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Comparing Quantitative Results of Zeta Factor Method using Various Values of Ionization Cross-sections

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The accuracy of a quantitative analysis in TEM-EDS is generally worse than that in SEM-EDS. There are two main reasons. One is the sample condition and orientation. The sample needs to satisfy uniformities of composition and thickness in a region of electron irradiation. Another reason is an inaccuracy of ionization cross-section used in quantifications such as the Cliff-Lorimer [1] and the Zeta factor [2]. There are some theoretical ionization cross-section tables available for the K line X-ray series. However, the result of quantitative analysis fluctuates depending on the values of ionization cross-section used in the calculation. A *k* factor of the Cliff-Lorimer method and a *zeta* factor of the Zeta factor method can be calibrated to obtain better quantitation. However, it is difficult to obtain thin standard specimen for a target sample. Especially, in Cliff-Lorimer method, the sample required to be no significant absorption of generated X-rays. Hence, it's difficult to calibrate these factors and therefore these factors obtained from experiments must be calculated from the theoretical ionization cross-section.

Here we present the results on our attempt to determine the most accurate ionization cross-section by comparing different Zeta factor methods of some samples, experimentally. The Cliff-Lorimer method was not used in this comparison because we cannot determinate the errors of sample thickness and density.

Table 1 shows the comparison of the quantifications by Zeta factor method on different ionization cross-sections. The EPMA result is from a bulk sample of San Carlos Olivine, analysed before making the thin sample. The solid angles were calculated by the Zeta factor method from a NiOx standard sample (Ted Pella Inc). Quantitative values are calculated by Zeta factor method on San Carlos Olivine using calculated solid angles. These EDS spectra were taken with a JEM-2100 equipped with 100 mm^2 SDD. Accelerating voltage was 200 kV and probe current was 53 pA. χ^2 is sum of squared residuals based on EPMA result. The solid angle and quantitative values fluctuate depending on the ionization cross-section values used. The difference of the solid angles shows one of absolute ionization cross-section. In this sample, the quantitative values of Paterson's ionization cross-section were the closest to the EPMA result and this ionization cross-section has good accuracy for the Olivine.

The PAP ionization cross-section was optimised for low accelerating voltage like EPMA. The other ionization cross-sections were calculated by fitting some measured data with Bethe model in TEM. However, the results from these three ionization cross-sections were different and we found that the Paterson's ionization cross-section has the best accuracy, experimentally.

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Table 1. Comparison of zeta factor results. Sample for solid angle and quantitative results is NiOx and Olivine, respectively.

	Solid angle [sr]	**Quant. values [mass%]**				
		O	**Mg**	**Si**	**Fe**	χ^2
EPMA		57.1	25.4	14.3	3.1	
Paterson J.H. [3]	0.68	55.3	25.7	14.4	4.6	5.6
Zaluzec N.J. [4]	0.49	52.9	26.2	15.2	5.7	25.9
Jakoby [5]	0.46	52.8	26.5	15.3	5.4	26.0
PAP [6]	0.59	51.9	26.4	15.6	6.1	38.7

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