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WETTING OF HEAT TRANSFER SURFACES WITH
LIQUEFIED METAL HEAT TRANSFER MEDIA
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Fig. 1.

Lead on SAE 1010 Steel Samples
Liquid Metal Applied at 800°F.

Fig. 2.

Lead on Tantalum Samples
Liquid Metal Applied at 1200°F.

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This invention relates, in general, to liquid-metal, heat-transfer systems and, more particularly, to a method for obtaining wetting of metallic heat transfer surfaces by liquefied-metal, heat-transfer media.

In modern power plants, liquid-metal, heat-transfer systems are becoming of increasing importance due to certain advantages which may be obtained by their use. This is particularly true in the field of atomic energy power generation where isolation of the reactor heat generator is simplified thereby and more efficient and compact design is facilitated.

Such systems present difficulties not often encountered in conventional systems. Among these difficulties is the problem of obtaining intimate contact of the heat transfer medium and the heat transfer surfaces. This is the result of the metallic medium failing to wet the heat transfer surface.

As a consequence of this failure of the heat transfer medium to come into close contact with the heated surface, film boiling of the medium results and the efficiency of the transfer of heat therebetweeen drops to a very serious degree. Moreover, dangerous overheating of the heated surface can then occur.

Now, it has been found that wetting of the heat transfer surface is promoted if such surface is subjected to a preliminary treatment and is then heated to at least a certain minimum temperature in vacuums of specific magnitudes. The heating may be conducted with the liquid metal in contact with the surface whereupon wetting is effected under the aforesaid conditions. However, in a modified method, wetting also occurs if the surface is heated to the required temperature under the said vacuum condition and is then cooled to a temperature somewhat above the melting point of the liquid metal before the surface is contacted therewith.

Accordingly, it is an object of the invention to provide improved operation of a liquid-metal, heat-transfer system.

Another object of the invention is to provide a method for promoting intimate contact between a metallic heat transfer surface and a liquid-metal, heat-transfer medium.

Still another object of the invention is to provide a method for promoting the wetting of a heat transfer surface by a liquid metal wherein the surface is subjected to heat treatment under high vacuum conditions.

Other objects and advantages will become apparent from consideration of the following description of the invention taken together with the accompanying drawings of which:

Figure 1 is a graphical illustration of the wetting of a steel surface by molten lead following a preheat vacuum treatment; and

Figure 2 is a graphical illustration of the wetting of a tantalum surface by molten lead following a preheat vacuum treatment.

The process of the invention is applicable to the conditioning of metallic heat transfer surfaces in any type of system in which certain liquid metal, i.e., molten metal, transfer media are employed. Such surfaces may be those of tubes, headers, coils, or the like, which are employed, for example, in the construction of boilers, heat exchangers, conduits and other components of power generating installations. These surfaces may likewise be those which are employed to transfer heat in atomic powered heat engines of either fixed or mobile types.

In particular, the process of the invention is applicable to those heat systems in which molten lead and bismuth or alloys thereof are employed as the liquid metal. The materials employed to construct the components of the heat exchange system may be various steels, including low carbon and stainless varieties (SAE 347 and 446); iron; molybdenum; tungsten; tantalum; columbium; cobalt; titanium; nickel; and zirconium.

In carrying out the process of the invention the heat transfer surface is given a preliminary conditioning treatment to remove gross surface impurities. Such treatment may include an abrasive cleaning as by grinding, sanding, buffing, or sand blasting and/or a degreasing operation using a chlorinated solvent as indicated by the nature of the impurities present on the surface. This treatment is intended to provide a fresh metal surface somewhat optimum results when employed with the principal treatment of the present process. Appropriate measures must then be taken to prevent the surface again becoming contaminated. Preferably, the subsequent steps of the process are initiated promptly.

