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Method and device for measuring a melt cooling curve

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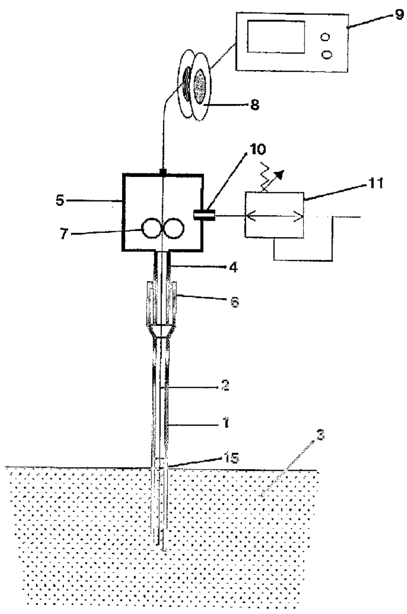
[Fortsetzung auf der nächsten Seite]

(54) Title: METHOD AND DEVICE FOR MEASURING A MELT COOLING CURVE

(54) Bezeichnung: VERFAHREN UND VORRICHTUNG ZUM MESSEN ABKÜHLKURVE VON SCHMELZEN



(57) Abstract: The invention relates to a method for measuring a melt cooling curve and/or a heating curve for a melt sample with the aid of an optical fibre, wherein the immersion end of an optical fibre provided at least partially with a free surface at a certain distance is encompassed with a theoretically heat-resistant reception space of the sample. Said immersion end of the optical fibre is immersed into the melt, thereby forming a sample in the sample reception space which is afterwards removed together with the sample and optical fibre from a molten metal and afterwards, the sample cooling curve of the sample and/or the temperature profile during heating are measured after the sample prehardening with the aid of a signal which is received by said optical fibre and transmitted to a measuring device. A corresponding measuring device and the use thereof are also disclosed.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren zum Messen der Abkühlkurve von Schmelzen und/oder der Aufheizkurve von Schmelzenproben mit einer optischen Faser, wobei ein mindestens teilweise eine freie Oberfläche aufweisendes Eintauchende der optischen Faser von einem temperaturbeständigen Probenaufnahmeraum beabstandet umgeben wird, dass die optische Faser mit ihrem Eintauchende in die Schmelze eingetaucht und dabei in dem Probenaufnahmeraum eine Probe gebildet wird, wobei danach der Probenaufnahmeraum mit der Probe und der optischen Faser aus der Metallschmelze herausgezogen und die Abkühlkurve der Probe und/oder, nach vorhergehender Erstarrung der Probe, der Temperaturverlauf beim Aufheizen anhand eines von der optischen Faser aufgenommenen und an ein Messgerät weitergeleiteten Signals gemessen wird. Des weiteren betrifft die Erfindung eine entsprechende Vorrichtung sowie deren Verwendung.

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Veröffentlicht:
— *mit internationalem Recherchenbericht*
— *vor Ablauf der für Änderungen der Ansprüche geltenden Frist; Veröffentlichung wird wiederholt, falls Änderungen eintreffen*

Zur Erklärung der Zweibuchstaben-Codes und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

Method and device for measuring the cooling curve of molten masses

The invention relates to a method for measuring the cooling curve of molten masses (melts) and/or the heating curve of melt samples with an optical fiber, wherein an immersion end of the optical fiber having an at least partially free surface is surrounded with a spacing by a temperature-resistant sample-receiving chamber, such that the optical fiber is immersed with its immersion end in the melt and in this way a sample is formed in the sample-receiving chamber, wherein the sample-receiving chamber with the sample and the optical fiber is thereafter pulled out of the molten metal, and the cooling curve of the sample and/or, after previous solidification of the sample, the temperature profile during heating is measured with reference to a signal obtained by the optical fiber and forwarded to a measurement device. In addition, the invention relates to a corresponding device as well as to its use. Here, melts are understood to include both melts of pure metals, such as iron, copper, or steel, or alloys, as well as cryolite melts, molten salts, or molten glass.

