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(54) **Title:** INHIBITING TOXICITY OF ACID SYSTEMS USED FOR TREATING METALS

(57) **Abstract:** In some examples of inhibiting toxicity of acid systems used for treating metals, a metal is pickled with a pickling composition that includes a first quantity of an acid and a second quantity of a corrosion inhibiting iodide salt. A mixture of the first quantity and the second quantity modify metallic properties of the pickled metal. The corrosion inhibiting iodide salt is added directly to the pickling composition independent of any other corrosion inhibitor or corrosion inhibiting polymer.



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## INHIBITING TOXICITY OF ACID SYSTEMS USED FOR TREATING METALS

### CLAIM OF PRIORITY

[0001] This application claims priority to U.S. Patent Application No.  
5 14/563,024 filed on December 8, 2014 the entire contents of which are hereby  
incorporated by reference.

### TECHNICAL FIELD

[0002] This disclosure relates to treating metals, for example, to improve  
corrosion resistance of the metals.

### 10 BACKGROUND

[0003] Metallic components made, for example, using carbon steel, can be  
treated using pickling systems to improve the resistance of the metallic component  
against conditions of the environment in which the components are implemented.  
Acids, for example, hydrochloric acid (HCl) and/or sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) are used in  
15 pickling treatments. However, the use of HCl is associated with problems due to its  
volatility which is enhanced by increases in temperature, acid strength, and iron  
content in the bath. This creates problems such as atmospheric pollution, pitting attack  
on the pickling material, and corrosion of surrounding structures, etc., in the pickling  
process. As a result, H<sub>2</sub>SO<sub>4</sub> is still in use in many steel plants. However, H<sub>2</sub>SO<sub>4</sub> acid  
20 pickling system is conventionally inhabited using high toxicity synthesized corrosion  
inhibitors.

### SUMMARY

[0004] This disclosure relates to inhibiting corrosivity of acid systems used for  
treating metals.

25 [0005] Some aspects of the subject matter described here can be implemented  
as a method of treating a metal. The treatment includes a pickling of the metal. The  
metal is pickled with a pickling composition that includes a first quantity of an acid  
and a second quantity of a corrosion inhibiting iodide salt. A mixture of the first

quantity of the pickling composition and the second quantity of the corrosion inhibiting iodide salt modifies metallic properties of the pickled metal.

[0006] This, and other aspects, can include one or more of the following features. The acid includes sulfuric acid. The corrosion inhibiting iodide salt includes potassium iodide. The second quantity of the corrosion inhibiting iodide salt is added directly to the first quantity of the pickling composition independent of any other corrosion inhibitor. The second quantity of the corrosion inhibiting iodide salt is mixed with the first quantity of the pickling composition at a temperature greater than room temperature. The temperature is at least about 60 °C. The temperature ranges between about 60 °C and 80 °C. The acid in the pickling composition has a molarity of either 0.5 M or 1.0 M. The metal comprises carbon steel. Pickling the metal with the pickling composition including the corrosion inhibition iodide salt includes soaking the metal in the pickling composition for a period of time. The period of time includes might be up to six hours. A ratio of the second quantity to the first quantity ranges between greater than 0 g of iodide salt per 100 mL of acid to about 0.5 g of iodide salt per 100 mL of acid. The first quantity and the second quantity are selected such that the pickling composition including the corrosion inhibiting iodide salt has a corrosion rate of less than about 0.05 lb/sq. ft.

[0007] Some aspects of the subject matter described here can be implemented as a method of pickling a metal. A first quantity of a pickling composition including an acid is obtained. The pickling composition modifies metallic properties of the pickled metal. A second quantity of a corrosion inhibiting iodide salt is obtained. To inhibit a corrosiveness of the pickling composition, the second quantity of the corrosion inhibiting iodide salt and no other corrosion inhibitor is mixed with the first quantity of the pickling composition. The metal is pickled with the pickling composition including the corrosion inhibiting iodide salt.

