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Deposition of Tungsten Thin Films by Magnetron Sputtering for Large-Scale Production of Tungsten Based Transition-Edge Sensors

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Abstract To cope with the foreseen demand for tungsten based TESs in the current and future phases of the CRESST experiment, we investigated the possibility to implement a reliable, simple and reproducible fabrication method using sputtering. In this contribution we present the method under development for tungsten deposition using conventional magnetron sputtering with xenon as sputtering gas. TESs with transition temperature (T_c) down to 15.0 mK have been obtained with transition width smaller than 1.0 mK. We also give a first assessment on the reproducibility of the process and present the potential for tuning of the T_c.

Keywords TES, Tungsten, Sputtering, Xenon, Dark matter, CRESST

1 Introduction

Superconducting transition edge sensors (TESs) are used as ultra sensitive thermometers to measure temperature changes in the range of μK. They have attracted quite some attention in the last few decades due to their sensitivity. Such sensors are widely adopted in low temperature detectors and are used in many different applications e.g. particle physics, astroparticle physics and astronomy.

1.1 TESs in CRESST Experiment

In the framework of the CRESST experiment (Cryogenic Rare Events Search with Superconducting Thermometers) which is a direct dark matter search experiment, tungsten TESs are used as sensing elements. Detectors in CRESST consist of a target crystal which has a mass of a few tens of grams, combined with a TES. These detectors are operated as cryogenic calorimeters at ~ 10 mK. The main detection channel is scattering of hypothetical

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dark matter particles off the target crystal nuclei. The deposited energy leads to a measurable temperature rise in the temperature sensor. We use tungsten-based TESs since tungsten has a $T_c \sim 15.4\ mK$ (in the bulk form) which is the closest to our design goal [1]. Taking into account that the measured signal is inversely proportional to the heat capacity of the sensor $\Delta T = \frac{\Delta E}{c}$, using tungsten at such low temperatures ensures a relatively low heat capacity of the sensors electronic system $c\ [J/(g\ K)] \approx 7.94 \times 10^{-6} \times T$ [2]. However, materials in the form of thin films often do not behave as they would do in the bulk form. The exact T_c and the width of the transition ΔT_c can always scatter above or below the standard value which is a strong function of the thin film quality [3].

So far we produce tungsten films in an optimized e-beam evaporation system. However, evaporation is a time consuming process (one week/run), involves high temperatures ($\sim 600\ ^{\circ}C$), has low throughput (1-4 sample/run) and has low yield. For the next CRESST upgrade a few hundreds of detectors are foreseen. Such a number of detectors will require more production capacity than that of our evaporation machine. Therefore a promising candidate to substitute evaporation is sputtering.

1.2 Superconducting tungsten thin films

The transition temperature as well as the transition shape (steepness) is what defines the quality of the detector. There are many aspects that may affect the transition. Unfortunately a full understanding of what governs the T_c in thin films has not yet been reached. Some probable causes that may affect the T_c will be explained in more detail in the following.

1.2.1 Impurities

The presence of impurities in tungsten films is known to strongly alter the T_c or completely suppress the transition. Contamination of films can originate from the the growth process itself. During film growth any volatile contaminant or gas impurity can be easily incorporated in the film. In addition the substrates may carry organic or inorganic residuals or water from the cleaning process. Even in highly controlled environments, tungsten may react with substrates at high temperatures or even at room temperature. Some common tungsten compounds have shown superconductivity at relatively higher temperatures. Since β-W [4] is known to be an unstable phase and to exist sometimes in grown tungsten films, less pure W films are also likely to be called β-W due to the difficulty of identifying amorphous or nanocrystalline films. Among impurities that cause higher T_c with respect to the nominal value we mention: W-silicide ($T_c < 2.84K$) [5], W-carbide ($T_c < 10K$) [6], W-nitride ($T_c < 4.85K$) [7], W-oxide ($T_c < 132K$) [8]. In contrast to these impurities, ferromagnetic impurities shift the T_c to lower temperatures or completely suppress it [9].

1.2.2 Argon retention

Retention of argon atoms in tungsten films cannot be easily avoided in sputtered films in Ar atmosphere. The consequent Ar amount is usually $\sim 1-2\%$ depending on the deposition parameters[10]. Such unavoidable impurity may cause no direct effect on tungsten films since there is no chemical reaction involved. However such retention may deteriorate the film quality. As a matter of fact, it has been shown that noble gas impurities like He may stimulate superconductivity in naturally not superconducting elements e.g. palladium in the form of a thin film when it was irradiated with low energetic He^{+} ions [11].

1.2.3 Stress

Stress is known to shift the T_c of superconductors (e.g. see [12]). Residual stress is an intrinsic feature of grown thin films and can be either tensile or compressive. It arises from the fact that, as the film grows, a columnar micro-structure dominates. A competition between the growing grains, a bombardment from the reflected neutral atoms, or a lattice parameter mismatch between the substrate and the film may lead to a stressed film (for more details see [10]). Another type of stress is thermal expansion coefficient mismatch between film and substrate since the film will be operated at cryogenic temperatures, the mismatch will be relatively large. The linear thermal expansion coefficient of tungsten is lower than almost all substrates and almost four times as that of Si [13, 14], hence it is not possible to avoid such mismatch.

Our approach to overcome this obstacle is to engineer our films to have a residual amount of stress after deposition that will cancel out the thermal expansion at low temperatures. Such engineering is possible by sputtering.

1.3 Xenon as sputtering gas

Mamor et al. [16] have shown a comparison of the residual stress in films produced with Ar vs. Xe as a function of sputtering gas pressure. Based on that one can infer that a stress-free film can be deposited at a pressure around 1 *Pa* for Xe compared with 7 *Pa* for Ar. The relatively high pressure for Ar may result in a higher Ar concentration in the film, which has to be avoided. On the contrary, Xe is less likely to be trapped due to its atomic size and the relatively low sputtering pressure.