Temperature measurement methods and devices, in which liquid temperatures are measured with the aid of optical fibers at high temperature, are known from EP 646 778 B1 among others. Additional devices are known from US 4,355,907. There, an immersion sensor is described, with which a sample of a molten metal is taken. The sample thereby adheres in a hollow space. A graphite plate is arranged between the hollow space and the optical fiber receiving the measurement values.

A sample vessel, into which molten metal is poured and in which the temperature of the molten metal is then measured by means of an optical fiber, is known from DE 36 31 645 A1. Other devices for measuring the temperature in molten metals are known from JP 62-185129 and JP 62-185130. In addition, methods for measuring the melting-point temperature in a smelting crucible with the aid of optical radiation are known from US 6,106,150, US 6,004,031, or from EP 802 401 A1.

According to a first aspect of the present invention, there is provided a method for measuring the cooling curve of melts and/or the heating curve of melt samples with an optical fiber, comprising the steps of providing an immersion end of the optical fiber with an at least partially free surface, surrounding the immersion end with a spacing by a temperature-resistant sample-receiving chamber, immersing the optical fiber with its immersion end in a melt, thereby forming a sample in the sample-receiving chamber, thereafter pulling the sample-receiving chamber with the sample and the optical fiber out of the melt, and measuring a cooling curve of the sample and/or measuring a temperature profile during heating after previous solidification of the sample, the measuring steps being performed with reference to a signal obtained by the optical fiber and forwarded to a measurement device, wherein both an immersion end face and a portion of a side wall of the immersion end of the optical fiber are brought into direct contact with the melt.

According to a second aspect of the present invention, there is provided a device for measuring the cooling curve of melts and/or the heating curve of melt samples, comprising an optical fiber configured to obtain a black-body radiation signal, a first end of the optical fiber being connected to a measurement device for detecting and processing a black-body radiation signal obtained by the optical fiber, a carrier for the optical fiber, a second end of the optical fiber being for immersion in a melt, the second end of the optical fiber being surrounded with a spacing by a temperature-resistant sample-receiving chamber comprising a structure and material suitable for withdrawing a sample from the melt to allow cooling or heating and obtain a black body radiation signal with the optical fiber, wherein both an end face and a portion of a side wall of the immersion end of the optical fiber have a free surface.

According to a third aspect of the present invention, there is provided cooling curve or heating curve measured by the method of the first aspect of the invention described above.

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According to a fourth aspect of the present invention, there is provided a device according to the second aspect of the invention described above when used for measuring the cooling curve of melts and/or the heating curve of melt samples.

The problem of the present invention is to improve the known methods or devices.

The problem is solved according to the invention by the features of the independent claims. Because both the end face and also a portion of the side wall of the immersion end of the optical fiber have a free surface or are brought into direct contact with the melt, the measurement accuracy and the response time can thereby be improved. Advantageous embodiments follow from the dependent claims. In particular, it is advantageous that the length of the portion of the side wall of the optical fiber in direct contact with the melt is at least 10 times, preferably at least 30 times, as large as the diameter of the free surface of the end face of the optical fiber, which is brought into direct contact with the melt for the measurement.

Preferably, after the immersion of the immersion end of the optical fiber in the melt, a reduced pressure is generated in the sample-receiving chamber, and melt is drawn into the sample-receiving chamber, which thereby significantly improves the sampling as such. It is also possible to bring the sample into the sample-receiving chamber by ferrostatic pressure. In addition, it is useful that, after the measurement of the cooling curve, the optical fiber be immersed again in the melt and that an increased pressure be generated in the sample-receiving chamber and liquid melt be forced out of the sample-receiving chamber. Naturally, the material can also be forced out after the measurement of the heating curve. It can also be useful that, after the measurement of the cooling and/or heating curve, the immersion end of the fiber and the end of the sample-receiving chamber filled with melt be cut off, in order to remove possibly damaged or expended material.

