

Coefficient of thermal expansion of nanostructured tungsten based coatings assessed by substrate curvature method

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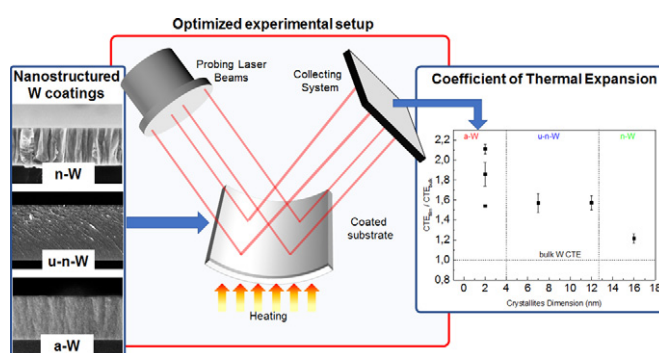
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HIGHLIGHTS

- Coefficient of thermal expansion and residual stresses of coatings measured by optimized optical implementation of the substrate curvature method
- Different nanostructures obtained by PLD result in different residual stresses and coefficient of thermal expansion.
- Nanocrystalline tungsten coatings show a higher compressive residual stress than amorphous like ones.
- The coefficient of thermal expansion is strongly correlated to the crystallites dimension.
- Amorphous like tungsten coatings have higher coefficient of thermal expansion than nanocrystalline tungsten ones.

GRAPHICAL ABSTRACT



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ABSTRACT

The in plane coefficient of thermal expansion (CTE) and the residual stress of nanostructured W based coatings are extensively investigated. The CTE and the residual stresses are derived by means of an optimized ad-hoc developed experimental setup based on the detection of the substrate curvature by a laser system. The nanostructured coatings are deposited by Pulsed Laser Deposition. Thanks to its versatility, nanocrystalline W metallic coatings, ultra-nano-crystalline pure W and W-Tantalum coatings and amorphous-like W coatings are obtained. The correlation between the nanostructure, the residual stress and the CTE of the coatings are thus elucidated. We find that all the samples show a compressive residual state of stress that decreases as the structure goes from columnar nanocrystalline to amorphous-like. The CTE of all the coatings is higher than the one of the corresponding bulk W form. In particular, as the grain size shrinks, the CTE increases from $5.1 \cdot 10^{-6} \text{ K}^{-1}$ for nanocrystalline W to $6.6 \cdot 10^{-6} \text{ K}^{-1}$ in the ultra-nano-crystalline region. When dealing with amorphous W, the further increase of the CTE is attributed to a higher porosity degree of the samples. The CTE of the coatings is also investigated as function of materials stiffness: when stiffness decreases, the CTE increases.

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1. Introduction

The objective of this work is the measurement of the residual stresses and of the coefficient of thermal expansion (CTE) of tungsten

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(W) coatings of different nanostructures, obtained by exploiting the versatility of the laser ablation technique (Pulsed Laser Deposition, PLD), and to investigate the correlation between these properties and the nanostructures.

It is well known that coatings are subjected to *residual stresses*, already present at the end of the deposition process. These stresses have two main origins. *Intrinsic stresses* are due to the deposition process itself, depend on the deposition conditions and on the mismatch of the properties between the coating and the substrate materials (e.g. lattice parameter) [1], and are developed during the deposition. When the sample is then cooled down from the deposition temperature to the room temperature, *thermal stresses* are typically developed, due to a thermal expansion mismatch between the coating and the substrate; these stresses also depend on the elastic properties of the deposited and the base materials. When the coated components operate at variable temperatures, additional thermal stresses are generated. All these stresses, typically intensified at the film-substrate interface, can lead to coating failure, by either cracking or delamination. Predicting and monitoring these stresses are crucial to guarantee the operational integrity of the coated devices. This requires the knowledge of the elastic moduli and the coefficient of thermal expansion (CTE) of the materials. In the case of coatings this cannot be taken for granted. Firstly, because the thermomechanical properties, which depend on the specific film structure and morphology, can be significantly different from the ones of the corresponding bulk form, and depend on the deposition process. Secondly, because, for coatings, direct measurement can be a challenging task.

A wide range of techniques is available to investigate the elastic properties of coatings; namely, nanoindentation [2,3] and various acoustic based techniques [4], including Brillouin spectroscopy [5–8], while little is known about the CTE of films. The standard techniques adopted to measure the CTE of bulk materials (e.g. dilatometry [9,10]) are usually not viable for coatings. Several unconventional techniques have been proposed, such as X-ray diffraction [11–13], ellipsometry [14] and different optical based techniques [15]. Among them, the optical implementation of the substrate curvature (SC) technique has been shown to be one of the most promising methods [13,16–20]. This method exploits laser beams to detect changes in the curvature radius of the coating-substrate system upon temperature variations [21]. The CTE of the coating can be then deduced if the CTE of the substrate and the elastic properties of both the film and the substrate are known (see Section 2).

