

Optimizing Deposition Techniques and Parameters for High T_c YBCO Thin Films

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Abstract: In this research article the goal is to optimise the critical deposition processes and process parameters to form high-quality, superior-properties superconducting thin film. Materials like amorphous MoSi, NbN, TiN and NbTiN are used in a variety of sophisticated superconducting devices, such as superconducting nanowire single photon detectors (SNSPDs). The atomic layer deposition and the ultra-high vacuum magnetron sputtering technique were performed under highly controlled conditions to obtain the thin films. The features of the film formed with each combination of the above parameters were investigated by adjusting each parameter one at a time. The accuracy of thickness and surface topographical characterisation was done by using atomic force microscopy (AFM), structural characterisation by high resolution transmission electron microscopy (HRTEM), surface characterisation of optical properties through variable angle spectroscopic ellipsometry (VASE) and confirmation of the superconducting behaviour was done by using cryogen-free electrical methods. The results show the surfaces are smooth, crystallinity is increased and the defect density is low for optimal deposition parameters. The structural changes improve some properties of the superconductor such as its increased critical current density (J_c), sharper colossal transition and transition temperature (T_c). The observed patterns were confirmed and supported by using statistical analysis (ANOVA, regression). A thorough method for optimising the process parameters is also improved by the study, and there is a good correlation between the deposition parameters and the thin film performance.

Keywords: Superconducting Thin Films, Deposition Optimization, Magnetron Sputtering, YBCO, Superconducting Transition Temperature (T_c), Critical Current Density (J_c), Thin Film Characterization, SNSPD

1. INTRODUCTION

In view of the need for cutting-edge electrical, quantum and sensing applications, High Temperature Superconducting (HTS) thin films have played a critical role in the research and development of superconductivity (Yue, Z. 2015). Because of their very low resistance and the observation of no expulsion of magnetic with current flow, they have high attractiveness for future applications including superconducting nanowire single photon detectors (SNSPDs), quantum computing devices, microwave resonators and high speed communication devices until reaching a certain superconducting transition Temperature (T_c) (Zhao, Y. 2015). Compounds with superconducting properties, e.g., NbN, NbTiN, TiN, or amorphous MoSi, are

especially attractive material candidates due to their outstanding superconducting properties, chemical stability, and suitability for thin film processing. Careful thin film deposition and process, however, are critical to development of highly conductive properties of these materials (Maldonado, P. 2015).

Various factors, including the sputtering power, chamber temperature and pressure, gas composition and film formation time, influence the very complex physical and chemical process required to form superconducting thin films (Tran, H. D. 2016). The process of fabricating a superconductor thin film is quite complicated, involving several parameters such as sputtering power, substrate temperature, substrate pressure, gas composition and time of forming the thickness of the film, which have important roles in determining the microstructure element and film quality (Shrivastava, S. K. 2017). Variations in these properties can result in significant variations of nucleation, grain growth, crystallinity, surface shape and defect level. These features of the microstructure of the material directly influence important superconducting properties, such as T_c , J_c and electrical resistivity (Abaloszewa, I. 2020). To achieve such uniform properties as uniform film thickness, smooth surface and high crystallinity and the least defects of the film (essential factors for the high performance of superconducting devices), it is essential to carefully optimize the deposition conditions (Ohnishi, T. 2018).

1.1 Background of the Study on High T_c YBCO Thin Films

Due to their perfect diamagnetism and low resistance at relatively high temperatures, high temperature superconductors (HTS) have gained a lot of attention. Among these is a highly popular one – Yttrium Barium Copper Oxide (YBCO) with a high superconducting transition temperature ($T_c = 90$ K), and good current-carrying capacity (Arpaia, R. 2017). A high-quality YBCO thin film is vital for a variety of new technologies under development, such as superconducting electronics, power transmission systems, magnetic sensors and quantum devices (Kumar, S. 2024). For achieving the highest superconducting properties, however, the deposition method and the required process parameters, such as the substrate temperature, pressure of oxygen gas in the atmosphere, and the film thickness, are of great importance (Zhou, X. 2024). All of these variables can significantly affect the crystal structure, crystallinity and oxygen content of films. The deposition condition is therefore crucial for characterizing and optimizing the structural integrity and superconducting properties of YBCO thin films, and

to enable the development and use of devices potentially useful for reliable, effective power use (De Angelis, M. 2022).

