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(54) **PUMPS FOR HOT AND CORROSIVE FLUIDS**

PUMPEN FÜR HEISSE UND KORROSIVE FLÜSSIGKEITEN

POMPES POUR FLUIDES CHAUDS ET CORROSIFS

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Description

TECHNICAL FIELD

5 **[0001]** The present disclosure relates to mechanical pumps for pumping hot, corrosive and erosive fluids like liquid metals and molten salts. The pumps can be of any construction or mechanism of operation, such as radial/centrifugal or axial, wherein at least one component is in contact with the liquid metal or molten salt.

BACKGROUND

10 **[0002]** The effective service life time of conventional mechanical pumps for liquid metals, normally made of iron base alloys, is typically only a few months or perhaps somewhat longer for molten salt pumps, depending on type of metal or salt and operating temperature.

15 **[0003]** Ceramic pumps or ceramic impellers that may have excellent corrosion and erosion resistance are known in the art, for example through US 3,776,660 and US 6,019,576. Suggested ceramic materials include graphite and silicon carbide, as disclosed for example in US 5,586,863. However, ceramic materials are mechanically sensitive and break easily when subjected to stress. For instance the fracture toughness of ceramic materials including graphite is generally considered to be too low for use as pump components.

20 **[0004]** Electro magnetic pump technologies have received a lot of interest in the nuclear industry. A NASA technical report, Liquid-Metal Pump Technologies for Nuclear Surface Power (NASA/TM-2007-214851) gives a review thereof. However, the use of electro-magnetic pumps for liquid metals is disadvantageous due to their poor efficiency.

25 **[0005]** Pumps for liquid metals and molten salts are used in metallurgical industry, metal coating industry and in chemical industry as well. Additionally there is a growing market for new energy production techniques using liquid metal or molten salts as an energy carrier and/or coolant, such as in liquid metal nuclear reactors, concentrated solar power plants (CSP) and in fusion reactors. Additionally, molten salts are also used for storage of thermal energy.

30 **[0006]** The pumps have shown to be a critical component in such nuclear or solar power plants since the overall energy efficiency is dependent on the maximum allowed fluid temperature and flow rate. Today, the maximum operating temperature and choice of fluid is basically limited by corrosion and erosion problems of the pump components. Temperatures of the liquid metals and molten salts used for energy production are typically in the range 200-500 °C but it is desirable to go higher in the future, possibly up to 800 °C.

[0007] US20040115079A1 disclose a pump for molten metal such as such as aluminium, copper, iron, zinc and alloys thereof having a protective coating. The protective coating is preferably formed of a ceramic and most preferably of nitridebonded silicon carbide. But other suitable, oxidation resistant materials may be used, such as aluminium oxide or other ceramics. Cement is used as an intermediate binding layer.

35 **[0008]** Lim Jun Et Al: "Design of alumina forming FeCrAl steels for lead or lead-bismuth cooled fast reac", JOURNAL OF NUCLEAR MATERIALS, discloses the use of TiN as precipitates into a FECrAl steel substrate to strengthen the steel, not as a part of part of an intermediate binding layer. The Al in the steel forms a protective AL₂O₃ layer. No intermediate binding layer is present.

40 **[0009]** Rivai et al.: "Compatibility of surface-coated steels, refractory metals and ceramics to high temperature lead-bismuth eutectic", PROGRESS IN NUCLEAR ENERGY, tests the compatibility of surface-coated steels, refractory metals and ceramics to high temperature lead-bismuth eutectic. The surface-coated steel material was Al-steel-sputtering coated STBA26 which was coated by physical vapor deposition (PVD) technique using the Unbalanced Magnetron Sputtering (UBMS) method. An oxide layer is formed in the aluminium coating. The Al layer is adhered directly to the steel without an intermediate binding layer comprises at least one layer of TiN.

45 **[0010]** Kura et al: "Corrosion behavior of Al-surface-treated steels in liquid Pb-Bi in a pot", JOURNAL OF NUCLEAR MATERIALS, discloses corrosion tests of Al-surface treated steels ion liquid Pb-Bi. The Al- surface is produced by gas diffusion or melt dipping. The Al layer is adhered directly to the steel without an intermediate binding layer comprises at least one layer of TiN.