W coatings are of particular interest in a wide range of technological applications, such as microelectronic and optoelectronic devices, absorption layers in X-ray lithography [22–24], and nuclear fusion reactors [25–27]. In this work we exploit the versatility of the PLD technique to deposit W-based coatings of different nanostructures, in order to investigate the relationship between nanostructure and properties, namely CTE and residual stresses. In particular, by tuning several process parameters (e.g. background gas pressure during deposition, laser fluence on target), PLD allows to grow both mono-elemental and multi-elemental coatings with tailored nanostructures, from amorphous to nanocrystalline, and morphologies, from porous to compact [5,28–30]. We deposit W-based coatings with three different nanostructures, namely (i) nanocrystalline W (n-W), (ii) ultra-nano-crystalline W (u-n-W) and (iii) amorphous-like W (a-W), and measure their properties.

Characterization is performed by an optimized SC setup that allows the CTE determination over a wide range of temperatures (25–1000 °C). An ad-hoc designed vacuum chamber is equipped with an optical system that drives a 2D pattern of parallel laser beams on the surface of the coated substrate, and detects the reflected beams by a CMOS sensor. The beam positions allow the direct determination of the substrate-coating curvature, also as function of temperature. From curvature measurements, the residual stresses and the CTE of

the coatings are derived, under the Stoney approximation [31], for known elastic moduli, which were previously measured by Brillouin spectroscopy (BS) [5].

2. The principle of obtaining the residual stress and the CTE of the coatings

Upon a temperature variation, the mismatch in the CTE between the coating and the substrate, combined with the dilation constraint represented by the film adhesion to the substrate, leads the sample to a progressive bending. The total bending depends on the difference between the CTEs of the two materials, on their thicknesses and their elastic moduli, and obviously on the temperature itself; it is well described by the continuum mechanics theory for multilayers [32]. In the case of a bilayer formed by a film much thinner than the substrate, such that the stress within the film can be taken as approximately uniform, the stress within the coating can be expressed in terms of the bending curvature radius R as:

$$\sigma_f(T) = \frac{E_s}{1-\nu_s} \frac{t_f}{t_s^2} \frac{1}{6} \left(\frac{1}{R(T)} - \frac{1}{R_0} \right) \quad (1)$$

In Eq. (1) E is the Young modulus, ν the Poisson's ratio and t is the thickness; the sub-indexes s and f stand for substrate and film respectively. $R(T)$ is the radius of curvature at temperature T and R_0 is the initial radius of curvature at a reference temperature. Eq. (1) is often known as Stoney's equation [31,33]. If the total film stress is only due to the thermal component, it is given by:

$$\sigma_f = \sigma_{thermal} = \frac{E_f}{1-\nu_f} (CTE_f - CTE_s) \Delta T, \quad (2)$$

and, taking the derivative of Eq. (2) over temperature:

$$\frac{d\sigma_f}{dT} = \frac{E_f}{1-\nu_f} (CTE_f - CTE_s), \quad (3)$$

such that:

$$CTE_f = CTE_s + \frac{d\sigma_f}{dT} \frac{1-\nu_f}{E_f} \quad (4)$$

Once the film stress is derived from the curvature measurement by Eq. (1), Eq. (4) is exploited to derive the CTE of the coatings. Eqs. (1)–(4) are written under the assumption of temperature independent elastic moduli, and allow to find a single value of CTE. It is well known that the above properties show some temperature dependence. A more detailed approach would therefore consider the temperature dependence of the elastic moduli of both the substrate and the film and of the CTE of the substrate, and would allow to find the temperature dependence of CTE of the film. However, the measurement of the temperature dependence of the elastic moduli of the coatings is a challenging task. This type of approach would be unavoidable if the explored temperature interval approaches the melting temperatures of either the substrate or the film. In our case, the maximum temperature remains far from both the melting and the recrystallization temperatures, at which more significant variations of the elastic moduli could be expected. Therefore, neglecting the temperature dependence of the elastic moduli introduces only small inaccuracies. This is experimentally confirmed by the good linearity of the $1/R$ vs. temperature curve (see Fig. 7), after the stresses relaxation occurring in the first cycles and discussed in Section 4.3.

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