# Preparation and Characterization of Pyroelectric Lithium Tantalate Coatings

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## Abstract

Marine reactors and floating nuclear power plants face challenges in boiling heat transfer within two-phase heat transfer systems due to wall temperature fluctuations induced by oceanic motions. The pyroelectric effect generated by lithium tantalate (LT), a pyroelectric material, in response to temperature variations can influence the wettability of heat transfer surfaces, and its application to such surfaces holds promise for improving heat transfer performance. This study achieves controlled preparation of LT via the sol-gel method and investigates the mechanism underlying the regulation of pyroelectric performance and wettability through changes in hydroxyl radical content. The results demonstrate that LT particle size increases with annealing temperature, while both increased coating thickness and decreased particle size contribute to enhanced pyroelectric performance. Fluorescence spectroscopy analysis reveals that the hydroxyl radical content in LT increases during heating, concurrently confirming LT's capability to regulate hydroxyl radical content throughout heating and cooling cycles. These findings indicate that LT coatings possess the ability to regulate the wettability of heat transfer surfaces in two-phase variable-temperature heat transfer systems.

## Full Text

## Preamble

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**Synthesis and Performance Characterization of Pyroelectric Lithium Tantalate Coatings**

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## Abstract

[Background]: The operation of marine reactors and floating nuclear power plants faces challenges from wall temperature fluctuations caused by oceanic motion, which affect boiling heat transfer in two-phase systems. The pyroelectric effect of lithium tantalate (LT), a material whose spontaneous polarization changes with temperature variations, can influence the wettability of heat transfer surfaces, offering potential for performance improvement. [Purpose]: This study aims to achieve controlled preparation of LT coatings via the sol-gel method and investigate the mechanism of pyroelectric performance and wettability regulation through hydroxyl radical content variation. [Methods]: The sol-gel method was employed for controlled synthesis of LT coatings, and the pyroelectric effect and wettability modulation mechanism were studied by monitoring hydroxyl radical content changes. [Results]: Results demonstrate that LT particle size increases with annealing temperature, while increased coating thickness and decreased particle size both enhance pyroelectric performance. Fluorescence spectroscopy analysis reveals elevated hydroxyl radical content during heating, confirming LT's ability to regulate hydroxyl radical concentration through heating-cooling cycles. Conclusions: These findings indicate that LT coatings possess the capability to regulate heat transfer surface wettability in two-phase variable-temperature systems.

**Keywords**: Pyroelectric effect; Lithium tantalate; Sol-gel method; Temperature-dependent wettability self-regulation

## Introduction

The development and utilization of marine energy constitute a vital component of global energy strategy. Marine reactors and floating nuclear power plants, as key technologies for marine energy exploitation, face heightened safety requirements and complex vapor-liquid phase behaviors during design and operation. In marine environments, two-phase heat transfer equipment must not only withstand natural factors such as wind and waves but also cope with resulting inclination and oscillation motions. These movements significantly affect bubble aggregation, diffusion behavior, and temperature distribution near heating walls, consequently influencing system heat transfer efficiency and safety. In bubble aggregation regions, boiling crises can occur due to dry patch formation or liquid film rupture at the bottom. In bubble diffusion regions, where coolant remains in single-phase or subcooled boiling states, the heat transfer coefficient (HTC) is lower than in saturated boiling. Therefore, enhancing HTC and safety margins in two-phase heat transfer systems represents a current research priority.

Surface wettability is a critical factor affecting near-wall boiling heat transfer. Hydrophobic surfaces can nucleate at lower wall superheat, achieving higher HTC at low heat flux through multiple nucleation sites. Hydrophilic surfaces enhance liquid replenishment in dry regions, suppress wall nucleation, and limit bubble accumulation, thereby increasing critical heat flux. In two-phase heat transfer systems, boiling performance is largely governed by bubble behavior, and the dynamic nature of boiling processes under oceanic motion creates varying demands for surface wettability, posing new requirements for surface modification technologies. Current surface modification methods only achieve static or irreversible wettability transitions, making them ill-suited for such dynamic scenarios.

