Machining chips of high pressure die-cast aluminum alloy 380 shown in Fig.4-2(a) were the raw material to be recycled. The chips were wet and covered with coolants when collected from the CNC machines. Fig.4-2(b) shows one of the recycled aluminum plate.



(a) (b)

Figure 4.2-2 (a) machining chips of aluminum alloy 380, and (b) a cast plate of the recycled alloy.

4.2.2. Cleaning

For safety and health considerations, wet machining chips were cleaned before refining process. Thermal method was employed in this study. Wet machining chips were loaded into a crucible and then, the crucible was heated up to the temperature of 400℃ for 45mins to 60mins in a furnace. With this kind of cleaning method, emulsions and coolant were easily burnt out. Then place those cleaned aluminum chips in a fume hood. Fig.4-3(a) showed a clay graphite crucible and a crucible holder used during cleaning and refining process.

4.2.3. Refining

300 grams of cleaned and dried chips were loaded into a clay-graphite crucible inside an electric resistance furnace. The chips inside the crucible was heated to 500℃ for 20 minutes of preheating to remove any entrapped moisture, and then refining flux was added into the crucible to cover the chips. Three different kinds of fluxes made by Basic Resources Inc. were selected for the purpose of comparison. They were Al-clean 101 [7], Al-clean 113 [8] and Al-clean 116 [9]. Two of them, Al-clean 101 and Al-clean 116 were fluoride-containing flux, and Al-clean 113 was fluoride-free flux. The chips/flux ratio was selected based on DOE. The crucible with chips and flux was held at 500℃ for 20 minutes.

After chips and flux were preheated, the temperature of the furnace was increased to a desired temperature for holding a fixed period of time given by the DOE.

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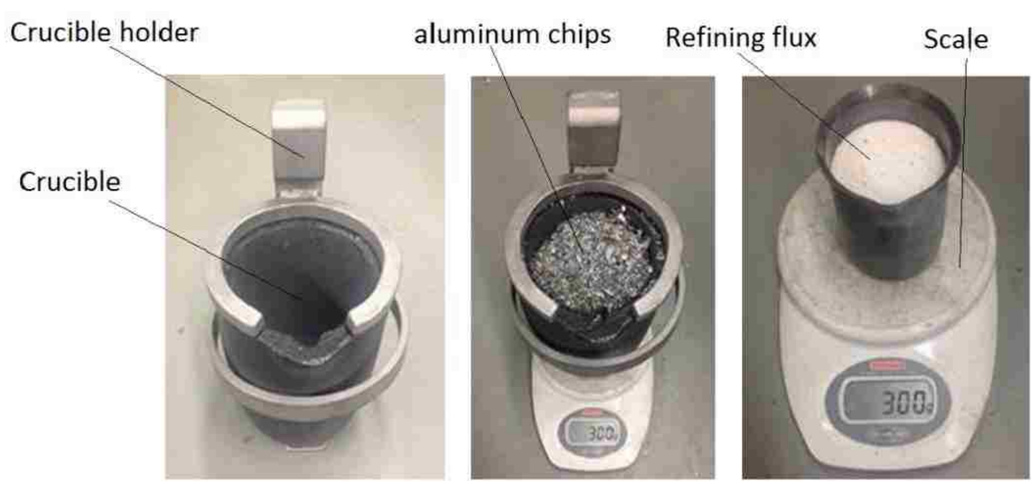


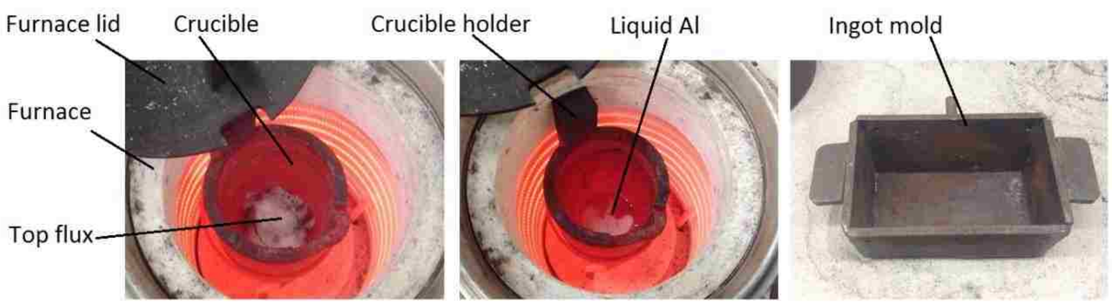
(a) (b) (c)

Figure 4.2-3 (a) crucible and its holder used in cleaning and refining process; (b) aluminum chips loaded into crucible; (c) refining flux.

4.2.4. Melting and casting

The slag floating on top of liquid aluminum was scooped out after the holding process. After removing the slag, the recovered liquid aluminum alloy was poured into an ingot mold and cast as a plate (Fig.4-1(b)). The solidified aluminum plates were quenched in water for analysis.

Fig.4-4(a) showed the melt mixture of the flux and chips in the crucible as the holding temperature reached 800℃, while Fig.4-4(b) depicted the recovered aluminum alloy after slag removal and before casting the alloy into the ingot mold (Fig.4-4(c)).



(a) (b) (c)

Figure 4.2-4 (a) melt mixture of the flux and chips; (b) recovered Al in the crucible; (c) ingot mold.

4.2.5. Recovery rate

Chips were weighed after cleaning and prior to refining experiments, while the recovered aluminum alloy in the form of the cast plate was weighed after refining experiments. The recovery rate of the chips was determined based on the following expression:

$$\text{Recovery rate }(\%) = \frac{\text{weight of recovered Al}}{\text{chips weight}} \times 100 \qquad (4\text{—}1)$$

here the weight of the cleaned and dried aluminum chips was 300 grams for each test of all the nine designed recycling experiments.

4.2.6. Tensile testing

The mechanical properties of the recycled aluminum were evaluated by tensile testing, which was performed at room temperature on a MTS criterion Tensile Test Machine (Model 43) equipped with a data acquisition system. Following ASMT B557 [10], 4 chosen flat tensile specimens (25 mm in gage length, 6 mm in width, and 3 mm in thickness) were machined from each recycled aluminum plate. The tensile properties, including ultimate tensile strength (UTS), 0.2% yield strength (YS), and elongation to failure (Ef) were recorded during the tests. Figs.4-5 and 4-6 show the dimensions of a tensile specimen and the tensile test machine, respectively.

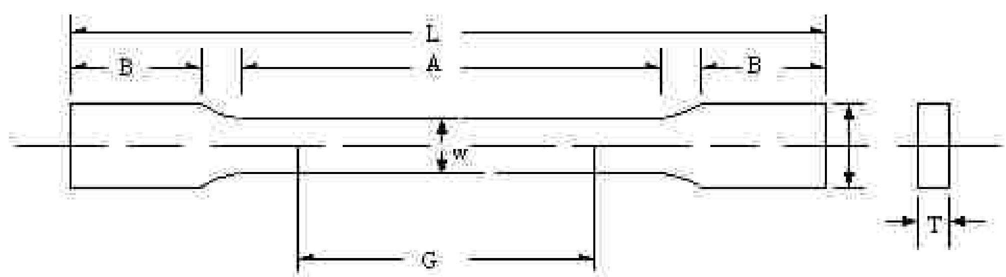


Figure 4.2-5 schematically illustration of Tensile Test Specimen (sub size)

G – Gage length: 25.4 ± 0.1 mm
W – Width: 6 ± 0.1 mm
T – Thickness: 3 mm ± 0.1 mm
R – Radius of fillet, min: 6 mm
L – Overall length, min: 100 mm
A – Length of reduced section: 32 mm
B – Length of grip section, min: 30 mm
C – Width of grip section: 10 mm

Figure 4.2-6 MTS criterion Tensile Test Machine (Model 43)

4.2.7. Microstructure Analysis

Specimens for microstructural analyses were cut from the interior of the components and prepared following the standard metallographic procedures. After proper polishing and etching, microstructural changes were examined on the surface of metallographic specimens obtained from as-cast samples using scanning electron microscopy (SEM) (Fig. 4-7).

Samples for metallographic observation were prepared by the following preparation procedure:

1. Samples were cut into rectangular shape;
2. Mounted with DIALLYL PHTHALATE (Mounting powder);
3. Ground with CARBIMET abrasive papers.

4. Polished with emery paper (to 1200 grades);
5. Fine polishes using 1 μm gamma alumina powder; and
6. Etched with 1% NaOH solution. Performed by submerging the sample into the etchant for about 40 seconds for SEM, rinsing with water and finally cleaned with ethanol specimen surface with running water and ethanol.
7. Specimens for SEM investigation were coated with either gold or carbon before being inserted into the microscope.

Figure 4.2-7 Scanning electron microscopy (FEI Quanta 200 FEG)

4.3. Taguchi design of experiment

4.3.1. Design of orthogonal array

Concluded from the experimental procedures, Table 4-1 gave the parameters selected for specific experimental parameters. Here four factors (flux type, chips/flux ratio, holding temperature and holding time during melting) with three levels were selected shown in Table 4-2. The factors and levels were used to design an orthogonal array L9 (34) for experimentation. Table 4-3 showed the experiment plan for this study, and these 9 experiments were conducted twice for consistency. Since each experiment was repeated once for verification, in total, the eighteen (18) tests were conducted base on the DOE given in Table 1 with four factors and three levels.

Table 4-1 Summary for experimental parameters

Flux type (Al-clean)	Chip/flux ratio	Heating time (mins)	Heating temperature (℃)	Holding time (mins)	Holding temperature (℃)	Stirring time (mins)
101	10:3			60	720	
113	10:4	20	500	75	760	5
116	10:5			90	800	

Table 4-2 Design factors and levels

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	101	10:3	60	800
2	113	10:4	75	760
3	116	10:5	90	720

Table 4-3 Designed experiment plans				
Experiment	A Flux Type (Al-clean)	B Chips/Flux Ratio	C Holding Time (mins)	D Holding Temperature (℃)
1	(1) 101	(1) 10:3	(3) 90	(2) 760
2	(2) 113	(1) 10:3	(1) 60	(1) 800
3	(3) 116	(1) 10:3	(2) 75	(3) 720
4	(1) 101	(2) 10:4	(2) 75	(1) 800
5	(2) 113	(2) 10:4	(3) 90	(3) 720
6	(3) 116	(2) 10:4	(1) 60	(2) 760
7	(1) 101	(3) 10:5	(1) 60	(3) 720
8	(2) 113	(3) 10:5	(2) 75	(2) 760
9	(3) 116	(3) 10:5	(3) 90	(1) 800

4.3.2. Signal-to-noise analysis with multiple characteristics

In process design, it is almost impossible to eliminate all errors caused by the variation of characteristics. An increase in the variance of multiple characteristics lowers the quality reliability of the recycling process. The Taguchi method uses signal-to-noise (S/N) ratio instead of the average value to interpret the trial results data into a value for the evaluation characteristic in the optimum setting analysis. To minimize the influence of the recovery rate and mechanical properties variation on the analysis of experimental data, the signal-to-noise(S/N) ratio was employed, which converted the trial result data into a value for the response to evaluate the recycling process in the optimal setting analysis. The S/N ratio consolidated several repetitions into one value which reflected the amount of variation present. This is because the S/N ratio can reflect both the average and the variation of the quality characteristics.

There are several S/N ratios available depending on the types of characteristics [11]: lower is best (LB), nominal is best (NB), and higher is best (HB). In the present study, recovery rates were treated as a characteristic value. Since the recovery rates of the recycling process were intended to be maximized, the S/N ratio for HB characteristics was selected, which was be calculated as follows:

$$S/N_{HB} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{\eta_{pi}^{2}}\right) \qquad (4—2)$$

where n is the repetition number of each experiment under the same condition for design parameters, andη_{pi} is recovery rate or mechanical properties of an individual measurement at the ith test.

The proposition for the optimization of recycling process with multiple performance characteristics (two objectives) using a weighting method is defined as the Eqs. (4-3) – (4-5):

$$Y_{SUM} = Y_P \times w \qquad (4—3)$$

where

$$Y_{SUM} = \begin{bmatrix}\eta_{1c}\\ \eta_{2c}\\ \vdots\\ \eta_{9c}\end{bmatrix};\quad Y_p = \begin{bmatrix}\eta_{11}\eta_{12}\eta_{13}\eta_{14}\\ \eta_{21}\eta_{22}\eta_{23}\eta_{24}\\ \vdots\quad\vdots\quad\vdots\quad\vdots\\ \eta_{91}\eta_{92}\eta_{93}\eta_{94}\end{bmatrix};\quad w = \begin{bmatrix}w_1\\ w_2\\ w_3\\ w_4\end{bmatrix} \qquad (4—4)$$

$$\sum_{i=1}^{4} w_i = 1 \qquad (4—5)$$

where w_1 ,w_2, $w_3$and w_4 are the weighting factor of recovery rate, yield strength, elongation and tensile strength, respectively. η_{jc} is the multi S/N ratio in the jth test, η_{ji} is the ith single response S/N ratio for the jth test; w_i is the weighting factor in the ith performance characteristics.

The objective function was formulated according to the previous optimization criteria:

$$\text{Maximize } f(X) = w_1 \cdot \eta_{recovery} + w_2 \cdot \eta_{YS} + w_3 \cdot \eta_{Ef} + w_4 \cdot \eta_{UTS} \qquad (4-6)$$

the above objective function is presented in an analytical form as function of input parameters since increased productivity and mechanical properties play the important roles during recycling of machining chips. However, in the actual manufacturing process, for different metal component specifications, the four characters should be considered as different critical roles by weighting factors. In this study, the case w_1= 0.4, w_2=0.2, w_3=0.2 and w_4=0.2 of weighting factors as multi-response were selected to combine the two sides for demonstrating recycling requirements.

4.3.3. Analysis of variance (ANOVA)

The analysis of variance (ANOVA) on the experimental results was performed to evaluate the source of variation during the recycling process. Following the analysis, it was relatively easy to identify the effect order of factors on recovery rate and mechanical properties of the recycled alloys as well as the contribution of factors to corresponding characteristics. In this study, the variation due to both the four factors and the possible error was taken into consideration. The ANOVA was established based on the sum of the square (SS), the degree of freedom (D), the variance (V), and the percentage of the contribution to the total variation (P). The five parameters symbols typically used in ANOVA [11] are described below:

1. Sum of squares (SS). SS_P denotes the sum of squares of factors A, B, C, and D; SS_e denotes the error sum of squares; SS_T denotes the total sum of squares.

The total sum of square SS_T from S/N ratio was calculated as:

$$SS_T = \sum_{i=1}^{m} \eta_i{}^2 - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \qquad (4\text{—}7)$$

where m is the total number of the experiments, and η_i is the factor response at the ith test.

The sum of squares from the tested factors, SS_p, was calculated as:

$$SS_P = \sum_{i=1}^{m} \frac{\left(S_{\eta_{jc}}\right)^2}{t} - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \qquad (4\text{—}8)$$

where m is the number of the tests (m= 9), j the level number of this specific factor p, t is the repetition of each level of the factor p, and $S_{\eta j}$ the sum of the multi-response S/N ratio involving this factor p and level j.

2. Degree of freedom (D). D denotes the number of independent variables. The degree of freedom for each factor (D_P) is the number of its levels minus one. The total degrees of freedom (D_T) are the number of total number of the result data points minus one, i.e. the total number of trials times the number of repetition minus one. And the degree of freedom for the error (D_e) is the number of the total degrees of freedom minus the total of degree of freedom for each factor.

3. Variance (V). Variance is defined as the sum of squares of each trial sum result involved the factor, divided by the degrees of freedom of the factor:

$$Vp\ (\%) = \frac{SS_P}{D_P} \times 100 \qquad (4\text{—}9)$$

4. The corrected sum of squares (SSp). SSp is defined as the sum of squares of factors minus the error variance times the degree of freedom of each factor:

$$SS_P{}' = SS_P - D_P V_e \qquad (4\text{—}10)$$

5. Percentage of the contribution to the total variation (P). Pp denotes the percentage of the total variance of each individual factor:

$$Pp\ (\%) = \frac{SS_P'}{SS_P} \times 100 \qquad (4—11)$$

4.4. Results and Discussion

The recovery rate and mechanical properties were selected as original responses. Analysis for recovery rate, yield strength, elongation and tensile strength were conducted based the S/N ratio. And a set of weighting factors combination was selected in this study for the multi-response S/N ratio calculated from Eqs. (4-3) to (4-6) to evaluate the effectiveness and efficiency of the recycling process and the quality of the recycled plate.

Table 4-3 gives the data of original results. The recovery rates were calculated with Eq. (4-1) using the weight of recycled aluminum. The yield strength, elongation and tensile strength were acquired based on tensile testing. Fig.4-8 shows a typical engineering stress and strain curve of the recycled A380 alloy

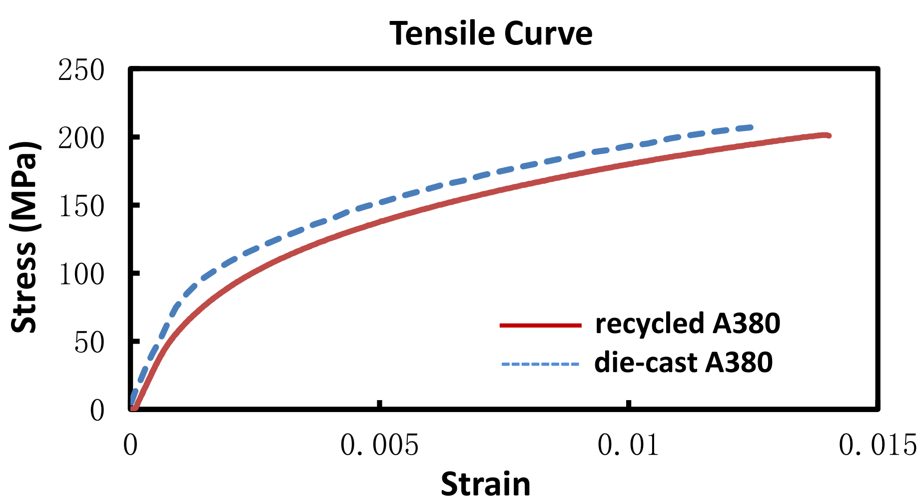


Figure 4.4-1 A typical engineering stress and strain curve of the recycled A380 alloy and die-cast A380 aluminum alloy

Table 4-4 Data of original results								
Experiment	Recovery rate (%)		Yield strength (MPa)		Elongation (%)		Ultimate tensile strength (MPa)	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
1	86.89	85.24	112.2	128.4	1.11	1.78	173.0	221.2
2	76.93	85.39	119.5	113.6	1.34	1.10	201.5	183.4
3	76.09	87.55	114.8	118.0	1.18	1.41	184.3	200.2
4	89.18	90.10	125.8	106.6	1.73	0.96	198.6	156.9
5	78.41	82.38	133.2	111.0	1.49	1.59	203.6	196.7
6	86.45	91.44	137.6	118.8	0.97	1.50	185.5	200.8
7	90.21	89.13	111.5	105.2	1.25	1.76	201.4	201.2
8	82.05	88.50	105.8	113.0	1.53	1.90	177.6	153.4
9	85.95	84.10	103.2	115.3	0.96	1.65	160.7	219.6

Table 4-5 S/N ratio of each response				
Experiment	S/N ratio of recovery rate	S/N ratio of yield strength	S/N ratio of elongation	S/N ratio of ultimate tensile strength
1	38.70	41.55	2.47	45.70
2	38.15	41.32	1.58	45.66
3	38.19	41.31	2.16	45.66
4	39.05	41.22	1.51	44.82
5	38.10	41.63	3.73	46.02
6	38.97	42.09	1.21	45.70
7	39.05	40.69	3.18	46.08
8	38.60	40.77	4.54	44.31
9	38.59	40.73	1.37	45.27

Since the four responses, recovery rate, yield strength (YS), elongation at fracture (E_f) and ultimate tensile strength (UTS) were intended to be maximized, the S/N ratio for HB (higher-is-better) characteristics was employed for all the four responses. The determined S/N ratios of the four responses were given in Table 4-5.

4.4.1. Effect of factors on S/N ratio of four responses

4.4.1.1. Recovery rate

The response of each factor to its individual level was calculated by averaging the S/N ratios of all experiments at each level for each factor. For the recovery rates, factor's mean S/N ratios for each level are summarized in Table 4-6, respectively. For instance, the mean S/N ratio (38.93) for flux type and level 1 was the average value of the S/N ratios of experiment No.1 (38.70), No.4 (39.05) and No.7 (39.05). The Delta value stands for the maximum difference of the S/N ratios for each factor

Table 4-6 S/N ratio of recovery rate

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	38.93	38.35	38.73	38.60
2	38.28	38.71	38.61	38.76
3	38.59	38.75	38.46	38.45
Delta	0.65	0.40	0.27	0.31
rank	1	2	4	3

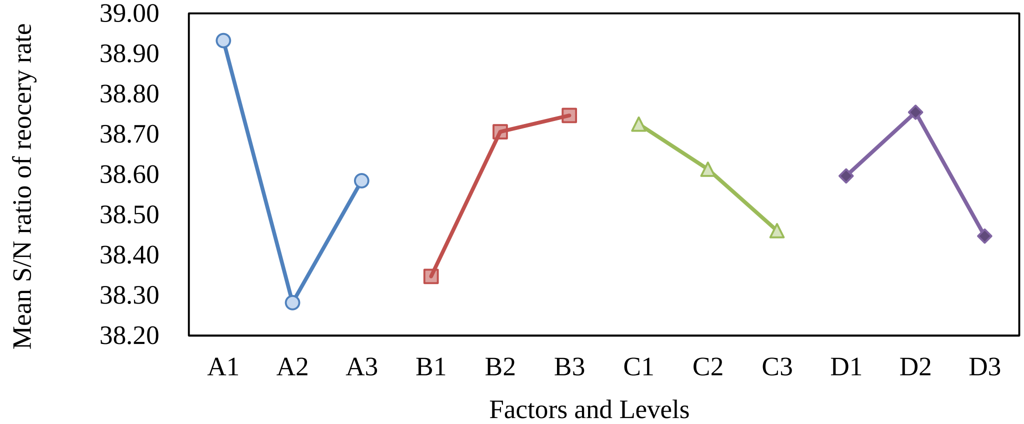


Figure 4.4-2 Effect on signal-to-noise graph for recovery rate

It is shown in Fig.4-9 that the mean S/N ratio of the factor flux type (factor A) reaches maximum using flux Al-clean 101 (level 1), and has the minimum value using flux Al-clean 113 (level 2). As the flux Al-clean 101 has a melting temperature around 500℃ and Al-clean 113 has a melting temperature between 690℃ and 705℃. The flux Al-clean 101 is more easily softened to have larger contact area with aluminum chips to achieve higher effectiveness at the relative low temperature.

The effect of the chips/flux ratio (factor B) on the mean S/N ratio of the recovery rate also plotted in Fig.4-9. The mean S/N ratio of recovery rate grows when additional flux added. It can be seen from the ratio 10:3 (level 1) to the ratio 10:4 (level 2) that the additional flux enhances the recovery rate. This might be because sufficient flux can greatly protect the aluminum chips from being oxidized during melting process. The curve seems to reach a plateau from the ratio 10:4(level 2) to 10:5 (level 3).This observation implied that the excessive amount of flux would result in minor effect on recovery rates. In the viewpoint of cost saving, the ratio 10:4 might be considered for recycling production. While the ratio 10:5 was employed, the recovery rate becomes the highest.

The lines plotted from C1 to C3 are the effect of holding time (factor C) on the mean S/N ratio of the recovery rate. The curve is much smoother without sharp fluctuations comparing to other plots, which means holding time has minor effect on the recovery rate. The mean S/N ratio decreases when extended holding times are employed. However, aluminum chips are more likely to be oxidized when being kept at elevated temperatures for a prolonged period of time. Thus, 60mins (level 1) is selected for its higher S/N ratio response.

The plot points D1 to D3 shows the holding temperature (factor D) on the mean S/N ratio of recovery rate. The mean S/N ratio reaches the peak at 760℃ (level 2), and then drops to720℃ (level 3).The working temperature of the three fluxes is within the temperature range of 700℃ to 800℃. Since the energy consumption for recycling is high and chips are more likely to be oxidized at high temperatures, the medium temperature is preferred.

Fig.4-9 also suggested the factors combination of A1, B3, C1 and D2 will maximize the recovery rate during this recycling process.

4.4.1.2. Yield strength

Table 4-7 listed the S/N ratio for the response yield strength. Fig.4-10 was plotted based on the data of Table 4-7 showed the effect of factors on S/N of yield strength.

From Table 4-7 and Fig.4-10, it can be seen that, among all the factors, the chips/flux ratio (factor B) has the greatest effect on yield strength. Holding temperature (factor D) has less influence, followed by the holding time (factor C) and the flux type (factor A). It also leads to the conclusion that the factor combination of A3, B2, C1, and D2 gives the highest yield strength.

