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High-Temperature Epitaxial Growth of Tantalum Nitride Thin Films on MgO Substrates: Structural Evolution and Potential for SQUID Applications.

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Abstract

The growth of superconducting tantalum nitride (TaN) thin films on magnesium oxide (MgO) substrates has been studied using pulsed laser deposition (PLD). This research examines how varying deposition parameters, substrate temperature, and ambient gas composition affect the structural, morphological, and superconducting properties of the films. X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), Atomic Force Microscope (AFM) and Transmission electron microscopy (TEM) results show that the TaN films possess excellent crystallinity and smooth surface morphology, with an optimal deposition temperature of 850 °C. The films exhibit superconducting transition temperatures (T_C) ranging from 5.0 K to 9.3 K, depending on the stoichiometry and deposition conditions. Electrical resistivity measurements further confirm the high quality of the films, reflected by their low residual resistivity ratios. These results indicate that PLD is a suitable method for producing high-quality TaN superconducting films.

This study emphasizes the role of deposition parameters in tuning the superconducting properties, particularly for applications in superconducting electronics and quantum computing. Additionally, these films are potentially used in susceptible devices like Superconducting Quantum Interference Devices (SQUIDs), with further improvement possible through optimizing stoichiometry and reducing oxygen impurities.

Keywords: TaN Thin Films, Epitaxial Growth, Structural evolution, Superconductivity, SQUID.

1. Introduction.

Superconductivity is a quantum mechanical phenomenon characterized by the complete absence of electrical resistance in certain materials when cooled below a critical temperature. Discovered in 1911 by Heike Kamerlingh Onnes, superconductors offer unique properties, including perfect conductivity and the expulsion of magnetic fields (the Meissner effect). These attributes make them highly valuable for various applications, such as lossless power transmission, magnetic levitation in transportation systems, and the creation of powerful electromagnets. Furthermore, superconductivity plays a vital role in the development of advanced technologies, including quantum computing and nanometric electronic devices. Despite significant progress, challenges remain in discovering materials that exhibit superconductivity at higher, more practical temperatures, which continues to be a major focus of research in the field.

In this last, efficient fabrication of materials with a low superconductive energy gap and an intermediate transition temperature (T_C), results essential for the development and enhancement of superconducting electronic devices in the GHz range. The transition-metal compounds, for example, the nitrides and carbides such as NbN, TiN, or TiC, TaN have previously shown superconductive transition temperatures of 2 to 10.4 K [1][2][3][4]. Also, these compounds are a significant class of materials, due to physical and chemical properties such as ultra-hardness (comparable to diamond) and high melting points around 3000 °C, those properties can be qualitatively understood by observing that the Fermi energy falls in a pronounced minimum of the density of states (DOS) [5].

Some reports have shown that tantalum nitride has a superconductive energy gap lower than NbN [6], the most commonly used material for single-photon detectors in the GHz range, so its material can be a better candidate for superconductive electronic devices. Depending on the amount of incorporated nitrogen, x, the tantalum nitride system TaN_x can be an insulator, semiconductor, or superconductor and also can exhibit a variety of crystallographic phases [7][8]. For example, Lee, H., and collaborators mentioned that Ta_2N thin films presented a high-temperature coefficient of resistance, so his group fabricated a resistor using this material as a diffusion barrier, with potential application for microelectronics [9]. On the other hand, the stoichiometric mononitride (TaN) phase with FCC structure exhibits superconductivity with a T_C of 8.15 K [10], if the thin films were grown epitaxially in an FCC substrate, the T_C can be pushed up to 10.8 K [11], so the Tc of TaN depends strongly on the crystallinity and stoichiometry of the thin films. Reports mentioned that Pulsed Laser

Deposition (PLD) in the reactive mode (RPLD) is an efficient method for the growth of high-quality thin films [12].

In the present work, superconducting tantalum nitride (TaN) thin films were synthesized using the pulsed laser deposition (PLD) technique. A high-purity tantalum (Ta) target (99.999%) was ablated in a nitrogen (N_2) atmosphere, with the pressure of N_2 being varied to investigate its effect on film properties. The substrate temperature was systematically altered to explore its impact on the growth dynamics and superconducting characteristics of the films. X-ray diffraction (XRD) analysis revealed that the TaN thin films exhibited excellent crystallinity, with sharp diffraction peaks indicating well-defined structural phases.

The deposition process was optimized by adjusting the substrate temperature, N_2 pressure, and laser energy, aiming to improve the superconducting transition temperature (T_C) of the films. Among the samples prepared, the film deposited at an optimal temperature demonstrated the highest T_C of 7.32 K, indicating superior superconducting behavior.

Analysis by XPS manifests the presence of stable oxygen impurities. The oxygen source is the residual gas inside the growth chamber, Victor Quintanar-Zamora et al obtained evidence that oxygen can occupy the N sites of the crystal without structural modifications [13].

These findings reinforce the potential of PLD as a viable method for fabricating high-quality TaN superconducting films, with controlled stoichiometry and phase purity. The optimal combination of N_2 pressure and substrate temperature appears crucial for achieving desirable superconducting performance, making these films promising candidates for applications in superconducting electronics.

