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Article

Enhancing Atomic Force Microscopy Sample Preparation Using a Modified Microwave-Assisted Drying System

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Abstract

Atomic Force Microscopy (AFM) is widely used for high-resolution topographic imaging, but sample preparation remains a critical factor influencing image quality. Wet preparation techniques improve nanoparticle dispersion but often introduce residual solvent layers that degrade imaging performance. This study investigates a modified microwave-assisted drying system designed to enhance AFM sample preparation. A commercial microwave was adapted with precise power, temperature, and time controls to optimize drying conditions and minimize aggregation. Various sample preparation methods were evaluated, including dry preparation, conventional wet preparation, and microwave-assisted drying. AFM characterization showed that the modified microwave system produced samples with more uniform morphology, reduced particle aggregation, and improved topographic resolution. Contact angle measurements indicated enhanced solvent removal, increasing hydrophobicity and better substrate interaction. These results demonstrate that controlled microwave-assisted drying effectively improves AFM imaging quality, offering a practical alternative to conventional drying methods.

Keywords: Atomic Force Microscopy, Microwave-Assisted Drying, Wet Preparation, Topographic Profile

INTRODUCTION

Atomic Force Microscopy (AFM) is a crucial analytical tool in various scientific fields, including physics, chemistry, biology, and medicine [1][2][3]. Its capability to generate high-resolution topographical and morphological images at the nanometer scale has established AFM as one of the most powerful techniques in nanotechnology research [4]. Unlike optical and electron microscopes, which rely on light or electron beams, AFM operates by sensing surface characteristics through a mechanical probe [5][6]. This technique uses Van der Waals, capillary, and adhesion forces between the cantilever tip and the sample surface at nanometer-scale distances [7]. During scanning, the cantilever tip moves in response to these forces, following the surface contours of the

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sample. The use of piezoelectric elements enables precise nanoscale movements, facilitating highly accurate surface mapping with minimal distortion or unintended magnification [8][9][10].

AFM provides various operational modes tailored to specific material characterizations, including topography imaging, electrical property analysis, nanomechanical property assessment, magnetic property examination, and thermal property evaluation [11][12][13]. Among these, topographical analysis is the most widely utilized method in Indonesia, particularly by researchers at Diponegoro University. This mode is versatile and applicable to various samples across multiple scientific disciplines.

A diverse array of materials, such as membranes, polymers, and thin films, can be effectively analyzed using AFM. However, powder-based samples, including nanosilver, nanogold, and various metal oxides, necessitate proper sample preparation to achieve high-quality imaging [14][15]. The sample preparation process is vital in enhancing image resolution and accuracy by addressing particle size, humidity, and material properties. For optimal imaging, the sample must be rigid, uniformly dispersed on the substrate, and possess a surface roughness lower than the substrate [16]. Improper sample preparation can degrade image quality due to artefacts introduced by residual solvent layers or water meniscus effects that interfere with cantilever movement [17].

There are currently two main atomic force microscopy (AFM) sample preparation techniques used in Indonesia: dry and wet preparation. Dry preparation involves directly immobilizing sample particles on a substrate, offering a simple, rapid method. However, this approach often results in uneven particle dispersion, which leads to aggregation and decreased imaging accuracy. In contrast, wet preparation uses a solvent to disperse the sample before drying, somewhat reducing aggregation. Nevertheless, a residual solvent layer may adhere to the particle surface, which could affect the final imaging results [16]. Therefore, an effective drying method is crucial for removing solvent residues and improving AFM imaging quality.

Previous studies have reported that conventional microwave-based drying can accelerate solvent evaporation, but often results in uneven heat distribution due to microwave hotspots [18]. These conditions can cause morphological distortion, particle size heterogeneity, and dispersion instability. Additionally, most commercial microwave systems do not precisely control parameters such as power, temperature, and time, making it difficult to achieve reproducible results [19]. These limitations demonstrate a research gap in the microwave-based AFM sample drying stage, particularly in parameter optimization to prevent aggregation and maintain particle morphology. Based on this knowledge gap, this study proposes developing a modified microwave

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system with more precise control over power, temperature, and time to improve and standardize the quality of AFM sample preparation.

The study aims to develop a specialized AFM sample drying system and evaluate its impact on imaging performance. Conventional drying methods, such as heating with a hot plate, are at risk of contamination due to exposure to environmental conditions. Another approach is to use a microwave reactor, which generates heat by interacting with the sample material. However, commercial microwave units lack precise parameter control, limiting their suitability for AFM sample preparation. To address this challenge, the study focuses on designing and optimizing a modified microwave reactor as a specialized tool for preparing powdered AFM samples to improve image quality and analytical reliability.