Pyroelectric materials, which exhibit temperature-dependent spontaneous polarization changes, offer a novel approach for dynamic wettability regulation. These materials can induce surface charge generation under temperature variations, subsequently producing hydrophilic hydroxyl radicals and hydrophobic superoxide radicals in coolant that influence wettability. Leveraging this characteristic, temperature-sensitive self-regulating wettability surfaces can be developed to achieve spontaneous reversible wettability regulation under temperature fluctuations in two-phase heat transfer systems, thereby enhancing HTC and safety margins. Lithium tantalate ($LiTaO_3$, LT), with its high pyroelectric coefficient, high Curie temperature (650°C), and low relative permittivity, is considered an ideal candidate for high-temperature heat transfer systems. Various techniques exist for LT coating preparation, including radio frequency magnetron sputtering, pulsed laser deposition, molecular beam epitaxy, metal-organic chemical vapor deposition, and sol-gel methods. Among these, the sol-gel method offers advantages of simple preparation, low cost, rapid film formation, and adjustable elemental ratios, making it a promising approach for LT coating synthesis.

Despite these advantages, in-depth research on controlled preparation of LT coatings via sol-gel methods and verification of pyroelectric materials in reversible wettability transitions remains lacking. This study addresses this gap by successfully preparing LT coatings using the sol-gel method and conducting detailed characterization of their structure and properties to confirm excellent crystallinity and pyroelectric performance. Through precise control of sol-gel parameters, controlled preparation of LT coatings was achieved, and their potential for temperature-sensitive wettability self-regulation was verified, providing a new solution for enhancing heat transfer performance in two-phase systems.

## 1.1 Preparation Method of Lithium Tantalate Coatings

This study employed the sol-gel method to prepare LT coatings, with the entire process comprising three main steps: LT sol preparation, spin-coating and curing, and annealing, as illustrated in [Figure 1: see original paper].

### 1.1.1 LT Sol Preparation

Anhydrous lithium acetate ($CH_3COOLi$, 98%, Grat) and 1,2-propanediol ($C_3H_8O_2$, 98%, Grat) were first mixed at a molar ratio of 1:10 and heated in an oil bath to 110°C with reflux heating for 3 hours at 10 rad/s magnetic stirring until complete dissolution. The mixture was then cooled to 40°C, and butyric acid ($C_4H_8O_2$, $ 99.8*{10}H{25}O_{5}Ta, 98^{+}$ to $Ta^{5+}$ in the precursor solution. The mixture was reheated in an oil bath to 130°C for 72 hours of reflux heating. After reaction completion, the obtained colloidal solution was left standing to promote full polycondensation between $Ta^{5+}$ and $Li^{+}$, forming stable Li-O-Ta bonds, ultimately yielding a 0.5 mol/L brownish-yellow LT sol.

### 1.1.2 Spin-Coating and Curing of LT Coatings

Titanium substrates (20 mm × 20 mm × 2 mm, Ti, $ 99.5*{3}H{6}$O, $ 99.5*{3}CH{2}$OH, $ 99.8_{2}$O, Inoke) for 25 minutes, followed by drying to ensure contaminant-free surfaces. Before spin-coating, substrates were fixed on a spin coater using a vacuum pump. The LT sol was uniformly applied, with spin-coating conditions set at 500 rpm for 15 seconds (dispersion) and 3000 rpm for 60 seconds (spinning). Immediately after spin-coating, samples were cured in a box furnace at 120°C. These spin-coating and curing steps could be repeated to control coating thickness.

### 1.1.3 Annealing of LT Coatings

Cured LT coatings were annealed in a tube furnace under atmospheric air pressure. The annealing process comprised three stages: first, heating from room temperature to 250°C and holding for 10 minutes to remove unreacted organics and provide a clean surface for subsequent high-temperature reactions; second, heating from 250°C to 450°C and holding for 10 minutes to decompose most byproducts and ensure impurity-free coatings; finally, heating from 450°C to different annealing temperatures (650°C or 700°C) and holding for 1 hour to achieve complete crystallization. After annealing, samples were removed when furnace temperature dropped below 30°C and stored under vacuum.

