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PREPARATION OF LOW-CARBON-CONTENT IRON FOR USE AS AN ACCELERATOR FOR CARBON DETERMINATIONS

by Emery J. Merkle and Judson W. Graab

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SUMMARY

Accelerator material for induction-heating determination of carbon content in materials containing less than 50 ppm was prepared by utilizing the decarburizing effect of moist hydrogen to purify a commercial iron accelerator. The material has a low and uniform carbon content, which makes it especially useful for determinations of this type.

INTRODUCTION

The determination of carbon by induction heating requires a material that will couple with the field of the furnace. An accelerator may be required when the physical nature of the sample, its limited quantity, or the particle size prevents direct coupling. Iron is one of the more common accelerators used for carbon determinations. However, the usefulness of iron is limited in some instances by its relatively high and variable carbon content. When a material having a carbon content below 50 ppm is analyzed, the carbon contained in 1 gram of accelerator may equal, or even greatly exceed, that of the sample. Moreover, the variation in carbon content in the accelerator makes precise determinations of carbon at this level virtually impossible. The obvious solution to the problem is to use an accelerator that has as low and as consistent a level of carbon as is obtainable. The decarburizing effect of moist hydrogen is recognized (ref. 1). The removal of carbon is based on the following reactions at elevated temperature:

$$(C) + 2H_2 \rightleftarrows CH_4 \qquad (1)$$

$$(C) + H_2O \rightleftarrows CO + H_2 \qquad (2)$$

In the reactions, (C) represents the carbon dissolved in iron.

An electrolytic iron reduced in moist hydrogen is commercially available as a reference grade material from the G. Frederick Smith Chemical Company. Because of its low carbon content, this material was tested as an accelerator.

It did not always couple effectively. This was attributed to the relatively large and uneven size of the particles.

This study demonstrates the ease with which an iron accelerator of low, uniform carbon content may be prepared. Accelerator material prepared in this manner is preferred for determining carbon content below 50 ppm. Its use is advocated for all refractory metals and refractory metal alloy systems.

EQUIPMENT

The following apparatus and reagents were used in this experiment: an iron chip accelerator, an induction furnace, a low-carbon analyzer, a tube furnace, a gas-washing bottle, commercial hydrogen, and a 5- by 90-centimeter Vycor tube fitted with standard taper glass adapter for tubing connections. The iron chip accelerator had a parts number 501-77, the induction furnace was model number 521, and the low carbon analyzer was model number 589-400 (Laboratory Equipment Company). The tube furnace was type MU-2013 (Hevi Duty).

PROCEDURE

A total of 400 grams of iron accelerator were screened on a 20-mesh sieve. The fines were discarded. Approximately 100 grams of the material retained on the sieve before purification were reserved for the determination of the carbon level. The balance of the material was loaded into the Vycor tube and distributed evenly in the heated portion of the furnace. As a safety precaution, the tube was purged with an inert gas before hydrogen was admitted. Hydrogen was then slowly passed through a gas-washing bottle containing water and then into the Vycor tube. The furnace was heated to 590° C for 3 hours and then cooled to room temperature while the flow of hydrogen was maintained. The inert gas was again used to displace the hydrogen before the treated material was removed. A second bottle of iron accelerator was treated as described, and a second treatment was performed the following day.

The carbon level of the iron, before and after treatment, was checked by burning 1-gram samples in the induction furnace for 2 minutes. The combustion products were determined with a chromatographic carbon analyzer. The carbon content of the iron was calculated by using a previously prepared calibration curve. The results of these tests are shown in table I. Each value represents a total of 10 replicate determinations run over a period of 2 days.

DISCUSSION OF RESULTS

The removal of carbon from iron by hydrogen treatment is considered to be a surface effect. Therefore, this procedure should be most effective with an iron that has a large surface-to-volume ratio. The accelerator used here consisted of flat plate-like particles. Table I shows that the carbon content of the iron has been reduced to less than 25 percent of its original value. The standard deviation column also demonstrates that the treated material has a

TABLE I. - CARBON CONTENT OF 1 GRAM OF IRON ACCELERATOR

BEFORE AND AFTER TREATMENT AT 590° C

Bottle	Treatment time, hr	Total carbon, μg	Standard deviation S,[a] μg carbon	Detection limit per gram sample (range of 3S), ppm
1	0	37.8	4.1	12.3
	3	8.5	1.2	3.6
2	0	37.5	2.7	8.1
	6	8.8	1.0	3.0

[a] $S = \sqrt{\frac{\sum d^2}{n - 1}}$

more uniform carbon content.

It is this greater uniformity, combined with the lower level of carbon, that makes the treated iron especially suitable as an accelerator for carbon determinations well below 50 ppm. The limit of detection was chosen as three times the standard deviation and represents the 99.7-percent confidence level. These data, shown in the last column of the table, demonstrate the superiority of the treated accelerator for use with material containing very low carbon.

The data for the second bottle of accelerator, which was treated twice, indicate that the second 3-hour treatment did not further reduce the carbon level. Further lowering of the carbon content may require a treatment that can penetrate well below the surface of the iron chips.

During reduction, the temperature should not exceed 590° C; otherwise sintering of the particles will occur. The results show that sufficiently large amounts of material, at least 300 grams, can be treated at one time without serious segregation problems.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 24, 1965.

REFERENCE

1. Lyman, Taylor, ed.: Metals Handbook. Vol. II. Eighth ed., ASM, c. 1964, pp. 67-68.