MICRO-XRF

Qualitative and quantitative analysis of a large welding joint with the M4 TORNADO

Application Note # XRF 464

Introduction

The M4 TORNADO micro-XRF spectrometer combines micrometer spatial resolution with a powerful set of analytical tools for qualitative and quantitative sample analysis. These tools are based on Bruker's unique position tagged spectroscopy (HyperMap) and include sophisticated functions for optimal display of measurement results, such as variations in intensities related to certain elements, and also for data cube mining.

For quantitative analysis a spectrum forward calculation is used instead of the usual peak deconvolution. Especially for metal alloys, this novel approach yields more stable results as the effects of peak overlap are significantly reduced and more fluorescence lines can be used for quantification.

Micro-XRF requires only little or no sample preparation. This reduction in pre-measure-

ment effort often calls for more after-measurement results evaluation by an experienced user. Thus, the full potential of the measurement technique can only be exploited, if data mining tools and quantification algorithm establish the means for flexible approaches.

This lab report exemplifies the advantages of a full sample scan compared to point analyses. It describes ways to obtain quantitative results from complex samples with HyperMap and the fundamental parameter (FP) based quantification method using the M4 TORNADO.

Sample

The investigated sample was a 50 mm x 70 mm cross section through a welding joint of two different steels.

Measurement conditions

The measurements were performed with a Bruker M4 TORNADO equipped with an Rh X-ray tube and a polycapillary lens. The following measurement conditions were used:

- tube voltage: 50 kV
- current: 200 µA
- pressure: 20 mbar
- spot size: <25 µm
- primary beam filter: 100 µm Al / 50 µm Ti / 25 µm Cu
- dwell time per pixel: 3 ms
- spatial resolution/pixel size: 14 µm
- scanned area: 70 mm x 47 mm
- total number of pixels: 14.6 Mio.

Results

The majority of materials are not homogeneous and it is challenging to find a representative location for quantitative analysis. Variations in the material composition, such as inhomogeneities, are an integral part of the sample properties. An element map or two-dimensional distribution analysis of the welding joint reveals significant compositional variations of the two steel alloys and the joint itself.

Figure 1 shows the distributions of the three relevant elements Fe, Mo, and Ni across the scanned area, revealing the complexitiy of the sample composition. Due to the different Fe intensities on the left and right side of the welding joint, two iron alloys can be distinguished. The welding joint itself is highly structured and consists mainly of Ni and Cr as can be seen in Table 1, where all found elements are displayed. Some areas also show a large Mo content. The results were obtained using a steel type calibration; their accuracy should be within ± 0.5 % (please refer to LR XRF 465).

Using HyperMap, the entire measured spectrum for each of 14.6 million pixels was saved. From this data cube, it is possible to extract the spectroscopic data for areas of interest, like an integral spectrum of an area, and perform a quantitative analysis therefrom. This was done for the two steel alloys (metal 1 and 2) and areas within the joint as marked in Figure 2.

Table 1 summarizes the quantification results. The composition of metal 1 (~ 9 wt% Cr and ~ 1 wt% Mo) reveals that this is a high temper-
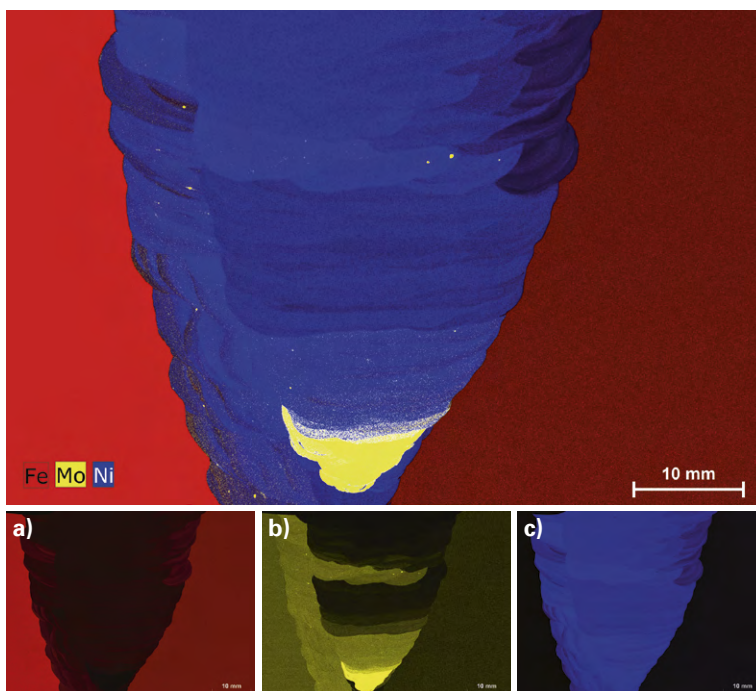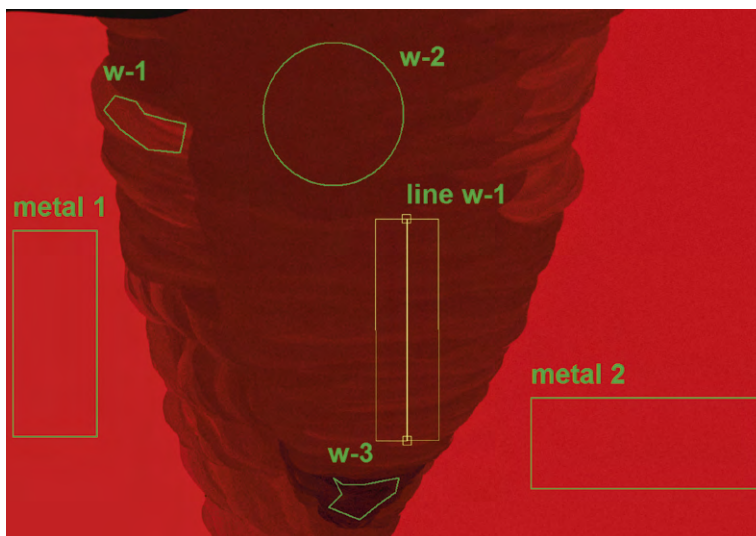
ature/high pressure steel (ASTM A335/P91). Metal 2, according to its composition, is a typical stainless steel (type 304). In order to weld these two metals together, a multitude of welding consumables was used in several steps. This is evident from both the varying elemental and quantitative compositions in the different regions w-1, w-2 and w-3.

