Development of multilayer coatings (Ni/C - Pt/C) for hard X-ray telescopes by e-beam evaporation with ion assistance

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ABSTRACT

A number of X-ray astronomical missions of near future (XEUS, Constellation-X, SIMBOL-X, HEXIT-SAT, NEXT) will make use of hard X-ray (10-100 keV) optics with broad-band multilayer coatings. To this aim we are developing a multilayer deposition technique for large substrates based on the e-beam deposition technique, improved by the implementation of an ion beam assistance device, in order to reduce the interfacial roughness and improve the reflectivity. The e-beam deposition with ion assistance keeps the film smoothness at a good level and takes the advantage of a reduction of the interlayer stresses[11]. This approach is well suited for the manufacturing of high-reflectance multilayer mirrors for hard X-rays space telescopes where, in addition to a high quality of the deposited films, a volume production is also requested. Moreover, we are also up-grading the replication technique by nickel electroforming, already successfully used for the gold coated soft X-ray mirrors of Beppo-SAX[1], XMM[2], JET-X/SWIFT[3] missions, to the case of multilayer coated mirrors. In this paper we will present the technique under development and the implemented deposition facility. Some preliminary, very encouraging, results achieved with the X-ray (8.05 and 17.4 keV) and topographic characterization on flat samples will be discussed.

Keywords: hard X-ray mirrors, e-beam evaporation, Ni electroforming replication, multilayer coatings

1. SCOPE OF THE WORK

The extension to the hard X-ray band (10 -100 keV) foreseen in next focusing X-ray telescopes will mostly implement multilayer-coated mirrors. A suitable technique for the production of multilayer mirrors is an extension of the Nickel electroforming replication from shaped mandrels[4,5]. This approach is very attractive since, in addition to get very good optical performances, in the case of multimodular telescopes it implies a reduction of cost and integration time since the shells are monolithic and many elements can be replicated from the same master.

In order to extend the Ni replication technique to the case to the case of multilayer coated mirrors, in the framework of a development project funded by ASI (the Italian Space Agency) which involves INAF/OAB, Media Lario S.r.l., LABEN, IASF, Politecnico di Milano, we are developing a multilayer deposition technique on mandrels based on the e-beam deposition. The choice of this kind of deposition is determined by the requirement of uniform coating of large surfaces, a feature which is achievable with difficulty with techniques like the ion-beam sputtering. We can, moreover, reuse the same coating facilities installed at Media Lario, which were used to produce the mirrors of XMM by Nickel electroforming replication.

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A crucial point is given by the surface quality of the mandrel (that has to be much better compared to the soft X-ray mirror with Au coating case) as the multilayer roughness growth process is very sensitive to the substrate smoothness level[13,14]. A superpolishing method has been developed at INAF OAB for this specific task: the Zeiss machines developed for the XMM project (now installed at OAB) are used for this application (fig.1). The improvement in smoothness of the superpolished surface in comparison to a SAX mandrel is evident (see tab.2).

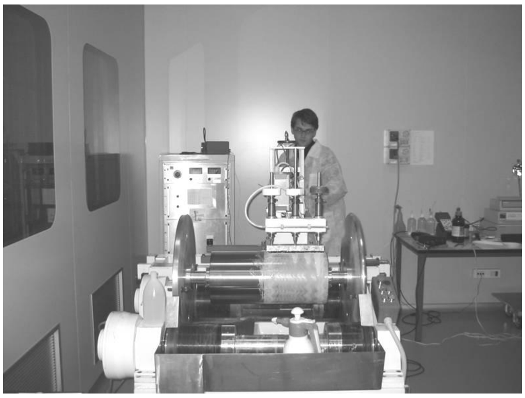


Fig.1. A Zeiss superpolishing machine installed at OAB working on a master mandrel

