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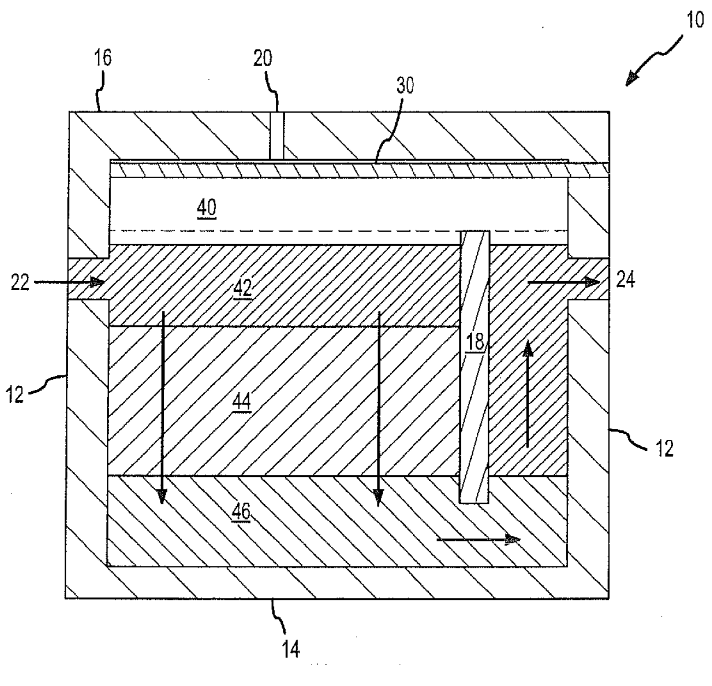

FIG. 1

(57) Abstract: Methods, apparatus and systems for protecting molten aluminum-magnesium alloys from oxidation during production processes are provided. The methods generally involve passing a carbon dioxide containing cover gas comprising at least about 5 volumetric percent up to 100 volumetric percent carbon dioxide over one or more surfaces of the molten aluminum-magnesium alloy, thereby forming a protective barrier on the surface of the molten aluminum-magnesium alloy.

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METHODS, SYSTEMS AND APPARATUS FOR PRODUCING ALUMINUM USING CARBON DIOXIDE AS A REACTIVE COVER GAS

CROSS REFERENCE TO RELATED APPLICATION

[0001] The present invention claims the benefit of U.S. Provisional Application Serial Number 60/890,985, entitled "METHODS, SYSTEMS AND APPARATUS FOR PRODUCING ALUMINUM USING CARBON DIOXIDE COVER GASES" filed on February 21, 2007, which is incorporated herein.

FIELD OF THE INVENTION

[0002] This invention relates to methods of restricting oxidation of aluminum and aluminum alloys (e.g., aluminum-magnesium alloys) during casting or other related ingot producing processes.

BACKGROUND OF THE INVENTION

[0003] Prior to casting, most alloys require some type of filtration to remove impurities. These impurities are typically oxide particles, but can be any second phase materials present in the molten aluminum that are not desired in the final product. Bed filtration is generally accepted to produce the highest quality of molten metal. Filtration media utilized in bed filtration generally include layers of alumina balls and irregular shaped alumina mesh. A plurality of layers of balls and mesh material of specific particle size ranges are typically used. Metal flows down through these layers and inclusions are captured. As molten metal is filtered, the bed eventually becomes loaded with inclusions and begins to develop a growing head drop from inlet to outlet (pressure drop) that eventually requires the bed to be removed from service and replaced.

[0004] There are various costs associated with running a bed filtration system. For example, there are costs associated with the materials and labor required to rebuild the beds between uses, along with the disposal cost for the spent filter media. The production downtime associated with changing out the beds can be significant. There are also the costs of electrical power and systems required to keep the bed hot between casts and maintain the desired temperature at the start of the cast. Finally, conventional beds are required to be skimmed after every alloy drop to minimize the skim layer (also referred to herein as dross) that develops during the cast. The dross that develops in the filter results in metal loss in two ways: metal is destroyed through oxidation and metal is lost from the system by entrainment in the dross. Skimming the filter disturbs the filtering unit and may result in inclusion releases and additional inclusions settling from the dross settling into the bed and consuming some of the capacity of the bed.

BRIEF DESCRIPTION OF THE FIGURES

[0005] Figure 1 is a schematic, cross-sectional view illustrating one embodiment of a filter bed useful in producing molten alloys.

[0006] Figure 2 is a graph illustrating results from a thermogravimetric analysis of an aluminum-magnesium alloy contacted with various gases.

[0007] Figure 3 is a graph illustrating results from a thermogravimetric analysis of an aluminum-magnesium alloy contacted with various gases.

[0008] Figure 4 is a graph illustrating results from a thermogravimetric analysis of an aluminum-magnesium alloy contacted with various gases.

[0009] Figure 5 is a schematic view of one embodiment of a casting pit layout.

[0010] Figure 6 is a graph illustrating the skim weight of various alloys generated in a bed filter relative to various cover gases.

[0011] Figure 7 is a graph illustrating the skim weight of various alloys generated in a bed filter relative to various cover gases.

DESCRIPTION OF THE INVENTION

[0012] The present invention creates an atmosphere containing carbon dioxide that behaves more like a reactive cover gas than an inert cover gas, which is an unexpected and surprising result. Typical reactive cover gases are gases used at low concentrations in a carrier gas, normally ambient air, that react with the molten metal, such as aluminum, at its surface to produce a tight, thin film, that suppresses substantially further oxidation. Also unexpected and surprising, the beneficial protective effects of the carbon dioxide can exist in very high concentration levels of oxygen and last at least one day after the carbon dioxide supply has been shutoff.

[0013] One aspect of the invention is the elimination of gaseous mixtures for cover or shielding containing conventional inhibiting agents such as fluorocarbons (for example, but not limited to, perfluoroketones, hydrofluoroketones, and mixtures thereof), fluorine (for example, but not limited to, difluoromethane, pentafluoroethane, 1,1,1,2-tetrafluoroethane, difluoroethane, heptafluoropropane, dihydrodecafluorpentane, hydrofluorethers, and mixtures thereof), and dilute sulphur hexafluoride mixtures, as well as all other conventional inhibiting agents not mentioned herein.

