

Trace and Small Phase Analysis : a New Generation of Column for Electron Microprobe

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The electron microprobe has become an essential technique for microanalysis in the field of geology, material sciences, microelectronics... Those fields are advancing and require techniques to detect and to measure light and heavy elements in lower concentration, located in particles and smaller phases or thinner layers. In this presentation, we propose to expose the improvements of the electron column of the Cameca SX100 microprobe to fit the specific requirements described above.

We can define several criteria to improve the microprobe column. The smaller phase analysis requires a bright source, an optimized focus of the electron beam, and also a stable low voltage. The thinner layers measurement implies low to high voltages with an optimal control of the voltage. The trace elements quantification needs high beam currents, and low to high voltages, depending on the phase size, or the excited x-ray lines. The light elements analysis requires low voltages and high currents. Hence, the requirements for the new generation of the electron column of the SX100 microprobe must emphasize on a minimal spot size, a high stable current, low to moderate voltages, but not excluding the high voltage to keep the versatility of the machine.

In order to satisfy those criteria, the electron column has been improved with the following characteristics :

- A versatile gun with good vacuum, allowing the use of a W filament for high voltage (up to 40 kV) and a LaB₆ or CeB₆ cathode for measurement at high spatial resolution. The power supply is designed to heat both cathodes at their optimal working point.
- An optimal alignment and new working mode of the column in order to optimize the beam density for high spatial resolution.
- A new beam current regulation with an extended range: from 0.5 nA up to 1 μA.

Those improvements compared with the usual column are obvious in terms of spatial resolution (Table 1 and Figure 1). The applications of the optimized electron column are numerous in EPMA for trace analysis or elemental mapping in small areas. We have compared the calculated analytical resolution for different pure standards (Si, Fe, Pb) at several beam currents with the W or LaB₆ gun with the new SX100 column. The analytical resolution is defined as the quadratic sum of the probe diameter and the ionization depth calculated with a $\varphi(\rho z)$ model [1]. The results of this comparison (Figure 2) clearly indicate that with the optimized column and LaB₆ cathode, it is possible to reach small probe size, compatible with the ionization depth obtained at voltages currently used in EPMA. Thus, an analytical resolution of 1 μm can be obtained at 15 kV and 100 or 1000 nA for light and heavy matrix, which makes it possible the trace elements measurement (concentration inferior to 0.01% weight) in small areas with a reasonable statistical precision. Those results will be discussed in order to evaluate the optimal working domain of the W gun and the Lab₆ gun.

[1] C. Merlet, Mikrochim. Acta 114/115 (1994) 363

Table 1 : Probe diameter for remarkable current values at 10 kV. Comparison between a tungsten (W) and a LaB6 cathode.

Current (nA)	Probe diameter (μm)		
	W and standard column	W with optimized column	LaB ₆ with optimized column
100	0,8	0,6	0,3
500	1,5	1,1	0,5
1000	2,0	1,4	0,6

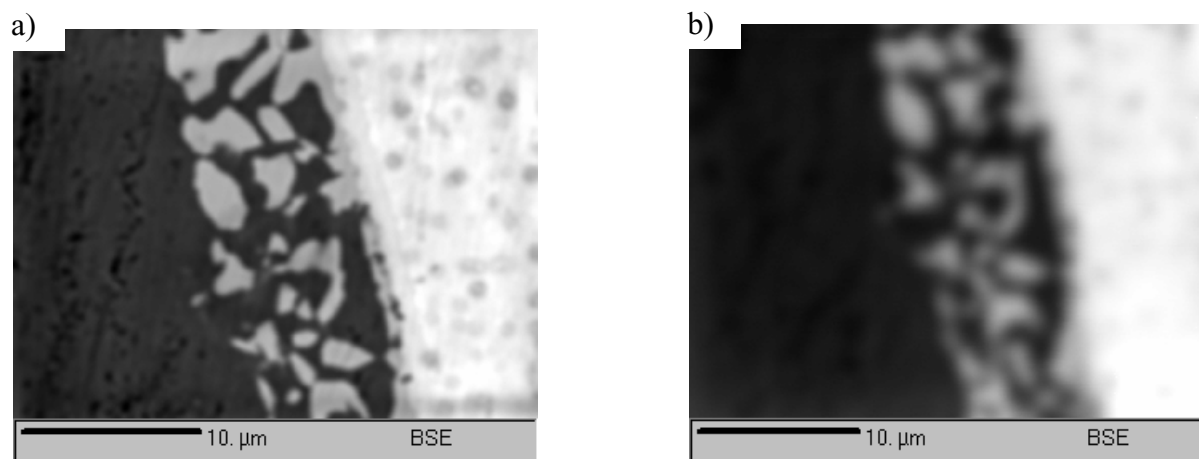


Figure 1 : BSE image on a bronze sample acquired at 10 kV, 100nA. The image a) is obtained with a LaB₆ cathode with the optimized column and the image b) is obtained with a W filament.

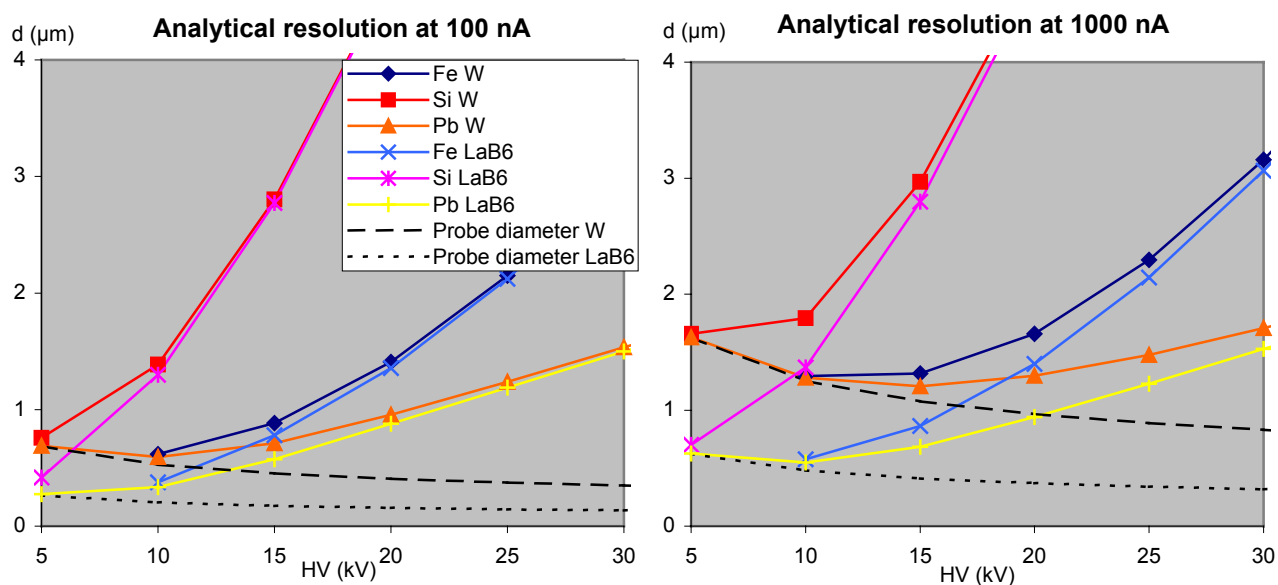


Figure 2 : Analytical resolution (d , in μm) for Fe $K\alpha$ in pure Fe, Si $K\alpha$ in pure Si and Pb $M\alpha$ in pure Pb obtained with LaB₆ and W cathode at 100 nA and at 1000 nA.