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(54) Title: A METHOD OF HEAT TRANSFER OF A NON-METALLIC OR METALLIC ITEM

(57) Abstract: The present invention relates to a method of heat treatment of non-metallic or metallic item comprising at least one step A) of heat transfer between the item and a heat transfer fluid A' comprising a fluid medium and nanoparticles, the heat transfer fluid having a heat transfer coefficient below the heat transfer coefficient of water.



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A method of a heat transfer of a non-metallic or metallic item

The present invention relates to a method of heat transfer of non-metallic or metallic item. In particular, it is well suited for steel, aluminum, stainless steel, copper, iron, copper alloys, titanium, cobalt, metal composite, nickel Industries or non-metallic industries such as plastics.

With a view of saving energy consumption, it is possible to improve the performance of heat exchangers systems and to introduce various heat transfer enhancement techniques. Most of techniques have focused on the improvement of heat transfer performance, i.e. the improvement of heat transfer coefficient. Usually, air pressure or fluids such as water, engine oil, ethylene glycol, etc. are used as heat transfer fluid sometimes with additives to improve heat transfer performance.

Additionally, recent investigations in nanotechnology have allowed the development of a new category of heat transfer fluid comprising nanoparticles. Such fluids also called "Nanofluids" are liquid suspension containing particles having at least one dimension below 100nm. These heat transfer fluids have usually an increased heat transfer coefficient.

Such fluids can be used for accelerating the heat transfer, for example the cooling, between a non-metallic or metallic and a heat transfer fluid. For example, in steel making industry, during the cooling process in a hot rolling process, the run-out table cools the steel strip from approximately 800–950°C at the entrance to 450–600°C at the exit. Thus, for some steel grades, a heat transfer fluid having high heat transfer coefficient is needed.

However, it is sometimes necessary to slow down the heat transfer between a non-metallic or metallic item and a heat transfer fluid. For example, during the manufacture of the medium- to high-carbon steels rods, the cooling is a step really important. Indeed, the cooling has to be really slow and controlled in order to obtain a rod having a fine pearlitic microstructure and of uniform quality. If the cooling is too rapid, the risk is to obtain martensitic structure which is detrimental for the manufacture of steel rods having improved strength and drawability.

The patent application EP0126481 discloses a method of direct heat treatment of a medium- to high-carbon steel rod by performing controlled cooling

on an expanded spiral coil of a hot rolled medium to high-carbon steel rod that has an austenitic structure. The cooling is performed by passage of the coil through a vessel containing a coolant composed of a gas bubble-water mixed fluid under a strong turbulent action which contains a uniform dispersion of oxidizing gas bubbles and which is held a predetermined temperature not higher than 95°C.

However, there is a risk not to have fine gas bubbles having a uniform size distribution. Indeed, the volume of bubble gas in a vessel is difficult to control. When the gas is blown into the warm water, the resulting bubbles are warmed up and the warm water evaporates into the bubbles until the equilibrium state is reached, and as a result, there occurs an almost instantaneous swelling of the bubbles. Additionally, a specific superficial velocity has to be respected to avoid the coalescence of gas bubbles together to form a single gaseous phase. Finally, a large volume of gas is needed in order to prepare the gas bubble-water mixed fluid containing oxidizing gas bubbles. Consequently, in industrial scale, the use of such fluid containing gas is difficult to handle and it is high costs effective.

The purpose of the invention is to provide an easy to implement method of heat treatment of a non-metallic or metallic item comprising at least one heat transfer between the item and a heat transfer fluid having a heat transfer coefficient below the heat transfer coefficient of water. In particular, there is a need to provide a method wherein the heat transfer can be slow and controlled.

This is achieved by providing a method of heat transfer between a metallic or non-metallic item and a heat transfer fluid according to claims 1 to 26.