[0008] This, and other aspects, can include one or more of the following features. A ratio of the second quantity to the first quantity ranges between greater than 0 g of iodide salt per 100 mL of acid to about 0.5 g of iodide salt per 100 mL of acid. The first quantity and the second quantity are selected such that the pickling composition including the corrosion inhibiting iodide salt has a corrosion rate of less

than about 0.05 lb/sq. ft. The acid in the pickling composition has a molarity of either 0.5 M or 1.0 M.

[0009] Some aspects of the subject matter described here can be implemented as a method of pickling a metal. A first quantity of a pickling composition including an acid is mixed with a second quantity of a corrosion inhibiting iodide salt that is independent of any corrosion inhibiting polymer. The pickling composition modifies metallic properties of the pickled metal.

[0010] This, and other aspects, can include one or more of the following features. The acid includes sulfuric acid. The corrosion inhibiting iodide salt includes potassium iodide.

[0011] The details of one or more implementations of the subject matter described in this specification are set forth in the accompanying drawings and the description below. Other features, aspects, and advantages of the subject matter will become apparent from the description, the drawings, and the claims.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0012] FIG. 1 shows a schematic of an example system for treating a metal.

[0013] FIG. 2 is a flowchart of an example process for treating a metal.

#### DETAILED DESCRIPTION

[0014] This disclosure describes inhibiting corrosivity of acid systems used for treating metals. The disclosure describes utilizing an iodide salt (e.g., potassium iodide) as an inhibitor for a pickling system that includes an acid (e.g., sulfuric acid). Doing so demonstrated a high protection to low carbon steel relative to certain commercially available inhibitor systems which resulted in high toxicity. Acid systems for treating metals can include different acids. For example, sulfuric acid and hydrochloric acid have been used in pickling treatments. Hydrochloric acid can sometimes be volatile, for example, at elevated temperatures and/or acid strengths, and/or when the iron content in the pickling treatment bath is high. The increased volatility of hydrochloric acid can result in negative effects, such as, atmospheric pollution, pitting attack on the treated metal, corrosion of surrounding structures, or other negative effects, in the pickling process. Sulfuric acid offers an alternative to

hydrochloric acid. Synthesized corrosion inhibitors that, in some cases, may include nitrogen, are often used with pickling systems that include sulfuric acid. The synthesized corrosion inhibitors exhibit high toxicity. Also, manufacturing such synthesized corrosion inhibitors can be complex and expensive.

5 [0015] As described below, an iodide salt, e.g., potassium iodide, can serve as an effective, yet cheaper alternative to synthesized corrosion inhibitors. Relative to certain synthesized corrosion inhibitors which include polymers, the iodide salt exhibits less toxicity in acid pickling systems that include sulfuric acid while exhibiting substantially similar corrosion inhibiting properties. The techniques  
10 described here can be implemented to treat metals, e.g., carbon steel such as low carbon steel or other metals and/or to descale heat exchange tubes (or both). Such treatment can prevent degradation of steel and other negative impacts that the conditions of the environment in which the steel is implemented can have on the steel. Pickling is also used to remove scale and corrosion products from boiler and  
15 distillation tanks.

[0016] FIG. 1 shows a schematic of an example system for treating a metal. FIG. 2 is a flowchart of an example process 200 for treating a metal. In some implementations, at 202, a first quantity of a pickling composition including an acid is obtained. The pickling composition 102 can modify metallic properties of the treated  
20 metal 104. For example, the pickling composition includes a pickling composition that can modify surface properties of metal, e.g., carbon steel. The pickling composition 102 can be placed in a container 100 in which metal 104 (for example, a metallic component) is placed. At 204, a second quantity of a corrosion inhibiting iodide salt is obtained. Examples of the first quantity and second quantity are described below. The  
25 second quantity of the corrosion inhibiting iodide salt 106 is mixed with the first quantity of the pickling composition 102. For example, the iodide salt 106 in a container 108 can be added to the pickling composition 102 in the container 100. The metal 104 is then treated with the pickling composition including the corrosion inhibiting salt. The iodide salts are adsorbed to the metal surfaces thereby minimizing  
30 acid contact to the metal surfaces.