2. Experimental Procedure.

The experiment is performed in a laser ablation system *RIBER LDM 32*. Consists of three stainless steel UHV vacuum chambers: introduction sample, PLD deposition, and analysis (X-Ray Photoelectron Spectroscopy, XPS), isolated by UHV gate valves and independently pumped by ion and Ti sublimation pumps. Every chamber has a base pressure of about 10^{-9} Torr. An Nd: YAG laser with a second and fourth harmonic generator (533 and 266 nm, respectively), is focused onto a 99.999% Ta target with 4 ns and *hv=1.5 eV*. The laser was operated at a frequency of 7.5 Hz; the substrate-to-target distance was 5 cm, while the incident energy density was *4.88 J/cm*^{2}. We studied the stoichiometry and properties of thin films as a function of gas pressure by introducing N_2 to realize RPLD. At this time, the ion pumps were closed and isolated to the PLD deposition

chamber, and a turbo pump was used to pump the gas down during film deposition. The films are transferred to the analysis chamber to characterize them in situ by XPS. Varying the N_2 pressure, p_{N2}, on the chamber (10^{-9} mTorr) and analyzing the films, we obtained TaN thin films at 90 mTorr of N_2. XPS data were collected using an *Al K α* monochromatic source and hemispherical analyzer from SPECS. The films were grown on (100) oriented single crystals substrates of MgO (purchased from Sigma Aldrich). To obtain an FCC structure, we were varying the deposition temperature (600-850 °C) using 90 mTorr of N_2. The XRD spectra were obtained with a Panalitycal X'Pert Pro MRD, and the ICSD powder diffraction database was used for the qualitative search-match phase identification. To identify the epitaxy of the films, TEM was obtained with a *Jeol JEM 2100F*. Finally, the curves R vs. T were analyzed using the Van der Pauw method in a *DynaCool Quantum Design* PPMS. AFM XE-70 Park Systems in contact mode was used to study the surface morphology of the films.

The synthesis protocol used in this study was modified from the work reported by Victor Quintanar-Zamora et al [13].

3. Results and discussions.

The atomic concentration of each component in the TaN thin films as a function of nitrogen pressure (pN_2) was analyzed using X-ray photoelectron spectroscopy (XPS). XPS measurements focused on the core levels of Ta_{4f}, N_{1s}, C_{1s}, and O_{1s}, with the peak areas calculated after linear background subtraction. This analysis provided insights into the stoichiometry and impurity levels in the films, which are crucial for optimizing their superconducting and structural properties.

The results indicate that nitrogen concentration increases significantly with rising nitrogen pressure. At 70 mTorr of p_{N_2}, the nitrogen concentration was measured at 32.5%, but this value rose sharply to 37.5% at 90 mTorr. Simultaneously, at 90 mTorr, the oxygen concentration dropped by 10.2%, and the carbon concentration fell to 0%. These reductions in oxygen and carbon impurities are essential for improving the film's quality, as both elements can introduce defects that degrade superconducting performance.

At a lower nitrogen pressure of 60 mTorr, the atomic concentrations of tantalum (Ta) and nitrogen (N) were found to be nearly equal. However, the concentrations of oxygen and carbon were relatively high at this pressure, which compromises the quality of the TaN films. Consequently, 60 mTorr was discarded as a viable pressure for synthesizing high-quality films due to the impurity levels.

The optimal stoichiometry for TaN thin films was achieved at 90 mTorr of p_{N_2}, where the nitrogen concentration reached its highest value, and oxygen and carbon impurities were minimized. This nitrogen pressure also aligns with the results from XRD and TEM, as the film deposited under these conditions exhibited the best crystallinity and epitaxial growth. The combination of optimal stoichiometry and high crystallinity, achieved by controlling both the deposition temperature and nitrogen pressure, plays a pivotal role in enhancing the superconducting properties of the TaN films.

Atomic concentrations for every p_{N2} are shown in Table 1. The low percentage of oxygen and carbon in the sample improves the epitaxial growth of the thin film; this is confirmed by the TEM and XRD results presented below.

Table 1. Quantification of TaN thin films for every P_{N_2}.

	10 mTorr	**30 mTorr**	**50 mTorr**	**60 mTorr**	**70 mtorr**	**90 mtorr**
% Ta	31.4	24.7	28.1	34.4	30.0	51.1
% N	2.4	1.8	5.6	26.6	32.2	36.5
% O	62.4	66.3	62.8	32.0	33.8	12.4
% C	3.8	6.8	3.5	7.0	4.0	0

Monitoring the positions of the $Ta_{4f\ 7/2}$ and $Ta_{4f\ 5/2}$ peaks can reveal any chemical state changes that tantalum (Ta) and nitrogen (N) may undergo during the ablation process. Figure 3 presents the deconvolution of high-resolution XPS peaks for Ta_{4f}, shown both before (Fig. 3a, 3b) and after (Fig. 3c, 3d) heating the substrate. The peaks were fitted using Gaussian functions. The relative atomic concentrations of Ta and N were calculated from the areas under the Ta_{4f} (Fig. 3 and Table 2) and N1s peaks after Touggard background subtraction, using CasaXPS software.

