Characterization of sputtered hafnium thin films for high quality factor microwave kinetic inductance detectors

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Abstract
Hafnium is an elemental superconductor which crystallizes in a hexagonal close packed structure, has a transition temperature $T_C \approx 400$ mK, and has a high normal state resistivity around $90 \mu \Omega$ cm. In Microwave Kinetic Inductance Detectors (MKIDs), these properties are advantageous since they allow for creating detectors sensitive to optical and near infra-red radiation. In this work, we study how sputter conditions and especially the power applied to the target during the deposition, affect the hafnium $T_C$, resistivity, stress, texture and preferred crystal orientation. We find that the position of the target with respect to the substrate strongly affects the orientation of the crystallites in the films and the internal quality factor, $Q_i$, of MKIDs fabricated from the films. In particular, we demonstrate that a DC magnetron sputter deposition at a normal angle of incidence, low pressure, and low plasma power promotes the growth of compressive (002)-oriented films and that such films can be used to make high quality factor MKIDs with $Q_i$ up to 600,000.

Keywords: kinetic inductance detectors, hafnium, sputter deposition, superconducting resonators

1. Introduction

Microwave Kinetic Inductance Detectors (MKIDs) [1] are superconducting detectors capable of measuring the arrival time and energy of single photons. Detection of individual photons from infrared [2–4] to x-rays [5] have been demonstrated using MKIDs. In these detectors, energy deposited onto the superconducting film breaks Cooper pairs into excited electrons (quasiparticles) whose presence affects the total kinetic energy of the super current. The superconducting film is patterned into a resonant circuit, and changes in the kinetic energy of the super current manifest as changes in surface inductance, $L^s$, of the resonant circuit. The resonant circuit is probed near its resonant frequency and the phase and amplitude of the transmitted probe signal are monitored for changes as quasiparticles are created.

A small superconducting gap energy, $2\Delta_0$, and a large kinetic inductance fraction are needed to make sensitive, responsive MKIDs. A small gap allows more quasiparticles to be created when a photon hits the detector resulting in greater sensitivity. A large surface inductance, $L^s$ a material property related to the kinetic inductance via a geometric proportionality constant, is valuable since the response of the detector is proportional to the fraction of kinetic inductance to total inductance in the resonator.

According to the BCS theory of superconductivity [6], the transition temperature of the superconductor, $T_C$, is related to the gap energy as $2\Delta_0 = 3.52k_B T_C$. Assuming that the film
is thin compared to its penetration depth, it can be derived [7] that

$$L_x = \frac{h}{\pi} \frac{\rho_n}{\Delta_0} = \frac{h}{\pi} \frac{R_n}{\Delta_0}$$ (1)

where $t$ the film thickness and $\rho_n$ ($R_n$) is the normal state resistivity (sheet resistance) just prior to the superconducting transition. Note that the easily measurable $T^C$ can be substituted for $\Delta_0$, assuming BCS superconductivity. Choosing a superconductor with a large $L_x \geq 10\hbar/\rho$ (high $\rho_n$) and low $T_C \leq 1$ K (low gap) allows us to design lumped element resonators in the 4–8 GHz band sensitive to optical and near infrared photons.

Only a few superconductors are known to possess relatively low $T^C$, relatively high $\rho_n$, and make high quality resonators. Examples include TiN [2, 8], PtSi [3] and granular aluminum [9]. Our interest in hafnium thin films stems from the fact that it has a low $T^C$ around 400 mK, a high $\rho_n$, and, as we have shown in a previous paper, can create resonators with $Q^s$ up to 190000 [10]. Moreover, a material with a larger $L_x$ can be made thicker for a given detector responsivity, which allows for more flexible resonators designs and could help to reduce noise due to phonon escape from the superconductor [11]. Additionally, as an elemental superconductor, we can reasonably expect to obtain highly uniform films across the wafer, in contrast to reactively sputtered TiN where the nitrogen gas flow distribution affects the superconducting gap uniformity of the deposited material [12, 13]. PtSi also has its disadvantages as Pt is an expensive material and the process requires a very precise control of the Pt and Si deposition rates as well as a fine control of the in-situ annealing of the bi-layer.

In order to improve hafnium MKID array performance, the research we present here is focused on better understanding the material properties and deposition conditions best suited for the fabrication of high quality factor hafnium resonators. This work adds to the existing literature in hafnium thin films developed for superconducting tunnel junction detectors [14, 15] and transition edge sensors [16].