50 SUMMARY

[0011] One object of the present disclosure is to provide a pump for hot and corrosive fluids wherein the pump has an extended service life.

[0012] A particular object is to provide a pump for molten lead or lead bismuth eutectic (LBE) for use in nuclear reactors.

55 **[0013]** This and other objects are achieved by the pump defined in independent claim 1. Further advantageous embodiments have been specified in the dependent claims.

DETAILED DESCRIPTION

[0014] Before the present invention is described, it is to be understood that the terminology employed herein is used for the purpose of describing particular embodiments only and is not intended to be limiting, since the scope of the invention will be limited only by the appended claims.

[0015] The present disclosure offers a solution to the shortcomings associated with mechanical pumps, mainly erosion and corrosion problems, when using high temperature fluids as energy carrier, energy storage and coolants for future energy production techniques as well as extending the service life time of pumps for the metallurgical industry etc.

[0016] By using a metallic substrate, a cermet or a cemented carbide substrate with a metallic binder phase which has good mechanical properties in combination with corrosion and erosion resistant coatings, which are sufficiently well matched regarding thermal expansion, thus enabling good coating adherence, it is possible to construct a mechanical pump for hot fluids with excellent service life time.

[0017] The outer surface layer must be hard enough to possess good erosion resistance. Among oxides, the better quality of alumina has 9 out of 10 on the Mohs hardness scale of abrasives and zirconia has a hardness of 8. Alumina and zirconia exhibit good corrosion resistance in for instance liquid lead and LBE. In general, these oxides are suitable for use when pumping a variety of different liquid metals and molten salts of technical interest.

[0018] In the same manner alumina has been shown to be highly corrosion resistant in sulphate, nitrate and carbonate molten salts of interest, as an example, for use in concentrated solar power (CSP) applications. Examples of technically interesting salts include, but are not limited to, sodium and potassium nitrates (Na, K)NO₃ and lithium, sodium and potassium carbonates (Li,Na,K)CO₃.

[0019] Several carbides and nitrides such as WC, TiC, B₄C, SiC, TaC, ZrC, TiN, ZrN, BN and carbo-nitrides such as TiCN and TiAlN have a Mohs hardness of 8.5-9.8 and exhibit good erosion resistance and possess sufficiently good high temperature oxidation and corrosion resistance in most of the liquid metals and molten salts of interest for the different industrial applications. For instance, TiC and ZrC have been tested in liquid Pb, Na and Li around 800°C and showed good resistance. However, the adherence of such layers is generally not sufficient for pump components exposed to fluids with high flow rates. For more demanding pump applications, such as in nuclear reactors, it is necessary to provide an additional outer oxide coating, preferably based on alumina or zirconia.

[0020] In the same manner alumina has been shown to be most resistant in molten salts such as potassium and sodium nitrates used for example in concentrated solar power systems (CSP).

[0021] In order to achieve perfect growth of an outer protective alumina scale by chemical vapour deposition (CVD) or physical vapour deposition (PVD) it is required to first provide an intermediate layer of a nitride to promote the ideal oxide structure. According to the invention this intermediate layer is TiN. The substrate metal should have a thermal expansion coefficient that is sufficiently close to the coating or coatings, i.e. in the case of alumina as an outer coating, the substrate could preferably be cemented carbide with a metallic binder.

[0022] Yttria-stabilized zirconia (YSZ), could for example be used on iron and nickel base alloy substrates since these have more similar thermal expansion coefficients. Suitable techniques to apply YSZ or Thermal Barrier Coatings (TBC) on a metal substrate are Electron Beam Physical Vapor Deposition (EBPVD), Air Plasma Spray (APS), High Velocity Oxygen Fuel (HVOF), Electrostatic Spray Assisted Vapour Deposition (ESAVD) or Direct Vapor Deposition.

