

Multilayer coatings for Synchrotron Radiation

V.V. Kondratenko, E.N. Zubarev, Y.P. Pershyn*, I.A. Kopylets⁺, A.Yu. Devizenko, L.E. Konotopsky, V.A. Sevryukova, V.S. Chumak

X-ray Optics Group at National Technical University “Kharkiv Polytechnic Institute”, Kyrpychova Street, 61002 Kharkiv, Ukraine

*pershyny@ukr.net +iakop@ukr.net

Our group has extensive experience in the fabrication and study of multilayer coatings, which are mainly used in X-ray optics as multilayer X-ray mirrors (MXMs).

These are conventional MXMs based on

- **Be** (Ti/Be),
- **B** (Mo/B, Mo/B₄C, Sb/B₄C, W/B₄C),
- **C** (Si/C, Co/C, Ni/C, W/C, Cr/C),
- **Mg** (Zr/Mg, Si/Mg),
- **Si** (Sc/Si, Mo/Si, W/Si),
- **Sc** (Cr/Sc, W/Sc) and
- **Ti** (W/Ti),

as well as heat- and radiation-resistant MXMs: Si/Mg₂Si, Mo₂B₅/B₄C, MoSi₂/Si, WC/Si, CrB₂/C, etc.

We have fabricated

- mirrors,
- monochromators,
- collimators,
- polarizers,
- objectives,
- diffraction gratings,
- supermirrors etc.

We have studied

- thermal and radiation stability of MXMs;
- early stage of reaction between layers at sub-nanometer level;
- have made estimation of thickness, composition and density of reaction products.

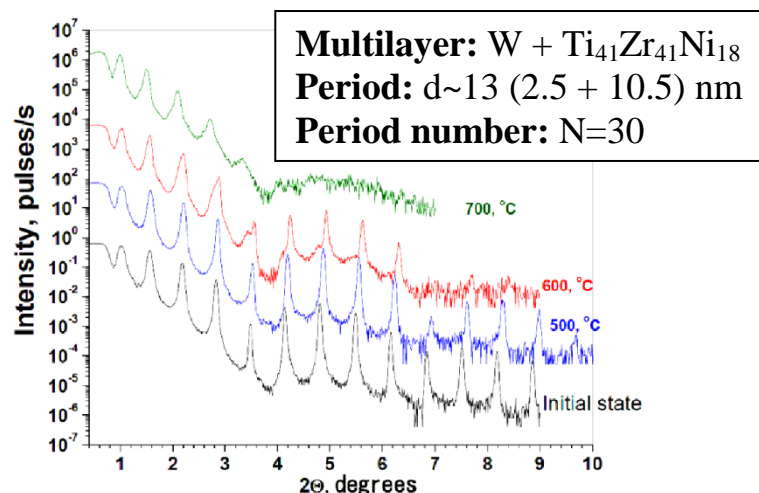
The following are some recent results on application of fabricated multilayers.

Multilayers (MLs) for radiation-resistant materials in nuclear and thermonuclear power engineering

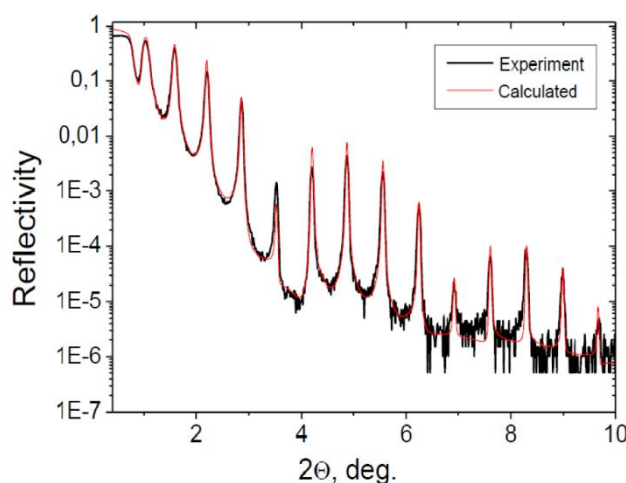
ML: **W + TiZrNi** (quasi-crystal)

W – small sputtering rate, high melting threshold, high heat conductivity

Quasi-crystal (QC) – reduces the impact of hydrogen on tungsten and steel



Small-angle X-ray reflectivity ($\lambda=0.154$ nm) for the **W/QC ML** ($d=13$ nm, $N=30$) in the initial state and after annealing at **500°C**, **600°C**, and **700°C** in vacuum.



