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## METHOD OF ANALYSIS WITH RADIOACTIVE MATERIAL

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This invention relates to a method of determining the concentration of an element in a mass while the mass is subjected to a treatment wherein the concentration is changed. The invention has particular relation to a method of analysis of a metal during the refining thereof.

In the refining of certain metals, the metal is placed in a furnace and subjected to extremely high temperatures in the presence of certain agents whereby the concentration of various elements in the metal is reduced to a desired value. This procedure is followed in the production of steel in which phosphorus and carbon are the major elements the concentrations of which are to be controlled. At various times during the refining process, samples of the steel are analyzed to determine the percentages of carbon and phosphorus therein. The analysis is then furnished to the furnace operator so that he may take proper steps for controlling the composition of the finished steel, as well as reducing the overall time required for the refining. It is highly desirable that the results of the analysis be made available to the operator very quickly after the taking of the sample. The quicker the information is received, the closer the operator can maintain conditions to the point of maximum efficiency in producing a steel of the desired composition.

Various methods have been devised for determining the percentage of carbon present in the steel, and some of these methods permit a rapid analysis to be made. However, the phosphorus content of the steel is customarily determined at various times during the refining by the regular chemical method which requires approximately one and one-half hours for each analysis. The time required for completion of the analysis is not of such tremendous importance at the beginning of the refining process, although it is obvious that a saving of time at any point increases the efficiency of operation. But after the refining has been in progress for a while, the detrimental effects of a long period of time between the taking of a sample and the completion of the analysis become more noticeable. Changes are taking place more rapidly and a critical point may pass while the operator is waiting for the results of the analysis. This is especially true if it is desired to produce a steel the composition of which is specified within close limits.

The necessity for speed in making an analysis is also apparent in the production of certain other alloys or in the production of any material where chemical concentrations are to be carefully controlled.

It is accordingly an object of my invention to provide a method for rapidly determining the concentration of an element in a mass while the mass is subjected to treatment wherein the concentration is reduced.

A further object of my invention is to provide a method for rapidly and inexpensively analyzing the concentration of an element in a metal at various times during the refining thereof.

Another object of my invention is to provide an accurate method for rapidly determining the concentration of phosphorus in steel at various times during the refining thereof.

The novel features that I consider characteristic of my invention are set forth with particularity in the appended claims. The invention itself, however, together with additional objects and advantages thereof will best be understood from the following description of a specific application of the invention.

My invention may be advantageously employed in the determination of the phosphorus content of steel at various times during the refining of the steel in an open-hearth furnace. In accordance with my invention, a small quantity of radioactive phosphorus is added to the charge of the furnace. To prevent any substantial loss of the radioactive phosphorus prior to its reduction by the usual operation of the open-hearth process, the radioactive phosphorus may be enclosed in a tightly sealed steel chamber. By the time the chamber melts, the charge is in a molten state and the radioactive phosphorus is thoroughly mixed therewith. Of course other methods of introducing the radioactive phosphorus may be used. Thereafter the amount of radioactive phosphorus in the steel is reduced in direct proportion to the reduction of the total phosphorus content.

A sample of the steel of a known or determinable quantity is drawn off and its radioactivity is measured with any suitable device, such as a Geiger-Mueller counter, an ionization chamber, or an electroscope. The same sample or one drawn at the same time is subjected to a chemical analysis to determine the percentage of phosphorus present in the steel. Thus the relationship between the radioactivity of the steel and the concentration of the phosphorus may be established. Thereafter samples of an equal quantity are drawn off at the various desired times. The radioactivity of each sample is measured. Then, as the relationship between the radioactivity of the steel and the concentration of the phosphorus is known, the percentage of phosphorus at the time of taking the sample may be derived.

By experiment, it has been discovered that approximately 0.15 millicuries of radioactive phosphorus per ton of steel is sufficient to give a counting rate of ten counts per second, using a Geiger-Mueller counter with a 1⅛ inch sensitive tube length. Under such circumstances, measurements for only a two-minute period enables a determination of the amount of phosphorus present within 3% of the phosphorus regardless of the amount of phosphorus per unit of steel. In addition, the accuracy can be further increased by increasing the amount of radioactive material used and/or by increasing the length of the measurement period. The accuracy thus available through the radioactive method compares favorably with that obtainable by the regular chemical method. Since the accuracy of the chemical method varies with the amount of phosphorus per unit of steel and that of the radioactive method does not, the latter is particularly advantageous when the percentage of phosphorus is low.

The great advantage of the radioactive method over the regular chemical method is that the concentration of phosphorus in the steel can be followed more closely because of the short time required for analysis. As the time required to measure the radioactivity of each sample is approximately three to four minutes after the sample has been prepared, one man can easily measure a sample every hour from each of ten or twelve furnaces. The time to prepare the sample is not greater than that to prepare a sample for the chemist. Therefore, the measurement time of three to four minutes for the radioactive method is to be compared with one and one-half hours for regular chemical analysis. As a result, the furnace operators are supplied with information which is sufficiently timely to enable them to increase the efficiency of the refining process. In addition to the saving in expenses afforded by an increase in the efficiency of the furnace operation, there is also a saving in the cost of making the necessary analysis as fewer chemists ae needed than if the analysis were made by the chemical method alone.