The following terms are defined:

- heat transfer fluid comprising nanoparticles (so-called Nanofluid) means a liquid suspension containing particles having at least one dimension below 100nm,
- laminar flow means a flow with a Reynolds number below a critical value of approximately 2300,
- turbulent flow means a flow with a Reynolds number larger than a critical value of about 4000,
- Percolation threshold concentration is the concentration of nanoparticles above which they are connected forming a long-range network. For heat transfer applications, it is suitable that such network connects the hottest part, i.e. the part where the heat starts to flow, of the fluid and the coldest part of the fluid, i.e. the

one where the heat is evacuated. In other words, below the Percolation threshold concentration, nanoparticles are not connected. When the Percolation threshold concentration is obtained, the network formed with nanoparticles, having higher thermal conductivity than the fluid medium, allows the heat carriers to take a path with much less thermal resistance, thus enhancing the thermal conductivity of the fluid, and therefore the heat transfer coefficient

- wt.% means percentage by weight,

- Graphite nanoplatelets means a multilayered system of graphene sheets having a thickness around between 5 and 20nm;

- Few layers graphene means a multilayered system of graphene sheets having a thickness between 1 and 5 nm and

- Graphene means a single-atom-thick sheet of hexagonally arranged, bonded carbon atoms, presenting usually a thickness below 1nm.

Other characteristics and advantages of the invention will become apparent from the following detailed description of the invention.

The invention relates to a method of heat treatment of non-metallic or metallic item comprising at least one step A) of heat transfer between the item and a heat transfer fluid A' comprising a fluid medium and nanoparticles, the heat transfer fluid having a heat transfer coefficient below the heat transfer coefficient of water.

Preferably, the method further comprises at least one step B) of heat transfer between the item and a heat transfer fluid B' comprising a fluid medium and nanoparticles, the heat transfer fluid B' having a heat transfer coefficient different from the heat transfer coefficient of A' and below the heat transfer coefficient of water.

More preferably, the method also comprises at least one step C) of heat transfer between the item and a heat transfer fluid C' comprising a fluid medium and nanoparticles, the heat transfer fluid C' having a heat transfer coefficient above the heat transfer coefficient of water.

Advantageously, the method also comprises at least one step D) of heat transfer between the item and a heat transfer fluid D' comprising a fluid medium and nanoparticles, the heat transfer fluid D' having a heat transfer coefficient

different from the heat transfer coefficient of C' and above the heat transfer coefficient of water.

According to the invention, the steps A), B), C), D) can be performed in any order, steps B, C and D being optional. In a preferred embodiment, the method of heat treatment according to the invention comprises steps A) and B) performed successively. In another preferred embodiment, step B) is performed before step A). Advantageously, the method of heat treatment according to the invention comprises steps A), B) and C) performed successively. They can also be performed in the following orders: B), C) and A) or C), A) and B). Preferably, the method of heat treatment according to the invention comprises steps A), B), C) and D) performed successively. They can also be performed in the following orders: B), C), A) and D) or A), C), D) and B).

In a preferred embodiment, the heat transfer fluid comprises nanoparticles chosen from graphite nanoplatelets, graphene, few layers graphene, TiO₂, ZnO₂, ZnO, Boron- nitride, copper, silica, montmorillonite, zeolite clinoptilolite, wollastonite, mica, zeolite 4A, Al₂O₃, silicate, pumice, calcium oxide or carbon nanotubes.

Preferably, the nanoparticles are multilayered nanoplatelets. Indeed, without willing to be bound by any theory, it seems that to obtain nanoplatelets morphology, nanoparticles should have a multilayer structure with weak interaction between layers, i.e. Van der Waals, hydrogen bond, mechanical bond, halogen bond, pi stacking, cation/anion-pi bonds, intercalation, salt bridges and polar-pi. This weak bonding together with a good thermal conductivity of the nanoplatelets raises the possibility of improving heat transfer coefficient of a fluid.

Advantageously, the thickness of nanoparticles is between 1 and 99.99 nm, preferably between 5 to 50 nm and more preferably between 5 to 15 nm.

Preferably, the lateral size of the nanoparticle is between 26 and 50µm, advantageously, between 35 and 45µm.

Preferably, the nanoparticle concentration is between 0.01 wt.% and 12 wt.%, advantageously between 2 and 8 wt.% and more preferably between 4 and 7 wt.%.