Table 4-7 S/N ratio of yield strength

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	41.15	41.39	41.36	41.09
2	41.24	41.64	41.10	41.47
3	41.37	40.73	41.30	41.21
Delta	0.22	0.92	0.27	0.38
rank	4	1	3	2

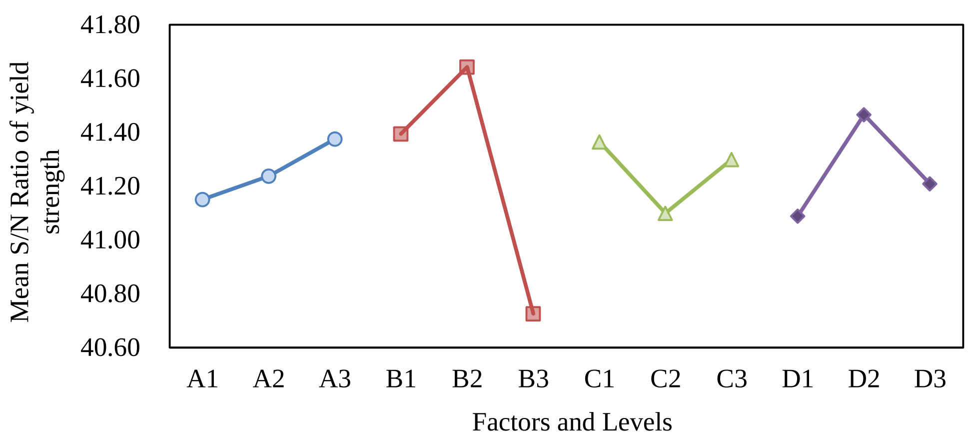


Figure 4.4-3 Effect on signal-to-noise graph for yield strength

4.4.1.3. Elongation

The S/N ratio for the response elongation is included in Table 4-8. Fig.4-11 was plotted based on the responses given in Table 4-8.

From the Fig.4-11 and the Table 4-8, it can be seen, that among all the factors, the flux type is the most significant factor, followed by the holding temperature while the chips/flux ratio and holding time had the least or almost no significance on elongation. The factor combination of A2, B3, C2, and D3 leads to the highest elongation of the recycled alloy.

Table 4-8 S/N ratio of elongation

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	2.39	2.07	1.99	1.49
2	3.29	2.15	2.73	2.74
3	1.58	3.03	2.52	3.02
Delta	1.71	0.96	0.74	1.54
rank	1	3	4	2

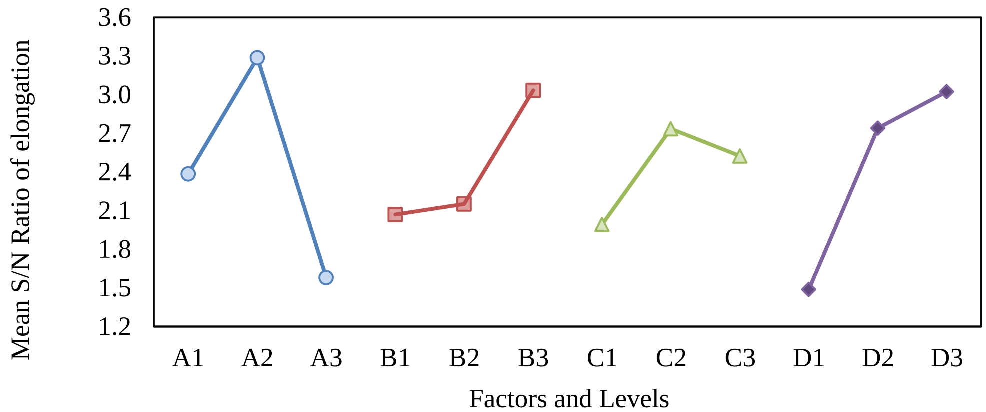


Figure 4.4-4 Effect on signal-to-noise graph for elongation

4.4.1.4. Tensile strength

Table 4-9 showed the S/N ratio for the response tensile strength. Fig.4-12 showed the effect of factors on S/N of tensile strength.

The rank in Table 4-9 indicates that the holding time during refining process exhibits the most significant influence on the tensile strength of the recovered alloy, the holding temperature has less effect followed by the chips/flux ratio, and flux type has little influence on tensile strength.

By selecting the highest value of the mean S/N ratio for each factor, the optimal level can be determined as A3, B1, C1 and D3 to obtain the highest tensile strength according to Table 4-9 and Fig.4-10.

Table 4-9 S/N ratio of tensile strength

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	45.53	45.67	45.81	45.25
2	45.33	45.51	44.93	45.23
3	45.54	45.22	45.66	45.92
Delta	0.21	0.45	0.89	0.68
rank	4	3	1	2

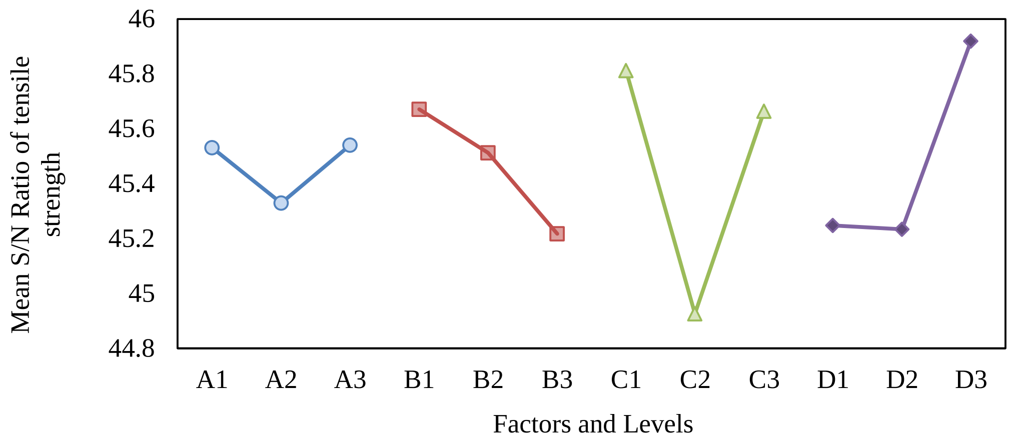


Figure 4.4-5 Effect on signal-to-noise graph for tensile strength

4.4.2. Effect of factors on S/N ratio of multi-response

The recovery rate and tensile properties were selected as responses to be investigated. A combination of weighting factors (w_1= 0.4, w_2=0.2, w_3=0.2 and w_4=0.2) were selected in this study for the multi-response S/N ratio calculated from Eqs. (4-3) to (4-6) to evaluate the effectiveness and efficiency of the recycling process and the quality of the recycled plates for different requirements.

Table 4-10 lists the S/N ratio of the multi-response, and Fig.4-13 shows the mean S/N ratio of the multi-response.

Table 4-10 S/N ratio of multi-response

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	33.39	33.17	33.32	33.00
2	33.28	33.34	33.20	33.39
3	33.13	33.29	33.28	33.41
Delta	0.25	0.18	0.13	0.41
rank	2	3	4	1

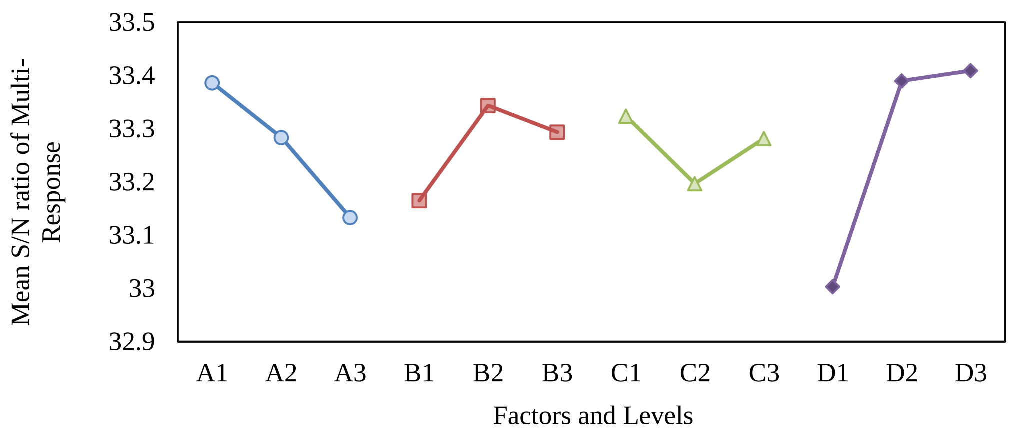


Figure 4.4-6 signal-to-noise graph of Multi-response

Fig.4-13 shows the effect of flux type (factor A) on the mean S/N ratio of both the recovery rate and tensile properties. The curve of factor A decreases from level 1 to level 3. It can be seen that the flux Al-clean 101 (level 1) have advantages over Al-clean 113 (level 2) and Al-clean 116 (level 3).

The plot of the chips/flux ratio (factor B) vs. the mean S/N ratio shown in Fig.4-13 starts with a low S/N ratio at the chips/flux ratio 10:3 (level 1) and reaches its peak at 10:4 (level 2), then decreases to the ratio 10:5 (level 3). It can be concluded that suitable amount of flux has the ability to reduce impurities and inclusions trapped in molten aluminum. The chips/flux ratio 10:3 (level 1) is insufficient to eliminate impurities while chips/flux ratio 10:5 (level 3) might introduce excessive foreign particles causing impurities and inclusion issues during refining and casting.

Fig.4-13 also shows the effect of the holding time (factor C) on the mean S/N ratio of multi-response. The mean S/N ratios are low at the holding time of 75mins (level 2), and the maximum value is at 60mins (level 1). And using 90mins (level 3) has similar but less effectiveness than 60mins. For the view of cost saving, the holding time of 60 mins is the best choice.

The effect of the holding temperature (factor D) on the mean S/N ratio is given in Fig.4-13. The mean S/N ratio reaches the maximum at 720℃ (level 3), and has similar effect at 760℃ (level 2). But the elevated temperature has limited effect on the mean S/N ratio.

By selecting the highest value of the mean S/N ratio for each factor, the optimal level can be determined. Hence, the optimum combination of the levels in terms of minimizing the porosity content for the present recycling process is A1, B2, C1 and D3; i.e., Al-clean 101 as the refining flux; 10:4 as the chips/flux ratio; 60mins as the holding time and 720℃ as the holding temperature.

4.4.3. Factor contributions

The contribution of each factor to the recovery rate can be determined by performing analysis of variance based on Eqs. (4-7) – (4-11). The results of analysis of variance (ANOVA) for the multi-response are summarized in Table 4-11.

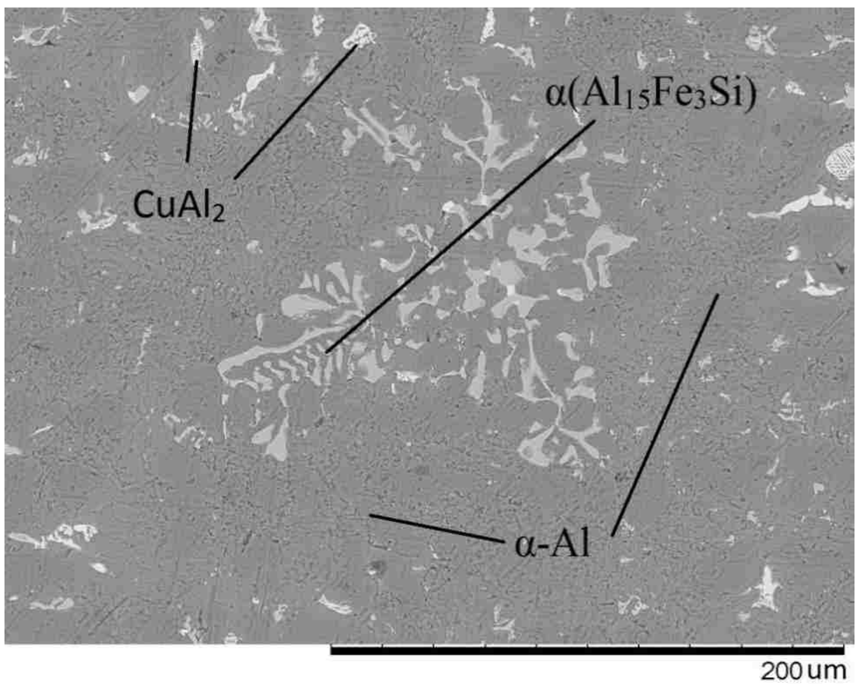
Table 4-11 Results of the ANOVA for multi-response

Factors	Degree of freedom (D)	Sum of squares (SS_p)	Variance (V)	Corrected sums of squares (SS_p')	Contribution	Rank
Flux type	2	0.10	0.05	0.10	19.95 %	2
Chips/flux ratio	2	0.05	0.03	0.05	10.42%	3
Holding time	2	0.02	0.01	0.02	5.12%	4
Holding temperature	2	0.31	0.16	0.31	64.51%	1
error		0.00	0.00		0	
Total		0.48			100%	

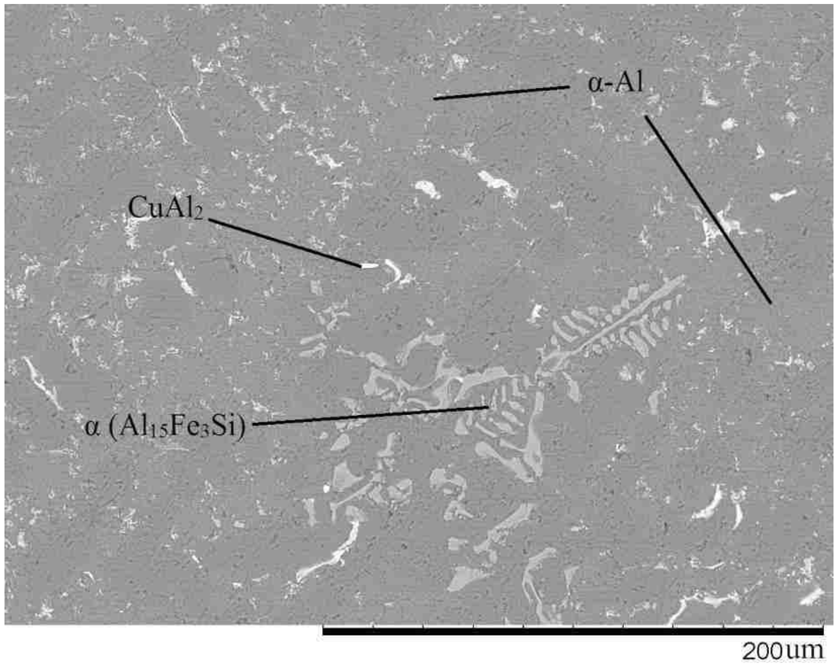
The results listed in Table 4-11 reveals the contribution of the four factors, i.e. the flux type, chips/flux ratio, holding time and holding temperature is 19.95%, 10.42%, 5.12% and 64.51%, respectively. The holding temperature has a contribution of 64.51 which is higher than the sum of the rest three factors. It has the major influence on the recovery rate and tensile properties. The flux type and chips/flux ratio made medium contribution while the holding time during the refining process both had minor effects on the recovery rate and tensile properties for their contribution percentages are around 10%.

4.4.4. Microstructure

Also, Fig.4-14 shows the microstructure of recycled aluminum of two confirmation runs. It is evident that the microstructure of the ingot mold-cast recovered aluminum alloy contained α ($Al_{15}Fe_3Si_2$) phase and β (Al_5FeSi) phase, Si phase, $CuAl_2$. The phase observation indicates that the recovered aluminum alloy possessed the same types of phases as those present in the die cast A380 given in references 3 and 12.



(a)



(b)

Figure 4.4-7 SEM micrograph showing microstructure of the recycled aluminum: (a) sample 2 test 8 (b) sample 3 test 4

4.5. Conclusions

The Taguchi method for the design of experiment has been used for optimizing the recycling process for the machining chips of high pressure die cast aluminum alloy A380. Four factors, three levels for each factor were designed based on Taguchi method. To achieve the maximum recovery rate and tensile properties, the signal-to-noise ratio of HB characteristics was employed to calculate the S/N ratio of recovery rate, yield strength, elongation and tensile strength. The optimum combinations were worked out based on the S/N ratio of each factor.

For each individual response, the optimum combinations were A1, B3, C1 and D2 for the highest recovery rate; A3, B2, C1, and D2 for the best yield strength; A2, B3, C2, and D3 for the highest elongation; A3, B1, C1 and D3 for the highest tensile strength.

For the multi-response objective, weighing factors were selected as w_1= 0.4, w_2=0.2, w_3=0.2 and w_4=0.2. The optimum combination (A1B3C1D2) was Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, and 60 mins as the holding time and 760℃ as the holding temperature. The holding temperature made the major contribution with the percentage of 64.51%, which was higher than the sum of the rest three factors. The chips/flux ratio made medium contribution while both the holding time and holding temperature during refining process had minor effect on the recovery rate and tensile properties for their low contribution percentages.

The microstructure of the recovered aluminum alloys contained the primary α-Al, Si phase, $CuAl_2$, Fe containing inter-metallic phases, which were almost the same as those present in the die-cast A380.

Acknowledgments

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5. CHAPTER V

OPTIMIZATION OF ALUMINUM CHIPS RECYCLING PROCESS FOR CORROSION RESISTANCE OF ALUMINUM ALLOY A380

To optimize recycling process, Design of Experiment (DOE) was utilized. In this study, Taguchi orthogonal array were designed based on four factors as flux types, chips/flux ratio, holding times and holding temperatures, and for each factor, three corresponding levels were also chosen. Recovery rate and corrosion resistance were selected as two individual responses to evaluate the effectiveness of the recycling process and the quality of the recycled alloy. Also, S/N ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data for optimization. Two sets of weighing factors were selected for the responses of recovery rate and corrosion resistance, respectively, for different requirement of the recycled alloy. The optimum combinations led to the highest recovery rate of by using Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, 60 minutes as the holding time and 760℃ as the holding temperature, while the combination using Al-clean 113 as the refining flux, 10:3 as the chips/flux ratio, 90 minutes as the holding time and 800℃ as the holding temperature made the recycling process effective considering both the recovery rate and corrosion resistance as objective functions.

5.1. Introduction

Aluminum alloys have been increasingly used in automotive industry. Among the aluminum usage in each vehicle, almost 35% of automotive aluminum components were manufactured by conventional high pressure die-casting (C-HPDC) processes [1]. C-HPDC components are manufactured along with considerable amount of aluminum waste in the forms of scrap, dross, and machining chips. The casting scrap is easily returned to melting; where by most of the metal is recovered and re-utilized in production processes. The study by Gronostajski and Matuszak [2] showed that, in the process of melting aluminum and aluminum alloy chips, on average, 10% of the metal was burnt and about 10% was lost because dross formed by mixing molten aluminum and slag were removed from the surface of liquid aluminum in the ladle. Also considering 8% loss of casting scraps, 72% aluminum would be recycled after casting. Thus the anticipated recovery rate of conventional recycling processes was around 72%. During the recycling of machining chips and melt dross, large amount of metal is lost as a result of oxidation, and the costs of labor and energy as well as the expenditure on environmental protection increase the general cost of the process. The chips as a by-product not only bring huge waste, but also could produce pollution to the environment. Also, due to high market demand for cost saving on die castings, the recovery of Al chips becomes critical for die casters.

However, recovery rates of the chips are often unknown to die casting shops since most chips are presently recycled externally and aluminum content in the chips depends on the practice of molten metal processing. Reducing the aluminum loss is the key to optimize the conventional recycling process. There are several influencing factors during the processes, such as flux types, amount of flux, stirring time, protective gas, holding time and holding temperature during melting, pouring temperature, etc., and for each factor, there are quantities alternative levels. To find the optimum process, many combinations of influencing factors and levels need to be experimented.

The Taguchi method uses a special design of orthogonal arrays to study all the designed factors with a minimum of experiments at a relatively low cost. Orthogonality means that factors can be evaluated independently of one another; the effect of one factor does not interfere with the estimation of the influence of another factor [3].

In this study, the Taguchi method for design of experiment (DOE) was used for the optimization of the recycling process for machining chips of high pressure die cast aluminum alloy A380. Since the preliminary results [4] indicates that the recovery rate was primarily determined by several key process parameters such as flux type, chips/flux ratio, holding time and holding temperature during melting, the present design of experiment took into account the influencing extent of each individual process parameter. This consideration led to the selection of those four influencing factors with three different levels. The results of the factor response analysis were used to derive the optimal level combinations. The contribution of each factor was determined by an analysis of variance. The chips collected directly from CNC machines were recycled with refining flux. The recovery rate of the recycled metal was determined based on weight measurements. To ensure the engineering performance of the recycled aluminum, corrosion behavior of the recovered aluminum alloy was analyzed.

5.2. Experimental Procedures

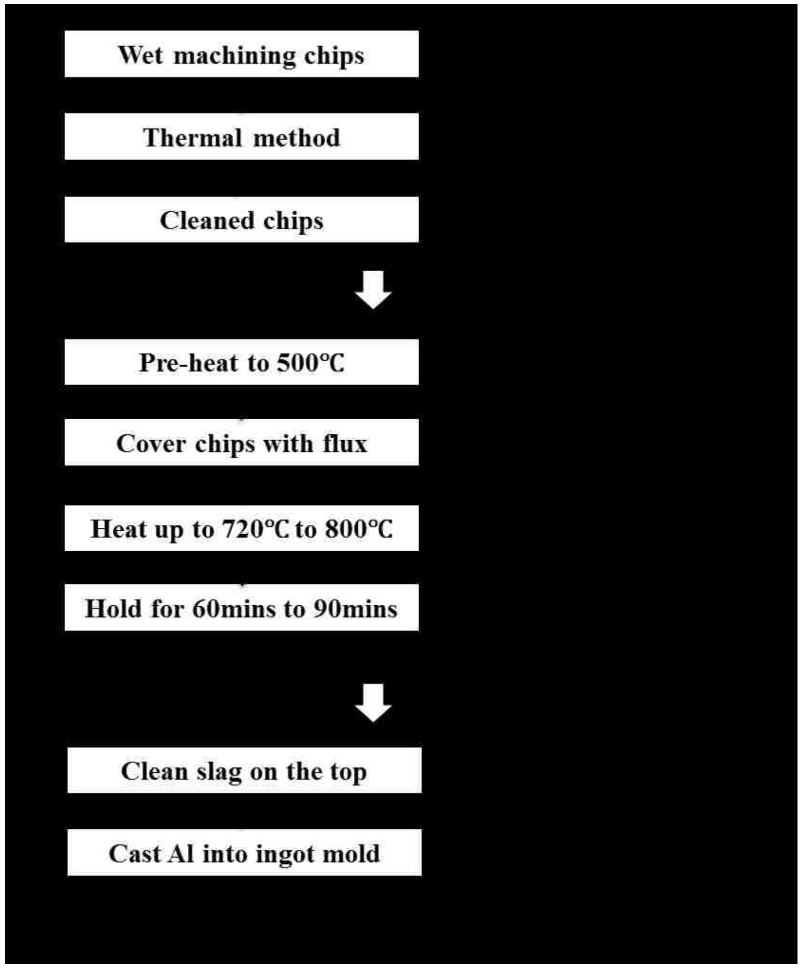
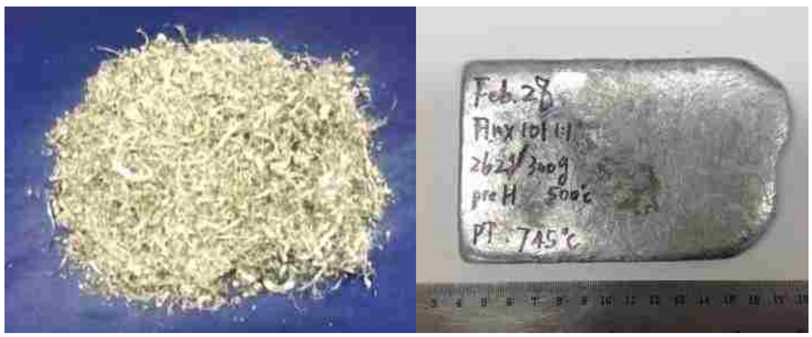


Figure 5.2-1 Flowchart of the recycling process

Fig.5-1 shows the flowchart of the recycling process used in this study. After cleaning, chips were loaded into a crucible and pre-heated to 500℃. Flux types and chips/flux weight ratio were selected as factors A and B in the DOE, respectively. The holding time and holding temperature were chosen as factors C and D.

5.2.1. Materials

Machining chips of high pressure die-cast aluminum alloy 380 shown in Fig. 5-2-(a) were the raw material to be recycled. The chips were wet and covered with coolants when collected from the CNC machines. Fig.5-2-(b) shows one of the recycled aluminum plate.



(a) (b)

Figure 5.2-2 (a) machining chips of aluminum alloy 380, and (b) a cast plate of the recycled alloy.

5.2.2. Cleaning

For safety and health considerations, wet machining chips were cleaned before refining process. Thermal method was employed in this study. Wet machining chips were loaded into a crucible and then, the crucible was heated up to the temperature of 400℃ for 45mins to 60 mins in a furnace. With this kind of cleaning method, emulsions and coolant were easily burnt out. Then place those cleaned aluminum chips in a fume hood. Fig.5-3(a) shows a clay graphite crucible and a crucible holder used during the cleaning and refining process.