[0023] A pump for hot and corrosive fluids according to aspects and embodiments of the present disclosure comprises at least one component coated with one or more wear and erosion resistant surface layers. This component can be an impeller vane, an impeller or a part of or the entire inner surface of the pump housing. The impeller is the most important part since it is subjected to high flow rates.

[0024] The substrate material may be selected from the following group of materials: steels, stainless steels, nickel-, cobalt-, molybdenum-, tantalum- and tungsten-based alloys, cermets and cemented carbides. A material of particular interest is tungsten carbide, which can be without a binder, or include a metallic binder.

[0025] The binder can comprise at least one metal selected from the group of Co, Ni, Fe, Cr, Al, Mn, Mo, V, Ti, Ta, Zr and Nb. According to a preferred embodiment the binder is chosen with respect to its stability in liquid lead and LBE at high temperatures and at low oxygen potentials.

[0026] The surface coating comprises at least one layer of refractory oxide, the oxides comprises aluminium oxide and zirconium oxide and stabilized or doped oxides thereof.

[0027] The total thickness of the surface coating is 0.2 μm to 1000 μm depending on the selected coating materials and application techniques. CVD coatings typically have a thickness of about 1 to about 20 μm and APS coatings typically have a thickness of about 50 to about 600 μm.

[0028] In a preferred embodiment, the pump comprises an impeller or at least one impeller vane made of a nickel-based alloy provided with a surface coating comprising one outer layer of yttria stabilized zirconia (YSZ).

[0029] Another preferred embodiment comprises of an impeller or at least one impeller vane wherein the substrate is a cemented carbide and the coating comprises at least one intermediate layer comprising TiN and a surface layer of Al₂O₃. Preferably, the cemented carbide comprises at least 80 % WC and a metallic binder, the metallic binder comprising

at least on metal selected from the group of Co, Ni, Fe, Cr, Al, Mn, Mo, V, Ti, Ta, Zr and Nb. The binder is chosen from Fe, Ni, Cr, Mo and Al, preferably the binder contains an amount of Al such that it easily can form aluminium oxide if the surface coating should be damaged.

[0030] A particular use of the coated cemented carbide is the use as a structural material for at least one pump component in a pump for pumping liquid lead or LBE, in particular in a nuclear reactor, wherein the liquid lead or LBE has an oxygen concentration below the lead oxide formation limit.

[0031] Another use is the pumping of molten salts, such as carbonate, nitrate and sulphate salts for use in energy applications such as CSP.

[0032] A nickel-based alloy comprising an outer layer of yttria-stabilized zirconia (YSZ) is also a suitable material for the above uses.

EXAMPLE 1

[0033] In the present example a laboratory-screening test of seven different materials were conducted in flowing lead, which simulates the erosive and corrosive environment a pump component would be exposed to in a liquid heavy metal. The materials and material combinations that were tested are presented in Table 1 below.

Table 1. Material samples subjected to comparative erosion and corrosion test	
No.	Description
1	Uncoated carbon steel
2	Uncoated austenitic stainless steel
3	Uncoated Kanthal APMT FeCrAl alloy
4	Pre-oxidized Kanthal APMT FeCrAl alloy, i.e. with a thin in-situ formed alumina scale
5	Commercial cemented carbide TiN coated tungsten carbide (WC + 6% Co)
6	Commercial Al ₂ O ₃ (ceramic)
7	Commercial thermal barrier coating (TBC) coated nickel-based alloy. Substrate, Haynes 230, air plasma spray (APS) coated with 360 µm yttria stabilized zirconia (YSZ) (ZrO ₂ with 7% Y ₂ O ₃) as a topcoat and 150 µm intermediate binder coating comprising of Ni-Co-Cr-Al-Y.

