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" THE CONTINUOUS PROCESS FOR THE SURFACE GRAINING OF ALUMINIUM FOIL FOR ALUMINIUM OFFSET LITHOGRAPHIC PLATES USED IN DUPLICATING MACHINE".

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi- 110001, India an Indian registered body incorporated under the Registration of Societies Act (Act of XXI of 1860).

The following specification describes the nature of this invention.

PRICE : TWO RUPEES

150486

This is an invention by Balkunje Anantha Shenoi, Scientist, Ramachandra Subramanian, Scientist, Venkatasubramanian Lakshminarasimhan, Scientist, Ramasubbu Venkatachalam, Senior Scientific Assistant, and Devaraj Kanakaraj, Senior Scientific Assistant, all of Central Electrochemical Research Institute, Karaikudi-623006, all Indian nationals.

The following specification relates to the method of continuously graining aluminium foils and providing a hard surface oxide coating thereon for use as off-set lithographic plates in printing industry.

The method consists in passing the aluminium foils through a series of tanks containing (1) an alkaline degreasing solution, (2) rinse water (3) nitric acid desmutting solution (4) rinse water (5) a dilute solution of hydrochloric acid and addition agents within specified composition ranges for electrochemical graining (of the surface) of the aluminium foil (6) rinse water (7) a solution containing chemicals to provide a hard surface oxide coating either chemically or electrochemically and (8) rinse water and air ~~drying~~ dryer.

The tank containing hydrochloric acid and the addition agents in the specified ranges is provided with a V shaped counter electrode; and an alternating current from a 3 phase 50 cycle transformer and of such intensity and electrical pressure to provide a current density on the foil in the range of 0.5 to 1.5 ampere per

square centimetre is passed. The line speed of travel of coil can be varied from 20 to 60 centimetres per minute. The line pass (i.e.) the length of the foil immersed in the electrolyte at a given instant is varied from 60 cms to 90 cms.

The oxide coating on the graining aluminium foil is formed either chemically or electrochemically in the tank containing either chemicals for conventional oxide coating process or chemicals for electrochemical anodising -- such as sulphuric acid, oxalic acid chromic acid or borates. The anodic coating may be obtained either by using alternating current from a step down transformer or direct current from a d.c. source such as rectifiers. The operating conditions and concentration ranges of chemicals for anodising are the same as in standard coventional anodising techniques hitherto adopted.

The chemical process for forming the oxide coating is by conversion coating process, the chemicals for the respective conversion coatings in the usual range and operating temperature and time being the same as in the standard procedures for obtaining such coatings.

The oxide coating on the grained aluminium foil may also be formed chemically by exposing the foil to hot water or steam at temperatures above 80°C for a specified time, the oxide coating formed being of the Boehimite type.

After the formation of the oxide coating to specified thickness by anyone of the above methods, the grained aluminium foil is rinsed dried and rolled. No sealing is done after the oxide coating formation.

Hitherto, the graining of the aluminium plates for lith graphic printing plates have been carried out by

1. Mechanical methods like wet graining in graining machines with glass, silica or marble balls and a suitable abrasive of given mesh size.
2. Mechanical methods like brush graining using special nylon brushes in brush graining machines.

3. Electrochemical graining in batches in solutions of hydrochloric acid with or without suitable addition agents.

This is open to the object that methods 1 and 2 are slow and require 60 to 120 minutes per plate to get the grained surface.

Method 1 is not suitable for graining this aluminium foils as these foils are easily damaged. On thicker aluminium sheets the grain size distribution is not uniform and the grains are also coarse. There is also the disadvantage of the abrasives getting embedded on to the aluminium surface.

Method 2 even though can be adopted for foils by suitable backing of the foils suffers from the disadvantage that it is very slow.

Method 3 is only a batch process. The time of graining is 10 to 15 minutes, and the voltage required is more than 20 volts (Indian Pat. No. 96606)

By the addition of certain organic compounds and narrowing down the spacing between the working electrode and counter electrode to a distance of less than 5 cms the time of graining had been brought down considerably in a subsequent patent (Pat.No. awaited) The minimum time claimed is 2 minutes with an a.c. voltage of 30 volts or more. The method, though good for aluminium sheets of thickness 1 mm and above, is not suitable for thin foils, and positioning of thin foils (.1 mm to 0.3 mm) of large areas within close distance of 5 cms apart is technically very difficult. Hence this method suffers from the following disadvantages when adopted for thin foils.

