

Aug. 26, 1941.

C. F. NORBECK

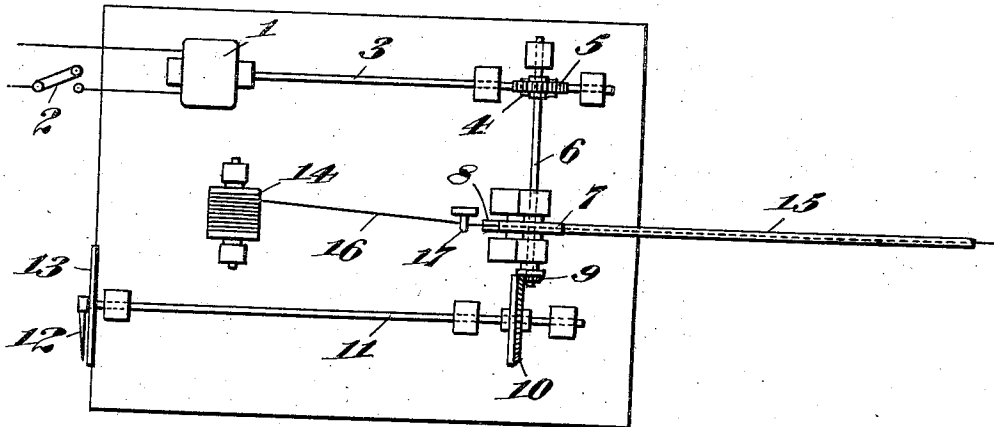
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METHOD OF CONTROLLING THE DEOXIDATION OF STEEL

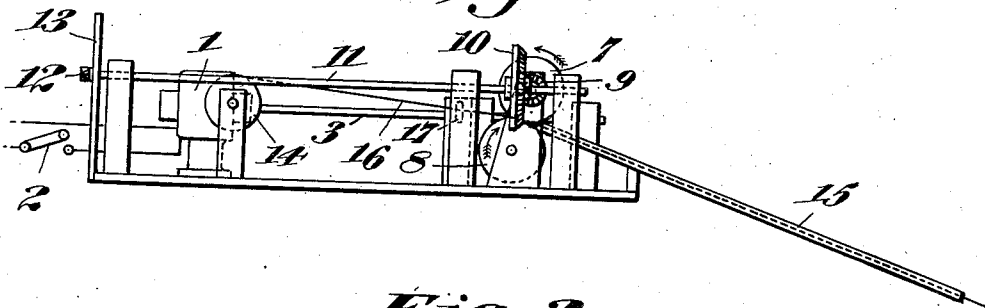
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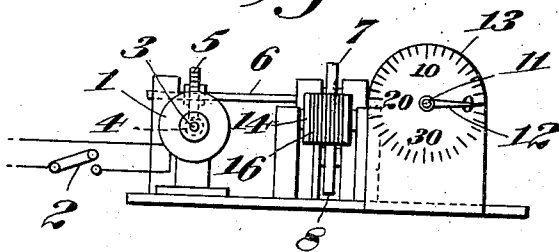
*Fig. 1.*



