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Judge et al.

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[45] **Oct. 28, 1975**

[54] **SAMPLING DEVICE FOR MOLTEN METAL**

[75] Inventors: **James R. Judge; Van L. Vierbicky,** both of Weirton, W. Va.

[73] Assignee: **National Steel Corporation,** Pittsburgh, Pa.

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[52] **U.S. Cl.** **73/425.6;** 73/DIG. 9

[51] **Int. Cl.²** **G01N 1/14**

[58] **Field of Search** ..... 73/425.4 R, 425.6, DIG. 9

[56] **References Cited**

UNITED STATES PATENTS

3,369,406	2/1968	Lowdermilk et al.	73/425.6
3,452,602	7/1969	Hackett	73/425.4 R
3,554,040	1/1971	Collins	73/425.4 R
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FOREIGN PATENTS OR APPLICATIONS

1,313,201	1/1966	France	73/DIG. 9
159,060	11/1963	U.S.S.R.	73/DIG. 9

Primary Examiner—Richard C. Queisser
Assistant Examiner—Joseph W. Roskos
Attorney, Agent, or Firm—Shanley, O'Neil and Baker

[57] **ABSTRACT**

A device for sampling heats of molten metal adapted to be connected to a source of vacuum to suck a sample of the molten metal into a refractory sample forming cylinder and attached disc mold chamber through the open end of the cylinder, the open end of the cylinder being closed and sealed prior to the sampling step by a metallic cap which will melt in the molten metal heat, the metal cap being hermetically sealed to the end of the cylinder by a hardened plastic applied as a liquid to the cap and contiguous portion of the cylinder, the plastic coating having a thickness and composition such that any surface flux material adhering to the sampling device as it is thrust into the heat will be removed completely from proximity to the lower end of the cylinder by the act of decomposition of the plastic.