As shown in Figure 1, at 60 mTorr of p_{N_2}, we identify contributions from tantalum oxide, nitride, carbide, and metallic tantalum. At 70 mTorr, contributions from these compounds are still present, along with tantalum oxynitride. *Liang Shi et al.* [14] synthesized TaN nanocrystals and reported that the $TaN_{4f\ 7/2}$ XPS peak appears at 23.5 eV. Similarly, Bernhard C. Bayer and colleagues [15] identified the $TaC_{4f7/2}$ XPS peak at 23.3 eV, and *Xiaoyun Yang* [16] synthesized TaON nanoparticles, reporting a photoemission peak at 25 eV.

In the deposition conditions of the thin layers of TaN, we found that increasing the nitrogen flow

substantially reduces the amount of oxygen in the films (see Table 1) compared to that reported by Victor Quintanar-Zamora et al [13], and it remains almost invariable when increasing the temperature to 850 °C (see table 2). Although higher temperatures can promote the desorption of impurities, including oxygen, in this case, the temperature range between 700°C and 850°C is not sufficient to drastically change the rate of oxygen incorporation or removal. Thus, any further increase in temperature beyond 700°C does not affect oxygen content due to limited oxygen availability and the dominant role of nitrogen flow.

These references are instrumental in the deconvolution of Figures 1 and 2. The separation between the peaks and the relationships between the areas were used to determine

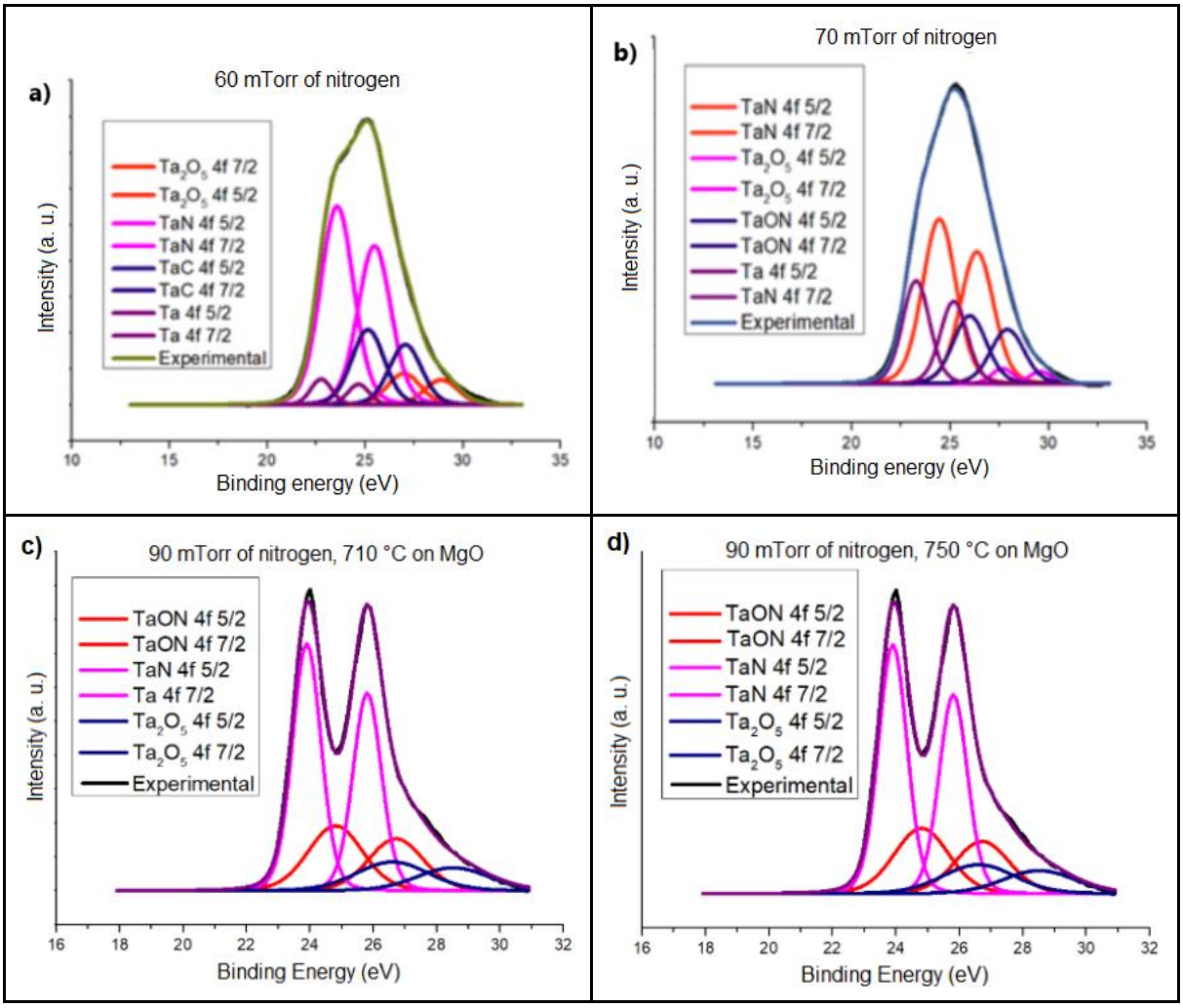

Figure 1. Comparison of Ta $4f_f$ XPS peaks determined for different samples.