1) non uniform current distribution resulting in non uniform grain structure
2) fast dissolution of the edges and corners
3) The probability of shorting of electrodes and 'arcing' will always be high, which will result in a possible damage to the plates as well as the electrical equipments.

The object of the present invention is to obviate these disadvantages by

(1) making the process a continuous one, in which the roll of aluminium foil will be driven through a series of processing tanks continuously at a selected speed of travel.

(2) formulating electrolytes of such chemicals and compositions that the etching of the surface is controlled to suit the specified grain density and size for the lithographic plates.

(3) selecting the line pass dimensions and line speed, the time of graining and time of anodising or of chemical oxide coating can be reduded to less than one minute.

To these ends the invention broadly consists in treating the aluminium foil successively as described hereunder.

1. The surfance contaminants and greases, lubricating oils etc., are removed from the surface of the foil by soak cleaning in an alkaline solution containing 3 to 10% by weight of NaOH at 30 to 35°C for 2 to 5 minutes.

2. This is followed by rinse in running water

3. The surface of the foil is desmutted in 5% nitric acid at 30 to 35°C for 2 minutes.

4. Rinse in tap water.

5. Electrochemically grained in a solution of hydrochloric acid of 0.25 N to 0.75N strength also containing a weak acid, such as boric acid, oxalic acid, tartaric acid, succinic acid or their salts in the range of 0.5% to 2% by weight. The necessary alternating current of 50 Hz is supplied from a 3 phase source. The driving potential of the supply is maintained between 4 volts and 20 volts depending on (1) the current density required to get the required grain size and distribution over foils and (2) the inter electrode spacing. The range of current density under which the graining takes place with uniform distribution of the grain size is between 0.5 amp to 1.5 amp. per square centimetere of the exposed surface area of the foil in the graining tank. The time required to get the desired grain size and distribution can be varied between 45 seconds and 180 seconds. The temperature of electrolyte is maintained between 25°C and 50°C the higher temperatures favouring a shorter time.

(6) The electrochemical graining step is followed by rinsing in running water.

(7) The oxide coating (to produce a hard surface) is formed by anodising in conventional anodising electrolytes like sulphuric acid, chromic acid, oxalic acid under the usual operating conditions either using an alternating current or a direct current. The oxide coating may also be formed by chemical methods such as M.B.V. process or in hot water kept at above 80°C.

(8) After the formation of oxide coating of required thickness, the foil is rinsed in running water and dried.

The process here before described, though suitable for continuous treatment of thin aluminium foils of 75 micron thickness, is by no means restricted to continuous treatment of foils alone. It has also been found that the sequence of operations here before described is also applicable to thicker aluminium sheets by batch process.

The following typical examples are given to illustrate the invention:

EXAMPLE - 1

Commercial pure Aluminium foil (2S) of thickness 90 microns is given pretreatment in 3% sodium hydroxide for a period of 90 seconds, at a temperature of 35° ± 1°C, rinsed in running water, desmutted in 5% V/V concentrated nitric acid (1.34 to 1.36 gm/cc) in water for 60 seconds at 30°C, rinsed and electrolytically grained in the following electrolyte as follows:

Electrolyte	0.5 N	Hydrochloric acid
	+ 2%	Boric acid
Temperature	40°C	
Time	60 seconds	
Power source	3 phase 50 Hz A.C	
Counter electrode	graphite	
Working electrode	Aluminium foil (2S) 90 micron thick of size 2.5 cm x 10 cm.	

Working electrode current density	0.5 amp/cm^2
Voltage across the electrodes	10 volts a.c

After the electro graining the foil is rinsed in running tap water, then in distilled water and anodised in the following solution.

Chromic acid	10 gms per litre of water
Temperature	35°C
D.C. voltage	30 V
Time	10 minutes

Hot rinsed and dried.