In addition to the measurement of the cooling curve of the melt or the heating curve of previously solidified melt samples, which can provide information on material properties, the bath temperature of the melt can also be measured. Advantageously, the immersion end of the optical fiber can be set in vibration at least intermittently, in order to prevent undercooling of the sample. The method can be used preferably for measuring the liquidus temperature and/or a phase transition temperature of the melt. Advantageously, the end face of the optical fiber has a free surface, in order to improve the signal reception. In particular, the optical fiber can be formed from sapphire or from quartz glass, particularly in order to be stable at higher temperature ranges.

Preferably, the sample-receiving chamber is formed as a tube, especially from quartz glass or from metal or ceramic. A slag cap can be arranged at the immersion end of the sample-receiving chamber, in order to prevent material lying on the melt to be analyzed from entering the sample-receiving chamber. The slag cap is typically made from a material, which melts or dissolves during the passage through the layer lying on or in the melt.

The sample-receiving chamber is preferably connected pneumatically to a device for generating an increased pressure or a reduced pressure, in order to be able to set and optionally to precisely regulate the necessary pressure. In addition, it is useful that the optical fiber be connected to a vibrator. The vibrator can be arranged, for example, on the carrier for the fiber. By the transfer of a vibration to the fiber and to the sample-receiving chamber, the vibrator has the effect that undercooling of the melt to be analyzed is prevented. For this reason, the coupling of the vibrator to the sample-receiving chamber is likewise usefully guaranteed.

The device according to the invention can be used both for measuring the bath temperature of the melt and also for measuring the liquidus temperature and/or a phase transition temperature of the melt.

An embodiment example of the invention is described in more detail below with reference to a drawing.

Shown in the drawing are:

Figure 1 a measurement device with carrier tube and

Figure 2 another embodiment of the measurement device.

The embodiment shown in Figure 1 has a replaceable carrier tube 1, through which the optical fiber 2 is guided. The carrier tube 1 can be replaced after use in the molten metal 3. For this purpose, it is removed from the connecting tube 4 of the housing 5, and a new carrier tube 1 is placed on the connecting tube 4 with a sealed connection 6. In the housing 5, a system of transport rollers 7 is arranged, with whose aid the optical fiber 2 is unwound from a spool 8 and fed to the molten metal 3. The immersion end of the fiber 3 has a free surface both at the end face and also at the portion of the side wall connected to the end face. The remaining portion of the fiber can have a coating, for example made from plastic, which can be removed, for example by combustion. The other end of the optical fiber is connected to a measurement device 9, which is used for signal reception and evaluation.

The housing 5 further contains a gas connection port 10, to which the increased pressure/reduced pressure unit 11 is attached.

The embodiment shown in Figure 2 has a cable box 12 as the core piece. In this cable box 12, the optical fiber 2 is wound on a roller 13. The optical fiber 2 is surrounded by a cladding tube 14, which is unwound together with the fiber 2 and fed to the molten metal 3 by transport rollers 7. The end of the optical fiber 2 facing away from the molten metal 3 is connected to the measurement device 9. Just like the housing 5 in the embodiment according to Figure 1, the cable box 12 is hermetically sealed and has a gas connection port 10. The increased pressure/reduced pressure unit 11 is connected to this gas connection port 10.

The optical fiber 2 has a free surface at its end facing the molten metal 3, both at the end face and also on the side wall, wherein the length of the free surface of the optical fiber 2, measured from the end face in the longitudinal direction, is more than 30 times the diameter of the end face of the optical fiber 2 intended for immersion in the molten metal 3. For measurement, the optical fiber 2 is immersed with its immersion end in the melt 3. Here, a reduced pressure is generated in the carrier tube 1 or the cladding tube 14, and a portion 15 of the melt is drawn into the tube. This bottom portion of the carrier tube 1 or the cladding tube 14 forms the sample-receiving chamber. The device with the sample-receiving chamber and the sample located therein (portion 15 of molten metal 3 drawn into the sample-receiving chamber) is pulled from the molten metal 3. Outside of the molten metal 3, the temperature is significantly lower than in the molten metal 3, so that the sample is cooled and the cooling curve is recorded with reference to the radiation signal obtained by the optical fiber 2 and forwarded to the measurement device 9. Here, one takes advantage of the known effect of a black-body radiation.