800℃, while Fig.5-4(b) depicted the recovered aluminum alloy after slag removal and before casting the alloy into the ingot mold (Fig.5-4(c)).

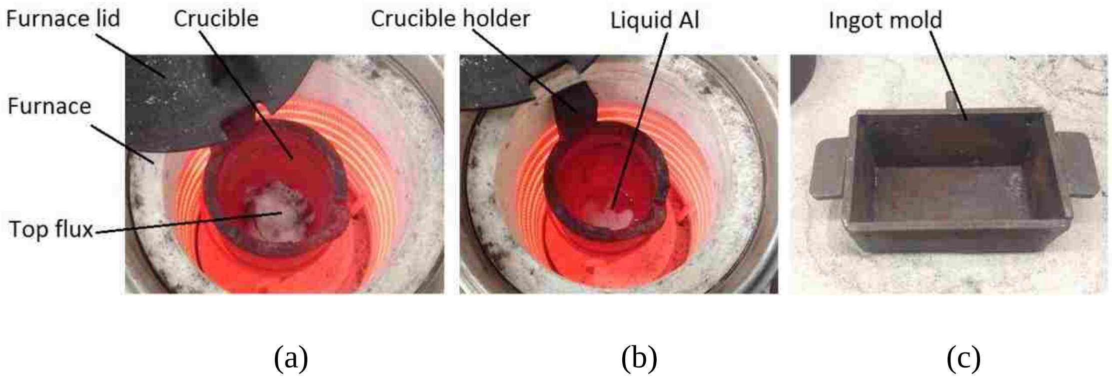


Figure 5.2-3 (a) melt mixture of the flux and chips; (b) recovered Al in the crucible; (c) ingot mold.

5.2.3. Recovery rate

Chips were weighed after cleaning and prior to refining experiments, while the recovered aluminum alloy in the form of the cast plate was weighed after refining experiments. The recovery rate of the chips was determined based on the following expression:

$$Recovery\ rate\ (\%) = \frac{weight\ of\ recovered\ Al}{chips\ weight} \times 100 \qquad (5\text{—}1)$$

here the weight of the cleaned and dried aluminum chips was 300 grams for each test of all the nine designed recycling experiments.

5.2.4. Polarization testing

Specimens for corrosion testing were cut from the tensile bar and prepared following the standard metallographic procedures. Samples were cut into rectangular shape; Polished with emery paper (to 800 grades); 1.0% NaCl solution was used in the polarization test. Fig.5-5 shows the schematic of the polarization testing equipment. Turn on the potentiostat and the software when the equipment was set. After the electrochemical testing system became stable (about 10 min), scans were conducted at a rate of 1 mV/s from –0.15 V versus open circuit potential in a more noble direction up to –0.75 V versus the reference electrode for recycled aluminum.

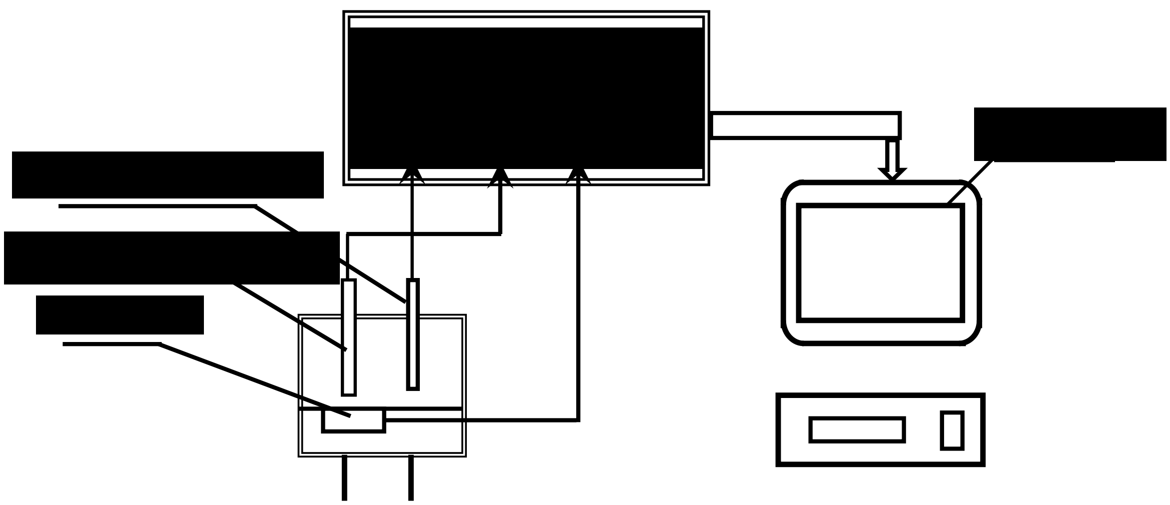


Figure 5.2-4 Schematic diagram of the polarization testing equipment CE: counter electrode, RE: reference electrode, WE: working electrode.

The calculation of the corrosion resistance of samples is based on the corrosion potential, the corrosion current density, and the anodic/cathodic Tafel slopes (β_a and β_c) which were derived from the measured polarization curves. Based on the approximately linear polarization at the corrosion potential (Ecorr), the value of corrosion resistance (Rp) was determined from the relationship [8,9]:

$$Rp = \frac{\beta_a \beta_c}{2.3 i_{corr}(\beta_a + \beta_c)} \qquad (5-2)$$

where i_{corr} is the corrosion current density.

5.3. Taguchi design of experiment

5.3.1. Design of orthogonal array

Table 5-1 Summary for experimental parameters

Flux type (Al-clean)	Chips/flux ratio	Heating time (mins)	Heating temperature (℃)	Holding time (mins)	Holding temperature (℃)	Stirring time (mins)
101	10:3			60	720	
113	10:4	20	500	75	760	5
116	10:5			90	800	

Table 5-2 Design factors and levels

Level	Factors			
	A Flux type (Al-clean)	B Chips/flux ratio	C Holding time (mins)	D Holding temperature (℃)
1	101	10:3	60	800
2	113	10:4	75	760
3	116	10:5	90	720

Concluded from the experimental procedures, Table 5-1 gives the parameters selected for specific experimental parameters. Here four factors (flux type, chips/flux ratio, holding temperature and holding time during melting) with three levels were selected shown in Table 5-2. The factors and levels were used to design an orthogonal array L_9 (3^4) for experimentation. Table 5-3 presents the experiment plan for this study, and this 9 experiments were conducted twice for consistency. Since each experiment was repeated once for verification, in total, the eighteen (18) tests were conducted base on the DOE given in Table 5-2 with four factors and three levels.

Table 5-3 Designed experiment plans

Experiment	A Flux Type (Al-clean)	B Chips/Flux Ratio	C Holding Time (mins)	D Holding Temperature (℃)
1	(1) 101	(1) 10:3	(3) 90	(2) 760
2	(2) 113	(1) 10:3	(1) 60	(1) 800
3	(3) 116	(1) 10:3	(2) 75	(3) 720
4	(1) 101	(2) 10:4	(2) 75	(1) 800
5	(2) 113	(2) 10:4	(3) 90	(3) 720
6	(3) 116	(2) 10:4	(1) 60	(2) 760
7	(1) 101	(3) 10:5	(1) 60	(3) 720
8	(2) 113	(3) 10:5	(2) 75	(2) 760
9	(3) 116	(3) 10:5	(3) 90	(1) 800

5.3.2. Signal-to-noise analysis with multiple characteristics

In process design, it is almost impossible to eliminate all errors caused by the variation of characteristics. An increase in the variance of multiple characteristics lowers the quality reliability of the recycling process. The Taguchi method uses signal-to-noise (S/N) ratio instead of the average value to interpret the trial results data into a value for the evaluation characteristic in the optimum setting analysis. To minimize the influence of the recovery rate and corrosion resistance variation on the analysis of experimental data, the signal-to-noise(S/N) ratio was employed, which converted the trial result data into a value for the response to evaluate the recycling process in the optimal setting analysis. The S/N ratio consolidated several repetitions into one value which reflected the amount of variation present. This is because the S/N ratio can reflect both the average and the variation of the quality characteristics. There are several S/N ratios available depending on the types of characteristics [10]: lower is best (LB), nominal is best (NB), and higher is best (HB). In the present study, recovery rates were treated as a characteristic value. Since the recovery rates of the recycling process were intended to be maximized, the S/N ratio for HB characteristics was selected, which was be calculated as follows:

$$S/N_{HB} = -10\,log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{\eta_{pi}^{2}}\right) \qquad (5\text{—}3)$$

where n is the repetition number of each experiment under the same condition for design parameters, andη_{pi} is recovery rate or corrosion resistance of an individual measurement at the ith test.

The proposition for the optimization of recycling process with multiple performance characteristics (two objectives) using a weighting method is defined as the Eqs. (5-4) – (5-6):

$$Y_{SUM} = Y_P \times w \tag{5—4}$$

where

$$Y_{SUM} = \begin{bmatrix} \eta_{1c} \\ \eta_{2c} \\ \vdots \\ \eta_{9c} \end{bmatrix}; \quad Y_p = \begin{bmatrix} \eta_{11}\eta_{12} \\ \eta_{21}\eta_{22} \\ \vdots \quad \vdots \\ \eta_{91}\eta_{92} \end{bmatrix}; \quad w = \begin{bmatrix} w_1 \\ \\ \\ w_2 \end{bmatrix} \tag{5—5}$$

$$\sum_{i=1}^{2} w_i = 1 \tag{5—6}$$

where w_1 and w_2 are the weighting factor of recovery rate and corrosion resistance, respectively. η_{jc} is the multi S/N ratio in the jth test, η_{ji} is the ith single response S/N ratio for the j th test; w_i is the weighting factor in the i th performance characteristics.

The objective function was formulated according to the previous optimization criteria:

$$Maximize\ f(X) = w_1 \cdot \eta_{recovery} + w_2 \cdot \eta_{corrosion} \tag{5—7}$$

the above objective function is presented in an analytical form as function of input parameters since increased productivity and corrosion resistance play the important roles during recycling of machining chips. However, in the actual manufacturing process, for different metal specifications, the two characters should be considered as different critical roles by weighting factors. When quality demand becomes critical, high weighting factors of corrosion resistance needs to be considered. For metal yield requirement, high recoveries require due to the consideration of cost saving. In this study, case 1 (1.0, 0), and case 2 (0.5, 0.5) with two different combinations of weighting factors were selected for demonstrating recycling requirements.

5.3.3. Analysis of variance (ANOVA)

The analysis of variance (ANOVA) on the experimental results was performed to evaluate the source of variation during the recycling process. Following the analysis, it was relatively easy to identify the effect order of factors on recovery rate and corrosion resistance of the recycled alloys as well as the contribution of factors to corresponding characteristics. In this study, the variation due to both the four factors and the possible error was taken into consideration. The ANOVA was established based on the sum of the square (SS), the degree of freedom (D), the variance (V), and the percentage of the contribution to the total variation (P). The five parameters symbols typically used in ANOVA [10] are described below:

1. Sum of squares (SS). SS_P denotes the sum of squares of factors A, B, C, and D; SS_e denotes the error sum of squares; SS_T denotes the total sum of squares.

The total sum of square SS_T from S/N ratio was calculated as:

$$SS_T = \sum_{i=1}^{m} {\eta_i}^2 - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \qquad (5-8)$$

where m is the total number of the experiments, and η_i is the factor response at the ith test.

The sum of squares from the tested factors, SS_p, was calculated as:

$$SS_P = \sum_{i=1}^{m} \frac{\left(S_{\eta_{jc}}\right)^2}{t} - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \qquad (5-9)$$

where m is the number of the tests (m= 9), j the level number of this specific factor p, t is the repetition of each level of the factor p, and $S_{\eta j}$ the sum of the multi-response S/N ratio involving this factor p and level j.

2. Degree of freedom (D). D denotes the number of independent variables. The degree of freedom for each factor (D_P) is the number of its levels minus one. The total degrees of freedom (D_T) are the number of total number of the result data points minus one, i.e. the total number of trials times the number of repetition minus one. And the degree of freedom for the error (D_e) is the number of the total degrees of freedom minus the total of degree of freedom for each factor.

3. Variance (V). Variance is defined as the sum of squares of each trial sum result involved the factor, divided by the degrees of freedom of the factor:

$$\text{Vp } (\%) = \frac{SS_P}{D_P} \times 100 \qquad (5\text{—}10)$$

4. The corrected sum of squares (SSp). SSp is defined as the sum of squares of factors minus the error variance times the degree of freedom of each factor:

$$SS_P{}' = SS_P - D_P V_e \qquad (5\text{—}11)$$

5. Percentage of the contribution to the total variation (P). Pp denotes the percentage of the total variance of each individual factor:

$$\text{Pp } (\%) = \frac{SS_P{}'}{SS_P} \times 100 \qquad (5\text{—}12)$$

5.4. Results and Discussion

5.4.1. Multi-response of S/N ratios

The recovery rate and corrosion resistance were selected as two original responses. Two combinations of weighting factors selected in this study for the multi-response S/N ratio were calculated from Eqs. (5-4) to (5-7) to evaluate the effectiveness and efficiency of the recycling process and the quality of the recycled plates for different engineering requirements. Fig.5-6 showed typical corrosion curve of recycled aluminum and die-cast A380 alloy.

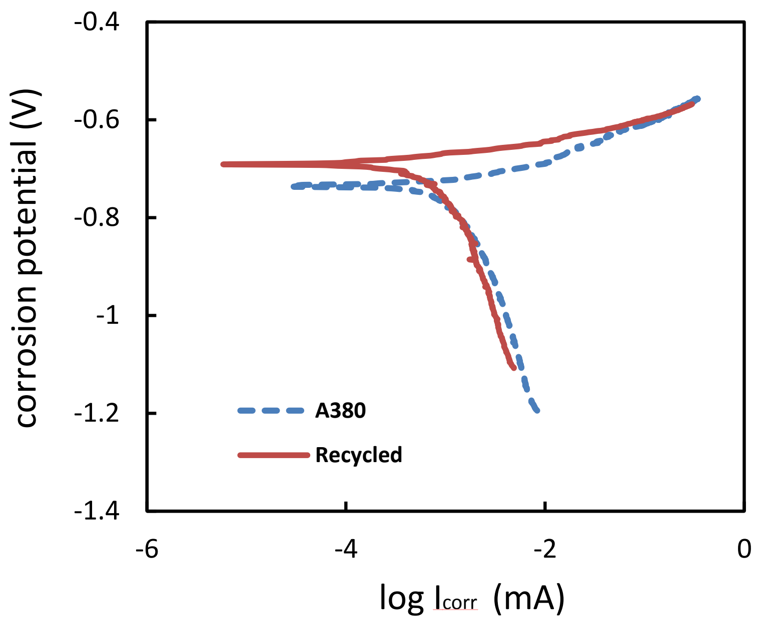


Figure 5.4-1 typical potentiodynamic polarization curve of experiment No.6

Tables 5-4 and 5-5 give the original data of recovery rate and corrosion testing results. The recovery rate was calculated with Eq. (5-1) using the weight of recycled aluminum. The corrosion resistance was calculated with Eq. (5-2).

Table 5-4 Data of original results of recovery rate

Experiment	Recycled Aluminum(g)		Recovery rate (%)	
	Test 1	Test 2	Test 1	Test 2
1	260.68	255.71	86.89	85.24
2	230.80	256.18	76.93	85.39
3	228.27	262.66	76.09	87.55
4	267.53	270.31	89.18	90.10
5	235.23	247.15	78.41	82.38
6	259.36	274.32	86.45	91.44
7	270.63	267.40	90.21	89.13
8	246.14	265.50	82.05	88.50
9	257.85	252.31	85.95	84.10

Table 5-5 Data of original results of corrosion resistance

Experiment	β_a (mV)		β_c(mV)		i_{corr} (μA)		Corrosion resistance (Ω)	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
1	0.050	0.041	0.327	0.352	0.383	0.425	49.232	37.531
2	0.033	0.038	0.389	0.397	0.267	0.356	49.535	42.312
3	0.040	0.036	0.506	0.456	0.593	0.692	27.179	20.961
4	0.030	0.039	0.495	0.521	0.442	0.705	27.824	22.361
5	0.052	0.048	0.557	0.595	0.439	0.651	47.103	29.650
6	0.030	0.032	0.575	0.410	0.914	0.675	13.563	19.126
7	0.027	0.030	0.523	0.478	0.629	0.484	17.747	25.362
8	0.041	0.030	0.489	0.502	0.784	0.663	20.974	18.562
9	0.037	0.039	0.600	0.589	0.652	0.771	23.240	20.623

Table 5-6 S/N ratio of multi-response objectives

Experiment	S/N ratio of Recovery rate	S/N ratio of Corrosion resistance	S/N ratio of Multi-response	
			case 1 (w_1=1.0, w_2=0)	case 2 (w_1=0.5, w_2=0.5)
1	38.70	32.51	38.70	35.60
2	38.15	33.16	38.15	35.66
3	38.19	27.41	38.19	32.80
4	39.05	27.84	39.05	33.44
5	38.10	31.00	38.10	34.55
6	38.97	23.89	38.97	31.43
7	39.05	26.26	39.05	32.66
8	38.60	25.87	38.60	32.23
9	38.59	26.77	38.59	32.68

Since the objectives, i.e., recovery rate and corrosion resistance was intended to be maximized; the S/N ratio for HB (higher-is-better) characteristics was used. The S/N ratio of these two responses was given in Table 5-6. The multi-responses of S/N ratio using two weighting factor combinations were also concluded in Table 5-6. The response of each factor to its individual level was calculated by averaging the S/N ratios of all experiments at each level for each factor.

5.4.2. Optimal recycling factors

With combinations of weighting factors, the factor's mean multi-response S/N ratios for each level are summarized in Table 5-7. For instance, the mean S/N ratio (38.93) for flux type and level 1 was the average value of the S/N ratios of experiment No.1 (38.70), No.4 (39.05) and No.7 (39.05) listed in Table 5-6.

The mean S/N ratio of the recovery rate was influenced by four factors, the flux type, chips/flux ratio, holding time and holding temperature. For each factor, the mean S/N ratios of case 1 (w_1=1.0, w_2=0) and case 2 (w_1=0.5, w_2=0.5) were plotted in Figs.5-7 based on the results given in Table 5-6

Table 5-7 The factor's Mean multi-response S/N ratio for each level with two weighting factors

level	Mean S/N ratio for case 1 (w_1=1.0,w_2=0)				Mean S/N ratio for case 2 (w_1=0.5, w_2=0.5)			
	A Flux type	B Chips/ flux ratio	C Holding time	D Holding temperat ure	A Flux type	B Chips/ flux ratio	C Holding time	D Holding temperat ure
1	38.93	38.35	38.73	38.60	31.13	32.10	30.55	31.13
2	38.28	38.71	38.61	38.76	31.65	30.46	30.12	30.41
3	38.59	38.75	38.46	38.45	29.58	29.80	31.69	30.82

.

It is shown in Fig.5-7 that mean S/N ratio of the factor flux type (factor A) reaches maximum using flux Al-clean 101 (level 1), and has the minimum using flux Al-clean 113 (level 2). As the flux Al-clean 101 has a melting temperature around 500℃ and Al-clean 113 has a melting temperature between 690℃ and 705℃. The flux Al-clean 101 is more easily softened to have larger contact area with aluminum chips to achieve higher effectiveness.

The effect of the chips/flux ratio (factor B) on the mean S/N ratio of the recovery rate also plotted in Fig.5-7. The mean S/N ratio of recovery rate grows when additional flux added. It can be seen that the additional flux enhances the recovery rate as the ratio 10:3 (level 1) changes to the ratio 10:4 (level 2). This might be because sufficient flux can greatly protect the aluminum chips from being oxidized during melting process. The curve seems to reach to a plateau from the ratio 10:4(level 2) to 10:5 (level 3).This observation implied that the excessive amount of flux would result in minor effect on recovery rates. In the viewpoint of cost saving, the ratio 10:4 might be considered for recycling production. While the ratio 10:5 was employed, the recovery rate becomes the highest.

The lines plotted from C1 to C3 are the effect of holding time (factor C) on the mean S/N ratio of the recovery rate. The curve is much smoother without sharp fluctuations comparing to other plots, which means holding time has minor effect on the recovery rate. The mean S/N ratio decreases when extended holding times are employed. However, aluminum chips are more likely to be oxidized when being kept at elevated temperatures for a prolonged period of time. Thus, 60mins (level 1) is selected for its higher S/N ratio response.

The plot points D1 to D3 shows the holding temperature (factor D) on the mean S/N ratio of recovery rate. The mean S/N ratio reaches the peak at 760℃, and then drops to 720℃. The working temperature of the three fluxes is within the temperature range of 700℃ to 800℃. Since the energy consumption for recycling is high and chips are more likely to be oxidized at high temperatures, the medium temperature is preferred.

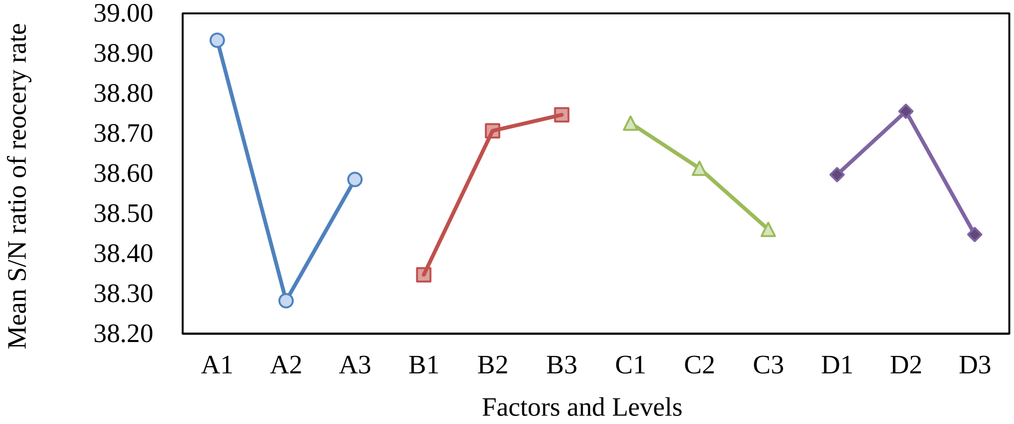


Figure 5.4-2 Multi-response signal-to-noise graph for case 1 (w_1 = 1.0, w_2=0)

By selecting the highest value of the mean S/N ratio for each factor, the optimal level can be determined. On this basis, the optimum combination of levels in terms of maximizing the recovery rate for this recycling process is A1, B3, C1 and D2; i.e. Al-clean 101 as the refining flux; 10:5 as the chips/flux ratio; 60mins as the holding time and 760℃ as the holding temperature.

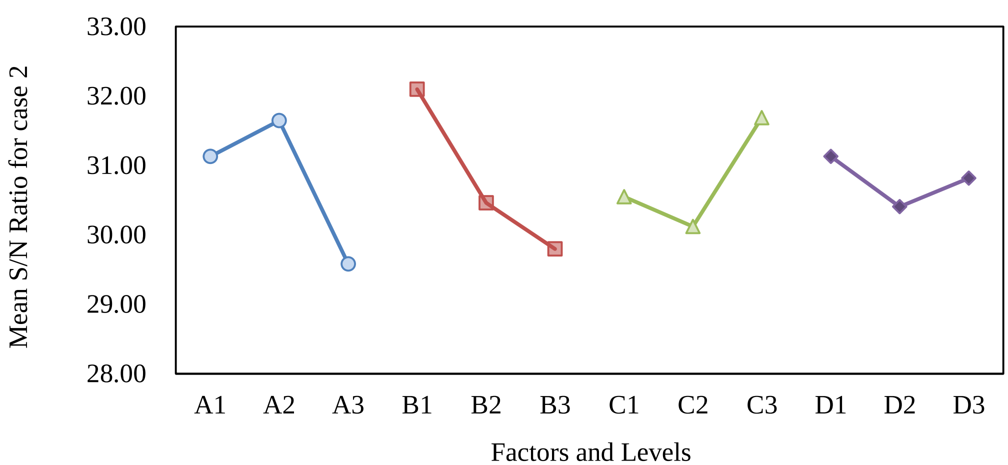


Figure 5.4-3 Multi-response signal-to-noise graph for case 2 (w1 = 0.5, w2=0.5)

For case 2, the recovery rate and corrosion resistance are taken into consideration simultaneously. Fig.5-8 shows the mean signal-to-noise ratio for case 2. By selecting the highest value of the mean S/N ratio for each factor, the optimal level can be determined. Hence, the optimum combination of the levels in terms of minimizing the corrosion resistance for the present recycling process is A2, B1, C3, D1; i.e., Al-clean 113 as the refining flux; 10:3 as the chips/flux ratio; 90 mins as the holding time and 800℃ as the holding temperature.

