Improvements in Laboratory Testing of DR Oxides at Midrex Technologies, Inc.

By Jeffrey Myers, Manager
Technical Center Operations
Midrex Technologies, Inc.

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INTRODUCTION

There are three main areas of concern in testing of DR ores: low temperature disintegration, reducibility and clustering. Midrex Technologies, Inc. (MTI) has historically used two tests (Hot Load and Linder) to evaluate these parameters.

The Hot Load and Linder tests have been in use at MTI and by its clients for over 35 years. An extensive historical database has been developed. The Linder test (Figure 1) is designed primarily for low temperature behavior, and is essentially similar to ISO 11257.

The Hot Load (clustering and reducibility) test (shown in Figures 2 and 3 on the following page) is run at 816° C, which was appropriate for DR operations for many years. Likewise, the ISO test for clustering (ISO 11256) is run at 850° C, and the reducibility test (ISO 11258) is performed at 800° C, all in the same general range.

Midrex Technology Center Wet Lab

Figure 1 - Linder Test for Low Temperature Disintegration

Figure 2 - Hot Load Test for Clustering and Reducibility

Figure 3 - Hot Load Test Showing Weights for Load

Improvements in coating technology, among other developments, have resulted in significant increases in operating temperatures at many MIDREX® Plants. As a consequence, there is some concern that the results from lower temperature tests may not necessarily be relevant to higher temperature operation. In response to this concern, a study was initiated at MTI to perform a series of higher temperature tests in an effort to define a correlation to actual plant performance.

IMPROVEMENTS IN LABORATORY EQUIPMENT

In order to assure the most consistent and reliable operation of the Hot Load, the control system was upgraded. In addition, data that previously had been manually recorded was directed to an automatic data acquisition system.

New test equipment corresponding to the ISO test procedures is being acquired to provide additional correlations with standardized testing.

COATING PROCEDURE FOR LABORATORY TESTING

The standard HL test uses pellets as supplied, so only pellets with coatings from suppliers can be tested easily. Since many producers add additional coating on site, it is useful to duplicate this in the lab so that the tested pellets more accurately reflect the actual characteristics of the pellets used in the plants.

A series of tests were performed to develop a reliable procedure for applying coatings to pellets on a lab scale. Application rates should be comparable to those in the plant (0.5 to 8 kg/tonne oxide), but the amount of pellets being coated in the lab is only 20-50 kg. Therefore, the amount of coating to be added to any one batch is only around 10 to 400 g, creating the challenge of making sure an even distribution of the coating is achieved on a reliably repeatable (and measurable) basis.

The approach used was to first mix the coating material (e.g., lime) with water to make a slurry, and then use the slurry to coat the pellets. This is similar to how coatings are applied in the commercial process. By weighing the pellets before and after coating, along with the weights of the coating and water, the net amount of coating material on the pellets can be calculated.

CORRELATION OF LABORATORY RESULTS WITH PLANT DATA

The next step was to collect samples and operating data from an operating plant to establish the correlation of laboratory results to actual data. This required collection of the following:

- Samples of oxide pellets, before and after coating (if possible)
- Samples of DRI from the corresponding time period
- Operating data from the corresponding time period

The first series of tests were conducted on uncoated pellets at various temperatures to examine the link between temperature and reducibility. Further testing was performed at test conditions that resulted in DRI characteristics that most closely matched those in the plant.

Coatings were then applied in the laboratory to match the coating amount applied in the plant. If coated pellets from the plant were available, they also were tested. The next step was to test the coated pellets in the laboratory and compare them to plant performance.

It is reasonable to assume that the results of standard lab testing are an indication of the performance of an oxide in a commercial furnace. Nevertheless, establishment of a reliable correlation to data obtained under carefully controlled conditions is an important step in verifying the utility of the test procedure in evaluation of the material.

IMPROVEMENTS IN ANALYTICAL EQUIPMENT

Iron totals and metallic irons have always been performed in-house by potassium dichromate titration. Improved quality assurance techniques, including triplicate analyses, scheduled check standards and automation of data storage and retrieval, have been instituted. New automatic titrators allow addition of the titrant with accuracies of down to 0.05 ml. All analytical procedures have been formalized and documented.