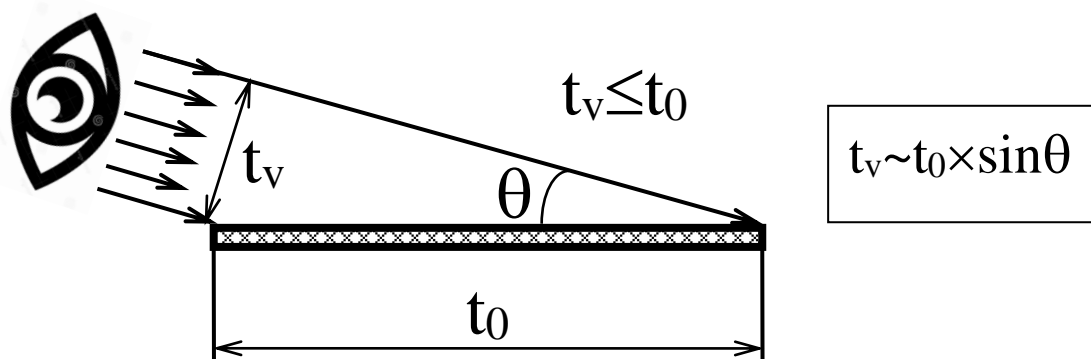
Measured and calculated small-angle X-ray reflectance ($\lambda=0.154$ nm) for the **W/QC ML** ($d=13$ nm, $N=30$) after annealing at **500°C**

Results of calculations ($T=500^\circ\text{C}$)

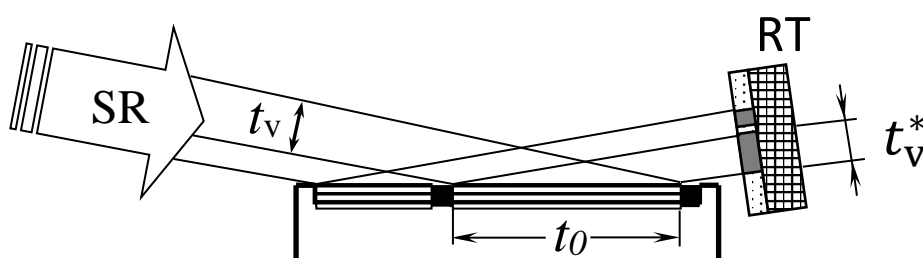
Layers	Materials	Thickness nm	Density g/cm ³	Roughness nm
top layer	W	2.57	16.9	0.44
ML (30 periods)	Ti ₄₁ Zr ₄₁ Ni ₁₈	10.26	6.3	0.37
	W	2.57	17.8	0.32

Reference: O.Yu. Devizenko, I.A. Kopylets et al. “Multilayer tungsten/quasi-crystal Ti-Zr-Ni systems as promising materials of protective elements a fusion reactor”, Problems of Atomic Science and Technology, 2 (150), 28-33 (2024).

