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Functional nano-structured tungsten based coatings for systems for energy production.

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Outline

The stability of heterophase interfaces between metal systems, their kinetic, structural, and thermomechanical properties are a matter of concern for high demanding applications involved in the development of technological coatings for the first wall materials in fusion reactors and their prototypes (ITER). We would like to discuss preliminary results of X-ray experiments on model coatings made of tungsten, in particular the problems related to strain inhomogeneity in metal films on heterophase substrates. These analyses of nanostructured thin layers can be performed using laboratory grazing incidence diffraction, allows the accurate extraction of quantitative relevant information about the structure (strain and atomic positions) and the microstructure (crystallite size and microstrain), selectively probing the material on a depth of few nanometers.

Phase analysis

Residual stress analysis

Crystallite size and strain determination

Study of the anisotropy in the lattice deformation

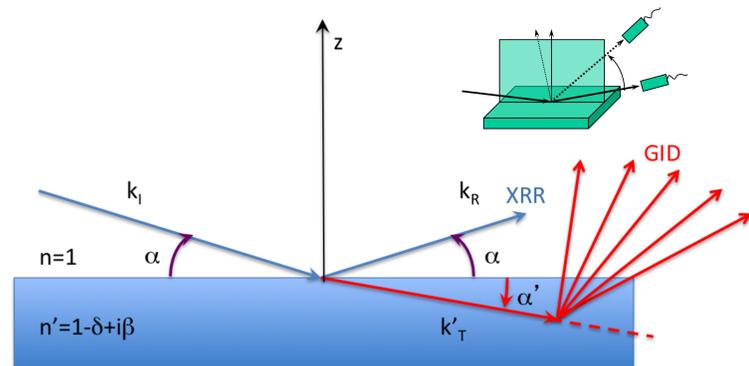
Investigation of gradients of microstructure parameters vs depth

Snell, Fresnel & XRD

$$\begin{cases} n \cos \alpha' = \cos \alpha \\ (a_I + a_R) n \sin \alpha' = (a_I - a_R) \sin \alpha \end{cases}$$

$$n = 1 - \delta + i\beta \equiv 1 - \frac{\alpha_c^2}{2} + i\beta; \alpha_c^2 \equiv \left(\frac{4\pi}{k^2}\right)\rho, \beta = \frac{\mu}{2k}$$

$$\begin{cases} \left(1 - \frac{\alpha_c^2}{2} + i\beta\right) \left(1 - \frac{\alpha'^2}{2}\right) = \left(1 - \frac{a^2}{2}\right) \rightarrow \alpha'^2 = \alpha^2 - \alpha_c^2 + 2i\beta \\ \left(1 + \frac{a_R}{a_I}\right) \alpha' = \left(1 - \frac{a_R}{a_I}\right) \alpha \rightarrow R \equiv \frac{a_R}{a_I} = \frac{\alpha - \alpha'}{\alpha + \alpha'} \end{cases}$$

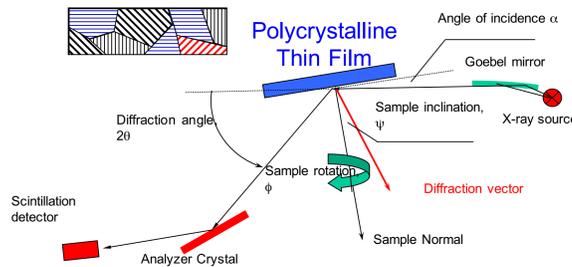


Differences between the specular reflectivity (XRR) and diffraction signals (GID).

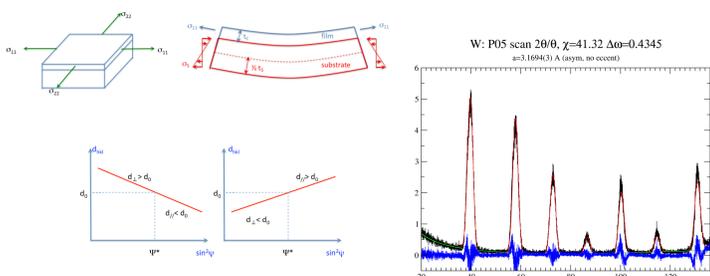
$$1 + R(\alpha) = T(\alpha) \rightarrow T(\alpha) = \frac{2\alpha}{\alpha + \alpha'}$$

$$T(\alpha) = 2 \frac{\alpha}{\alpha_c} \text{ for } \alpha \leq \alpha_c$$

$$T(\alpha) = \frac{2\alpha / \alpha_c}{\alpha / \alpha_c + \sqrt{(\alpha^2 / \alpha_c^2 - 1)}} \text{ for } \alpha > \alpha_c$$