## 1.2 Structural Characterization Methods for Lithium Tantalate

To comprehensively understand the microstructural features of prepared LT coatings, this study employed a JSM-7800F ultra-high resolution thermal field emission scanning electron microscope (SEM) and a Navigator-100 high-throughput SEM at Shanghai Jiao Tong University Analysis Center for surface morphology characterization. Phase composition, a key determinant of physical properties, was analyzed using X-ray diffraction (XRD) with a D8 ADVANCE Da Vinci multifunctional X-ray diffractometer. This mature phase analysis technique provides detailed information on crystal structure, interplanar spacing, and crystal orientation. Analysis of obtained diffraction

patterns enables identification of dominant crystal phases and assessment of crystallinity. These characterization steps provide both surface morphology information and in-depth understanding of phase composition and crystal structure, forming the basis for further pyroelectric performance analysis.

## 1.3 Pyroelectric Performance Characterization Methods

This study aimed to verify LT's temperature-sensitive wettability self-regulation capability, focusing on evaluating hydroxyl radicals generated during temperature changes. Pyroelectric material spontaneous polarization varies with temperature, causing surface charge changes that react with hydroxide ions in water to generate hydroxyl radicals, as shown in Equation (1). To detect hydroxyl radical generation, terephthalic acid (TA) was selected as a probe molecule. TA rapidly reacts with hydroxyl radicals through aromatic ring hydroxylation to form highly fluorescent 2-hydroxyterephthalate ions (2-HTA), as shown in Equation (2). The fluorescence intensity of this reaction product, measurable via fluorescence spectroscopy, correlates positively with hydroxyl radical concentration.

Experiments utilized a temperature gradient created by two water baths at different temperatures. First, 10 ml of 1$\times$10^{-3}$ mol $\cdot$ $L^{-1}$ TA solution was heated to 50°C in a beaker. Meanwhile, LT samples were placed in another water bath at a different temperature. The LT sample was then immersed in the preheated TA solution for 2 minutes before rapid transfer to a 20°C water bath. This thermal cycling was repeated five times to simulate practical temperature variations. Immediately after cycling, the reacted solution was analyzed using an FLS1000 fluorescence spectrometer at Shanghai Jiao Tong University Instrument Analysis Center. With excitation wavelength set at 315 nm and emission at 425 nm, the fluorescence intensity of 2-HTA was measured. Comparison with a standard 2-HTA solution established a linear relationship between fluorescence intensity at 425 nm and 2-HTA concentration, as shown in Figure 2: see original paper. Figure 2: see original paper presents the standard curve fitting results, correlating fluorescence intensity with apparent hydroxyl radical concentration, enabling evaluation of LT coating pyroelectric performance and wettability self-regulation potential.

## 2.1 Structural Characterization of Lithium Tantalate

The sol-gel method enables controlled preparation of LT, allowing regulation of substrate, coating thickness, crystallinity, and particle size—all parameters influencing pyroelectric performance.

### 2.1.1 Effects of Substrate and Thickness

Figure 3: see original paper shows LT coatings prepared on Ti substrates exhibiting significant cracks and particle accumulation, substantially reducing coating

quality. Crack formation is attributed to thermal expansion coefficient mismatch between LT coatings and Ti substrates, causing stress accumulation during high-temperature annealing. Particle accumulation results from impurity incorporation during coating cracking. To address this, a platinum (Pt) interlayer was introduced between Ti and LT, creating Ti-Pt substrates. Figure 3: see original paper demonstrates that Pt incorporation yields smooth, uniform coating surfaces, significantly improving quality through better thermal expansion coefficient matching and reduced interfacial stress.

In pyroelectric materials, pyroelectric potential is proportional to thickness, while thinner materials are favored for faster response times and minimal impact on surface thermal resistance and mechanical properties. Thus, tradeoffs between pyroelectric potential and thickness must be considered. Coating thickness can be controlled by repeating spin-coating and curing steps. Cross-sectional SEM images in Figure 3: see original paper and (d) show coatings after different spin-coating cycles (3 and 6 times). Figure 3: see original paper reveals uniform banded material accumulation with a thickness of approximately 250 nm, confirming successful and controllable synthesis. Increasing cycles to 6 times increased thickness to approximately 450 nm, as shown in Figure 3: see original paper, accompanied by more pronounced disordered stripe-like accumulation, indicating cumulative morphological effects from repeated synthesis.