EXAMPLE -2

76 micron foil, 100 mm wide commercial purity Aluminium (3S) in the form of a coil is electrograined in a continuous processing plant with the line speed of 46 cm per minute and line pass in the graining tank is 60 cm. The pretreatment condition is the same as stated in Example 1.

The electrolyte for electrograining is as follows:

Electrolyte: 0.65 N Hydrochloric acid
2% W/v citric acid

Temperature : 45 °C

Current through the cell: 500 amps.

Current source : 50 Hz 3 phase continuously variable a.c.

Counter electrode: Thick Al sheet

Cell voltage : 9 volts

Spacing of electrodes: 5 cm

The foil is rinsed in tap water and deionised water and then treated to get a Boehmite aluminium oxide coating as follows:

Solution	deionised water
Temperature	90 to 95 °C
Time	3 minutes

The coil is then air dried and rolled up in the take off roll.

EXAMPLE - 3

90 micron foil and 50 mm width 2S purity was treated in the continuous processing plant as before upto the pretreatment stages. Then electrograined in the tank as follows:

Electrolyte:	3% V/V Hydrochloric acid in water(1.18 g/cc) containing 20 grams of oxalic acid.
Temperature	35°C
Current through the cell.	350 amps.
Current source	50 Hz 3 phase stepdown transformer with stepless regulator control.
Counter electrode	Graphite panels
Cell voltage	15 volts
line speed.	60 cms per minute

Spacing of the electrodes: 4.5 cms

The foil is rinsed in running tap water and in deionised water and treated in a solution containing 5% W/V of sodium carbonate plus 2% W/v of Potassium chromate at 95 °C for 2 minutes. The foil is then rinsed, dried and wound on a spool.

The following are the advantages of the invention:

1) In the continuous prodessing, thin aluminium foils are held and moved in tension at a fixed distance as close as required from the counter electrodes without any chance for shorting or arcing.
2) As the electrolyte near the electrode surface is under constant agitation the current density is higher than in batch process and the time of graining is shortened to less than 1 minute.
3) Because of electrode movement and agitation thereof the grainning is finer and more uniform without preferential dissolution at edges or corners.
4) The rate of production is high

5) Both the surfaces are uniformly grained such that two sides of the foil can be used for printing in duplicating machines.

6) Damages due to manual handling is completely eliminated.

7) The process is also amenable for batch processing of thicker aluminium sheets which cannot be obtained in the form of rolled coils.

8) In the present invention higher concentrations of hydrochloric acid is used which makes the problem of electrolytic solution control and maintanance less critical than in the earlier methods.

9) The voltage necessary for graining even in extreme conditions of high current densities is only 10 volts in comparison with the 20 to 40 V claimed in earlier work.

Dated this 26th day of December, 1978.

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150486

THE PATENTS ACT. 1970

COMPLETE SPECIFICATION

(Section—10)

" THE CONTINUOUS PROCESS FOR THE SURFACE GRAINING OF ALUMINIUM FOIL FOR ALUMINIUM OFFSET LITHOGRAPHIC PLATES USED IN DUPLICATING MACHINE".

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi- 110001, India an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed :—

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150486

This is an invention by Balkunje Anantha Shenoi, Scientist; Ramachandra Subramanian, Scientist; Venkatasubramanian Lakshminarasimhan, Scientist; Ramasubbu Venkateshalam, Scientist and Devraj Kanagaraj, Senior Scientific Assistant, all of Central Electrochemical Research Institute, Karaikudi 623 006, and all are Indian nationals.

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This invention relates to a continuous process for the production of grained aluminium foils and providing a hard surface oxide coating thereon for use as offset lithographic plates in printing industry or in office duplicating machines.

Hitherto the graining of the aluminium plates for lithographic printing plates have been carried out by

i) mechanical methods like wet graining in graining machines with glass, silica or marble brass balls and a suitable abrasive of given mesh size

ii) mechanical methods like brush graining using special nylon brushes in brush graining machines

iii) electrochemical graining in batches in solutions of hydrochloric acid with or without suitable addition agents.

This is open to the objection that methods 1 and 2 are slow and require 60 to 120 minutes per plate to get the grained surface.