Instrument	**Scan Length (μm)**	**SAX #12 mandrel rms (Å)**	**Superpolished mandrel rms (Å)**
WYKO -2.5 X	***6000.0***	***N. A.***	***10.1***
WYKO -20 X	***660.0***	***7.6***	***3.0***
AFM	***10.0***	***6.2***	***2.4***
AFM	***1.0***	***3.4***	***1.8***

Tab. 2: The roughness values[6] achieved by the new lapping method on a prototype mandrel surface are compared to those of the SAX mandrel #12

2. HARD X-RAY EVAPORATED MULTILAYER MIRROR PRODUCTION

In our production method setup Pt/C or Ni/C multilayer are deposited on a rotating mandrel by e-beam evaporation in the existing facility at Media Lario (see fig. 3,4,5). The thickness monitoring is demanded to a quartz microbalance. The adopted e-beam method permits a high deposition rate (more than 0.2 nm/sec, on non-rotating samples). Some crucibles are available on a carrousel to allow the alternate deposition of different materials.

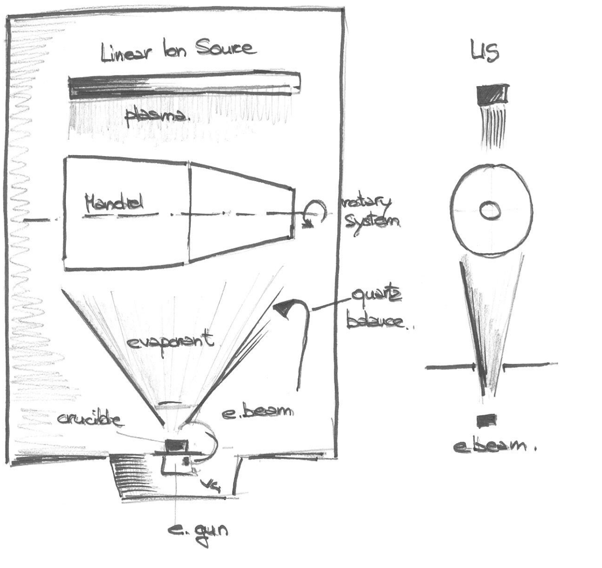


Fig.3: The deposition facility in ion-assistance configuration front view (left) and side view (right). The rotating mandrel is coated with a multilayer film by the e-beam evaporated material, while an ion flux source “assists” the deposition

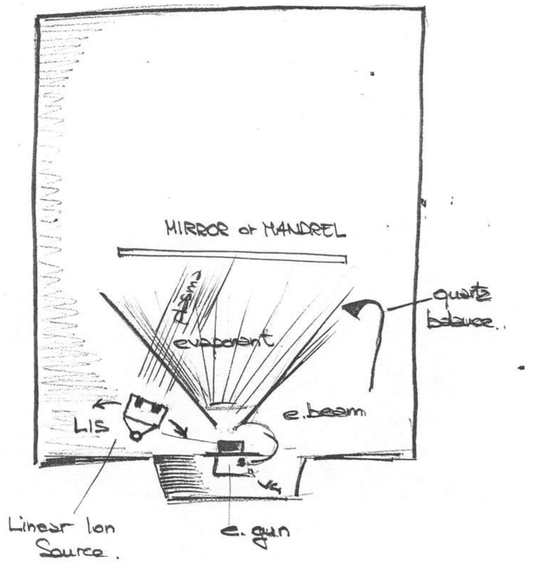
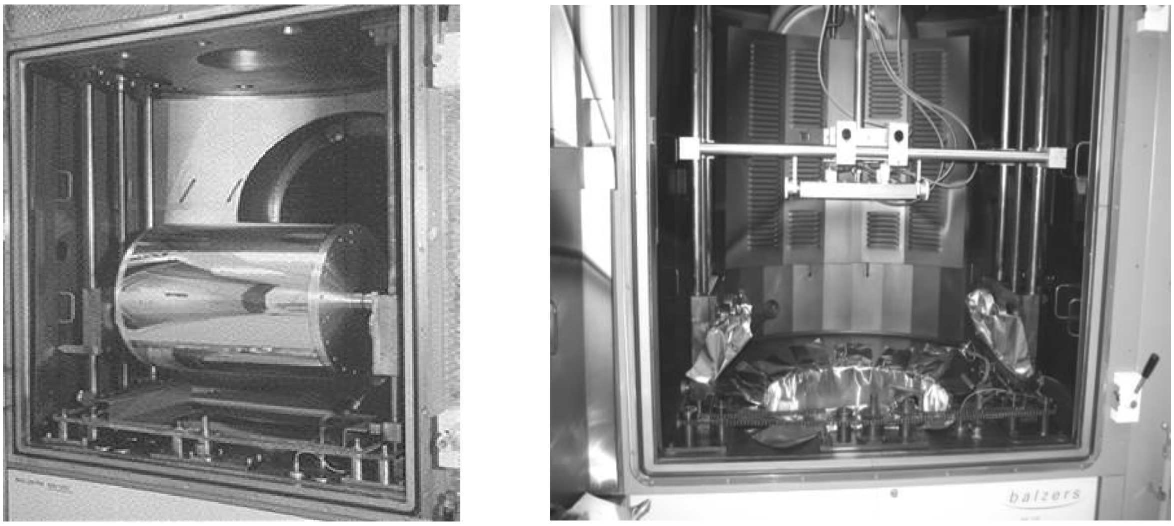


