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(54) **METHOD FOR THE APPLICATION OF AN ANTICORROSIVE, PROTECTIVE, NIOBIUM-OXIDE COATING APPLIED BY THERMAL SPRAYING**

VERFAHREN ZUM AUFTRAGEN EINER ANTI-KORROSIVEN NIOB-OXID SCHUTZSCHICHT MITTELS THERMISCHEN SPRITZENS

PROCÉDÉ D'APPLICATION D'UN REVÊTEMENT À BASE D'UN OXYDE DE NIOBIUM PAR PULVÉRISATION THERMIQUE

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Description

[0001] Description of the Patent of Invention for "Niobium-based Compositions and Coatings, Niobium Oxides and Their Alloys Applied by Thermal Spraying and Their Use as an Anticorrosive".

THECHNICAL FIELD

[0002] This invention refers to the application of niobium oxide coatings by means of thermal spraying for the purpose of an anticorrosive protection in highly corrosive environments, mainly those wihc present high temperatures, show presence of gases such as H₂S, SO₂, CO₂ as well as organic and inorganic acids.

PRIOR TECHNIQUES

[0003] The Brazilian Patent Application n° PI 0102414-0 refers to niobium-based compositions and coatings, niobium oxides and their possible associations with other oxides and their use by means of common painting techniques and not by electrodeposition through molten salts or equivalents thereof used in electroplating, it being understood that same is proposed to neutralize the highly corrosive effect of naphthenic acids and sulphur components, which very quickly destroy carbon steel and special alloys such as stainless steel of virtually all chromium alloys and nickel alloys families, and for use not only as a coating in petroleum refining units, but also in industrial units which present similar problems.

SUMMARY OF THE INVENTION

[0004] In its most general aspect, this invention proposes the use niobium oxides as an anticorrosive coating applied by thermal spraying over carbon steel surfaces and other metallic materials of current use in industrial centers. Such coatings are known for example in the prior art JP62158834.

DETAILED DESCRIPTION OF THE INVENTION

[0005] This invention refers to niobium oxide formulations by granulometric classification that allows their application by thermal spraying equipment.

[0006] The components referred to above obey the following characteristics:

Boiling point:	a maximum of 2000°C
Density:	4.47-8.0 g/cm ³
% niobium oxide:	99.4
Sulphur ppm:	10
Fe ppm:	229
Pb ppm:	< 1
Granulometry:	[-180 to 45 μm]

[0007] Thermal spraying is a coating process in which metallic or non-metallic materials in the form of a powder or a wire are molten in the nozzle of appropriate guns, and then projected under pressure towards the surface to be coated. Due to the high pressure, these molten materials come out from the guns as microdroplets which, upon approaching, the substrate become grouped and then are deposited in the form of flakes", sometimes also called "pancakes". From this moment on, there are depositions of successive layers and thus a thermo-sprayed characteristic coating provided with superposed pancakes". These flakes are affixed to the substrate by mechanical anchoring processes, and therefore there is the need of a prior preparation of the substrate so as to create the required anchoring conditions.

[0008] The surface to be sprayed thermically must be previously cleaned. The cleaning process consists of the following phases:

1 - Surface Pre-cleaning.

It consists of the removal of sludge, corrosion products, coatings residues, insoluble incrustations, scales and gross particulate through mechanical processes (hydro-jetting or abrasive blasting), chemical processes (degreasing, acid cleaning) or thermal processes (burning, direct flame, reducing atmosphere).

2 - Final Cleaning.

It consists of the preparation of the surface by the blasting process so as to eliminate products that prevent the contact between the coating and the substrate. The surface must achieve a Sa3 cleaning quality and a given roughness, that is surface conditions allowing for the adherence of the coating to the substrate.

3 - Preheating.

[0009] Preheating during its pre-cleaning is, in fact, a cleaning by the action of a flame and its purpose is to provide the burning and volatilization of greases, oils and humidity retained at the metal substrate in the event of failure of the other cleaning methods. It can also be used after the final cleaning so as to reduce all residual tensions (which have an influence upon the adhesion and cohesion of the layer) and to remove any residual humidity. The preheating temperature values depend further upon the material of the layer, of the type of the substrate, and of its physical properties.

[0010] The coating process refers to the example 1 which consists of the application of niobium oxide by thermal spraying to the flame. Example 1: Application of a niobium oxide layer over a 3mm thick carbon steel plate.

[0011] Initially, the surface is blasted, to get it cleaned and to obtain the desired roughness, with white aluminium oxide granulometry 30 alundum 38 A; to obtain the Sa3 cleaning degree by comparison with all surface quality standards as published by NACE RM 01/70 rule. Then the surface must be heated for removal of humidity; to -control the heating so that the temperature is not in excess of 150°C; in the sequence, to apply a pre-layer of 40A1-60Nb agglomerate. Finally, to fill up the equipment with niobium oxide with a granulometry between -180 to 45 μm, to adjust the parameters and carry out the application.

[0012] Parameters for application of niobium oxide:

Oxygen pressure: 2.0 to 4.0 kg/cm²

Acetylene pressure 0.5 to 1.0 kg/cm²

Deposition rate regulation(*): 5-15

(*): Depending on the thickness of the layer to be applied, the deposition rate must be altered.

[0013] Upon application the deposited layer must have the following characteristics:

A - Thickness

Verify the thickness using a thickness meter specific for measuring non magnetic layers over magnetic substrates.