Subsequent to the preliminary treatment, the surface to be treated is arranged in such a manner that it may be simultaneously subjected to both controlled high temperatures and high vacuums either in contact with the liquid metal or in such a way that the surface may be later contacted therewith under maintained vacuum; however, at a lower temperature. These operations may be conducted in either appropriate vacuum apparatus or closed systems may be treated, in situ. In the latter case, it is necessary only to provide means for evacuating the system, in situ, while supplying external heat as by actuating the heat source normally employed with the vacuum.

The usual means employed to introduce the liquid metal may be employed to contact such material with the treated surface.

It will be appreciated that the phenomena of wetting cannot be rigidly defined since there is a wide intermediate range between the unwet and fully wet states. However, quite reliable, reproducible and consistent indications of wetting can be obtained by either of the methods described hereinafter.

Wetting of a surface may be conveniently determined by measuring the angle of contact between a liquid globule and the solid surface. Observations indicate that the droplet shape does not change on cooling once the droplet shape has become established at a given temperature. Therefore the measurement is most easily made on cooled samples. Relevant to the present disclosure, the convention was adopted that "wetting" of a metal surface has occurred if the liquid-solid contact angle is less than 90°.

In conducting such a "wetting" test, there may be employed a vacuum furnace provided with accurate temperature control means and means for supporting the surface to be tested in a horizontal position. If the preheat method is to be employed means are required for supporting a solid piece of the heat transfer metal in a cooler portion of the furnace during the preliminary heating and for disposing such metal upon the heat transfer surface when the temperature is lowered as indicated in the foregoing. Polishing of the heat transfer metal surface to remove stains and foreign particles and
scraping of the heat-transfer metal (Pb or Bi) to remove oxides, etc., assures reproducibility and accuracy of the test.

The technique employed in performing the test consists in placing the cleaned and polished surface to be tested in a horizontal position in the vacuum furnace. Also, the scraped portion of heat transfer metal (Pb, Bi or alloy) is placed on the surface in accordance with the first method or is suspended in a cooler position of the furnace if the second method is being employed. Then the system is evacuated and heated to the required minimum temperature for a short period of time and is then cooled. In the second method, the system is cooled to a lower temperature but above the melting point of the liquid metal. The heat transfer material is then placed on the surface, which is allowed to melt and the droplet is allowed to stabilize at the given temperature. Subsequently, the system is cooled and the contact angle of the droplet is measured.

Results obtained by the foregoing method of the wetting of SAE-1010 steel surfaces by lead applied at 800° F. after the surface had been preheated to various temperatures are illustrated in Fig. 1 of the drawing. The contact angles, indicated therein, fall in a range between the curves of maximum (+) and minimum values (−), with consistent wetting, as indicated by a less than 90° contact angle, occurring at about 1600° F. Results of a similar experiment wherein lead was applied at 1200° F. to a surface which had been previously heated to a higher temperature are illustrated in Fig. 2 of the drawing. As may be seen therein, the surface must be heated in a high vacuum to above about 1600° F. to consistently effect wetting by molten lead applied subsequently at 1200° F.

Results obtained by this method and the necessary conditions are also summarized in the following table.

<table>
<thead>
<tr>
<th>Table 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metal Surface</td>
</tr>
<tr>
<td>Low carbon steel (ribbon)</td>
</tr>
<tr>
<td>Low carbon steel (SAE 1000)</td>
</tr>
<tr>
<td>Tantalum</td>
</tr>
</tbody>
</table>

The foregoing method is not suitable for determining the degree of wetting of surfaces totally immersed in the liquid metals and, accordingly, there was developed a "wetting detector" method for use under these conditions. This method operates on the principle that when two parallel plates of the material to be tested are brought together in a liquid metal there will be a particular separation distance at which capillary pressure will cause the liquid metal to break away and withdraw from between the plates. This maximum distance "d" between the plates is related to the liquid-metal, surface tension, "γ"; the contact angle, "θ", between the liquid metal and the surface of the plates; and the liquid static pressure, "p", at the gap, by the following approximate relation:

\[ d = \frac{2γ}{p \cos θ} \]

It will be noted that, for a positive liquid pressure, only values of the contact angle exceeding 90 degrees give a physically real value of "d." When the contact angle is less than 90 degrees the liquid metal does not break away and the test indication is that the metal surface is wet.

