In situ Multilayer deposition studies by X-ray Energy Dispersive Method

J. C. Malaurent*, H. Duval*, G. Cauchon**, P. Dhez**, O. Hainaut**, M, Idir**
and J.P. Chauvineau***.

** Laboratoire de Physique des Solides, Bat 510 Univ. Paris XI, F- 91405 Orsa= y.*

*** Laboratoire de Spectroscopie Atomique et Ionique, Bat 350, Univ. Paris XI, F- 91405 Orsay.*

**** Institut d'Optique Theorique et Applique d'Orsay, Bat 503 Univ. Paris XI, F- 91405 Orsay.*

As demonstrated early by E. Spiller, in-situ X-ray reflectivity is adequate to provide basic information on multilayers during deposition and on coating methods. Only few practical methods exist because the substrate must be fixed during coating and the vacuum compatible reflectometer shoud be without moving part.

Spiller's in-situ reflectometor uses a monochromatic soft x-ray line, at fixed incidence, produced by a windowless x-ray tube inside the evaporator. In 1994 another in-situ x-ray reflectivity method was developed but with source and detector outside. It used a classical x-ray tube, a curved Si111 crystal and a CCD detector with a phosphor converter (1). The small angular divergence of the prepared monochromatic beam is suitable to get reflectivity at 8 keV versus incidence angle. Compared to Spiller's method, the 8 keV photons are able to penetrate all the stack, in addition the angular opening of the monochromatic beam give the possibility to follow simultaneously the reflectivity from the total reflection region to Bragg peaks.

Reflectivity by an Energy Dispersive Method (EDM) is convenient for in-situ reflectivity tests. As demonstrated few years ago, it is well-suited to study multilayers on specular (2) and off-specular (3) conditions over a broad energy spectrum, typically 5 to 50 keV. By initially setting the incidence angle, the first order Bragg peak can be brought to any given energy. Dependence of the calculated reflectivity on possible indices uncertainties, versus energy, were pointed out but appear to be quite low due to the evaluated indice table accuracy (4).

Mo/Si multilayers deposited on (111) silicon wafers by electron beam evaporation and by ion beam sputtering have been studied experimentally by EDM reflectivity with simultaneous monitoring by an in-situ Spiller's type reflectometer at 44=C5. Even for the first deposited periods, EDM permits to observe the secondary fringes appearing between the total reflection limit and the first Bragg peak. Peak reflectivity and band pass of the Bragg peaks have also been followed during deposition. Like others reflectivity methods, EDM is sensitive to interface roughness during the deposition of successive periods and roughness estimates were performed. Experimental results were compared to corresponding calculated models.

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