METHODS

This study was conducted in three main stages: fabrication of a modified microwave drying system, analysis of sample preparation techniques, and characterization of AFM imaging results.

Fabrication of a modified microwave drying system

In the first stage, a specialized microwave-based drying system was developed to enhance the preparation of powder samples using the wet preparation technique. Commercial microwave ovens are designed primarily for food heating and lack the precision required for controlled sample drying. To address this limitation, modifications were made to three key components: power control, temperature regulation, and time control. These modifications allowed for precise user control over the drying process, enabling the optimization of conditions specific to different sample types (Figure 1). The modified system was designed to ensure uniform heating and efficient solvent removal without introducing contaminants or affecting sample morphology.

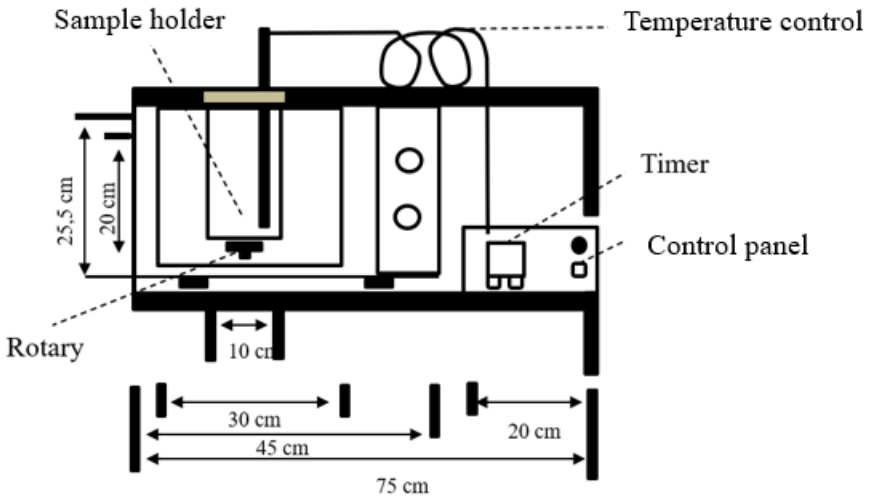

Figure 1. Schematic of the modified microwave reactor as a supporting tool for wet preparation of powder samples

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Sample preparation techniques

The second stage involved comparing different sample preparation techniques to assess their impact on AFM imaging quality. Five preparation methods were employed: dry preparation, wet preparation, wet preparation with hotplate drying, wet preparation using a commercial microwave, and wet preparation using the modified microwave reactor. Nanosilver powder was used as the model sample in all experiments.

In the dry preparation technique, a layer of carbon tape was attached to the sample holder, and nano silver powder was carefully sprinkled onto the surface. The sample was then evenly spread using a spatula to ensure uniform coverage. In contrast, wet preparation involved dispersing nano silver powder in distilled water at a concentration of 0.1 mg/mL, followed by mechanical stirring using a shaker to ensure homogeneous dispersion. A controlled volume of this dispersion was then drop-cast onto the sample holder and left to dry at room temperature for 24 hours, forming a thin film.

Modified wet preparation techniques were introduced to investigate the effect of drying conditions. In the hotplate-assisted method, the sample was placed on a hotplate set to 80°C for 3 hours until complete drying. For microwave-assisted drying, two approaches were compared: the use of a commercial microwave and the custom-modified microwave reactor. The sample was exposed to 300 W of microwave power for 1 hour in the commercial microwave method. The modified microwave system adjusted precise power, temperature, and time parameters to achieve optimal drying while minimizing sample aggregation and morphological distortions.

Table 1. Parameters of each AFM sample preparation method

Method	Dispersion volume (µL)	Drying time	Temperature / Power
Dry Preparation	–	–	–
Wet Preparation	Controlled	24 h	Room temperature
Wet Preparation + Hotplate	Controlled	3 h	80°C
Wet Preparation + Commercial Microwave	Controlled	1 h	300 W
Wet Preparation + Modified Microwave Reactor	Controlled	Optimized	Precise power, temperature, and time control

Characterization of AFM results

The final stage focused on characterizing the prepared samples using NX10 AFM (Figure 2). Image acquisition and processing were conducted using SmartScan and XEI Park System software. The effectiveness of each preparation method was evaluated based on several key parameters, including

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surface topography, morphological uniformity, and aggregation/agglomeration behavior. Contact angle measurements were also performed to assess the solvent's residual moisture content and hydrophilicity on the sample-substrate interface. These analyses aimed to determine the most effective preparation technique for obtaining high-quality AFM images with minimal artifacts and optimal surface resolution.