*Fig. 2.*



*Fig. 3.*



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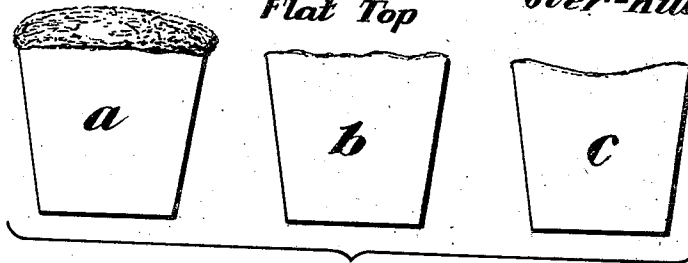
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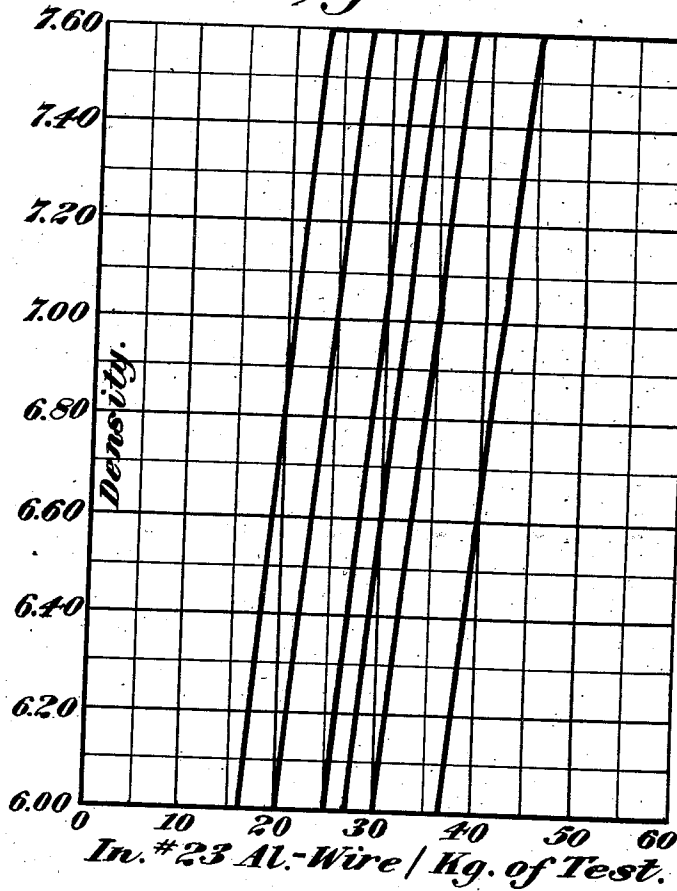
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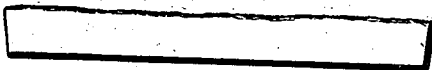
*Fig. 4.* Under-Killed. Killed To Flat Top. Over-Killed.



*Fig. 5.*



*Fig. 6.*



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# UNITED STATES PATENT OFFICE

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## METHOD OF CONTROLLING THE DEOXIDATION OF STEEL

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3 Claims. (Cl. 75-58)

This invention relates to a new, direct, and rapid method of determining the degree of oxidation of a heat of molten steel. The invention is of particular benefit in the production of "rimming" steel, but it is also very useful in the production of semi-killed and killed steel.

Molten steel in the furnace contains a certain amount of oxygen absorbed from the charge, from ore additions, and from the furnace atmosphere. That the steel may solidify properly either as "rimming" steel or as semi-killed or killed steel, and that the desired grain size of the final product may be obtained, exactly the right amounts of deoxidizer must be added to the steel in the furnace and ladle, the amounts of deoxidizer, of course, depending on how much oxygen is present in the molten steel. While misjudgments in the amount of deoxidizer added in the furnace and ladle can sometimes be rectified by making deoxidizing additions in the mold, the latter procedure is generally undesirable.

In rimming steel, if too little deoxidizer is added in the ladle the gas evolution resulting from the reaction of iron oxide and carbon in the steel may cause the steel to fall sharply in the mold. Rather large additions of deoxidizers to the mold may then be required which may contaminate the steel. A sharply falling steel also tends to cause the defect known as "lamination." If too much deoxidizer is added in the ladle the steel will rise sharply in the mold. Such steel invariably gives poor surface in rolling. In semi-killed steel, the addition of too little deoxidizer in the ladle may cause a "blowy" structure, while too much will cause excessive pipe. In killed steel, too little deoxidizer will also give a blowy ingot or casting; too much deoxidizer will cause excessive pipe if no hot top is used. Either too little or too much deoxidizer, for the degree of oxidation of the steel, may give a grain size other than that required, so that exact grain size control is not attained.

For all types of steel, therefore, it is important to know the degree of oxidation of the bath while the steel is still in the furnace, so as to be able to add exactly the right amount of deoxidizer to the furnace or ladle.

