GOVERNMENT OF INDIA : THE PATENT OFFICE : 214, ACHARYA JAGADISH BOSE ROAD, CALCUTTA-17.

Complete Specification No. 133404,

Application and Provisional Specification filed on 29th October 1971.

Complete Specification filed on 27th January 1973.

Acceptance advertised on 20th July 1974.

Index at acceptance—12C[XXXIII(2)], 142 [LXVI(6)], 154C [XXXVII(1)].

PROVISIONAL SPECIFICATION

IMPROVEMENTS IN OR RELATING TO THE SPANGLE FINISH ON TINNED STEEL.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH,

RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES Act (ACT XXI OF 1860).

The following specification describes the nature of this invention :—

This is an invention by BALKUNJE ANANTHA SHENOI, Scientist, Mrs. INDIRA RAJAGOPALAN, Scientist and HANDADY VENKATAKRISHNA UDUPA, Director, all of the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, and all are Indian Nationals.

This invention relates to improvements in or relating to decorative effects on electrotinned or hot dipped tin plates on steel or any other metal.

Hitherto it has been proposed to get patterns on tinned steel by costly and laborious engraving procedures or by elaborate electromarking procedures. To adopt the above procedures, the thickness of tin coatings should be at least 10 thou or more. The reproducibility and reliability of the markings by the above procedures depend upon a number of factors and mainly upon the skill of the labourer. Moreover, the above procedures offer some mechanical defects after the final process. Moreover, the finishes obtained by the above processes do not have sufficient eye appeal also. Because of the fact that higher thickness of plates are required and the manual skill involved in the above processes, these are highly costly too.

Hence, a process has been worked out by a relatively simple technique and the pattern that is obtained by the present process cannot even be duplicated by the other engraving procedures. The object of the invention is to obviate the disadvantages by resorting to recrystallisation procedures (i.e. by heating the hot dipped or electrotinned steel to the recrystallisation temperature of tin), etching the recrystallised tinned steel by choosing suitable etchants so as to reveal the grain patterns, followed by flash plating of tin and colouring the same to give rise to multicoloured, multigrained structured patterns on tinned steel specimens. The etching procedure is to be carefully chosen, making use of the chemical amisotropy of the etchant. The etchant should not be too corrosive to attack the tinned steel in no time or too slow in which case one will get a uniform dissolution leading to a smooth surface and not a multitoned effect.

To these ends, the invention broadly consists in electro plating of tin on steel or hot dipping of tin on steel followed by recrystallisation and revelation of grains so as to product a multitoned effect and is coloured by using suitable treatment (electrochemical, chemical or physical methods).

The Plating Procedures

The mild steel panels of required size are polished with different grades of emery down to 520 grade, and polished. The panels after vapour degreasing, are cathodically cleaned in 5% sodium hydroxide solutions with steel anode of same size at 30-50°C at a current density of 50 asf for 2 minutes. They are washed in water and rinsed in distilled water. The specimen is made as a cathode in the acid tin plating bath of the following composition under the conditions given below :

Stannous sulphate :	80 g/l
Sulphuric acid :	20 g/l
CTAB	5 g/l
pH	2-3
Current density	0.5-3 A/dm^2
Time	2-30 minutes

Till the thickness of the plate is 2-10/ μ

The electroplated tin panels are washed well to free it from the traces of acid and heated in a furnacls at 100-200°C for 5-10 minutes to allow the crystae (of tin) to grow. The specimen is cooled rapidly to get finer crystal pattern greater than 1 cm size or very slowly to get coarser crystal size of 1 cm or more. The specimens are etched in any one of the following solution and the optimum conditions of etching are worked out so as to have maximum relief/grain pattern and minimum rate of attack on tin.

SOLUTION 1

Ferric chloride :	20-100 gms
Sodium sulphide :	0.01-0.1 gm
Water	1 litre
Temperature	30-50°C
Time	2-5 seconds

Price : TWO RUPEES.