5.4.3. Factor contributions

The contribution of each factor to the recovery rate can be determined by performing analysis of variance based on Eqs. (5-8) – (5-12). The results of analysis of variance (ANOVA) for case 1 (w_1=1.0, w_2=0) and case 2 (w_1=0.5, w_2=0.5) are summarized in Table 5-8 and Table 5-9, respectively.

Table 5-8 Results of the ANOVA for case 1 (w1=1.0, w2=0)

Factors	Degree of freedom (D)	Sum of squares (SS_p)	Variance (V)	Corrected sums of squares (SS_p’)	Contribution	Rank
Flux type	2	0.64	0.32	0.64	54.13%	1
Chips/flux ratio	2	0.29	0.15	0.29	24.75%	2
Holding time	2	0.11	0.06	0.11	9.04%	4
Holding temperature	2	0.14	0.07	0.14	12.08%	3
error		0.00	0.00		0	
Total		1.17			100%	

Table 5-9 Results of the ANOVA for case 2 (w1=0.5, w2=0.5)						
Factors	Degree of freedom (D)	Sum of squares (SS_p)	Variance (V)	Corrected sums of squares (SS_p')	Contribution	Rank
Flux type	2	6.94	0.40	3.47	34.58%	2
Chips/flux ratio	2	8.39	1.80	4.19	41.80%	1
Holding time	2	3.95	1.45	1.98	19.67%	3
Holding temperature	2	0.79	3.87	0.39	3.94%	4
error		0.00	0.00		0.00	
Total		20.08			100%	

Table 5-8 gives the contribution of the four factors in case 1, i.e. the flux type, chips/flux ratio, holding time and holding temperature is 54.13%, 24.75%, 9.04% and 12.08%, respectively. The flux type makes a contribution of 54.13%, higher than the sum of the rest three factors, which has the major influence on the corrosion resistance of the recycled alloy. The chips/flux ratio takes the second place with a contribution of 24.75%. The holding time and holding temperature has minor influence on recovery rate for both of their contributions are around 10%.

Table 5-9 shows the contribution of the four factors in case 2, i.e. the flux type, chips/flux ratio, holding time and holding temperature is 34.58%, 41.80%, 19.67% and 3.94%, respectively. Chips/flux ratio has a contribution of 41.80% and flux type has a contribution of 34.58%, which are the two major influencing factors. The holding time makes medium contribution while holding temperatures during the refining process has little effect on case 2 for its contribution percentages are below 5%.

5.5. Conclusions

The Taguchi method for the design of experiment has been used for optimizing the recycling process for the machining chips of high pressure die cast aluminum alloy A380. Four factors, three levels for each factor were designed based on Taguchi method. To achieve the maximum recovery rate and corrosion resistance, the signal-to-noise ratio of HB characteristics was employed to calculate the S/N ratio of recovery rate and corrosion resistance. The optimum combinations were worked out based on the S/N ratio of each factor.

For the multi-response objective case 1, the metal yield was the only requirement for the recycling process. The optimum combination (A1B3C1D2) was Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, and 60 mins as the holding time and 760℃ as the holding temperature. The flux type made the major contribution to recovery rate with the percentage of 54.13%, which was higher than the sum of the rest three factors. The chips/flux ratio made medium contribution while both the holding time and holding temperature during refining process had minor effect on the recovery rate for their low contribution percentages.

For the multi-response objective case 2, weighing factors were selected as w_1= 0.5, w_2=0.5. The optimum combination (A2B1C3D1) was Al-clean 113 as the refining flux, 10:3 as the chips/flux ratio, and 90 mins as the holding time and 800℃ as the holding temperature. The chips/flux ratio made the major contribution with the percentage of 41.80% and followed by flux type. Holding time made medium contribution while temperature during refining process had minor effect for its low contribution percentages.

Acknowledgments

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5.6. References

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6. CHAPTER VI

GENERAL CONCLUSION AND FUTURE WORKS

6.1. General conclusions

In order to fulfill the objectives stated in Chapter 1, firstly, twenty-two trials of recycling experiments were conducted under several conditions varying from different cleaning methods and influencing factors during refining process. Based on the results of the preliminary study, experimental plans were designed via the Design of Experiment to optimize the refining process with the objective recovery rate, porosity content, mechanical properties and corrosion resistance. Four influencing factors (flux types, chips/flux ratios, holding times and holding temperatures) were selected as four factors and for each factor, three corresponding levels were also chosen to create Taguchi orthogonal array. Signal-to-noise (S/N) ratios for multiple characteristics and analysis of variance (ANOVA) were utilized to analyze experimental data. Optimum combinations of factor were analyzed and concluded for individual response and multi-response. The main conclusions from this study can be summarized as following:

1. For cleaning method, acetone was a good agent to remove oil emulsion presented on the surface of aluminum machining chip in organic solvent cleaning.
2. The highest recovery rate of the recycled aluminum alloy A380 was 92.03% when the experimental conditions of Al-clean 101 for the refining flux; 10:5

for the chips/flux ratio; 60mins for the holding time and 760℃ for the holding temperature were applied.

3. The mechanical properties of the recovered aluminum were as good as those of the die-cast A380 aluminum alloy. The microstructure of the recovered aluminum alloys also contained the primary α-Al, Si phase, $CuAl_2$, Fe containing inter-metallic phases, which were almost the same as those present in the die cast A380. Despite of high concentration of silicon and low concentration of magnesium, the recovered aluminum alloy had an acceptable chemical composition.

4. When the metal yield was the only requirement for the recycling process, the optimum combination (A1B3C1D2) could reach the highest S/N ratio of recovery rate, i.e. Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, and 60mins as the holding time and 760℃ as the holding temperature. The flux type made the major contribution to recovery rate with the percentage of 54.13%, which was higher than the sum of the rest three factors. The chips/flux ratio made medium contribution of 24.75% while both the holding time and holding temperature during refining process had small effects on the recovery rate for their low contribution percentages of 12.08% and 9.04%, respectively.

5. To take both recovery rate and porosity content into consideration, weighing factors w_1=0.5, w_2=0.5 were selected. A2B2C1D1 was concluded as the optimized combination, i.e. Al-clean 113 as the refining flux; 10:4 as the chips/flux ratio; 60mins as the holding time; 800℃ as the holding temperature.

The holding temperature makes the major influence with a contribution of 51.49%, and the chips/flux ratio and the holding time had the moderate effects of 23.91% and 19.28% on the porosity, respectively, while flux type has a minor influence with the contribution of 5.32%.

6. To evaluate both the recovery rate and mechanical properties during the refining process, weighing factors were selected as w_1= 0.4, w_2=0.2, w_3=0.2 and w_4=0.2 for recovery rate, YS, E_f and UTS, respectively. The optimum combination (A1B3C1D2) had the highest S/N ratio for this multi-response, i.e. Al-clean 101 as the refining flux, 10:5 as the chips/flux ratio, and 60 mins as the holding time and 760℃ as the holding temperature. The holding temperature made the major contribution with the percentage of 64.51%, which was higher than the sum of the rest three factors. The chips/flux ratio made medium contribution while both the holding time and holding temperature during refining process had minor effects on the recovery rate and tensile properties for their low contribution percentages.

7. For the requirement of recovery rate and corrosion resistance, weighing factors were selected as w_1= 0.5, w_2=0.5. The optimum combination (A2B1C3D1) was Al-clean 113 as the refining flux, 10:3 as the chips/flux ratio, and 90 mins as the holding time and 800℃ as the holding temperature. The chips/flux ratio made the major a contribution with the percentage of 41.80% and followed by flux type. Holding time made medium contribution while the holding temperature during refining process had little effect for its low contribution percentages.

6.2. Future works

Organic solvent such as acetone solvent is good for chips cleaning. However, acetone is a flammable liquid which has a certain level of hazard to store and transport. Thermal method has comparable cleaning effectiveness as solvent cleaning, but gas emission is observed during heating process, which is not as environment-friendly as solvent cleaning method. Further studies should be carried out on other cleaning methods such as hot pressing techniques.

The recovery rate of the A380 alloy reaches as high as 92.03% via the Design of Experiment and the data analysis. But, chemical reactions between chips and fluxes and physical recovery mechanisms are still unclear and need to be investigated in details.

The developed cleaning and refining processes were only validated under lab conditions. Large-scale experiments on manufacturing sites should be performed to prototype the developed recycling process for industrial application.