Figure 3 displays the profile along line w-1. This line object was drawn into the data cube from top to bottom and then widened to sum all spectra perpendicular to the profile and therewith increase the statistics of the spectra and the representativeness of the profile.

Fe Mo Ni

a) b) c)

Figure 1

Distribution of Fe, Mo, and Ni in the welding joint and the adjacent metals 1 and 2 as shown in Figure 2

Figure 2

Element map of Fe with five selected areas and a line profile

w-1 w-2

metal 1

line w-1

metal 2

w-3

	Ti	V	Cr	Mn	Fe	Ni	Cu	Nb	Mo
metal 1	0.004	0.16	8.52	0.36	89.78	0.12	0.15	0.07	0.84
metal-2	0.39	0.04	18.91	1.68	69.58	8.74	0.36	0.01	0.29
w-1	0.12	0.03	15.09	1.37	19.68	61.32	0.01	1.20	1.19
w-2	0.07	0.02	15.74	5.59	9.04	68.04	0.01	1.35	0.14
w-3	0.11	Trace	19.88	0.61	3.28	67.14	0.01	2.94	6.04

Results of the quantitative evaluation

The individual spectra of the complete line profile were quantified and the mass fractions of the elements as a function of the distance are plotted. Fe shows large variations along the profile. Most significant composition changes can be seen for Mn, which is at > 7 % in the beginning and after ~ 12 mm drops down to 1.5 %, whereas the Mo content begins at 0.1 % and increases step-like to 1.5 % in the lower part of the welding joint.
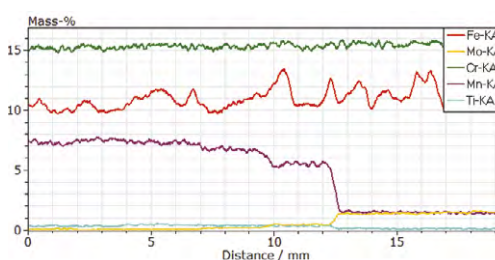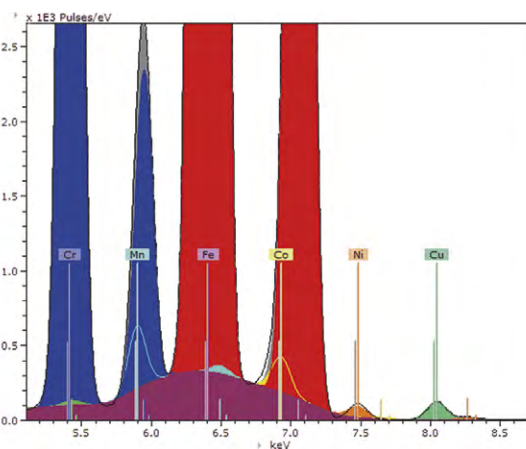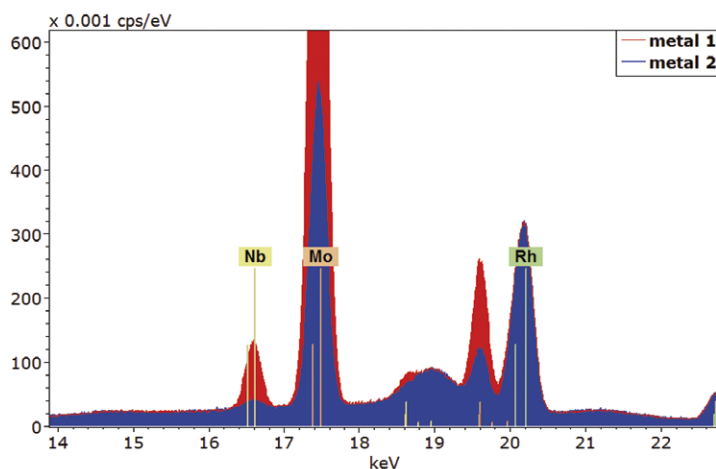