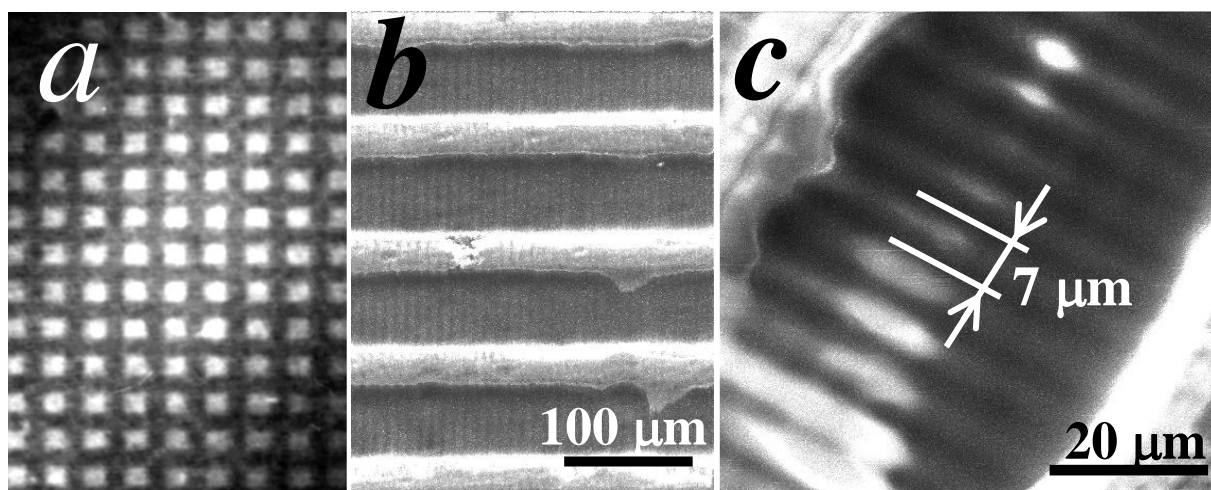
Reflective multilayer (ML) X-ray masks



A layout showing the principle of one-dimensional compression



Schematic drawing of a reflective X-ray mask and its application for normal exposure of the recording element surface (SR – synchrotron radiation, RT – recording tool).

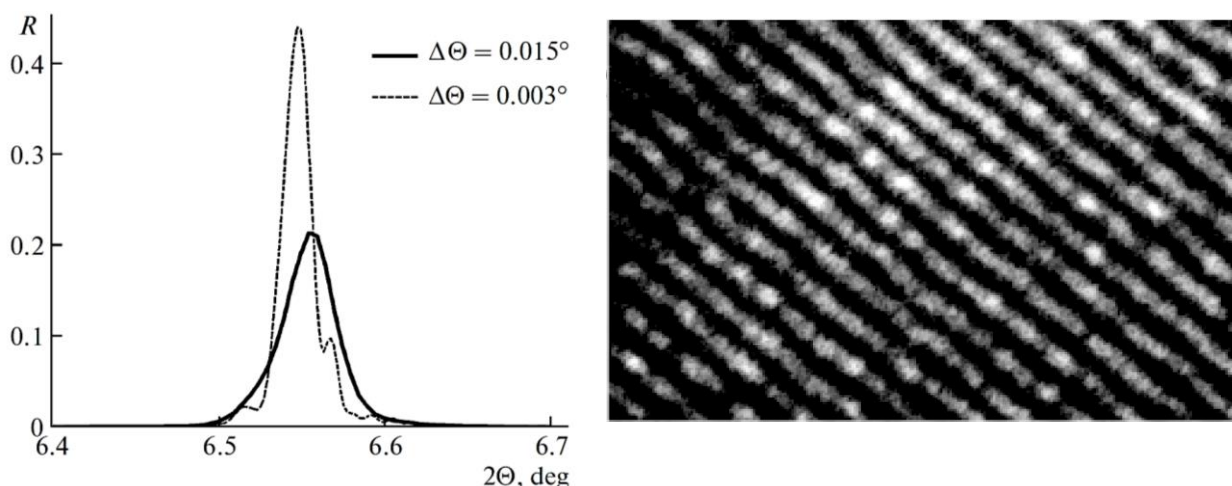


(a) Electron microscopy grid (rectangular openings of $\sim 50 \mu\text{m}$). (b) SEM image of exposed photoresist SU-8 using WC/Si ML mask with a pattern of (a). The pattern shrinkage is ~ 14 ; $\theta = 4^\circ$; $\lambda \sim 0.35 \text{ nm}$; the mask-photoresist distance is $\sim 40 \text{ mm}$. General view shows 4 periods in horizontal direction. (c) An enlarged image of the imprint part from (b). The measurements are made at the MAX II synchrotron (Lund, Sweden).

