

Quantified X-Ray Mapping in the WDS Mode

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Keywords: EPMA, segregation, mapping

Abstract. In material science, there is an increased demand for mapping of microstructural components and their composition. EPMA (Electron Probe Micro Analysis) with WDS (Wavelength Dispersive Spectrometry) is known as having high spectral resolution and sensitivity, but in practice considered to be slow in mapping applications. The present work describes a development of EPMA including design of both instrumental hardware and software related to electronics and calibration.

Instrument: Modified ARL-SEMQ DELL GX1-500

Number of WDS spectrometers: 6 (which means 6 elements simultaneously)

Calibration: **Certified Reference Materials** (scan area 2x2 mm)

Specimen size: 1 x 1 mm to 57 x 64 mm

Accelerating voltage: 7-25 kV; sample current: 2-10 μA (means high intensity)

Point size: 1x1 to 100 x 100 μm (each analysis is an average over the point size; step length = point size)

Analysis time per point: 0,05 to 0,2 sec.

Number of analysis points: 10 000 to 200 000

Total analysis time: 1 to 6 h

1. Introduction

Computerised EPMA-WDS is a well-established technique having high X-ray spectral resolution and sensitivity [1]. The objective of the present work is to summarize the development of a microanalytical laboratory comprising optimised EPMA-WDS instrumentation regarding analytical performance and analysis time in elemental concentration mapping applications.

2. Experimental

Aiming for a high performance microanalytical laboratory, a natural benchmarking was the ordinary melt shop laboratory, where advanced spectrometry techniques are used. The EPMA instrument used in elemental concentration mapping is a modified ARL-SEMQ DELL GX1-500 with six wavelength dispersive spectrometers. The original instrument was modified with modern computers, electronics, X-ray detectors, step motors for high precision in control of specimen stage and spectrometer settings. The electron optics was optimised for high sample currents 2-10 μA , resulting in high X-ray intensities meeting demands for fast analyses giving good pulse counting statistics. The high intensities are needed to minimize the analysis time per point which reduces the total time for the mapping. For calibration of the instrument Certified Reference Materials (CRMs) are used. Further, the repeatability in the stage movement using step motors is critical to get good spatial resolution in the assessment raster. This has been developed from both hardware and software point of view to get automated routines. Two computers are involved, one in the control of the microprobe hardware and one for calculations and processing of the mapping images. Software was also developed for on-line evaluation communication using remote desktop access.

3. Results and Discussion

The following parameters can be used in a wide range of applications:

- 6 WDS spectrometers, all with double monochromator crystals. Simultaneous determination of six elements (high concentration elements in combination with low concentration elements). It should be noted that the analysis is non-destructing and additional elements, 7 – 12, can be assessed in a second run. Further, areas can be selected for repeated analysis with a higher resolution using shorter steps (“higher magnification”).
- Specimen size: 1x 1 mm to 57 x 64 mm
- Accelerating voltage: 7-25 kV; sample current: 2-10 μ A.
- Point size: 1x1 to 100 x 100 μ m (each analysis is an average over the point size; the step = point size)
- Analysis time per point: 0,05 to 0,2 sec.
- Number of analysis points: 10 000 to 200 000
- Total analysis time: 1 to 6 h
- Calibration: Certified Reference Materials (scan area 2x2 mm)
- Polished specimen (non-destructing analyses)

An illustrative example is EPMA mapping of segregation in the mid-thickness of a conticast low alloy construction steel [2]. The concentrations according to heat analysis is C 0,28 %; Mn 0,85%; P 0,009 %; Cr 0,60 %; Ni 0,52 %; Mo 0,33%.

Certified Reference Materials: NBS no 1161 – 1165 (for calibration an area 2 x 2 mm was scanned, that is 20 x 20 = 400 points à 100x100 μ m)

Analytical conditions:

Specimen size: 20 x 42 x 10 (mm) and scanned area 16 x 39 (mm).

Raster: 1570 rows and 640 columns (1 004 800 analyses).

Point size: A square 25 x 25 (μ m). About 98 % of the scanned area 16 x 39 mm was analysed, because of resetting time of the pulse counters.

Accelerating voltage: 25 kV.

Sample current: 8 μ A.

Fig. 1 shows the EPMA Mn- mapping in the mid-thickness region of a conticast slab.

The concentration scale uses colours for different concentration ranges obtained in the analyses. The three squares A – C show the distributions of concentrations at different “magnifications”. The dendritic structure is shown with good resolution. Semi-macro segregates are found in the upper part and a centre line segregation, about 1,5 -2 mm wide, at height 13 mm in Fig.1.