### 2.1.2 Effects of Preparation Parameters

This study systematically varied curing time, sol settling time, and annealing temperature to investigate their effects on LT coating crystallinity, quality, particle size, and pyroelectric performance, with specific parameters shown in [Figure 4: see original paper]. SEM images of six LT coatings in Figure 4: see original paper reveal significant surface morphology variations. Coatings in Figure 4: see original paper and (b) consist of fine, uniformly distributed particles, indicating good uniformity and quality. In contrast, Figure 4: see original paper shows larger irregular particles resulting from excessive grain growth during annealing, with grain size increasing at 700°C compared to 650°C, demonstrating annealing temperature's regulatory effect on grain growth. Figure 4: see original paper, (e), and (f) exhibit obvious cracks and particle agglomeration—clear coating defects. Excessive sol settling time may cause agglomeration due to enhanced interparticle interactions, while overly short or long curing times can induce internal stresses and cracking.

XRD patterns of the six coatings, compared with standard $LiTaO_3$ cards in [Figure 5: see original paper], show diffraction peaks at 2 angles of 23.7°, 32.8°, 34.8°, 39°, 40.1°, 42.6°, 48.6°, 53.4°, and 54.8°, corresponding to LT crystal planes (012), (104), (110), (006), (113), (202), (024), (116), and (211), confirming LT crystal presence. Comparison of XRD patterns with SEM images reveals that annealing temperature, curing time, and sol settling time all affect crystal structure and quality. Notably, the 650°C-annealed coating (b) shows clearer diffraction peaks than the 700°C coating (c), indicating that 650°C an-

nealing yields more ideal crystal structures, while higher temperatures may cause excessive grain growth or structural damage. Curing time significantly affects crystallinity and thermal stability—the 10-minute cured coating (d) shows more regular surface morphology with fewer cracks and particle accumulations compared to 15-minute and 20-minute coatings (e) and (f), suggesting excessive curing time may cause surface damage and affect crystalline quality. Sol settling time also markedly influences crystalline quality; the 24-hour settled coating (a) exhibits superior crystallinity compared to 48-hour and 72-hour coatings, as excessive settling time leads to irregular gel precipitation and impurity formation, compromising uniformity. Additionally, $TiO_2$ diffraction peaks detected in surfaces (b), (d), (e), and (f) likely result from Ti substrate surface cracking and exposure at high temperatures, causing titanium-oxygen reactions. Future work should optimize substrate treatment and high-temperature processing to avoid undesirable side reactions.

Particle size effects on coating quality were further investigated by comparing size distributions of coatings annealed at different temperatures (650°C and 700°C), as shown in [Figure 6: see original paper]. Results demonstrate that particle size increases with annealing temperature, rising from 22.64 nm at 650°C to 218.68 nm at 700°C. During final annealing stages, grain growth occurs due to reduced grain boundary energy and atomic diffusion driving forces. Higher temperatures activate atomic thermal motion, accelerating growth rates. However, excessive grain growth adversely affects performance—larger particles reduce coating porosity and may introduce grain boundary defects, compromising mechanical strength and thermal stability.

## 2.2 Pyroelectric Performance of Lithium Tantalate Coatings

### 2.2.1 Pyroelectric Performance of LT Coatings with Different Thicknesses and Particle Sizes

As shown in Figure 7: see original paper, temperature change-induced pyroelectric effects generate positive and negative surface charges. Positive charges react with hydroxide ions in water to form hydrophilic hydroxyl radicals, while negative charges react with dissolved oxygen to create hydrophobic superoxide radicals. Without temperature variation, pyroelectric materials exhibit no spontaneous polarization, and thus no radicals are generated. Hydrophilic hydroxyl radicals promote water spreading, enhancing surface hydrophilicity/wettability, whereas hydrophobic superoxide radicals reduce water spreading, decreasing wettability. Thus, regulating radical generation enables effective surface wettability control.

Based on these characteristics, performance verification experiments were conducted on LT surfaces with different thicknesses and particle sizes, using LT without temperature variation as a control group. Results in Figure 7: see original paper show that all LT surfaces exhibit peaks at 425 nm under heating,

indicating hydroxyl radical generation, while the control group shows none, confirming LT's pyroelectric effect. Using the linear relationship from Figure 2: see original paper, quantitative analysis reveals that the 450 nm-thick LT surface produces 15.27% higher hydroxyl radical concentration than the 250 nm-thick surface. For different particle sizes, the 22.64 nm LT surface shows a 12.47% increase compared to the 218.68 nm surface. These results verify that LT coatings generate more hydroxyl radicals during heating and reveal significant effects of coating thickness and particle size—thicker coatings and smaller particles both enhance pyroelectric performance and radical generation.