2. REVIEWS OF LITERATURE

Gao, P. (2019) In order to produce high-quality YBCO thin films with enhanced superconducting characteristics, many research have emphasised the optimisation of pulsed laser deposition (PLD) parameters. It is reported that these factors of mainly substrate temperature and oxygen partial pressure have a significant effect on crystallinity and phase purity into the YBCO films. By using growth substrates at temperatures between 750 and 800°C and by applying a controlled oxygen environment during normal growth conditions, the proper epitaxial growth and incorporation of oxygen atoms are made possible which is required to attain a high superconducting transition temperature (T_c). Structural instability is possible at higher oxygen pressures while at lower oxygen pressures less oxygen vacancy contributes adversely to the superconducting properties. Furthermore, the deposition rate and the film's homogeneity change based on the energy density, frequency pulse, and laser. We have consistently obtained smooth film surfaces, robust c-axis texturing, and T_c values close to the bulk cuprate value of 90 K under almost ideal PLD circumstances. The findings highlight the significance of adjusting the deposition conditions to guarantee a balance between the superconducting properties and the kinetics of film development.

Li, Q. (2016) Magnetron sputtering has been thoroughly investigated as an effective technique for producing YBCO thin films, particularly for industrial and large-scale applications. Experimental research has shown that each sputtering technique, including RF and DC, can accurately regulate the film's composition and thickness. The Ar/O₂ gas ratio, sputtering power, and film formation pressure in the chamber all have a significant impact on the composition and microstructure of the film. The ideal oxidation state and crystalline structure of Y, Ba, and Cu—essential components in the development of the superconducting phase—are ensured by an oxygen-rich environment. However, if the sputtering power is too great, the film may sustain damage and re-sputtering effects, which would lower the film's quality. Additionally, it has been reported that in order to enhance T_c and restore oxygen in the lattice, post-deposition annealing is necessary under an oxygen environment. Improved alignment, defect density, and critical current density (J_c) are made possible by the optimised sputtering conditions achieved

on the resulting films. These experiments have shown that, if the right parameters are used, sputtering may also be a practical method for producing PLD-quality films.

Selvamanickam, V. (2015) The two primary techniques that should be taken into consideration as an economical way to create YBCO thin films are chemical solution deposition (CSD) and metal-organic decomposition (MOD). According to published research, precursor characteristics such precursor chemistry, concentration in solutions, and heat treatment profiles have an impact on the film's quality. Phase purity and superconducting characteristics depend on the breakdown temperature and oxygen annealing phases. In order to achieve uniform grain development and remove organic residues from melts, multi-step annealing processes including pyrolysis and crystallisation phases were advantageous. Additionally, selecting various substrates and preparing their surfaces are highly valued. Optimal processing conditions may result in T_c comparable to that attained by PLD produced films, even though the CSD techniques may initially produce films with poorer crystallinity than the vacuum-based approaches. If the process parameters can be effectively optimised to prevent errors and provide consistent superconducting qualities, CSD is a highly promising method for commercial use that is both scalable and cost-effective.

Miura, M. (2016) improved YBCO thin film deposition, sophisticated optimisation approaches such combinatorial methods and in-situ monitoring techniques have recently been investigated. Techniques such as reflection high energy X-ray diffraction (RHEED) and real-time spectroscopic ellipsometry have been used to track the growth dynamics of films and parameters during deposition. Such feedback has been demonstrated to enable fine tuning of surface morphology, crystallinity and film thickness. Also, the design of experiments (DOE), a statistical optimisation technique, and machine learning have been used to find optimum set of parameter values. The techniques have drastically cut down on trial-and-error experimentation, and increased the reproducibility. Besides, it was found that buffer layers to minimize the lattice mismatch and improve epitaxial growth are also very important. These higher state-of-the-art optimisations make films superior to their usual counterparts in terms of their excellent superconducting properties such as best T_c , strong J_c and low surface roughness. With this growing path, YBCO thin film studies can be propelled to logical and data-driven optimisation.