Inhomogeneous strain in films



The regular ψ -scan method unfortunately probes a volume that changes as a function of y , averaging out the strain in the film, and preventing a mapping of the local strain

Measuring the local strain in glancing geometry

$$\psi = \alpha - \theta(hkl)$$

ψ angle should be re-encoded in glancing geometry

$$\tau = \frac{\sin \alpha \sin(2\theta - \alpha)}{\mu [\sin \alpha + \sin(2\theta - \alpha)]}$$

The depth τ probed by X-rays is now a function of the impinging angle

$$\langle \epsilon_{\varphi\psi} \rangle = \frac{\int_0^t \epsilon_{\varphi\psi}(z) \exp\left(-\frac{z}{\tau}\right) dz}{\int_0^t \exp\left(-\frac{z}{\tau}\right) dz}$$

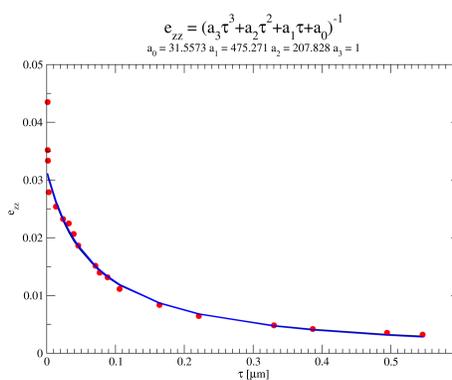
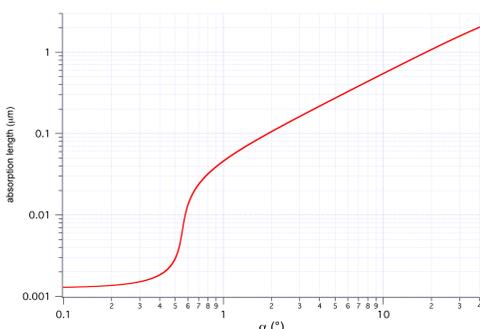
The average strain in a film of thickness t is still averaged by the way we measure (Fredholm's integral)

$$\langle \epsilon_{\varphi\psi} \rangle = \frac{1}{\tau} \int_0^\infty \epsilon_{\varphi\psi}(z) \exp\left(-\frac{z}{\tau}\right) dz = \frac{1}{\tau} \mathcal{L}\left(\epsilon_{\varphi\psi}(z), \frac{1}{\tau}\right)$$

If t is larger than the probed depth τ the Fredholm's integral becomes a Laplace transform that can be inverted, thus providing the value of the local strain at each value z in the film

$$\epsilon_{\varphi\psi}(z) = \mathcal{L}^{-1}\left(\tau \langle \epsilon_{\varphi\psi} \rangle, z\right)$$

Snell's law adapted to XRD: it provides the encoding of the probed depth information by the impinging beam angle



Depth profile of residual stresses in W films can be obtained using GID. A limitation of the method is that the material should be quasi-isotropic (untextured).

This requirement can be further relaxed if the anisotropy ratio of the material is small, that is the case of many metals like W and Mo. The result shows that the strain is almost uniform in most of the film but it rapidly increases close to the surface. In a layer below the surface ($< 100\text{nm}$) the strain increases very fast. A small part of this effect seems related to the presence of a very thin oxide layer (thickness of about 1 nm).

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Benefits

The analysis of the structures and microstructures of nanostructured thin layers can be performed using laboratory grazing incidence diffraction, provided accurate corrections are performed to handle the instrumental broadening effects related to the experiment geometry for an impinging beam close to the critical angle. These procedures allow the accurate extraction of quantitative relevant information about the structure and the microstructure, selectively probing the material on a depth of few nanometers.

Grazing incidence X-ray diffraction can be a workhorse technique for deriving crystallite size in nanoscale systems due to its non-destructive character, the fast data collection and the relative simplicity of the experimental setup. This information is relevant for an accurate description of nanocrystalline systems prepared as thin films and it can significantly improve the knowledge of their structure-properties relationships.

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