Fig.4: The deposition facility in open substrates coating configuration - front view. This arrangement can be used to coat planar mirrors or open curved surfaces, e.g. the XEUS segmented mirrors.

The facility has been upgraded by installing a linear Ar ion source, that assists the deposition to improve smoothness, adhesion, hardness, stability of the growing multilayer on the mandrel[12]. The ion source provides a homogeneous ion flux upon the whole mandrel length.

The current setup of the linear ion source is shown in the fig. 3. The evaporate (Pt, Ni, C) condenses on the rotating mandrel: the growing film is then exposed to Ar ion flux incoming from the Linear Ion Source. This approach is well suitable to make monolithic replicated mirrors: an alternative setup (open substrates coating configuration) may be used (see fig. 4) to coat planar or curved, segmented surfaces, as the XEUS mirrors.



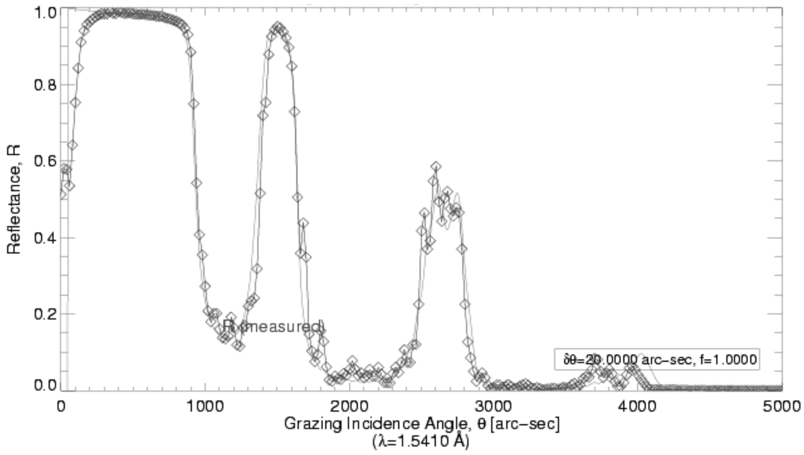
(A) (B)

Fig.5: Front view of the *Balzers* coating chambers installed at Media Lario. (A) A first open chamber hosting a gold coated mandrel used to make a XMM shell mirror: the chamber is large enough to host large mandrels e.g. those foreseen for the SYMBOL-X optics. (B) A second coating chamber of the same model: the Linear Ion Source is installed in the chamber upper part. The evaporation device (e gun + crucibles) is located in the cavity visible in the lower part of the image .