16 Claims, 3 Drawing Figures

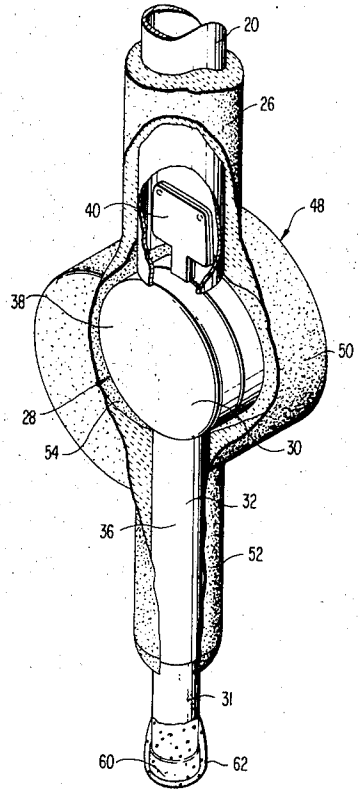


FIG. 1

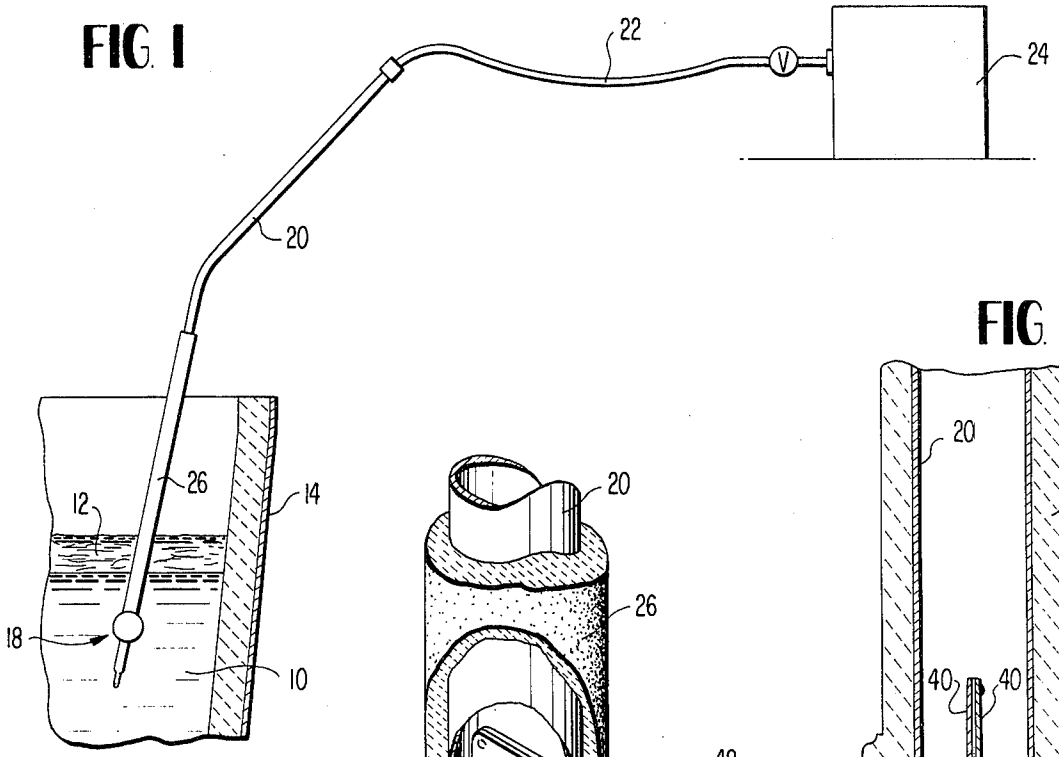


FIG. 2

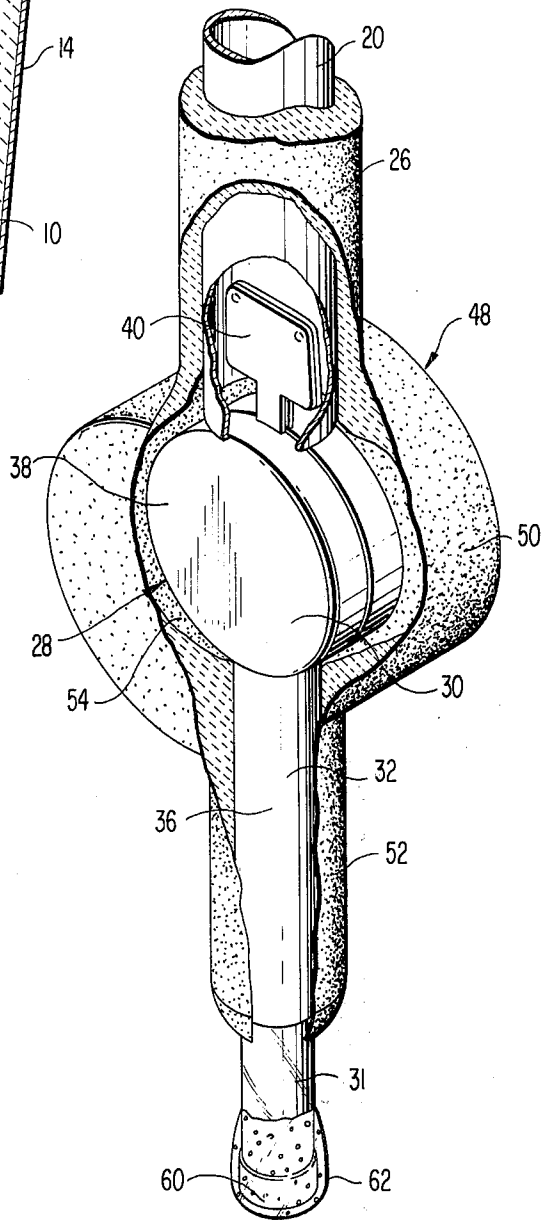
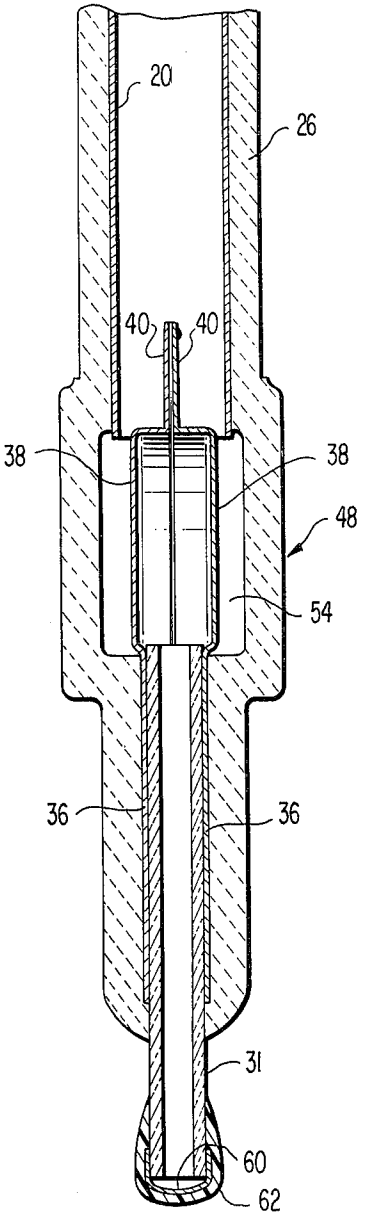


