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Analysis of Boron by epma: correction for dual Mo and Si interferences for phases in the Mo-B-Si system

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Analysis of compounds in the Mo-B-Si system is of interest in material science research [1,2]. Boron can be detected with synthetic defractors (LDEs) with 2ds ranging from 200 to 100 Å (Table1). Boron Ka, 67.7 Å , suffers from pathological interference by Mo Mz at 64.35Å (Figure 1). This severe difficulty has forced researchers to analyze only Mo and Si and assume B content from the initial starting material composition, e.g. Huebsch et al [2].

In 1995, we began to investigate direct epma of B [1]. We observed the additional complication of the low wavelength background amplitude changing with varying Si content. We have now identified this as due to a strong 6th order reflection of Si Ka "off the end" of the 200 Å LDE (at 42.7 Å), complicating background modeling for B Ka and the Mo Mz overlap. The use of PHA does not improve the situation.

Research was conducted at UW-Madison utilizing a Cameca SX51 electron microprobe, outfitted with a “large area” 200 Å LDE (Ovonyx) as well as anti-contamination devices (cold finger and baffle, and air jet). To minimize a large matrix correction for B, we use an accelerating voltage of 7 kV; materials are not coated. Standards are pure B, Mo and Si. Mo La is analyzed with a PET crystal, and Si Ka with TAP. Nunes et al [1] found no cause for concern regarding B Ka peak shifts or shapes of the various B-phases considered.

We now have software that applies a simultaneous correction for both the Mo and Si interferences [3,4]. With the wide range between the low wavelength side of Mo Mz and the high wavelength side of B Ka (38 Å or 20000 sin theta units) it is essential that a curved (exponential) background be used (Figure 2). The software used to apply these features is Probe for Windows (Advanced Microbeam Inc); the overlaps are iterated within the matrix correction until convergence. The matrix correction is Armstrong’s phi-rho-z (modified from Brown and Bastin) [5], with Henke MACs.

Table 2 presents data for the pure end members and demonstrates the ability of the dual corrections with curved background to yield significantly improved analyses for B. It is also essential for the software to report negative K ratios to indicate the effectiveness of the corrections. Some analyses of MoB and the T2 phase (Mo5SiB2) are shown in Table 3, and are compared to the nominal/stoichiometric values of these phases – recognizing that there are solubility ranges present. Determining the solubility ranges – particularly for B – is in fact a key motivation for this research. These analyses show small differences in atomic percentages compared to analyses we did previously with less robust interference correction

software [1]. We are currently evaluating the effect of utilizing other MACs for B, as well as alternate standards.

References

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[3] Donovan, Snyder and Rivers 1993 Microbeam Analysis **2**, 23-28.
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[5] Armstrong 1988 Microbeam Analysis pp. 239-246.

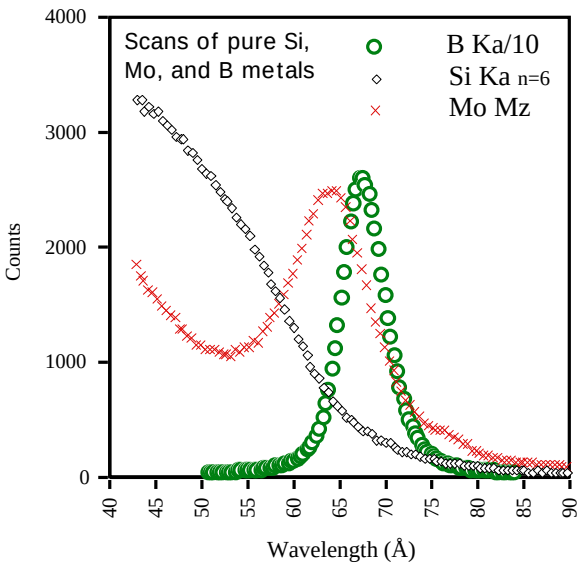

Figure 1

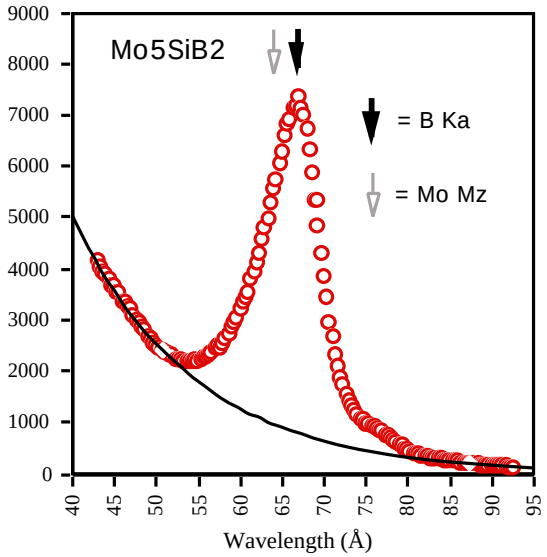

Figure 2

Table 1

LDE type	Mo-B4C	Ni-C
2d (A)	200	100
Sin Theta	0.35	0.71
Peak c/s/nA	2574	14
P/B	15.1	4.5

Table 2

	without interference correction on B Ka				with Mo-Si interference corrections for B Ka			
sample	B wt%	Si wt%	Mo wt%	Sum wt%	B wt%	Si wt%	Mo wt%	Sum wt%
pure Si	7.3	101.2	0	108.5	0*	100.6	0	100.6
pure Mo	5.1	0.04	100.3	105.4	0.16	0	100.4	100.5
pure B	99.8	0.06	0	99.9	99.4	0.07	0.06	99.6

* K-ratio = -.0005

Table 3

	B wt%	Si wt%	Mo wt%	Sum wt%	B at%	Si at%	Mo at%	Sum at%
MoB-nominal	10.13	0.00	89.87	100.00	50.00	0.00	50.00	100.00
MoB-experimental	10.00	0.03	90.60	100.64	49.44	0.07	50.49	100.00
Mo5SiB2-nominal	4.08	5.31	90.61	100.00	25.00	12.50	62.50	100.00
Mo5SiB2-exp.	4.60	5.27	89.61	99.49	27.52	12.12	60.36	100.00