Residual Stresses in Tungsten Thin Films for Single Photon Detectors

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Abstract

The residual stress in 20 nm thick tungsten films deposited on silicon substrate by dc magnetron sputtering is investigated. The sample was held in a continuous flow cryostat, which was capable of achieving temperatures as low as 8 K. The cryostat was mounted on a goniometer to enable the angle-dispersive x-ray diffraction measurements. X-ray diffraction was used to monitor the shift of the α-W {110} Bragg reflection at room temperature and 8 K. From the shift of the {110} Bragg reflection, the total residual stress was estimated at about 6.0 GPa. After applying corrections for the thermal stress in the film, the residual intrinsic stress is estimated at 5.8 GPa.

1. Introduction

Tungsten superconducting transition edge sensors (TES) are used in different astrophysics and astronomy applications, and as single-photon detectors that are necessary in quantum information experiments.[1,2] In comparison to other photon detectors, the TES provides both the energy and the time-of-arrival of the absorbed photon instead of sacrificing one detail for the other.[3] Such detectors consist of tiny squares of thin tungsten films and operate at temperatures close to zero Kelvin. Operation at such low temperatures changes properties of tungsten from metallic to superconducting when the films reach the superconducting transition temperature (Tc). This is important because close to the superconducting transition temperature, very small thermal changes, such as the absorption of a single photon, increase the film electrical resistance. The result is the production of an electrical output signal, which directly corresponds to the detection of the absorbed photon energy.[4] In order to reduce the dark count rates (intrinsic to a TES), and increase the absorption of a TES, antireflective (AR) coatings can be used on the metal films. Such coatings include silicon oxide and silicon nitride, but such an addition changes the superconducting properties of W thin films.[1]

Mechanical, physical, and optical properties of W thin films depend on the preparation conditions,[5,6,7] the thickness of the film,[5] and the difference in thermal expansion coefficient between the film and the substrate.[8] Therefore, the residual stress will be sample specific and highly dependent on the initial preparation conditions. In general, sputtered thin metal films tend to have high residual stresses.[5]

The total residual stress in a thin film results from both the intrinsic stress and thermal stress.[9] That is,

$$\sigma_{total} = \sigma_{in} + \sigma_{th} \quad (1)$$

Intrinsic (growth) stress is due to metastable film nucleation and growth. Thermal stress depends on the difference between thermal expansion coefficients of the film and substrate. In this study the internal thermal stress and total residual stress in a W thin film are examined by x-ray diffraction (XRD). It is of interest to observe the changes in residual stress of both the W thin film grown on the Si substrate and the same film that was additionally capped by an AR coating. Here, we report on the results obtained on an uncoated film. The analysis gained from this research will aid our understanding of the changes in the superconducting properties of the W film when incorporated in a multilayer stack.

2. Experimental

2a. Film Deposition

The thin W film was deposited onto a 375 μm thick Si wafer by dc magnetron sputtering. The deposition rate was adjusted to result in a film thickness of 20 nm and the Ar pressure during deposition was 9 mTorr. The sample was found to be a mixture of α-W (bcc structure), and metastable β-W (A15 cubic structure) phases. In general, residual stress can influence the superconducting critical transition temperature, Tc, of the thin film both directly and by inducing the $\beta \rightarrow \alpha$ phase transformation. The Tc for a pure α and β phase are ~15 mK and 1-4 K respectively. The Tc of the sample was determined as ~120 mK. Therefore, our sample primarily consists of the bcc structure.

2b. XRD Measurements

A standard two-circle powder goniometer was used for XRD measurements. It was set up in the Bragg-Brentano parafocusing geometry. The goniometer had a vertical θ-2θ axis and a radius of 22 cm. The radiation was excited at 43 kV and 39 mA. The CuKα x-ray radiation was collimated by Soller slits and a 2 mm divergence slit. The diffracted beam was collimated with Soller slits and a 0.2 mm receiving slit.[10] A Ge solid-state detector and a single-channel analyzer were used to select the CuKα wavelength. This experimental setup is illustrated in Figure 4.