Instead of or in addition to the cooling curve, the sample can be heated/melted after solidification/cooling, for example by immersion of the sample-receiving chamber of the sampling device in the melt. In this manner, the heating curve is likewise recorded and evaluated as a temperature-time diagram.

The cooling curve/heating curve gives information about the liquidus temperature and/or the solidus temperature, because at this temperature in a temperature-time diagram, a temperature plateau is registered over a short time. Likewise, phase transitions within the cooling molten metal can be identified by temperature plateaus in the temperature-time diagram. As long as the immersion end of the optical fiber 2 is itself located in the molten metal 3, its actual bath temperature can be measured.

After measuring the cooling curve, the optical fiber 2 can be immersed again in the molten metal 3. The sample thereby melts. After melting, the heating curve can be determined. Thereafter, an increased pressure is generated via the gas connection port 10 in the measurement device, especially within the carrier tube 1 or the cladding tube 14, so that the liquid molten sample is forced out of the sample-receiving chamber. The device can then be used for new sampling. If necessary, in the embodiment according to Figure 1, the carrier tube 1 must be replaced and the optical fiber 2 is then guided into the new carrier tube 1. In the embodiment according to Figure 2, the immersion end of the cladding tube 14 is cut off with the optical fiber 2 as well as melt residue possibly contained in the cladding tube 14, as soon as this immersion end has become unusable. The optical fiber 2 is then unwound together with the cladding tube 14 from the spool 13.

In addition, the optical fiber is connected to a vibrator, not shown in the drawing. The vibrator can be arranged, for example, on the carrier 1 for the fiber 2, and by the transfer of a vibration to the fiber 2 and to the sample-receiving chamber, the vibrator has the effect that undercooling of the melt to be analyzed is prevented. For this reason, the rigid coupling of the vibrator on the sample-receiving chamber is useful and also sensible.

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The claims defining the invention are as follows:

1. A method for measuring the cooling curve of melts and/or the heating curve of melt samples with an optical fiber, comprising the steps of providing an immersion end of the optical fiber with an at least partially free surface, surrounding the immersion end with a spacing by a temperature-resistant sample-receiving chamber, immersing the optical fiber with its immersion end in a melt, thereby forming a sample in the sample-receiving chamber, thereafter pulling the sample-receiving chamber with the sample and the optical fiber out of the melt, and measuring a cooling curve of the sample and/or measuring a temperature profile during heating after previous solidification of the sample, the measuring steps being performed with reference to a signal obtained by the optical fiber and forwarded to a measurement device, wherein both an immersion end face and a portion of a side wall of the immersion end of the optical fiber are brought into direct contact with the melt.

2. The method according to claim 1, wherein a length of the portion of the side wall of the optical fiber located in direct contact with the melt is at least ten times as large as a diameter of the immersion end face of the optical fiber located in direct contact with the melt.

3. The method according to claim 2, wherein the length of the portion of the side wall of the optical fiber located in direct contact with the melt is at least thirty times as large as the diameter of the immersion end face of the optical fiber located in direct contact with the melt.

4. The method according to any one of claims 1 to 3, further comprising, after immersion of the immersion end of the optical fiber in the melt, the step of generating a reduced pressure in the sample-receiving chamber such that melt is drawn into the sample-receiving chamber.

5. The method according to claim 4, further comprising, after measuring the cooling curve, the steps of immersing the optical fiber again in the melt, and generating an increased pressure in the sample-receiving chamber such that melt is forced out of the sample-receiving chamber.

6. The method according to claim 4 or 5, further comprising, after measuring the cooling curve and/or the heating curve, the step of cutting off the immersion end of the fiber and an end of the sample-receiving chamber filled with melt.