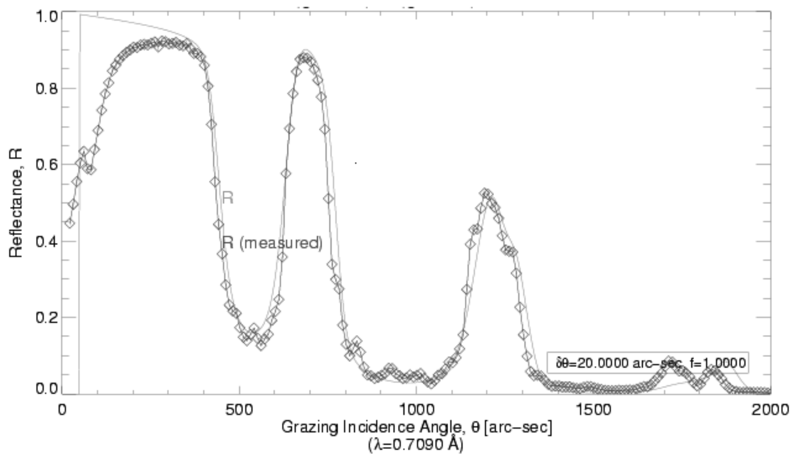
3. Ni/C MULTILAYER MIRRORS

As a first step of the project multilayer coatings with constant period have been deposited by e-beam evaporation in order to test the feasibility of the process. The first multilayer prototypes were deposited on superpolished silicon wafers without ion-assistance. Although the e-beam deposition without assistance is commonly believed to be a not optimal deposition method to produce multilayer coatings, we have indeed obtained very encouraging results and we foresee to start soon the deposition on mandrel with the ion assistance.

An example of deposited Ni/C multilayer has been characterized in X-ray reflectivity at the two standard energies of 8.05 keV and 17.4 keV. The reflectivity (see fig. 6) at the first peak turns out to be very good (95% at 8.05 keV). The model is well fitted with a roughness of 4 Å assuming a C density of 1.6 g/cm^3, and 3 Å assuming the C bulk value (2.3 g/cm^3). A further improvement of the film smoothness may be expected by the use of the ion assistance. The 2nd peak dispersion indicates the presence of a drift in the layer thickness: this is confirmed by the Transmission Electron Microscope performed on a multilayer section (see fig. 7), that showed, indeed, a very promising film quality as the layers are amorphous and homogeneous. The absence of crystallites in the structure will allow a stress reduction, which is very important to coat optics which have to operate in the extreme orbital environmental conditions. The period instability may be caused by evaporation rates or density fluctuations: special care is now devoted to the stability of the process conditions. It is worth noting that a part of the reflectivity enhancement may be caused by the lower value of the C density, which increases the contrast density absorber/spacer.



(A)



(B)

Fig.6: X-ray reflectivity test at 8.05 keV (A) and 17.4 keV (B) performed on a Ni/C multilayer with 19-bilayers. The reflectivity profile is fitted with IMD. The first peak reflectivity is over 93% @ 8.05 keV. In both cases there is an evidence of layer thickness instability: the period drift has been strongly reduced in the successive multilayers. The roughness value inferred from the model is about 3 Å, assuming the bulk value density for the Carbon (2.3 g/cm^3).

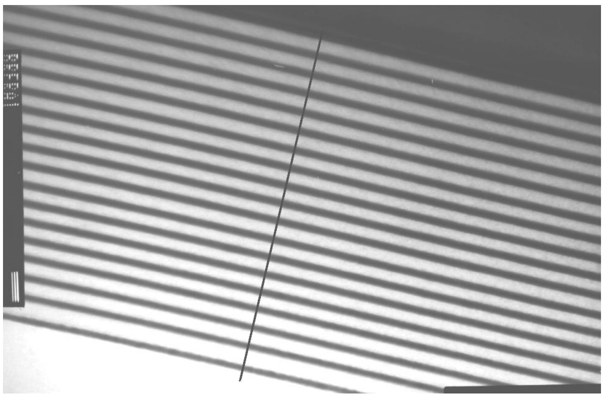


Fig.7: TEM section of the X-ray measured Ni/C multilayer (taken at CNR-IMEM, Parma, Italy). Overall thickness: 219 nm. The Ni layers are the dark bands, the C or Si are the grey bands. The layers are completely amorphous and homogeneous.