2. Hafnium film deposition

Hafnium films are deposited on 100 mm diameter a-plane sapphire substrates in a load-locked ultra-high vacuum AJA ATC-2200 sputtering system with a typical base pressure of $6 \times 10^{-10}$ Torr. The argon working gas cylinder has a certified purity of 6 N and it goes through an additional gas purifier before entering the sputter chamber. The hafnium sputter target used has a nominal purity of 3N5 (99.95 wt%), but the actual ingot chemistry was measured to have 0.16 wt% zirconium; additionally, impurities measuring above 1 ppm-wt are 55 ppm-wt oxygen, 16 ppm-wt carbon, 6.6 ppm-wt niobium, and 1.4 ppm-wt iron. Two different gun-substrate configurations were used for the hafnium film deposition (figure 1). One where the sputter gun is tilted at an angle of 19.2° off normal and the distance between the center of the target and the closest point to it on the substrate is 15.4 cm. Another, where the target is concentric with the substrate and there is 19.5 cm vertical distance between their centers. For each configuration, the substrate is rotating. All guns have a balanced magnet configuration and 75 mm diameter target. The deposition pressure and DC power are varied within the ranges 1–15 mTorr and 20–660 W respectively. Argon flow is kept at 30 sccm for all depositions. The deposition rate for each condition is measured with a step profilometer and is used to deposit 125 nm thick hafnium films. This thickness gives the resonators on our fabrication mask the total inductance required to achieve their designed resonant frequency.

3. Hafnium film characterization

Superconducting transition temperature. The superconducting transition temperature for each sample is measured in a Leiden Cryogenic dilution fridge with a base temperature of 60 mK, and they are given in the figure 2. The $T^C$ of the films are found to be between 435 mK and 355 mK depending on the deposition conditions. We measure a residual-resistivity ratio
found to be better than \( \sim 20\% \) despite many optimizations in the deposition process [18]. We believe that our sheet resistance uniformity is improvable by using a larger sputter target and positioning the substrate farther away from it during deposition, but it will be necessary to check that such changes do not negatively impact the resonator quality factors.

The kinetic inductance of the films is calculated using equation (1) and the measured \( R^s\) and \( T^s\); figure 2 shows the value of the surface inductance for the different deposition conditions. It is found to be in the range 15–20 pH/\( \square \).

Crystallography. The crystallographic orientations and texture of the films are determined with a Panalytical XPERT MRD (Materials Research Diffractometer) PRO equipped with a Pixcel 3D x-ray diffractometer detector. The x-ray diffraction (XRD) patterns of 125 nm thick hafnium films grown on sapphire at 5 different plasma powers at a normal incidence are shown in figure 3. A diffraction pattern for an hafnium film deposited with the tilted gun configuration is also given (in red). The analyses were made in the 20 – \( \omega \) mode between 30\( ^\circ \)–37\( ^\circ \). The patterns show that the films are in the hexagonal phase [19, 20] and are strongly oriented along the (010), m-plane, and (002), c-plane, axes since only those two peaks are visible (no other peaks are visible when the analysis is performed on the full 20–80\( ^\circ \) except the one from the sapphire substrate). The two peaks are shifted toward lower angles from the theoretical peak position (vertical dashed lines at 32.3\( ^\circ \) and 35.5\( ^\circ \)) which indicates that the films are under compression. Assuming our hafnium is predominantly polycrystalline and only oriented along two axes, the fraction of (010) and (002) crystal in the film can be computed by comparing the intensities of the peaks. Figure 3 shows the percentage of (010)-oriented crystals in the film as a function of the plasma power used for the deposition. We show that by changing the plasma power during the deposition we are able to control the preferred orientation of the hafnium from a 3\% (100)-oriented film at 20\( W \) to 61.1\% at 660\( W \).

Assuming the diffraction peaks are only broadened due to the size of the crystallites, the Scherrer formula, \( D = \frac{K\lambda}{\beta} \cos(\theta) \) [21], can be used to estimate their sizes. \( D \) is the size of the crystallite, \( K \) is the shape factor and typically equals 0.9, \( \lambda = 0.1546\text{ nm} \) is the x-ray source wavelength, \( \beta \) is the full width at half maximum of the peak in radian and \( \theta \) is the Bragg angle in radian. For the films deposited at a normal incidence, the crystallites’ dimensions range from 12 nm to 30 nm, and their dimensions are about 40 nm for the hafnium deposited at an angle of 19.2\( ^\circ \).