A direct way to determine the degree of oxidation of the bath would be chemically to analyze the oxygen content of the molten steel. While such chemical analysis methods are known, they have not been practical in helping to make any particular heat of steel, since they take about an hour or more, by which time the heat would ordinarily already be tapped. Moreover, if the heat

were to be held in the furnace until the oxygen analysis were completed, the oxygen content of the heat would no longer be that indicated by the analysis, because of changes occurring during the comparatively long interval required for the analysis. As a result, the practice has been to resort to the following indirect methods of determining the degree of oxidation of the bath: (1) Analyze the carbon and manganese content of the steel in the bath. (2) Analyze the iron oxide content of the slag above the bath. Both of these types of analyses are faster than an analysis for oxygen of the molten bath.

These indirect methods have serious drawbacks, however. In general it is true that the higher the content of carbon and manganese in the steel, the lower the oxygen content. But this relationship depends on equilibrium being attained within the molten steel which does not always occur, so that frequently there are wide variations in the oxygen content of steels of the same carbon and manganese content. In rimming steels in particular, which are generally low in carbon, the carbon and residual manganese content give but inaccurate indications of the oxygen content. In the time available it is almost impossible to determine the carbon content of the bath closely enough to obtain accurately the relationship between the carbon content and the oxygen content of the molten bath.

It is also difficult, in low carbon steel, to judge the oxygen content of the bath indirectly from the iron oxide content of the slag. In general, the more iron oxide in the slag the more oxidized will be the bath. This holds accurately, however, only when equilibrium between bath and slag is attained, which does not always occur. The relationship only holds for the so-called "active" oxygen content of the slag and not for the "fixed" oxygen combined as compounds of iron oxide with lime and silica, and there is inadequate knowledge of how to ascertain the "active" oxygen content of the slag from the usual analysis for iron oxides. Moreover, although the iron oxide content of the slag can be determined fairly rapidly, say in 20 minutes, even this time interval is fairly long, particularly when the bath is changing rapidly so that the oxygen content at tap may be considerably different from that at the time the sample was taken.

The new direct and rapid method of this invention for determining the degree of oxidation of the bath utilizes, instead of chemical analysis, physical criteria in the appearance and condition of solidified test samples of the molten steel

which indicate the degree of oxidation very accurately.

To a spoonful of the molten steel taken from the furnace aluminum wire is added at a constant rate until a characteristic film appears on the surface of the molten metal. This end point indicates that the sample has been killed to a flat topped condition and in pouring the metal into the usual type of sample test mold this can be verified by observing whether the steel solidifies flat without either rising or piping. It will be evident to those skilled in the art that the steel of the sample presents the same surface appearance as a flat topped ingot of commercial semi-killed steel.

The flat topped condition does not mean that sufficient aluminum has been added to the molten metal to combine with all of the iron oxide present so as to form aluminum oxide. For this a large excess of aluminum would have to be added over that needed to combine quantitatively with the iron oxide. A large excess of aluminum is in fact added in the method of chemical analysis referred to on page 3, line 14, in which the aluminum oxide formed on adding aluminum to the molten steel is determined in order to arrive at its iron oxide content. In the physical method of this invention, however, only just enough aluminum is added to obtain that balanced deoxidized condition at which the surface of the molten steel just begins to show a film—this also being the point at which the test mold solidifies exactly flat without rising or falling. A bubble or blister will appear on the flat top surface of the test indicating that a small amount of carbon monoxide gas produced by the reaction of carbon with iron oxide still remained in the aluminum treated steel. At this stage most of the iron oxide in the steel is converted to aluminum oxide but a considerable residuum of iron oxide remains. Nevertheless, the determination is reproducible and the end point of a flat topped test always denotes the same degree of deoxidation.

The use of the physical criteria or end points makes the procedure very rapid. A test with the required number of check determinations may be run in about 5 minutes. The procedure has also proved very accurate and in this respect has the following advantages over the chemical analysis method. Since an excess of aluminum is not added there is little chance of some of the aluminum being consumed in deoxidizing slag entrained in the metal or surrounding the slagged spoon. All of the limited amount of aluminum combines first with the iron oxide in the molten metal whereas if an excess is used some of the excess tends to deoxidize any slag present and thus introduces an error. In the physical method the whole test ingot serves as the sample. In the chemical method, on the other hand, drillings must be used of limited portions of the test ingot and an error may enter in because of segregation of the aluminum oxide.

