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<p>(21) International Application Number: PCT/SE99/02393</p> <p>(22) International Filing Date: 16 December 1999 (16.12.99)</p> <p>(30) Priority Data: 9804419-1 18 December 1998 (18.12.98) SE 9902407-7 24 June 1999 (24.06.99) SE</p> <p>(71) Applicant (for all designated States except US): SINTERCAST AB [SE/SE]; P.O. Box 102 03, S-100 55 Stockholm (SE).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): POPELAR, Patrik [CA/SE]; Prinsgatan 13 B, S-641 30 Katrineholm (SE). ANDERSSON, Conny [SE/SE]; Albro Gård, S-635 13 Eskilstuna (SE).</p> <p>(74) Agents: BERG, S., A. et al.; Albihns Patentbyrå Stockholm AB, P.O. Box 5581, S-114 85 Stockholm (SE).</p>		<p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: METHOD FOR PREDICTING THE MICROSTRUCTURE OF SOLIDIFYING CAST IRON</p> <p>(57) Abstract</p> <p>By studying heat transfer in a sample vessel containing a sample of molten cast iron, it is possible to carry out accurate predictions of the microstructure in which the molten cast iron sample will solidify. This method is also highly suitable for automatisation by using a computer.</p>			

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METHOD FOR PREDICTING THE MICROSTRUCTURE OF SOLIDIFYING CAST IRON

The present invention relates to an improved method for predicting the micro-structure with which a certain cast iron melt will solidify. The invention also relates to an apparatus for carrying out the method.

10

Background of the invention

WO86/01755 (incorporated by reference) discloses a method for producing compacted graphite cast iron by using thermal analysis. A sample is taken from a bath of molten cast iron and this sample is permitted to solidify during 0.5 to 10 minutes. The temperature is recorded simultaneously by two temperature responsive means, one of which is arranged in the centre of the sample and the other in the immediate vicinity of the vessel wall. So-called cooling curves representing temperature of the iron sample as a function of time are recorded for each of the two temperature responsive means. According to this document it is then possible to determine the necessary amount of structure-modifying agents that must be added to the melt in order to obtain the desired microstructure.

WO92/06809 (incorporated by reference) describes a specific method for evaluating the cooling curves obtained by the method of WO86/01755. According to this document, thermal analysis is carried out in a sample vessel coated with a material consuming the active form of the structure-modifying agent. This material may comprise oxides of Si, Mn, Fe, K and Na. An early plateau in the cooling curve recorded by a certain temperature-responsive means located near the vessel wall indicates that flake graphite has been formed due to interaction with the coating. It is then possible to determine whether any structure-modifying agent has to be added to the melt in order to obtain compacted graphite cast iron by using calibration data.

5 When casting compacted graphite cast iron in a commercial large-scale foundry, it is of utmost importance that accurate and reliable predictions of the micro-structure of the castings can be carried out. Sometimes cooling curves that are difficult to interpret are accepted as CGI despite the fact that some flaky graphite has been formed. In order to improve the accuracy of the evaluation, there is thus
10 a need for alternative methods for evaluating cooling curves which methods can compensate for deviations from the normal appearance of the cooling curves.

Summary of the invention

15 Now, it has turned out that by studying heat transfer in a sample vessel containing a sample of molten cast iron, it is possible to carry out accurate predictions of the microstructure in which the molten cast iron sample will solidify. This method is also highly suitable for automatization by using a computer.

20 Definitions

The term "cooling curve" as disclosed herein refers to graphs representing temperature as a function of time, which graphs have been recorded in the manner disclosed in WO86/01755 and WO92/06809.

25 The term "heat generation curve" as utilised herein relates to a graph showing the heat that is generated in a certain zone of a molten cast iron as a function of time. For the purposes of the present invention, the heat generation curves herein are determined for a zone located in the centre of a molten cast iron sample (the A zone), and in the periphery of a molten cast iron sample (the B zone), respectively. Below, methods for determining heat generation curves will be further described.

35 The term "sample vessel" as disclosed herein, refers to a small sample container which, when used for thermal analysis, is filled with a sample of molten metal.

5 The temperature of the molten metal is then recorded during solidification in a suitable way. Preferably the sample vessel is designed in the manner disclosed in WO86/01755, WO92/06809, WO91/13176 (incorporated by reference), WO96/23206 (incorporated by reference) or PCT/SE98/02122.