We performed a texture measurement to understand the rotational orientation of the crystallites, which cannot be ascertained by the 20 – \( \omega \) peaks alone. In a pole figure, the intensity of the signal is proportional to the number of grains oriented along a particular direction, and a peak at the center corresponds to crystal oriented perpendicular to the surface of the film. The very weak signal in the center of the (010) pole figure, figure 3(a), shows that for the hafnium deposited at a gun angle of 19.2\( ^\circ \) the crystal planes are not growing perpendicular to the substrate but tilted by 55\( ^\circ \). The (002) pole figure, figure 3(b), exhibits a strong peak at the center meaning that the crystallites are growing perpendicular to the substrate. In
Figure 3. Left $2\theta - \omega$ scans of 125 nm thick hafnium films on sapphire deposited at different gun tilts and plasma powers. The patterns are shifted on the logarithmic intensity scale for clarity. Top right The ratio of intensity of the (010) peak to the (002) in percent as function of the deposition power is given. Bottom right (a) and (b) Pole figure of (010) and (002) for the film deposited in the tilted gun configuration. (c) and (d) Pole figure of the (010) and (002) for the film deposited at 660 W at a normal incidence. The intensity colormap is shown in a square root scale for clarity.

Figure 4. Average stress in the hafnium films deposited on a 4'' wafer for the two deposition configurations. Left At a fixed deposition plasma power of 100 W and within the 1–5 mTorr pressure range. Right At a deposition pressure of 2.5 mTorr and for deposition power between 20 W and 660 W. The error bars represent the standard deviation of the measurement.

The average stress of a 125 nm thick hafnium film deposited on a 4'' diameter substrate is determined by measuring the change in radius of curvature prior to and after deposition by using a Tencor Flexus tool. Figure 4 shows the stresses obtained for a wide range of deposition conditions. At a fixed deposition plasma power of 100 W and for the tilted gun geometry, the stress changed from compressive to tensile when the pressure is increased. At a normal angle of incidence, only compressive films can be deposited within the 1-5 mTorr pressure range. The stress for the films deposited at 2.5 mTorr and a power between 20 and 660 W are always found to be compressive. In the case of the films deposited at a 90° angle, the stress in the films is between -800 and -1700 MPa. Compressive hafnium films have been previously been studied by, Turner et al [22], and have been used for the fabrication of the high performance MKID array by Zobrist et al [10].

At low deposition pressures or short target-to-substrate distance, sputtered atoms impinge upon the substrate without having lost energy to scattering events. The impinging atoms can have enough energy to create local defects as a result of the shockwave created by their impact in a phenomena known as peening. Peening provides a mechanism for the growth of dense, compressive films [23–25]. While we have not measured the density of our films, their tendency to have gigapascal scale compressive stress, our sputter pressure, and the target-substrate geometry lead us to suspect that peening plays a dominant role in the growth of our films.
Curiously, we find that the stress of our films as a function of the sputter power follows two different trends depending on the sputter gun configuration. Increasing the powers gives decreasing stress for the normal incidence configuration while the opposite is true for the tilted gun configuration. Meanwhile, we find that the only way to obtain tensile films is to increase the deposition pressure. We expect that in this situation the impinging atoms reach the substrate with an insufficient energy to move to sites forming a closely packed atomic arrangement resulting instead in a porous, void-rich microstructure [26].

**Film Morphology.** The surface and cross section of hafnium films are shown in figure 5. Columnar growth is observed in all films and the crystallites are contained within the columns. Measuring from the SEM images, the crystalline grains are approximately 32 nm for the tilted gun sputter configuration and 17 nm for the normal configuration. This accords with the estimate based on the Sherrer formula.

We also remark that the hafnium films readily charge up when imaged in the SEM. This is unusual for a metal. The film deposited at 90° shows some dark areas on the surface that have not been identified.