It is apparent that the advantages to be derived from the use of this method of analysis are not confined to the determination of the phosphorus content in producing open-hearth steel. It is well known that the production of other metals, alloys and other materials require careful control of the chemical concentration of a particular element. If that element can be made radioactive in a form such that its reduction will be directly proportional to the reduction of the main body of the element, this method of analysis may be employed to advantage. Further, if a suitable radioactive isotope of the particular element to be followed is not available, a radioactive isotope of another element may be used in the practice of this method providing the chemical behavior of the other element is such that there would be a definite known relationship in the change in concentration of the other element and the element to be followed.

Although I have shown and described a specific application of my invention, I am fully aware that many other applications thereof are possible. My invention, therefore, is not to be restricted except insofar as is necessitated by the prior art and by the spirit of the appended claims.

I claim as my invention:

1. The method of determining the concentration of an element in a mass at various times while the mass is subjected to a treatment which tends to change the concentration of said element in said mass, a portion only of said element in the mass being radioactive, which comprises determining the relationship between radioactivity of the mass and concentration of the element and subsequently measuring the radioactivity of the mass at said various times whereby the concentration at each of said times may be derived from said relationship.

2. The method of determining the concentration of an element in a mass at various times while the mass is subjected to a treatment which tends to change the chemical equilibrium between said element and other components of said mass, a portion only of said element in the mass being radioactive, which comprises measuring the radioactivity of the mass and determining by chemical analysis the concentration of the element in the mass as composed when the radioactivity is measured to establish the relationship between radioactivity of the mass and concentration of the element, and subsequently measuring the radioactivity of the mass at said various times whereby the concentration at each of said times may be derived from said relationship.

3. The method of determining the concentration of an element in a mass at various times while the mass is subjected to a treatment at high temperatures wherein the concentration is changed, a portion of said element in the mass being radioactive and the concentration of the element being known prior to said treatment, which comprises measuring the radioactivity of the mass prior to said treatment to establish the relationship between radioactivity of the mass and concentration of the element and subsequently measuring the radioactivity of the mass at said various times, whereby the concentration at each of said times may be derived from said relationship.

4. The method of determining the concentration of an element in a normally non-radioactive mass at various times while the mass is subjected to a treatment wherein the concentration is reduced, which comprises mixing a small quantity of the same element which is radioactive in the mass in a form such that the concentration of the radioactive portion is decreased in direct proportion to the decrease in concentration of the rest of the element, determining the relationship between radioactivity of the mass and concentration of the element and subsequently measuring the radioactivity of the mass at said various times whereby the concentration at each of said times may be derived from said relationship.

5. The method of determining the concentration of an element in a normally non-radioactive mass at various times while the mass is subjected to treatment wherein the concentration is reduced, which comprises mixing a small quantity of a radioactive material in the mass having such a chemical behavior that the concentration of the material is reduced in a definite relationship to the reduction in concentration of said element, determining the relationship between radioactivity of the mass and concentration of said element and subsequently measuring the radio-

activity of the mass at said various times whereby the concentration at each of said times may be derived from said two relationships.

6. The method of determining the concentration of an element in a normally non-radioactive mass at various times while the mass is subjected to a treatment wherein the concentration is reduced, which comprises mixing a small quantity of the same element which is radioactive in the mass in a form such that the concentration of the radioactive portion is decreased in direct proportion to the decrease in concentration of the rest of the element, then measuring the radioactivity of the mass and determining by chemical analysis the concentration of said element in the mass as composed when the radioactivity is measured to establish the relationship between radioactivity of the mass and concentration of the element, and subsequently measuring the radioactivity of the mass at said various times, whereby the concentration at each of said times may be derived from said relationship.

7. The method of determining the concentration of an element in a metal at various times during the refining thereof wherein the concentration is reduced, the metal being in a molten state with a portion of said element being radioactive, which comprises determining the relationship between radioactivity of the metal and concentration of the element and subsequently measuring the radioactivity of the metal at said various times, whereby the concentration at each of said times may be derived from said relationship.

8. The method of determining the concentration of an element in a metal at various times during the refining thereof wherein the concentration is reduced, the metal being in a molten state with a portion of said element being radioactive, which comprises measuring the radioactivity of the metal and determining by chemical analysis the concentration of the element in the metal as composed when the radioactivity is measured to establish the relationship between radioactivity of the metal and concentration of the element, and subsequently measuring the radioactivity of the metal at said various times whereby the concentration at each of said times may be derived from said relationship.

9. The method of determining the concentration of phosphorus in steel at various times during the refining thereof wherein the concentration is reduced, the steel being in a molten state with a portion of said phosphorus being radioactive, which comprises determining the relationship between radioactivity of the steel and concentration of the phosphorus and subsequently measuring the radioactivity of the steel at said various times, whereby the concentration at each of said times may be derived from said relationship.

10. The method of determining the concentration of phosphorus in steel at various times during the refining thereof wherein the concentration is reduced, the steel being in a molten state with a portion of said phosphorus being radioactive, which comprises measuring the radioactivity of the steel and determining by chemical analysis the concentration of the phosphorus in the steel as composed when the radioactivity is measured to establish the relationship between radioactivity of the metal and concentration of the phosphorus, and subsequently measuring the radioactivity of the steel at said various times, whereby the concentration at each of said times may be derived from said relationship.

11. The method of determining the concentration of phosphorus in steel at various times during the refining thereof by the open-hearth process, which comprises adding a small quantity of radioactive phosphorus to the steel, then measuring the radiactivity of the steel and determining by chemical analysis the concentration of phosphorus in the steel as composed when the radioactivity is measured to establish the relationship between radioactivity of the steel and concentration of the phosphorus, and subsequently maesuring the radioactivity of the steel at said various times, whereby the concentration at each of said times may be derived from said relationship.

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