[0017] The acid in the pickling composition can include sulfuric acid. The corrosion inhibiting iodide salt can include potassium iodide. The second quantity of

the iodide salt can be added directly to the first quantity of the pickling composition independent of any other corrosion inhibitor. In other words, no corrosion inhibitor other than the second quantity of the iodide salt need be added to the pickling composition to inhibit corrosion of the treated metal.

[0018] Some example conditions for mixing the pickling composition and the corrosion inhibiting iodide salt are described here. In one example, 0.5 M sulfuric acid at about 60 °C can be mixed with potassium iodide at a concentration ranging between about 0.075 g per 100 mL to about 0.30 g per 100 mL. In another example, 0.5 M sulfuric acid at about 80 °C can be mixed with potassium iodide at a concentration ranging between about 0.10 g per 100 mL to about 0.50 g per 100 mL. In a further example, 1 M sulfuric acid at about 60 °C can be mixed with potassium iodide at a concentration ranging between about 0.10 g per 100 mL to about 0.40 g per 100 mL. In an additional example, 1 M sulfuric acid at about 80 °C can be mixed with potassium iodide at a concentration ranging between about 0.05 g per 100 mL to about 0.40 g per 100 mL. Thus, the temperature at which the pickling composition and the iodide salt are mixed might be greater than room temperature, e.g., can be about 60 °C. Further, for any concentration of acid and at any temperature, the ratio of the quantity of the iodide salt to the quantity of the acid can range between greater than 0 g of iodide salt per 100 mL of acid to about 0.5 g of iodide salt per 100 mL of acid. Also, in general, a quantity of the corrosion inhibiting salt and a quantity of the acid in the pickling composition can be selected such that the pickling composition has a corrosion rate of less than about 0.05 lb/sq. ft.

[0019] Results of corrosion tests performed on a metal, e.g., 1018 cold roll steel, are described below. An analysis of the cold roll steel revealed the composition shown in Table 1. In the experiments described below, the average weight of the metal sample was about 18 g. The metal sample was exposed to about 200 mL of pickling composition described below.

Table 1: Analysis of Cold Roll Steel

| <b>Carbon (C)</b> | <b>Manganese (Mn)</b> | <b>Phosphorus (P)</b><br>Max | <b>Sulfur (S)</b><br>Max |
|-------------------|-----------------------|------------------------------|--------------------------|
| 0.15-0.2          | 0.6-0.9               | 0.04                         | 0.05                     |

[0020] In the experiments performed to obtain the results described below, a first quantity of a pickling solution including sulfuric acid was mixed with a second

quantity of iodide salt. To inhibit a corrosiveness of the pickling composition, no corrosion inhibitor other than the iodide salt was mixed with the first quantity. In particular, no synthesized corrosion inhibiting materials such as polymers were mixed either with the pickling composition or with the iodide salt. The cold roll steel was treated with the pickling composition, for example, by soaking the cold roll steel in the mixture of the pickling composition and the iodide salt, under experimental conditions shown in the following tables.

[0021] Experiment I

[0022] Potassium iodide having a concentration in a range between about 0 g to about 0.3 g was mixed with 100 mL of 0.5 M sulfuric acid at about 60 °C for about six hours. The corrosion rate and the inhibition efficiency measured for the different concentrations of potassium halide in sulfuric acid are shown in Table 2.

Table 2: Effect of iodide concentration on inhibition of 0.5 M H<sub>2</sub>SO<sub>4</sub> acid at about 60 °C

| Potassium iodide concentration (g/100 ml H <sub>2</sub> SO <sub>4</sub> ) | Temp., °C | Soaking Time, hr | Corrosion Rate, lb/ft <sup>2</sup> | I E, wt% |
|---------------------------------------------------------------------------|-----------|------------------|------------------------------------|----------|
| 0                                                                         | 60        | 6                | 0.28024                            |          |
| 0.075                                                                     | 60        | 6                | 0.00240                            | 99.14    |
| 0.1                                                                       | 60        | 6                | 0.00233                            | 99.167   |
| 0.2                                                                       | 60        | 6                | 0.00232                            | 99.17    |
| 0.3                                                                       | 60        | 6                | 0.00095                            | 99.66    |

[0023] Experiment II

[0024] Potassium iodide having a concentration in a range between about 0 g to about 0.4 g was mixed with 100 mL of 1 M sulfuric acid at about 60 °C for about six hours. The corrosion rate and the inhibition efficiency measured for the different concentrations of potassium halide in sulfuric acid are shown in Table 3.