Measurements were taken in a step-scanning mode. The strong α-W {110} peak was scanned both at 293 K and at 8 K. The 2θ range scanned was 39.5° - 41.5°. The step size and scan time for this run were 0.02° and 25 sec respectively. The Bragg law was used to determine the lattice interplanar spacing for the crystallographic planes parallel to the sample surface. It follows that

$$2d \sin \theta = \lambda \qquad (2)$$

or

$$d = \lambda/(2 \sin \theta) \qquad (3)$$

where,

d = lattice spacing (Å)
θ = Bragg reflection position (Deg)
λ = wavelength = 1.5406 Å.

The angles and lattice spacing are illustrated in Figure 1.

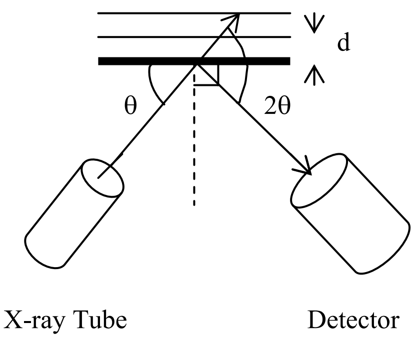

FIG. 1
X-rays exit the x-ray tube and impinge on the sample at an angle θ. X-rays leave the sample with an angle of 2θ, relative to the incident beam direction, towards the Ge detector.

2c. Stress Determination

Residual stress in thin films is due to extrinsic (thermal) and intrinsic stresses. This correlation is expressed by equation (1).

2c1. Thermal Stress

Thermal stresses in thin films are caused by a difference in thermal expansion coefficients (α) between the film and the substrate. The thermal expansion coefficient is defined through the change in length per unit temperature:

$$\Delta L = \alpha\, L_o\, \Delta T \qquad (4)$$

and

$$\alpha = \Delta L/(L_o\, \Delta T) \qquad (5)$$

where,

$\Delta L = L_f - L_o$
L_f = length at 8 K
L_o = length at 293 K
$\Delta L/ L_o$ = fractional thermal linear expansion
α = thermal expansion coefficient (K^{-1})
$\Delta T = T - T_o$ = change in temperature (K).

The length change parallel to the surface, however the x-ray measurements give the interplanar spacing perpendicular to it.

The residual stresses due to external thermal effects, σ_{th}, can be calculated from

$$\sigma_{th} = (E_f/1-\nu_f) \int_{To}^{T} \Delta\alpha \, dT' \qquad (6)$$

which can be approximated by

$$\sigma_{th} \approx (E_f/1-\nu_f)\ \Delta\alpha\ \Delta T \qquad (7)$$

where E_f is Young's modulus (410 Gpa) and ν_f is the Poisson's ratio (0.28) of the thin film.[11, 12] $\Delta\alpha$ is the difference between the average thermal expansion coefficients of the film and substrate between the temperature interval of 8 to 293 K.

The thermal linear expansion of W is larger than that for Si, which means that the W contracts more than Si from room temperature to 8 K. Because the substrate mechanically constrains the film, a Si wafer will not allow the W film to contract as much as it would without the substrate. Figure 2 uses arrows to depict the amount each material would like to contract.

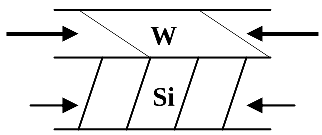

FIG. 2

W is constrained by Si at low temperature. Si, contracts less than W does from 293 to 8K, and because Si is much thicker, does not allow W to completely contract. This creates an extrinsic residual stress in the sample.

The XRD results show that the film contracts more than the substrate does. It was expected that the film would be under tensile stress because of the thickness of the film, the pressure during deposition, and by analyzing published thermal data.[5,8] The results showed that the thin W film was under tension.