In a preferred embodiment, the heat transfer fluid has a dispersing agent being a non-surface active polymer, a surfactant or a mixture thereof. The surfactant can be cationic, anionic, amphoteric or non-ionic.

For example, the dispersant agent can be polyvinylpyrrolidone, polysaccharides, sulphated polysaccharides, linear alkylbenzene sulfonates, lignin sulfonates, di-alkyl sulfosuccinates, quaternary ammonium compounds, sodium stearate or a mixture thereof.

Preferably, the nanoparticles concentration / dispersing agent concentration ratio is between 3 and 18. More preferably, the nanoparticles concentration / dispersing agent concentration ratio is between 4 and 15, advantageously between 4 and 8 and preferably being between 4 and 6.

Without willing to be bound by any theory, it seems that when the above ratio is controlled and the Percolation threshold concentration reached, the heat transfer fluid according to the invention allows for a higher thermal conductivity and therefore a higher heat transfer coefficient in laminar regime flow. Indeed, the dispersing agent would be able to avoid deposition and agglomeration of nanoparticles. For instance, if the dispersing agent is a surfactant, the nanoparticle would be enclosed by a micelle consisting in a core of hydrophobic molecules and a shell of hydrophilic molecules. Such micelle structure allows dispersing nanoparticles within the fluid. However to obtain percolation, in other words the formation of the long-range network formed by the nanoparticles, the degree of dispersion of nanoparticles has to be limited.

Preferably, the heat transfer fluid comprises a fluid medium chosen from water, ethylene glycol, ethanol, oil, methanol, silicone, propylene glycol, alkylated aromatics, liquid Ga, liquid In, liquid Sn, potassium formate or a mixture thereof. Gallium, Indium and Tin can be used as heat transfer fluid, in particular for the cooling of a metallic item. Indeed, the melting point of gallium is of 30°C, the one of indium is 157°C and the one of tin is of 232°C. For example, they can be used to cool down computer chips or laboratory equipments such as neutron sources.

The heat transfer fluid is preferably manufactured by the following steps:

- A. the provision of nanoparticles according to the present invention,
- B. the provision of a fluid medium,

C. the adjustment of the nanoparticle concentration in order to achieve percolation and

D. the mixing of the nanoparticles with the fluid medium.

According to the invention, the flow of the heat transfer fluid can be in a laminar or turbulent flow regime. In a laminar flow regime, the heat transfer coefficient is proportional to the thermal conductivity. On the contrary, in turbulent flow regime, the heat transfer coefficient depends on a set of thermo-physical properties such as viscosity.

According to the invention, the heat transfer step is between a metallic or non-metallic item and the heat transfer fluid. Preferably, the metallic item, being for example a metallic substrate, is made of aluminum, steel, stainless steel, copper, iron, copper alloys, titanium, cobalt, metal composite, nickel and the non-metallic is made of plastics.

In the prior art, the heat transferring using water as fluid medium can usually be realized by one mode. The mode is called "contact water" which means that water is used to cool or heat an object by being in direct contact with it.

According to a preferred embodiment of the invention, the item, being metallic, is a metallic substrate and the heat transfer fluid is directly in contact with it. In this case, the heat transfer can be realized by jet impingement cooling, pool boiling, spray cooling or micro-channel cooling.

For example, in the steel making industry, the heat transfer by contact water cooling can be implemented:

- in sprays chambers of continuous casters and hot rolling process such as the cooling process on the run-out table,
- In coke ovens for gas treatment and quenching of coke,
- during the slag quenching in blast furnaces, basic oxygen furnaces and electric arc furnaces.

Preferably, the method of the heat treatment according to the invention further comprises at least one heating step. For example, the heating step is performed at a temperature between 0 to 1200°C.

The method according to the present invention can be performed for example in the construction industry, in particular in the method for manufacturing

the steel beams. For example, structural beams according to EP1961887 are produced by cutting a specific sinusoidal line in a starting H-section, being a steel plate in a form of H, by flame cutting. Then, two obtained T-sections are shifted and welded by arc welding. Usually, after the arc welding, it is known to cool down.

- 5 This cooling step is important since it define the final microstructure of the welded and therefore, the final mechanical properties.