3. METHODOLOGY

3.1 Research Design and Experimental Approach

The current work optimises the deposition methodology using a methodical and experimental research strategy, in order to obtain a high quality thin film of a super conductor, the deposition parameters were optimized. One of the main goals is to understand the relationship between the structure, optical and superconducting properties with the deposition parameters of NbN, NbTiN, TiN and amorphous MoSi materials. For controlled experimentation, the one variable at a time (OVAT) approach was used, changing only one deposition parameter each experiment, while maintaining the remaining ones at a fixed value. With this method it was possible to measure, with accuracy, the impact of each of those factors on the film performance. All of the study procedure involved thin film deposition, multi-technique characterisation, gathering and interpretation of data. With several deposition runs carried out under the identical deposition circumstances, great care was taken to guarantee uniformity and reproducibility. Furthermore, its design is interdisciplinary which includes statistical analysis, condensed matter physics and material science etc. For the creation of a dependable thin film of superconductors for cutting-edge applications like superconducting nanowire single photon detectors (SNSPD), this methodical technique makes the optimisation process repeatable and methodical.

3.2 Materials, Deposition Techniques, and Process Parameters

Attempts have been made in experiments to synthesis superconducting thin films from various target materials with high purity such as Nb, Ti and MoSi alloys. Materials selected as suitable substrates (silicon or sapphire) were based on the suitability for superconducting applications and thermal stability. The thin films were deposited using Ultra-high vacuum (UHV) reactive DC magnetron sputtering and atomic layer deposition (ALD) was employed in the production of TiN films for thickness control. All the parameters such as sputter power, chamber pressure, substrate temperature, Ar/N₂ gas flow ratio and deposition time were carefully controlled and optimized. The target material was hit by high energy argon ions in the sputter process, which knocked out atoms from the target and deposited the atoms on the substrate to create the desired thin film coating. Even in this field of development some intriguing discoveries were made; superconducting nitrides like NbN and NbTiN were found to be formed by reactive sputtering in a nitrogen environment. Those parameters had to be carefully selected and optimized to

control the film thickness, crystallinity, phase purity, and defect density. High quality thin films with a quality suitable for application in superconducting devices as a whole were obtained.

3.3 Characterization Techniques and Measurement Procedures

Using a variety of cutting-edge characterisation techniques, the physical characteristics and a portion of the superconducting property of the deposited thin films were examined. The layer thickness and shape were investigated using Atomic Force Microscopy (AFM) and the step height measuring technique at the nanoscale. High-Resolution Transmission Electron Microscopy (HRTEM) was used to structurally characterise them, enabling a thorough analysis of the crystal structure, lattice orientation, grain size, and interface quality. The Variable Angle Spectroscopic Ellipsometry (VASE) approach was used to assess optical characteristics such as refractive index, extinction coefficient, etc. This method allowed for the precise determination of film thickness and optical constants across a wide wavelength range. Using AFM in tapping mode, high-resolution topographical pictures of the surfaces were obtained in order to further characterise their grain distribution and roughness. The electrical and superconducting characteristics were compared using a cryogen-free closed-cycle measurement device that measured resistance as a function of temperature ($R-T$) down to the cryogenic temperatures. The observations made it possible to calculate the critical current density (J_c), transition breadth, and superconducting transition temperature (T_c). A comprehensive analysis of thin film performance and quality was made possible by a combination of characterisation functions.

3.4 Data Analysis, Modeling, and Optimization Framework

Relationships between the aforementioned deposition factors and the thin-film characteristics were determined by methodically analysing the experimental data and using both analytical and statistical techniques. Mathematical models and governing equations were used to quantify important parameters such as deposition rate, layer thickness, superconducting transition temperature, and critical current density. Scatter plots were used to graphically display data from the data acquisition system, and trend analysis and ideal process conditions were identified. Regression analysis was utilised to describe the dependence of superconducting characteristics on deposition factors, and statistical techniques (descriptive statistics) were employed to evaluate consistency and repeatability of experimental data. To lessen the random fluctuation that may lead to trend observations, several of the parameters have statistically

significant variation. In order to evaluate measurement errors and verify the accuracy of the data, uncertainty calculations were also performed. The best deposition conditions for optimising the superconducting performance were found using the combination of analytical modelling and statistical validation. In terms of developing excellent thin film for complex application involving superconductor, this is a good approach.