**Optical Film Properties.** The room temperature optical reflectance, transmittance and absorbance have been measured within the 200 nm–2000 nm range with a Shimadzu UV3600 spectrometer equipped with an integrating sphere. The spectra are plotted in figure 6. This room temperature measurement allows us to set an upper limit to the quantum efficiency of the material for use in detectors sensitive to these wavelengths. Because these measurements are made at room temperature, we adjust the film thicknesses to match the resistivity of a 125 nm thick film just above the superconducting transition temperature. Using RRRs of 1.6 and 1 for hafnium and PtSi the spectra are acquired from films 245 nm and 60 nm thick, respectively. We compare Hf to PtSi because we have previously established that PtSi is suitable for making high quantum efficiency MKIDs sensors [3, 4]. We find that the Hf has a lower transmittance than PtSi, but they have similar reflectance and absorbance.

**4. Resonator fabrication**

To make the MKIDs, 125 nm thick Hf films first undergo a dehydration bake at 135 °C. We then spin on a 80 nm thick layer of DUV-42P6 adhesion promoter followed by a 800 nm thick layer of UV6-0.8 imaging photoresist. The resist is patterned with an MKID test geometry using an ASML PAS 5500/300 DUV stepper which has a resolution better than 200 nm for dense patterns. The resist is developed in AZ MIF 300 and the adhesion promoter layer is removed in the etch chamber as part of hafnium etch. The hafnium is etched in a PlasmaTherm SLT 700 reactive ion etcher (RIE) which has 4½ parallel plates and water cooling to 20 °C for the substrate. (We note that wafer temperature never exceeded 41 °C during the etch and deposition for all deposition conditions.) The etch recipe is BCl3 and Cl2 flowed at 60 sccm and 40 sccm, respectively; 5 mTorr process pressure; 100 W RF power at 13.56 MHz. The etch rate was measured with a step profiler and is found to be 0.41 nm/s and uniform over a full 4½ wafer. The resist is then removed with solvents, gold bond pads are added (via a lift-off process) on the side of the chip to ensure a good thermalization of the devices to the bath temperature and finally diced. The chip dimensions are 13 × 13 mm.

The etch profile is an important parameter to control to make MKID devices. The smallest feature on our lumped element test geometry is the gap between the meandered inductor trace which is 500 nm wide. Vertical walls are needed to avoid
Table 1. Summary of the properties of 125 nm hafnium films deposited at 2.5 mTorr argon pressure, with different plasma powers and gun angles. Resistivity (with standard deviation over the wafer) and $t^C$ (± 5mK thermometer calibration) at the center of the wafer, preferred crystal orientation and stress (with standard deviation over the wafer) in the films. The average and standard deviation of the internal quality factor $Q^i$ for MKIDs made out of the specific films are given. The device studied in Zobrist et al [10] was fabricated at JPL. That hafnium was DC magnetron sputtered at normal incidence using a 6″ target at 2.5 mTorr. We scaled the 350 W power used on the 6″ target to what it would be on a 3″ target keeping power density fixed and it gives a power of 125 W.

| Target Power [W] | $R^C$ [Ω□]       | $R^a$ uniformity | $T^C$ [mK] | $L^S$ [pH□] | $t_{(002)}^C$ [%] | $t_{(002)}^S$ [%] | $\sigma$ [MPa] | $Q^i$          |
|------------------|-------------------|------------------|------------|-------------|-------------------|-------------------|------------|--------------|
| Normal configuration |
| 660              | 7.30 ± 0.28       | 11.6 %           | 385        | 15.3        | 61.1 %            | −722 ± 50         | −325 000±30 000 |
| 350              | 7.60 ± 0.32       | 12.2 %           | 364        | 17.0        | 48.7 %            | −820 ± 42         | 174 000±17 000  |
| 100              | 8.25 ± 0.33       | 11.3 %           | 403        | 16.5        | 19.3 %            | −1314 ± 50        | 405 000±22 000  |
| 40               | 10.20 ± 0.25      | 7.3 %            | 410        | 20.6        | 3.7 %             | −1408 ± 51        | 605 000±80 000  |
| 20               | 8.35 ± 0.22       | 8.1 %            | 435        | 15.8        | 3.0 %             | −1432 ± 48        | 515 000±95 000  |
| Tilted configuration |
| 350              | 6.46 ± 0.13       | 7.0 %            | 355        | 13.2        | 2.0 %             | −1154 ± 71        | 16 800±2000    |
| Normal configuration - Zobrist et al [10] |
| 125°             | 8.11 ± 0.26       | 12.1 %           | 395        | 16.7        | 38 %              | −1236             | 190 0000      |

Figure 7. SEM images of the profile of a hafnium film etched by reactive ion etching in a mixture of BCl$_3$/Cl$_2$.

shorts. Figure 7 shows SEM images of the etch profile of an hafnium film deposited on a silicon substrate. Our etch process produces sidewalls that are approximately 110°. Occasional etch residue, which degrades MKID performance [27], is observed in the etch trenches and on the etch side wall.