Samples heated at 700 °C and 750 °C (Figures 1c and 1d) present better stoichiometry of TaN than Figures 1a and 1b; had a lower atomic concentration of Ta_2O_5 and the doublet shape is more defined. Table II shows the atomic concentration of heated samples of 700 °C to 850°C using 90

mTorr of p_{N2}. The tantalum concentration does not vary much as the temperature changes, but nitrogen and oxygen do vary. Incorporation of oxygen into TaN was studied in detail in the mentioned previous work [13].

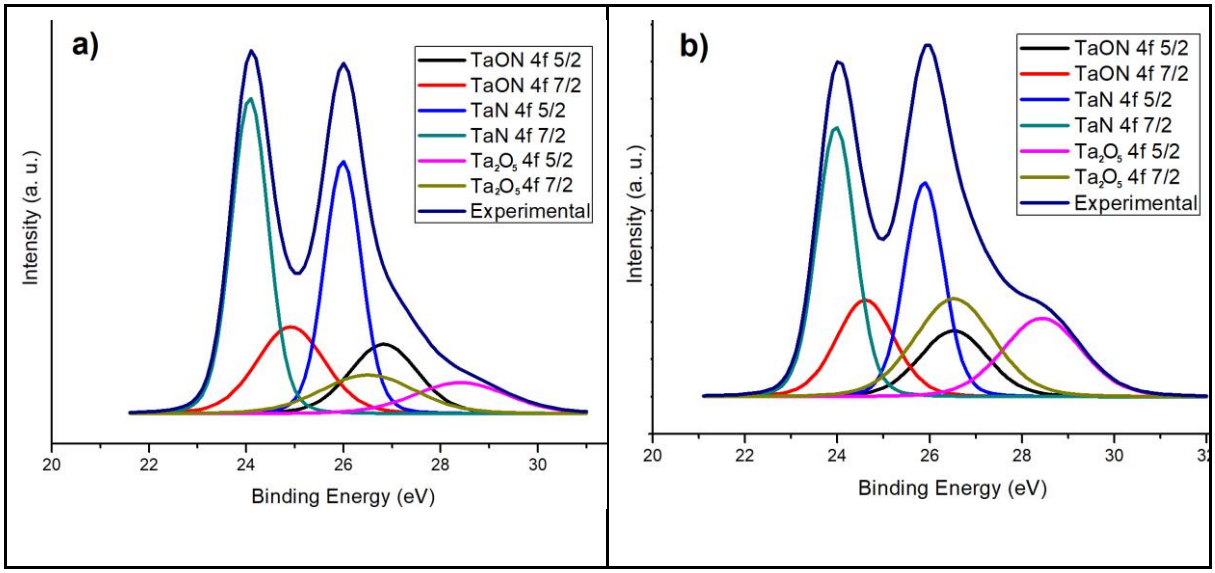

Figure 2. Ta 4f XPS doublets of samples heated at a) 800 °C and b) 850 °C

When the temperature is increased, the tantalum allows greater incorporation of oxygen, forming a lattice interstitial. This is confirmed by the concentration of the sample heated to 850 °C (Table II) and with the Ta_2O_5 doublet's intensity in Figure 2b; the intensity of the Ta_2O_5 doublet of Figure 4a is less than Figure 2b.

Table II. Quantification of heated samples.

	% Ta	% N	%O
700 °C	50.96	36.58	12.45
750 °C	51.86	34.81	13.31
800 °C	48.15	38.84	12.99
850 °C	47.62	37.12	15.25

The effect of deposition temperature on the structural evolution of tantalum nitride (TaN) thin films deposited on magnesium oxide (MgO) (100) substrates was investigated using X-ray diffraction (XRD) and transmission electron microscopy (TEM). Figure 3 illustrates the XRD patterns of TaN films deposited at various temperatures, revealing critical insights into phase development, crystallinity, and epitaxial growth as the temperature increased.

At 600 °C, the XRD results show a low crystallinity film, as evidenced by the weak intensity of the (111) δ-TaN reflection. The δ-TaN phase is identified by its characteristic reflections at (111), (200), (220), and (311) positions, based on JCPDS Card No. 00-0040-0829. Additionally, a weak β-TaN phase, identified from the (110) reflection (JCPDS Card No. 00-039-1485), is present, though the overall crystallinity is poor at this temperature. As the deposition temperature is increased to 700 °C, a significant change in the film's phase composition is observed. A mixed phase of δ-TaN (JCPDS File No. 49-1283) and β-TaN (JCPDS Card No. 39-1485) appears, with the cubic δ-TaN phase becoming more prominent compared to the hexagonal β-TaN phase. The increased crystallinity is evident from the sharper and more intense diffraction peaks. The coexistence of both cubic and hexagonal phases at 700 °C suggests an intermediate stage of structural evolution, where the cubic δ-TaN phase begins to dominate the overall microstructure.