To add the aluminum wire to the test spoon at a constant rate and to record the exact amount added, a portable "aluminum feed apparatus" has been devised to practice this invention. In this apparatus a motor run by a battery (a clock work mechanism may also be used) unwinds aluminum wire from a spool and feeds the wire through a delivery tube into the spoon of molten metal until the "end point" is noted and the switch is turned off. A meter re-

records the inches of wire fed in each test. Referring to the drawings:

Fig. 1 is a plan view of the aluminum feed apparatus; Fig. 2 is a side view; and Fig. 3 is an end view. The apparatus is mounted on a suitable base. The motor 1 started by the switch 2, turns the motor shaft 3 on which is mounted the worm 4 which engages worm wheel 5 driving shaft 6. To this is attached a feed roll 7 which frictionally drives another feed roll 8. The aluminum wire is gripped between these rolls and is unwound from the spool 14, guided by guide 17, and fed through the delivery tube 15. To the shaft 6 is also attached a beveled gear 9 which drives another beveled gear 10 to which is attached the shaft 11 and indicator 12. As this turns it indicates the amount of wire fed on scale 13 which is attached to the base of the apparatus.

The determination is made as follows. A test spoon is thoroughly slagged in the furnace and a spoonful of molten metal is withdrawn. The metal in the spoon is skimmed of slag with a scraper and the aluminum wire is fed in by means of the apparatus until a film is observed to form over the surface. The meter records the number of inches of aluminum wire fed into the spoon. All the metal in the spoon is then poured into a special vertical type test-mold and the appearance of the top of the test is carefully observed.

If the test metal sinks during freezing without bleeding the test is considered over-killed. If it freezes over flat, but bleeds very slightly at the top forming a small bubble or two on the surface, it is considered as just right. If it rises in freezing and bleeds considerably it is under-killed. If the appearance of the solidified test does not indicate that the desired exact end point has been obtained the test is repeated. Usually one or two, or at the most three, tests are sufficient to obtain a satisfactory flat topped test. Fig. 4 illustrates the appearance of solidified test ingot *a* being under-killed, *b* killed to flat top, and *c* over-killed.

In heats of steel in which the residual carbon content at tap is over 0.11 per cent, casting the sample into vertical test molds may result in more piping than occurs in lower carbon steel. Estimation of the proper "end point" then becomes more difficult. This difficulty can be overcome by casting the deoxidized steel into a pancake shaped mold, so that the steel solidifies rapidly with no piping. The end point can then be more easily estimated from the appearance and number of gas bubbles forming on the top surface of the cast pancake. The appearance of such a cast pancake is illustrated in Fig. 6.

When the test is just right it is removed from the mold and measured or weighed. The weight of aluminum wire fed to produce the flat topped condition, divided by the weight of the test ingot, gives the desired "oxidation factor" which can be conveniently arrived at with a minimum of calculation by means of specially prepared tables. For example, for a certain "oxidation factor," adding 40 lbs. of aluminum to the ladle of a 100-ton rimming steel heat might be found to give satisfactory rimming action in the molds, whereas for the same "oxidation factor" on a similar heat adding 30 lbs. of aluminum to the ladle might be too little and 50 lbs. might be too much. With a higher "oxidation factor" it might be found by trial that adding 60 lbs. of

aluminum to the ladle will give a satisfactory rimming action for the same grade of steel, whereas 50 lbs. might be too little and 70 lbs. might be too much. The correlation between "oxidation factor" and the proper deoxidation addition is determined empirically by observing the behavior of a large number of heats; the correlation varies somewhat for different shops and of course for different grades of steel. The test is now completed. The entire test including the necessary calculations, takes less than 5 minutes.

The relation between the "oxidation factor" shown by the test and the necessary deoxidation treatment for the heat of steel is determined empirically by observing a large number of heats. Based on such data a deoxidation schedule is set up for the particular size heat, grade of steel, etc. In extended operations in large open hearth shops it has been found that deoxidation control based on the test described in this invention is more accurate and successful than control based on the old indirect method of calculating from the iron oxide content of the slag and the carbon and manganese content of the bath. As a rule the use of the new method is less expensive, as some of the chemical determinations required by the old method can be dispensed with.

