Application note

Analyzing the metallization of DRI for process optimization using ARL X'TRA Companion X-ray Diffractometer

Authors

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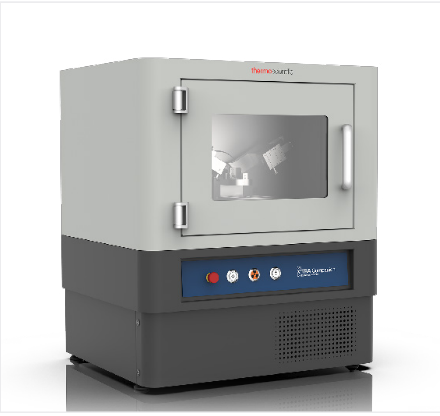


Figure 1. ARL X'TRA Companion X-ray diffraction system.

Introduction

Direct Reduced Iron (DRI) is a crucial material in the steelmaking industry, produced by reducing iron ore in its solid state using natural gas, or increasingly, hydrogen (H_2). This process is favored for its lower carbon footprint compared to traditional blast furnace methods, aligning with global climate change initiatives. Stringent regulations with ambitious targets for reducing greenhouse gas emissions are driving the demand for "Green Steel." The adoption of DRI is pivotal for industries aiming to meet these regulatory requirements and contribute to sustainable development. Utilizing hydrogen as a reducing agent further enhances the environmental benefits, as it results in water vapor rather than carbon dioxide as a byproduct.

The commercial impact of direct reduced iron (DRI) is substantial, particularly within the context of the broader $1.55 trillion global steel market. While the DRI market was valued at approximately $115 billion in 2021, it is expected to grow at a CAGR of around 9% till 2032. This growth outpaces the steel market's projected CAGR of 5%, driven by increasing demand from sectors such as construction, automotive, and infrastructure. The adoption of DRI offers a cost-effective and environmentally friendly alternative, driving innovation and competitiveness within the steel sector.

X-ray diffraction (XRD) is an analytical technique employed to characterize the crystalline structure of DRI. One of the key quality parameters assessed by XRD is metallization, which refers to the percentage of metallic iron present in the DRI. High metallization levels indicate a more efficient reduction process and higher quality product. XRD provides detailed insights into phase composition and purity, essential for quality control and optimizing production processes, ensuring the final product meets stringent industry standards. Through accurate measurement of metallization, manufacturers can enhance the process performance to increase efficiency and reduce costs.

Instrument & software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available on demand. Thanks to the innovative solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion XRD provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (laboratory information management system).

Experimental

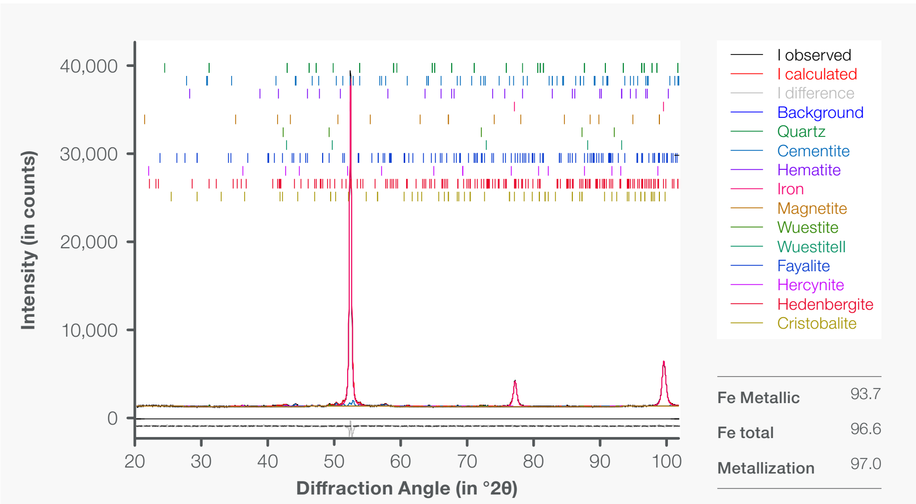
A set of six DRI samples with reference values from X-ray fluorescence spectroscopy (XRF) combined with wet chemical analysis was selected. The samples cover a representative concentration range from low to high metallization. The samples were manually pressed in top loading sample cups and measurements (10 minutes) in reflection mode were performed using Co Kα (1.79030 Å) radiation with sample spinning. (c.f. Figure 2). A Rietveld refinement using Profex software was carried out, including crystalline silicate and aluminate phases which contribute to the gangue. $Fe_{metallic}$ is calculated from the iron quantities of Fe and Cementite while Fe_{total} shows the total amount of Fe in all phases. The metallization is calculated as the ratio of $Fe_{metallic}$ out of Fe_{total}.