At 750 °C, the XRD patterns show a further increase in the dominance of the cubic δ-TaN phase, indicating a structural transition towards a more stable phase at higher temperatures. The β-TaN phase is significantly reduced, and the δ-TaN phase becomes the major phase present in the film. This increased phase purity and the sharper diffraction peaks indicate enhanced crystallinity, aligning with the structural requirements for high-performance superconducting films.

The most significant structural improvement is observed at a deposition temperature of 850 °C. At this temperature, the XRD patterns demonstrate epitaxial growth of the TaN thin film on the MgO substrate. The (200) and (220) reflections of the δ-TaN phase show high intensity, while the (111) and (311) peaks exhibit lower intensity. The sharpness and intensity of these peaks indicate a highly crystalline film with excellent alignment to the MgO (100) substrate. The epitaxial relationship between the film and substrate is confirmed by the matching orientations of the lattice planes, suggesting a coherent interface between the two materials. Similar results were reported by *Chaudhuri* and collaborators [17] and *T. Elangoval* [18].

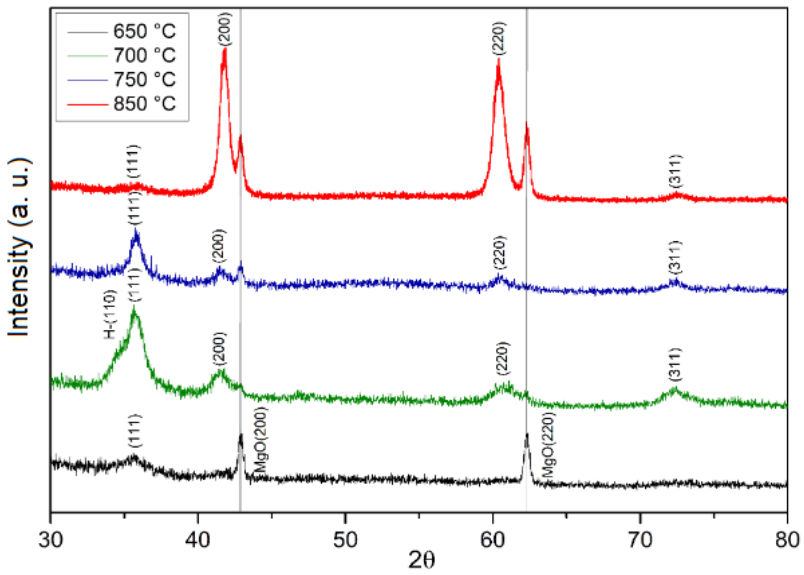

Figure 3. XRD spectra of TaN thin films on MgO) substrates at different temperatures.

Figure 4 shows how the lattice parameter and interplanar spacing decreased as temperature deposition increased. The lattice parameter decreased from 4.36 to 4.32 Å, as the temperature deposition increased. In fact, at 850 °C, the adatom surface mobility and surface diffusion of the deposited atoms are further increased, which is also proved by *Elangoval et al* [19], *Adamik et al* [20], and *Cheng et al* [21] research.

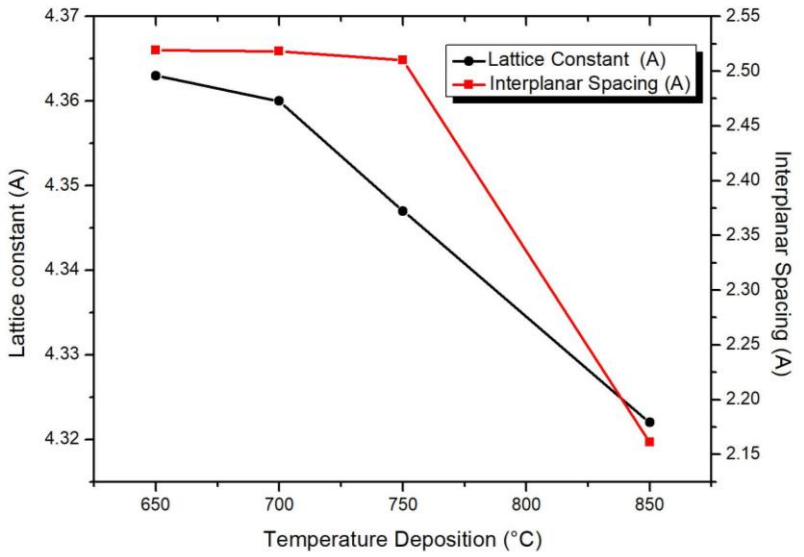

Figure 4. Lattice constant and Interplanar Spacing of TaN thin films deposited at different temperatures.