[0014] Several embodiments of the present invention encompass the beneficial protective effects of the carbon dioxide that can exist in very high concentration levels of oxygen. The carbon dioxide containing gas generally comprises at least about 5 volumetric percent carbon dioxide so as to facilitate the formation of the thin film. In one embodiment, the carbon dioxide containing gas comprises at least about 10 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises at least about 20 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises at least about 50 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises not greater than about 80 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises not greater than about 90 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises not greater than about 95 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises not greater than about 99 volumetric percent carbon dioxide. In one embodiment, the carbon dioxide containing gas comprises 100 volumetric percent carbon dioxide. All of the above volumetric percentages are at standard temperature and pressure. In one embodiment, the remainder of the carbon dioxide containing gas may be made up with ambient air. In one embodiment, such as when the amount of carbon dioxide in the carbon dioxide containing gas is at least about 5 volumetric percent, the remainder of the carbon dioxide containing gas may be made up with oxygen. In one embodiment, the remainder of the carbon dioxide containing gas may be made up with one or more of an oxidizing gas (e.g., ambient air, oxygen) and inert gases (e.g., helium, nitrogen, argon). In one embodiment, the carbon dioxide containing gas consists essentially of carbon dioxide and ambient air. In one embodiment, the carbon dioxide containing gas consists essentially of carbon dioxide and oxygen. In one embodiment, the carbon dioxide containing gas consists essentially of carbon dioxide, ambient air and oxygen. In another embodiment the carbon dioxide containing gas consists of carbon dioxide and another inert gas, such as one or more of helium, nitrogen or argon. However, the above listing of mixtures is not intended to limit the invention but to present examples. Therefore, one skilled in the art based on the teachings herein will be able to determine other air or other inert gas mixture concentrations with carbon dioxide that produces a tight, thin film sufficient to inhibit oxidation of the molten metal.

[0015] Another embodiment of the present invention that illustrates the beneficial protective effects of the carbon dioxide can exist in very high concentration levels of oxygen includes relatively low amounts of carbon dioxide, where, for example, the head space of a bed filter is only partially inerted atmosphere, for example, 12%.

[0016] Another embodiment of the present invention does not purge the casting apparatus of the carbon dioxide containing gas. Since carbon dioxide (density 1.98 g/L) is heavier than air (density 1.2 g/L) and argon (density 1.784 g/L), an effective protective shroud will maintain longer than lighter gases because it will not disperse into the atmosphere as readily. This phenomenon will allow for continued casting after passing of the carbon dioxide containing gas is terminated or the allow for the casting operation to be halted for a period to of up to one (1) day for maintenance or operator availability.

[0017] In view of the foregoing, a broad objective of the present invention is to increase the efficiency of deep bed filtration processes in the production of aluminum and aluminum alloys (e.g., aluminum-magnesium alloys). Another objective is it increases the usable life if a bed filter so as to reduce operational costs. A related objective is to decrease the amount of oxidation of aluminum and aluminum alloys during production processes.

[0018] In addressing one or more of the above objectives, the present inventors have recognized that a carbon dioxide containing gas can be utilized during filtration, casting, or other ingot producing processes to lower the amount of surface oxidation of aluminum and aluminum alloys. By utilizing carbon dioxide gases, dross formation is reduced and, with respect to filtering, the need for skimming can be reduced while increasing useful bed lifetime. One embodiment of a deep bed filter is illustrated in Figure 1. In the illustrated embodiment, the bed 10 includes sidewalls 12 interconnected with a bottom 14 and a top 16. The bed further includes a gas injection inlet 20, a molten metal inlet 22, and a molten metal outlet 24. A heater 30 is disposed below the top 16 of the bed 10. The bed 10 further includes a baffle 18 for configuring the flow of molten alloy through the bed 10. In operation, molten alloy 42 flows through the inlet 22 of the bed 10 through two separate layers of filtering media, the first being alumina mesh 44 and the second being balls of alumina 46. The molten alloy 42 flows downward through the first and second layers of filtering media 44, 46, around baffle 18, and up and out of the bed via outlet 24.

[0019] During operation, a carbon dioxide containing gas is injected via the gas injection inlet 20 into the head space 40 of the bed 10. The carbon dioxide gas generally comprises at least about 5 volumetric percent carbon dioxide (at STP) with the remaining portion comprising ambient air or other gases. It has been unexpectedly found that the use of a carbon dioxide containing gas has greater oxidation resistance than gases containing air diluted with an inert gas. It has been unexpectedly found that the use of the carbon dioxide containing gas as a cover gas facilitates production of a thin protective film on the surface of the molten aluminum, particularly with respect to molten aluminum-magnesium alloys. While the mechanism of film formation is not precisely understood, the thin protective film resists permeation of oxygen into the molten aluminum-magnesium alloy, and thus restricts further oxidation of the molten aluminum-magnesium alloy. In the illustrated embodiment, the carbon dioxide containing gas in the head space 40 interacts with the molten alloy 42 to create the thin film (not illustrated). This thin film creates a barrier between the molten allow 42 and the gases within the head space and restricts diffusion of oxygen to the surface of the molten alloy, thereby restricting further oxidation of the molten alloy and thus lowering the amount of dross / skim produce during filtering operations.

[0020] The pressure within the head space 40 is generally slightly above atmospheric pressure so as to ensure that other gases do not infiltrate the filter box and contact the alloy. However, it is within the contemplation of this invention that yet another aspect of the invention is that carbon dioxide can be effectively perform as an inhibiting agent in a unsealed or pressurized system, which means that ambient air is allowed to leak or permeate into the head space 40, for example. For this example, the flow rate of the carbon dioxide containing gas is generally related to the size of the bed 10. In one embodiment, the flow rate of carbon dioxide into the bed is at least 5 SCFH, such as at least about 25 SCFH. In one embodiment, the flow rate of carbon dioxide into the bed is not greater than 400 SCFH, such as not greater than 100 SCFH.