10 The term "sampling device" as disclosed herein, refers to a device comprising a sample vessel equipped with at least two temperature responsive means for thermal analysis, said means being intended to be immersed in the solidifying metal sample during analysis, and a means for filling the sample vessel with molten metal. The sample vessel is preferably equipped with said sensor in the manner disclosed in fig. 2 in WO96/23206 or PCT/SE98/02122.

15

20 The term "structure-modifying agent" as disclosed herein relates to compounds affecting the morphology of graphite present in the molten cast iron. Suitable compounds can be chosen from the group of magnesium and rare earth metals such as cerium, or mixtures of these compounds. The relationship between the concentration of structure-modifying agents in molten cast irons have already been discussed in the above cited documents WO92/06809 and WO86/01755.

25 The term "CGI" as disclosed herein refers to compacted graphite cast iron.

The term "SGI" as disclosed herein refers to spheroidal graphite cast iron.

Figures

30 The invention will now be described with reference to the accompanying figures in which:

35 Fig. 1 schematically outlines a diagram of a sample vessel that can be used in connection with the present invention. In such a sample vessel the heat is uniformly transported in all directions. A sample of molten cast iron comprised in

5 such a vessel can therefore be regarded as a freezing sphere. In this figure, the freezing sphere is divided into two zones A and B. The radii r_1 and r_2 relate to the average radii of zones A and B, respectively;

10 Fig. 2 discloses cooling curves and corresponding heat generation curves for CGI. In figures 2 - 7, the following abbreviations are used: T_A = centrally recorded cooling curve, T_B = peripherically recorded cooling curve, P_A = heat generation in the centre, and P_B = heat generation in the periphery;

15 Fig. 3 discloses cooling curves and corresponding heat generation curves for a low nodularity CGI. During the measurement flake graphite is formed in the sample vessel due to reaction with the wall coating;

Fig. 4 discloses cooling curves and corresponding heat generation curves for cast iron comprising a high carbon equivalent content;

20 Fig. 5 discloses cooling curves and corresponding heat generation curves for a near eutectic iron;

25 Fig. 6 discloses cooling curves and corresponding heat generation curves for a eutectic or hypereutectic iron; and

Fig. 7 discloses cooling curves and corresponding heat generation curves for grey flake iron; and

30 Fig. 8 is a schematic presentation of an apparatus for controlling production of compacted graphite cast iron according to the present invention.

Detailed description of the invention

5 As already disclosed above, the present invention relates to predicting the micro-
structure in which a certain molten cast iron sample will solidify by measuring
the heat evolution in the sample. In particular, the invention relates to determin-
ing the heat transfer between two sample zones (one zone in the centre of the
sample and one peripheral zone encompassing the central zone) and the sur-
10 roundings. By studying graphs presenting the derivative of heat as a function of
time, it is possible to carry out accurate predictions.

The thermal balance of any uniform element can be described by the relation:

15 $Q_{\text{stored}} = Q_{\text{generated}} + Q_{\text{in}} - Q_{\text{out}}$ (1)

where Q_{stored} is the amount of heat stored by the heat capacity of the material,
 $Q_{\text{generated}}$ is the amount of heat generated by the volume of material, Q_{in} is the heat
transferred into the material from its surroundings and Q_{out} is the heat transferred
20 from the sample volume to its surroundings.

When carrying out the present invention, it is advantageous to use a sample ves-
sel as disclosed in SE 9704411-9. In such a sample vessel, the heat transport in a
sample contained in the vessel is approximately the same in all directions. From
25 now on, the heat transport between the centre (Figure 1, zone A) and the more
peripheral part (Figure 1, zone B) of the molten cast iron contained in a sample
vessel is described. As the A zone is situated in the centre of the freezing sphere,
no heat will be transported into the zone and Q_{in} is therefore equal to zero. Ap-
propriate substitutions in relation (1) above give the following equation:

30

$$C_p m_A dT_A/dt = Q_{\text{genA}} + 0 - 4\pi k_e [(T_A - T_B)/(1/r_1 - 1/r_2)] \quad (2)$$

where C_p is the heat capacity per unit mass, m_A is the mass of the A zone, dT_A/dt
is the temperature change of the A zone per unit time, k_e is the effective heat

5 transfer coefficient of the material, and $(1/r_1 - 1/r_2)^{-1}$ is the mean distance for heat transport. The radii r_1 and r_2 are both defined in fig. 1. T_A and T_B are the temperatures in the A zone and B zone, respectively.

