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(54) **Title**: ENAMEL POWDER AND ENAMEL SLURRY FOR PRODUCING AN ENAMEL COATING ON A METALLIC SUBSTRATE, METHOD FOR MANUFACTURING AN ENAMEL SLURRY AND USE OF THE ENAMEL COATING FOR HEAT EXCHANGERS

(57) **Abstract**: The invention concerns the enameling of aluminium-containing metallic objects that may be exposed to high thermal and mechanical loads. A preferred field of application of the invention is the coating of heat exchangers for the heating of liquids, in particular water, so that by an enameling the heat exchanger at least in portions thereof is protected from hot flame gases and condensing as well as condensed fluids. This is achieved with an enamel powder for the manufacture of an enamel slurry for producing an enamel coating on a metallic aluminium- containing substrate, which enamel powder is a mixture of an enamel frit and up to 15 % of a powder-form oxidic light metal compound and up to 20 % of a high-grade steel powder and with an enamel slurry containing such enamel powder and a special liquid such that the resulting enamel coating is resistant to high thermal and mechanical loads.

Enamel powder and enamel slurry for producing an enamel coating on a metallic substrate, method for manufacturing an enamel slurry and use of the enamel coating for heat exchangers

Field

The invention relates to the protection of aluminium-containing metallic objects that may be exposed to high thermal and mechanical loads. A preferred field of application of the invention relates to heat exchangers for the heating of liquids, in particular water.

Background

Heat exchangers of aluminium are known. An example of a known heat exchanger is described in EP-0 889 292 A2 and shown in figure 1 thereof. Preferably, an aluminium heat exchanger, for example for a central heating installation, is manufactured in a casting process, e.g. a sandcasting process. The heat exchanger includes at least one flue gas draft and water channels which are integrally formed during the casting procedure, for example by using a sand mold and sand cores. Optionally, the aluminium walls of the heat exchanger also bound a burner chamber in which a burner may be accommodated. Alternatively, a burner may be connected to a side the heat exchanger, e.g. the top side, so that the flames produced by the burner extend into a burner chamber which may be formed by an upstream part of the at least one flue gas draft. An example of such a configuration is shown in figures 1 and 2 of EP 1722172 A2. The water channels are preferably arranged in such a manner that the aluminium walls that bound the at least one flue gas draft and the burner chamber are cooled by the water present in the water channels. In use, very hot flue gasses directly contact the aluminium heat exchanger walls in the at least one flue gas draft of the heat exchanger. The walls of the aluminium heat exchanger may be provided with a surface area enlarging structures, such as fins of pins that protrude in the at least one flue gas draft. Optionally, the walls may also be provided with surface area enlarging pins and or fins that extend into the water channels. Thus, a very direct transfer of heat from the flue gas via the aluminium walls to the water in the water channel may be effected. Because the aluminium heat exchangers may be manufactured as a monocast, manufacturing costs may be relatively low.

Aluminium is a suitable material because it has a very good heat conductivity properties. The surface of aluminium will automatically be covered with a thin layer of aluminium oxide which is vacuum-tight and prevents a further corrosion. Advantageously, the material of a heat exchanger comprises an alloy that in addition to aluminium also contains silicon and magnesium. The silicon here serves for better castability of the liquid alloy. This is of importance for being able to cast fine structures such as the fins and/or pins that provide a better heat transfer as a consequence of the enlarged heat transfer surface area. Magnesium, in turn, is added to the alloy to increase mechanical hardness.

Summary

When the known heat exchanger is used, fuel is combusted and the flue draft gases which are produced may contain combustion products are formed which are detrimental to the aluminium. Especially for heat exchangers with very high efficiency, the flue draft gases are cooled down to such an extend that condensation of water occurs within the heat exchanger. In both gas and oil, which are the common fuels used for combustion, sulfur is contained. During the burning process, this sulfur reacts with oxygen to form sulfur dioxide. Additionally NO_x is formed in the burning process. During the subsequent condensation, in which water is formed, the water reacts with the sulfur dioxide and the NO_x so that and sulfurous acid and Nitric acid are formed. Upon evaporation of the condensate, the concentration of the acids increases so that the acids may react with the amphoteric aluminium to form deposits. The surface of the heat exchanger is affected by these kind of deposits which may impede heat transmission and which may lower the efficiency of the heating system. Additionally, the deposits may clog the flue gas drafts. Finally, the life of the system is decisively reduced.

It is an object of the invention to alleviate the problem mentioned above.

This object can not be achieved by utilization coatings comprising organic materials, as has been shown by tests with a wide range of organic coating materials, as the high temperature that arises upon the combustion of oil, gas, etc., destroys the organic coating in less than no time.