2c2. Intrinsic Stress

The other contribution to the residual stress is the intrinsic stress. Such stresses are developed during deposition and are of primary interest because they affect film microstructure and properties. The intrinsic stress is difficult to calculate, but it can be determined from the total residual stress of the sample. The total residual stress can be found from:

$$\sigma_{total} = (-E_f/\nu_f)\ (\Delta d/\ d_o) \qquad (8)$$

A simple calculation yields the thermal linear expansion from the measured lattice spacing values. It follows that for an isotropic material,

$$\Delta d/\ d_o = \nu\ \Delta L/\ L_o \qquad (9)$$

where,

$\Delta d = d_f - d_o$ = change in lattice spacing (Å)
d_f = lattice spacing at 8 K (Å)
d_o = lattice spacing at 293 K (Å)
ν = Poisson's ratio

Combination of equations (8) and (9) give,

$$\sigma_{total} = -E_f\ (\Delta L/\ L_o) \qquad (10)$$

Finally, using equations (1), (7), and (10), it follows that,

$$\sigma_{in} = -\ E_f\ [(\Delta L/\ L_o) + 1/(1-\nu_f)\ \Delta\alpha\ \Delta T] \qquad (11)$$

Because W is isotropic, this leads to a straightforward analysis of the extrinsic stress through XRD.

2d. X-ray Peak Profile Analysis

XRD peak patterns were analyzed using the computer program WinFit!.[12] This program allows for a range selection, manual peak insertion, and the choice of the Pearson VII asymmetric function for curve fitting. A fit for each peak is displayed (if more than one is selected), as well as the final curve fitted to the scan. The numerical results of interest yield the peak position and maximum intensity.

3. Results and Discussion

3a. X-ray Diffraction

XRD measurements confirmed that the film was primarily composed of the α-W structure. The α-W {110} peak was selected for analysis and XRD scans were taken at room temperature (293 K). The cryostat with the sample was subsequently cooled down to 8 K and the measurements were repeated. Comparisons between the two scans portray a definite peak shift with the decrease in temperature. (See Figure 3.)

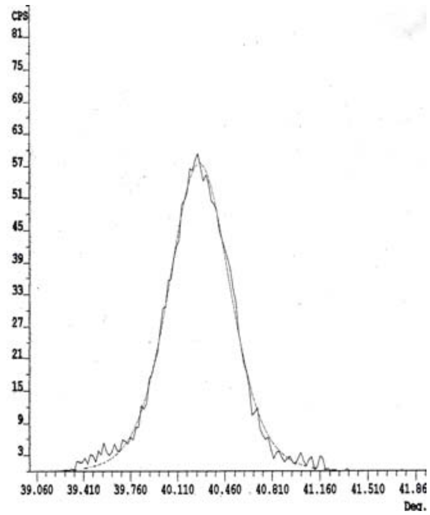

FIG. 3a

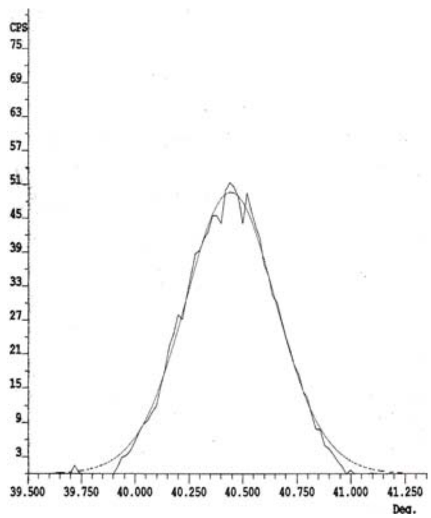

FIG. 3b

Intensity as a function of 2θ for W thin films at 293 K (3a) and 8 K (3b).

Using WinFit![12] to individually fit each peak, precise 2θ values were obtained, and corresponding lattice spacing were calculated using equation (3). All of the measured and calculated values are shown in Table 1.

TABLE 1: XRD Peak Measurements

	W	**8K**	**293K**
2θ (deg)		40.4444	40.2708
d (Å)		2.2284788	2.237684
$\Delta d/ d_o$		-0.0041137	
ν		0.28	
$\Delta L/ L_o$		-0.0146918	

3b. Extrinsic Residual Stress

Equation (7) was used to calculate the thermal (extrinsic) stress in the sample due to the change in the temperature. Previously published thermal expansion coefficients[12] were used in the calculations. Between 293 and 8 K, the average α_W was 2.25 $x10^{-6}$ K^{-1} and the average α_{Si} was 1.3 $x10^{-6}$ K^{-1} . The extrinsic residual stress was therefore estimated as 150 MPa.