10 From equation (2) we can isolate the heat generation term and calculate the average bulk heat generation in the A zone:

$$Q_{\text{genA}} = C_p m_A dT_A/dt + 4\pi k e [(T_A - T_B)/(1/r_1 - 1/r_2)] \quad (3)$$

15 In equation (3) all variables are constants except dT_A/dt and $(T_A - T_B)$. Accordingly, equation (3) can be simplified to:

$$Q_{\text{genA}} = k_1 dT_A/dt + k_2 (T_A - T_B) \quad (4)$$

20 where k_1 and k_2 are constants. Hence a heat generation curve corresponding to the A zone can be calculated from a set of cooling curves recorded in the centre and periphery of a sample of molten cast iron.

Heat balance for the B zone resembles the balance of zone A, but heat is transferred both into the zone (from the A zone) and out from it (to the surroundings).
25 Hence term Q_{in} in relation (1) is not equal to zero. Appropriate substitutions in relation (1) give the following equation:

$$C_p m_B dT_B/dt = Q_{\text{genB}} + 4\pi k e [(T_A - T_B)/(1/r_1 - 1/r_2)] - [h(T_B - T_S) + \epsilon A_B \sigma (T_B^4 - T_S^4)] \quad (5)$$

30 where, in addition to those variables defined in connection with equations (2) and (3) above, h is the convective heat loss to the surroundings, T_S is the temperature of the surroundings, and $\epsilon A_B \sigma$ is the appropriate constant in Stefan-Boltzmann law of radiation.

5 Regarding the last term, we can assume that the ratio between total radiative heat loss and convective heat loss is constant:

$$C = \epsilon A_B / h \quad (6)$$

10 Accordingly, we can isolate the heat generation term of zone B, Q_{genB} :

$$Q_{genB} = C_p m_B dT_B/dt - 4\pi k e [(T_A - T_B)/(1/r_1 - 1/r_2)] + h[(T_B - T_S) + C\sigma(T_B^4 - T_S^4)] \quad (7)$$

15 In equation (7) all variables are constant except dT_B/dt , $(T_A - T_B)$ and T_B . Accordingly, equation (7) can be simplified to:

$$Q_{genB} = k_3 dT_B/dt - k_4 (T_A - T_B) + k_5 T_B + k_6 T_B^4 - k_7 \quad (8)$$

20 By applying equations (4) and (8) on cooling curves recorded in the manner disclosed in WO86/01755 and WO92/06809 we can determine the corresponding heat generation curves. Figs. 2A-6A disclose different kinds of cooling curves and Figs. 2B-6B corresponding heat generation curves.

25 As already mentioned, these calculations are based on a situation where heat is uniformly transported in all directions. The skilled person can of course determine other equations corresponding to other heat transport conditions.

30 There are larger differences between different kinds of heat generation curves compared to corresponding cooling curves. For CGI (Fig. 2), the curve representing heat release in the B zone comprises two distinct peaks, one smaller and one larger, whereas the heat release for low nodularity CGI (Fig. 3) is so much larger that these two peaks hardly can be separated. This also applies to curves related to high carbon equivalent cast iron (Fig. 4). A primary austenite peak is visible the A zone curve. In heat generation curves representing near eutectic iron

5 (Fig. 5) a small austenite peak is still visible, whereas the peak has disappeared in the curves relating to eutectic or hypereutectic iron (Fig. 6). As regards grey flaky iron (Fig. 7), the first peak of the heat generation curve representing the B zone is dominating.

10 It is preferred to carry the prediction method by using a computer-controlled system, especially when a large number of measurements must be carried out. Such a system is outlined in fig. 8. A sample of a molten cast iron is taken and transferred to a sample vessel 22. During the measurement of a particular sample the two temperature-responsive means 10,12 send signals to a computer means 14 in order to generate cooling curves and corresponding heat generation curves, e.g. by applying equations (3) and (6). The computer has access to calibration data, e.g. previously recorded model curves corresponding to a known amount of structure-modifying agent or a known microstructure, in a ROM unit 16 and calculates the amount of structure-modifying agent that has to be added to the melt.

15 In a preferred embodiment this is implemented as an expert system. The amount of structure-modifying agent to be added is signalled to a means 18 for administering structure-modifying agent to the melt 20 to be corrected, whereby the melt is supplied with an appropriate amount of such agents.