Reference: V.S. Chumak, S. Peredkov et al. “Reflective X-ray masks for X-ray lithography”, J. Micromech. Microeng. v. 34, 045008 (10pp) (2024).

High-resolution multilayer X-ray mirrors

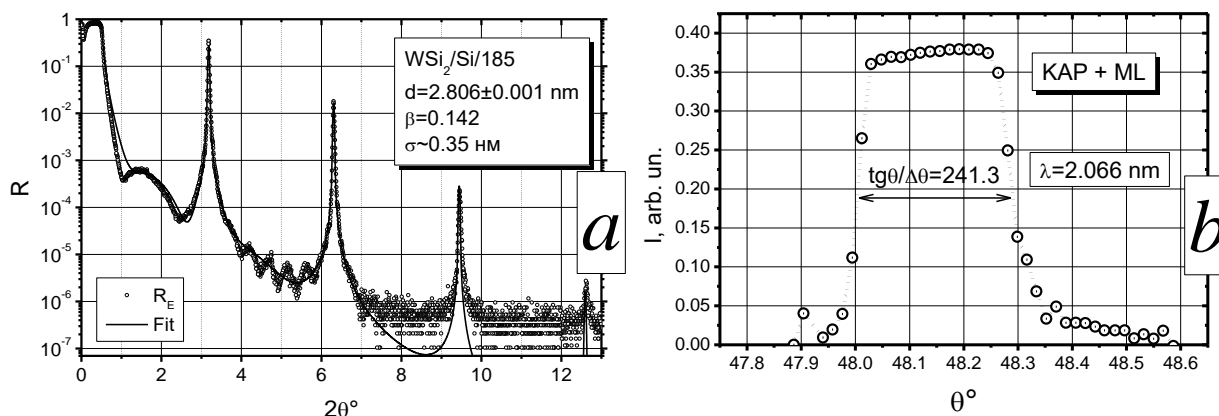
ML: W/B₄C; period $d=t_W+t_{B_4C}=1.35$ nm; $\beta=t_W/d\sim 0.4$; $\lambda=0.154$ nm; $\lambda/\Delta\lambda\sim 200$.



Reflectance of the 1st diffraction maximum for a W/B₄C ML ($d=1.35$ nm) recorded with CuK α_1 radiation ($\lambda=0.154$ nm) at an incident beam divergence ($\Delta\theta$) of 0.015° and 0.003° (left). Cross-sectional TEM view of a 1.35-nm W/B₄C ML (right).

Reference: I.A. Kopylets, V.V. Kondratenko, E.N. Zubarev, and D.V. Roshchupkin, “Formation of short-period multilayer W/B₄C compositions”, Technical Physics, v. 57, No. 12, pp. 1709–1715 (2012).

ML: W/Si; period $d=t_W+t_{Si}\approx 2.81$ nm; $\beta=t_W/d\sim 0.14$; $\lambda\sim 2.1$ nm; $\lambda/\Delta\lambda\sim 240$.



(a) Measured small-angle X-ray reflectogram (circles) and fitting curve (line) for W/Si ML with thin W-layers ($\lambda=0.154$ nm). **(b)** Reflection curve of W/Si ML (period $d\sim 2.8$ nm) at ω -scan with synchrotron radiation ($\lambda=2.066$ nm) using organic KAP crystal as a monochromator.

Reference: Y.P. Pershyn, A.Yu. Devizenko, V.V. Kondratenko, H. Modrow, F.-J. Hormes, “Structural and X-ray optical characteristics of W/Si multilayer X-ray mirrors,” Metallofiz. Noveishie Tekhnol., v. 38, No. 3, p. 367-388 (2016).

Thank you
for your attention