Analysis of Materials for Production and From Production

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Abstract

The metal plant will have major and minor process streams of materials. Materials purchased for the production are well characterized, with a certificate from the supplier, and there will be an onsite laboratory for critical quality checks - and underneath all this is an extensive support structure and knowledge base including instrument suppliers, ISO standards and suppliers of Reference Materials.

But for some minor process streams, some recurring waste, and incidental unknown materials this support structure might be not-so-helpful, or maybe non-existing. Hydro Aluminium has addressed this issue. Starting with experience from our onsite analysis of materials for production, principally quality control, Hydro Aluminium has established a practical approach to what we call Complex Analysis. Some of the methodology will be described and examples will be given that include random dust, new and used refractory, new and used cell materials, and electrolyte bath mixed in with other material in several forms.

Complex Analysis uses smart sampling, imagery and XRD phase analysis supported with XRF to understand the material. The XRF matrix modelling is supported with combustion analysis, electrochemical titration and XRD to include the light elements. Risk evaluation and HSE concerns are addressed in all these steps.

Hydro Aluminium has established an effective operational procedure for handling a wide and increasing range of materials from production. And an added benefit is that when materials begin to be well characterized, they can be established as internal Reference Materials, and used when building calibrations.

Keywords: Aluminium reduction technology, Material characterization, X-ray analysis, Spent production materials, Waste to value.

1. Introduction

1.1 Safety

Safety precautions must be respected such as wearing gloves, eye protection, breathing mask and handling in a ducted fume hood when required.

This type of analysis work will see many and different types of samples; there will be fine powders and there will be dusting. Some will have fluorides or adsorbed gases including HF(ad) and sulfur oxide $SO_3(g)$ on the surface.

1.2 Key Analysis Methods

The characterization of materials is much enhanced by XRD phase analysis, especially when using Rietveld modelling for quantitative determination. The methodology has evolved around this, and the flowchart illustrate steps that usually give a good analysis. As a first step, it is helpful to have the approximate XRF composition, the raw data, to enable early start of the XRD phase determination. It is normally known where the material is from, and a probable composition, and an XRD scan will confirm and clarify further.

XRF - Elements	Omnian - elements F to U
Raw Data	XRF without supporting input
	★
XRD Phases	With major elements, start
Search & Match	HighScore Search & Match
Usually can start with	Assemble Phase Set
existing Phase Set	ICDD PDF5+
Finalizing XRF Eleme	ntal Matrix Model
Finalizing XRF Eleme	Leco-C
Finalizing XRF Elemen Supporting analysis into XRF Model	Leco-C ← Leco-N&O Titration E
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Finalizing XRF Element Supporting analysis into XRF Model Helps build better element-matrix mod Repeated to Build th XRD Quantitative wi	tal Matrix Model Leco-C ↓ Leco-N&O Titration-F Select Balance els XRD elements XRD-N Search e Phase Set th Riqas and HighScore

Figure 1. Flowsheet: The yellow boxes are the steps and the text next to it gives relevant information. "Favourite Flow" is the learning step, in "Finalizing XRF" the important XRF model is developed and the work finishes with the XRD model.

Sometimes a good starting XRD Phase Set might already exist, and just some small Search & Match based on the XRF elements will be used to enhance the Set.

The supporting analyses are a powerful tool when integrated into the XRF model. C, N, O or F, and more, can be added as a Known into the matrix model, at the known, fixed value.

Another powerful tool is to set one of the major components as "Balance". E.g. for spent lining, C-Balance is logical as C is major, and is not quantified by the XRF analysis itself. But as Balance, C is actively integrated in the element matrix.



documenting well with photos, and a log of actions and results. In our system we can go back more than 5 years, to compare with a current material and handling, this is very helpful.

Figure 8. Improvement XRD - better counting statistics with better array detector. In lower pane old scan is on right Y2 axis, illustrating count difference.

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10. References

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