4. Pt/C MULTILAYER MIRRORS

The choice of the couple for multilayer optics for hard X-rays telescopes is a fundamental step: our current design foresees the deposition of 150 Pt/C graded bilayers in order to cover a wide spectral range in reflectivity. We have chosen the couple Pt/C because of:

1. the high contrast density which reduces the number of necessary bilayers,
2. they have similar CTEs (Pt: 8.8×10^{-6} K^{-1}, C: 7.1×10^{-6} K^{-1}) which reduces greatly the thermal stress which arises in every layered structure,
3. C is a spacer with a minimal photoelectric absorption coefficient
4. the couple Pt/C has a good chemical stability and it is suitable to make stable, abrupt interfaces,
5. the very high K-edge energy of the Pt allows a good reflectivity up to 80 keV (so a large reflectivity band is allowed and the telescope is sensitive also in correspondence to e.g. the ^{44}Ti line at 68 keV in Supernova Remnants.

A number of Pt/C multilayer, flat test prototypes were deposited on Silicon Wafers returning very high peak reflectivities: the 8.05 keV X-ray reflectivity showed well defined and narrow peaks, with reflectivities close to 83% (see fig. 8). The roughness derived from the fit is very good (3 Å, starting from a Silicon wafer substrate). Moreover, the Pt has deposited at a density close to its bulk value (21.1 g/cm^3) while the Carbon deposits at only 1.6 g/cm^3, which returns a good interface reflectivity. Incoming TEM sections will provide us a direct measurement of the stack parameters.

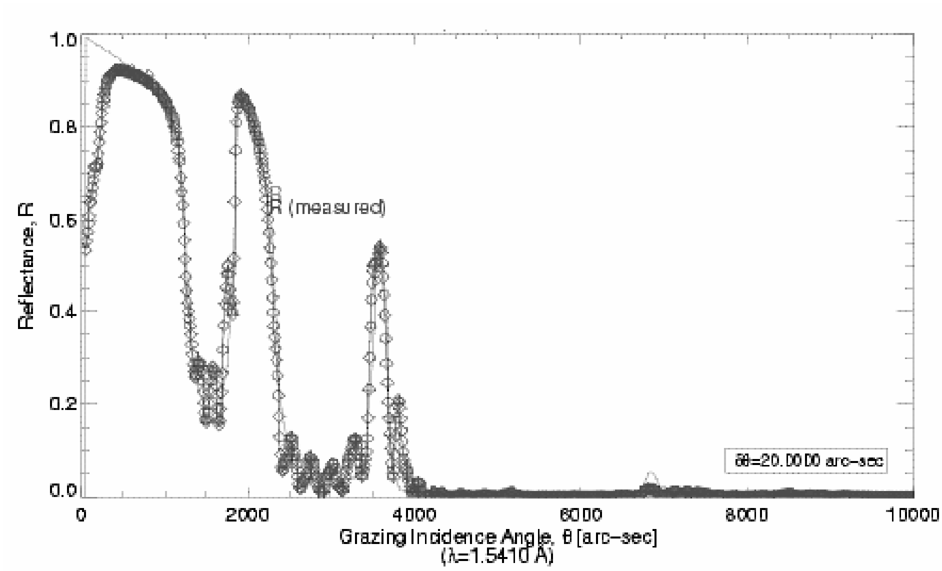


Fig. 8: A 8.05 keV reflectivity scan of a 20-bilayer Pt/C multilayer on flat Si wafer substrate. The period is about 9.3 nm and $\Gamma \sim 0.33$, modeled with a roughness value $\sigma = 3$ Å. The peaks are clearly defined as the period is better kept constant.