Table 3: Effect of iodide concentration on inhibition of 1 M H<sub>2</sub>SO<sub>4</sub> acid at 60°C

| Potassium iodide concentration (g/100 ml H <sub>2</sub> SO <sub>4</sub> ) | Temp., °C | Soaking Time, hr | Corrosion Rate, lb/ft <sup>2</sup> | I E, wt% |
|---------------------------------------------------------------------------|-----------|------------------|------------------------------------|----------|
| 0                                                                         | 60        | 6                | 0.501954623                        |          |
| 0.1                                                                       | 60        | 6                | 0.00184                            | 99.63    |
| 0.2                                                                       | 60        | 6                | 0.00100                            | 99.79    |
| 0.3                                                                       | 60        | 6                | 0.00050                            | 99.90    |
| 0.4                                                                       | 60        | 6                | 0.00044                            | 99.91    |

[0025] Experiment III

[0026] Potassium iodide having a concentration in a range between about 0 g to about 0.5 g was mixed with 100 mL of 0.5 M sulfuric acid at about 80 °C for about six hours. The corrosion rate and the inhibition efficiency measured for the different concentrations of potassium halide in sulfuric acid are shown in Table 4.

5 Table 4: Effect of iodide concentration on inhibition of 0.5 M H<sub>2</sub>SO<sub>4</sub> acid at 80°C

| Potassium iodide concentration (g/100 ml H <sub>2</sub> SO <sub>4</sub> ) | Temp., °C | Soaking Time, hr | Corrosion Rate, lb/ft <sup>2</sup> | I E, wt% |
|---------------------------------------------------------------------------|-----------|------------------|------------------------------------|----------|
| 0                                                                         | 80        | 6                | 0.308457656                        |          |
| 0.1                                                                       | 80        | 6                | 0.00763                            | 97.52    |
| 0.2                                                                       | 80        | 6                | 0.00243                            | 99.21    |
| 0.3                                                                       | 80        | 6                | 0.00194                            | 99.19    |
| 0.4                                                                       | 80        | 6                | 0.00109                            | 99.65    |
| 0.5                                                                       | 80        | 6                | 0.00068                            | 99.78    |

[0027] Experiment IV

[0028] Potassium iodide having a concentration in a range between about 0 g to about 0.5 g was mixed with 100 mL of 1 M sulfuric acid at about 80 °C for about six hours. The corrosion rate and the inhibition efficiency measured for the different concentrations of potassium halide in sulfuric acid are shown in Table 5.

10 Table 5: Effect of iodide concentration on inhibition of 1 M H<sub>2</sub>SO<sub>4</sub> acid at 80°C

| Potassium iodide concentration (g/100 ml H <sub>2</sub> SO <sub>4</sub> ) | Temp., °C | Soaking Time, hr | Corrosion Rate, lb/ft <sup>2</sup> | I.E, wt% |
|---------------------------------------------------------------------------|-----------|------------------|------------------------------------|----------|
| 0                                                                         | 80        | 6                | 0.60895                            |          |
| 0.05                                                                      | 80        | 6                | 0.01109                            | 98.18    |
| 0.1                                                                       | 80        | 6                | 0.00618                            | 98.77    |
| 0.2                                                                       | 80        | 6                | 0.00376                            | 99.38    |
| 0.3                                                                       | 80        | 6                | 0.00307                            | 99.50    |
| 0.4                                                                       | 80        | 6                | 0.00252                            | 99.59    |

[0029] In sum, this disclosure describes mixing a first quantity of a pickling composition including an acid with a second quantity of a corrosion inhibiting iodide salt. The acid in the pickling composition can include sulfuric acid. The corrosion inhibiting iodide salt can be independent of any corrosion inhibiting polymer. A metal, e.g., carbon steel, can be treated in the mixture to modify surface properties of the metal while inhibiting corrosion. Adding the halide directly to the pickling

composition without any synthesized corrosion inhibitor can decrease a toxicity of the pickling composition.