[0021] While the use of a carbon dioxide containing gas to restrict surface oxidation of aluminum and aluminum alloys (e.g., aluminum-magnesium alloys) has been described in relation to a filter bed, a carbon dioxide containing gas could also be used to restrict oxidation of molten aluminum and aluminum alloys (e.g., aluminum-magnesium alloys) in other processes and/or apparatus, such as during casting or other ingot production processes. For example, the carbon dioxide containing gas could be utilized in casting troughs, in-line degassing units, other types of filtration systems such as ceramic foam bowls and rigid media filters, ingot casting molds, such as direct chilled, horizontal direct chilled or electromagnetically chilled molds, and in shape casting systems such as permanent molds and sand molds. Moreover, while the present disclosure has been mainly with reference to aluminum-magnesium alloys, it is anticipated that the carbon dioxide containing gas could be utilized to restrict oxidation of various molten aluminum metals, such as substantially pure molten aluminum, as well as other aluminum-containing alloys.

Examples

[0022] Example 1. Samples of aluminum-alloy 5083 were held under ambient air, argon and 50:50 CO_2/air atmospheres at three different holding temperatures (1250°, 1300°F, 1350°F). Thereafter, a thermogravimetric analysis (TGA) was completed. The TGA results are illustrated in Figures 2, 3 and 4. The weight increases shown in these plots are the result of the increase weight of the samples due to oxidation because the weight of the oxide is greater than the weight of the base metal. Thus, the change of weight with time is an measure of the rate of oxidation of the sample. The results indicate that, with increasing holding temperature, the oxidation rate of the samples increases dramatically in air. The oxidation rate is reduced with an argon inert atmosphere, as expected, but surprisingly the oxidation rate is even further reduced using a mixture of ambient air and carbon dioxide.

[0023] Example 2. A filter box, similar to that illustrated in Figure 1, was produced. The alumina mesh layer generally had a size of from about +3 mesh to about -6 mesh. The balls of alumina had a diameter of about three-quarters of an inch. As illustrated in Figure 5, the filter box (A94) was placed in-line with a furnace (40K), a degassing unit (A622), and a casting pit. Tests were run with the filter bed in either a standard flow profile (as illustrated in Figure 1) or an upflow profile (an inverted configuration of Figure 1), with various flow rates of argon and/or CO_2 gas flowing into the headspace of the filter box. Two different alloys (5182, 3004) were utilized during the test. During tests where ingots were cast, LiMCA (liquid metal cleanliness analysis), PoDFA (porous disc filtration analysis), and AlSCAN (hydrogen in metal analysis) measurements were made. Each day of casting generally produced two casts, and the filter bed was skimmed following the second cast of the day. Skim weights were recorded, as well as the oxygen level in the filter box. There were a number of tests where there was no alloy cast, but gas settings were changed and the bed filter was skimmed. In all cases, the recorded skim weight represented skim that formed during a period of approximately 24 hours at the given condition. Table 1, below, provides some of the test results. Graphs of the skim weight relative to the type of incoming gas are illustrated in Figure 6 (standard flow profile) and Figure 7 (upflow profile).

Table 1 - Test Results (LiMCA, Oxygen and Skim Weight data)

Test	Bed Configuration	Alloy	Ar (SCFH)	CO_2 (SCFH)	% O_2	Skim Weight (lbs)	Note
1	Standard	5182	50	0	0.7	19	No Cl2 to A622
2	Standard	5182	50	0	0.8	17	Only 1 drop
4	Standard	5182	50	0	0.9	23	2nd drop
6	Standard	5182	25	25	0.4	0	2nd drop
7	Standard	5182	50	0	3.4	47	Only 1 Drop
9	Standard	5182	0	50	2.9	14	2nd Drop
10	Standard	5182	0	25	3.1	2	1st Drop
12	Standard	5182	0	5	12.2	11	2nd Drop
12a	Standard		0	5	8.8	0	No Cast - Gas Change
12b	Standard		7	0	4.0	13	No Cast - Gas Change
14	Standard	5182	0	0	18.9	12	2nd Drop
18	Standard	5182	0	0	12.3	120	2nd Drop
20	Standard	3004	0	0	8.4	95	2nd Drop
20a	Standard	3004	50	0	3.2	0	No Cast - Gas Change
20b	Standard	3004	0	25	1.0	11	No Cast - Gas Change
22	Standard	3004	0	25	1.0	16	2nd Drop
24	Standard	3004	50	0	1.2	16	2nd Drop
26	Upflow	5182	0	5	3.3	6	2nd Drop
28	Upflow	5182	0	5	5.6	3	2nd Drop
30	Upflow	5182	5	0	2.1	8	2nd Drop
30a	Upflow	5182	5	0	2.3	13	No Cast - Gas Change
30b	Upflow	5182	25	0	15.1	9	No Cast - Gas Change
32	Upflow	5182	0	5	9.6	6	2nd Drop
34	Upflow	5182	0	0	18.8	14	2nd Drop
36	Upflow	3004	0	0	9.9	70	2nd Drop
38	Upflow	3004	0	5	2.5	14	2nd Drop
38a	Upflow	3004	0	50	20.5	1	No Cast – Air Pumped In
38b	Upflow	3004	50	0	8.8	12	No Cast – Air Pumped In

[0024] The skim weights from the use of carbon dioxide were less than the skim weights from the use of ambient air. Unexpectedly, the skim weights from the use of carbon dioxide were also generally less than that of the skim weights from the use of argon, indicating that the carbon dioxide may be interacting with the surface of the molten aluminum-magnesium alloy to produce a thin protective film that restricts further oxidation of the aluminum-magnesium alloy.

[0025] While various embodiments of the present invention have been described in detail, it is apparent that modifications and adaptations of those embodiments will occur to those skilled in the art. However, it is to be expressly understood that such modifications and adaptations are within the spirit and scope of the claims and doctrines of equivalence.

What is claimed is:

1. A method for minimizing oxidation of molten aluminum comprising the steps of:

a. flowing the molten aluminum through a casting apparatus;

b. passing a cover gas containing carbon dioxide over a surface of the molten aluminum to react the carbon dioxide with the molten aluminum to form an oxidation resistant film;

c. shutting off the cover gas containing carbon dioxide; and

d. optionally, repeating the steps a-c with a delay of no more than one (1) day after the step c,

whereby the residual benefits of the cover gas containing carbon dioxide over a surface of the molten aluminum begin to deteriorate after one (1) day the shutting off the cover gas containing carbon dioxide.

2. The method of Claim 1 wherein the casting apparatus is one of a bed filter, a ceramic foam filter bowl, a rigid media filter, an in-line degassing system, and an ingot casting mold.

3. The method of Claim 2 wherein the ingot casting mold is one of a direct chill ingot casting mold, an electromagnetic ingot casting mold and a horizontal direct chill ingot casting mold.