The variation of resistance of TaN films with T_C values is shown in Figure 5, measured from room temperature down to liquid helium temperature. The measurement of the film deposited at 650 °C was not performed due to its low crystallinity. As mentioned previously, a mixed δ-TaN and β-TaN

phases are identified in the film deposited at 700 °C. The mixture of these phases results in the absence of superconductivity [21]. Upon increasing to 750 °C, a superconducting transition occurs at T_C=5.66 K, which has sample δ-TaN as the significant phase. T_C's highest value was obtained on the film deposited at 850 °C (T_C=7.32 K). As shown in Figure 3, that sample shows an epitaxial growth, crystallinity, and T_C higher than the others.

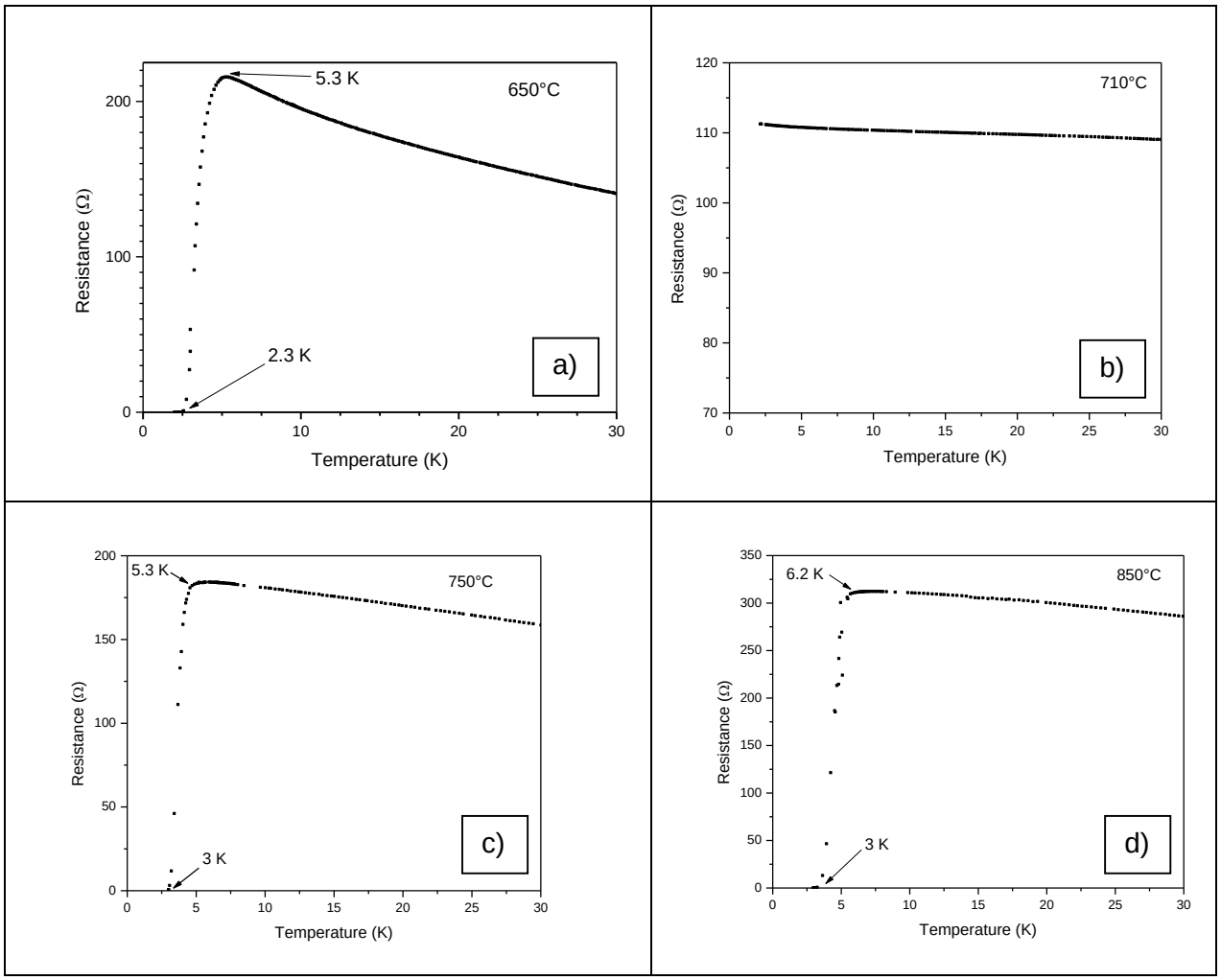

Figure 5. variation of resistance of TaN films grown to a) 650 ºC, b) 710 ºC, c) 750 ºC, and d) 850 ºC,

Cross-sectional transmission electron microscopy samples were prepared by a 4 kV Ar-focused ion beam (FIB) for transmission electron microscopy observation; Au film was evaporated above TaN before the FIB process to protect the sample (Figure 6a). The sample shows 70 nm of thickness (Figure 6b) and can distinguish the Au protective layer used for the FIB process. Figure 6c shows the epitaxial growth in the (100) MgO direction, and Figure 6d shows a region away from the substrate; the interplanar spacing of the (200) plane was calculated (d=2.176 Å) with Digital Micrograph software. It is observed that there are low crystallinity regions and others that have

planes with a growth in the direction of the substrate. Figure 6e shows the electron diffraction pattern of TaN; it presents the (200) and (220) planes because the sample contains TaN as a major phase.