SOLUTION 2

Nitric acid	2-5%
Inhibitor	0.1% g/l
Time	2-5 seconds
Temperature	30-60°C

SOLUTION 3

Sulphuric acid	5-10%
Inhibitor	0.1 g/l
Temperature :	30-40°C

SOLUTION 4

Hydrochloric acid :	20-50 cc
Nitric acid	10-40 cc
Water to	1 litre
Time	2-5 seconds
Temperature	30°C

The etched panels are washed thoroughly and electroplated with metals such as cadmium, tin, or lead in which cases a thin layer of metal is reinforced over the multigrained patterns and rinsed. The panels are coloured by using coloured lacquers or by chemical or electrochemical methods.

A typical solution for getting a rainbow pattern consists of the following :

1.	Ammonium molybdate :	20-50 g/l
	Nickel sulphate	10-20 g/l
	Boric acid	10-20 g/l
	pH	4-5
	Current density	10 mA/sq.cm
	Temperature	30-70°C
	Time :	2-4 minutes
2.	Nickel sulphate :	100 g/l
	Zinc sulphate :	40 g/l
	Ammonium sulfocyanide	24 g/l
	pH	5
	Current density	5-10 mA/cm^2
	Time	2 secs to 5 minutes

The specimen is made as the cathode in the above solution with lead as the anode and finished panel will have the multicolours in the grains.

Any of the standard chemical colouring procedure recommended for colouring of tin can be adopted for the multigrained tinned steel also.

Alternately the hot dipped tin also after recrystallisation procedures listed above can be flow brightend, etched, rinsed, bright plated with cadmium, tin etc and coloured to get an attractive mottled, spangled or multitoned effect.

The following is the main advantage of the invention :

1. It increases the aesthetic appeal of the product suitable for decorstive effects like making of toys, boxes, furniture, refrigerator and a number of decorative items.

R. Bhaskar Pai

Patents Officer

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

Dated this 26th day of October 1971.

Dipti Printing, Calcutta-4-150 Copies.

Complete Specification No. 133404.

IMPROVEMENTS IN OR RELATING TO OBTAINING SPANGLE FINISH ON ELCTROPLATED OR TINNED STEEL.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.

RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATON 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 :—

This is an invention by BALKUNJE ANANTHA SHENOI, Scientist, Mrs. INDIRA RAJAGOPALAN, Scientist and HANDADY VENKATAKRISHNA UDUPA, Director, all of the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, and all are Indian Nationals.

This invention relates to improvements in or relating to obtaining spangle finish on electroplated or tinned steel and has particular reference to spangle finish coatings on electrotinned or hot dipped tin plates.

Hitherto it has been proposed to get patterns on tinned steel by costly and laborious engraving procedures or by elaborate electromarking procedures. To adopt the above procedures, the thickness of tin coatings should be at least 10 thou or more. The reproducibility and reliability of the markings by the above procedures depend upon a number of factors and mainly upon the skill of the labourer. Moreover, the above procedures offer some mechanical defects after the final process. Moreover, the finishes obtained by the above processes do not have sufficient eye appeal also. Because of the fact that higher thickness of plates are required and the manual skill involved in the above processes, these are highly costly too.

Hence, process has been worked out by a relatively simple technique and the pattern that is obtained by the present process cannot even be duplicated by the other engraving procedures. The object of the invention is to obviate the disadvantages.

According to the present invention, there is provided a process for obtaining spangle finish on electroplated or tinned steel so as to produce a multitoned effect of recrystallisation by heating and then etching it in a suitable etchant.

The invented process thus resorts to recrystallisation procedures (i. e., by heating the hot dipped or electro-tinned steel to the recrystallisation temperature of tin), etching the recrystallised tinned steel by choosing suitable etchants so as to reveal the grain patterns, followed by flash plating of tin and colouring the same to give rise to multicoloured, multigrained patterns on tinned steel specimens. The etching procedure is to be carefully chosen to give the best relief. The etchant should not be either too corrosive to attack the tinned steel in no time or too slow in which case, one will get a uniform dissolution leading to a smooth surface and not a multitoned effect.

To these ends, the invention broadly consists in electroplatin of tin on steel or hot dipping of tin on steel followed by recrystallisation and revelation of gains so as to produce a multitioned effect and is coloured by using suitable treatment (electrochemical, chemical or physical methods).