4. The method of Claim 1 wherein the casting apparatus is a continuous casting system.

5. The method of Claim 4 wherein the continuous casting system is one of a roll caster and a bar caster.

6. The method of Claim 1 wherein the cover gas comprises at least about 5 volumetric percent carbon dioxide.

7. The method of Claim 1 wherein the cover gas comprises at least about 10 volumetric percent carbon dioxide.

8. The method of Claim 1 wherein the cover gas comprises at least about 20 volumetric percent carbon dioxide

9. The method of Claim 1 wherein the cover gas comprises at least about 50 volumetric percent carbon dioxide.

10. The method of Claim 1 wherein the cover gas comprises no greater than about 80 volumetric percent carbon dioxide.

11. The method of Claim 1 wherein the cover gas comprises no greater than about 90 volumetric percent carbon dioxide.

10. The method of Claim 1, wherein the cover gas comprises no greater than about 95 volumetric percent carbon dioxide.

12. The method of Claim 1 wherein the cover gas comprises no greater than about 99 volumetric percent carbon dioxide.

13. The method of Claim 1 wherein the cover gas comprises no greater than about 100 volumetric percent carbon dioxide.

14. The method of Claim 1 wherein the cover gas comprises does not comprise

fluorocarbons mixtures.

15. The method of Claim 1 wherein the cover gas comprises does not comprise fluorine mixtures.

16. The method of Claim 1 wherein the cover gas comprises does not comprise dilute sulphur hexafluoride mixtures.

17. The method of Claim 1 wherein the cover gas comprises does not comprise additional inhibiting agents.

18. The method of Claim 1 wherein the cover gas comprises further comprises an inert gas.

19. The method of Claim 18, wherein the inert gas is helium, nitrogen, or argon.

20. The method of Claim 1 wherein the cover gas further comprises an oxidizing gas.

21. The method of Claim 20 wherein the oxidizing gas is ambient air.

22. The method of Claim 20 wherein the oxidizing gas is oxygen.

23. The method of Claim 1 wherein the step b occurs in a partially inerted atmosphere.

24. The method of Claim 1 wherein the step b in a 12% inerted atmosphere.

25. The method of Claim 1 further comprising the step of heating the cover gas between about 1250° to about 1350°F.

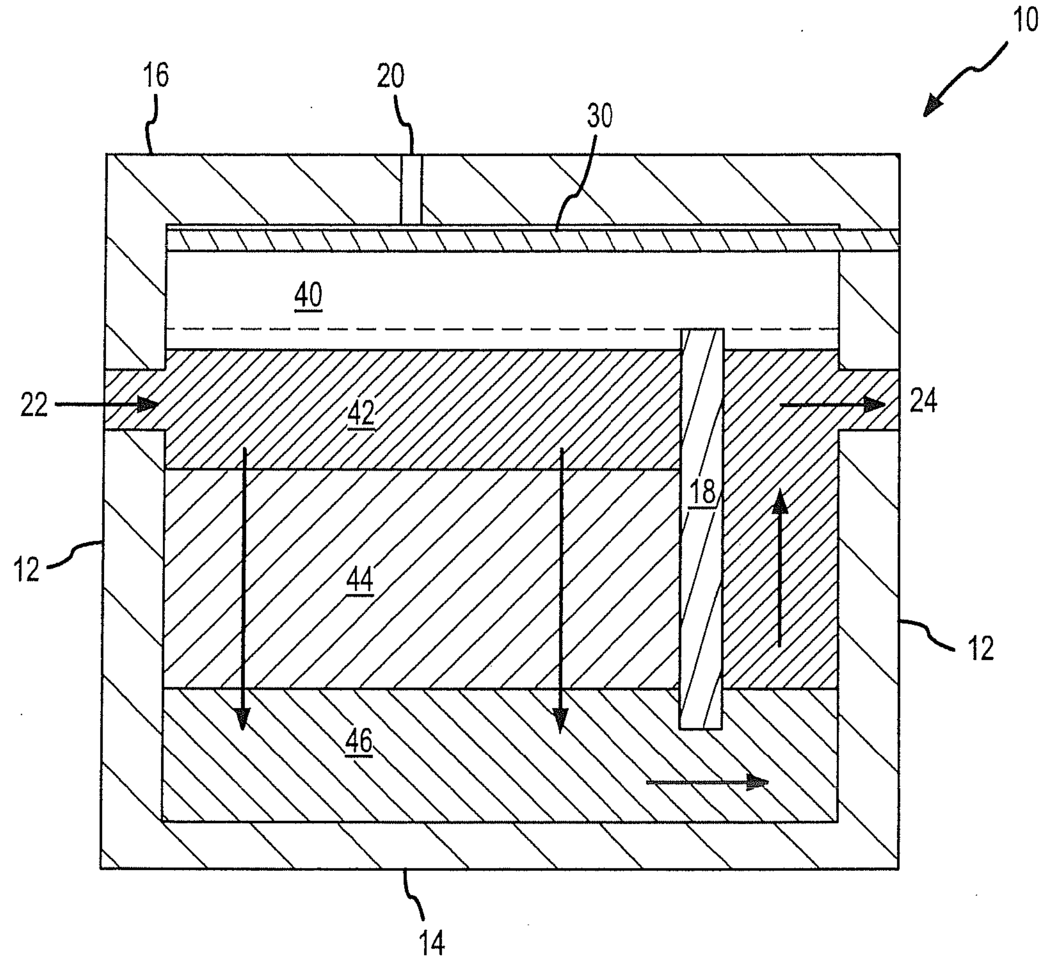
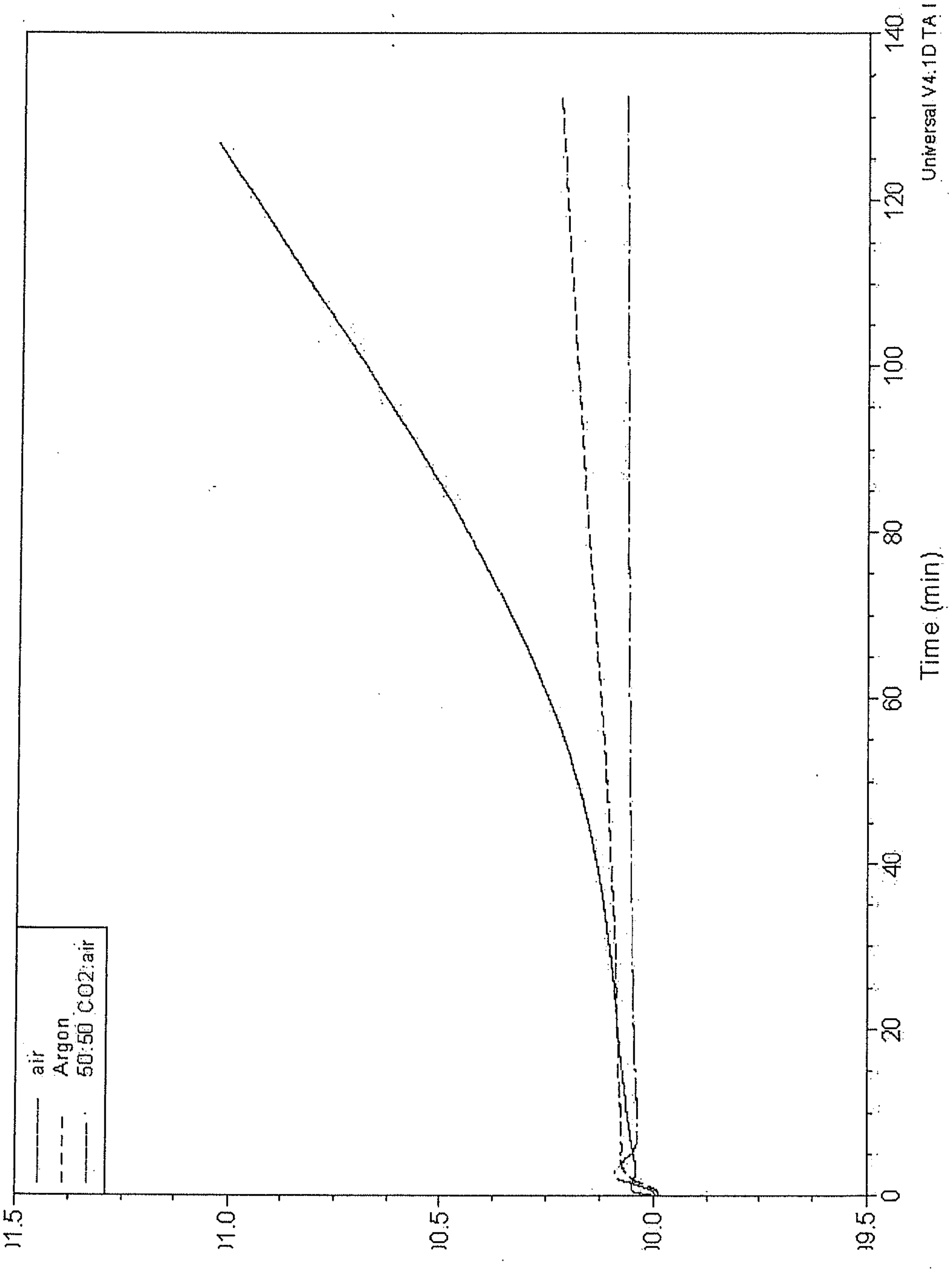