FIG. 3



SAMPLING DEVICE FOR MOLTEN METAL

BACKGROUND OF THE INVENTION

In the steel industry a continuing problem is the rapid procurement of solidified samples of molten steel during processing to be used for constituent analysis. Samples for this purpose have been obtained in the past by dipping into the molten metal in the bath or in a ladle with a spoon to bring out a little of the melt and then pouring some of this molten metal into a mold to obtain a satisfactory ingot which could be used for analysis. This method often resulted in a sample unrepresentative of the bath because of changes in composition taking place in the spoon.

There are two desirable shapes in present day testing, pin-type samples and flat disc shaped samples. It has been proposed to combine these two types of samples into one called lollipop. This type of testing shape is recovered in present practice by utilizing an immersible mold structure which upon insertion into a molten bath becomes filled with molten metal which solidifies to form the test piece. In this operation the apparatus for obtaining the test piece has an open end through which the molten metal can enter the mold by hydrostatic action. In some cases this open end is closed before insertion into the bath to be sampled by closure means which will melt at the time of insertion of the apparatus into the bath. The closure member is designed to melt after the immersible end of the apparatus has passed through the slag layer which overlies the bath. In the case of sampling the molten metal in a continuous casting mold, the slag is formed by casting powder. The molten metal entering the sample mold has been made to do so by hydrostatic pressure in the bath. Where the molten steel being sampled is not killed it has been suggested that the cap which closes the lower end of the mold inserted into the bath may be formed of a deoxidizing material for the metal entering the mold.

One of the difficulties encountered in obtaining pin and lollipop sample specimens has been the entry of impurities into the sample which are traceable to the slag or casting powder adhering to the closed end of the mold as it passes through the slag layer.

One form of apparatus for obtaining a sample is illustrated in U.S. Pat. No. 3,686,949 where the apparatus incorporating the sample mold is in the form of a dipper. In that environment also the problem of impurities from the slag getting into the sample is present. The patentee proposes using a "cover . . . formed of laminated assemblies of paper, plastic, various metals, or combinations thereof. For example, an exposed upper layer of sheet aluminum or paper melts and vaporizes with enough turbulence to avoid such undesired slag deposits on (the) cover . . . " (col. 15, lines 4–9)

Additional forms of sampling apparatus are shown in U.S. Pat. Nos. 3,452,602 and 3,646,816. In each of these patents the open end of the sampling device which is inserted into the body of molten metal to be sampled is closed by metal covers which may be of deoxidizing material in respect to the metal being sampled.

None of the devices illustrated in the aforegoing mentioned patents depends upon suction for drawing the molten metal to be sampled into the mold. U.S. Pat. No. 3,534,614 does use suction but in that case the suction is derived from an evacuated and sealed glass tube, the lower end of which is exposed for ready melting on exposure to the liquid metal whereby the liquid metal is sucked into the remaining portion of the tube.

Recognizing the advantages of using suction to fill the mold with metal to be sampled, while avoiding the problems involved in sealed evacuated sample molds, applicant has invented a sampler which is lowered into a bath or pool of the liquid to be sampled on the end of a long conduit having a suction pump or the like connected to the upper end. This arrangement requires that the means for closing the lower end of the device have a specialized sealing action since failure of the seal between the cover and the opening at the lower end prior to the proper time for drawing in the sample will result in slag and impurities being sucked into the mold. For this reason the types of covers and caps heretofore proposed have not been satisfactory for this kind of sampling device.