5. REPLICATED Pt/C MULTILAYER MIRRORS

A very important step of our development is the verification of the feasibility of the replication process with multilayer coatings. To this aim we have deposited 20 Pt/C bilayers on a superpolished flat Ni Kanigen sample, the same material which is used to produce the mirror master mandrels. The coating has been Ni electroformed and replicated: the result is a transfer of the multilayer structure on the electroformed Ni mirror (see fig. 9). The reflectivity scan shows a clear constant period multilayer, with clear and narrow peaks.

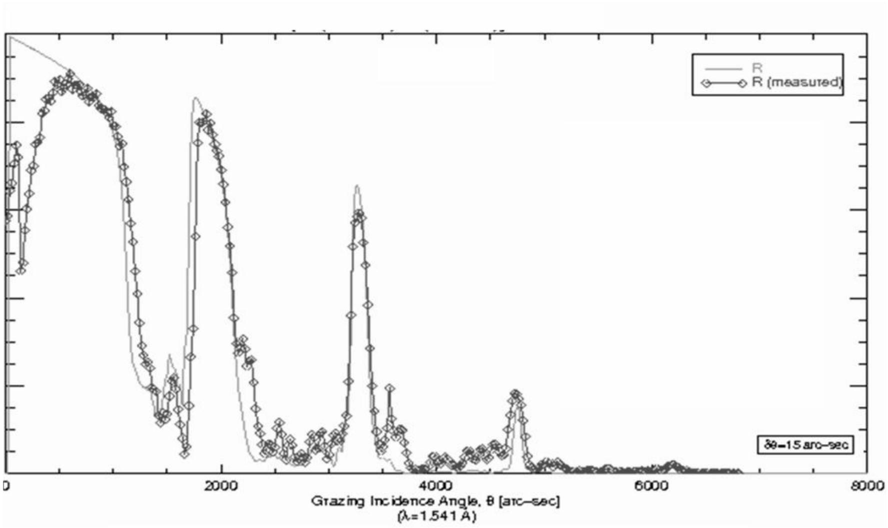


Fig. 9: A 8.05 keV reflectivity scan of a 20 bilayer Pt/C multilayer, after Nickel electroforming and replication. The multilayer was previously evaporated on a flat superpolished Nickel Kanigen sample. The multilayer structure was preserved after the replication.

6. CONCLUSION AND FINAL REMARKS

The deposition technique based on e-beam deposition for multilayers we are developing looks very promising. A great advance is expected in the next depositions by the increase of the bilayer number on a rotating mandrel, and from the ion assistance utilization. The final aim is the production of a graded multilayer, high reflectance mirror shell replicated from a superpolished mandrel with 280 mm of diameter.

An important spin-off application of this work is the deposition of Mo/Si multilayers mirrors for EUV lithography for nanoelectronic components. A number of flat Mo/Si prototypes has already been deposited with e-beam and the achieved roughness (see fig. 10) is very good (3.1 Å), without ion assistance. The future developements will aim to improve the surface roughness in order to achieve a very effective EUV reflectance.