FIG. 1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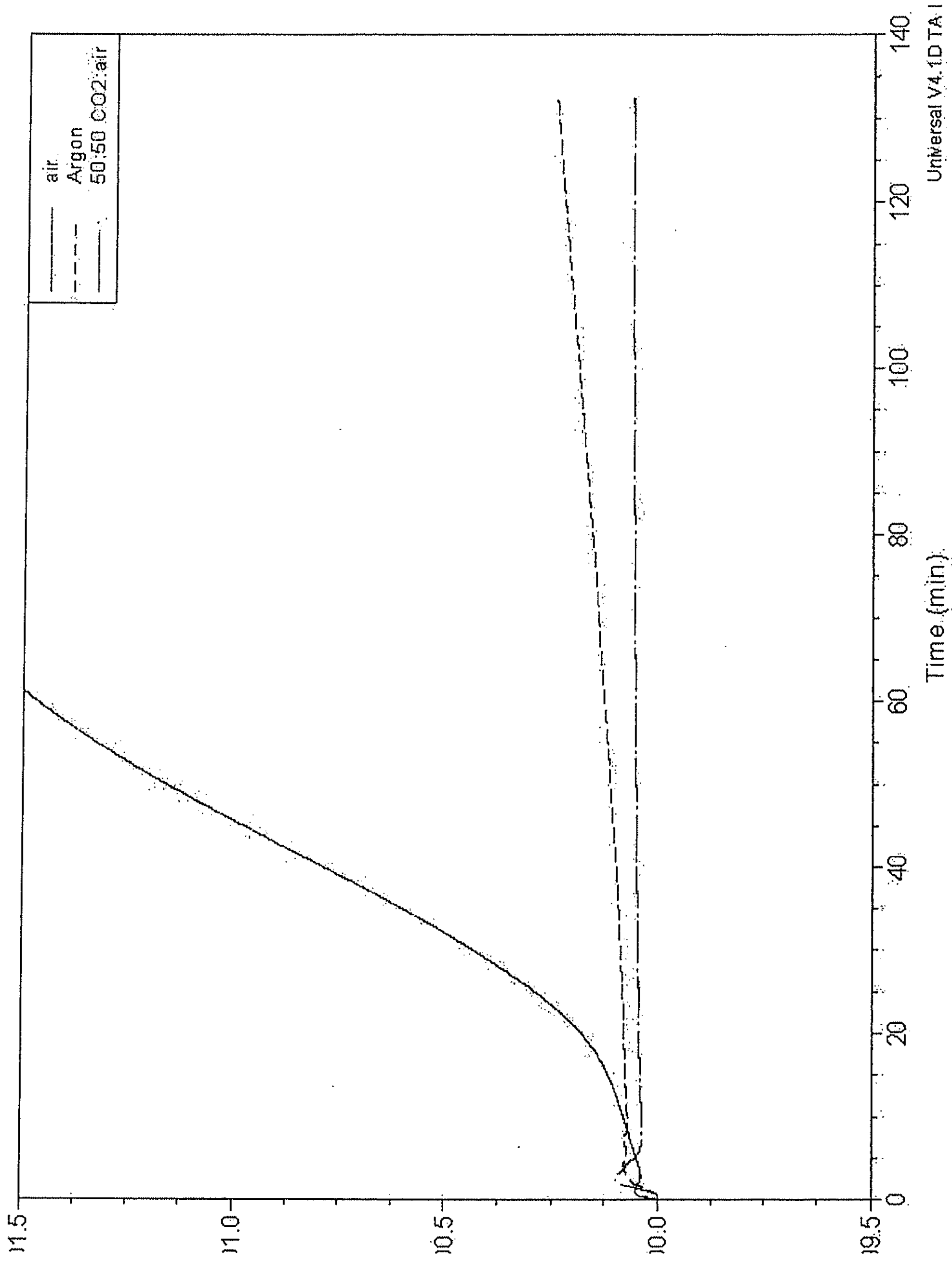
Argon
50:50 CO2:air
0
20
40
60
80
100
120
140
Time (min)
Universal V4.1D TA I
11.5
11.0
10.5
10.0
9.5