Method 1 is not suitable for graining thin aluminium foils as these foils are easily damaged. On thicker aluminium sheets the grain size distribution is not uniform and the grains are also coarse. There is also the disadvantage of the abrasives getting embedded on to the aluminium surface.

Method 2 even though can be adopted for foils by using a suitable backing of the foils, suffers from the disadvantage that it is very slow.

Method 3 is only a batch process. The time of graining is 10 to 15 minutes and the voltage required is more than 20 volts as described in Indian Patent No 96606. By the addition of certain organic compounds and narrowing down the spacing between the working electrode and counter electrode to a distance of less than 5 cms the time of graining had been brought down considerably by a further modification of the said process. The minimum time required is still 2 minutes with an a.c. voltage of 20 volts or more. This method though good for aluminium sheets of thickness 1 mm and above, is not suitable for thin foils and positioning of thin foils

(0.1 mm to 0.3 mm) of large areas within close distance of 5 cms apart is technically very difficult. Hence this method suffers from the following disadvantages when adopted for thin foils

i) non uniform current distribution resulting in non uniform grain structure

ii) fast dissolution of the edges and corners and the

iii) the probability of shorting of electrodes and 'arcing' will always be high, which results in a possible damage to the plates as well as the electrical equipments

The object of the present invention is to obviate these disadvantages by

i) making the process as a continuous one, in which the roll of aluminium foils is driven through a series of processing tanks continuously at a selected speed of travel

ii) formulating electrolytes of such chemicals and compositions that the etching of the surface is controlled to suit the specified grain density and size for the lithographic plates and

iii) by selecting the line pass dimensions and line speed, the time of graining and time of anodising or of chemical oxide coating can be reduced to less than three o minutes.

This invention accordingly provides a continuous process for the production of grained aluminium foils for use in offset litho printing or in office duplicating machine which consists in subjecting aluminium foil from a roll to the successive steps of (i) cleaning in a alkaline solution of sodium hydroxide (ii) washing in running water, (iii) treatment in nitric acid (iv) washing in running tap water (v) subjecting the treated aluminium foil to electrochemical graining in the electrolytic bath consisting of a solution of a hydrochloric acid and a weak aliphatic acid or inorganic acid at a current density of 0.6 to 1.5 A/cm^2 sq.cm derived from a.c. source at a temperature of between 25 - 50°C (vi) rinsing the thus electrochemically grained foil in running tap water, (vii) treating in nitric acid (viii) washing in running tap water and then in deionized

water, (ix) forming an oxide coating on the grained foil surface either in a conventional anodising electrolyte under usual conditions or by chemical oxidation in a conventional chemical conversion coating process and (x) washing the foil in deionised water to obtain the aluminium foil with the desired size and distribution of grains.

To these ends the process of this invention broadly consists in treating the aluminium foil successively as described hereunders

1) The surface contaminants and greases and lubricating oils are removed from the surface of the foil by soak cleaning in an alkaline solution containing 3 to 10% by weight of NaOH at 30 to 35°C for 2 to 5 minutes.

2) This is followed by rinse in running water

3) The surface ~~xxxxxxxxxxxx~~ of the foil is desmutted in 5% nitric acid (Sp.gr 1.41 - 1.42) at 30 to 35°C for 2 to 5 minutes.

4) Rinse in tap water

5) Electrochemically grained in a solution of hydrochloric acid of strength 0.25N to 0.75N containing a weak organic or inorganic acid. The organic acid may be an aliphatic having one or more COOH group with or without hydroxyl groups in the chain of which the chain length is limited to 5 to 10 carbon atoms such as formic acid, oxalic acid, succinic acid, tartaric acid, citric acid, lactic acid or their salts. The inorganic acid may be boric acid. The necessary alternating current of 50 Hz is supplied from a 3 phase source. The driving potential of the supply is maintained between 4 volts and 20 volts depending on (a) the current density required to get the required grain size and distribution over foils and (b) the inter electrode spacing. The range of current density under which the graining takes place with uniform distribution of the grain size is between 0.5 amp to 1.5 amp per square centimetre of the exposed surface area of the foil in the graining tank. The time required to get the desired grain size and distribution can be varied between 45 secs and 120 secs. The temperature of the electrolyte is maintained between 25°C and 50°C, the higher temperature favouring a shorter time.

