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Calculating Thin Film Strain From XRD Data

Menghitung Regangan Film Tipis dari Data XRD

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Abstract

This study aims to investigate the crystallite size and microstrain of Cu plate samples subjected to various hot water treatment times using the Williamson-Hall method and XRD profiles. The estimated crystallite sizes obtained from this method were compared with scanning electron microscope (SEM) observations, showing good agreement. The Williamson-Hall method was also utilized to determine microstrain, and the influence of temperature on the mechanical properties of copper was examined, highlighting the dominant role of dynamic softening mechanisms in copper deformation. Line broadening analysis was employed to compare crystallite size and microstrain, demonstrating the usefulness of the Williamson-Hall method in cases where both factors contribute to line broadening. The research underscores the reliability of the Williamson-Hall method in accurately determining crystallite size and microstrain from XRD line broadening analysis. The findings reveal that increasing treatment time leads to higher strain in thin films, and the calculated crystal size aligns well with reference measurements. This study provides valuable insights for designing and synthesizing copper-based materials with improved structural integrity and desired mechanical properties.

Highlights:

- Accurate characterization: The Williamson-Hall method and XRD profiles are reliable techniques for determining crystallite size and microstrain in Cu plate samples, validated by comparison with SEM observations.
- Temperature influence: Temperature plays a crucial role in the mechanical properties of copper, with dynamic softening mechanisms dominating the deformation behavior.
- Line broadening analysis: Line broadening analysis allows for the comparison of crystallite size and microstrain, demonstrating the utility of the Williamson-Hall method in cases where both factors contribute to line broadening.

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Introduction

Different synthesis methods used to create nanostructured materials can result in a variety of structural flaws, including lattice strain. The diffraction peaks may get broader in crystals with nanometer-sized particles in the range of 10 nm to 100 nm. The crystallite size and lattice strain are determined by the widening of the diffraction peak. Scherrer's formula can be used to determine the crystallite size. In addition to these methods, the Williamson-Hall method was also used to determine the size of crystallites. The lattice strain is also calculated using this method. As a result, ZnO nanopowders were created in this work by precipitation and calcined at various temperatures. The Williamson-Hall (W-H) method was used to determine the crystallite size and lattice strain..

Thin film microstrain

Measurement of strain or deformation in the individual grains of thin films is referred to as thin film microstrain. To carry out microstrain measurements, different techniques like EBSD, -HRXRD, Raman microspectrometry, and X-Ray diffraction (XRD) can be employed. The values of microstrain measured in thin films containing multiple crystals can offer insights into various structural parameters, including crystallite size, dislocation density, and the sensitivities of the techniques employed for mapping microstrain in thin films. [1].

Williamson-Hall (W-H) Analysis

X-ray profile analysis is a simple and powerful tool to estimate the crystallite size and lattice strain. This method is attributed to G. K.Williamson and his student, W. H. Hall [2]. It relies on the principle that the approximate formulae for size broadening, BL, and strain broadening, ße, vary quite differently with respect to Bragg angle, 0.

Literature Review

ZnO nanopowders were created by Mote et al. using coprecipitation methods. At 450 °C, grains with an average size of roughly 50 nm were created. Using W-H analysis and several models, such as the uniform deformation model, the uniform deformation stress model, and the uniform deformation energy density mode, the physical parameters such as strain, stress, and energy density values were also computed. [3].

Fe-doped ZnO nanoparticles with a hexagonal wurtzite shape have been created by Prabhu et al. Williamson-Hall (W-H) analysis and a size-strain plot were used to examine the effects of crystallite sizes and lattice strain on the peak broadening of Fe doped ZnO nanoparticles. All of the samples' XRD peaks had their strain, stress, and energy density characteristics computed using the size-strain plot method (SSP), the uniform stress deformation model (USDM), the uniform deformation energy density model (UDEDM), and the uniform deformation model (UDM). studies from W-H analysis, SSP, and TEM studies were corroborated by the mean particle size of Fe doped ZnO-NPs[4].

Using nanocrystalline Ni and Cu powders, Brandstetter et al. utilized Williamson-Hall analysis to determine grain size and lattice strain[5].

Similar to this, Zak et al. used the sol-gel process to create ZnO nanopowders. The sample has a hexagonal wurtzite phase and is crystalline in form. XRD was used to investigate the ZnO nanoparticles' phase evolution. To investigate the individual effects of crystallite sizes and lattice strain on the peak broadening of the ZnO nanoparticles, the Williamson-Hall analysis and size-strain plot approach were utilized. ZnO-NPs that were calcined at 750 °C showed a nonuniform strain and an average particle size of roughly 20 nm in the TEM image. The TEM results and the W-H technique results were in good agreement[6].

In order to measure the lattice strain on ZnO particles produced by a straightforward, surfactant-assisted combustion synthesis, Prabhu et al. also used the W-H method[7].

ZnO flat thin film was created by Thool et al. using chemical bath deposition methods. The Scherrer method and the Williamson-Hall method were used to evaluate the crystallite size and lattice strain from X-ray line broadening[8].

After heating the precursor at 350 degrees Celsius, Sarma et al. generated ZnO nanopowders using an easy, cost-effective precipitation technique. The W-H method was used to analyze the line broadening of ZnO nanoparticles caused by the small crystallite size and strain[9].

Aim of this study

The objective is to calculate strain and crystallite size from XRD data using Willium- Hall analysis.

Method

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We will calculate crystallites size and strain from XRD data using Williamson-Hall (W-H) plot method. The broadening (B_T)of the peaks in the XRD data is caused by the combined effects of crystallite size (B_D) and microstrain (B_e).