The utilization of known inorganic coating materials, on the other hand, led to glass ceramic coating materials in the form of enamels with a small softening interval. Low-melting enamels are required because aluminium has a low melting point and the temperature which is required for the application of the enamel on the aluminium surface should be lower than the melting temperature of the aluminium. In fact, the softening interval of known low-melting enamels is so small that these enamels do not exhibit any decisive resistance to high thermal and mechanical loads which are caused by quick temperature changes due to the hot flue gases and even flames that intermittently contact the coated surface due to switching on and off of the burner.

Thus, in order to improve the corrosion protection and reduction of deposit formation on aluminium-containing metallic objects, the object of the invention is to provide a low melting enamel with good heat conductive properties that is resistant to high thermal and mechanical loads.

According to a first aspect of the invention, a coating material is provided that is defined in claim 1. This coating material comprises an enamel powder for manufacturing an enamel slurry for producing an enamel coating on a metallic aluminium-containing substrate, wherein the powder comprises an enamel frit which is mixed with up to 15 % of a powder-form oxidic light metal compound and up to 20 % of a high-grade steel powder.

According to a second aspect of the invention, an enamel slurry according to claim 5 is provided. The enamel slurry for producing an enamel coating on a metallic aluminium-containing substrate comprises an amount of water and a mixture, wherein the mixture includes:

- an enamel powder according to the invention; and
- up to 10 % of alcohol, up to 2.5 % of calcium formate, up to 6 % of boric acid, up to 6 % of potassium hydroxide solution, and up to 6 % of water glass.

In a further elaboration of the slurry, the amount of water in the slurry is such that the slurry has a density of substantially 1.85 g/cm^3.

The percentages mentioned herein, are percentages by weight. The enamel coating obtained with this slurry is optimal for its purpose, i.e. coating aluminium containing substrates that are subjected to high temperature and mechanical loads. This is due to the interaction of the constituents, which make their contributions by virtue of their properties. Thus, the oxidic light metal compound increases the heat resistance by virtue of its melting point of 2,050 °C. The mixture of boric acid, potassium hydroxide solution and water glass replaces the usual antisettling agent clay, whose decomposition is precluded by reason of the low firing temperature, and increases the viscosity of the enamel slurry, so that, for example, spraying of the enamel is made possible. The addition of calcium formate also influences, in the interaction with the potassium ions available, the viscosity of the enamel slurry, so that applying the enamel to the substrate in the draining procedure is made possible. The addition of alcohol improves the stability of the admixture of high-grade steel powder in the enamel slurry, in particular in that larger accumulations of the metal on the surface of the enamel slurry are avoided. The admixture of the high-grade steel powder brings about the improvement of the heat conductivity and hence the reduction of the mechanical load by reduction of the difference of the heat expansion coefficients of the substrate and the enamel.

The enamel frit itself is manufactured in a known manner, in that, for example, different raw materials such as quartz, borax, feldspar, soda, saltpeter, cryolite, and others where applicable, are fused together at temperatures of up to 1,400 °C over a prolonged period of time in a rotary furnace or a melter over an appropriate period of time, thereupon the molten mixture is quenched in water or via water-cooled rollers and finally ground in a drum mill.

The coating which will be obtained with the enamel powder according to the invention and with the slurry according to the invention has a larger softening interval as a consequence of which the resistance against high thermal and mechanical loads is very good. Additionally, the coating will also have excellent heat conductive properties which is of vital importance in the application in heat exchangers.

In an embodiment, the enamel powder may contain, besides the enamel frit, 8 to 15 % of powder-form oxidic light metal compound and 8 to 20 % of high-grade steel powder. Good results have been obtained with an embodiment in which the oxidic light metal compound was corundum. The high-grade steel powder is preferably uncoated.

In an embodiment of the slurry, the mixture may contain, besides enamel powder, 1 to 10 % of alcohol, 0.1 to 2.5 % of calcium formate, 2 to 6 % of boric acid, 2 to 6 % of potassium hydroxide solution, 2 to 6 % of water glass.

Preferably, the alcohol is a monovalet alcolhol.

These embodiments provide to an enamel coating that meets the requirements which are necessary in the fierce environment existing in the flue gas drafts of a aluminium heat exchanger and that is resistant to the high temperature and mechanical loads existing in such environment.