4. RESULTS AND DISCUSSION

4.1 Integrated Analysis of Deposition Parameters and Film Growth

The sputtering power, the sputtering chamber pressure, the sputtering substrate temperature, the gas composition of sputtering gases, the duration of sputtering and other parameters are the determining factors of the growth behavior of the superconducting thin film in the experimental results. The parameters have an effect on the nucleation, atomic mobility and phase formation processes that in turn control the superconducting properties of the films.

The deposition rate is represented by:

$$R = \frac{YIM}{\rho AN_A e}$$

where R is the deposition rate, Y is the sputtering yield, I is the discharge current, M is atomic mass, (ρ) is the density, A is the deposition area, N_A is Avogadro's number and (e) is the electronic charge. It's shown that the higher the sputtering power, the higher the deposition rate until a high power, above which defects and surface roughness are introduced.

The effect of chamber pressure is controlled by mean-free path:

$$\lambda = \frac{kT}{\sqrt{2}\pi d^2 P}$$

The mean free path, λ , is proportional to the molecular diameter (d), the Boltzmann constant (k) and the inverse of the pressure (P) and the inverse of the temperature (T). The energy particle deposition due to lower pressure increases (λ) leading to better crystallinity.

There are two factors that change the film thickness: deposition rate and deposition time.

$$t = R \times T$$

A graph of where (t) is thickness and (T) is deposition time. This relation line was applied to predict/control the growth of films. These results verify that the optimised values of these parameters produce uniform, dense and highly crystalline superconducting films.

4.2 Thickness and Uniformity Analysis

Thickness analysis revealed that the superconducting property was affected directly along with the electrical property. The experimentally determined thickness was checked with AFM and was found to be correlated with:

$$t = R \times T$$

In addition, the following methods for verifying and deposition rate was tested:

$$R = \frac{t}{T}$$

For the purpose of calculating uniformity of thickness across the substrate the following was used:

$$U = \left(1 - \frac{t_{max} - t_{min}}{2t_{avg}}\right) \times 100$$

The thickness (tmax), (tmin) and (tavg) is the maximum, minimum and average thickness respectively. The experimental results demonstrated uniformity greater than 95% which meant uniform deposition.

The dependence of superconductivity transition temperature with respect to film thickness is:

$$T_c(d) = T_{c0} \left(1 - \frac{d_c}{d}\right)$$

The transition temperature at thickness d is represented by a. Let (Tc(d)) denote the transition temperature at a certain thickness, (Tc0) represent the transition temperature in the bulk form and (dc) represent the critical thickness. The results revealed that ultrathin films had decreased (Tc) around it owing to the finite size effects.

The dependence of electrical resistance on thickness is given by the following:

$$R_s = \frac{\rho}{t}$$

Sheet resistance, [Rs] and resistivity, [ρ]. Thinner films showed higher resistance values, further indicating the requirement of the optimum thickness. In summary, the thickness optimization with 5-10nm value showed high superconducting performance and reliability of the device.

4.3 Structural and Morphological Characterization

Structural characterisation by HRTEM has confirmed the crystallinity and phase formation of the films. The wavelengths of the electrons are:

$$\lambda = \frac{h}{\sqrt{2m_e eV}}$$

This is due to the following equation: where (h) is Planck's constant, (m_e) is the mass of an electron, and (V) is the accelerating voltage.

Bragg's law analysis of the crystallographic structure was performed:

$$n\lambda = 2d\sin\theta$$

interplanar spacing, d, and diffraction angle, (θ) are as indicated. Earlier this was confirmed to form the desired superconducting phases, NbN and NbTiN.

Grain size estimate made by using the Scherrer equation:

$$D = \frac{K\lambda}{\beta\cos\theta}$$

where (D) is crystallite size, (K) is shape factor, (β) is the FWHM and (θ) is the diffraction angle.

The dislocation density was estimated from the following formula:

$$\rho = \frac{1}{D^2}$$

This indicated a lower defect density and higher superconductivity when the grains were bigger.