[0030] Particular implementations of the subject matter have been described. Other implementations are within the scope of the following claims.

5 [0031] What is claimed is:

## CLAIMS

1. A method of pickling a metal, the method comprising:  
pickling a metal with a pickling composition comprising a first quantity of an acid and a second quantity of a corrosion inhibiting iodide salt, wherein a mixture of  
5 the first quantity of the pickling composition and the second quantity of the corrosion inhibiting iodide salt modify metallic properties of the pickled metal.
2. The method of claim 1, wherein the acid comprises sulfuric acid.
3. The method of claim 1, wherein the corrosion inhibiting iodide salt comprises potassium iodide.
- 10 4. The method of claim 1, wherein the second quantity of the corrosion inhibiting iodide salt is added directly to the first quantity of the pickling composition independent of any other corrosion inhibitor.
5. The method of claim 1, wherein the second quantity of the corrosion inhibiting iodide salt is mixed with the first quantity of the pickling composition at a temperature  
15 greater than room temperature.
6. The method of claim 5, wherein the temperature is at least about 60 °C.
7. The method of claim 6, wherein the temperature ranges between about 60 °C and 80 °C.
8. The method of claim 1, wherein the acid in the pickling composition has a molarity  
20 of either 0.5 M or 1.0 M.
9. The method of claim 1, wherein the metal comprises carbon steel.
10. The method of claim 1, wherein pickling the metal with the pickling composition including the corrosion inhibiting iodide salt comprises soaking the metal in the pickling composition for a period of time.
- 25 11. The method of claim 10, wherein the period of time comprises at up to about six hours.

12. The method of claim 1, wherein a ratio of the second quantity to the first quantity ranges between greater than 0 g of iodide salt per 100 mL of acid to about 0.5 g of iodide salt per 100 mL of acid.

13. The method of claim 1, wherein the first quantity and the second quantity are  
5 selected such that the pickling composition including the corrosion inhibiting iodide salt has a corrosion rate of less than about 0.05 lb/sq. ft.

