AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

ED-XRF 145

SPECTRO XEPOS (XEP05)

Analysis of Ferro Alloys Prepared as Fused Beads

Introduction

X-ray fluorescence is a widely used technique for analyzing ferro alloys. For a fast sorting, samples can be prepared as powders in cups. Pressed pellets are chosen as sample preparation when higher accuracy is required but special calibrations are neccessary, especially if the grain size of the sample differs from available reference samples.

To avoid the influence of different grain sizes, the samples can also be prepared as fused beads. When preparing fused beads, the sample material must be oxidized to avoid damage of the platinum crucible. Standard operation procedures describe the process and list the required chemicals. With that approach even different types of ferro alloys can be analyzed with one method.

The SPECTRO XEPOS can be offered as a package of:

- Instrument
- Fusion technique
- Calibration samples
- Validation samples
- Consumables
- Installation
- Calibration

Sample Preparation

The samples were prepared as fused beads by the company Fluxana according to a predefined sample preparation procedure.

Instrumentation

The samples were excited for 240 s by an air cooled low-power thick binary Pd/Co alloy anode end window X-ray tube combined with an innovative monochromator which also polarizes the primary tube spectrum.

The illustration 1 shows the schematic setup of this excitation principle. The main benefit for this monochromatic excitation is the boost in sensitivity for the analysis of the light elements like Al and Si.

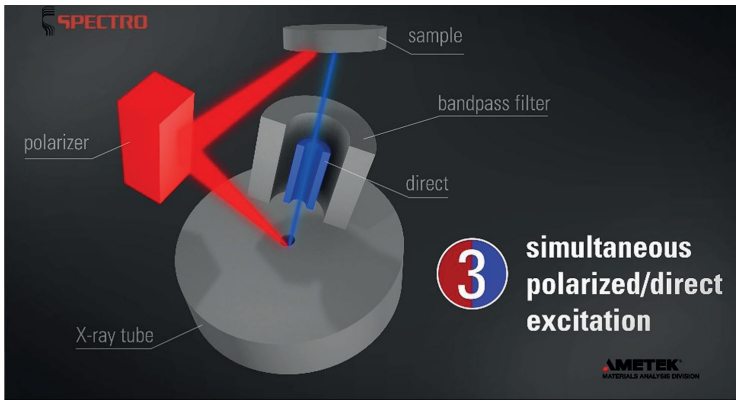

Illustration 1: Schematics of the combined direct/polarized excitation

A high resolution SDD was used to record the fluorescence radiation from the sample. The resolution for the SDD was ≤130 eV for the Mn-Kα-line.

The measurements were done in vacuum. The components are packaged in a compact bench top housing with a small footprint. All measurement parameters are controlled by a PC.

Calibration and Validation

For calibration a set of commercially available fused beads from Fluxana was used, which also includes several validation samples. For this application report, reference samples for FeSi, FeMn, FeMo, FeW and FeSiMn were used in one calibration using empirical matrix corrections.

The following graphs show as examples the correlation for the analysis of Si, Mn, Mo, and W in the various samples.