The foregoing test may be conveniently performed in a vacuum oven by supporting a flat plate of the test metal near the lower portion of a cavity formed therein and employing as the movable surface, the flat end of a micrometrically adjustable probe which is operated from outside the oven through an appropriate vacuum seal. Liquid metal, in a clean state, is introduced into the cavity to a level sufficient to provide any convenient value of static pressure "p." Heat is then applied under high vacuum conditions. It will be understood that this method will generally be employed to determine the degree of wetting for liquid metals in contact with the heat transfer metal surface. However, the results obtained by this method correlate closely with those obtained by the droplet method.

Employing vacuums of the order of 0.03 micron, results obtained when the surface was heated in contact with the liquid metal may be summarized as follows: Stainless steels (S. A. E. 347 and 446) were subjected to temperatures in the range of 1000° F. to 1800° F. and were found to be wet by lead and bismuth at 1600° F. Unless heated to this temperature, wetting was erratic and not uniformly good.

Molybdenum and tungsten were subjected to temperatures in the range of 1500° to 1800° F. and it was found that a temperature of 1800° F. was required to effect adequate wetting by lead and bismuth. Below this temperature the surface was either not wet or was only partially wet.

Titanium, zirconium, and cobalt were heated to temperatures in the range of 1500° to 1800° F. resulting in wetting by both lead and bismuth; however, these surfaces are attacked in this temperature range by the liquid metals. Therefore, it is usually advisable to employ a lower working temperature once the wetting has been effected.

Nickel was wet by bismuth at temperatures as low as 1600° F.; moreover, the vacuum requirements were found to be less rigorous with this material. With pressures of about 1.0 micron, adequate wetting was still obtained. Results with molten lead and a nickel surface should closely parallel the behavior with bismuth.

In the foregoing, results have been described with reference to lead and bismuth as individual liquid metals; however, similar results will also be obtained with alloy mixtures of these two metals. In practicing the invention it is important to maintain the vacuum during the time following the initial heating and until the liquid metal is contacted with the surface as exposure to even very low air pressures vitiates the conditioning treatment.

While in the foregoing there has been described what may be considered to be preferred embodiments of the invention, modifications may be made therein without departing from the basic concept of the invention, and it is intended to cover all such as fall within the scope of the appended claims.
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5 irons, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium by a liquid-metal, heat transfer medium selected from the group consisting of lead, bismuth and alloy mixtures of lead and bismuth, the steps comprising preheating said surface to a temperature above about 1000°F, in a vacuum of below about 1.0 micron pressure, and continuously maintaining said vacuum while contacting said surface with the liquid-metal, heat-transfer medium, whereby said surface is wet by said liquid medium.

3. In a process for promoting the wetting of a metallic heat transfer surface formed of a material selected from the group consisting of low carbon and stainless steels, iron, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium by a heat transfer medium selected from the group consisting of Bi, Pb, and alloys of Bi and Pb, the steps comprising subjecting said surface to a temperature above about 1000°F, and contacting said surface with said heat transfer medium at a temperature above the melting point thereof, whereby said surface is wet by said heat transfer medium.

4. The process as defined in claim 3 wherein said vacuum is maintained at a pressure value below about 0.03 micron Hg.

5. In a method of promoting the wetting of a metallic heat transfer surface formed of a material selected from the group consisting of low carbon and stainless steels, iron, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium with a liquid lead heat transfer medium, the steps comprising applying and maintaining a vacuum of below about 0.03 micron Hg on said surface, heating said surface to at least 1000°F under said vacuum, and contacting said surface with said medium in said vacuum and while at a temperature above the melting point thereof, whereby said surface is wet with liquid lead.