Figure 2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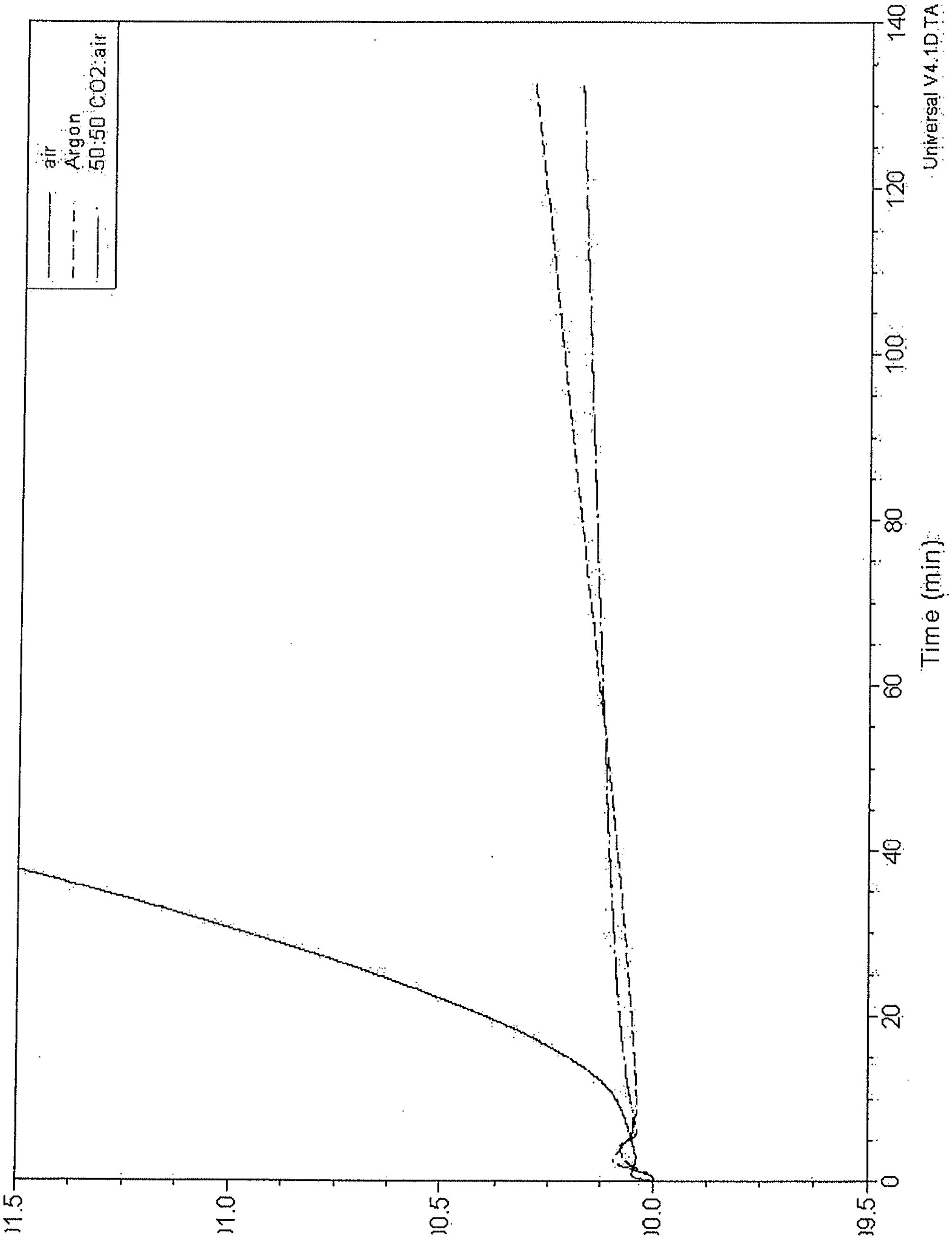
Argon
50:50 CO2:air
0
20
40
60
80
100
120
140
Time (min)
Universal V4.1D TA I

Figure 3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Argon
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11.5
11.0
10.5
10.0
9.5
0
20
40
60
80
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Time (min)
Universal V4.1D TA I