Another object of the invention is achieved by providing a method for manufacturing an enamel slurry for producing an enamel coating on a metallic aluminium-containing substrate. The method includes:

- mixing enamel frit with up to 10 % of alcohol, up to 2.5 % of calcium formate, up to 6 % of boric acid, up to 6 % of potassium hydroxide solution, up to 6 % of water glass;
- stirring into the mixture up to 15 % of an oxidic light metal compound and up to 20 % of a high-grade steel powder in order to obtain a mixture including the enamel powder according to any one of claims 1-4; and
- adding water until the mixture has a density of approximately 1.85 g/cm^3.

In an embodiment, the method for manufacturing an enamel slurry may include that the high-grade steel powder together with the mixture is ground for 1 minute.

In an embodiment, the alcohol may added to the mixture after the grinding process.

In an embodiment, 1 to 10 % of quartz with a grain size > 50 µm may added during the grinding process.

The invention also provides a method for manufacturing an aluminium heat exchanger including a casted heat exchanger body bounding at least one flue gas draft and at least one water channel, wherein the method comprises:

- at least partially filling the flue gas draft with a enamel slurry according to any one of claims 5-8;

- draining the slurry from the flue gas draft;

- firing the heat exchanger body so as to form the enamel coating.

With this method, only the parts of the heat exchanger that are exposed to the aggressive constituents in the flue gases and the condensate may be covered which beneficial in view of costs.

In an embodiment, the aluminium surfaces of the heat exchanger body that have to be coated may be subjected to a pretreatment before the at least partially covering of the flue gas draft with an enamel slurry, wherein the pretreatment comprises at least partially removing the aluminium oxide layer from the surfaces to be coated.

Thus, a better bonding between the enamel coating and the aluminium walls of the heat exchanger will be obtained.

In an embodiment, the pretreatment may include a chemical treatment including subjecting the surface to an alkaline detergent followed by neutralization with diluted salpeter.

In an alternative embodiment, the pretreatment may include an annealing process followed by a cooling down. Due to the different thermal expansion coefficients of aluminium and aluminium oxide, the thin layer of aluminium oxide will be removed.

In an embodiment, the method may include:

- blowing dry the flue gas draft with a blow dryer after the draining and before the firing.

In order to obtain a durable coating, the firing may include heating the heat exchanger body at a temperature in the range of 510 °C to 610 °C for at least 60 minutes. Depending on the size of the heat exchanger this time may be longer. For a 28 kW heat exchanger, the firing time may be approximately 90 minutes.

Finally, the invention provides a heat exchanger including a casted aluminium heat exchanger body bounding at least one flue gas draft and at least one water channel, wherein at least a part of the flue gas draft is coated with a enamel coating formed from a slurry including the enamel powder according to the invention.

In an embodiment of the heat exchanger, the enamel coating may be formed from a slurry according to the invention.

The manufacture of an enamel slurry is advantageously implemented in that calcium formate is provided as saturated solution in water and is added in small portions until the intended viscosity is reached.

Preferably, the high-grade steel powder has a grain size < 50 µm.

For a casted aluminium heat exchanger with narrow flue gas drafts and surface enlarging pins and fins, the method described above including filling the flue gas drafts with slurry and draining the flue gas drafts is most practical to guarantee a good coverage of all parts of the flue gas draft with the slurry. However, the invention does not exclude other means for applying the slurry according to the invention on a substrate surface. For example, the application of the enamel slurry to the substrate, or the heat exchanger body, may be done by pouring on, or, alternatively spraying. After enamel slurry has been applied, it is dried and then fired. Temperature and duration of the firing depend on the volume of the substrate object. In this regard, it has proven advantageous to use for this an oven with air circulation, so as to utilize the convective flow of heat as well as possible.

In the following, the enameling of a heat exchanger is explained in more detail by way of exemplary embodiment.

First, the heat exchanger body consisting of a silicon- and magnesium-containing aluminium alloy is subjected to an annealing process during up to 60 minutes in a gas-heated oven at at least 350 °C in order to remove any adherent dirt and grease residues and to break up the aluminium oxide layer, in order to be able to apply the enamel slurry to the desired surfaces of the heat exchanger body. The enamel slurry is manufactured in a porcelain-walled mill, which is filled with porcelain balls for up to about 70 %. In the mill, 500 kg of suitable enamel frit as well as 50 kg of corundum, 50 kg of high-grade steel powder, 3 liters of methyl alcohol, 1 kg of calcium formate, 10 kg of boric acid, 20 kg of potassium hydroxide solution, 30 kg of water glass and 250 liters of water are added, mixed and ground, until the enamel slurry has a density of 1.85 gcm^{-3} and a grinding fineness, measured with a special sieve, of 3 parts according to Pemco. The finished enamel slurry is applied to at least a part of the heat exchanger surfaces which are influenced by flue gases and condensate during operation of the heat exchanger, for instance by pouring the enamel slurry on these surfaces or spraying it on by means of a spray gun. After run-off of the excess enamel slurry, the enameling of the heat exchanger is completed in that the adherent enamel slurry without prior drying is fired in a gas furnace for 90 minutes at 550 to 610 °C.