Not only is the sealing between the cap and the lower end of the sampling device important where suction is applied to the entire interior of the sampling device from the operator's end but there is the further effect of the pressure differential exerted on the cap or cover by the suction at the time the cap is first subjected to the cap or cover by the suction at the time the cap is first subjected to the high temperature of the bath as the cap or cover enters the slag layer. Softening of the cap at this point could result in collapse of the cap. More important, premature melting or even vaporization of the cap will result in the liquid cap metal or decomposition products being sucked into the mold thereby polluting the sample.

Thus the fact that suction applied at the operator's position is now recognized as the best method for obtaining a sample has introduced new problems, solution of which has been applicants' desideratum.

BRIEF DESCRIPTION OF THE DRAWINGS

In the accompanying drawing which illustrates a specific embodiment of applicants' invention:

FIG. **1** is a view of the preferred embodiment and the environment of use;

FIG. **2** is a perspective view of the preferred embodiment with parts broken away for better illustration; and

FIG. **3** is a view in cross section taken on the line **3—3** of FIG. **2.**

DESCRIPTION OF PREFERRED EMBODIMENT

Referring to the drawing and especially FIG. **1,** a bath or pool of molten steel **10** having on its surface a layer of slag or casting powder **12** is held within a refractory lined receptacle, treating vessel or tundish **14.** Where the receptacle is a continuous casting mold the refractory of course is not present. A sampling device indicated generally at **18** is shown projecting through the layer of slag **12** into the bath of molten metal **14.** The sampling device is carried on the lower end of a long, rigid conduit or pipe **20** which has attached to its upper end by means of a flexible hose **22** an evacuating device **24** such as a mechanical roughing vacuum pump. The portion of pipe **20** projecting into the bath is protected by a refractory sheath **26.**

Referring to FIG. **2,** the sampling mold component of sampling device **18** is indicated generally at **28.** This mold is made up of a hollow, flat metallic section indicated at **30** for forming the desired disc sample, a fused quartz tube **31** for forming the desired pin sample and

a cylindrical metallic holder **32** for the quartz tube carried by section **30.** Where desired the sample mold need not include provision for forming a disc sample. The metallic portion of mold **28** is made up of two identical halves each half being made up of a semi-cylindrical portion **36,** a pan shape portion **38** and projecting ear means **40** for holding the component halves together but sufficiently spaced so as to place the interior of the disc shaped mold in gaseous communication with the space surrounding the disc shaped mold. Ear means **40** are formed from a couple of slightly tapered ears which are spot welded together, the resulting structure being wedgeable into the lower end of pipe **20** to hold sample mold **28** rigidly in place at the lower end of pipe **20.**

The two pan shaped elements **38** form the disc sample mold section and the semi-cylindrical elements **36** carried by pan shaped elements **38** embrace and hold fused quartz tube **31** which is at least as long a desired pin sample. The open upper end of quartz tube **31** opens into the interior of disc sample mold **30** and therefore the interior of quartz tube **31** is in open communication with the interior of disc sample mold **30.**

Preferably integrally molded on the lower end of refractory sheath **26** is an impervious refractory casing indicated generally at **48** formed of a section **50** surrounding and in spaced relation to disc sample mold **30** and an integral depending tubular portion **52** preferably molded around the cylindrical metallic holder section **36** with the lower portion of tubular portion **52** molded around and holding quartz tube **31** in place while leaving the lowermost portion of quartz tube **31** exposed. Since casing **48** is integrally molded onto sheath **26** and tubular extension **52** is integrally molded onto enlarged portion **50** of casing **48** and since tubular extension **52** of the refractory casing is molded onto and in sealing relation with quartz tube **31,** the interior of refractory casing **48** can be subjected to the vacuum generated at means **24** between mold section **30** and section **50** of the refractory casing. Space **54** acts as thermal insulator and conduit for applying vacuum to mold **30** interior. This last is because the pan shaped halves of mold **30** are slightly spaced and not in sealing contact with each other and when subatmospheric gas pressure present in the cavity **54** it is present within the disc sample mold **30** and the interior of fused quartz tube **44.**