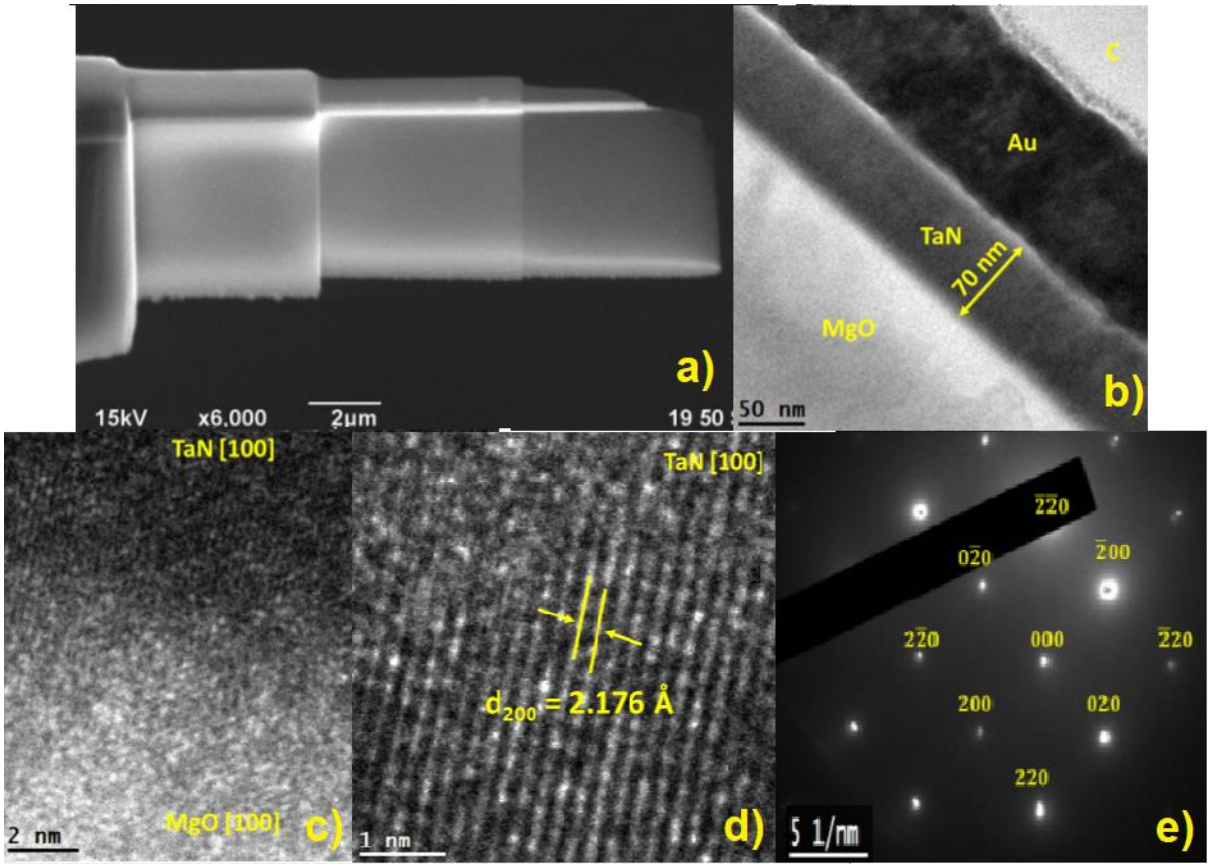

Figure 6. a) SEM image of the cross-section of TaN thin film prepared with FIB. b) TEM analysis of the lateral region of TaN film deposited at 750 °C. c) TEM micrography at the interface between MgO substrate and TaN thin film. d) Transversal section showing the interplanar spacing of TaN. e) Indexed electron diffraction pattern of TaN thin film.

TEM analysis provides further confirmation of the epitaxial growth and crystallinity of the TaN thin films. The interplanar spacing was calculated using Digital Micrograph software for both the MgO substrate and the TaN film. For the substrate, the interplanar spacing was found to be d = 2.115 Å, while the film's interplanar spacing was slightly larger, d = 2.166 Å. This slight mismatch is typical for epitaxial growth, where the film adapts to the lattice parameters of the substrate (Figure 6a.).