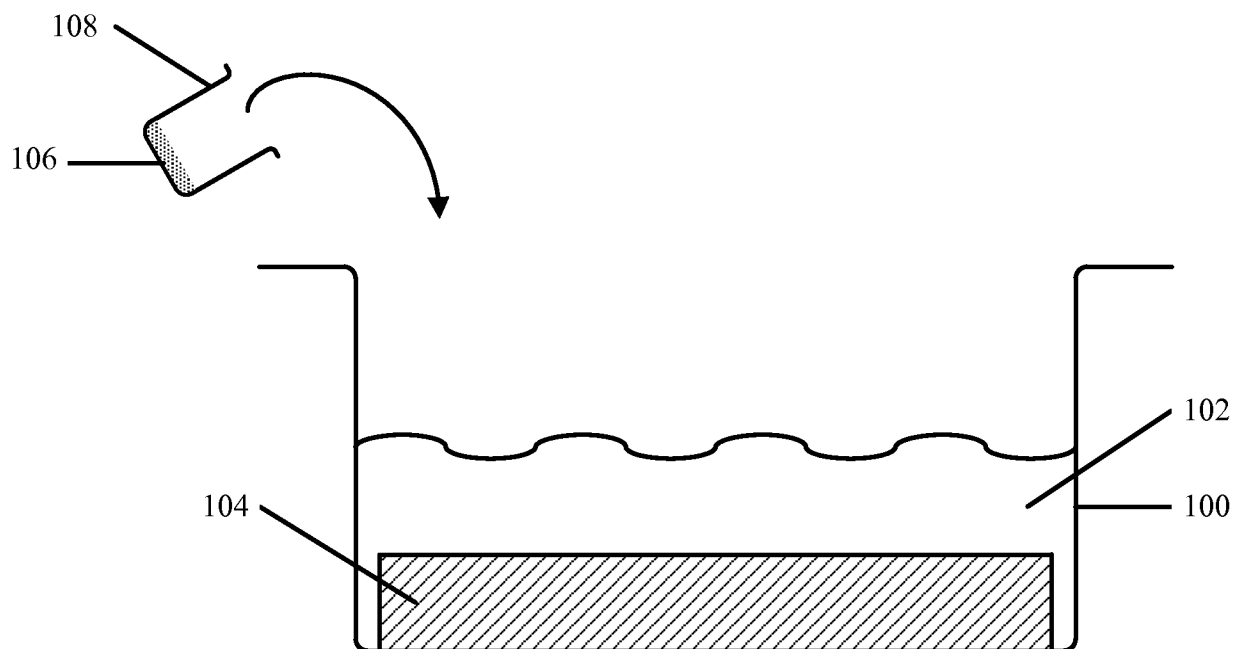
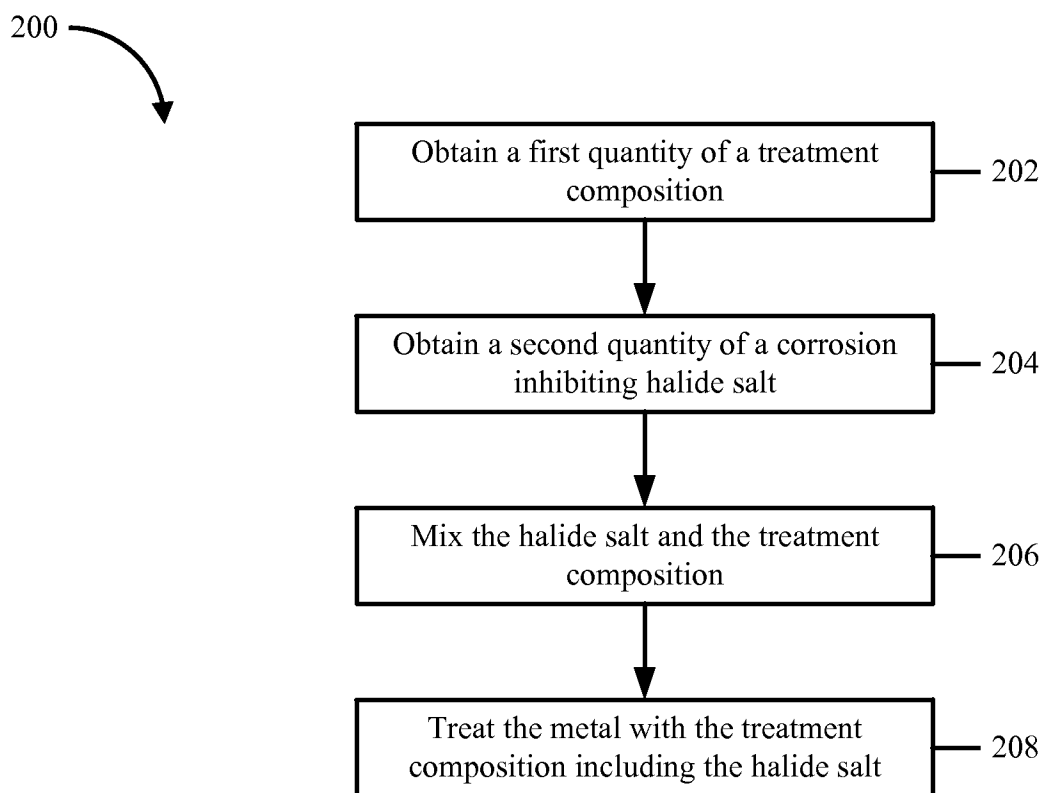
14. A method of pickling a metal, the method comprising:  
obtaining a first quantity of a pickling composition comprising an acid, the  
pickling composition to modify metallic properties of the pickled metal;  
10 obtaining a second quantity of a corrosion inhibiting iodide salt;  
to inhibit a corrosiveness of the pickling composition, mixing the second  
quantity of the corrosion inhibiting iodide salt and no other corrosion inhibitor with the  
first quantity of the pickling composition; and  
pickling the metal with the pickling composition including the corrosion  
15 inhibiting iodide salt.