### 2.2.2 Enhancement Mechanism of Pyroelectric Effect

Sufficient potential is required for radical generation. Using the pyroelectric equation [27] (Equation (3)), this study analyzed thickness effects. In the equation, $U$ represents pyroelectric potential, $\Delta T$ temperature change, $l$ coating thickness, $p$ pyroelectric coefficient, and  dielectric constant. Pyroelectric potential correlates positively with thickness, enabling thicker coatings to generate more hydroxyl radicals during heating.

At the nanoscale, reduced LT particle size increases specific surface area, enhancing synergistic effects among thermoelastic, dielectric, and piezoelectric effects that amplify pyroelectric response. Decreased surface atomic coordination number intensifies spontaneous polarization in nanocrystals, increasing local polarization [28]. Research shows both piezoelectric and pyroelectric potentials increase with decreasing nanocrystal size or increasing shape factor. Equation (4) illustrates how nanocrystal dielectric constant depends on size and shape, where $_{33}\widehat{\ }\infty$ is the bulk value, $S$ is solid-gas transition entropy, $R$ is the ideal gas constant, $D$ is nanocrystal characteristic size, $h$ is lattice constant, and $\lambda$ is shape factor. This phenomenon ultimately yields greater pyroelectric potential at smaller particle sizes, generating more hydroxyl radicals during heating. Future optimization may involve fine-tuning material properties such as dielectric constant, crystal structure, and thickness.

### 2.2.3 Verification of Pyroelectric Effect on Surface Wettability Regulation

Figure 7: see original paper compares fluorescence spectra of TA solutions after five heating-cooling cycles with LT coatings. Results show significantly higher peaks at 425 nm in heated solutions versus cooled solutions, indicating hydroxyl radical generation during heating and replacement by superoxide radicals during cooling, reducing hydroxyl radical content by 7.50% compared to heated solutions. This verifies that LT's pyroelectric effect modulates hydroxyl and superoxide radical concentrations under temperature variation, thereby changing surface wettability through temperature control and demonstrating LT's potential for temperature-sensitive wettability self-regulation.

## Conclusions

This study successfully prepared LT coatings via the sol-gel method and systematically investigated effects of key process parameters on coating quality, thickness, crystal structure, particle size, and pyroelectric performance. The findings are summarized as follows:

1. Substrate selection significantly affects coating quality. Introducing Pt as an interlayer substantially improved LT coating-Ti substrate interface bonding and reduced stress accumulation from thermal expansion coefficient mismatch. Adjusting spin-coating cycles effectively controlled coating thickness, thereby influencing pyroelectric performance.

2. Preparation parameters regulate coating crystallinity, thickness, and particle size. Curing time, sol settling time, and annealing temperature are critical for crystallinity. Optimal parameters were identified as 15 minutes curing, 24-hour sol settling, and 650°C annealing, yielding LT coatings with optimal crystallinity and pyroelectric performance. Spin-coating cycles control thickness, while LT particle size increases with temperature.

3. Coating thickness and particle size significantly affect pyroelectric characteristics. Increased thickness and decreased particle size enhance pyroelectric performance. Fluorescence spectroscopy confirms increased hydroxyl radical content during heating and validates LT coatings' ability to regulate hydroxyl radical concentration through thermal cycles.

This study successfully produced high-crystallinity, high-performance pyroelectric LT coatings with controlled preparation via sol-gel parameter optimization, demonstrating temperature-sensitive wettability self-regulation capability. These results provide preliminary exploration for large-scale production and application of LT coatings in two-phase heat transfer systems, offering new solutions for efficient marine energy utilization and green energy system transformation.

## Author Contributions

Miaoxin Ma: Drafted the manuscript; conducted research; acquired data; analyzed/interpreted data.
Hui He: Conceived and designed experiments; critically reviewed intellectual content.
Qi Lu: Performed statistical analysis; acquired funding; provided administrative, technical, or material support.
Xiaojing Liu: Acquired funding; provided administrative, technical, or material support; supervised the project.

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*Note: Figure translations are in progress. See original paper for figures.*