In order to preserve the interior of the sample molds free from contamination prior to taking a sample, the lower open end of quartz tube **31** is closed by a metal cap **60,** which in turn is enveloped by a plastic coating **62** entirely covering the cap and extending beyond the cap around and in continuous sealing contact with the exterior surface of fused quartz tube **31** in the neighborhood of the cap **60** as to seal cap **60** to the lower end of quartz tube **31** and thereby maintain the interior of the entire system under subatmospheric pressure generated at means **24.** Metal cap **60** is preferably formed of a metal, such as mild steel, which has no constituents which would interfere with test results.

The coating **62** is a continuous, homogeneous plastic coating. The composition of this plastic and the thickness of the plastic coating enter into the present invention in addition to the sealing function of the plastic. The composition of this plastic material and the thickness of the plastic coating is such that introduction and movement of the sampling device into and through the surface layer of slag or fluxing material on the molten metal and thence into the body of molten metal to be sampled will result in the plastic material decomposing into gaseous form at a rate and in a quantity to remove completely from proximity to the exterior walls of the lower end portion of the fused quartz tube any slag or fluxing material which otherwise would be adhering to the lower end portion of the fused quartz tube so as to contaminate the sample entering the mold.

The coating of plastic material is preferably applied as a liquid and the resulting coating hardened or allowed to harden to form the final protective coating. The viscosity of the liquid plastic material is controlled or the number of applied coats is used to achieve the desired thickness of the final coating layer. The coating need extend no further up the fused quartz tube than is necessary to adequately seal the joint between the metal cap and fused quartz tube. If the plastic coating material application is too thin for a given plastic material, insufficient liberation of gas takes place to accomplish the desired purposes of the invention. If the layer of coating material is too thick, melting of the cap is delayed to a point of time where the mold itself is damaged by the heat of the melt.

Choice of the plastic covering in the present invention must be such as to solve the basic requirements for successful sampling of molten steel baths and especially in the continuous casting mold environment. The final solidified plastic material coating must be strong enough to mechanically hold the cap on the fused quartz tubing before and during initial insertion of the sample through the slag or fluxing material on the surface of the liquid metal. The final solidified plastic material coating must result in a vacuum tight sealing action between the metal cap and the fused quartz tubing prior to and during initial insertion of the sampler through the slag and/or casting powder materials covering the liquid metal. The solidified plastic material coating must have a low decomposition activity or boil off so as to give protection to the metal cap for a sufficient time, e.g. one to two seconds, to avoid adhesion to and consequent contamination of the exterior of the metal cap by the slag or casting powder material until the intake opening end of the fused quartz tube can penetrate to the liquid metal. The solidified plastic material coating is preferably an organic material that will boil free of the metal cap without leaving a decomposition product of its own, including carbon, carbonaceous residues and/or residues of elements of analytic interest in respect to the sample that might be taken into the sample as the cap melts. The solidified plastic material coating must have little or no affinity for moisture that could be accidentally introduced into the liquid steel and cause serious accidents.

Examples of the best plastics invetigated by the applicant are acrylic resins derived from acrylic and methacrylic acids and copolymers of these or copolymers formed utilizing as one reactant acrylic or methacrylic acid or a derivative of one of these acids, all of such resins being well known in the art. The brand names of some of these resins are Lucite, Plexiglass and Pli-bond cement. These resins are described in "Acrylic Resins" by Milton Horn, Reinhold Publishing Corporation 1960, Library of Congress Catalog Card Number 60-8707, especially pages 1-13 and 114-117 and in "Handbook of Common Polymers" pages 87-99, The Chemical Rubber Co., Cleveland, Ohio, Library of

Congress Catalog Card Number 74-173090, both of these publications being incorporated by reference herein.