For a primer the thickness of the layer applied must be in the range of 100μm.B

B - Adherence

Glue to the applied layer with an adhesive, a peg of 25.4 mm in diameter which upon curing is tensioned in a tension machine. The adherence value must be between 10 and 40 Mpa.

C - Electrode Power

Up to 100°C the electrochemical power measured in relation to the saturated calomel over a carbon steel substrate must show the value of -600 +/- 50mV_{ecs}.

D - Instilling of HCl of P.A. purity

It must not show deterioration in the coating and show yellow oxidation characteristic of the oxide reaction.

[0014] The above defined parameters should not be deemed to be restricted to the scope of this invention, as other forms of application by thermal spraying can be used.

Claims

1. Method for the application of an anticorrosive niobium oxide coating having a melting point upto a maximum of 2000°C, a density of 4.47-8 g/cm³ and a granularity of -180 - 45 μm. The said coating being applied by thermal spraying and the substrate being prepared by applying a pre-layer to provide the required anchoring conditions, **characterised in that** the said pre-layer is an agglomerate of 40 Al - 60 Nb.

2. Method according to any of the above claims, **characterized by** using an oxygen pressure of 2.0 to 4.0kg/cm² and an acetylene pressure of 0.5 to 1.0kg/cm² during coating.

3. Method according to claim 2, **characterized by** a deposition rate regulation of 5-15.

4. Method according to any of the above claims, **characterized by** said preparation including pre-cleaning and final cleaning.

5. Method according to claim 4, **characterized by** said final cleaning being achieved by means of blasting.

6. Method according to claim 4 or 5, **characterized by** said preparation also including heating to remove humidity.

7. Method according to claim 5, **characterized by** said blasting being performed to achieve a Sa3 cleaning degree by comparison with all surface quality standards as published by NACE RM01/70 rule.

5 **Patentansprüche**

1. Verfahren zum Auftragen einer antikorrosiven schützenden Niob-OxidBeschichtung, welche einen Schmelzpunkt bis zu einem Maximum von 2000°C, eine Dichte von 4.47 bis 8 g/cm³ und eine Korngröße von 180-45 µm aufweist, wobei die Beschichtung durch thermisches Spritzen aufgebracht wird und das Substrat durch Aufbringen einer Vorschicht vorbereitet wird, um die erforderlichen Bindungsbedingungen herzustellen, **dadurch gekennzeichnet, dass** die Vorschicht ein Agglomerat aus 40A1-60Nb ist.
2. Verfahren nach Anspruch 1, **gekennzeichnet durch** die Verwendung eines Sauerstoffdrucks von 2.0 bis 4.0 kg/cm² und eines Azetylendrucks von 0.5 bis 1.0 kg/cm² während der Beschichtung.
3. Verfahren nach Anspruch 2, **gekennzeichnet durch** eine Abscheidungsratensteuerung von 5 bis 15.
4. Verfahren nach einem der vorhergehenden Ansprüche, **gekennzeichnet dadurch, dass** die Vorbereitung eine Vorreinigung und eine Endreinigung umfasst.
5. Verfahren nach Anspruch 4, **dadurch gekennzeichnet, dass** die Endreinigung durch Reinigungsstrahlen erreicht wird.
6. Verfahren nach Anspruch 4 oder 5, **dadurch gekennzeichnet, dass** die Vorbereitung auch ein Aufheizen zum Entfernen von Feuchtigkeit umfasst.
7. Verfahren nach Anspruch 5, **dadurch gekennzeichnet, dass** das Reinigungsstrahlen durchgeführt wird, um einen Sa3-Reinheitsgrad im Vergleich mit allen Oberflächenqualitätsstandards wie in der NACE RM01/70-Regel veröffentlicht zu erreichen.

Revendications

1. Procédé pour l'application d'un revêtement protecteur anticorrosion d'oxyde de niobium de point de fusion ne dépassant pas 2 000 °C, de masse volumique 4,47 à 8 g/cm³ et de granularité allant de -180 à 45 µm, ledit revêtement étant appliqué par pulvérisation thermique et le substrat étant préparé par application d'une pré-couche servant à fournir les conditions d'ancrage requises, **caractérisé en ce que** ladite pré-couche est un agglomérat de 40 A1 - 60 Nb.
2. Procédé selon la revendication 1, **caractérisé en ce que** l'on utilise une pression d'oxygène de 2,0 à 4,0 kg/cm² et une pression d'acétylène de 0,5 à 1,0 kg/cm² pendant l'application du revêtement.
3. Procédé selon la revendication 2, **caractérisé par** une régulation de la vitesse de dépôt de 5-15.
4. Procédé selon l'une quelconque des revendications précédentes, **caractérisé par le fait que** ladite préparation comprend un nettoyage préalable et un nettoyage final.
5. Procédé selon la revendication 4, **caractérisé par le fait que** ledit nettoyage final est effectué par décapage au jet.
6. Procédé selon la revendication 4 ou 5, **caractérisé par le fait que** ladite préparation comprend aussi un chauffage destiné à éliminer l'humidité.
7. Procédé selon la revendication 5, **caractérisé par le fait que** ledit décapage au jet est réalisé de manière à obtenir un degré de nettoyage Sa3 par comparaison avec toutes les normes de qualité de surface publiées par la règle NACE RM01 /70.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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- JP 62158834 B [0004]