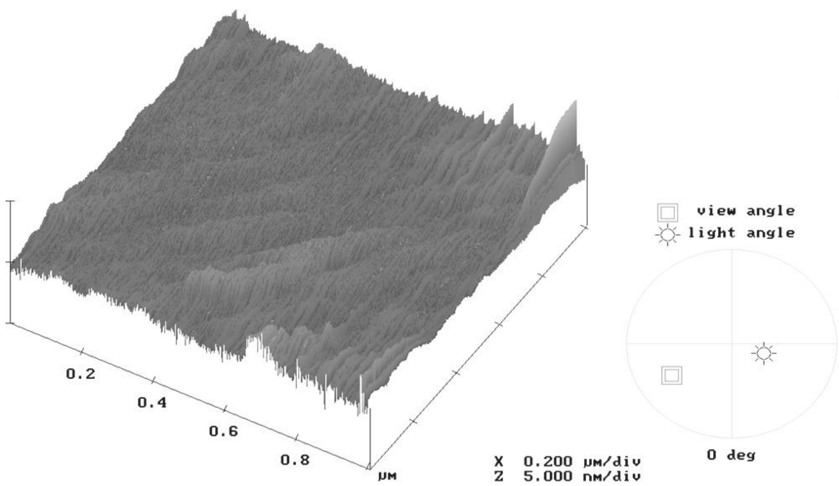


Fig. 10: An AFM map (1 micron scan) of a Mo/Si multilayer sample deposited on Silicon Wafer (20 bilayers). The surface roughness is 3.1 Å, very near to the substrate roughness value.

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REFERENCES

1. O. Citterio, G. Bonelli, G. Conti, E. Mattaini, E. Santambrogio, B. Sacco, E. Lanzara, H. Brauninger, and W. Burkert, "Optics for the X-ray imaging concentrators aboard the X-ray astronomy satellite SAX", *Appl. Opt*., **27**, 1470 (1988).
2. D. N. Burrows, J. E. Hill, J. A., Nousek, A. A. Wells, A. D. Short, R. Willingale, O. Citterio, G. Chincarini, and G.Tagliaferri, "Swift X-Ray Telescope", *SPIE Proc*., **4140**, 64 (2000).
3. P. Gondoin et al., "Calibration of the first XMM flight mirror module I – image quality", *SPIE Proc*. **3444**, 278 (1998)
4. G. Pareschi et al. "The HEXIT (High Energy X-ray Imaging Telescope) balloon-borne mission", Proc. of the 16th ESA Symposium on European Rocket and Balloon Programmes and Related Research", St. Gallen (CH) 2-5 June 2003
5. O. Citterio, P. Cerutti, F. Mazzoleni, G. Pareschi, E. Poretti, P. Laganà, A. Mengali, C. Misiano, F. Pozzilli, and E. Simonetti, "Multilayer optics for hard X-ray astronomy by means of replication techniques", *SPIE Proc*., **3766**, 310 (1999).
6. Citterio et al., "Development of of soft and hard X-ray optics for astronomy: progress report 2 and considerations on material properties for large-diameter segmented optics of future missions", *SPIE Proc*., **4496**, 23 (2001)
7. O. Citterio, M. Ghigo, F. Mazzoleni, G. Pareschi, L. Peverini, "Development of soft and hard X-ray optics for astronomy", *SPIE Proc*., **4138**, 43 (2000).
8. S. E. Romaine, A. M. Hussein, J. Everett, A. M. Clark, R. J. Bruni, P. Gorenstein, M. Ghigo, F. Mazzoleni, and O. Citterio, "Application of multilayer coatings to replicated substrates", *SPIE Proc*., **3444**, 564 (1998).
9. G. Pareschi, V. Cotroneo, "Soft (0.1 - 10 keV) and hard (>10 keV) x-ray multilayer mirrors for the XEUS astronomical mission", *SPIE Proc.* **5168,** 53 (2004)
10. G. Pareschi, et al., "Nickel replicated multilayer optics for soft and hard X-ray telescopes", *SPIE Proc.*, **4012,** 284 (2000)
11. E. Zoethout et al., "Stress Mitigation in Mo/Si Multilayers for EUV Lithography", *SPIE Proc.*, **5037**, 872 (2003)
12. T. R. Culver et al. "Developements in Ion Assisted Multilayer Coatings", *SPIE Proc.*, **1848**
13. D. G. Stearns, D.P. Gaines, D.W. Sweeney and E.M. Gullikson, "Nonspecular x-ray scattering in a multilayer-coated imaging system", Journal of Applied Physics, **84**, 2 (1998)
14. D. G. Stearns et al., "Defects from substrate particles depend on the sputter deposition process", Solid State Technology, nov 2000, 95
15. E. Spiller, "Soft X-ray optics", *SPIE Press*, 1994