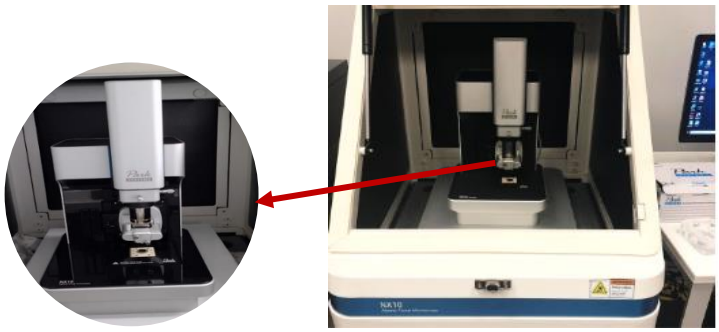

Figure 2. Atomic Force Microscopy NX10 Park System

RESULT AND DISCUSSION

The microwave system was modified by adapting a commercial microwave with three main control elements: power control, temperature control, and time control. The addition of a temperature control system allows for the regulation of the actual temperature within the sample, which is placed in a glass tube equipped with a thermocouple. This system ensures that the set temperature depends on the applied power, where an increase in power accelerates the achievement of the desired temperature. With this modification, the reactor chamber inside the microwave becomes more controlled, enabling the determination of optimal conditions for each sample type to enhance preparation effectiveness.

The temperature control system is designed separately from the main system. It consists of several components: a miniature circuit breaker (MCB), temperature display, indicator lamp, on/off switch, and a thermocouple placed inside a glass tube or sample chamber in the microwave. This setup allows for real-time monitoring of the actual sample temperature. Additionally, the power and time settings of the microwave, which were originally discrete, have been modified to be continuous, providing greater flexibility for users. The sample chamber is made of glass material to minimize interactions between microwaves and the thermocouple and certain sample types that may interact directly with microwaves. The temperature control system and microwave are integrated into a single framework, ensuring a well-coordinated system. The modification details can be seen in Figure 3.

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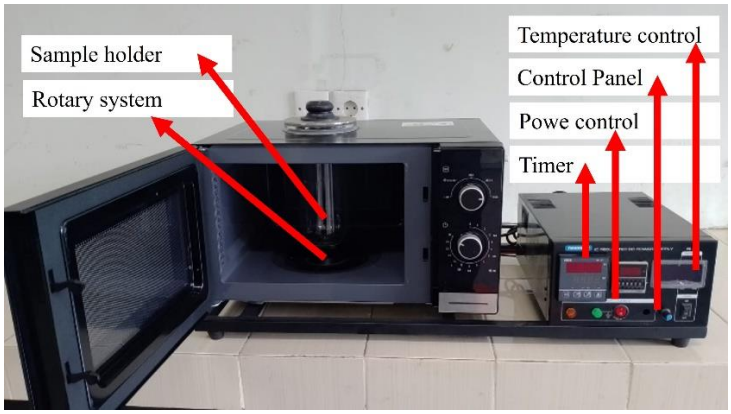

Figure 3. Fabrication details of the modified microwave reactor for sample drying

Figure 4 presents the morphological analysis of sample surfaces prepared using various methods, including dry preparation, wet preparation (WP), WP furnace, WP hotplate, WP microwave, and WP modified microwave. Morphological defects in thin films are indicated by the symbols "○", "□", and "x". Visually, samples prepared using the dry preparation method exhibit significant structural damage, with massive aggregation and highly heterogeneous particle distribution. This phenomenon occurs due to overlapping particles on the substrate without sufficient order in thin-film formation. Meanwhile, the wet preparation method produces blurred and less defined surfaces, likely due to residual solvent covering the film surface. These findings indicate that dry preparation and wet preparation methods without supporting tools do not result in proper dispersion, contributing to defects in thin-film growth on the substrate. In contrast, WP with a modified microwave produces a more homogeneous surface morphology, with a more uniform particle size distribution and minimal aggregation across most areas.