[0034] An erosion test facility was constructed by the inventors. In the erosion test facility, housing up to 10 kg of lead, a theoretical flow rate up to 10 m/s could be achieved by rotating the liquid metal using a rotating disk with a diameter of 15 cm. The disk, immersed into the liquid metal, was rotated using an electric motor coupled to the disk using a stainless steel shaft. Air ingress was reduced to a minimum using standard vacuum copper sealing, and the amount of dissolved oxygen in the liquid lead was controlled by means of an Ar-H₂-H₂O gas mixture. The test samples were placed close to the periphery of the rotating disk, where the highest flow rates were expected. The facility was heated using a 1000 W band heater mounted on the outside of facility and the temperature was controlled using thermocouples of type K and a PID regulator. The test conditions are shown in Table 2.

Table 2. Test conditions	
Amount of lead	10 kg (approximately 1 dm ³)
Flow rate (theoretical)	7 m/s
Temperature	550°C
Oxygen concentration	10 ⁻⁷ weight-%
Exposure time	300 h

[0035] The samples were removed after 300 h and post-exposure examination was carried out using a standard optical microscope reaching magnifications up to about 1000x. The tested materials were evaluated with respect to before and after appearance, color and erosion cavitation, as well as from the amount of lead attached to the sample, i.e. wettability.

[0036] The screening test revealed large differences in erosion and corrosion resistance between the tested specimens. The uncoated steel samples 1 - 3 showed severe erosion and corrosion damage, meaning a noticeable shift in appearance

from smooth metallic surfaces to dark uneven and notched surfaces. The least damage was found on the uncoated FeCrAl alloy, sample 3. The coated samples, including the pre-oxidized FeCrAl alloy, sample 4, showed no or little damage.

[0037] Sample 6, the commercial Al_2O_3 -ceramic and sample 7, the YSZ coated Nibase ally, were virtually unaffected. The commercial cemented carbide sample, coated with TiN, showed a significantly better erosion resistance than the uncoated steels in the test.

[0038] The screening test thus confirmed that structural components of a pump for liquid lead could be effectively protected from the bulk metal by stable coatings. Particular useful coatings comprise Al_2O_3 and ZrO_2 .

[0039] The screening test thus confirmed that structural components of a pump for liquid lead could be effectively protected from the bulk metal by stable coatings. Particular useful coatings comprise Al_2O_3 and ZrO_2 . Pumps with such coatings or including components with such coatings are well suited for use in different systems for the generation of energy, such as but not limited to concentrated solar power and nuclear energy. They are particularly well suited for use in lead or LBE cooled reactors in the nuclear industry.

[0040] The invention has mainly been described above with reference to a few embodiments. However, as is readily appreciated by a person skilled in the art, other embodiments than the ones disclosed above are equally possible within the scope of the invention, as defined by the appended patent claims.

Claims

1. A pump for pumping molten metal or molten salt, said pump comprising at least one component manufactured of a substrate and coated with one or more wear and erosion resistant surface layers, wherein said substrate is provided with at least one intermediate binding layer and one outer layer, and said outer layer comprises at least one refractory oxide **characterised in that** the intermediate binding layer comprises at least one layer of TiN
2. The pump according to claim 1, wherein the thickness of the surface coating is 0.2 μm to 1000 μm .
3. The pump according to claim 1, wherein the thickness of the surface coating is 5 - 600 μm .
4. The pump according to any one of the preceding claims, wherein the intermediate binding layer having a thickness of 0.1 - 5 μm .
5. The pump according to any one of the preceding claims, wherein the substrate is a cemented carbide and the refractory oxide outer layer comprises at least 90 % Al_2O_3 .
6. The pump according to claim 5, wherein the outer layer comprises at least 99 % Al_2O_3 .
7. The pump according to claim 5, wherein the cemented carbide comprises at least 80 % WC and a metallic binder, said metallic binder comprising at least one metal selected from the group of Co, Ni, Fe, Cr, Al, Mn, Mo, V, Ti, Ta, Zr and Nb.
8. The pump according to claim 7, wherein said metallic binder comprises Co and/or Fe and/or Ni and/or Cr and/or Mo in combination with Al.
9. The pump according to claim 8, wherein the binder is a Fe-Al, Ni-Al or a Ni-Cr-Mo-Al binder.