3c. Intrinsic Residual Stress

The intrinsic residual stress was investigated by taking the difference between the total and thermal residual stresses. The total residual stress was calculated using equation (10) and the changes in d spacing from table 1. The W thin film had a total residual stress of 6.0 GPa. Thus, intrinsic stress was dominant in the W thin film.

The thin film was expected to be in a state of tensile stress. Using equation (11) it was established that the intrinsic residual stress in the observed W thin film was 5.8 GPa, and thus the thin film was under tension.

Residual stresses in thin W films depend on many factors, and though such stresses have been extensively studied, understanding and applying the results can be challenging. Both the intrinsic and extrinsic stresses contribute to the total residual stress of a thin film. The extrinsic stress, or thermal stress, for W and Si was expected to be very small, which is one of the reasons that this combination is used in devices.

4. Conclusions and Future Research

In this study, the intrinsic residual stress in 20 nm thick W thin films was examined using XRD. The intrinsic residual stress was found to be tensile and of the magnitude of about 5.8 GPa. It is an order of magnitude larger than the thermal residual stress due to a difference in thermal expansion coefficient between W and Si. The residual stress will provide useful information for understanding the changes in the superconducting properties of the thin film when the AR coating is applied.

In order to establish a thorough understanding of the residual stress in W thin films, measurements and analysis of the intrinsic stress present in the sample will be additionally investigated using the XRD $\sin^2\psi$ method. In addition to this, systematic error analysis needs to be done to verify the validity of measured interplanar spacing. Finally, an XRD analysis of a W thin film grown on a Si substrate and capped with a AR coating will need to be completed using the same methodology, in order to fully understand the changes that occur both when the coating is applied.

Acknowledgements

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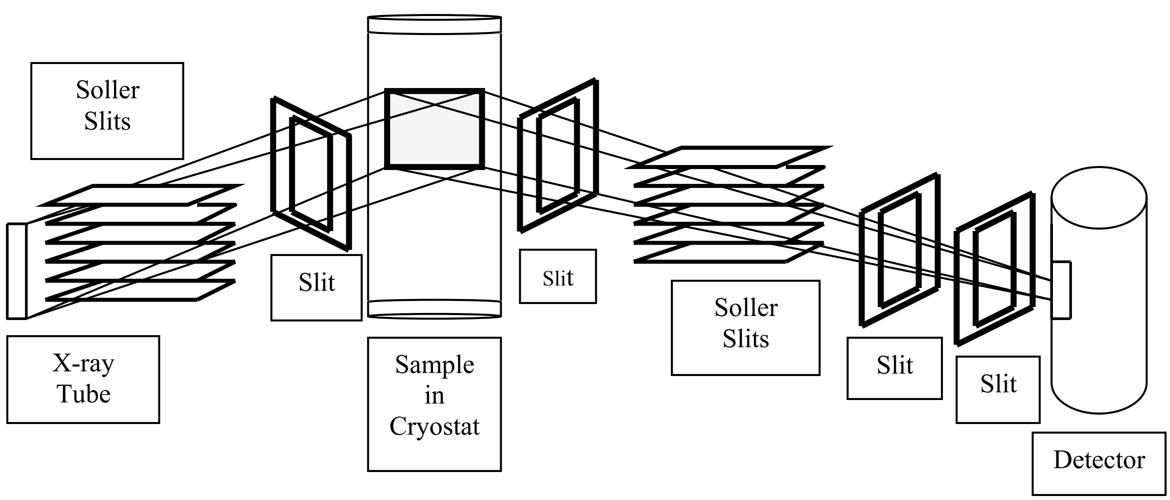

FIG. 4
Pictorial of X-ray path form tube to detector.

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