The resonators are designed to have resonant frequencies around 5 GHz and to have a spacing of 2 MHz. In order to compare the performances of each chip, nine resonators with a simulated coupling quality factor $Q^C$ of 40 000 are analyzed.

5. Resonator measurements

The chips are cooled down in a BlueFors dilution refrigerator to a base temperature of ~ 12 mK. Resonators are first found manually with an Agilent 20 GHz vector network analyzer. Then, an automated readout system which uses a synthesizer and digitizer is used to find the power at which each resonator starts to bifurcate [28]. This system then acquires the complex IQ transmission data by sweeping the microwave probe through the resonance from low to high frequency using a probe power that is 14 dBm below bifurcation. The resulting IQ curves are fitted with the model proposed by Khalil et al [29]. From the fitted curves, the intrinsic quality factor of each resonator is computed.

An MKID test chip was fabricated out of the Hf film produced by each sputter condition. In figure 8, we plot the average $Q^i$ of the nine resonators described in the previous section for these sputter conditions. All the hafnium MKIDs made from films deposited in the tilted gun configuration gave low quality resonators with $Q_i < 17000$. We are able to obtain $Q^i$ as high as 600 000 in the normal incidence gun configuration, at low power.

We find that our hafnium resonators have $Q^i$ on par with that measured from PtSi$^i$ [17]. The same one layer MKID test geometry was used for both the PtSi$^i$ and Hf film resonators.

Figure 8. Except for the Zobrist et al point, each data point is the average internal quality factor from nine resonators on a single test chip made from a Hf film deposited at the power indicated on the x axis. From chip to chip, the same nine resonators are included in the average. The quality factor for the film deposited at an angle of 19.2° is also shown. The error bars represent the statistical standard deviation of the internal quality factor of the resonators. The Zobrist et al point comes from the average over 10 resonators (also around 5 GHz) of a slightly different resonator geometry from a different MKID test chip mask.
6. Conclusions

In conclusion, we have studied the properties of sputtered hafnium films and MKIDs made from them while varying several sputter deposition parameters. We have shown that we are able to control the crystal orientation by growth conditions. Sputtered hafnium grows along the (010), m-plane, and (002), c-plane, crystal planes, and deposition at a normal angle of incidence promotes crystal growth perpendicular to the substrate. By decreasing the deposition power, the film tends to be mostly (002)-oriented.

When the hafnium is sputtered at a normal angle of incidence to the substrate, we are able to make MKID resonators with internal quality factor \(Q_i\) as high as 600,000. We have observed this effect in two different sputter systems. Such \(Q_i\) is about a factor of 3 higher than the \(Q_i\)s achieved in our previous hafnium MKID paper [10]. We have shown that resonator \(Q_i\) increases with decreasing the sputter power; this coincides with an increase in the \(002\) plane to \(010\) ratio, compressive film stress, and smaller crystals grains as measured by scanning electron microscope. When the hafnium is deposited at a gun angle of 19.2°, 17,000 is the highest resonator \(Q_i\) we can obtain.

The high \(Q_i\) resonators have a superconducting transition temperature of 435 mK and a surface inductance of 16 pH/cm. From our optical reflectance measurement, we expect the quantum efficiency of sputtered Hf MKIDs for the detection of photons in the UVOIR band to be on par with PtSi\(^2\) resonators [17] which we currently use in our detector arrays [3, 4]. The sheet resistance uniformity obtained with hafnium is in line with PtSi\(^2\) and routinely better than TiN\(^3\) and methods for improving it are clear, e.g. using a larger target and placing the substrate farther away from it.

Our research has not determined why a certain combinations of sputter gun angle, pressure, and power yield to high \(Q_i\) MKID resonators, but we have identified film characteristics, such as crystal structure, texture, film stress, and grain size which correlate with high \(Q_i\).

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