APPENDICES

Appendix I : Original Data of Tensile Testing

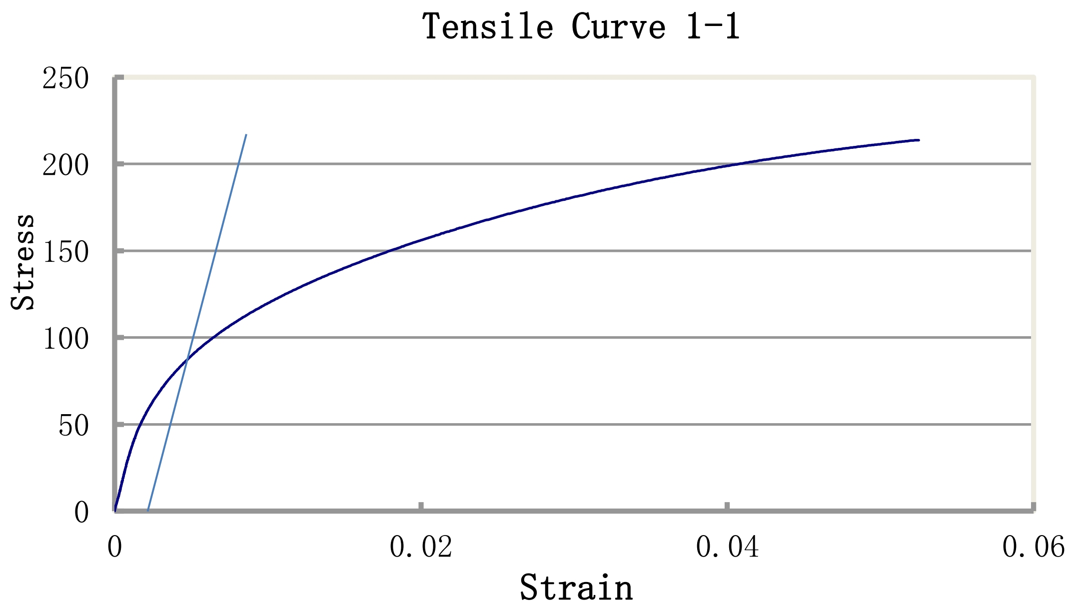

Figure I-1 Tensile curve for sample 1 DOE#1 test 1

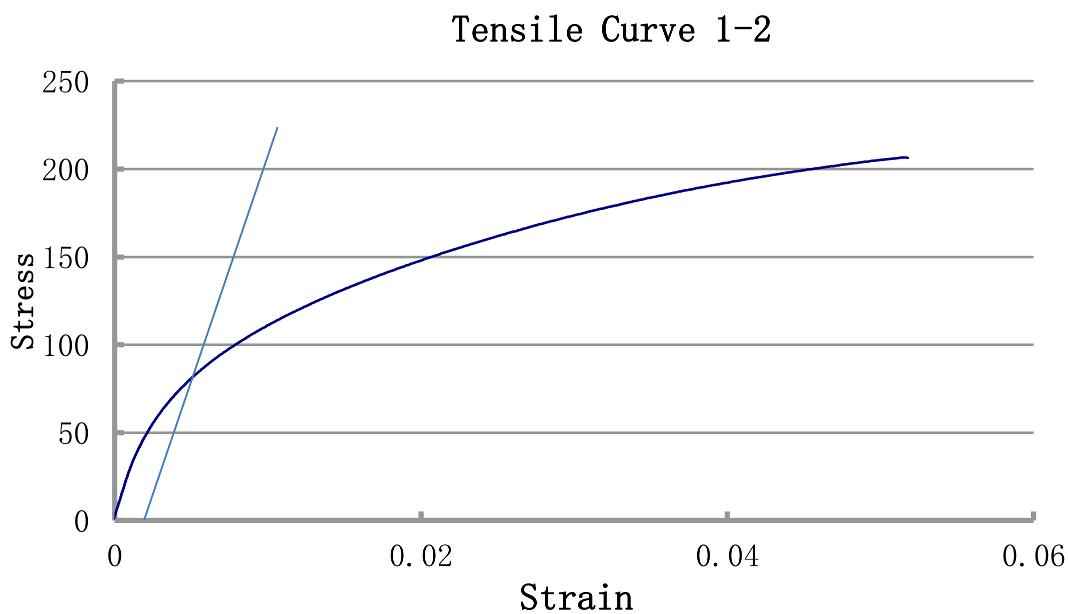

Figure I-2 Tensile curve for sample 2 DOE#1 test 1

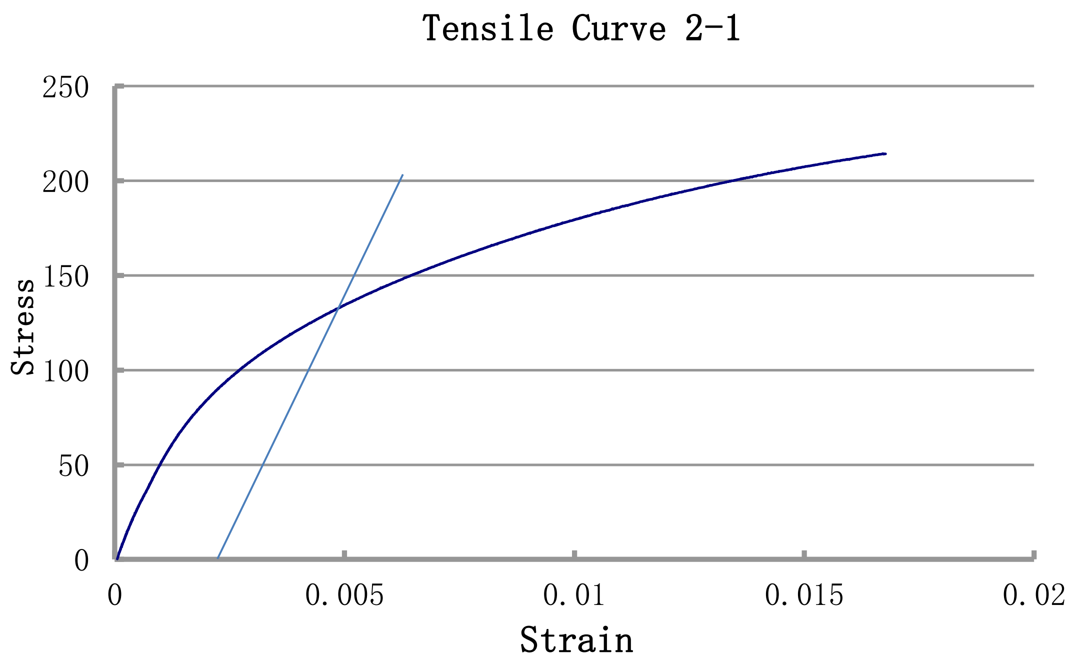

Figure I-3 Tensile curve for sample 1 DOE#1 test 2

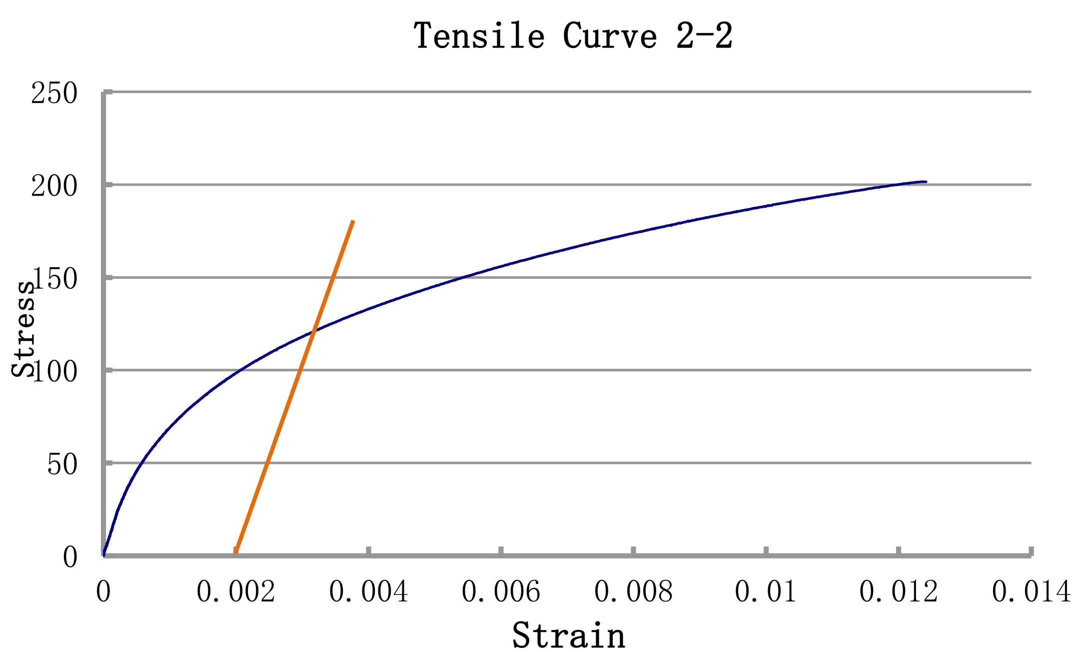

Figure I-4 Tensile curve for sample 2 DOE#1 test 2

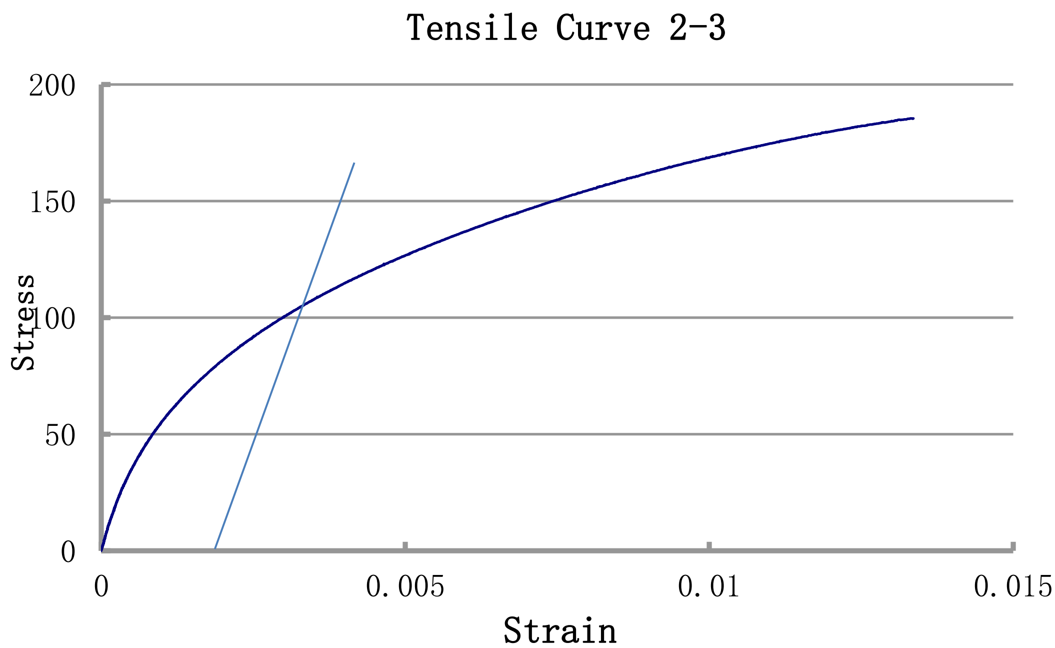

Figure I-5 Tensile curve for sample 3 DOE#1 test 2

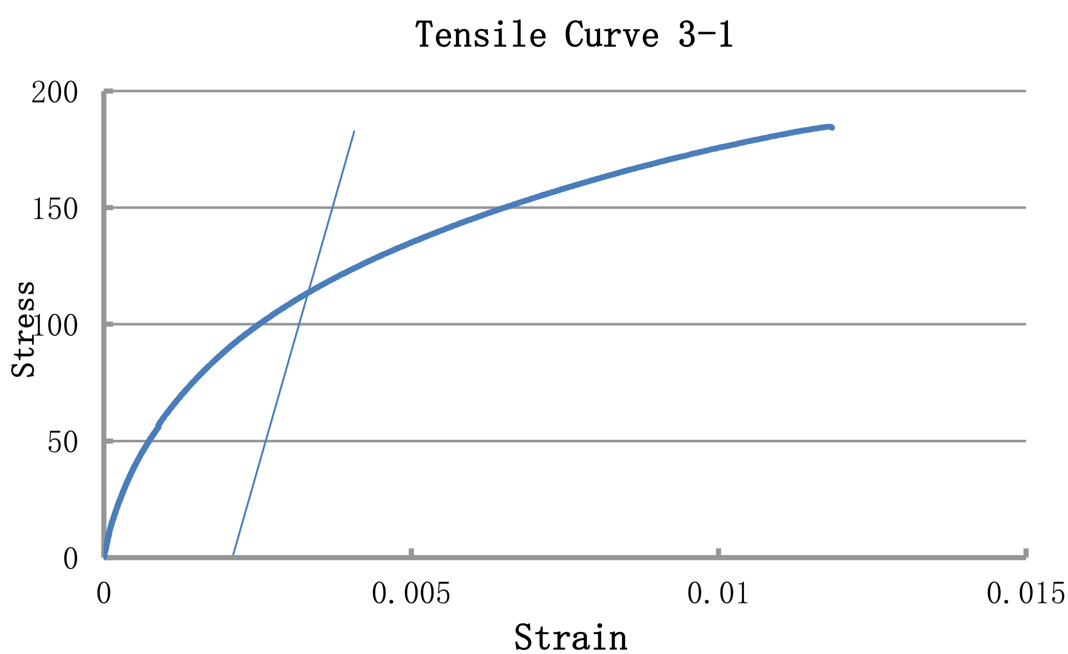

Figure I-6 Tensile curve for sample 1 DOE#1 test 3

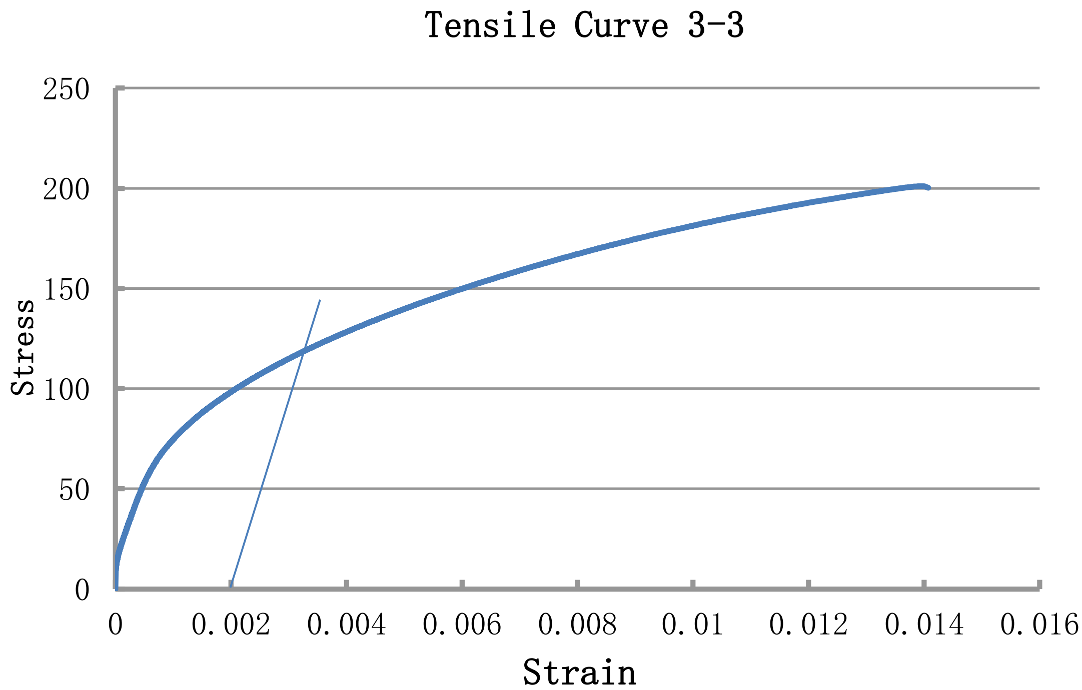

Figure I-7 Tensile curve for sample 3 DOE#1 test 3

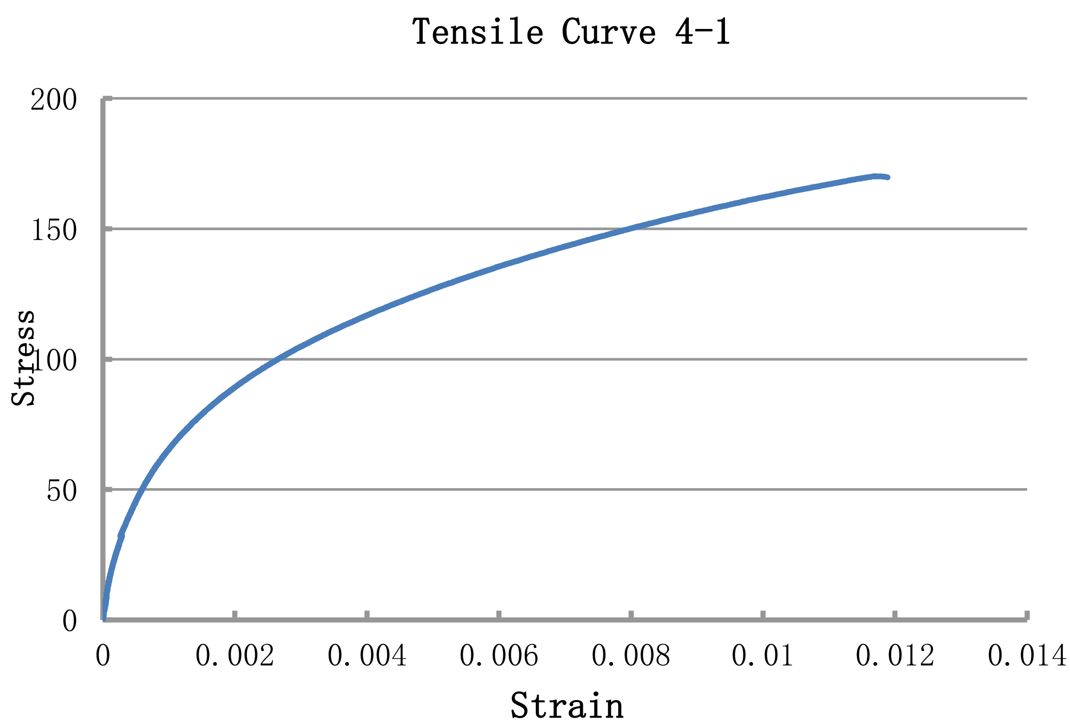

Figure I-8 Tensile curve for sample 1 DOE#1 test 4

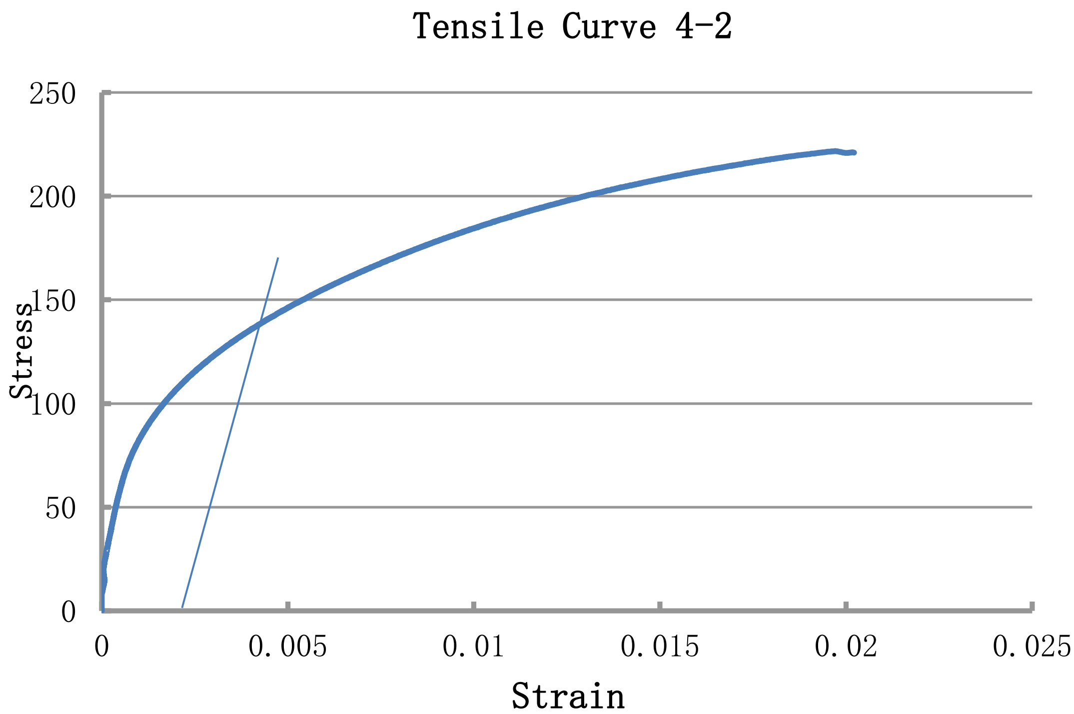

Figure I-9 Tensile curve for sample 2 DOE#1 test 4

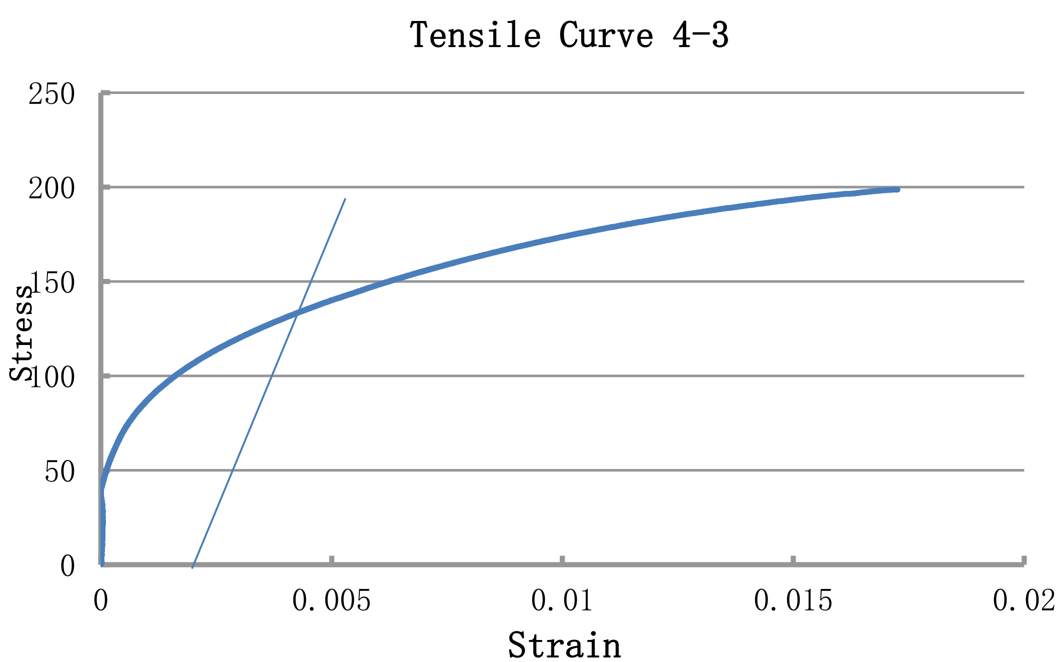

Figure I-10 Tensile curve for sample 3 DOE#1 test 4

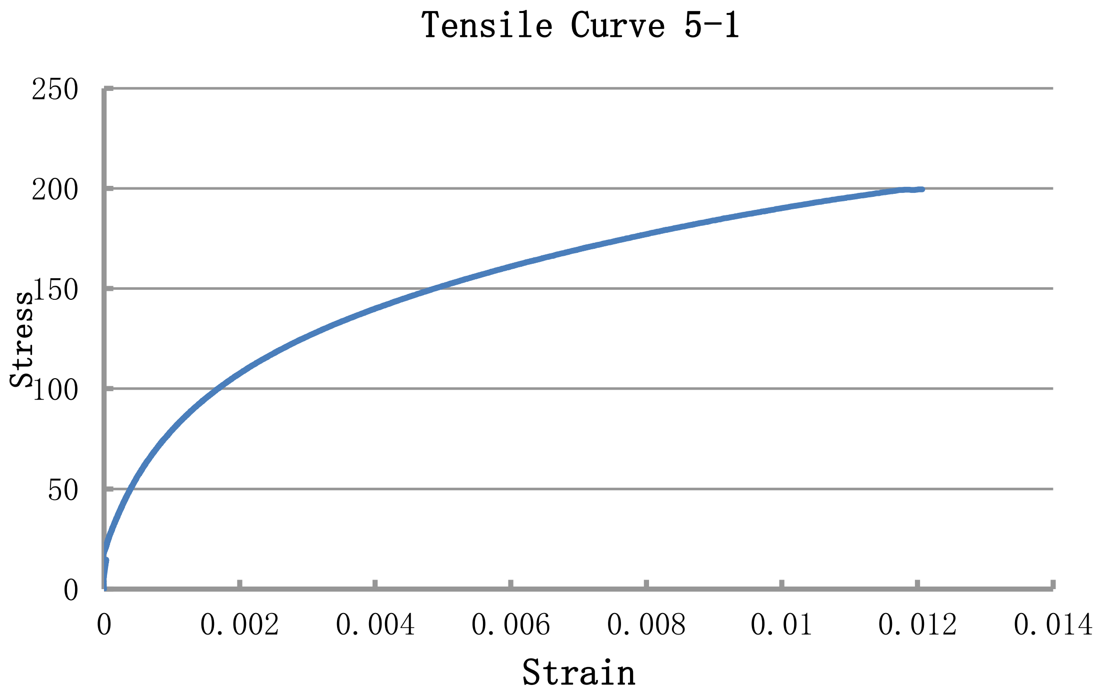

Figure I-11 Tensile curve for sample 1 DOE#1 test 5

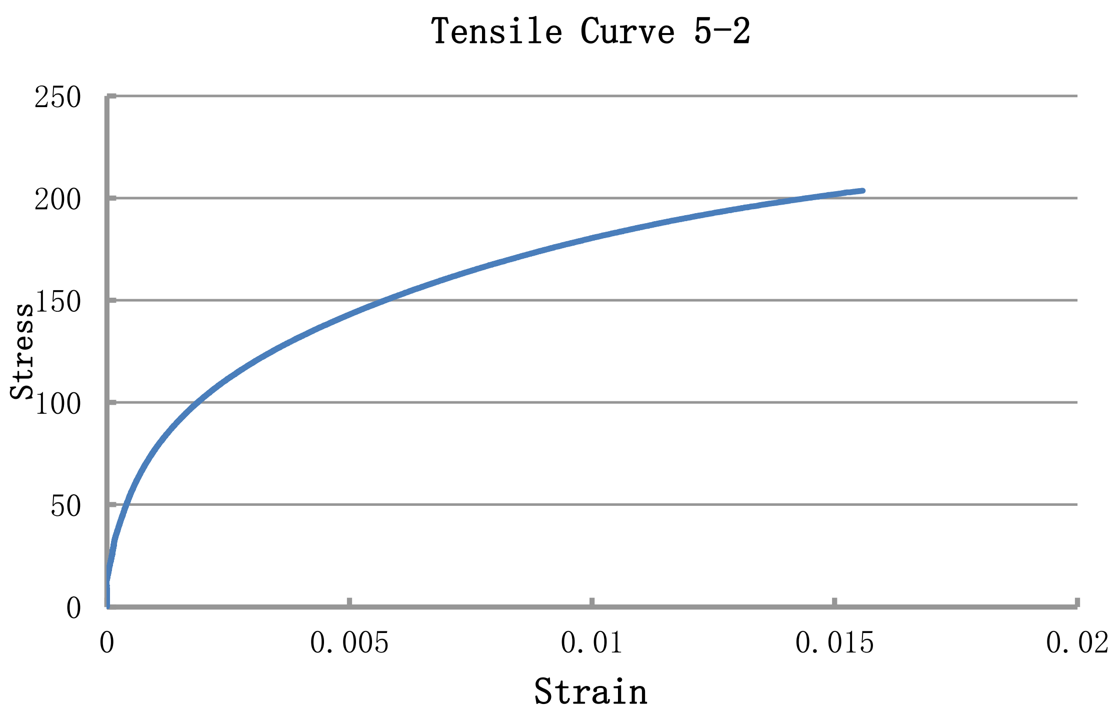

Figure I-12 Tensile curve for sample 2 DOE#1 test 5

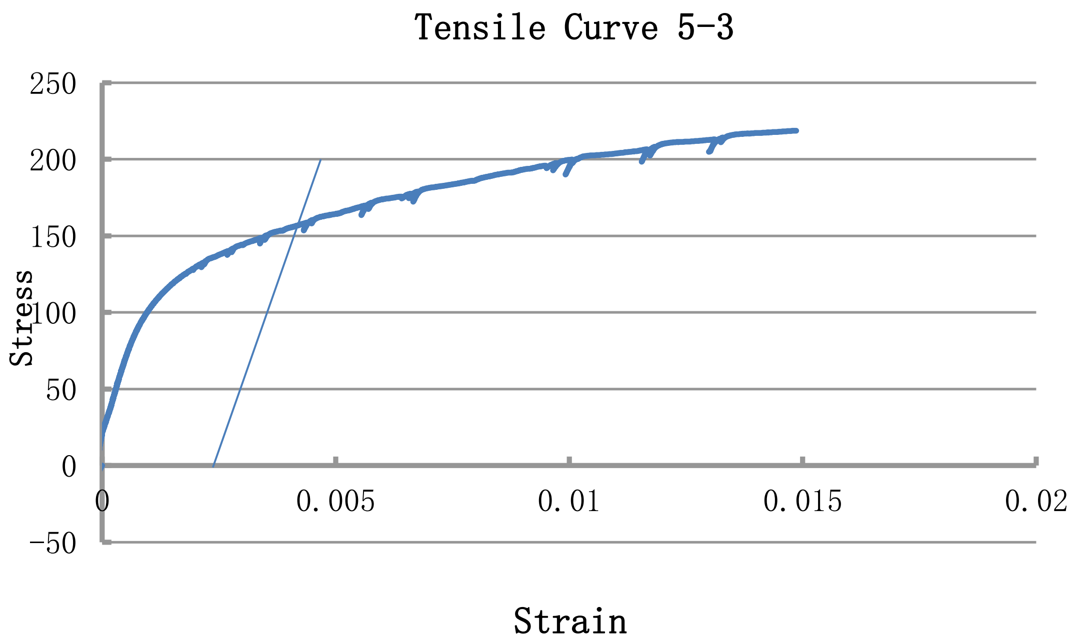

Figure I-13 Tensile curve for sample 3 DOE#1 test 5

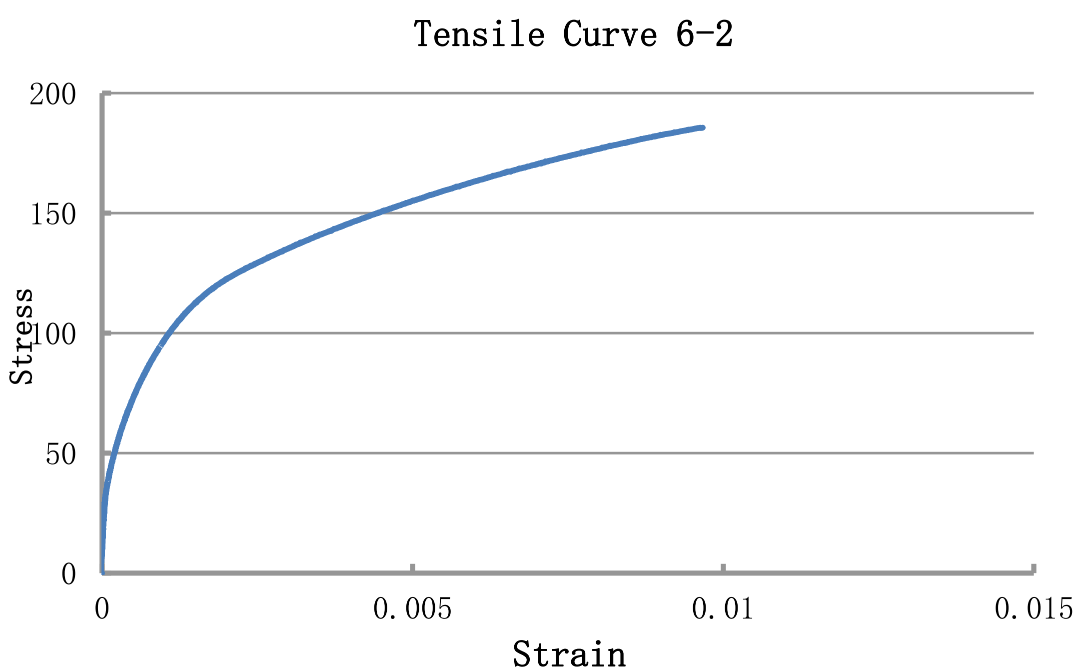

Figure I-14 Tensile curve for sample 2 DOE#1 test 6

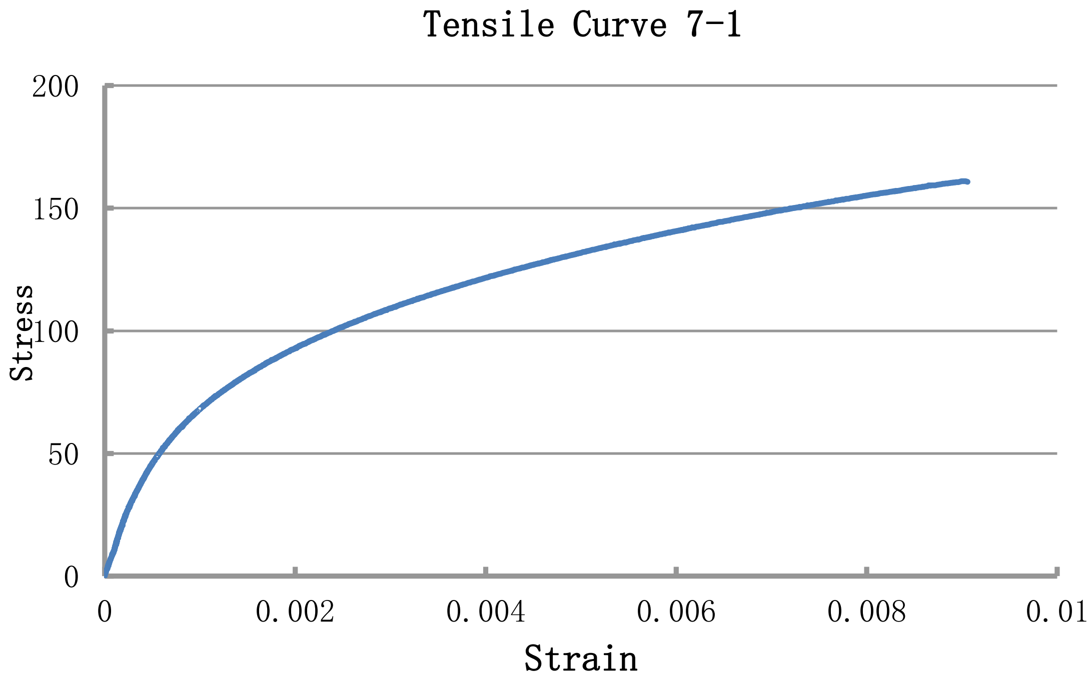

Figure I-15 Tensile curve for sample 1 DOE#1 test 7

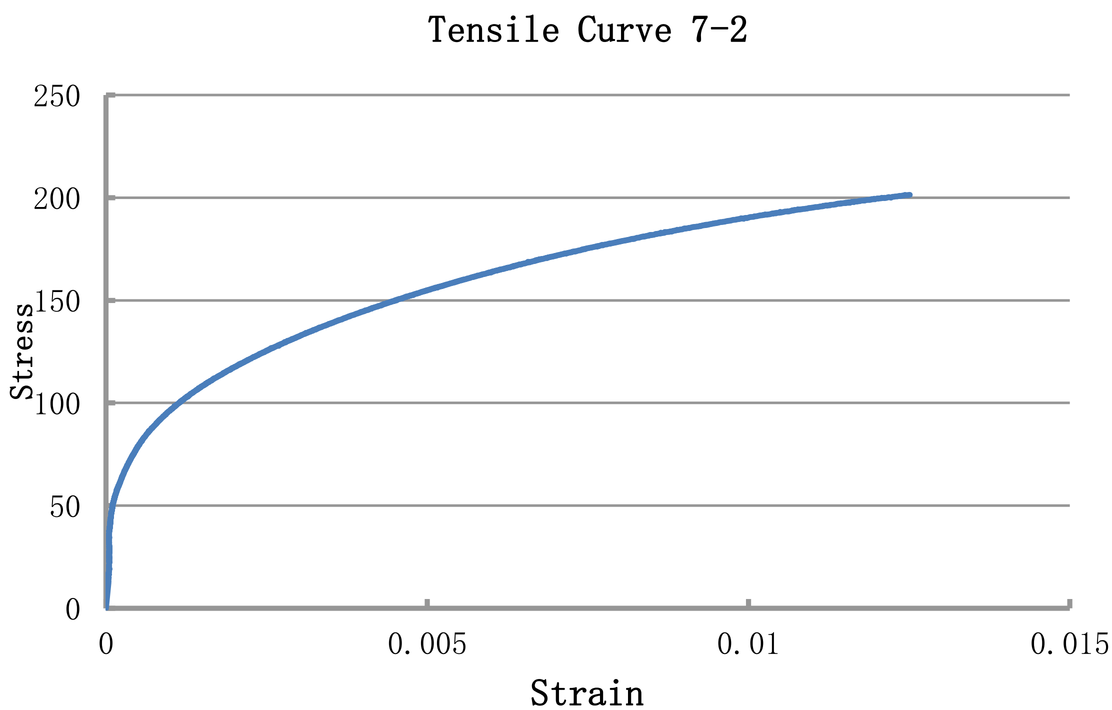

Figure I-16 Tensile curve for sample 2 DOE#1 test 7

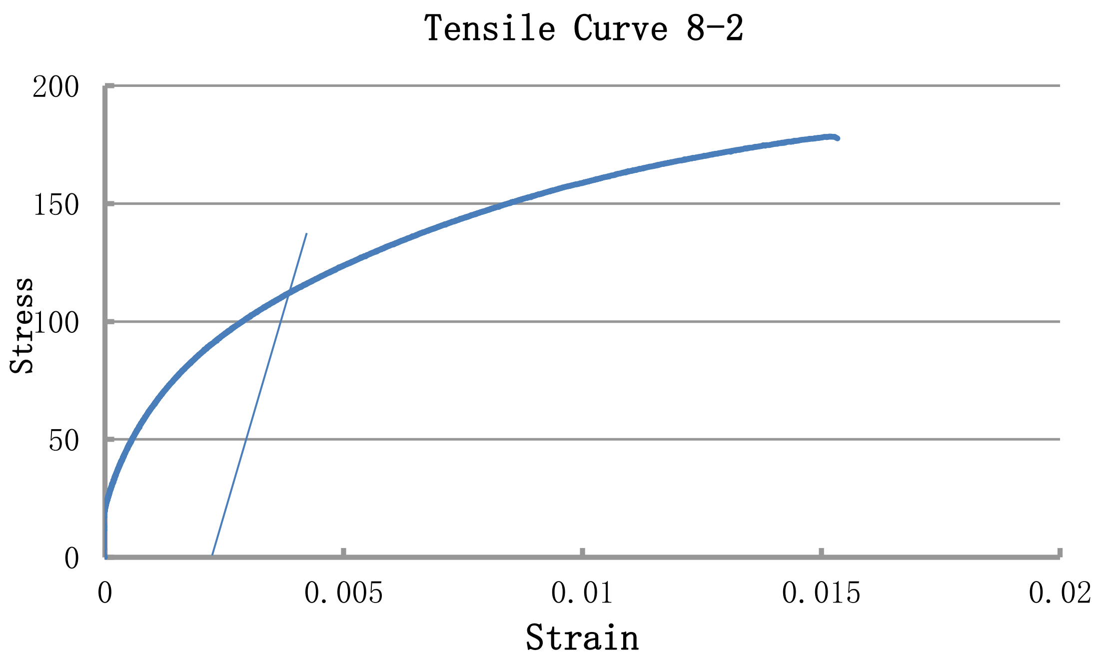