7. The method according to any one of claims 1 to 6, further comprising measuring a bath temperature of the melt with the optical fiber.

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8. The method according to any one of claims 1 to 7, further comprising the step of setting the immersion end of the optical fiber into vibration at least intermittently.

9. The method according to any one of claims 1 to 8, wherein a liquidus temperature and/or a solidus temperature and/or a phase transition temperature of the melt are determined.

10. The method according to any one of claims 1 to 9, wherein the optical fiber comprises quartz, glass or sapphire.

11. The method according to any one of claims 1 to 10, wherein the sample-receiving chamber has a form of a tube.

12. The method according to any one of claims 1 to 11, wherein the sample-receiving chamber comprises quartz glass.

13. The method according to any one of claims 1 to 12, wherein the sample-receiving chamber comprises metal or ceramic.

14. The method according to any one of claims 1 to 13, wherein a slag cap is arranged on an immersion end of the sample-receiving chamber.

15. The method according to any one of claims 1 to 14, wherein the sample-receiving chamber is connected pneumatically to a device for generating increased pressure or reduced pressure.

16. The method according to any one of claims 1 to 15, wherein the optical fiber is connected to a vibrator.

17. A method as claimed in claim 1, substantially as hereinbefore described with reference to any one of the accompanying drawings.

18. A device for measuring the cooling curve of melts and/or the heating curve of melt samples, comprising an optical fiber configured to obtain a black-body radiation signal, a first end of the optical fiber being connected to a measurement device for detecting and processing a black-body radiation signal obtained by the optical fiber, a carrier for the optical fiber, a second end of the optical fiber being for immersion in a melt, the second end of the optical fiber being surrounded with a spacing by a temperature-resistant sample-receiving chamber comprising a structure and material suitable for withdrawing a sample from the melt to allow cooling or heating and obtain a black body radiation signal with the optical fiber, wherein both an end face and a portion of a side wall of the immersion end of the optical fiber have a free surface.

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19. The device according to claim 18, wherein a length of the free surface of the side wall of the optical fiber is at least ten times as large as a diameter of the free surface of the end face of the optical fiber.

20. The device according to claim 19, wherein the length of the free surface of the side wall of the optical fiber is at least thirty times as large as the diameter of the free surface of the end face of the optical fiber.

21. The device according to any one of claims 18 to 20, wherein the optical fiber comprises quartz, glass, or sapphire.

22. The device according to any one of claims 18 to 21, wherein the sample-receiving chamber has a form of a tube.

23. The device according to any one of claims 18 to 22, wherein the sample-receiving chamber comprises quartz glass.

24. The device according to any one of claims 18 to 23, wherein the sample-receiving chamber comprises metal or ceramic.

25. The device according to any one of claims 18 to 24, wherein a slag cap is arranged on an immersion end of the sample-receiving chamber.

26. The device according to any one of claims 18 to 25, wherein the sample-receiving chamber is connected pneumatically to a device for generating increased pressure or reduced pressure.

27. The device according to any one of claims 18 to 26, wherein the optical fiber is connected to a vibrator.

28. The device according to any one of claims 18 to 27, which is adapted for determining a liquidus temperature and/or a solidus temperature and/or a phase transition temperature of the melt.

29. The device according to any one of claims 18 to 28, wherein the carrier for the optical fiber comprises a carrier tube.

30. The device according to any one of claims 18 to 28, wherein the carrier for the optical fiber comprises a cladding tube.

31. The device according to claim 30, wherein the cladding tube and optical fiber are unwound together from a spool.

32. A device as claimed in claim 18, substantially as hereinbefore described with reference to any one of the accompanying drawings.

33. Cooling curve or heating curve measured by the method of any one of claims 1 to 17.

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34. A device according to anyone of claims 18 to 32 when used for measuring the cooling curve of melts and/or the heating curve of melt samples.