2 Experimental Methods

Tungsten films ~ 200 *nm* were deposited on single crystal Si(100) substrates with ~ 30 *nm* thermally grown SiO_2. We used a commercial sputtering device device at Max Planck Institute for Plasma Physics in Garching. Substrates where cleaved from a 6 inch wafer into (10×20) mm^2. The samples were degreased using acetone in an ultrasonic bath for 5 mins followed by IPA for another 5 mins then were dried with nitrogen gas. The machine has two cathodes in a confocal configuration with $25°$ inclination to the substrate holder and a center to center distance ~ 10 *cm*. A 3 inch tungsten target of 99.95% purity (Lesker) was used. The base pressure before deposition was $\sim 2.0 \times 10^{-6}$ *Pa*. The chamber pressure during deposition was varied between $0.6 - 2.5$ *Pa* for different runs in steps of 0.1 *Pa* while keeping all other parameters constant to study the effect of Xe pressure on T_c. Xe gas with purity of $5N.3$ was admitted to the chamber using a mass flow controller (MFC) with flow between $9 - 25$ *sccm* to adjust the corresponding pressure. 300 *W* DC was applied on the magnetron during all runs. Prior to the deposition, the samples were plasma cleaned using 50 *W* RF bias for 5 *min*. While the magnetron shutter was closed, the target was sputtered clean for 5 *min* using the same power of deposition. The deposition time was 5 mins for all runs. To accurately study the effect of one parameter (pressure) while keeping all other parameters constant and without breaking the vacuum, an in-situ substrate holder was developed which has a capacity of six samples and can expose one sample at a time to the magnetron. Gas pressure was adjusted without turning off the magnetron. The deposited films were then structured using a positive-tone photoresist into TES structure of

(6×8) mm^2. The uncovered W was wet etched. Then a 1 mm wide stripe was structured using a negative-tone photoresist and a 500 nm Au layer was deposited in a lift-off process for the purpose of thermal anchoring. A similar process was done to deposit two aluminum bias bond pads on both ends. For T_c measurements the samples were mounted on a copper holder and bonded using an aluminum wire for bias current and a gold wire for thermal anchoring. The holder was mounted in a dilution refrigerator that has a base temperature of $< 10mK$. The TESs were voltage biased using a 40 $m\Omega$ and the resistance was measured using a SQUID amplifier[17].

3 Results

Fig. 1 shows typical transition curves (R vs T) of two W-TESs deposited using 0.6 Pa Xe. The two TESs that were structured on the same substrate to study the homogeneity of the film as well as the possible scatter in the in T_c in the same deposition run. The films have a $T_c \sim 16$ mK and ΔT_c 0.4 mK. The two TESs have a difference in T_c of ~ 0.5 mK which is acceptable for our purpose.

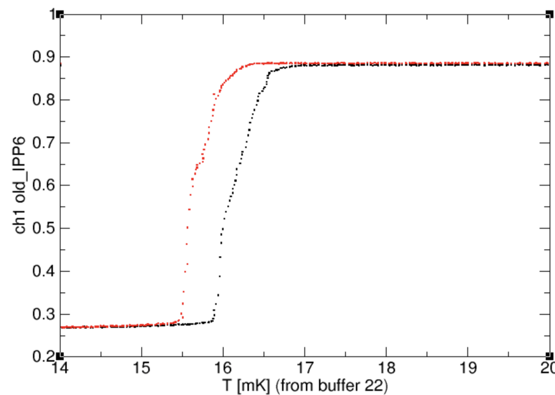

Fig. 1 A comparison between two 8 mm^2 TESs structured on the substrate. They were both biased with the same current of 1.0 μA. A shift of 0.5 mK can be seen.

Fig. 2 shows the T_c of W films vs. the Xe pressure. Despite the still low statistics, it is possible to presume a linear correlation between deposition pressure and T_c.

4 Discussion

This work was a clear proof of principle that sputtering can produce films with properties that fit the requirements of CRESST experiment. A method that is able to provide a suitable quality for large TES production has been demonstrated. Yet further investigation is needed to understand in details what governs the transition and how to improve the sensitivity of a TES. Further study of the reproducibility is under investigation in a dedicated machine at our institute. So far the films were grown on Si substrates with ~ 30 nm thermally grown SiO_2. However the next step is to study films deposited on different substrates following the

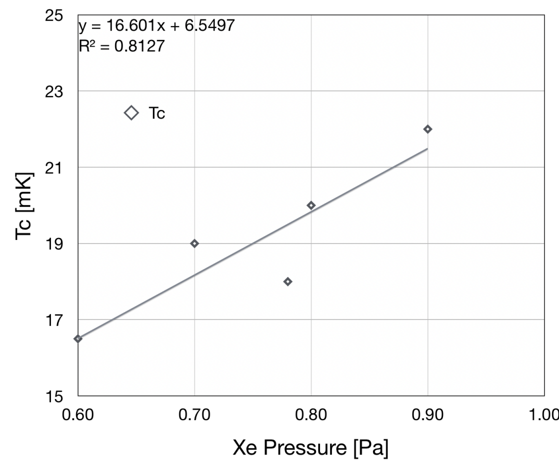

Fig. 2 T_c of films that were deposited with Xe as a function of sputtering pressure. The pressure was varied in steps of ~ 0.1 *Pa*

experimental requirements of probing a completely new parameter space towards low mass dark matter.

5 Conclusion

Sputtering W films for TES fabrication with Xenon gas showed that a TES with T_c down to 15 mk with transition width smaller than 1 mk is achievable. Tuning of T_c may be possible by controlling the Xe pressure during the sputter process.

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