Patentansprüche

1. Pumpe zum Pumpen von geschmolzenem Metall oder geschmolzenem Salz, die Pumpe umfassend zumindest eine Komponente, die aus einem Substrat hergestellt und mit einer oder mehreren verschleiß- und erosionsbeständigen Oberflächenschichten beschichtet ist, wobei das Substrat mit mindestens einer Zwischenbindungsschicht und einer Außenschicht versehen ist und die Außenschicht mindestens ein feuerfestes Oxid umfasst, **dadurch gekennzeichnet, dass** die Zwischenbindungsschicht mindestens eine Schicht aus TiN umfasst.
2. Pumpe nach Anspruch 1, wobei die Dicke der Oberflächenbeschichtung 0,2 μm bis 1000 μm beträgt.
3. Pumpe nach Anspruch 1, wobei die Dicke der Oberflächenbeschichtung 5 - 600 μm beträgt.

4. Pumpe nach einem der vorhergehenden Ansprüche, wobei die Zwischenbindungsschicht eine Dicke von 0,1 - 5 μm aufweist.
5. Pumpe nach einem der vorhergehenden Ansprüche, wobei das Substrat ein Sinterkarbid ist und die Außenschicht aus feuerfestem Oxid mindestens 90 % Al_2O_3 umfasst.
6. Pumpe nach Anspruch 5, wobei die Außenschicht mindestens 99 % Al_2O_3 umfasst.
7. Pumpe nach Anspruch 5, wobei das Sinterkarbid mindestens 80 % WC und ein metallisches Bindemittel umfasst, wobei das metallische Bindemittel mindestens ein Metall umfasst, das aus der Gruppe von Co, Ni, Fe, Cr, Al, Mn, Mo, V, Ti, Ta, Zr und Nb ausgewählt ist.
8. Pumpe nach Anspruch 7, wobei das metallische Bindemittel Co und/oder Fe und/oder Ni und/oder Cr und/oder Mo in Kombination mit Al umfasst.
9. Pumpe nach Anspruch 8, wobei das Bindemittel ein Fe-Al-, Ni-Al- oder ein Ni-Cr-Mo-Al-Bindemittel ist.

Revendications

1. Pompe pour pomper un métal fondu ou un sel fondu, ladite pompe comprenant au moins un composant fabriqué à partir d'un substrat et revêtu d'une ou plusieurs couches de surface résistantes à l'usure et à l'érosion, dans laquelle ledit substrat est doté d'au moins une couche de liaison intermédiaire et d'une couche extérieure, et ladite couche extérieure comprend au moins un oxyde réfractaire, **caractérisée en ce que** la couche de liaison intermédiaire comprend au moins une couche de TiN.
2. Pompe selon la revendication 1, dans laquelle l'épaisseur du revêtement de surface est de 0,2 μm à 1000 μm .
3. Pompe selon la revendication 1, dans laquelle l'épaisseur du revêtement de surface est de 5 à 600 μm .
4. Pompe selon l'une quelconque des revendications précédentes, dans laquelle la couche de liaison intermédiaire a une épaisseur de 0,1 à 5 μm .
5. Pompe selon l'une quelconque des revendications précédentes, dans laquelle le substrat est un carbure cémenté et la couche extérieure en oxyde réfractaire comprend au moins 90 % d' Al_2O_3 .
6. Pompe selon la revendication 5, dans laquelle la couche extérieure comprend au moins 99 % d' Al_2O_3 .
7. Pompe selon la revendication 5, dans laquelle le carbure cémenté comprend au moins 80 % de WC et un liant métallique, ledit liant métallique comprenant au moins un métal choisi dans le groupe constitué par Co, Ni, Fe, Cr, Al, Mn, Mo, V, Ti, Ta, Zr et Nb.
8. Pompe selon la revendication 7, dans laquelle ledit liant métallique comprend du Co et/ou du Fe et/ou du Ni et/ou du Cr et/ou du Mo en combinaison avec de l'Al.
9. Pompe selon la revendication 8, dans laquelle le liant est un liant de type Fe-Al, Ni-Al ou Ni-Cr-Mo-Al.

REFERENCES CITED IN THE DESCRIPTION

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