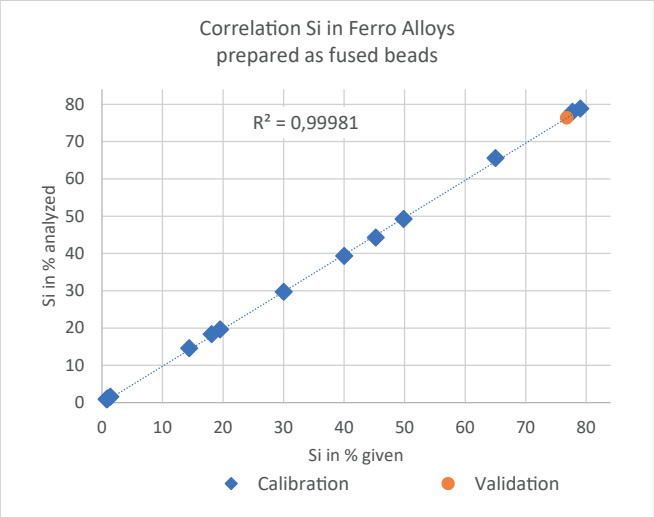

Figure 1: Correlation plot for Si

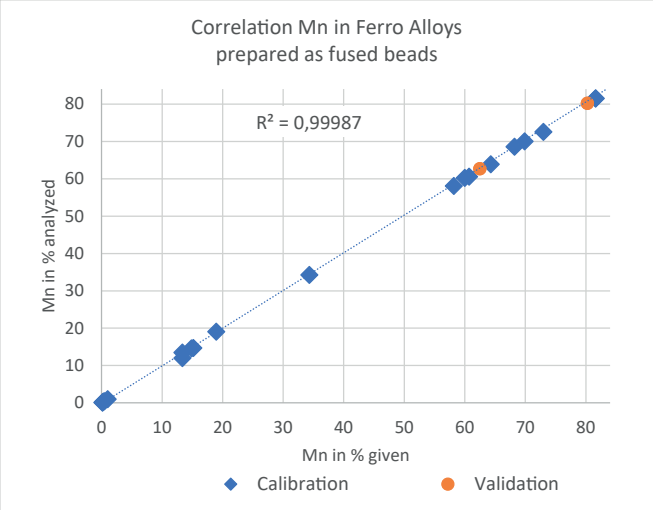

Figure 2: Correlation plot for Mn

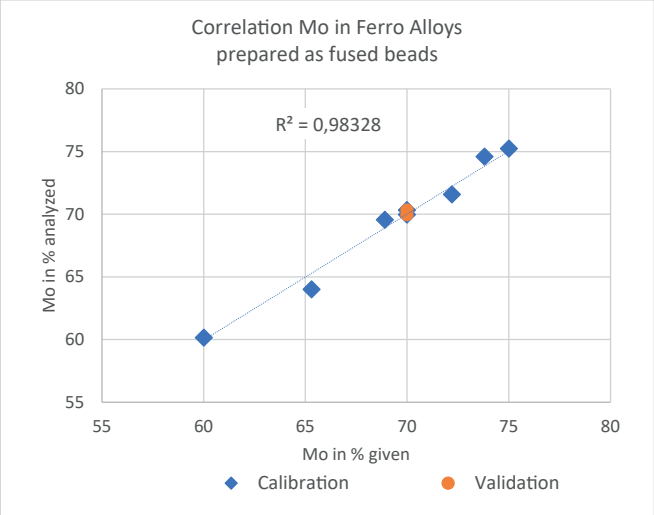

Figure 3: Correlation plot for Mo

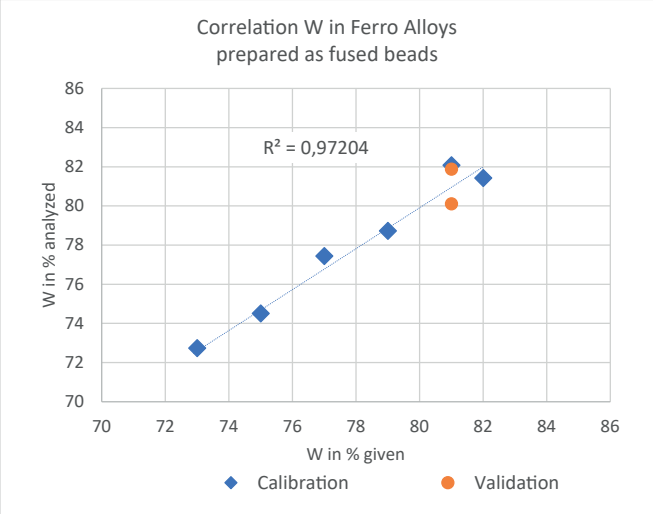

Figure 4: Correlation plot for W

Besides these four elements, the method also covers the analysis of the content of the elements Al, P, and Fe.

Summary

The example results show the capability to analyze ferro alloys, prepared as fused beads, with good accuracy using the SPECTRO XEPOS.

The correlation plots show high correlation coefficients. The results of the validation samples show a good agreement between expected and analyzed concentrations.

A fast screening of ferro alloys samples can even be done with samples prepared as powders in cups. Pressed pellets could be an alternative as sample preparation if well characterized reference samples with comparable grain size are available for calibration.

In view of size, power and cost the SPECTRO XEPOS meets also the requirements of modern laboratories.

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