Figure 4

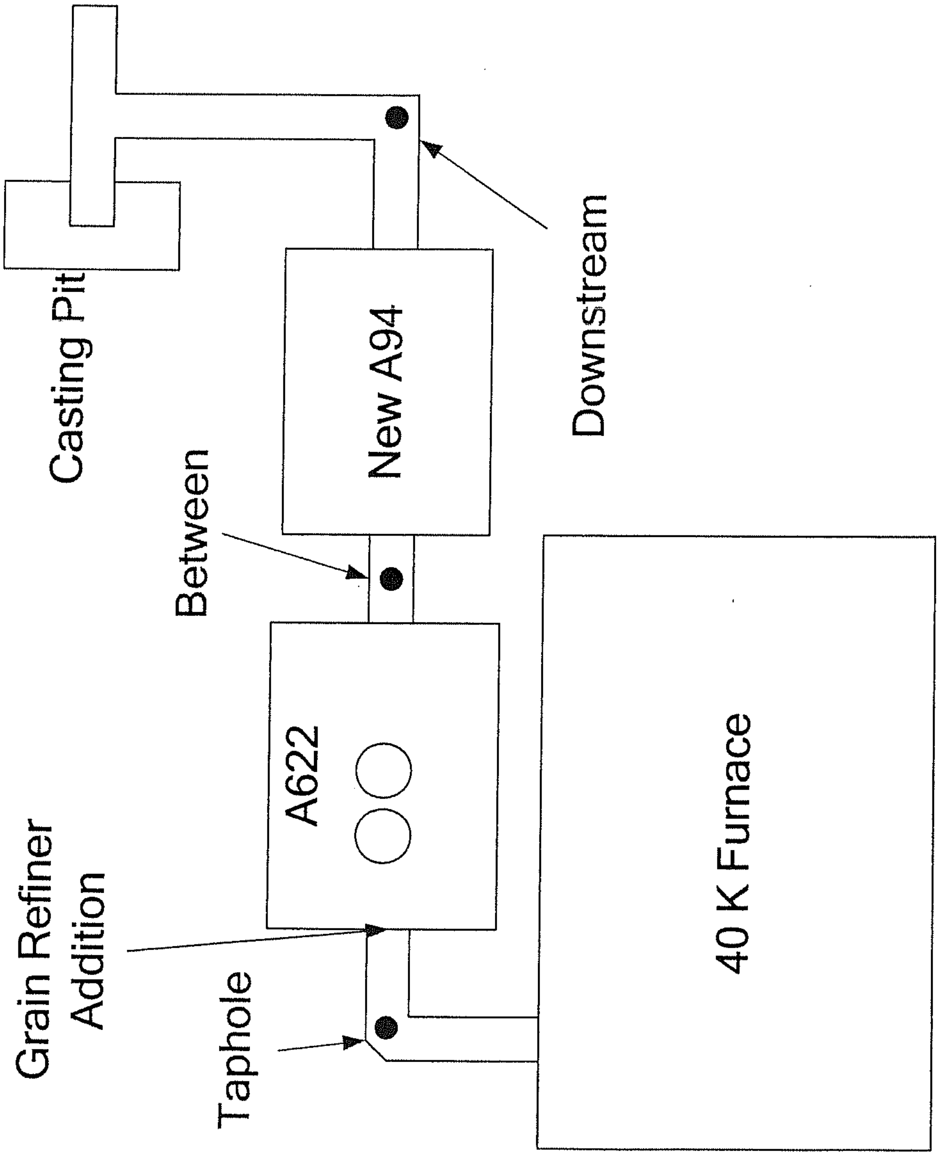
Casting Pit
New A94
Downstream
Between
A622
40 K Furnace
Grain Refiner
Addition
Taphole

Figure 5

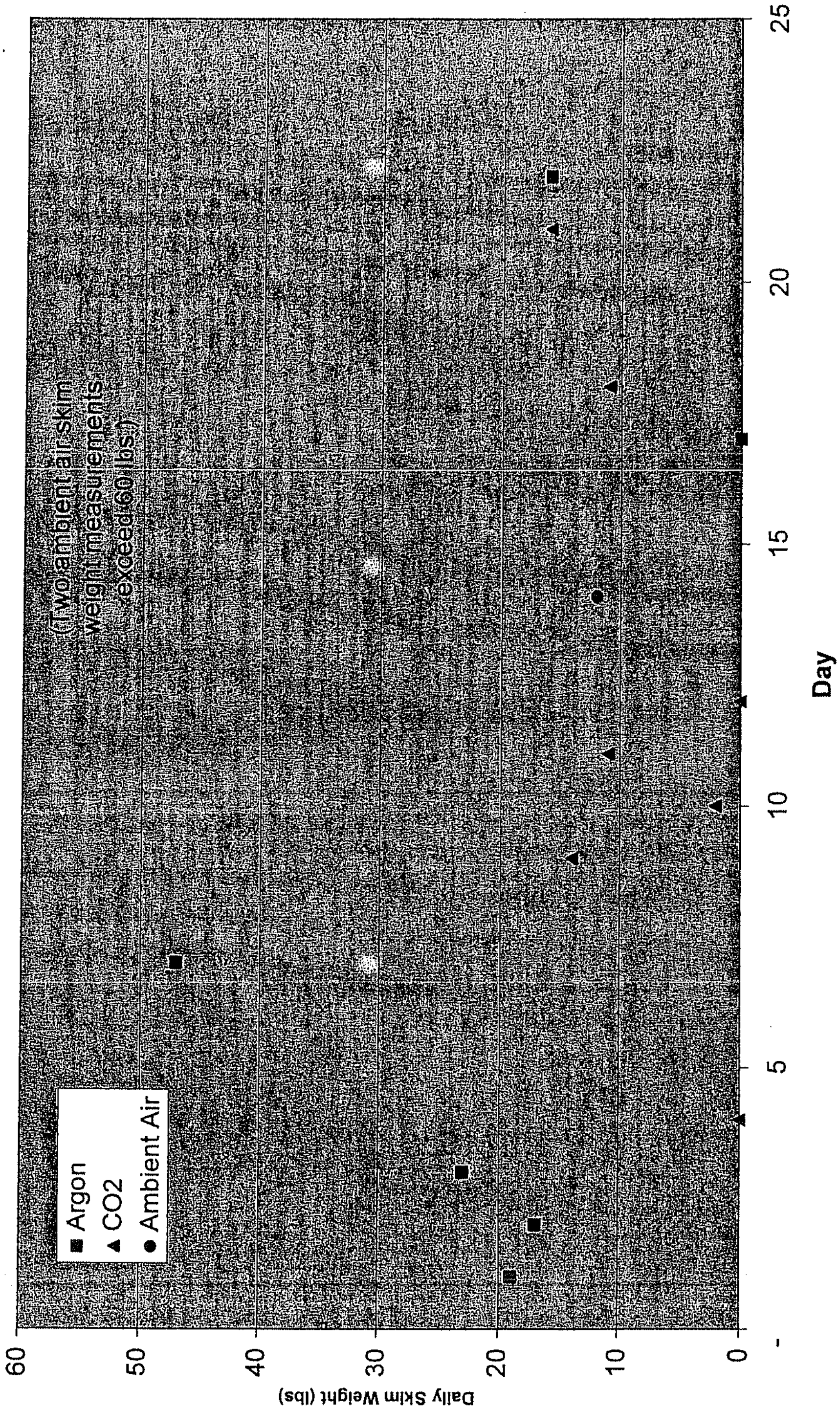
Argon
CO2
Ambient Air
(Two ambient air skim weight measurements exceed 60 lbs.)
Daily Skim Weight (lbs)
Day
0
10
20
30
40
50
60
-
5
10
15
20
25