Figure 3

Line profile w-1 as cut from the datacube in Figure 2

Figure 4a displays the comparison of the spectra for metal 1 and metal 2 in the energy range for the elements Nb and Mo. The detection of Nb in very low concentrations from 700 ppm down to 100 ppm illustrates the high sensitivity of micro-XRF or XRF in general. Figure 4b shows the forward calculated convolution of the sum spectrum of metal 1 where Cu has a concentration of 0.15 %. This illustrates that in the related energy region the sensitivity of the M4 TORNADO is still sufficient for a reliable analysis of common steel alloy elements, even though it is not as high as in the Zr–Mo region.

As mentioned in the measurement conditions section, the measurement for the quantitative analysis was done using a strong primary beam filter. When the sample is scanned without a filter, diffraction peaks which are common for crystalline samples such as metal alloys will obstruct the peaks of minor and trace elements, here mainly V, Ni and Cu. These diffraction peaks, however, also provide information on the welding process.

Figure 5 reveals the lateral distribution of different crystallites in the sample. This kind of plot can be obtained by marking diffraction peaks
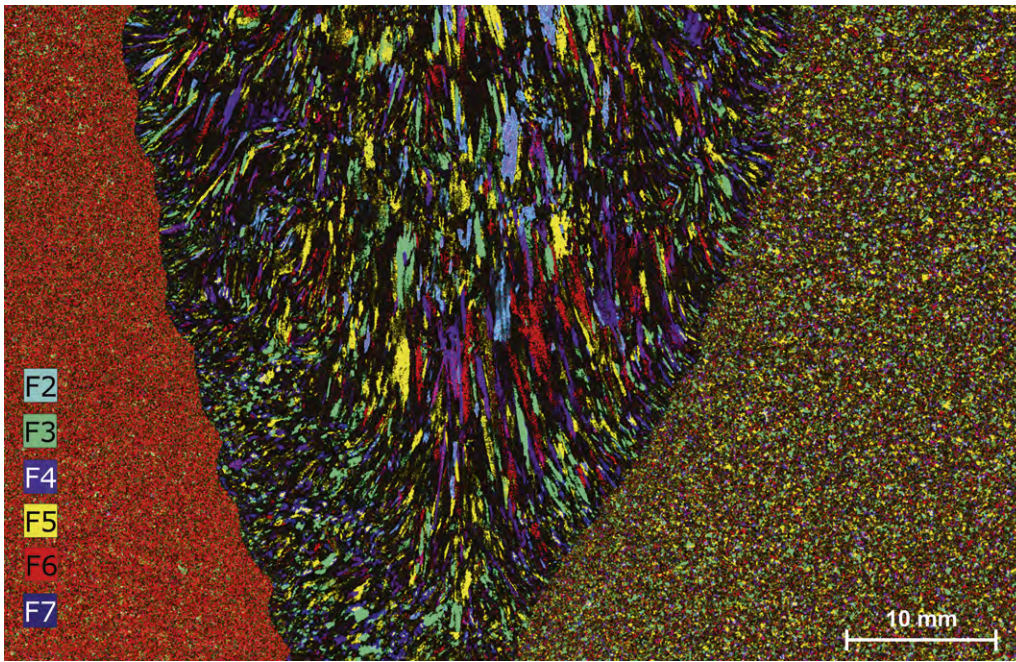
Figure 4

Illustration of the sensitivity of the M4 TORNADO in different spectral regions. Spectra for metal 1 and 2 (left) and convoluted sum spectrum (right).

in the map sum spectrum and subsequently display their distribution (please refer to LR XRF 463).

In the welding joint the size and the growth direction of the individual crystal domains are obviously related to the temperature gradient. At the interface between the joint and the adjacent metals, where the cooling is much faster, the diffraction patterns are very small and directed perpendicular to the interface. With an increasing distance to the metal, the crystallites become larger as the cooling in the center of the welding joint is slower and they had more time to grow.

Especially for the quantification of structured samples, the HyperMap functionality is vital as it allows to scan a sample and afterwards identify and select areas from the datacube which can be considered homogeneous. The subsequent FP quantification yields high accuracy and allows the identification of the qualitative and quantitative sample composition. Multiple data evaluation tools support an in-depth investigation of complex samples. Even diffraction peak distributions can be evaluated to provide even more information about the investigated sample.

Conclusion

The M4 TORNADO with the spacious measurement chamber and the exclusive analytical performance make this instrument an essential tool for modern material analysis.

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