The plastic can be applied, preferably in liquid form by dipping, in one or more layers dependent on the viscosity, the layers being intimately bound to each other to form a homogeneous and impervious coating, the coating adhering to the cap and tubing to seal the cap and tubing joint. The solidified plastic should have a thickness sufficient to last for approximately one to two seconds while penetrating a layer of casting powder on molten steel in a continuous casting mold, such thickness in one case being 0.010 inches. If the plastic is too thick, too much volatile gas is generated and the cap can be blown off the tube or removed by the activity of the vaporizing plastic before the opening end of the tube has penetrated through the slag layer to the molten steel or the cap can be kept from melting for too long a time, thus allowing adverse decomposition of the quartz tubing. The use of a liquid plastic which is allowed to harden or polymerize after dipping or painting it on helps insure freedom from large trapped air pockets and moisture that could blow the plastic cap off prematurely. Instead of applying plastic by dipping or painting, a preformed thin plastic cap can be placed on the metal cap and the end of the tube and heated to near liquid state but there appears to be no advantage for this way of arriving at the desired coating.

Experimentation has proved that some forms of plastic materials are not suitable for purposes of the present invention. Thus an amberoid cement subjected to the same temperature conditions as those existing while sampling a molten steel bath left a slight residue and took about a second longer than materials named above. A proprietary plastic sold under the name Dip 'n Grip showed a gross carbonaceous residue and prolonged disintegration time of 12 seconds. demonstrating that some types of plastic are not usable in the environment of the present invention.

We claim:

1. A molten metal sampling device for use with a source of vacuum comprising

a. an elongated tubular support member open at both end portions and connectable at its upper end portion to the source of vacuum,

b. a gas impervious, thermal insulating, refractory casing supported by and hermetically sealed to the lower end portion of the tubular support member,

c. a cylindrical mold open at each end, one end portion of the cylindrical mold projecting into and being supported by and in hermetically sealed relation to the refractory casing with the opening in the upper end portion in communication with the interior of the refractory casing and with the lower end portion of the cylindrical mold exposed,

d. a metal cap closing the opening in the lower end portion of the cylindrical mold while leaving exposed a portion of the exterior walls of the cylindrical mold contiguous to the metal cap, and

e. a continuous, homogeneous and impervious plastic material coating covering the metal cap and at least a portion of the exposed exterior walls of the portion of the cylindrical mold contiguous to the metal cap, the plastic material coating hermetically sealing the connection between the metal cap and the cylindrical mold,

f. the thickness of the plastic material coating and the composition of the plastic material coating being such that on movement of the sampling device into a body of molten metal to be sampled through a surface layer of slag or fluxing material, the plastic material coating will decompose into gaseous form at a rate and in a quantity to remove completely from proximity to the lower end exterior walls of the cylindrical mold with the desired sample of the molten metal.

2. A device as claimed in claim **1** in which

g. the character of the plastic material coating results from dipping the metal cap and said portion of the exposed sidewalls of the lower portion of the cylindrical mold into a body of liquid plastic material and hardening the resulting coating.

3. The device claimed in claim **2** in which

h. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

4. The device claimed in claim **1** in which

g. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

5. A molten metal sampling device for use with a source of vacuum comprising

a. an elongated tubular support member open at both end portions and connectable at its upper portion to the source of vacuum,

b. a sample mold chamber supported by the tubular support member, the interior of the mold chamber being in communication with the open lower end of the tubular support member,

c. an elongated sample cylindrical mold having exterior walls and openings at both end portions into the interior, the cylindrical mold having the opening at its upper end portion in communication with the interior of the mold chamber,

d. a gas impervious, thermal insulating refractory casing surrounding and spaced from the mold chamber and surrounding and in hermetically sealed relation to the lower end portion of the tubular support member and the upper end portion of the cylindrical mold,

e. a metal cap closing the opening in the lower end portion of the cylindrical mold while leaving exposed a portion of the exterior walls of the cylindrical mold contiguous to the metal cap, and

f. a continuous, homogeneous and impervious plastic material coating covering the metal cap and at least a portion of the exposed exterior walls of the portion of the cylindrical mold contiguous to the metal cap, the plastic material coating hermetically sealing the connection between the metal cap and the cylindrical mold,

g. the thickness of the plastic material coating and the composition of the plastic material coating being such that on movement of the sampling device into a body of molten metal to be sampled through a surface layer of slag or fluxing material, the plastic material coating will decompose into gaseous form at a rate and in a quantity to remove completely from proximity to the lower end exterior walls of the cylindrical mold slag or fluxing material which otherwise might enter the cylinder with the desired sample of the molten metal.