In an attempt to approximate the end point for a flat topped test even more accurately if possible than can be done in the procedure described, the density of the test ingots was studied and a modification of the above described procedure worked out which is also comprised in this invention.

The density of test ingots can be rapidly determined by weighing with a spring balance for example, first by suspending the ingot in air thus getting the weight in air and then immersing the ingot in water and thus getting the weight in water. It was found that the densities of under-killed tests increased rapidly with small increments of added aluminum wire until the flat topped condition was reached when the density remained constant or fell off as piping occurred. Thus, the density of under-killed tests is extremely sensitive to variations in the amount of aluminum added. Moreover, in plotting the densities of a series of samples taken from any heat against the amount of aluminum wire added, series of parallel lines were obtained for the tests from different heats, except for those few heats in which pig iron was added to the molten steel within 20 minutes before taking the tests. In the few heats to which pig iron was added to the molten steel within 20 minutes before taking the tests, the slopes of the lines were irregular.

In the great majority of the heats, therefore, in which parallel lines are obtained it is readily possible to extrapolate very accurately the results of only 1 or 2 under-killed tests to the end point, i. e., to arrive at the amount of aluminum which would be required to produce a flat topped test. This is illustrated in Fig. 5. Knowing the amount of aluminum required to produce the determined relatively low density the amount of aluminum required to give the density 7.6 of a flat-topped test can be obtained by extrapolating along a line parallel to the heavy steeply sloping lines shown in Fig. 5. By such extrapolation of the density values of under-killed tests it may be possible to obtain a more accurate "end point" than can be obtained by observing the film formation.

Since under-killed tests can be used it also

becomes possible to weigh out the desired amount of aluminum beforehand and place this in the test mold itself instead of feeding the aluminum to the slagged spoon. This eliminates any possibility of error due to aluminum deoxidizing slag on the walls of the spoon. It also dispenses with the use of the aluminum feed apparatus. The density method as described above has been found very practical and accurate. It may be used either by itself, or as an auxiliary method in combination with the aluminum feed apparatus for determining the flat topped condition more accurately perhaps than can be done by observing the appearance of the solidified test.

It will be clear, therefore, that the desired degree of deoxidation of the sample is indicated by an "end point" which may be successfully determined by any one or a combination of all of the above described methods, that is by noting when incipient filming of the surface of the molten sample occurs, by noting that the appearance of the sample as solidified in a test mold is that of a flat topped test, and by determining the density of the deoxidized sample and extrapolating from this value to the amount of aluminum required to give a flat topped test.

Having thus described my invention what I claim as new and desire to secure by Letters Patent is:

1. A process of controlling the deoxidation of a heat of steel which comprises the steps of taking a small sample of the steel, adding deoxidizer to the sample until the sample has the flat top characteristic of semi-killed steel, thus determining the amount of deoxidizer required to bring the sample to the flat top state, comparing this amount of deoxidizer with the amounts of deoxidizer required for samples of previous heats of steel of varying degrees of oxidation to ascertain a previous heat having a similar degree of oxidation, and adding deoxidizer to the heat of steel in amount as indicated by such similar previous heat to give the desired degree of deoxidation.

2. A process of controlling the deoxidation of a heat of steel which comprises the steps of taking a small sample of the steel before ladle additions, adding aluminum to the sample until the sample has the flat top characteristic of semi-killed steel, thus determining the amount of aluminum required to bring the sample to the flat top state, comparing this amount of aluminum with the amounts of aluminum required for samples of previous heats of steel of varying degrees of oxidation to ascertain a previous heat having a similar degree of oxidation, and adding aluminum to the heat of steel in amount as indicated by such similar previous heat to give the desired degree of deoxidation.

3. A process of controlling the deoxidation of a heat of steel which comprises the steps of taking a small sample of the steel before ladle additions, adding a predetermined amount of deoxidizer to the sample, measuring the density of the sample and computing therefrom the amount of deoxidizer required to give a flat topped sample, comparing this amount of deoxidizer with the amounts of deoxidizer required for samples of previous heats of steel of varying degrees of oxidation to ascertain a previous heat having a similar degree of oxidation, and adding deoxidizer to the heat of steel in amount as indicated by such similar previous heat to give the desired degree of deoxidation.

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