Generally, water is used as heat transfer fluid to cool down the welded beam. However, the cooling with water is too fast so there is a risk that the welding point has a brittle microstructure. It is also possible to cool down the beam with air.

- 10 However, such cooling will take too much time. Thus, in this case, it is necessary to have a heat transfer fluid having a heat transfer coefficient lower than the one of water but higher than the one of air in order to obtain the good cooling rate and consequently, to reach the desired welding point microstructure.

- 15 With the method according to the present invention, it is possible to have such heat transfer fluid. Indeed, the heat treatment, being the cooling of the welded beam, can be performed with a heat transfer fluid A') comprising water as a fluid medium and Graphite nanoplatelets having a lateral size of 40 μm and a thickness of 10 nm. The concentration of the nanoparticles is of 7wt.%. The fluid A' further comprises 1wt.% of polyvinnypyrrolidone as dispersing agent. The
20 nanoparticles concentration / dispersing agent concentration ratio is 7. The heat transfer reduction with respect to the one of water is of -53% in turbulent regime flow at 25°C. Thus, this fluid A') allows for a slower cooling than water and higher than air.

- 25 When two slow cooling steps have to be performed with two heat transfer fluids having different heat transfer coefficient, both being below the one of water, the above fluid A') and another heat transfer fluid B') can be used. For example, the heat transfer fluid B') comprises water as fluid medium and graphite nanoplatelets having a lateral size of 40 μm and a thickness of 10 nm. The concentration of nanoparticles is of 10wt.%. The fluid B' further comprises 1wt.%
30 of polyvinnylpyrrolidone as dispersing agent. The nanoparticles concentration / dispersing agent concentration ratio is 10. The heat transfer reduction with respect to the one of water is of -75% in turbulent regime flow at 25°C.

The method according to the present invention can also be performed for example in any method for manufacturing a metallic substrate, in particular iron alloys. Indeed, after the annealing of such metallic substrate, a cooling is usually performed in order to obtain the desired microstructure. The cooling step can be
5 achieved with the method according to the present invention using a heat transfer fluid having a heat transfer coefficient below the one of water. This results in a controlled cooling rate that allows microstructural design as a function of the desired mechanical properties and final applications.

CLAIMS

1. Method of heat treatment of a non-metallic or metallic item comprising at least
5 one step A) of heat transfer between said item and a heat transfer fluid A'
comprising a fluid medium and nanoparticles, the heat transfer fluid having a
heat transfer coefficient below the heat transfer coefficient of water.
2. Method according to claim 1, comprising in addition at least one step B) of
10 heat transfer between the item and a heat transfer fluid B' comprising a fluid
medium and nanoparticles, the heat transfer fluid B' having a heat transfer
coefficient different from the heat transfer coefficient of A' and below the heat
transfer coefficient of water.
- 15 3. Method according to claim 1 or 2, comprising in addition at least one step C) of
heat transfer between the item and a heat transfer fluid C' comprising a fluid
medium and nanoparticles, the heat transfer fluid C' having a heat transfer
coefficient above the heat transfer coefficient of water.
- 20 4. Method according to claim 3, comprising in addition at least one step D) of
heat transfer between the item and a heat transfer fluid D' comprising a fluid
medium and nanoparticles, the heat transfer fluid D' having a heat transfer
coefficient different from the heat transfer coefficient of C' and above the heat
transfer coefficient of water.
- 25 5. Method according to anyone of claims 1 to 4, the heat transfer fluid comprises
nanoparticles chosen among graphite nanoplatelets, graphene, few layers
graphene, TiO₂, ZnO₂, ZnO, Boron- nitride, copper, silica, montmorillonite,
30 zeolite clinoptilolite, wollastonite, mica, zeolite 4A, Al₂O₃, silicate, pumice,
calcium oxide or carbon nanotubes or any mixture thereof .

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6. Method according to anyone of claims 1 to 5, wherein the nanoparticles are multilayered nanoplatelets.

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7. Method according to anyone of claims 1 to 6, wherein the thickness of the nanoparticles is between 1 and 99.99 nm.