Figure I-17 Tensile curve for sample 2 DOE#1 test 8

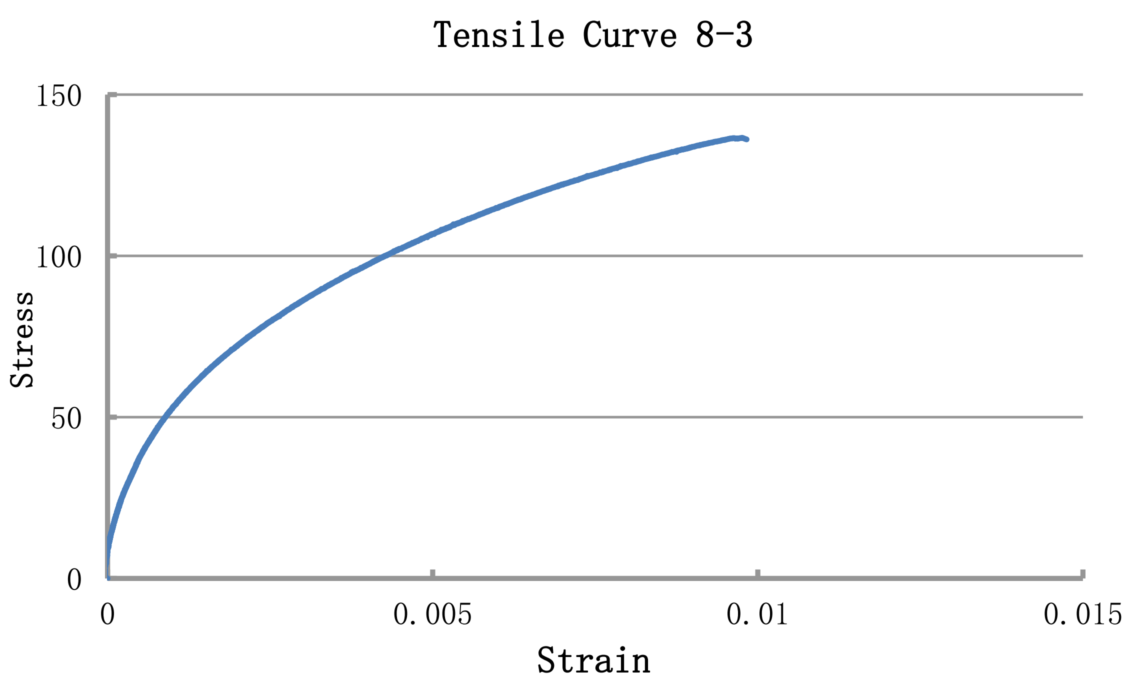

Figure I-18 Tensile curve for sample 3 DOE#1 test 8

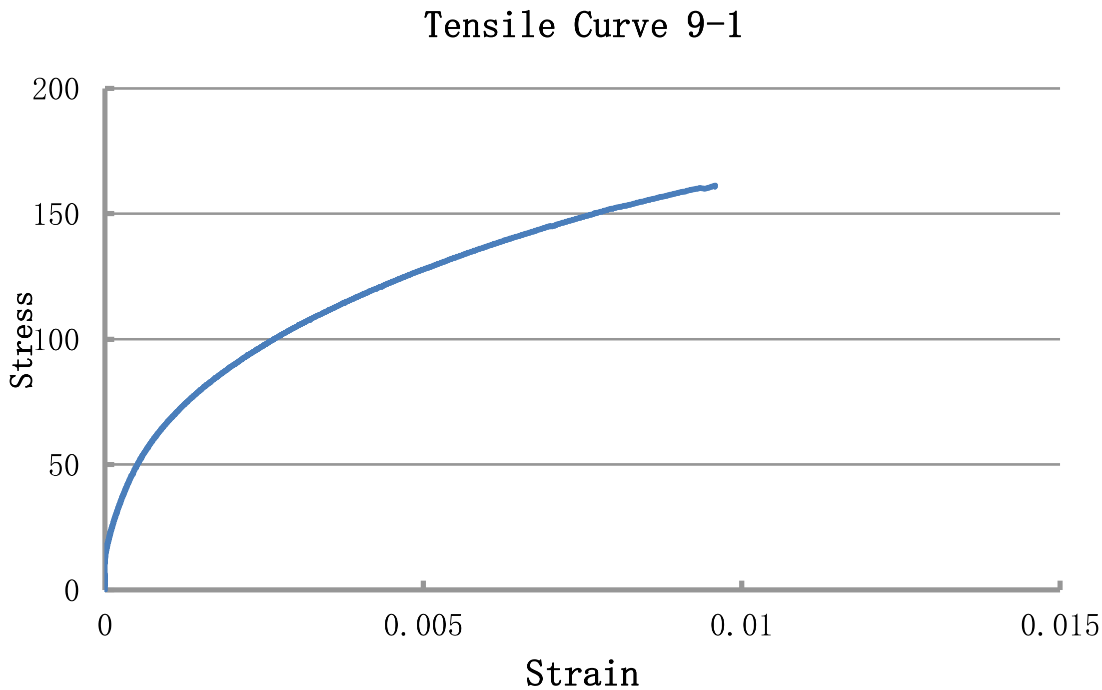

Figure I-19 Tensile curve for sample 1 DOE#1 test 9

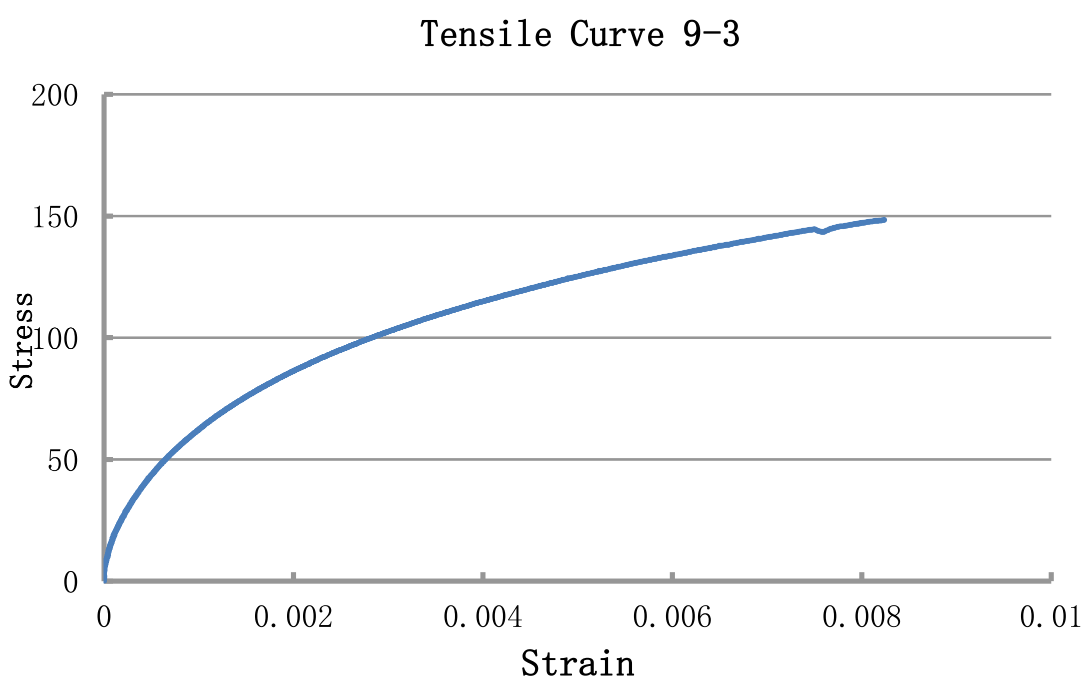

Figure I-20 Tensile curve for sample 3 DOE#1 test 9

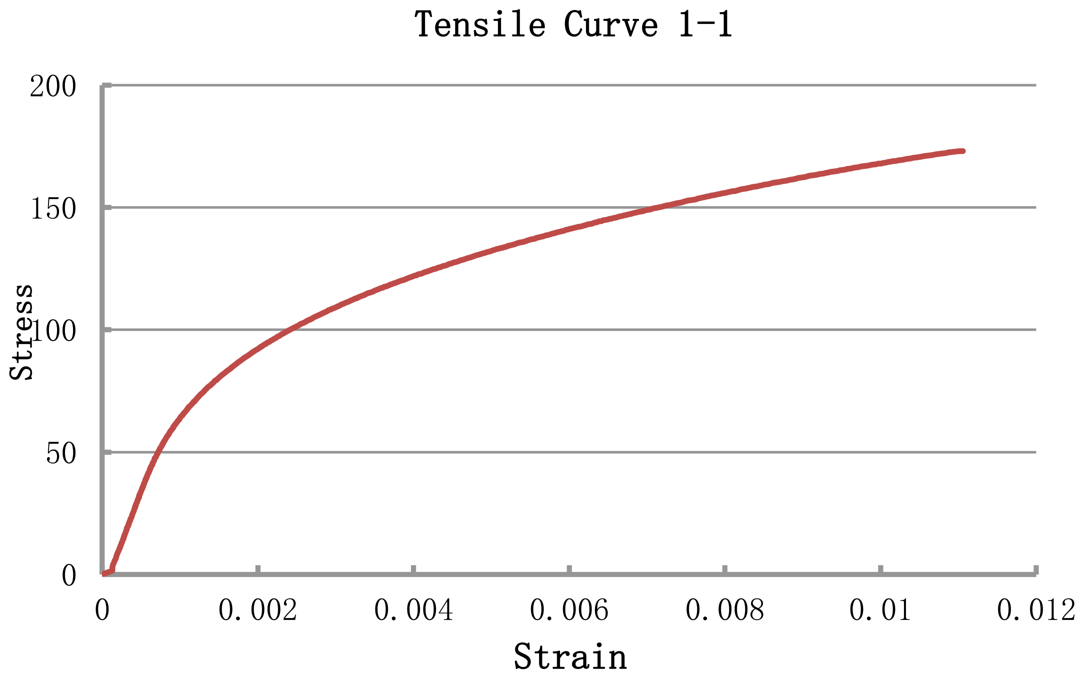

Figure I-21 Tensile curve for sample 1 of DOE#2 test 1

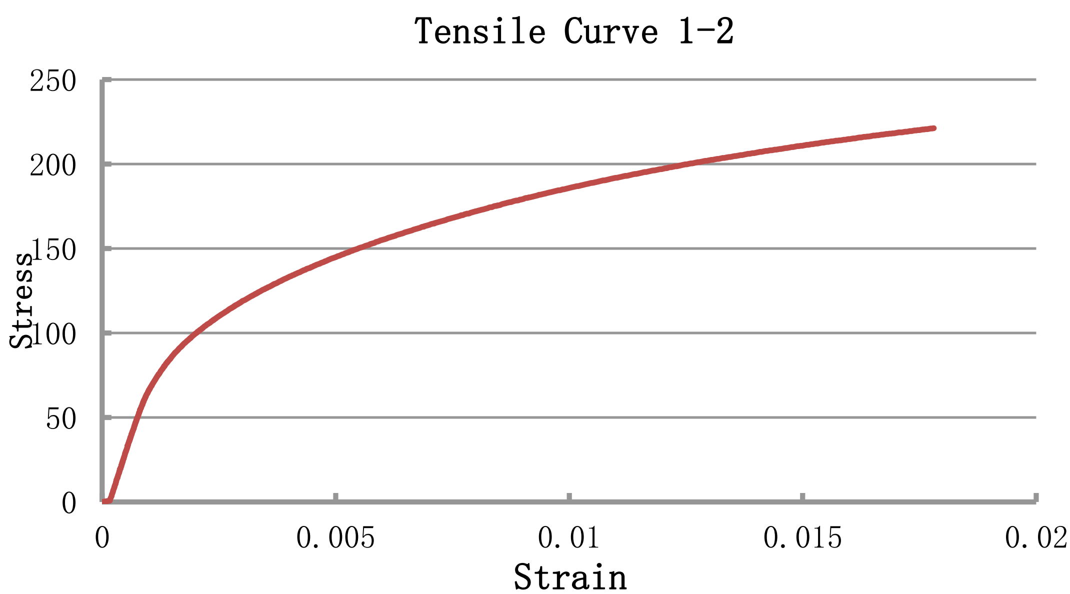

Figure I-22 Tensile curve for sample 2 of DOE#2 test 1

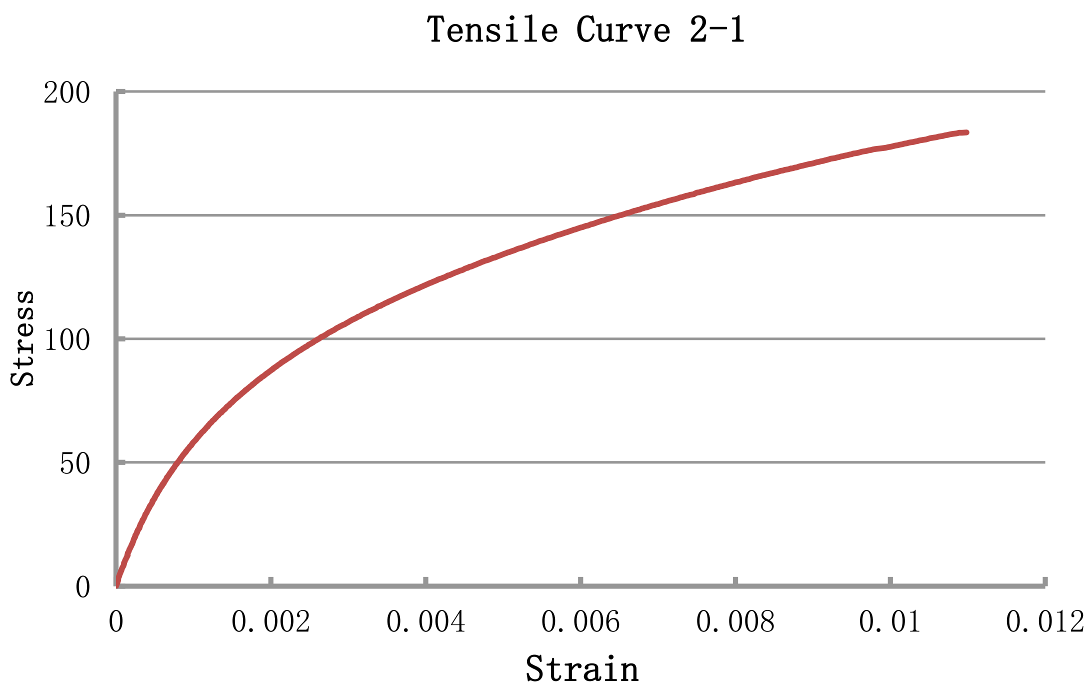

Figure I-23 Tensile curve for sample 1 of DOE#2 test 2

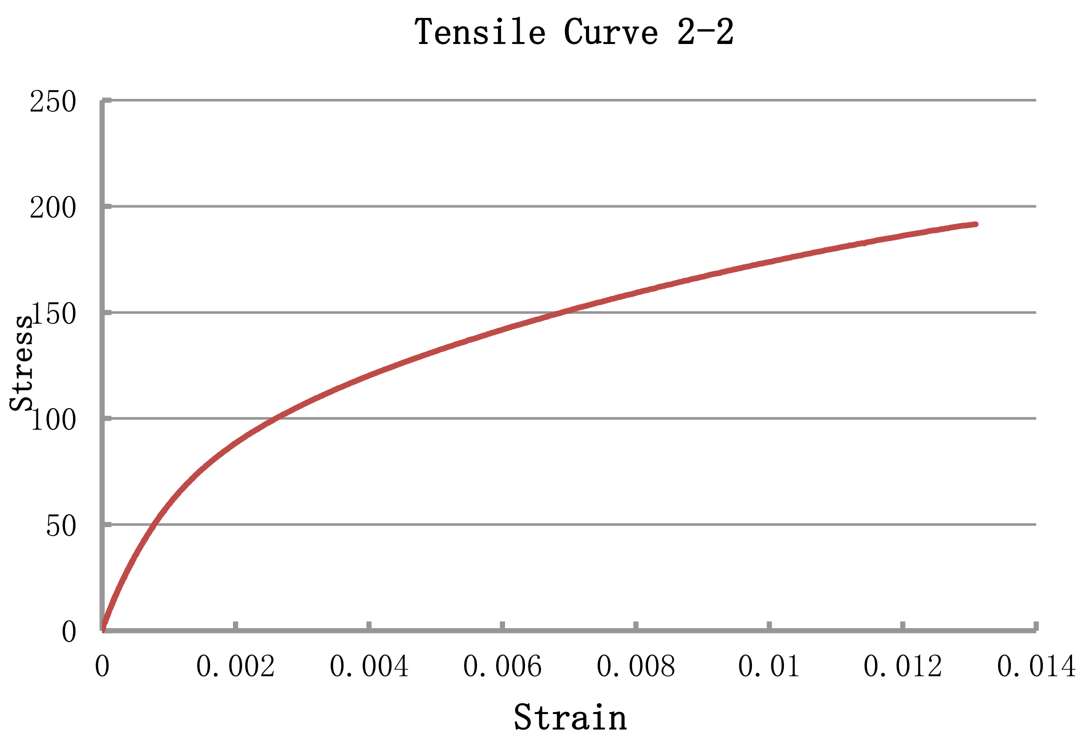

Figure I-24 Tensile curve for sample 2 of DOE#2 test 2

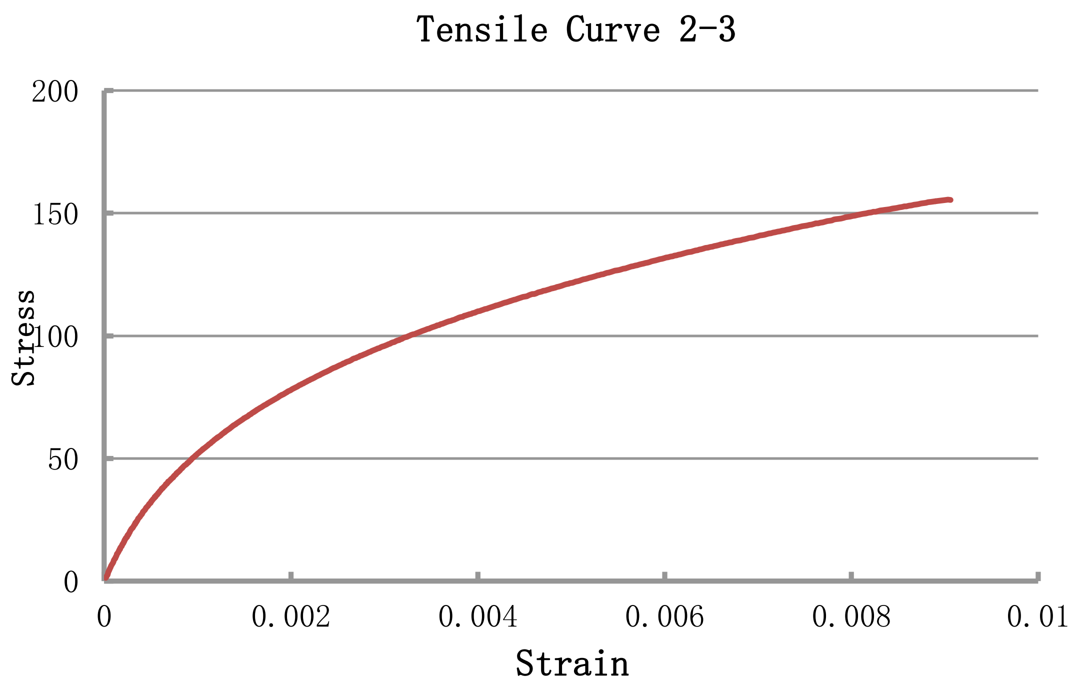

Figure I-25 Tensile curve for sample 3 of DOE#2 test 2

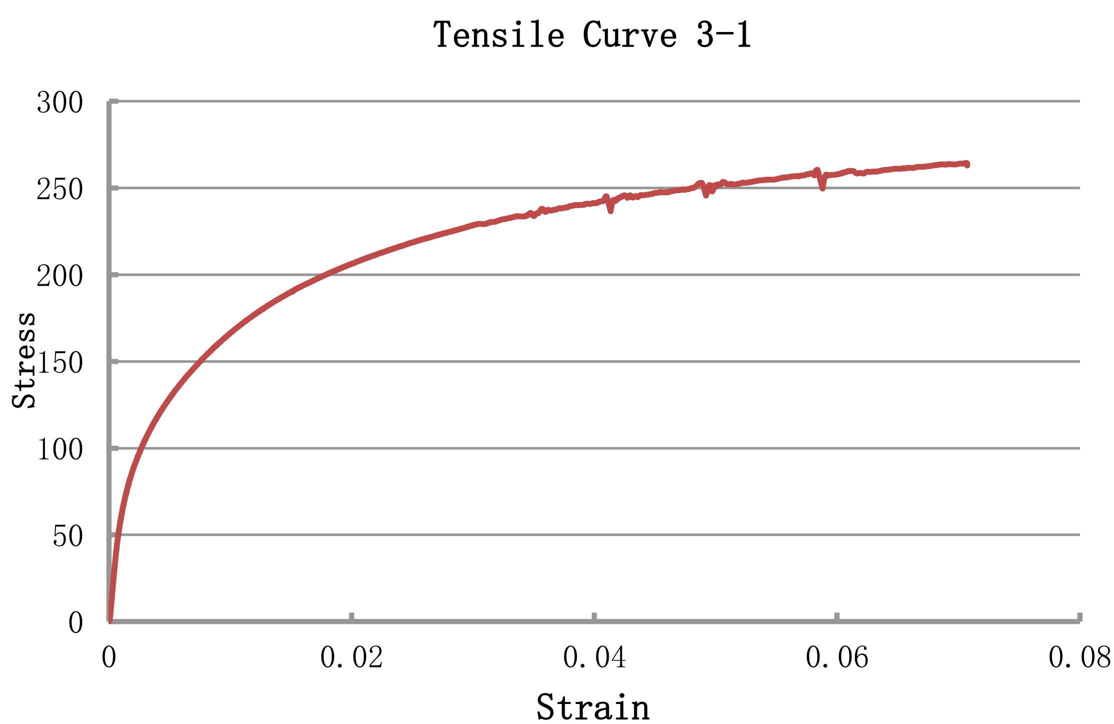

Figure I-26 Tensile curve for sample 1 of DOE#2 test 3

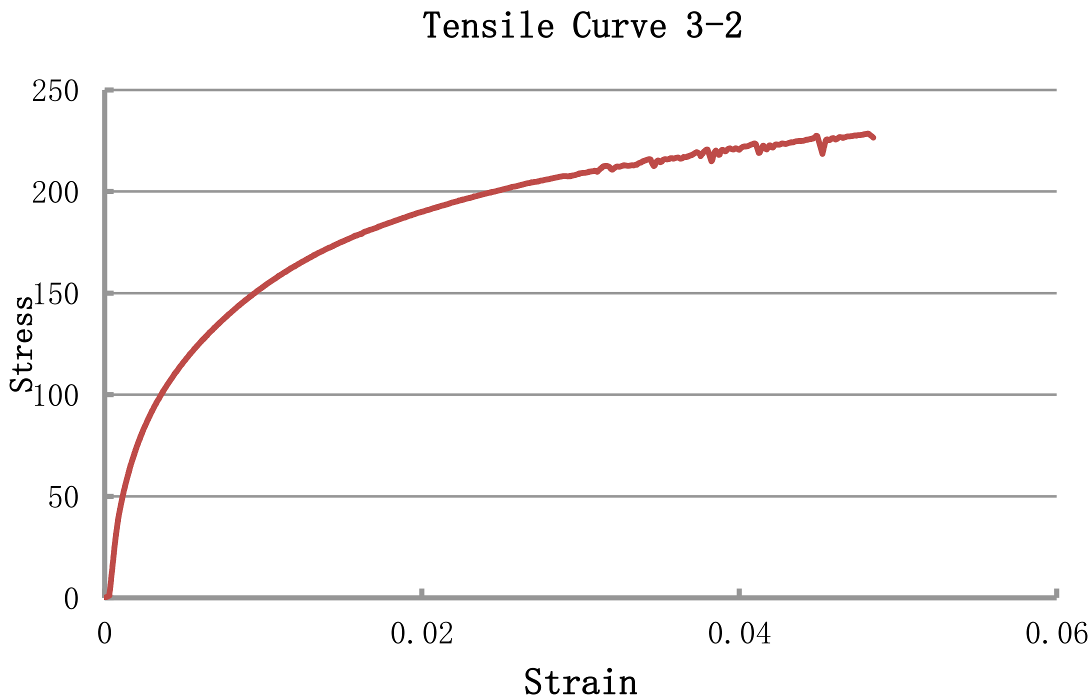

Figure I-27 Tensile curve for sample 2 of DOE#2 test 3

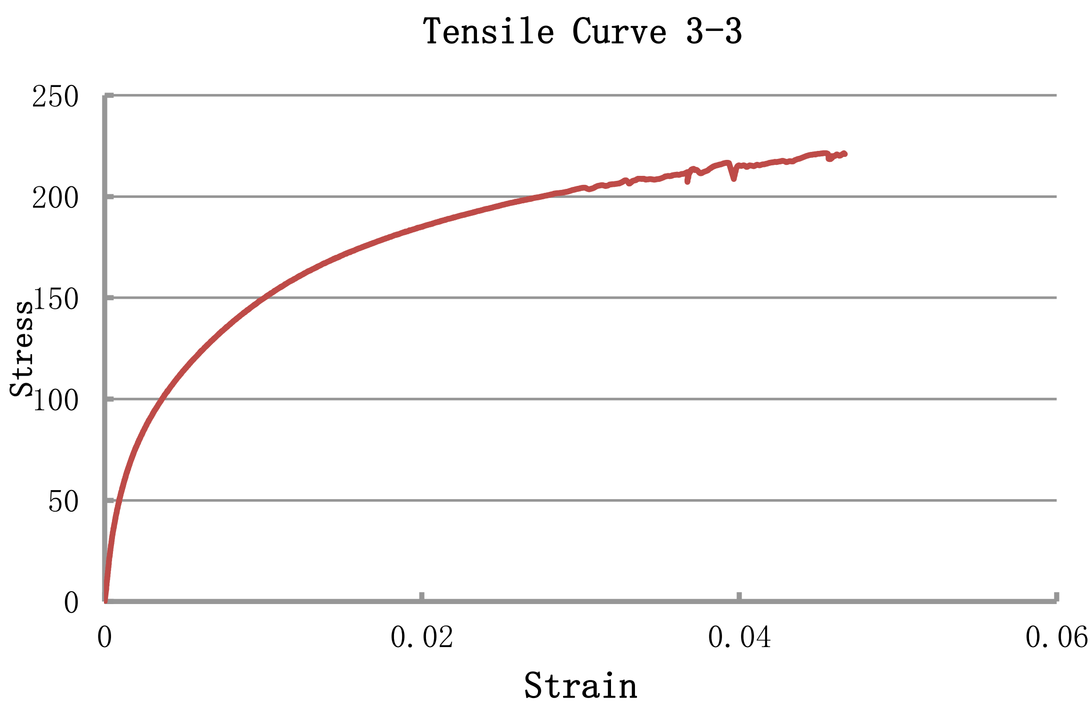

Figure I-28 Tensile curve for sample 3 of DOE#2 test 3

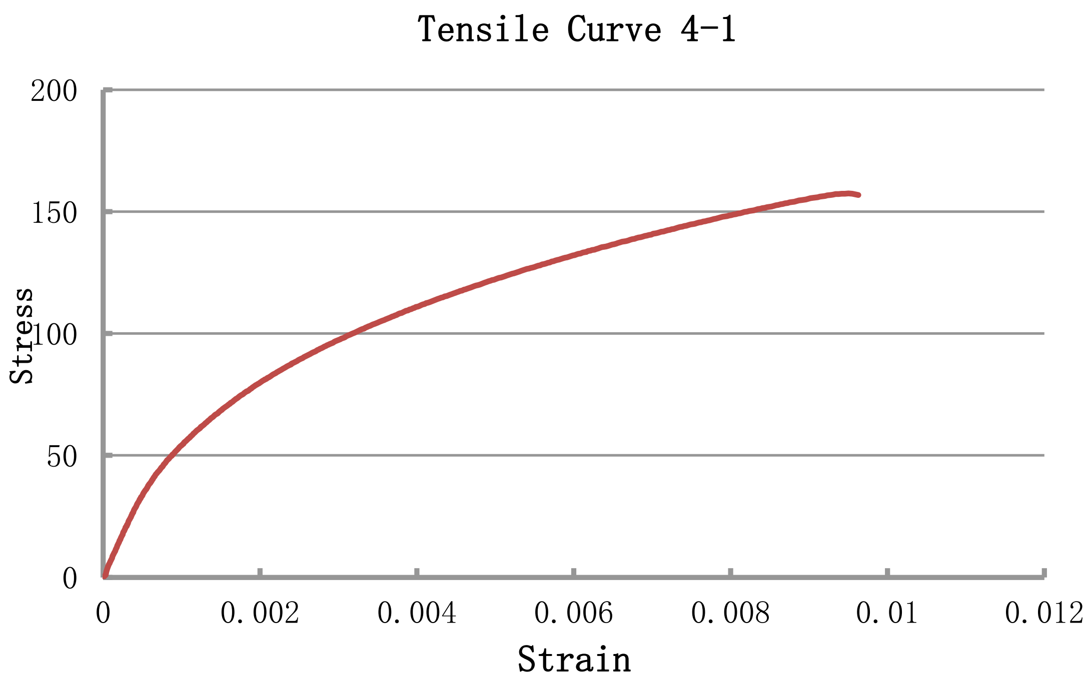

Figure I-29 Tensile curve for sample 1 of DOE#2 test 4

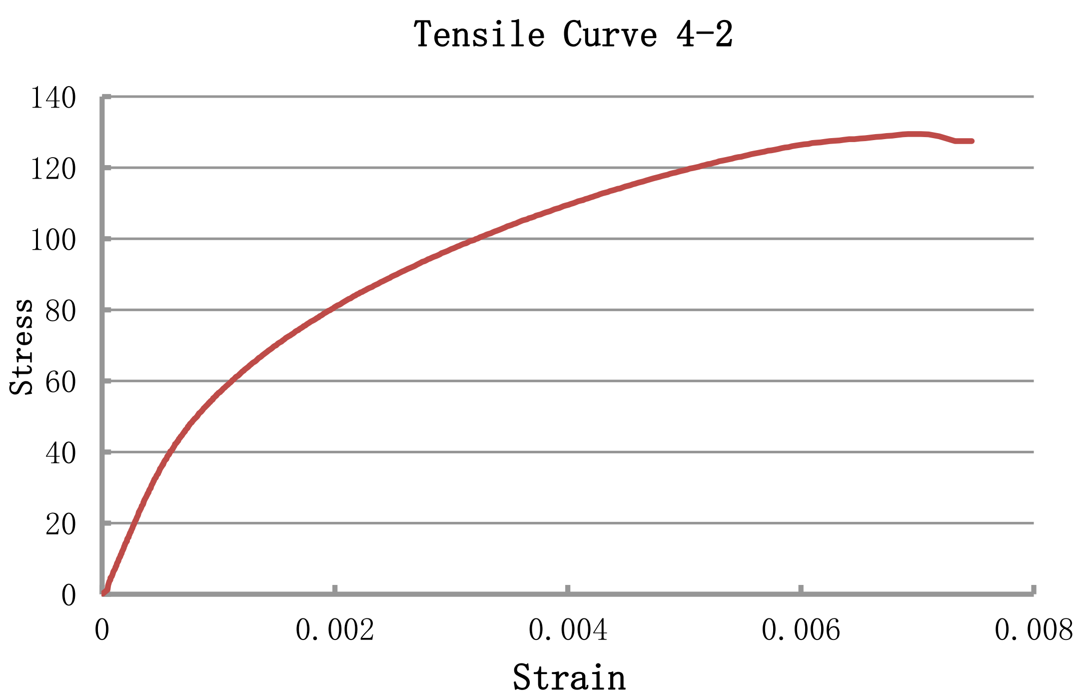

Figure I-30 Tensile curve for sample 2 of DOE#2 test 4

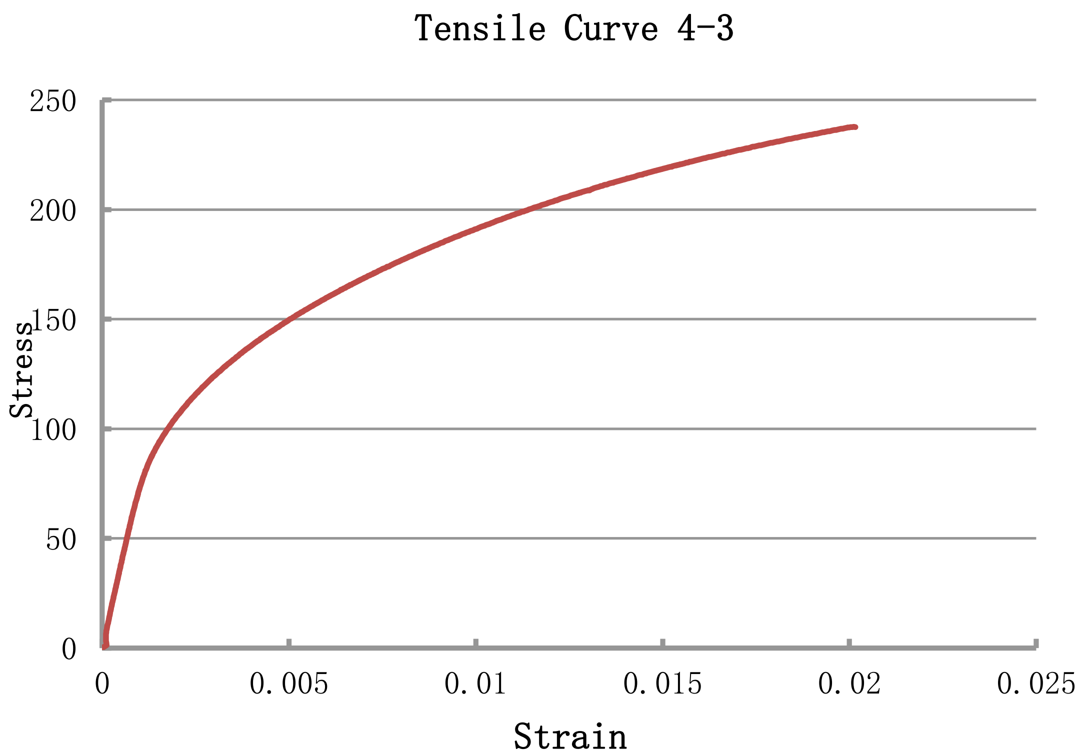

Figure I-31 Tensile curve for sample 3 of DOE#2 test 4

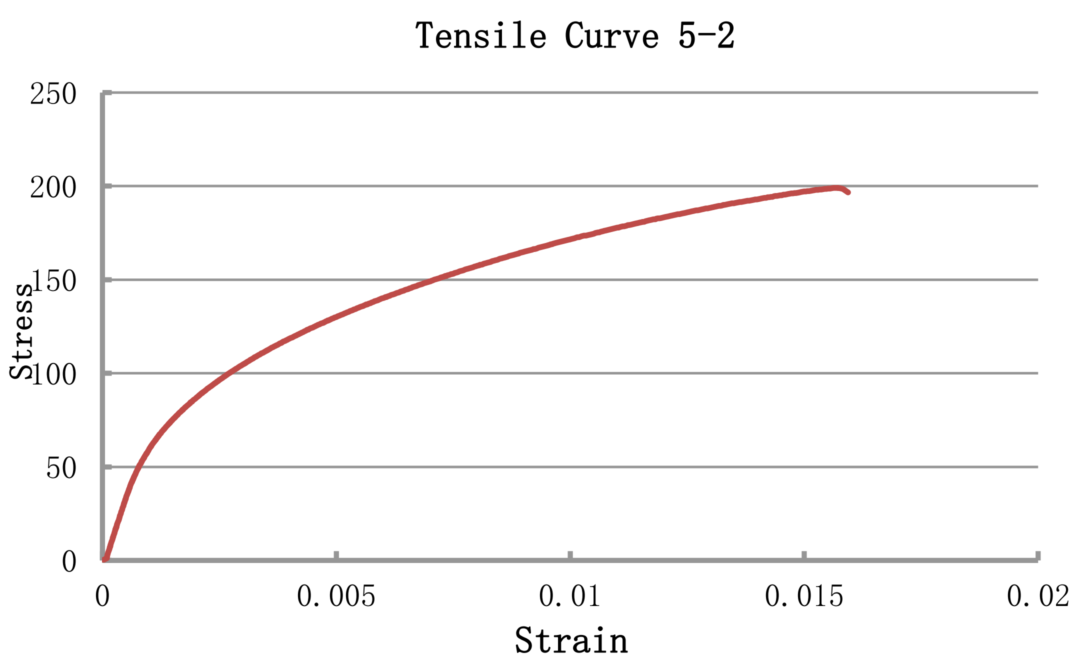

Figure I-32 Tensile curve for sample 2 of DOE#2 test 5

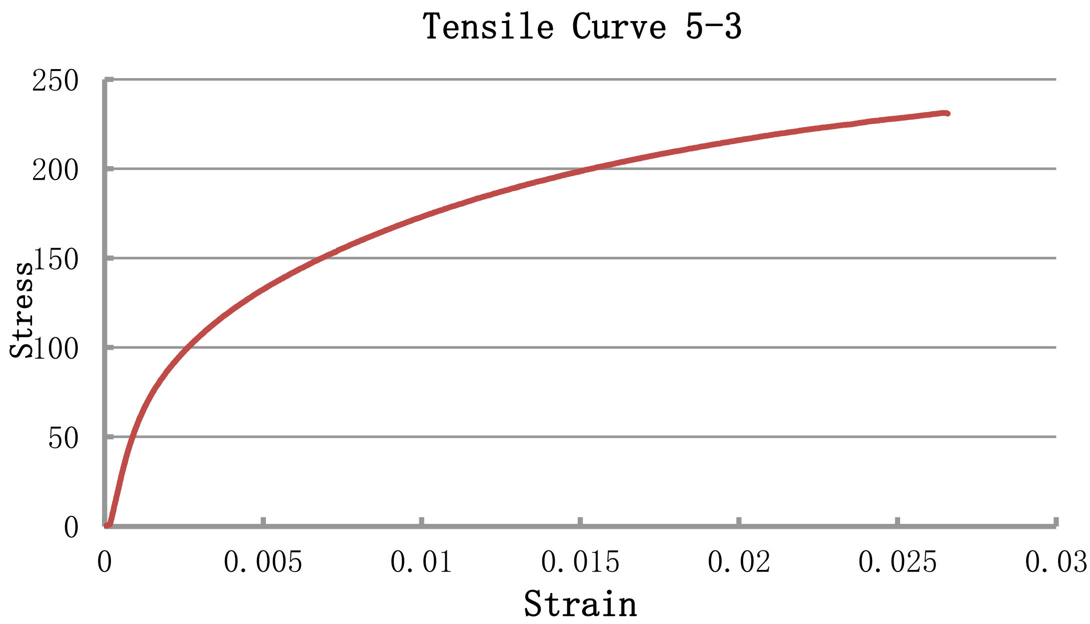

Figure I-33 Tensile curve for sample 3 of DOE#2 test 5