THE PLATING PROCEDURES

The mild steel panels of required size are polished with different grades of emery down to 520 grade, and a polished. The panels after vapour degreasing are cathodically cleaned in 5% sodium hydroxide solutions with steel anode of same size at 30-50°C at a current density of 50 asf for 2 minutes. They are washed in water and rinsed in distilled water. The specimen is made as a cathode in the acid tin plating bath of the following composition under the conditions given below :

Stannous sulphate	80 g/l
Sulphuric acid	20 g/l
CTAB	5 g/l
pH	2-3
Current density	0.5-3 A/dm^2
Time	2-30 minutes
Till the thickness of the plate is	2-10 microns

The electroplated tin panels are washed well to free it from the traces of acid and heated in a furnace at 100-200°C for 5-10 minutes to allow the crystals (of tin) to grow. The specimen is cooled rapidly to get finer crystal pattern lesser than 1 cm size or very slowly to get coarser crystal size of 1 cm or more. The specimens are etched in any one of the following solutions and the optimum conditions of etching are worked out so as to have maximum relief/grain pattern and minimum rate of attack on tin.

SOLUTION 1

Ferric chloride	20-100g
Sodium sulphide	0.01-0.1g
Water	1 litre
Temperature	30-50°C
Time	2-5 seconds

SOLUTION 2

Nitric acid	2-5%
Tetra alkyl Ammonium halide	0.1 g/l
Time	2-5 seconds
Temperature	30-60°C

SOLUTION 3

Sulphuric acid	5-10%
Benzotriazole	0.1 g/l
Temperature	30-50°C
Time	2-5 seconds

SOLUTION 4

Hydrochloric acid	20-50 cc
Nitric acid	10-40 cc
Water to	1 litre
Time	2-5 seconds
Temperature	30°C

The etched panels are washed thoroughly and electroplated with metals such as cadmium, tin or lead in which cases a thin layer of metal is reinforced over the multigrained patterns and rinsed. The panels are coloured by using coloured lacquers or by chemical or electrochemical methods.

A typical solution for getting a rainbow pattern consists of the following :

1.	Ammonium molybdate	20-50 g/l
	Nickel sulphate	10-20 g/l
	Boric acid	10-20 g/l
	pH	4-5
	Current density	10m. amp/sq.cm
	Temperature	30-70°C
	Time	2-4 minutes
2.	Nickel sulphate	100 g/l
	Zinc sulphate	40 g/l
	Ammonium sulphocyanide	24 g/l
	pH	5
	Current density	5-10mm A/sq. cm
	Time	2 secs-5 mts.

The specimen is made as the cathode in the above solution with lead as the anode and the finished panel will have the multicolours in the grains.

Any of the standard chemical colouring procedure recommended for colouring of tin can be adopted for the multigrained tinned steel also.

Alternately, the hot dipped tin also after recrystallisation procedures listed above can be flow-brightened, etched, rinsed, bright plated with cadmium, tin etc. and coloured to get an attractive mottled, spangled or multitoned effect.

The following is the main advantage of this invention :

1. To increase the aesthetic appeal of the products such as toys, boxes. furniture, refrigerator or like articles.

WE CLAIM :

1. A process for obtaining spangle finish on electroplated or tinned steel so as to produce a multitoned effect of recrystallisation by heating and then etching it in a suitable etchant.

2. A process for obtaining spangle finish on electroplated or tinned steel as claimed in Claim 1 wherein the plated part is heated from 100°-200°C for recrystallation.

3. A process for obtaining spangle finish on electroplated or tinned steel as claimed in Claim 1 wherein the recrystallised part is etched in any one of the etching solutions described herein.

4. A process for obtaining spangle finish on electroplated or tinned steel as claimed in any of the preceding claims which consists of the following steps :-

(i) tinning or electroplating of tin over mild steel substrate,

(ii) heating the tinned surface for recrystallisation, and

(iii) etching the recrystallised surface.

5. A process for obtaining spangle finish on electroplated or tinned steel substantially as hereinbefore described.

R. Bhaskar Pai

Patents Officer

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.

Dated this 23rd day of January 1973.

Dipti Printing, Calcutta-4-150 Copies.