8. Method according to claim 7, wherein the thickness of the nanoparticles is between 5 to 50 nm.

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9. Method according to claim 8, wherein the thickness of the nanoparticles is between 5 to 15 nm.

10. Method according to anyone of claims 1 to 9, wherein the lateral size of the nanoparticles is between 26 and 50 μ m.

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11. Method according to claim 10, wherein the lateral size is between 35 and 45 μ m.

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12. Method according to anyone of claims 1 to 11, wherein the nanoparticles concentration is between 0.01 – 12 wt.%.

13. Method according to claim 12, wherein the nanoparticles concentration is between 2 and 8 wt.%.

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14. Method according to claim 13, wherein the nanoparticles concentration between 4 and 7 wt.%.

15. Method according to anyone of claims 1 to 14, wherein the heat transfer fluid further comprises a dispersing agent.

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16. Method according to claim 15, wherein the dispersing agent is a non-surface active polymer or a surfactant or a mixture thereof.

17. Method according to claim 16, wherein the surfactant is a cationic, anionic, amphoteric or non-ionic one.
- 5 18. Method according to claim 17, wherein the dispersing agent is chosen from polyvinylpyrrolidone, polysaccharides, sulphated polysaccharides, linear alkylbenzene sulfonates, lignin sulfonates, di-alkyl sulfosuccinates, quaternary ammonium compounds and sodium stearate or a mixture thereof.
- 10 19. Method according to anyone of claims 16 to 18, wherein the nanoparticles concentration / dispersing agent concentration ratio is between 3 and 18.
20. Method according to anyone of claims 1 to 19, wherein the fluid medium is chosen among water, ethylene glycol, ethanol, oil, methanol, silicone,
15 propylene glycol, alkylated aromatics, liquid Ga, liquid In, liquid Sn, potassium formate and any mixture thereof.
21. Method according to anyone of claims 1 to 20, wherein the heat transfer fluid is in laminar or turbulent regime flow.
- 20 22. Method according to anyone of claims 1 to 21, wherein the item, being metallic, is made of aluminum, steel, stainless steel, copper, iron, copper alloys, titanium, cobalt, metal composite or nickel.
- 25 23. Method according to anyone of claim 1 to 22, wherein the metallic item is a metallic substrate and the heat transfer is such that the heat transfer fluid is directly in contact with the metallic substrate.
- 30 24. Method according to claim 23, wherein the contact between the metallic substrate and the heat transfer fluid is realized though jet impingement cooling, pool boiling, spray cooling or micro-channel cooling.

25. Method according to anyone of claims 1 to 24, wherein the heat treatment of non-metallic or metallic item further comprises at least one heating step.

5 26. Method according to claim 25, wherein said one heating step is performed at a temperature between 0 and 1200°C.

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2015/002394

A. CLASSIFICATION OF SUBJECT MATTER

INV. C09K5/10 B22D11/124 C21D1/00
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)

C09K B22D C21D

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2009/065107 A1 (HO GEORGE [TW] ET AL) 12 March 2009 (2009-03-12) paragraphs [0002] - [0008], [0029] - [0032]; figures 1-4; examples -----	1-26
X	US 2012/186789 A1 (SEDAROUS SALAH S [US] ET AL) 26 July 2012 (2012-07-26) paragraphs [0002], [0006], [0007], [0012], [0070]; figure 1; tables 1,2 -----	1,5,12, 15,17
X	US 2013/341028 A1 (CHRISTIAN CHAD F [US] ET AL) 26 December 2013 (2013-12-26) paragraphs [0002], [0015], [0026], [0031], [0034], [0036], [0055], [0056]; examples; table 2 ----- -/-	1-26

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

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Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2015/002394

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GOPALAN RAMESH ET AL: "Review of thermo-physical properties, wetting and heat transfer characteristics of nanofluids and their applicability in industrial quench heat treatment", NANOSCALE RESEARCH LETTERS, vol. 6, no. 1, 1 January 2011 (2011-01-01), page 334, XP055297238, ISSN: 1556-276X, DOI: 10.2355/isijinternational.50.124 Introduction; Discussion; Table 1 -----	1-26
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/IB2015/002394

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