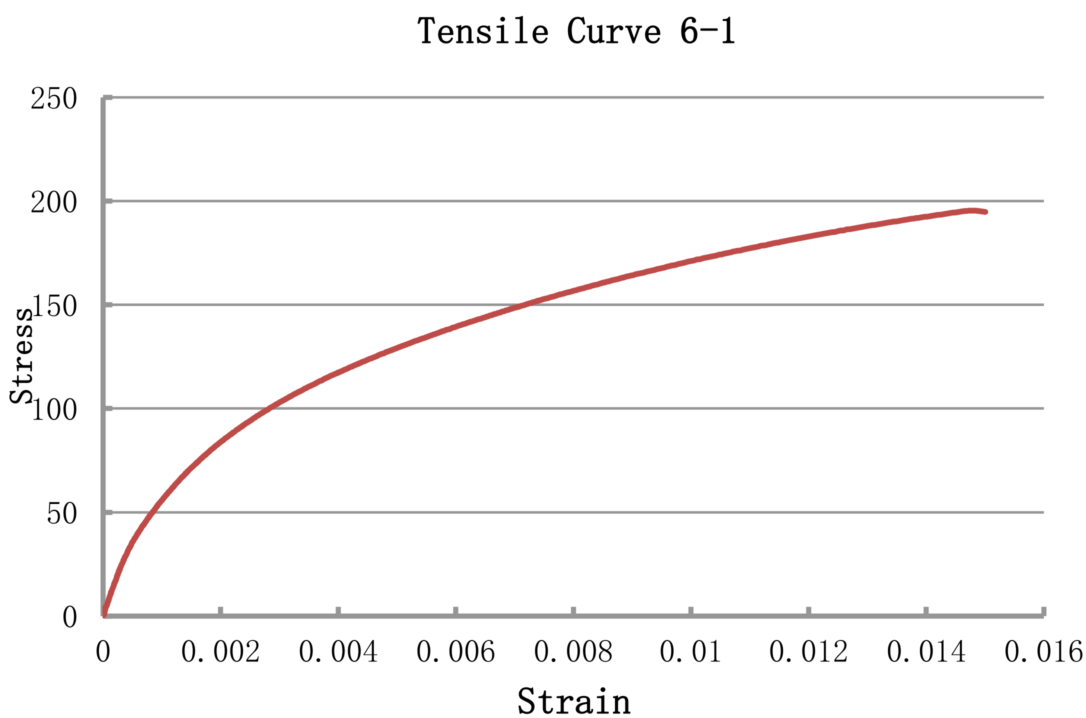

Figure I-34 Tensile curve for sample 1 of DOE#2 test 6

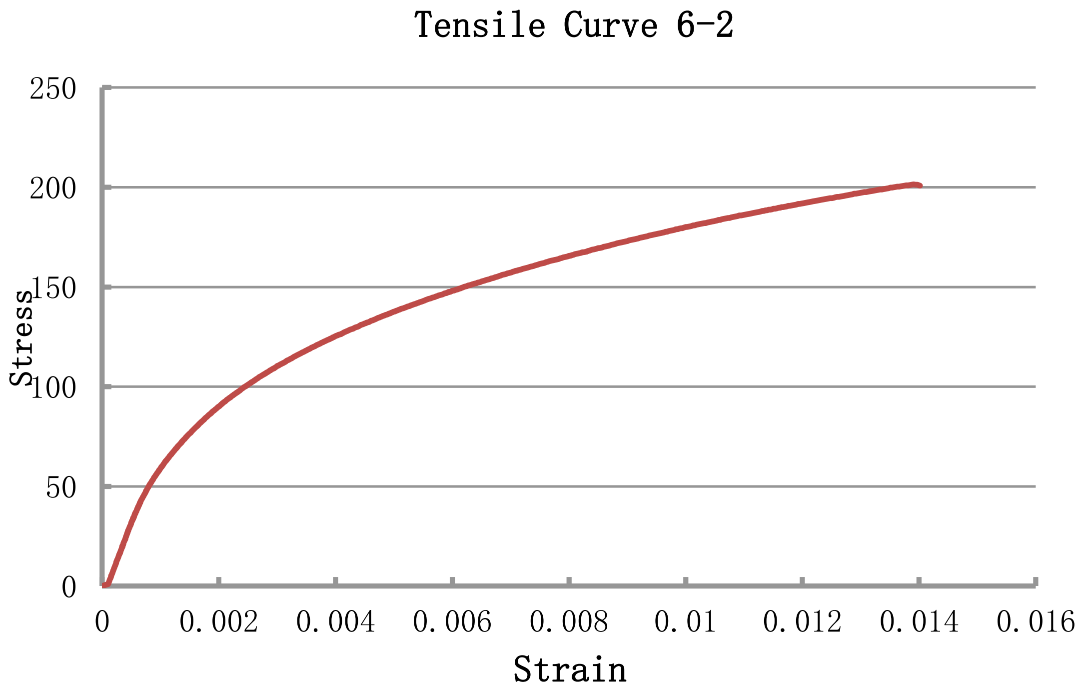

Figure I-35 Tensile curve for sample 2 of DOE#2 test 6

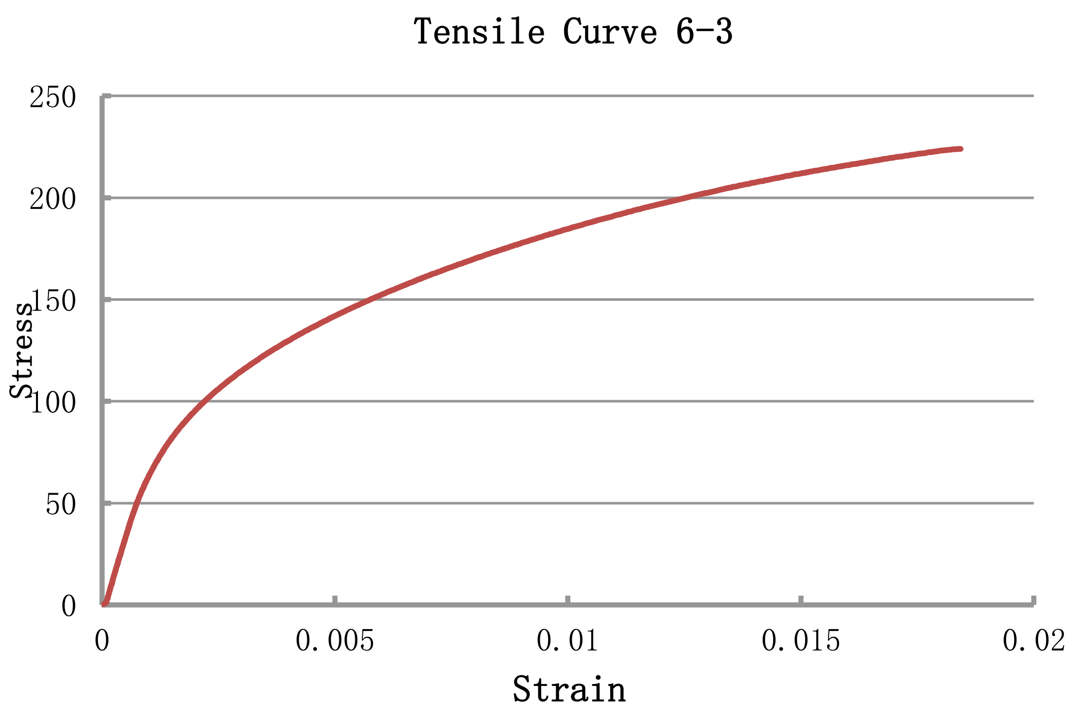

Figure I-36 Tensile curve for sample 3 of DOE#2 test 6

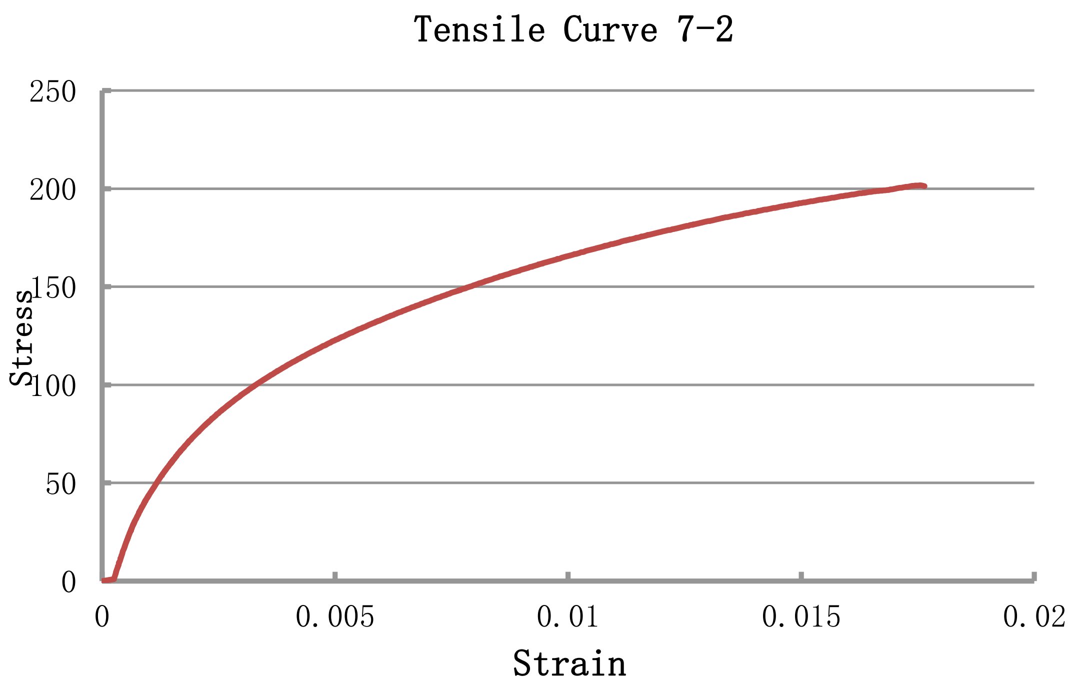

Figure I-37 Tensile curve for sample 2 of DOE#2 test 7

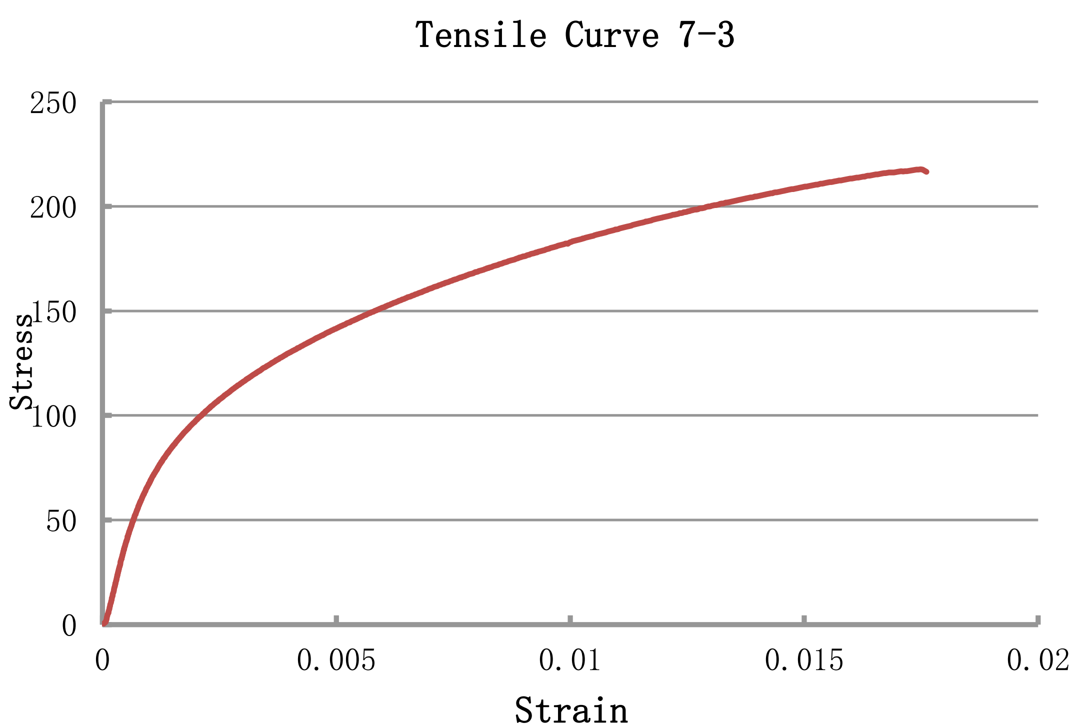

Figure I-38 Tensile curve for sample 3 of DOE#2 test 7

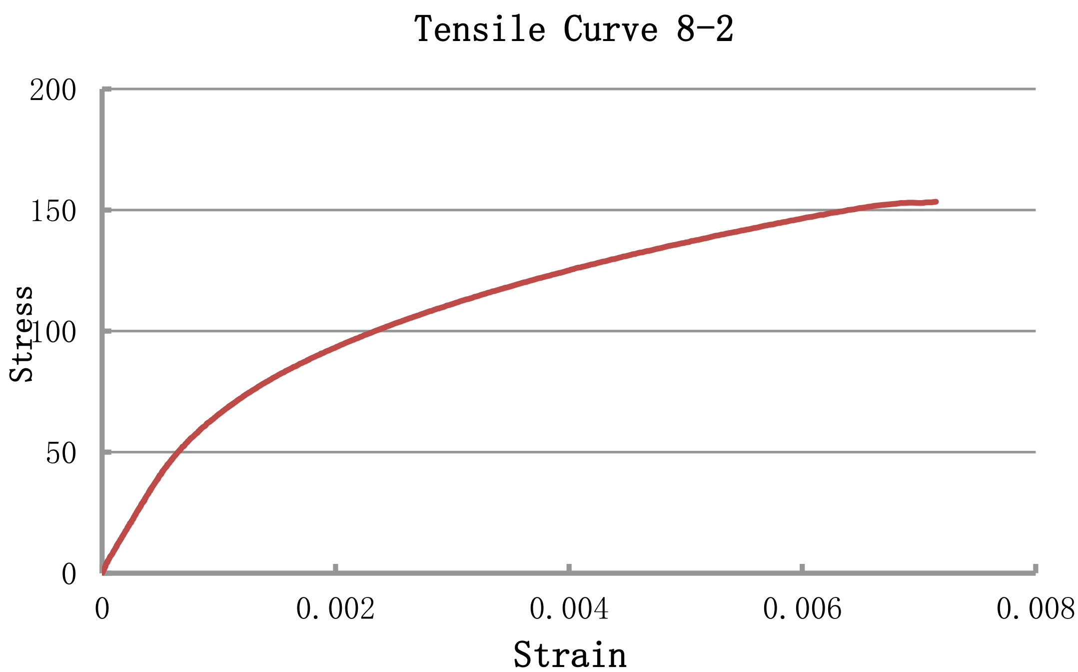

Figure I-39 Tensile curve for sample 2 of DOE#2 test 8

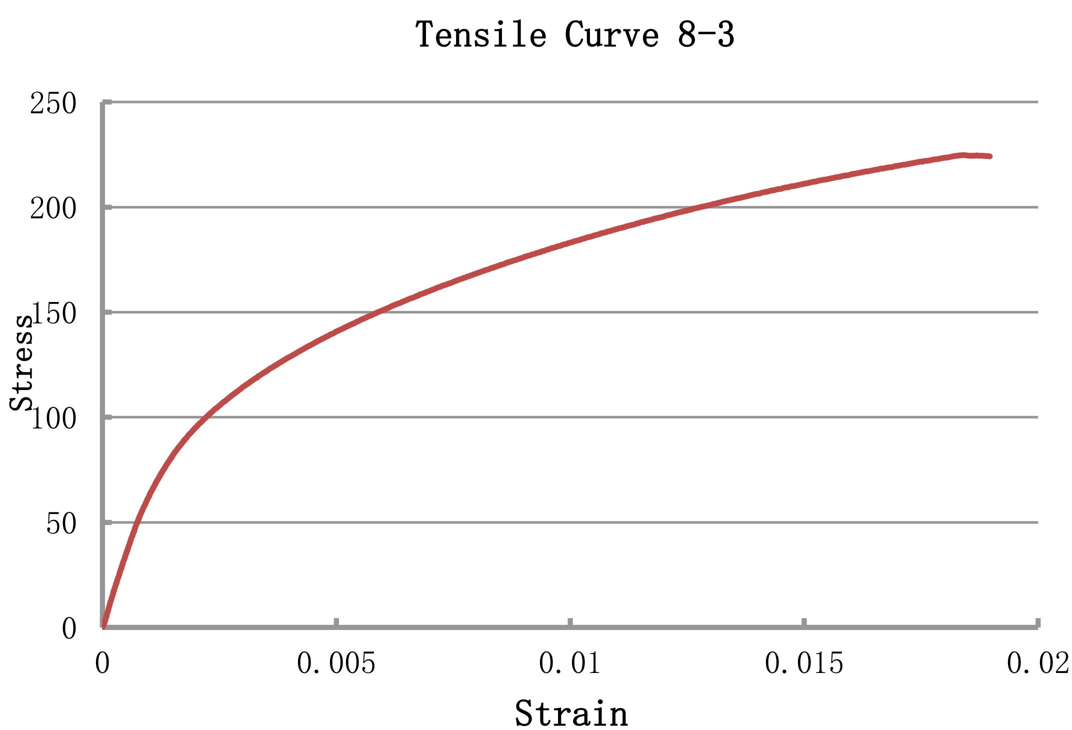

Figure I-40 Tensile curve for sample 3 of DOE#2 test 8

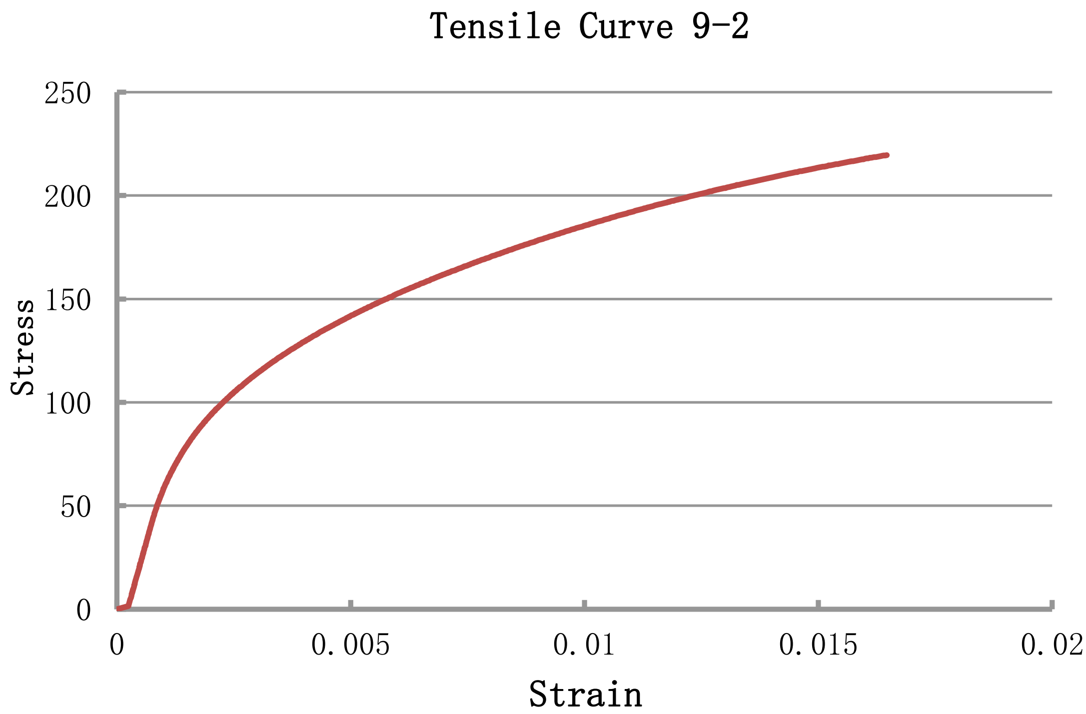

Figure I-41 Tensile curve for sample 2 of DOE#2 test 9

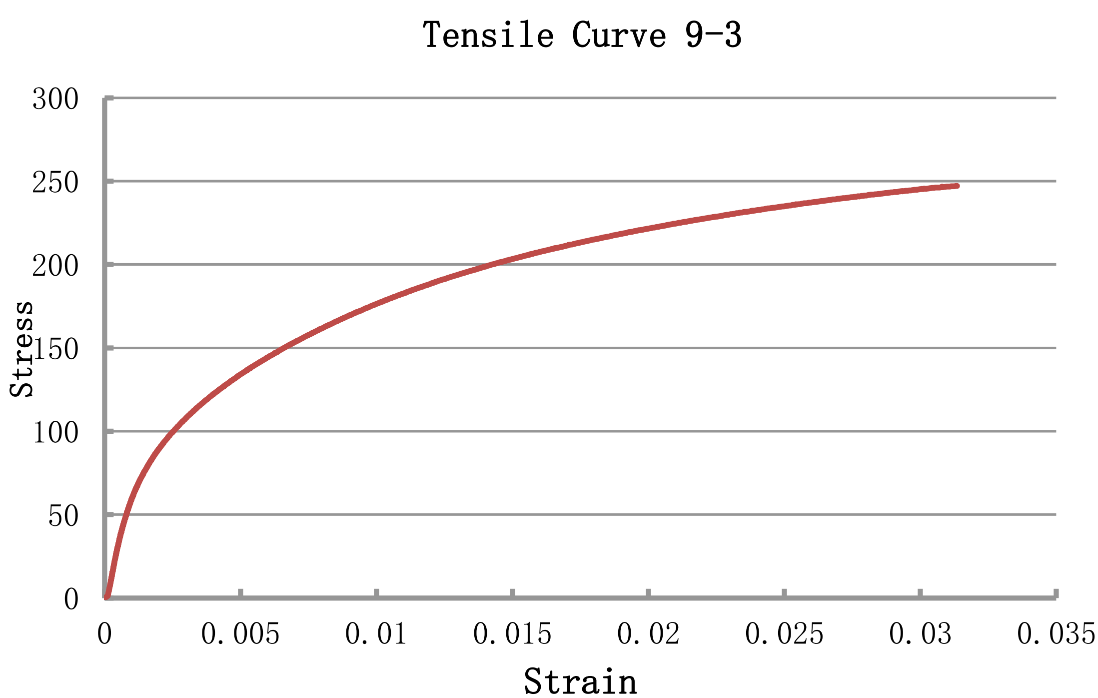

Figure I-42 Tensile curve for sample 3 of DOE#2 test 9

Appendix II： Original Data of Corrosion Testing

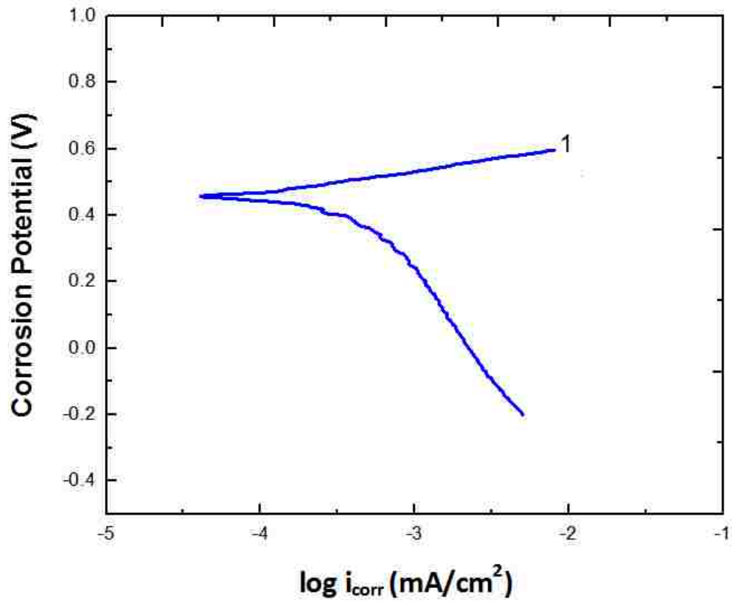

Figure II-1 Typical corrosion curve of experiment No.1

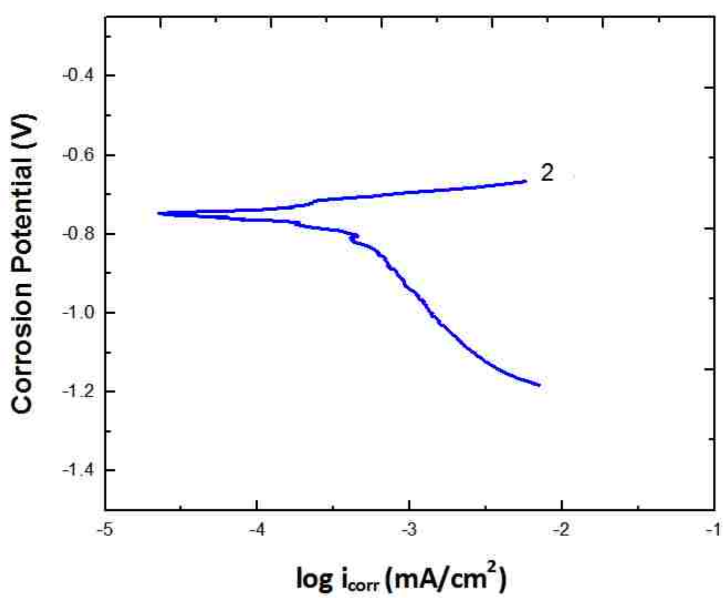

Figure II-2 Typical corrosion curve of experiment No.2

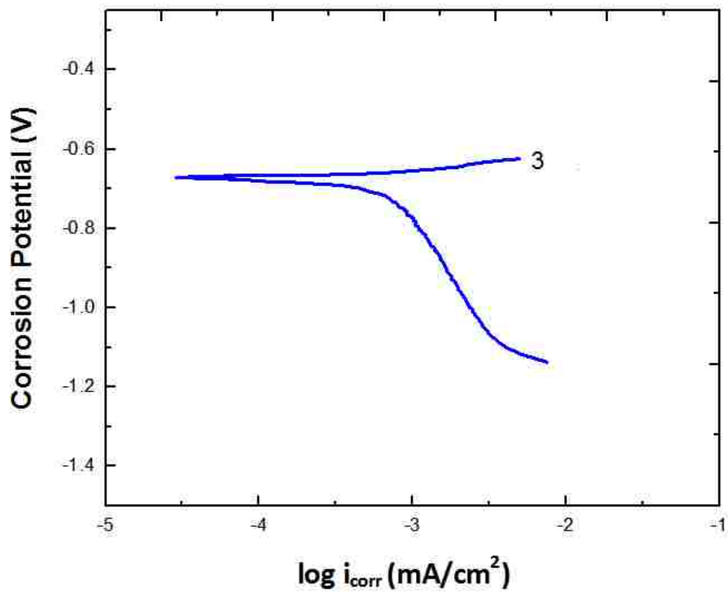

Figure II-3 Typical corrosion curve of experiment No.3

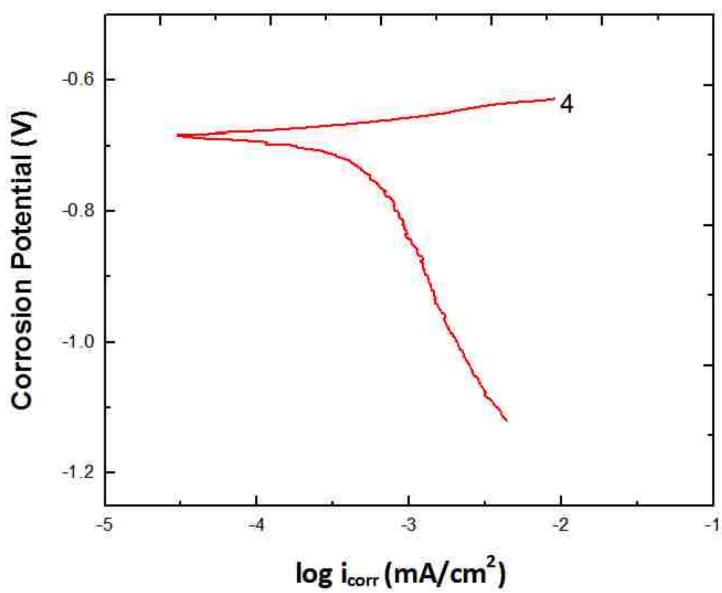

Figure II-4 Typical corrosion curve of experiment No.4

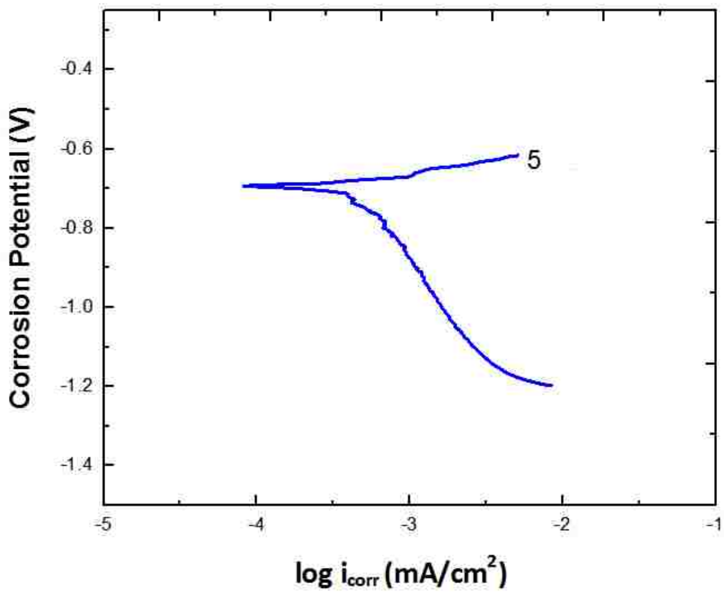

Figure II-5 Typical corrosion curve of experiment No.5

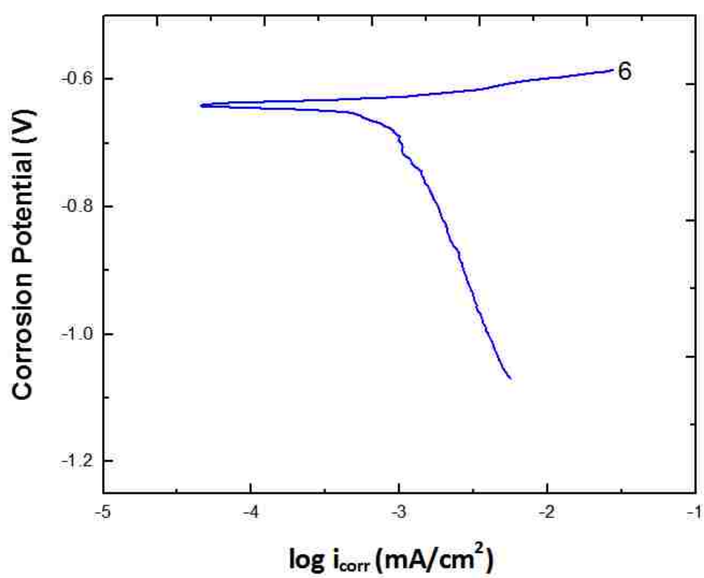

Figure II-6 Typical corrosion curve of experiment No.6

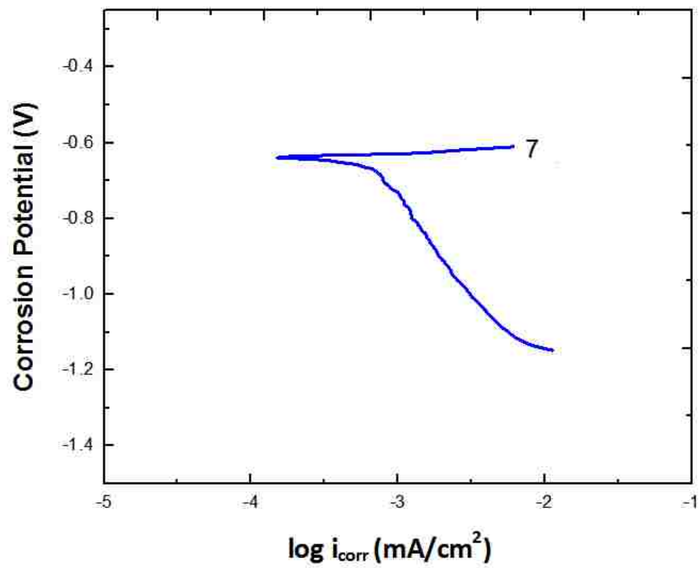

Figure II-7 Typical corrosion curve of experiment No.7

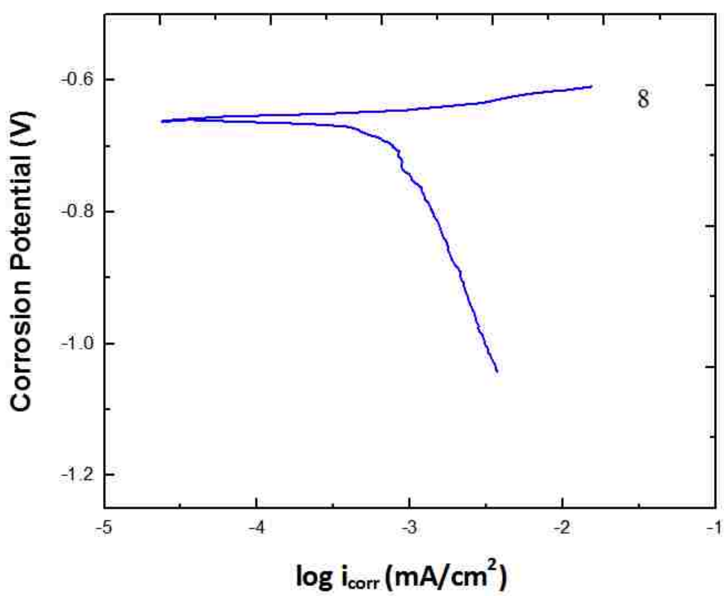