Using AFM, surface morphology analysis was performed and the RMS roughness was obtained as:

$$R_q = \sqrt{\frac{1}{N} \sum_{i=1}^N (z_i - \bar{z})^2}$$

and average roughness:

$$R_a = \frac{1}{N} \sum_{i=1}^N |z_i - \bar{z}|$$

Results demonstrated that optimized films resulted in a surface roughness of less than 1nm RMS, that is a smooth surface. Smooth morphology had the advantage of a lessening of electron scattering and an enhancement of current transport as compared with rough films, which exhibited poorer superconducting properties.

4.4 Optical and Superconducting Properties

Complex reflectance ratio was used to evaluate the optical characterisation by VASE for obtaining both refractive index and extinction coefficient:

$$\rho = \frac{r_p}{r_s} = \tan \Psi, e^{i\Delta}$$

The value of complex refractive index can be described as:

$$N = n - ik$$

and the dielectric function is:

$$\varepsilon = (n - ik)^2$$

The absorption coefficient of the mixture is given by:

$$\alpha = \frac{4\pi k}{\lambda}$$

The band gap value was calculated by applying the Tauc relation:

$$(\alpha h\nu)^m = A(h\nu - E_g)$$

Optimized films exhibited higher refractive index and lower extinction coefficient values which were positive sign of dense and uniform structure for the films. The superconducting properties were studied based on resistance temperature relations:

$$R = \rho \frac{L}{A}$$

and normalized resistance:

$$R_N = \frac{R(T)}{R_{300K}}$$

Transition temperature was calculated as:

$$T_c = \frac{T_{90} + T_{10}}{2}$$

and transition width:

$$\Delta T_c = T_{90} - T_{10}$$

The critical current density was measured by the following procedure:

$$J_c = \frac{I_c}{A}$$

The upper critical field was estimated that:

$$H_{c2}(0) = \frac{\Phi_0}{2\pi\xi^2}$$

Optimized films demonstrated the superior superconducting characteristics of higher (T_c), higher (J_c) and narrower transition width as obtained from the results.

4.5 Statistical Validation and Optimization for SNSPD Applications

The results were statistically analysed for reliability. The arithmetic average was used and calculated as follows:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

Variance and the standard deviation were provided by:

$$s^2 = \frac{\sum (x_i - \bar{x})^2}{n - 1}, s = \sqrt{s^2}$$

Coefficient of variation was:

$$CV = \frac{s}{\bar{x}} \times 100$$

The most commonly used expression of regression analysis was:

$$Y = a + bX$$

and multiple regression as:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_n X_n$$

The coefficient of determination was:

$$R^2 = 1 - \frac{\sum (y_i - \hat{y}_i)^2}{\sum (y_i - \bar{y})^2}$$

The F-statistic was used in analysis of variance (ANOVA):

$$F = \frac{MS_{\text{between}}}{MS_{\text{within}}}$$

The values of uncertainty analysis were obtained in the following manner:

$$u_c = \sqrt{u_1^2 + u_2^2 + \dots + u_n^2}, U = ku_c$$

The correlations of the deposition parameters with the superconducting properties was confirmed using statistical results. Optimized films demonstrated low variability, high repeatability and good statistical differences in performance. Optimized NbN and NbTiN films provided the ideal thickness of 5–10 nm, high T_c , high J_c values and smooth surface morphology for SNSPD applications. High detection efficiency, low noise and stable operation of the device are required for these qualities.

Table 4.1: Key Equations Used in Data Analysis

Category	Equation	Purpose
Deposition	$R = \frac{t}{T}$	Deposition rate calculation
	$t = R \times T$	Film thickness estimation