6) The electrochemical graining step is followed by rinsing in running water.

7) The grained surface is then treated in 10% V/V nitric acid(Sp. gr. 1.41 - 1.42) followed by running tap water and finally in deionised water.

8) The oxide coating(to produce a hard surface) is formed by anodising in conventional anodising electrolytes like sulphuric acid, chromic acid, oxalic acid under the usual operating conditions either using an alternating current or a direct current. The oxide coating may also be formed by chemical methods such as ASV. process or in hot water kept at above 80°C.

9) After the formation of oxide coating of required thickness the foil is rinsed in running water followed by washing in deionised water and dried.

The process hereinbefore described, though suitable for continuous treatment of thin aluminium foils of 75 to 200 micron thickness, is by no means restricted to continuous treatment of foils alone. It has also been found that the sequence of operations hereinbefore described is also applicable to thicker aluminium sheets by batch process.

A continuous process for the production of grained aluminium foils for use in offset lithoprinting or in office duplicating machine which consists of (i) cleaning in alkali solution containing 3 to 7% W/V sodium hydroxide kept at 25-50°C (ii) washing in running tap water (iii) treatment in 5-15% V/V (Sp.gr. 1.41-1.42) nitric acid at 30-35°C (iv) washing in running tap water wherein the treated aluminium foils is electrochemically grained in a solution of hydrochloric acid of strength ranging between 0.25N and 0.75N containing 0.5 to 2% of a weak aliphatic acid or inorganic acid of which the organic acid containing 2-10 carbon atoms with one or more carboxyl group with or without hydroxyl group such as acetic acid, formic acid, tartaric acid, lactic acid, citric acid or their salts, the inorganic acid being boric acid at a current density 0.5 to 1.5A/sq.cm derived from a three phase 50 cycle a.c. source, the temp. of the said graining

solution is kept between 25-50°C for a period of 45 to 100 secs (v) rinsing in running tap water (vi) treated in a nitric acid of sp.gr. 1.41 - 1.42 (5 - 15% V/V) kept at 30-35°C (vii) washing in running tap water and then in deionized water (viii) forming an oxide coating either in a conventional anodising electrolyte like sulphuric acid of sp.gr. 1.84 (10% V/V), oxalic acid (5% W/V) chromic acid 10% W/V under usual conditions using either a.c. or d.c. current or by chemical oxidation in hot water kept at 85-95°C for a period of 90 - 180 secs or in conventional chemical conversion coating process (ix) washing the foil in deionized water to get the desired size and distribution of grains.

The following typical examples are given to further illustrate the inventions

Commercial pure aluminium foil (2S) of thickness 90 microns is given pretreatment in 3% sodium hydroxide for a period of 90 seconds, at a temperature of 35°C ± 1°C rinsed in running water, desmutted in 5% V/V nitric acid (Sp.Gr. 1.41 - 1.42) in water for 60 seconds at 30°C, rinsed and electrolytically grained in the following electrolyte as follows

Electrolyte	0.5 N hydrochloric acid + 2% Boric acid
Temperature	40°C
Time	60 seconds
Power source	3 Phase 50 Hz A.C.
Counter electrode	Graphite
Working electrode	Aluminium foil (2S) 90 micron thick of size 2.5 cm x 10 cm
Working electrode current density	0.5 amp/cm^2
Voltage across the electrodes	10 volts a.c.

After the electro graining the foil is rinsed in running w tap water then in distilled water and anodised in the following solution.

Chromic ac[illegible]	10 gms per litre of water
Temperature	35°C

D.C. Voltage	30 V
Time	10 minutes

Hot rinsed and dried.

Example 2

75 micron foil, 100 mm wide commercial purity aluminium (3S) in the form of a roll is electrograined in a continuous processing plant with a linear speed of 46 cm per minute and line pass in the graining tank is 60 cm. The pretreatment conditions is the same as stated in example 1.