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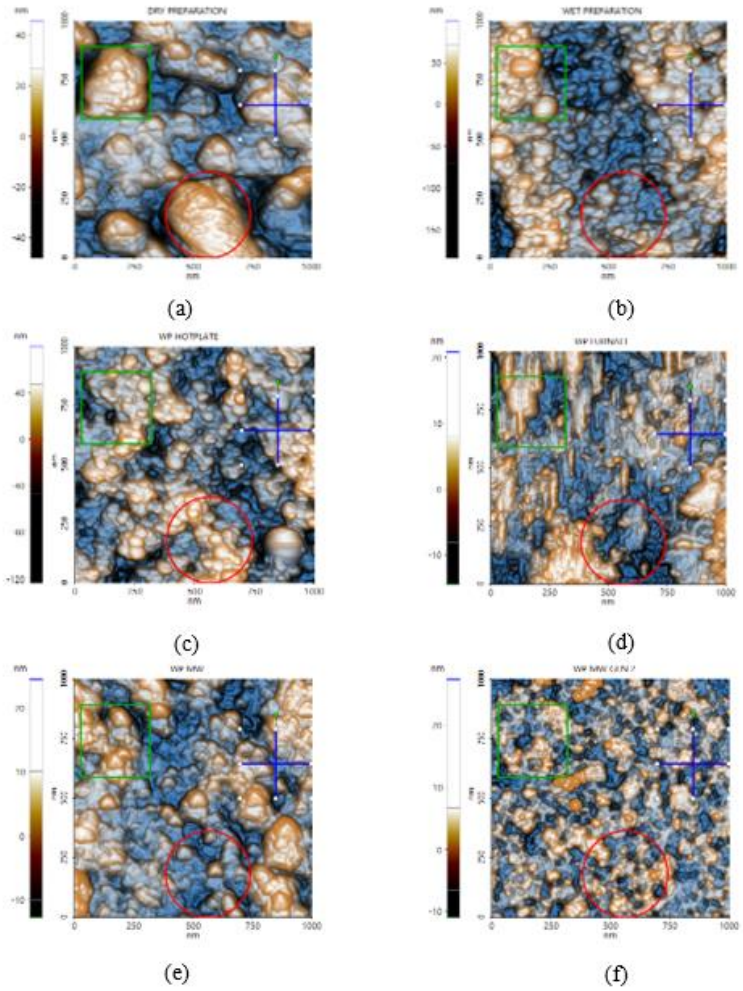

Figure 4. Surface morphology of thin films using the (a) dry preparation, (b) wet preparation, (c) WP furnace, (d) WP hotplate, (e) WP microwave, and (f) modified WP microwave methods

Figure 5 shows the topography of nanosilver films prepared using four wet preparation methods. Aggregate and agglomerate formations are reflected in graphs that resemble "mountain" or "hill" shapes, where larger and more numerous aggregates lead to increased graph fluctuations. For WP methods using a furnace and hotplate (Figures 5a and 5b), the resulting topography images show differences in sample shapes, despite both methods employing the same drying principle of heat transfer through convection and conduction. Furnace drying in a closed system leads to changes in sample shape and size due to direct heat interaction. Conversely, in the hotplate method, heat flow is reduced by the open environment, preventing significant shape alterations. The aggregate topology graphs for both methods exhibit similar distribution patterns.

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For WP methods using a commercial microwave and a modified microwave (Figures 5c and 5d), microwave-assisted heating results in better topography than other methods. Although aggregation and agglomeration are still observed in WP with a commercial microwave, their intensity is lower than in WP without supporting tools. Meanwhile, the WP method using a modified microwave shows a significant improvement in particle and aggregate distribution homogeneity, as evidenced by a more stable aggregation pattern in the graph. This phenomenon likely occurs because microwave heating results in uneven energy distribution, where materials with high microwave absorption, such as solvents, absorb more energy compared to opaque materials like nanosilver [20][21].