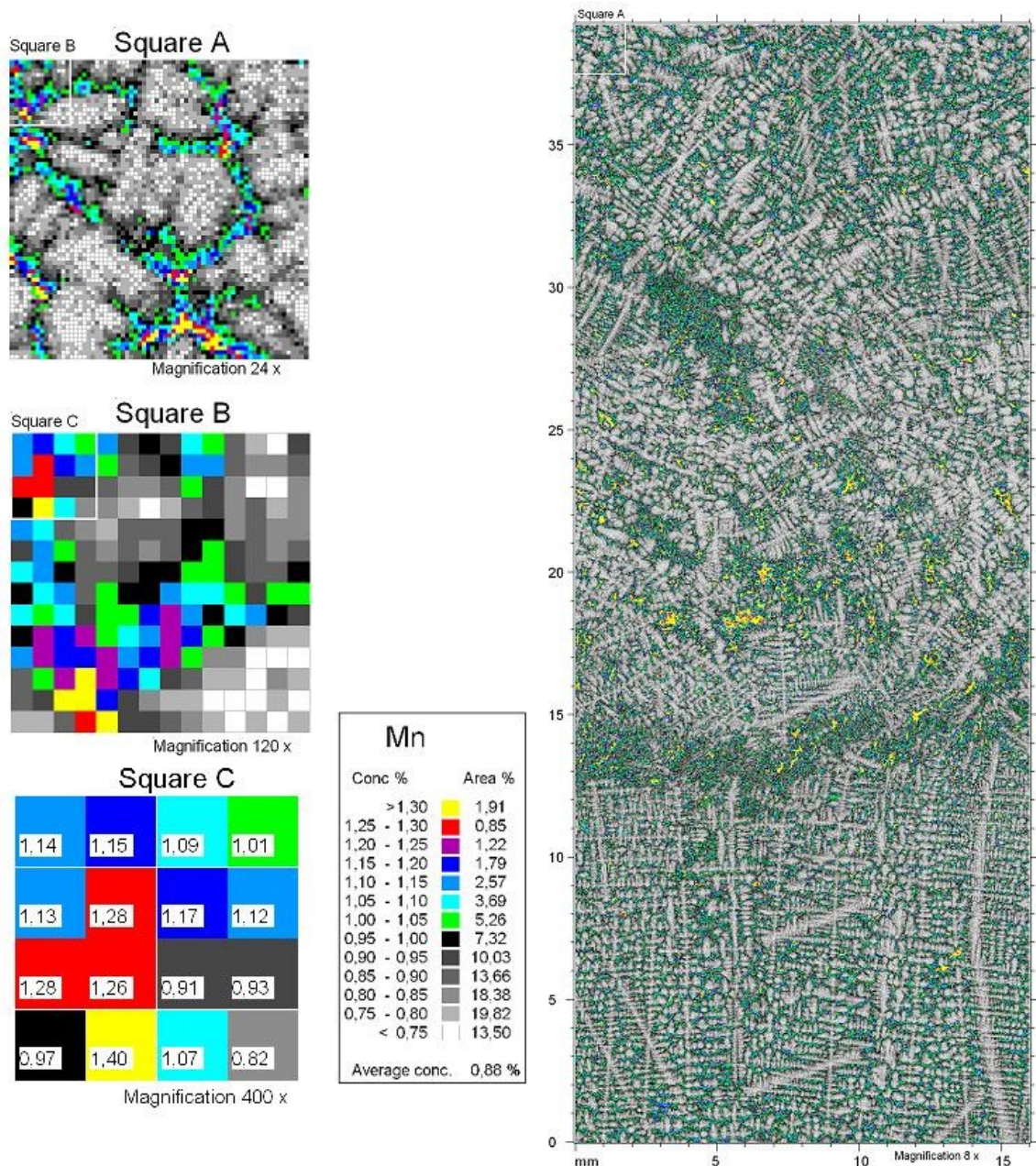


Figure 1. EPMA Mn- mapping (16x39 mm). Point size 25 x 25 μm .

Good resolution of dendritic pattern

Columnar crystal structure at bottom, centre line segregation at about $y = 13 - 15$ mm.

Table 1 summarizes the average concentrations from the mapping in the segregated mid-thickness region compared to the heat composition.

Table 1. Average composition from mapping compared to heat composition (wt%)

Method	Cr	Ni	Mo	Mn	P	C
Mapping	0,63	0,52	0,31	0,88	0,010	0,28
Heat	0,60	0,52	0,33	0,85	0,009	0,28
Difference	+0,03	0,00	-0,02	+0,03	+0,001	0,00

This means a **relative** deviation from the heat composition in the range 5-10%, maximum. The segregated mid-thickness part might explain the deviation.