15. The method of claim 14, wherein a ratio of the second quantity to the first quantity ranges between greater than 0 g of iodide salt per 100 mL of acid to about 0.5 g of iodide salt per 100 mL of acid.

16. The method of claim 14, wherein the first quantity and the second quantity are  
20 selected such that the pickling composition including the corrosion inhibiting iodide salt has a corrosion rate of less than about 0.05 lb/sq. ft.

17. The method of claim 14, wherein the acid in the pickling composition has a molarity of either 0.5 M or 1.0 M.

18. A method of pickling a metal, the method comprising:
- mixing a first quantity of a pickling composition comprising an acid, the pickling composition to modify metallic properties of the pickled metal, with a second quantity of a corrosion inhibiting iodide salt that is independent of any corrosion
  - 5 inhibiting polymer; and
  - pickling the metal with the pickling composition including the corrosion inhibiting iodide salt.
19. The method of claim 19, wherein the acid comprises sulfuric acid.
20. The method of claim 19, wherein the corrosion inhibiting iodide salt comprises
- 10 potassium iodide.

**FIG. 1****FIG. 2**

## INTERNATIONAL SEARCH REPORT

International application No

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A. CLASSIFICATION OF SUBJECT MATTER  
 INV. C23G1/08 C23G1/04  
 ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
 C23G C23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, COMPENDEX, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                           | Relevant to claim No.  |
|-----------|------------------------------------------------------------------------------------------------------------------------------|------------------------|
| X         | CN 1 108 703 A (SHANDONG PROV METALLURG SCIENC [CN])<br>20 September 1995 (1995-09-20)<br>page 3; claims; example 1<br>----- | 1-5,<br>9-16,<br>18-20 |
| X         | US 2 708 184 A (HAGER KARL F ET AL)<br>10 May 1955 (1955-05-10)<br><br>column 1, lines 35-47; example 1<br>-----             | 1-4,<br>8-10,<br>12-20 |
| X         | US 3 773 465 A (KEENEY B ET AL)<br>20 November 1973 (1973-11-20)<br>claim 1; example VI<br>-----                             | 1,4-7,<br>9-16,18      |
| Y         | US 2 511 988 A (FRANCIS MYERS HENRY ET AL)<br>20 June 1950 (1950-06-20)<br>claims<br>-----<br><br>-/-                        | 1-20                   |



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

22 December 2015

Date of mailing of the international search report

12/01/2016

Name and mailing address of the ISA/

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Mauger, Jeremy

## INTERNATIONAL SEARCH REPORT

International application No

PCT/US2015/055657

| C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT |                                                                                                                                                                                                                                                                                                    |                       |
|------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| Category*                                            | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                                                                                                                 | Relevant to claim No. |
| Y                                                    | <p>HEUSLER K E ET AL: "THE INFLUENCE OF IODIDE IONS AND CARBON MONOXIDE ON THE ANODIC DISSOLUTION OF ACTIVE IRON", JOURNAL OF THE ELECTROCHEMICAL SOCIETY, ELECTROCHEMICAL SOCIETY, vol. 108, 1 August 1961 (1961-08-01), pages 732-740, XP001057483, ISSN: 0013-4651<br/>Discussion<br/>-----</p> | 1-20                  |

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2015/055657

| Patent document<br>cited in search report |   | Publication<br>date | Patent family<br>member(s) | Publication<br>date |
|-------------------------------------------|---|---------------------|----------------------------|---------------------|
| CN 1108703                                | A | 20-09-1995          | NONE                       |                     |
| US 2708184                                | A | 10-05-1955          | NONE                       |                     |
| US 3773465                                | A | 20-11-1973          | NONE                       |                     |
| US 2511988                                | A | 20-06-1950          | NONE                       |                     |