20

5 Claims:

1. A process for producing a CGI casting or optionally a SGI casting, requiring a sampling device, at least two means for recording cooling curves, and a means for administering structure-modifying agent(s) to a molten cast iron from which said casting is to be produced, said method comprising the steps of:
 - 10 a) providing a sample of the molten cast iron from which said casting is to be produced, and adding said sample to a sample vessel;
 - 15 b) simultaneously recording at least two cooling curves, at distances r_1 and r_2 , etc., from the centre of the sample vessel;
 - 20 c) determining at least two heat generation curves by using the information obtained in b);
 - 25 d) comparing the heat generation curves obtained in c) with previously determined heat generation curves representing known concentrations of structure-modifying agents which previously determined heat generation curves have been obtained under same conditions as in step a), b) and c);
 - 30 e) determining the amount of structure-modifying agent to be added to the melt based upon the comparison in step d);
 - 35 f) causing said means for administrating structure-modifying agent to administrate a correct amount to the melt; and
 - 40 g) carrying out the casting operation in a per se known manner.

5 2. A process according to claim 1, **characterized** in that said sample vessel is of
a type wherein heat is substantially uniformly transported in all directions
when it is filled with molten cast iron.

10 3. A process according to claim 1 or claim 2, **characterized** in that compacted
graphite cast iron is produced.

15 4. A process for determining the amount of structure-modifying agent that has to
be added to a certain cast iron melt in order to obtain CGI or optionally SGI, re-
quiring a sampling device, and at least two means for recording cooling curves,
said method comprising the steps of:

20 a) providing a sample of the molten cast iron from which said casting is to be
produced, and adding said sample to a sample vessel;

25 b) simultaneously recording at least two cooling curves, at distances r_1 and r_2 ,
etc., from the centre of the sample vessel;

30 c) determining at least two heat generation curves by using the information ob-
tained in b);

35 d) comparing the heat generation curves obtained in c) with previously deter-
mined heat generation curves representing known concentrations of structure-
modifying agents which previously determined heat generation curves have been
obtained under same conditions as in step a), b) and c);

40 e) determining the amount of structure-modifying agent to be added to the melt
based upon the comparison in step d);

5 5. An apparatus for establishing, in real time, an amount of a structure-modifying agent to be added to produce a CGI casting or optionally a SGI casting, the apparatus comprising:

10 a first temperature sensor (10), arranged at a distance r_1 from the centre of a sample vessel but inside said sample vessel, for recording a first cooling curve;

a second temperature sensor (12), at a distance r_2 (r_2 being larger than r_1) from the centre of said sample vessel but inside said sample vessel, for recording a second cooling curve;

15 a computer device (14) for determining an amount value (Va) of a structure-modifying agent to be added to the melt;

a memory means (16) which is provided with pre-recorded calibration data;

20 the computer being set up to determine heat generation curves for one central zone and one peripheral zone in said sample vessel by applying

i) the thermal balance formula:

$$Q_{\text{stored}} = Q_{\text{generated}} + Q_{\text{in}} + Q_{\text{out}}$$

25 where Q_{stored} is the amount of heat stored by the heat capacity of the material, $Q_{\text{generated}}$ is the amount of heat generated by the volume of material, Q_{in} is the heat transferred into the material from its surroundings and Q_{out} is the heat transferred to the surroundings; and

ii) the first and second cooling curves recorded by said first and second temperature sensors (10, 12);

30 the computer being set up to determine an amount value (Va) of a structure-modifying agent to be added to the melt by comparing said heat generation curves with pre-recorded calibration data.

5 6. A computer program product for use in an apparatus for establishing, in real time, an amount of a structure-modifying agent to be added to a cast iron melt (20) during the process of producing a compacted graphite iron casting;

wherein the apparatus has

10 a first temperature sensor (10), arranged at a distance r_1 from the centre of a sample vessel but inside said sample vessel, for recording a first cooling curve;

 a second temperature sensor (12), at a distance r_2 (r_2 being larger than r_1) from the centre of said sample vessel but inside said sample vessel, for recording a second cooling curve;

15 a computer device (14) for determining an amount value (V_a) of a structure-modifying agent to be added to the melt;

 a memory means (16) which is provided with pre-recorded calibration data;

 the computer program product comprising:

20 a recording medium and a computer-readable code means for directing the computer device to determine heat generation curves for one central zone and one peripheral zone in said sample vessel by applying

i) the thermal balance formula:

$$25 \quad Q_{\text{stored}} = Q_{\text{generated}} + Q_{\text{in}} + Q_{\text{out}}$$

where Q_{stored} is the amount of heat stored by the heat capacity of the material, $Q_{\text{generated}}$ is the amount of heat generated by the volume of material, Q_{in} is the heat transferred into the material from its surroundings and Q_{out} is the heat transferred to the surroundings; and

30 ii) the first and second cooling curves recorded by said first and second temperature sensors (10, 12); and

 a recording medium and computer-readable code means for determining an amount value (V_a) of a structure-modifying agent to be

5 added to the melt by comparing said heat generation curves with pre-
recorded calibration data.