In regions farther from the substrate, the interplanar spacing was measured as d = 2.169 Å, indicating that the cubic face-centered cubic (FCC) structure persists throughout the film, maintaining a high degree of crystallinity. Figure 6d and Figure 6e confirm the presence of the FCC phase, with clear identification of the (200) and (220) planes, consistent with the XRD results and

ICDD Card No. 00-049-1283. The film's lattice parameter, calculated from the interplanar spacings, closely matches the values obtained from the XRD analysis, further validating the structural data. Figure 4 summarizes the lattice parameters of the TaN films at various deposition temperatures. The results show a clear trend: as the deposition temperature increases, the lattice parameter decreases. This is attributed to the reduced interplanar spacing at higher temperatures, likely caused by increased atomic mobility and improved crystallinity during growth. The trend aligns with previous studies on high-temperature epitaxial growth, where elevated temperatures promote denser packing and tighter atomic arrangements within the crystal lattice.

XPS results obtained in this work show a significant decrease in oxygen concentration in the TaN films when increasing the nitrogen flow, and it remains almost invariant when increasing the substrate temperature up to 850 °C.

Overall, the film deposited at 850 °C exhibits the highest degree of crystallinity, as demonstrated by both XRD and TEM analysis. The epitaxial alignment of the TaN film with the MgO substrate is critical for achieving superior superconducting properties, as high crystallinity and minimal defects reduce electron scattering, improving the film's superconducting transition temperature (T_C). The combination of cubic phase dominance, epitaxial growth, and precise lattice matching with the MgO substrate makes this film an excellent candidate for superconducting applications, particularly in devices such as Superconducting Quantum Interference Devices (SQUIDs).

The TEM and XRD analyses suggest that further refinement of the deposition process, such as eliminating oxygen impurities and fine-tuning the nitrogen stoichiometry, could result in even higher critical temperatures. The high-quality epitaxial TaN films grown at 850 °C show great potential for integration into advanced superconducting technologies, where low resistivity and high T_C are essential for device performance.

Figure 7 shows micrographs obtained on the AFM XE-70 Park Systems, which indicates low roughness of the films even at high growth temperatures (850 °C). suggesting that the deposition parameters significantly influence both the structural and superconducting properties of the TaN thin films but not in the roughness.

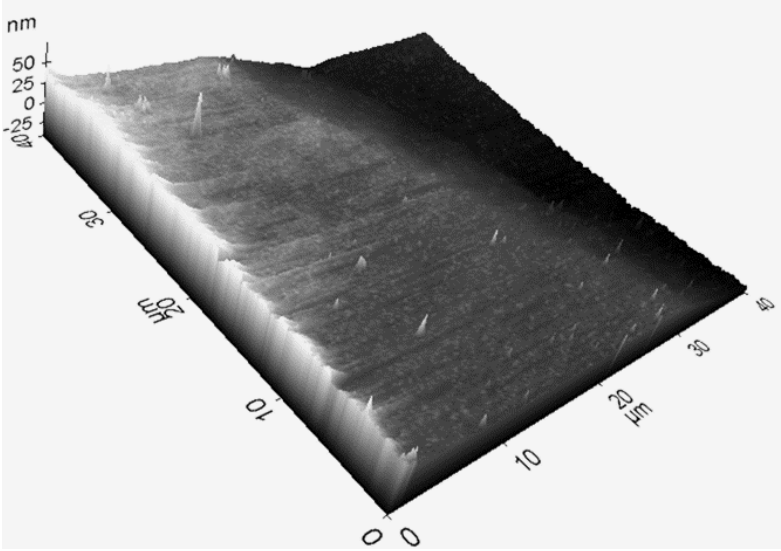

Figure 7. Micrograph of TaN film deposited at 750 °C obtained with AFM XE-70 Park Systems

Conclusions.

Tantalum nitride thin films were synthesized by the reactive pulsed laser deposition technique. It has been found the films can be presented with different critical temperatures depending on the synthesis conditions: deposition temperature and stoichiometry. Films growing at 650 and 700 °C do not show superconductivity; otherwise, films at 750 and 850 °C have a critical temperature of 5.66 and 7.32 K, respectively. XPS results show the presence of oxygen that was difficult to eliminate; even so, they were superconducting. The presence of oxygen does not cause the sample to lose superconductivity, as long as its structure remains cubic. Quantification shows that tantalum allows greater incorporation of oxygen at high temperatures.

XPS analysis demonstrates that nitrogen pressure critically influences both the stoichiometry and impurity content of TaN thin films. The nitrogen concentration at 90 mTorr provides the best stoichiometric balance while minimizing the concentrations of oxygen and carbon impurities. This pressure, in combination with an optimal deposition temperature of 850 °C, results in the growth of high-quality, epitaxial TaN films with excellent structural and superconducting characteristics.

XRD and TEM analysis shows a high crystalline deposition at high temperatures, sample deposited at 700 °C presents hexagonal and cubic mixed phases, and at 850 °C the sample shows an epitaxial growth with MgO substrate. Also, the lattice parameter and interplanar spacing were decreased when the temperature was increased. It is estimated that eliminating oxygen impurities will achieve a higher critical temperature; also, the sample growing at 850 °C can be useful for the fabrication of a Superconductive Quantum Interference Device (SQUID).

Atomic force microscopy (AFM) analysis further confirmed the smooth surface morphology of the films, suggesting that the deposition parameters significantly influence both the structural and superconducting properties of the TaN thin films but not in the roughness.

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