6. In a method of promoting the wetting of a metallic heat transfer surface formed of a material selected from the group consisting of low carbon and stainless steels, iron, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium with a liquid lead heat transfer medium, the steps comprising subjecting said surface to a temperature above the melting point thereof, whereby said surface is wet with liquid bismuth.

7. In a method of promoting the wetting of a metallic heat transfer surface formed of a material selected from the group consisting of low carbon and stainless steels, iron, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium with a liquid lead heat transfer medium, the steps comprising subjecting said surface to a temperature above 800°F, and contacting said surface with said medium while at a temperature above about 800°F, whereby said surface is wet with liquid bismuth.

8. In a process for wetting a stainless steel heat transfer surface with a liquid metal heat transfer medium selected from the group consisting of lead, bismuth, and alloys of lead and bismuth, the steps comprising heating said surface to a temperature of at least about 1600°F and under a vacuum of the order of 0.03 micron, continuously maintaining said surface under said vacuum, and finally contacting the surface with the said liquid heat transfer medium while maintaining said vacuum.

9. In a process for wetting a stainless steel heat transfer surface with a liquid metal heat transfer medium selected from the group consisting of lead, bismuth, and alloys of lead and bismuth, the steps comprising heating said surface to a temperature of at least about 1600°F and under a vacuum of the order of 0.03 micron, continuously maintaining said surface under said vacuum, and finally contacting the surface with the said liquid heat transfer medium while maintaining said vacuum.

10. In a process for wetting a heat transfer surface selected from the group consisting of molybdenum and tungsten with a liquid metal heat transfer medium selected from the group consisting of lead, bismuth, and alloys of lead and bismuth, the steps comprising heating said surface to a temperature in the range of 1500°F to 1800°F and under a vacuum of the order of 0.03 micron, continuously maintaining said surface under said vacuum, and finally contacting the surface with the said liquid heat metal transfer medium while maintaining said vacuum.

11. In a process for wetting a heat transfer surface selected from the group consisting of tantalum, columbium, titanium, zirconium, and cobalt with a liquid metal heat transfer medium selected from the group consisting of lead, bismuth, and alloys of lead and bismuth, the steps comprising heating said surface to a temperature in the range of 1500°F to 1800°F and under a vacuum of less than about 1 micron, continuously maintaining said surface under said vacuum, and finally contacting the surface with the said liquid heat metal transfer medium while maintaining said vacuum.

12. In a process for wetting a nickel heat transfer surface with a liquid metal heat transfer medium selected from the group consisting of lead, bismuth, and alloys of lead and bismuth, the steps comprising heating said surface to a temperature of at least about 1000°F and under a vacuum of less than about 1 micron, continuously maintaining said surface under said vacuum, and finally contacting the surface with the said liquid heat metal transfer medium while maintaining said vacuum.

13. In a method for promoting the wetting of a metallic heat transfer surface formed of a material selected from the group consisting of low carbon and stainless steels, iron, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium with a liquid-metal heat transfer medium selected from the group consisting of lead, bismuth and alloy mixtures of lead and bismuth, the steps comprising heating said surface to a high temperature above about 1000°F, subjecting the heated surface to a vacuum where the pressure is below about 1.0 micron, continuously maintaining said surface under said vacuum, and contacting said surface with said heat transfer material while under said vacuum and at a temperature above the melting and below the boiling point thereof.

14. In a method for promoting the wetting of a metallic heat transfer surface formed of a material selected from the group consisting of low carbon and stainless steels, iron, molybdenum, tungsten, tantalum, columbium, cobalt, titanium, nickel and zirconium with a liquid lead-bismuth heat transfer medium, the steps comprising applying and maintaining a vacuum of below about 0.03 micron Hg on said surface, heating said surface to at least 1000°F under said vacuum, and contacting said surface with said medium in said vacuum and while at a temperature above the melting point thereof, whereby said surface is wet with liquid lead.

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