6. A device as claimed in claim **5** in which

h. the character of the plastic material coating results from dipping the metal cap and said portion of the exposed sidewalls of the lower portion of the cylindrical mold into a body of liquid plastic material and hardening the resulting coating.

7. The device claimed in claim **6** in which

i. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

8. The device claimed in claim **5** in which

h. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

9. A molten metal sampling system comprising

a. an evacuating means for continuously supplying a vacuum

b. an elongated tubular support member open at both end portions and connectable at its upper end portion to the means for supplying vacuum,

c. a gas impervious, thermal insulating, refractory casing supported by and hermetically sealed to the lower end portion of the tubular support member,

d. a cylindrical mold open at each end, one end portion of the cylindrical mold projecting into and being supported by and in hermetically sealed relation to the refractory casing with the opening in the upper end portion in communication with the interior of the refractory casing and with the lower end portion of the cylindrical mold exposed,

e. a metal cap closing the opening in the lower end portion of the cylindrical mold while leaving exposed a portion of the exterior walls of the cylindrical mold contiguous to the metal cap, and

f. a continuous, homogeneous and impervious plastic material coating covering the metal cap and at least a portion of the exposed exterior walls of the portion of the cylindrical mold contiguous to the metal cap, the plastic material coating hermetically sealing the connection between the metal cap and the cylindrical mold,

g. the thickness of the plastic material coating and the composition of the plastic material coating being such that on movement of the sampling device into a body of molten metal to be sampled through a surface layer of slag or fluxing material, the plastic material coating will decompose into gaseous form at a rate and in a quantity to remove completely from proximity to the lower end exterior walls of the cylindrical mold with the desired sample of the molten metal.

10. A system as claimed in claim **9** in which

h. the character of the plastic material coating results from dipping the metal cap and said portion of the exposed sidewalls of the lower portion of the cylindrical mold into a body of liquid plastic material and hardening the resulting coating.

11. The system claimed in claim **10** in which

i. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

12. The system claimed in claim **9** in which

h. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

13. A molten metal sampling system comprising

a. an evacuating means for continuously supplying a vacuum

b. an elongated tubular support member open at both end protions and connectable at its upper portion to the means for supplying vacuum,

c. a sample mold chamber supported by the tubular support member, the interior of the mold chamber beihg in communication with the open lower end of the tubular support member,

d. an elongated sample cylindrical mold having exterior walls and openings at both end portions into the interior, the cylindrical mold having the opening at its upper end portion in communication with the interior of the mold chamber,

e. a gas impervious, thermal insulating refractory casing surrounding and spaced from the mold chamber and surrounding and in hermetically sealed relation to the lower end portion of the tubular support member and the upper end portion of the cylindrical mold,

f. a metal cap closing the opening in the lower end portion of the cylindrical mold while leaving exposed a portion of the exterior walls of the cylindrical mold contiguous to the metal cap, and

g. a continuous, homogeneous and impervious plastic material coating covering the metal cap and at least a portion of the exposed exterior walls of the portion of the cylindrical mold contiguous to the metal cap, the plastic material coating hermetically sealing the connection between the metal cap and the cylindrical mold,

h. the thickness of the plastic material coating and the composition of the plastic material coating being such that on movement of the sampling device into a body of molten metal to be sampled through a surface layer of slag or fluxing material, the plastic material coating will decompose into gaseous form at a rate and in a quantity to remove completely from proximity to the lower end exterior walls of the cylindrical mold slag or fluxing material which otherwise might enter the cylinder with the desired sample of the molten metal.

14. A system as claimed in claim **13** in which

i. the character of the plastic material coating results from dipping the metal cap and said portion of the exposed sidewalls of the lower portion of the cylindrical mold into a body of liquid plastic material and hardening the resulting coating.

15. The system claimed in claim **14** in which

j. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

16. The system claimed in claim **13** in which

i. the plastic material coating comprises at least one substance selected from the group consisting of resins derived from acrylic and methacrylic acids.

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