	$\lambda = \frac{kT}{\sqrt{2}\pi d^2 P}$	Mean free path (pressure effect)
Thickness & Electrical	$T_c(d) = T_{c0}(1 - \frac{d_c}{d})$	Thickness dependence of (T_c)
	$R_s = \frac{\rho}{t}$	Sheet resistance
Structural	$n\lambda = 2d\sin\theta$	Crystal structure (Bragg's law)
	$D = \frac{K\lambda}{\beta\cos\theta}$	Grain size (Scherrer equation)
Morphology	$R_q = \sqrt{\frac{1}{N}\sum(z_i - \bar{z})^2}$	RMS surface roughness
Optical	$N = n - ik$	Complex refractive index
	$\alpha = \frac{4\pi k}{\lambda}$	Absorption coefficient
Superconductivity	$T_c = \frac{T_{90} + T_{10}}{2}$	Transition temperature
	$J_c = \frac{I_c}{A}$	Critical current density
	$H_{c2}(0) = \frac{\Phi_0}{2\pi\xi^2}$	Upper critical field
Statistics	$\bar{x} = \frac{1}{n}\sum x_i$	Mean
	$R^2 = 1 - \frac{\sum(y_i - \hat{y}_i)^2}{\sum(y_i - \bar{y})^2}$	Regression fit quality
	$F = \frac{MS_{\text{between}}}{MS_{\text{within}}}$	ANOVA significance

In order to comprehend and correlate experimental results with deposition parameters and thin-film characteristics, basic analytical equations (Table 4.1) were used to summarise experimental data. The most significant physical processes related to film growth are captured by these equations together, including the deposition kinetics and control of film thickness, the evolution of the film's structure, surface morphology, optical response, and superconducting characteristics. This can be used in conjunction with experiments to investigate the effect of processing parameters on the significant properties such as transition temperature, critical current density and surface roughness of products quantitatively. Furthermore, the use of statistical formulae rendered the results reliable, repeatable and meaningful. The consequence is that this collection of equations can be used to optimize the conditions for deposition. And to fabricate a better superconducting thin film suitable for use in the latest devices.

4.6 Discussion

Experimental observations indicate that the deposition parameters play a crucial role in the structure, optical, and superconducting properties of thin film. From the foregoing observations on film quality, it is clear that application of ideal sputtering condition, control of sputtering power, setting an optimum chamber pressure and a proper flow ratio of Ar/N₂ gases are effective in improving the film quality, such as uniformity of film thickness, crystallinity of films, and reduction of defect density. The films produced under these ideal conditions had a better surface morphology with reduced rms roughness, which decreased electron scattering and improved the film's superconducting performance. The need of maintaining the films in the ideal thickness range to guarantee reduced potential finite-size effects and the maintenance of high T_c values was further supported by the association between thickness and superconducting transition temperature. The critical current densities were found to rise with increasing grain size, owing to the fact that carrier mobility increases with decreasing dislocation density, as indicated by the structural analysis. These findings were further supported by optical characterisation, which showed that denser and more homogenous films had a lower extinction coefficient and a higher refractive index. Still, the changes found were sufficiently large and reproducible to be confirmed statistically. Overall, the discussion emphasizes the need for accurate control and optimization of deposition parameters, as well as the potential for using these 2D materials in emerging applications like superconducting nanowire single photon detectors and other quantum devices.

5. CONCLUSION

The authors of the current work succeeded in the preparation of high-quality superconducting thin films such as NbN, NbTiN, TiN and even amorphous films of MoSi by optimizing the methods and process parameters of their preparation. Relationships between the structural, optical, and superconducting properties were studied by a systematic experiment method with special emphasis on some of the most important deposition parameters, such as the sputter power, pressure in the chamber, deposition temperature, flow ratio of the gases present during the experiment and deposition time. The results conclusively proved that if good surface morphology, crystallinity and defect density and uniform thickness are to be obtained, these parameters must be well controlled. Under optimal conditions, the films exhibited the greatest superconducting properties including a high transition temperature (T_c), a high critical current density (J_c) and a narrow transition width. These characteristics are crucial for dependable device functioning. Advanced characterisation techniques, such as AFM, HRTEM, VASE, or cryogen-free electrical measurements, have enabled to build a complete picture of the well-documented relationship of the microstructure with the superconducting properties. In addition, the observed patterns were replicated and found to be statistically significant, thus making the credibility of the optimisation strategy more reliable. Other applications such as single photon detection with a superconducting nanowire single photon detector (SNSPD) which is very sensitive to the homogeneity and quality of the surface required thickness control in the nanometre range. Finally, the results have shown a full understanding of the control of the deposition process to achieve superior performance for a thin-film device, and helped to improve understanding and control of the deposition process for next generation of superconducting and quantum electronic devices.

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