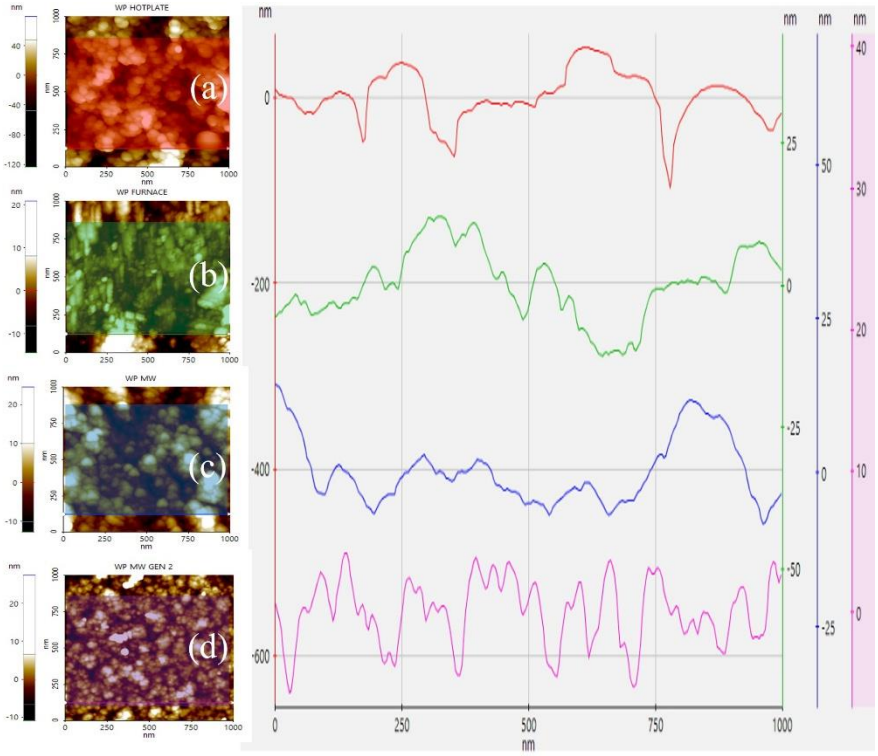

Figure 5. Film contour profiles and topographic regularity graphs of (a) WP furnace, (b) WP hotplate, (c) WP microwave, and (d) modified WP microwave

Figure 6 presents the contact angle analysis of films prepared using the WP furnace, WP hotplate, WP microwave, and WP modified microwave, with respective contact angles of 39.3º, 38.4º, 55.5º, and 57.1º. Material characteristics based on contact angle values are classified as hydrophilic (<30º), partially wetted (30-80º), and hydrophobic (>80º) [22]. These results indicate that films prepared using a modified microwave exhibit the highest contact angle among all methods, suggesting increased hydrophobicity. Consequently, this method produces films with lower water interaction than other WP methods.

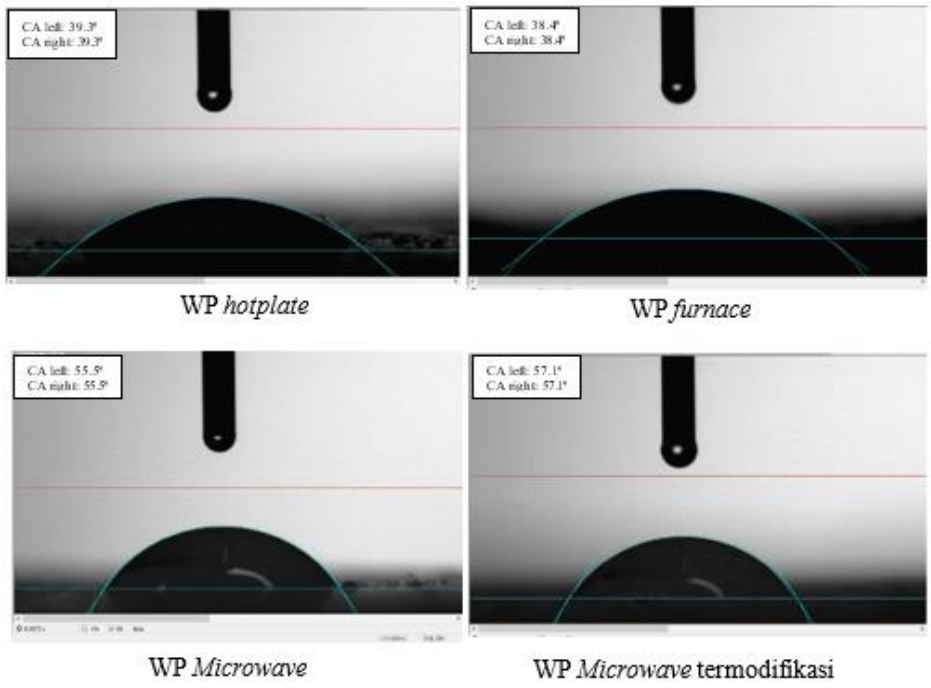

Figure 6. Contact angle analysis of (a) WP furnace, (b) WP hotplate, (c) WP microwave, and (d) modified WP microwave

CONCLUSION

This study demonstrates that a modified microwave-assisted drying system significantly improves AFM sample preparation by optimizing drying conditions and reducing solvent residue. Compared to conventional methods, microwave-assisted drying enhances nanoparticle dispersion, minimizes aggregation, and improves topographic imaging quality. Contact angle analysis further confirms superior solvent removal, leading to increased hydrophobicity. The findings highlight the potential of controlled microwave drying as a reliable and efficient technique for AFM sample preparation, contributing to improved analytical accuracy in nanomaterial characterization.

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