A second example shows segregation studies in compacted graphite iron (CGI) using EPMA WDS mapping [3]. From the microstructure investigation of color etched specimens, areas for

mapping were selected and marked with Vickers indentations. The mapping was performed on the polished surface and comprised the elements Mn, C, P, Si, Cu and Mg, calibrated using Certified Reference Materials. An efficient procedure in mapping is to initially produce an overview which can be compared with the etched condition and the as polished condition. Areas for secondary mapping at higher spatial resolution (higher magnification) can be chosen and marked using on-line internet facilities for remote desktop access.

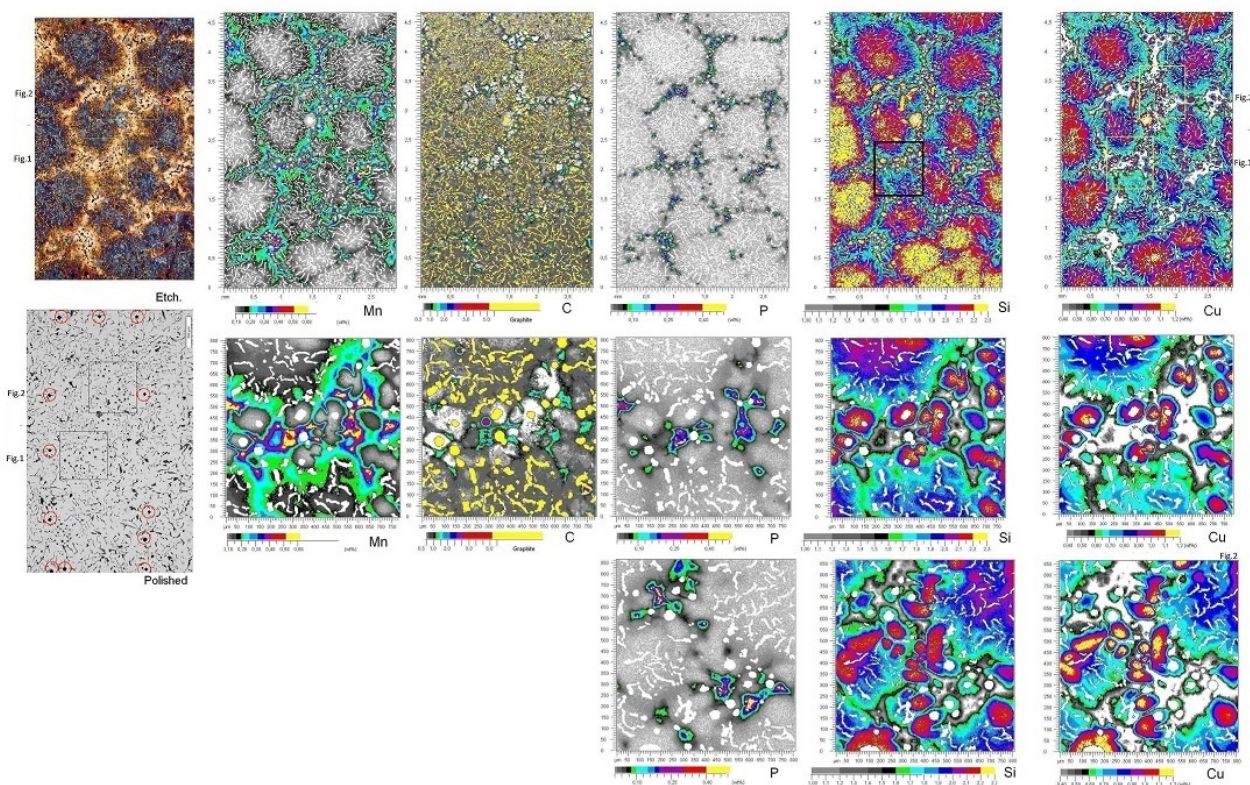


Figure 2. EPMA mapping of CGI. Micrographs of tint-etched and polished surface, respectively. The corresponding concentration maps for Mn, C, P, Si and Cu, respectively, are shown with color scales for the concentrations

4. Conclusion

A microanalytical laboratory has been developed meeting demands for high resolution EPMA mapping.

A quantitative tool for analysis of segregates is high resolution EPMA including elemental concentration mapping and image analysis. Quantitative accuracy is obtained using calibration with Certified Reference Materials.

Recent work in cooperation with research groups shows that computerised EPMA can be used in various development works: alloying/ thermodynamics, casting/solidification, welding, heat treatment etc.

References

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