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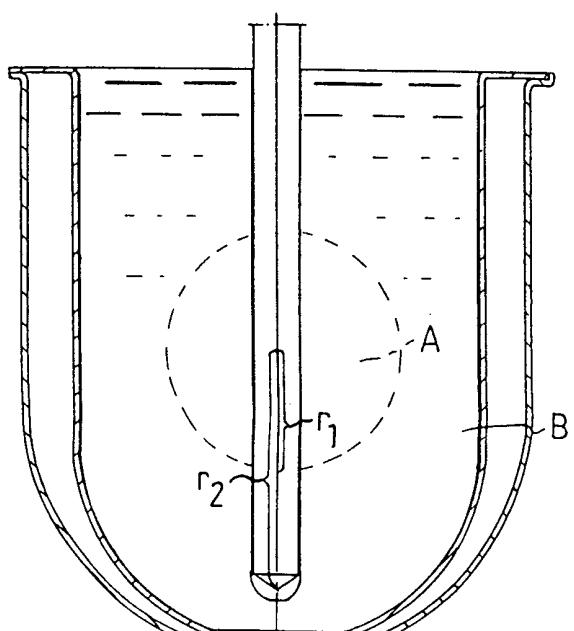


FIG.1

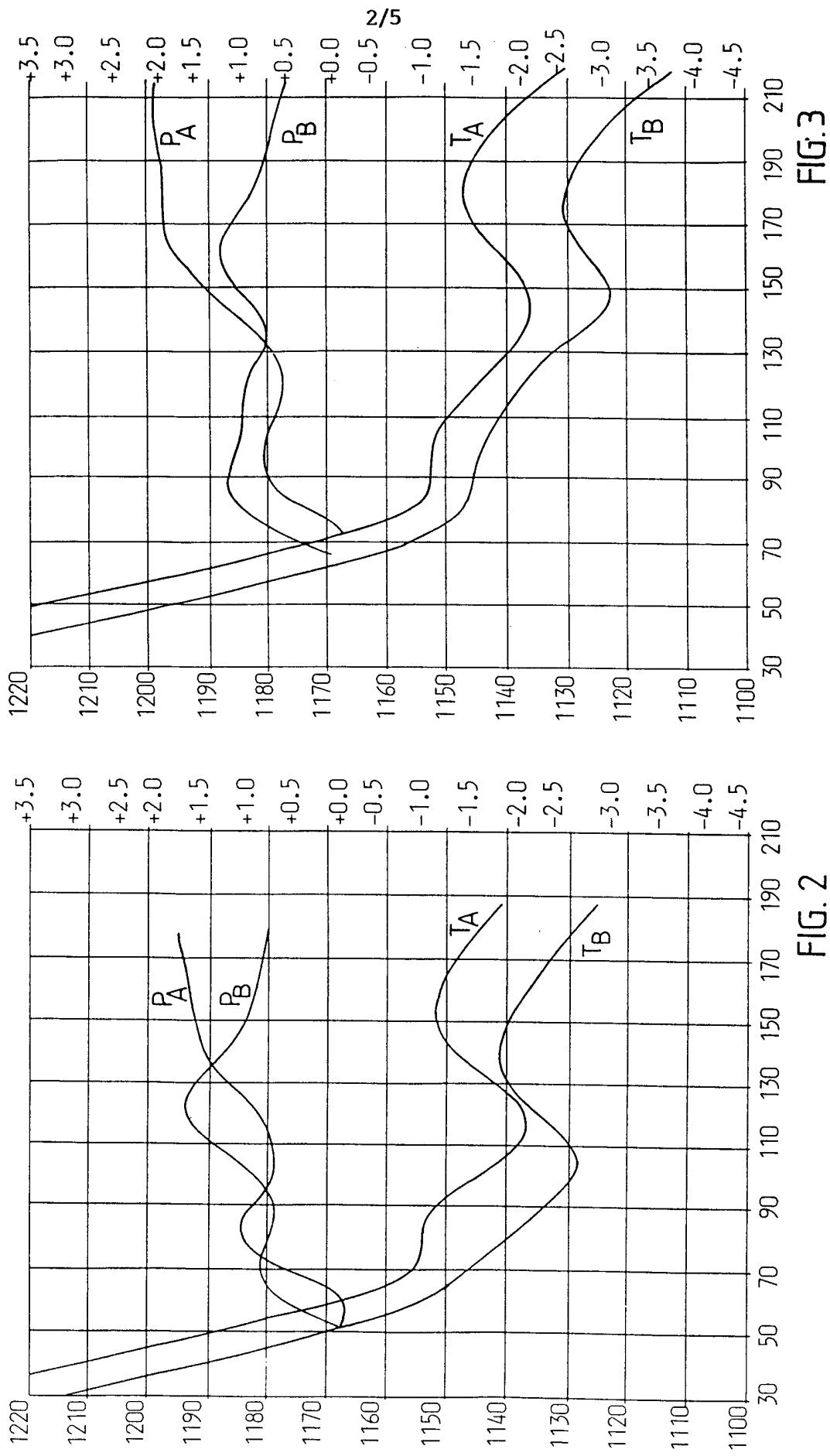


FIG. 2

FIG. 3

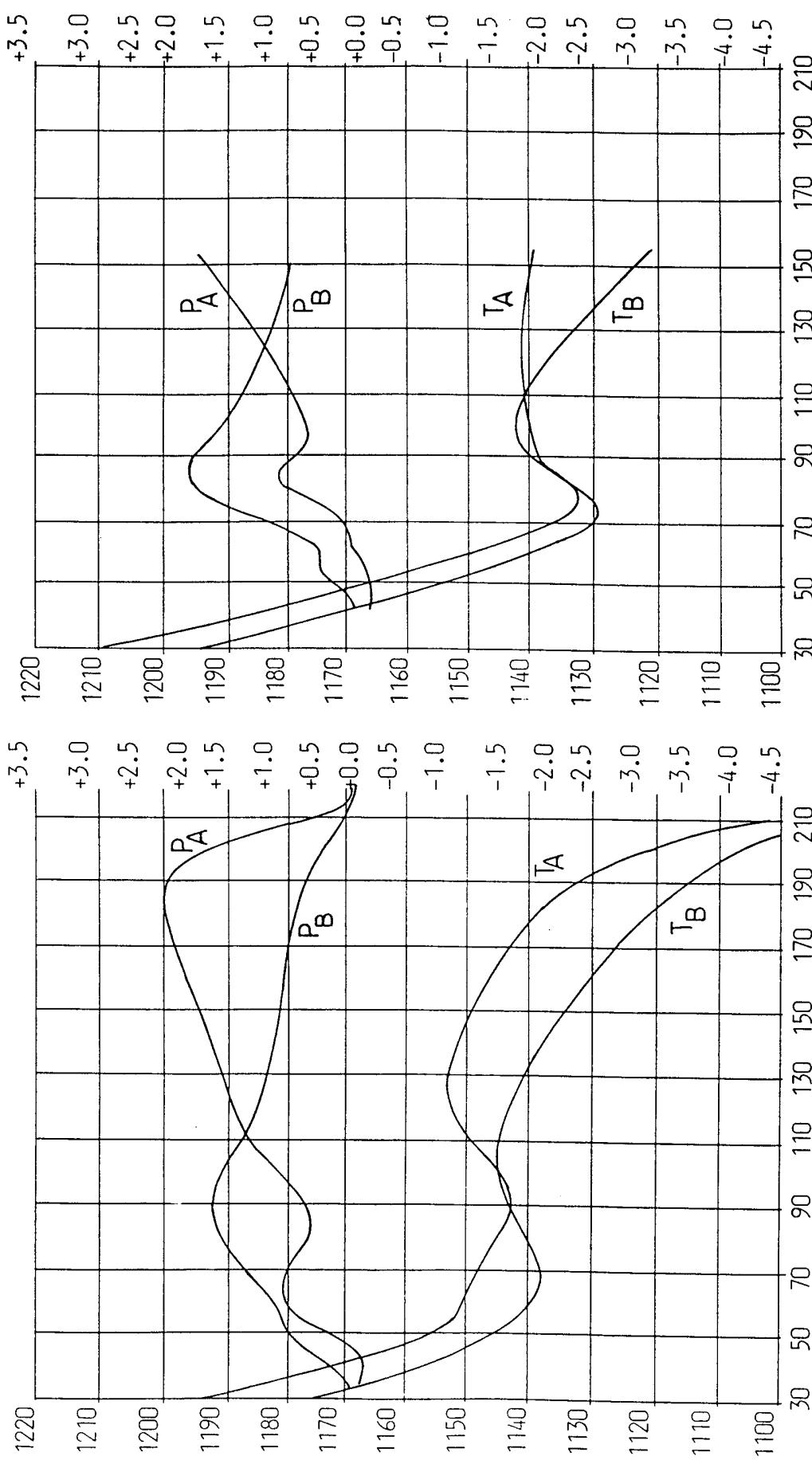


FIG. 4

FIG. 5

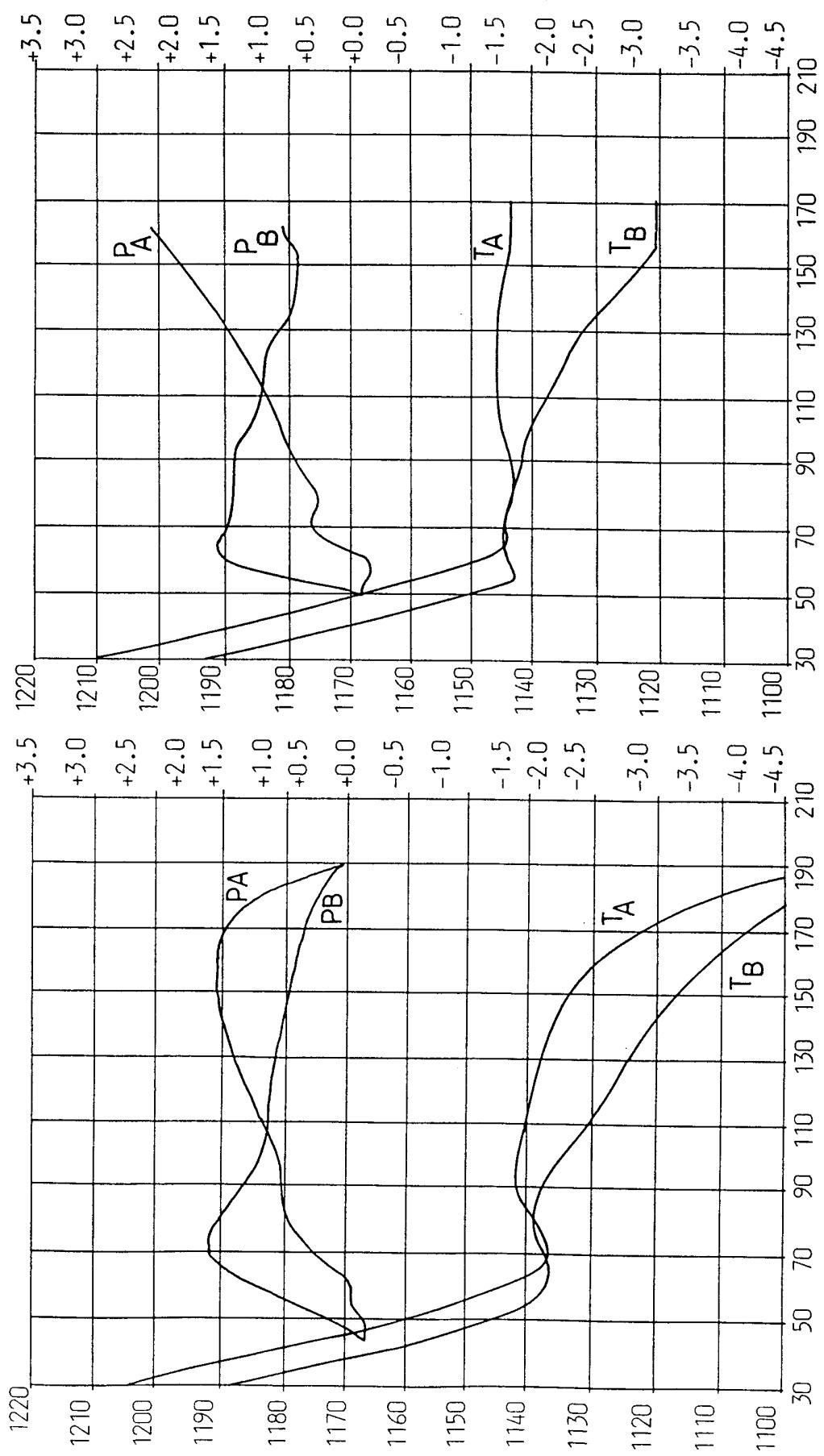
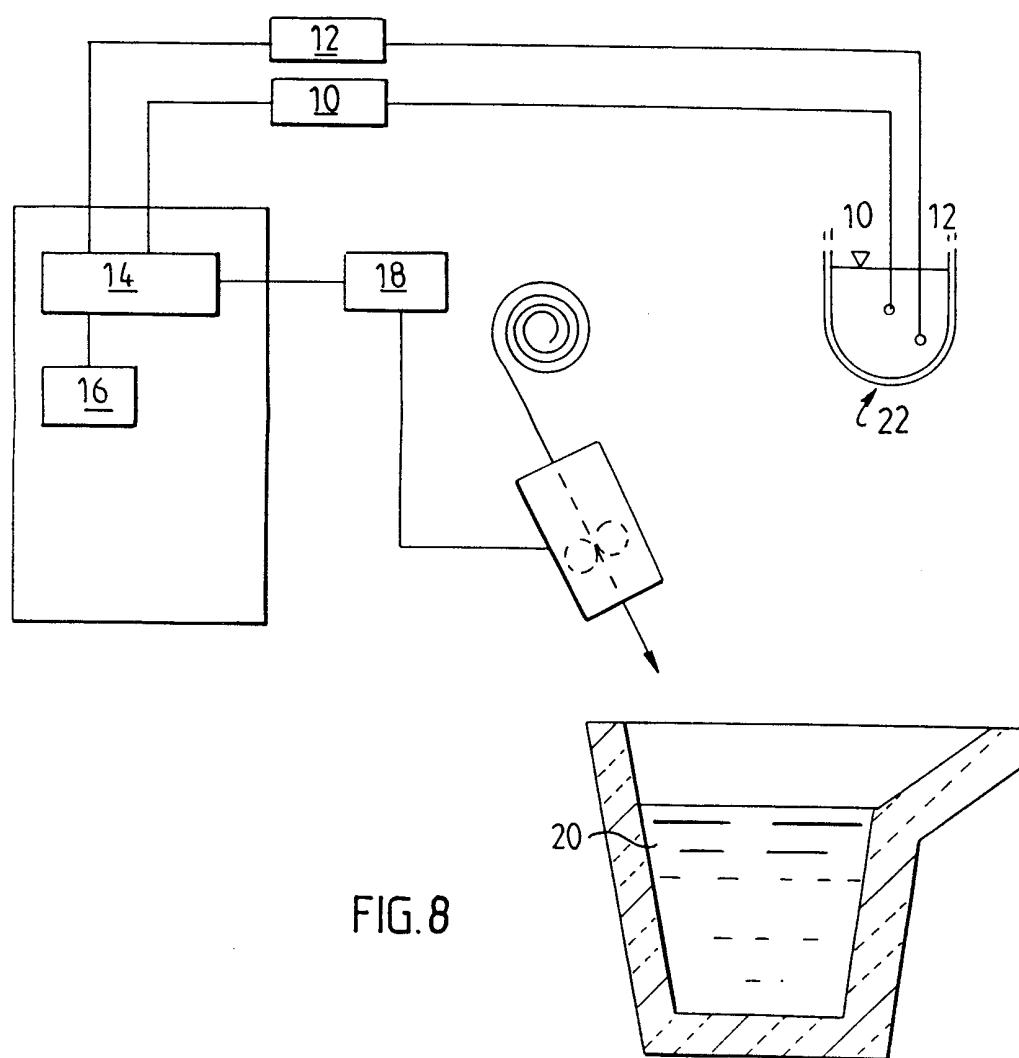


FIG. 6

FLAKE IRON FIG. 7

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INTERNATIONAL SEARCH REPORT

International application No.

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A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C22C 33/08

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	Gjuteriet, Volym, Nr 3, 1996, Jon Nilsson, "Utvecklingsprojekt på segjärnsområdet för optimerad gjutgodskvalitet" page 18 - page 19 --	1-3,4,5,6
Y	DE 2849598 A1 (DR. K. ABLEIDINGER & CO.), 7 June 1979 (07.06.79), page 3, line 12 - page 8, line 32, figures 1-4 --	1-3,4,5,6

Further documents are listed in the continuation of Box C. See patent family annex.

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INTERNATIONAL SEARCH REPORT

International application No.

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

02/12/99

International application No.
PCT/SE 99/02393

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