Figure II-8 Typical corrosion curve of experiment No.8

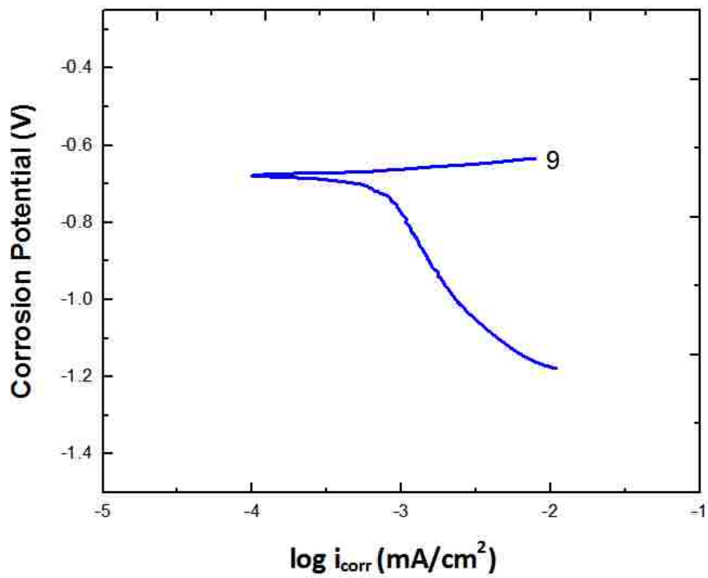

Figure II-9 Typical corrosion curve of experiment No.9

Appendix III : Copyright releases from publications

Chapter II

From:	Matt Baker <mbaker@tms.org>
Subject:	RE: Permission
Date:	Tue, 28 Apr 2015 at 2:17 pm
To:	Bojun Xiong <bojun@uwindsor.ca>

Dear Bojun,

The letter serves as formal permission for you to publish the pre-publication author version of your paper "Recycling of Die Cast Aluminum A380 Machining Chips" in your thesis. The paper should include a credit line that indicates that the final version was published in Light Metals 2015, copyright 2015 by The Minerals, Metals & Materials Society. Please let me know if I can be of further assistance.

Best regards,

Matt Baker

Matt Baker | The Minerals, Metals & Materials Society

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Chapter III

From:	**CIRP-JMST** <laszlo.monostori@sztaki.hu>
Subject:	Submission confirmation
Date:	Tue, 31 Mar 2015 at 1:57 pm
To:	Bojun Xiong <bojun@uwindsor.ca>

Dear Mr. Bojun Xiong,

We have received your article "Process Optimization for Recycling of Machining Chips of Die Cast Aluminum Alloy A380" for consideration for publication in CIRP Journal of Manufacturing Science and Technology.

Your manuscript will be given a reference number once an editor has been assigned.

To track the status of your paper, please do the following:

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CIRP Journal of Manufacturing Science and Technology.

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