The electrolyte for electrograining is as follows:

Electrolyte	0.65N hydrochloric acid + 2% W/V citric acid
Temperature	45°C
Current density	1.0 amp/dm^2
Current source	50 Hz 3 phase continuously variable a.c
Counter electrode	Thick Al sheet
Cell voltage	9 volts
Spacing of electrodes	5 cm

The foil is rinsed in tap water and deionised water and then treated to get a Boehmite aluminium oxide coating as follows:

Medium	Deionised water
Temperature	90 to 95°C
Time	3 minutes

The foil is then air dried and rolled up in the take up roll.

Example 3

90 micron aluminium foil of 50 mm width of 2S grade was treated in the continuous processing plant as before upto the pretreatment stages. Then electrograined in the tank as follows:

Electrolyte	3% W/V hydrochloric acid in water (Sp.gr. 1.18) containing 20 gms of oxalic acid/litre.

Temperature	35°C
Current source	50 Hz 3 phase septdown transformer with stepless regulator control
Current density	1.5 amp/cm^2
Counter electrode	Graphite panels
Cell voltage	15 volts
Line speed	60 cms per minute
Spacing of the electrodes	4.5 cm

The foil is rinsed in running tap water and in deionised water and treated in a solution containing 5% V/V of sodium carbonate plus 2% W/V of potassium chromate at 95°C for 2 minutes. The foil is then rinsed, dried and wound on a spool.

The following are the advantages of the inventions

1) In the continuous processing, thin aluminium foils are held and moved in tension at a fixed distance as close as required from the counter electrodes without any chance for shorting or arcing.

2) As the electrolyte near the electrode surface is under constant agitation the current density is higher than in batch process and the time of graining is shortened to less than 1 minute.

3) Because of electrode movement and agitation thereof the graining is finer and more uniform without preferential dissolution at edges or corners.

4) The rate of production is high

5) Both the surfaces are uniformly grained such that two sides of the foil can be used for printing in duplicating machine.

6) Damage due to manual handling is completely eliminated

7) The process is also amenable for batch processing of thicker aluminium sheets which cannot be obtained in the form of rolls.

8) In the present invention higher concentration of hydrochloric acid are used which makes the problem of electrolytic solution control and maintenance less critical than in the earlier methods.

9) The voltage necessary for graining even in extreme conditions of high current densities is only 10 volts in comparison with the 20 to 40V claimed in earlier work.

WE CLAIM:

1) A continuous process for the production of grained aluminium foils for use in offset lithoprinting or in office duplicating machine which consists in subjecting aluminium foil from a roll to the successive steps of (i) cleaning in an alkali solution of sodium hydroxide, ii) washing in running tap water, iii) treatment in nitric acid iv) washing in running tap water v) subjecting the treated aluminium foil to electrochemical graining in an electrolytic bath consisting of a solution of hydrochloric acid and a weak aliphatic acid or inorganic acid at a current density 0.5 to 1.5 A/sq.cm derived from a.c. source at a temperature between 25-50°C vi) rinsing the thus electrochemically grained foil in running tap water vii) treating in nitric acid, viii) washing in running tap water and then in deionised water, ix) forming an oxide coating on the grained foil surface either in a conventional anodising electrolyte under usual conditions or by chemical oxidation in a conventional chemical conversion coating process and x) washing the foil in deionised water to obtain the aluminium foil with the desired size and distribution of grains.

2) A process as claimed in claim 1 wherein the electrochemical graining treatment in step (v) is carried out using a three phase 50 Hz a.c. source at a current density of 0.5 A/sq.cm.

3) A process as claimed in claim 2 wherein the temperature of the electrolytic solution is kept at 40°C.

4) A process as claimed in any of the preceding claims wherein the foil is processed at a linear speed of 35-70 cm/minute.

4) A process as claimed in claim (1) wherein the electrolytic solution used in step (v) consists of hydrochloric acid in the concentration range between 0.25 to 0.75 N containing 0.5 to 2% of an aliphatic acids with 2- 10 carbon atoms having one or more carboxylic group with or without a hydroxyl group such as acetic acid, formic acid, tartaric acid, lactic acid, citric acid, succinic acid or their salts or an inorganic acid such as boric acid.

5) A continuous process for the production of grained aluminium foils for use in offset litho printing or in office duplicating machine substantially as herein described and illustrated.

Dated this 18th day of March 19[illegible]

Scientist(Patents)
Council of Scientific & Industrial Research