Results & Discussion

$Fe_{metallic}$, Fe_{total}, and metallization were calculated from the quantitative refinement (e.g. Table 1). The quantities show good linear correlation with reference values (Figure 2). Deviations are most likely due to the partially amorphous gangue with its quantity not being accessible in the XRD analysis. The SEE (standard error of estimate) of the Metallization is calculated as 2.53.

Your Benefits

The ARL X'TRA Companion XRD yields data perfectly suited to analyze DRI samples. Utilizing **one-click Rietveld refinement** for full quantification and calculation of process parameters in **seconds**, enables **ease of use** for operators and **reliefs training constraints**. Tracking of $Fe_{metallic}$, Fe_{total}, and metallization allows constant **process optimization and assessment of the metallization stage** which **saves cost**.



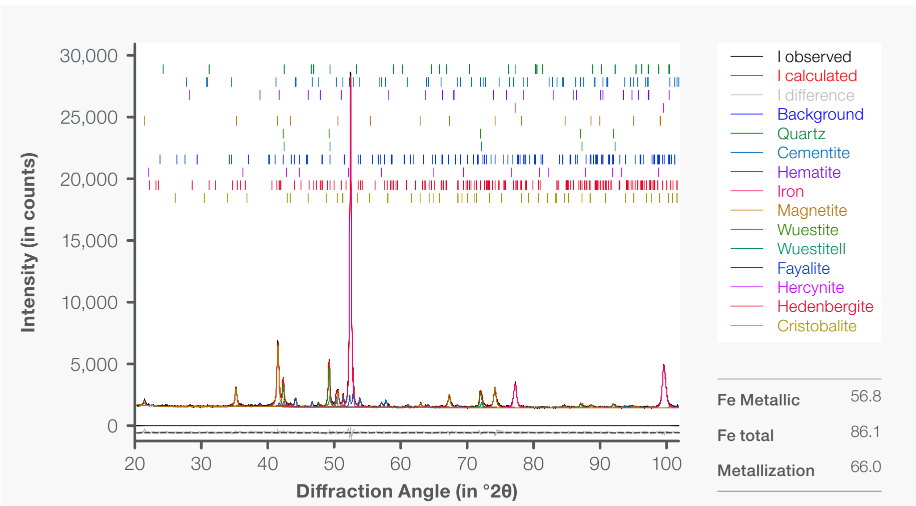
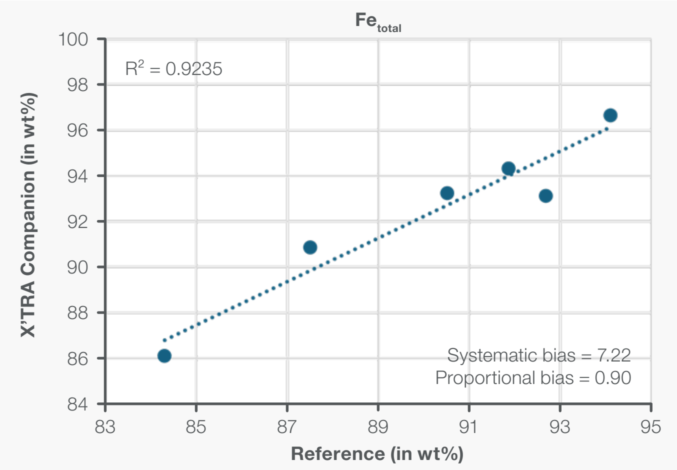
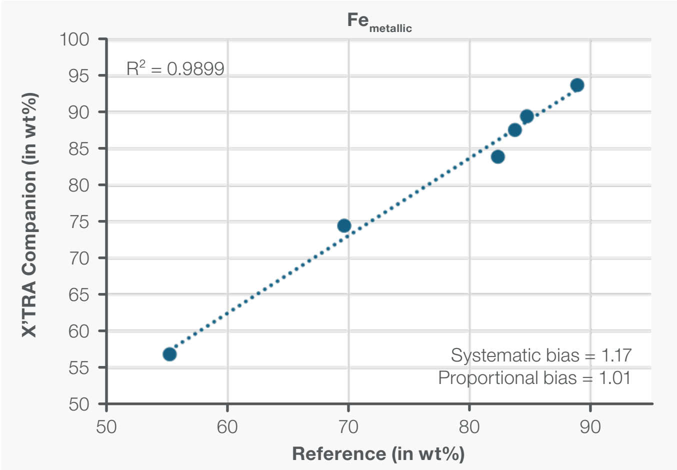