Dated 24 May, 2007

Heraeus Electro-Nite International N.V.

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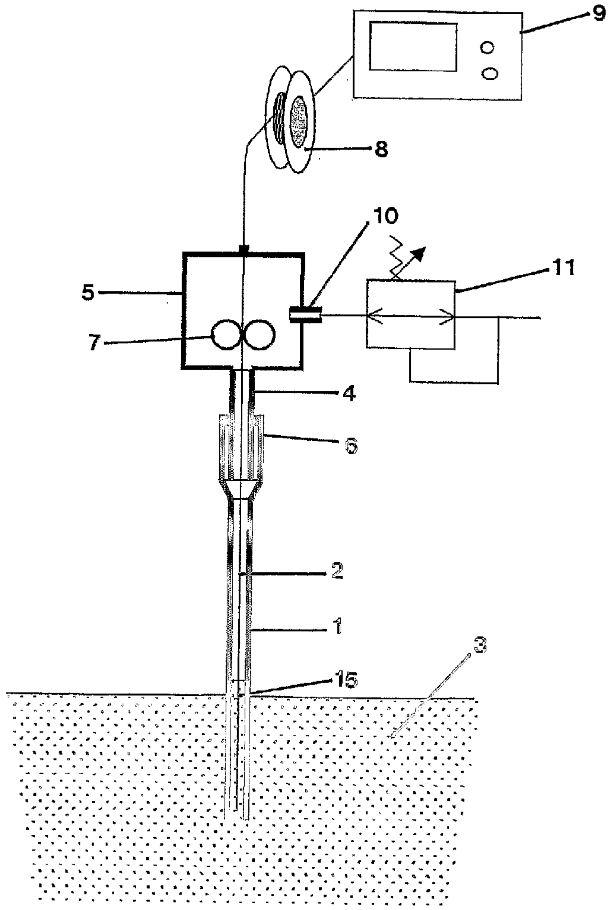

Fig. 1

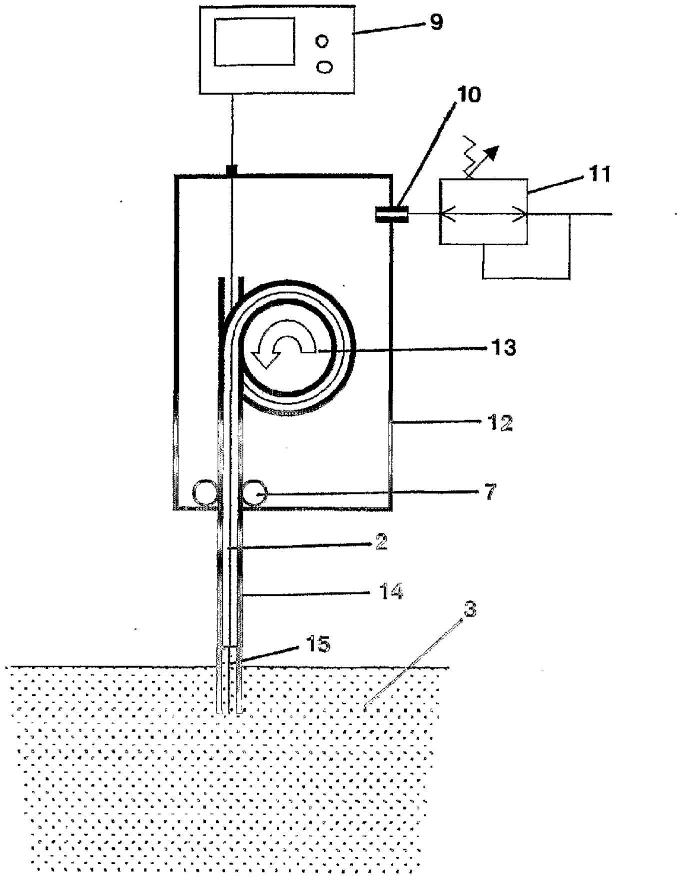

Fig. 2