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XMF 104 X-ray Micro Analyser

Application of Micro Beam X-ray Analysis in Corrosion Studies

Introduction

This application notes highlights the utility of micro-beam x-ray characterization in the field of corrosion metallurgy. In this study, micro beam x-ray elemental analysis was carried out on a corroded super-heater tube of a waste incineration plant. Elemental composition on the various parts of the corroded tube is essential for understanding the nature and cause of corrosion.

Such a study was possible due to the high resolution of Unisantis XMF-104 X-ray Micro Analyser which allows precise characterization of the mixture of salts and oxides, forming the deposits.

Unisantis XMF-104 X-Ray Micro Analyser

Application Note: 502

In cooperation with Chem4tech – University of Brescia (ITALY)

Application Background

Corrosion is an expected mechanism that can lead superheater tubes to failure. Localized or generalized loss of thickness occurs because of corrosion by the products of combustion (external) or from steam, especially when some contaminated water, coming from the drum, flows through the superheater (internal) after some process abnormality.

In this study, Micro-XRF measurements have been carried out on a superheater tube used extensively in waste incineration plant which experienced a leakage after six years of usage. Such a study throws light on the understanding of corrosion mechanisms which take part in the tubes

The picture of the corroded superheater tube is given in Fig1. The thick and porpous white deposits marked in the picture were scraped out and X-ray microanalysis was carried out on them.

Fig 1. Picture of the corroded portions of the superheater tube.

Instrumentation

Measurements were performed using a Unisantis XMF-104 X-Ray Micro Analyser , equipped with a 50 W Molybdenum tube and a high resolution two–stage Peltier cooled, compact Si-PIN detector.

This reduces the dimensions of the instrument significantly and also eliminates the need for cooling of the detector with liquid nitrogen.

The XMF uses a Polycapillary focusing lens which provides the analyst an intensely focused x-ray beam of very small diameter. Typical dimensions of the focal spot at the exit of the lens vary between 50 and 250 microns.

Results

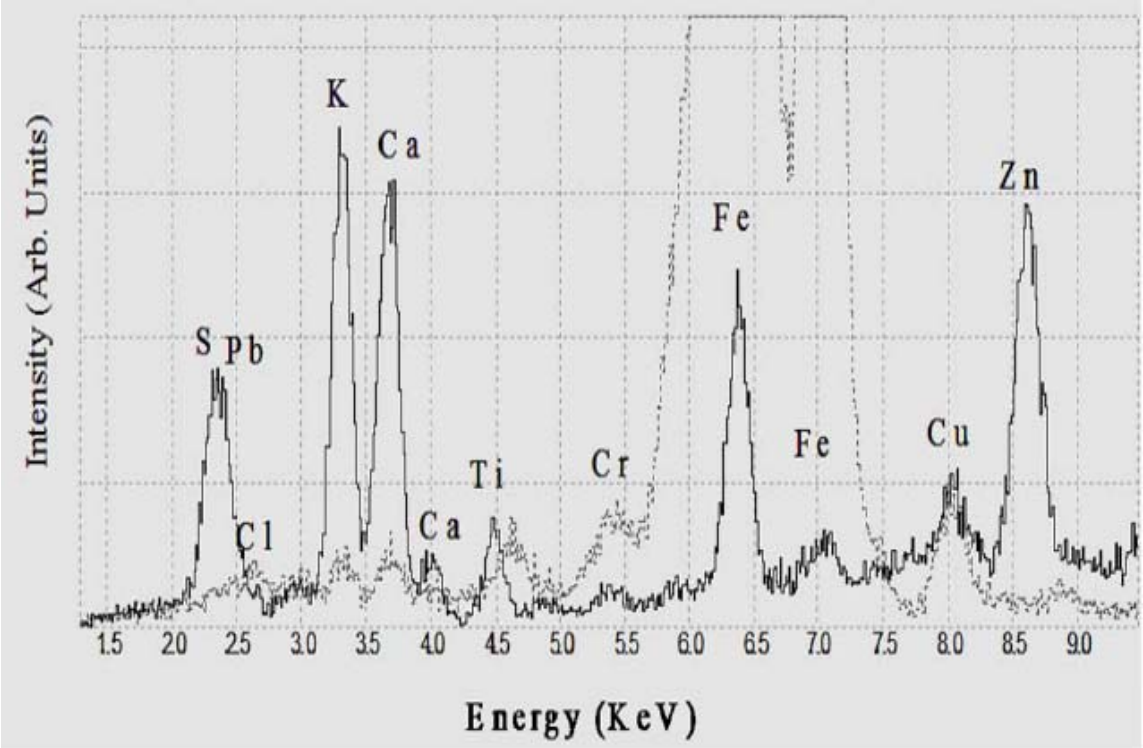

Fig 2. XRF Spectrum of white deposits on the corroded superheater tube

The microanalysis EDXRF spectrum obtained from the porous white deposits on the superheater tube is given in Fig 2.

The continuous line indicates the elemental spectrum obtained from the powder and the dotted line spectrum indicates the one obtained from the steel surface of the superheater tube.

Discussion

***μXRF** analysis (Fig.2) showed that the thick and porous white deposit mainly contains Sulphur, Potassium, Sodium, Lead and Calcium with small amounts of Chlorine and Silicon. The corroded steel surface was found to mainly contain Aluminium and Iron (Fig.2 - continuous line).*

In contrast, the analysis of the thin and adherent rust layer on the corrosion front, indicates the presence of mainly Iron along with a significant amount of Chlorine. Small quantities of Sodium, Copper, Calcium Potassium, Silicon and Zinc were also found. It is interesting to observe that in this case there is no evidence of Sulphur (Fig. 2 - dotted line).

Conclusion

From the comparison between micro-XRF and XRD analysis (not reported here), it was observed that the white fly-ash deposit is a mixture of crystalline sulphate phases, comprising mainly of ***$K_2Ca(SO_4)_2 \cdot (H_2O)$ (syngenite), $K_3Na(SO_4)_2$*** *(aphthitalite), $CaSO_4$* ***(anhydrite) and Pb*** *sulphates. Peaks, of chlorides like NaCl (halite), were also found in the XRD patterns.*

It can be concluded that HCl-induced corrosion of low-alloy steel is significantly retarded with addition of SO_2, because sulphates are formed instead of chlorides. Sulphate forms non-corrosive deposits, reducing the corrosion from chlorine attack. When the SO_2/HCl ratio is over 2, the formation of sulphate salts are favoured over the chloride salts and thus chlorides do not form at a sufficient concentration to induce corrosion.

Such a study was possible due to the high resolution EDXRF spectrum obtainable using Unisantis XMF-104 Micro Analyser, equipped with a low power tube. Optimal collimation of the primary x-ray beam from the low power tube was obtained using Kumakhov' polycapillary lens.

Other unique advantages of Unisantis XMF-104 are:

- No counter gas
- No external water cooling for x-ray tube
- Small footprint

Reference

The above study was carried out at Chem4Tech, University of Brescia, Italy under the expert guidance of Dr. L. Depero and her research team.

Unisantis S.A. is a global leader in the development and manufacture of innovative X-Ray analytical instrumentation, complete solutions and software for structure and elemental analysis using proprietary Kumakhov's optics best known for accurate beam collimation. Success in research has enabled Unisantis S.A. to develop new cutting - edge X-ray technology, applications and products for the market. Our products have particular applications in material characterization, life science and industrial analysis.

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