Figure 2. XRD patterns of two DRI samples (Sample 1 left; Sample 6 right); Values for $Fe_{metallic}$, Fe_{total}, and metallization are shown.





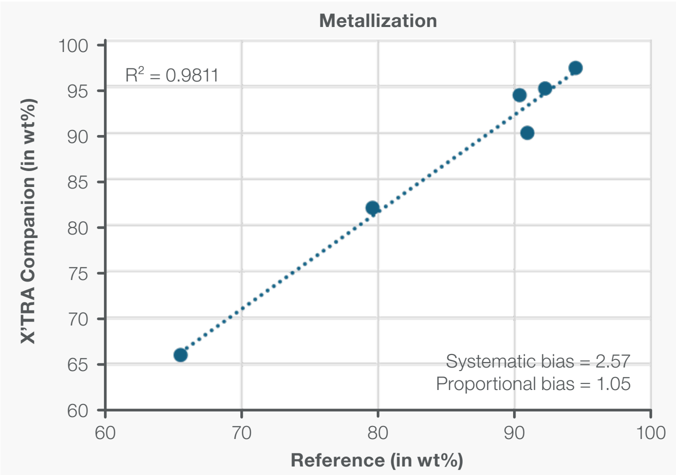


Figure 3. Linear correlation between the reference and values determined by XRD of $Fe_{metallic}$, Fe_{total}, and metallization. R^2 values, systematic, and proportional bias are shown in the graphs.

Quantity (in wt %)	Quartz SiO_2	Cementite Fe_3C	Hematite F_2O_3	Iron Fe	Magnetite Fe_3O_4	Wustite FeO	Wustite II $Fe_{0.925}O$
Sample 1	0.3	7.3	0.3	86.9	0.5	1.2	0.5
Sample 2	0.9	23.2	0.3	67.8	0.9	2.8	1.5
Sample 3	0.5	24.9	0.4	64.4	1.0	0.0	4.6
Sample 4	0.7	10.0	1.1	74.6	2.6	5.0	1.8
Sample 5	0.1	4.2	6.6	70.5	10.6	3.6	0.0
Sample 6	0.2	10.2	1.9	47.3	21.3	10.3	4.8

Quantity (in wt %)	Fayalite Fe_2SiO_4	Hercynite $FeAl_2O_4$	Hedenbergite $CaFeSi_2O_6$	Cristobalite SiO_2	Fe_{total} (Reference)	$Fe_{metallic}$ (Reference)	Metallisation (Reference)
Sample 1	0.6	1.2	1.1	0.1	96.6 (94.1)	93.7 (88.9)	97.0 (94.5)
Sample 2	0.3	0.6	1.6	0.0	94.3 (91.9)	89.4 (84.7)	94.8 (92.2)
Sample 3	1.1	1.2	1.9	0.0	93.1 (92.7)	87.6 (83.8)	94.1 (90.4)
Sample 4	1.3	1.2	1.8	0.0	93.2 (90.5)	83.9 (82.3)	90.0 (90.9)
Sample 5	0.7	1.5	2.0	0.3	90.9 (87.5)	74.4 (69.7)	81.9 (79.6)
Sample 6	1.1	1.1	1.9	0.0	86.1 (84.3)	56.8 (55.2)	66.0 (65.5)

Table 1. Results of Rietveld refinement of six DRI samples including $Fe_{metallic}$, Fe_{total}, and metallization values.

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