Figure 6

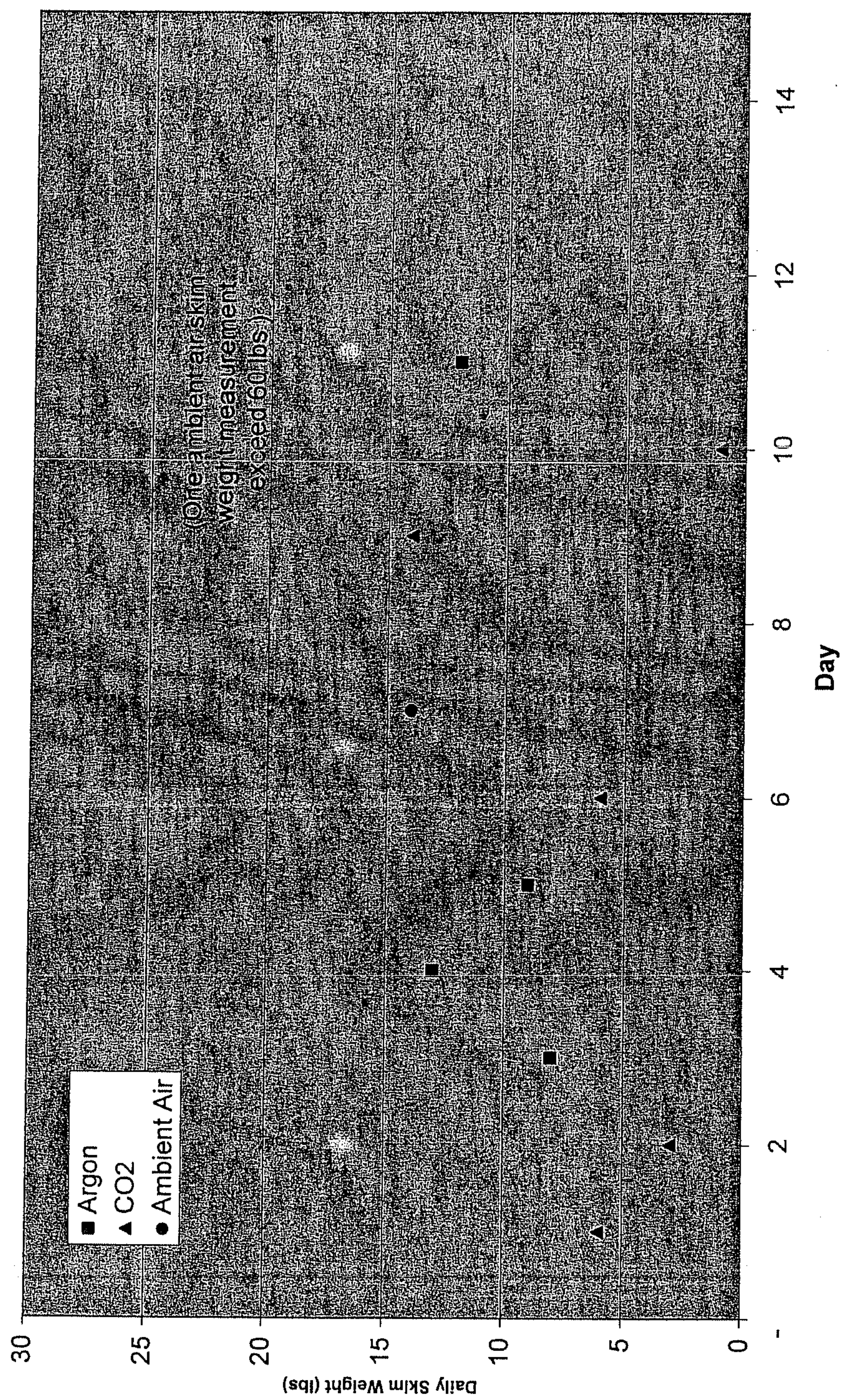
Argon
CO2
Ambient Air
(One ambient air skim weight measurement exceed 60 lbs.)
Daily Skim Weight (lbs)
0
5
10
15
20
25
30
-
2
4
6
8
10
12
14
Day

Figure 7

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2008/054539

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B21/06

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C22B

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

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

EPO-Internal, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 52 043730 A (HAGINOYA IKUO) 6 April 1977 (1977-04-06) abstract	1-13, 18-25
X	DATABASE WPI Week 1986 Derwent Publications Ltd., London, GB; AN 1986-255224 XP002483927 -& JP 61 182869 A (KUBOTA LTD) 15 August 1986 (1986-08-15) abstract	1-13, 18-25
P,X	WO 2007/072831 A (TAIYO NIPPON SANSO CORP [JP]; NAGAKURA YUTAKA [JP]; TOKUDA KENJI [JP];) 28 June 2007 (2007-06-28) abstract	1-13, 18-25

-/--

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
20 June 2008	01/07/2008

Name and mailing address of the ISA/	Authorized officer
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4

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	US 2008/000647 A1 (LULY MATTHEW H [US] ET AL) 3 January 2008 (2008-01-03) the whole document	1-13, 18-25
P,X	DATABASE WPI Week 2007 Derwent Publications Ltd., London, GB; AN 2007-527186 XP002483928 -& CN 1 928 138 A (SUZHOU RES INST OF NONFERROUS [CN]) 14 March 2007 (2007-03-14) abstract	1-13, 18-25

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Claims Nos.: 14-17

Claims 14-17 are unclear due to the contradictory wording "comprising does not comprise" used. As a result, no search can be performed on such an unclear subject-matter.

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guideline C-VI, 8.2), should the problems which led to the Article 17(2)PCT declaration be overcome.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2008/054539

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 14-17
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers allsearchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search reportcovers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2008/054539

Patent document cited in search report		Publication date	Patent family member(s)			Publication date
JP 52043730	A	06-04-1977	JP	987945	C	26-02-1980
			JP	54020164	B	20-07-1979
JP 61182869	A	15-08-1986	NONE			
WO 2007072831	A	28-06-2007	JP	2007167863	A	05-07-2007
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CN 1928138	A	14-03-2007	NONE			