Claims

1. An enamel powder for manufacturing an enamel slurry for producing an enamel coating on a metallic aluminium-containing substrate, comprising an enamel frit admixed with up to 15 % of a powder-form oxidic light metal compound and up to 20 % of a high-grade steel powder.
2. The enamel powder according to claim 1, wherein the mixture contains, besides the enamel frit, 8 to 15 % of powder-form oxidic light metal compound and 8 to 20 % of high-grade steel powder.
3. The enamel powder according to claim 1 or 2, wherein the oxidic light metal compound is corundum.
4. The enamel powder according to claim 1, 2 or 3, wherein the high-grade steel powder is uncoated.
5. An enamel slurry for producing an enamel coating on a metallic aluminium-containing substrate, wherein the slurry comprises an amount of water and a mixture, wherein the mixture includes:

   - an enamel powder according to claim 1; and
   - up to 10 % of alcohol, up to 2.5 % of calcium formate, up to 6 % of boric acid, up to 6 % of potassium hydroxide solution, and up to 6 % of water glass.
6. The enamel slurry of claim 5, wherein the amount of water such that the slurry has a density of substantially 1.85 g/cm^3.
7. The enamel slurry according to claim 5 or 6, wherein the mixture contains, besides enamel powder, 1 to 10 % of alcohol, 0.1 to 2.5 % of calcium formate, 2 to 6 % of boric acid, 2 to 6 % of potassium hydroxide solution, 2 to 6 % of water glass.
8. The enamel slurry according to any one of claims 5-7, wherein the alcohol is a monovalent alcohol.
9. A method for manufacturing an enamel slurry for producing an enamel coating on a metallic aluminium-containing substrate, wherein the method includes:

- mixing enamel frit with up to 10 % of alcohol, up to 2.5 % of calcium formate, up to 6 % of boric acid, up to 6 % of potassium hydroxide solution, up to 6 % of water glass;
- stirring into the mixture up to 15 % of an oxidic light metal compound and up to 20 % of a high-grade steel powder in order to obtain a mixture including the enamel powder according to any one of claims 1-4; and
- adding water until the mixture has a density of approximately 1.85 g/cm^3.

10. The method for manufacturing an enamel slurry according to claim 9, wherein the calcium formate is added dissolved in water.
11. The method for manufacturing an enamel slurry according to claim 9 or 10, wherein the high-grade steel powder together with the mixture is ground for 1 minute.
12. The method for manufacturing an enamel slurry according to claim 11, wherein the alcohol is added to the mixture after the grinding process.
13. The method for manufacturing an enamel slurry according to any one of claims 11-12, wherein during the grinding process 1 to 10 % of quartz with a grain size > 50 µm is added.
14. A method for manufacturing an aluminium heat exchanger including a casted heat exchanger body bounding at least one flue gas draft and at least one water channel, wherein the method comprises:
    - at least partially filling the flue gas draft with a enamel slurry according to any one of claims 5-8;
    - draining the slurry from the flue gas draft;
    - firing the heat exchanger body so as to form the enamel coating.
15. The method according to claim 14, wherein the aluminium surfaces of the heat exchanger body that have to be coated are subjected to a pretreatment before the at least partially filling of the flue gas draft with an enamel slurry, wherein the pretreatment comprises at least partially removing the aluminium oxide layer from the surfaces to be coated.
16. The method according to claim 15, wherein the pretreatment includes a chemical treatment including subjecting the surface to an alkaline detergent followed by neutralization with diluted salpeter.

17. The method according to claim 15, wherein the pretreatment includes an annealing process followed by a cooling down.
18. The method according to any one of claims 14-17, including:
    - blowing dry the flue gas draft with a blow dryer after the draining and before the firing.
19. The method according to any one of claims 14-18, wherein the firing includes heating the heat exchanger body at a temperature in the range of 510 °C to 610 °C for at least 60 minutes.
20. A heat exchanger including a casted aluminium heat exchanger body bounding at least one flue gas draft and at least one water channel, wherein at least a part of the flue gas draft is coated with a enamel coating formed from a slurry including the enamel powder according to claim 1.
21. The heat exchanger according to claim 20, wherein the enamel coating is formed from a slurry according to any one of claims 5-8.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
INV. C03C8/02 C03C8/14 C03C8/18 C03C14/00 F24H1/00
F24H9/00
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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)
C03C F24H

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, WPI Data

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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