The procedure for metallic iron has been modified from the ISO procedure to improve turnaround time and safety; however, the calculation of metallization is functionally equivalent in both methods.

Improvements in C and S determination have been made through equipment upgrade (new LECO CS-230), improved operator training, triplicate analyses and review before release and improved calibration techniques.

Hot Load and Linder test results have historically been limited to Fe total, metallization, C, and S. Analysis of other oxides was performed at an outside lab and conducted only on the oxide, not on the product.

An ion coupled plasma (ICP) optical spectrometer was installed in 2007. The ICP (Figure 4) is now used for analysis of several elements on a standard basis for every sample. This expanded capability allows for more detailed reporting of the results and provides information about oxide chemistries, alkalis, volatile elements, such as zinc and lead, and residual metals.

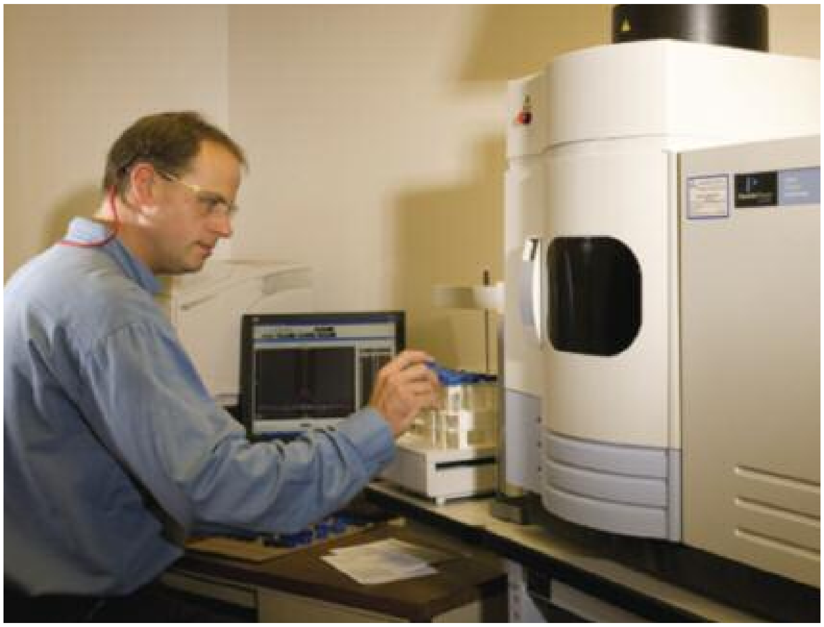

Figure 4 - ICP for Elemental Analysis

Sample preparation procedures are critical for the ICP. Extensive development went into creating the optimal combination of acid dissolution with microwave treatment (Figure 5)

Figure 5 - Microwave Digestion for the ICP

In June 2008, we added a combination X-ray fluorescence/ X-ray diffraction (XRF/XRD) instrument. This will provide independent confirmation of the elemental analyses performed by the ICP. More importantly, it will provide the opportunity to define the nature of the compounds present in the ores and allow detailed characterization of the mineralogy. Test programs are envisioned in several areas:

First, it is likely that previously unexplained variations and differences in oxide behavior (e.g. reducibility, rate of carbon pickup and strength) may be related to differences in mineralogy. The goal is to determine which mineralogical characteristics are most relevant to these parameters. This is a complex task that will require extensive investigation and analysis.

Second, we should be able to use the elemental analyses, along with the compound data, to “fingerprint” specific ore sources. This would allow identification of the source of an unknown iron ore once a sufficient data base has been established.

CONCLUSION

A major program to improve equipment and test procedures has been undertaken at the Midrex Technologies, Inc. Research and Development Technology Center. Improvements have been made in both laboratory equipment and procedures. Additional improvements have been made in the areas of furnaces and equipment for testing reducibility and clustering, including the capability to perform standard ISO testing, along with other test procedures developed by MTI. Analytical capabilities have been strengthened by the addition of new titration equipment, new carbon/sulfur analyzer, new microwave digester and ICP and new XRF/XRD.