Total broadening = Broadening due to crystallites size + Broadening due to strain

Where:

$B_T = B_D + B_e$

B_T is the total broadening

B_D is broadening due to crystallite size

B_e is the broadening due to strain

From the Scherer equation, we know that,

$B_D = k\lambda/(D\cos\theta)$

Where

B_D is FWHM (ie broadening of the peak) in radians k= 0.9 is the shape factor, λ= 0.15406 nm is the wavelength of X-ray source, D is the crystal size, is the peak position in radians similarly.

The XRD peak broadening due to micro strain is given by

$B_e = 4\varepsilon\tan\theta$ (2-3)

Where B_e. is broadening due to strain and θ is the peak position in radians.

Substituting equation (2-2) and (2-3) in equation (2-1),

we obtain:

$B_T = k\lambda/(D\cos\theta) + 4\varepsilon\tan\theta$

As we know that $\tan\theta = \sin\theta/\cos\theta$

Therefore, equation (2-4) can be written as

$B_T = k\lambda/(D\cos\theta) + 4\varepsilon\sin\theta/\cos\theta$

Multiplying both sides by cosθ,

$\cos\theta \times B_T = \cos\theta \times k\lambda/(D\cos\theta) + \cos\theta \times 4\varepsilon\sin\theta/\cos\theta$

$B_T\cos\theta = k\lambda/D + 4\varepsilon\sin\theta$

Equation (2-5) represent a straight line, in which is the gradient (slope) of the line and kλ/D is the y- intercept.

Consider the standard equation of a straight line,

$y = mx + c$

Where is the slope of line and is the y- intercept

Comparing equation (2-5) with equation (2-6), we have:

$y = B_T\cos\theta$ i

$m = \varepsilon$ ii

$x = 4\sin\theta$ iii

$c = k\lambda/D$ iv

We will plot (4 sin θ) on x-axis and (B_T cosθ) on y-axis.

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The value of m which represent gradient (slope) of the line it will be the value of the strain ε. Finally, we will calculate crystallites size from the y-intercept

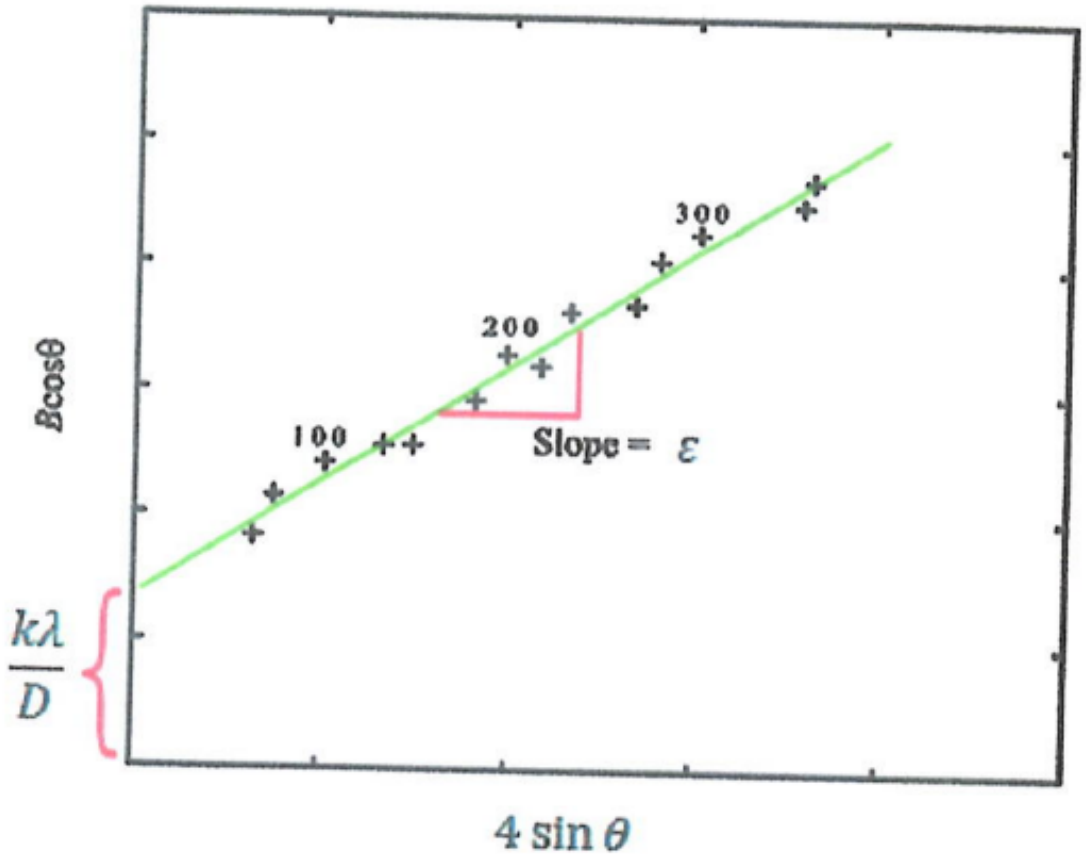

Figure 1.

We need to follow the following steps to calculate the strain from XRD data:

1- By using originLab program we will determain 20 (degree) for each peak and FWHM, BT (in degree) from XRD data.

2- Convert and FWHM BT from degree to radians.

3- Determain the value of B_T cosθ and 4 sinθ

4- Plot B B_T cosθ on y-axis and 4 sinθ on x-axis to obtain the final W-H plot in origin Lab program as scatter graph.

5- Preform liner fitting for the scatter graph.

6- The slope of the plot is the strainε .

The XRD data adoptede from the refreance [10].

Results and Discussions

Figures 3-1 show XRD images of the Cu samples for various hot water treatment time.

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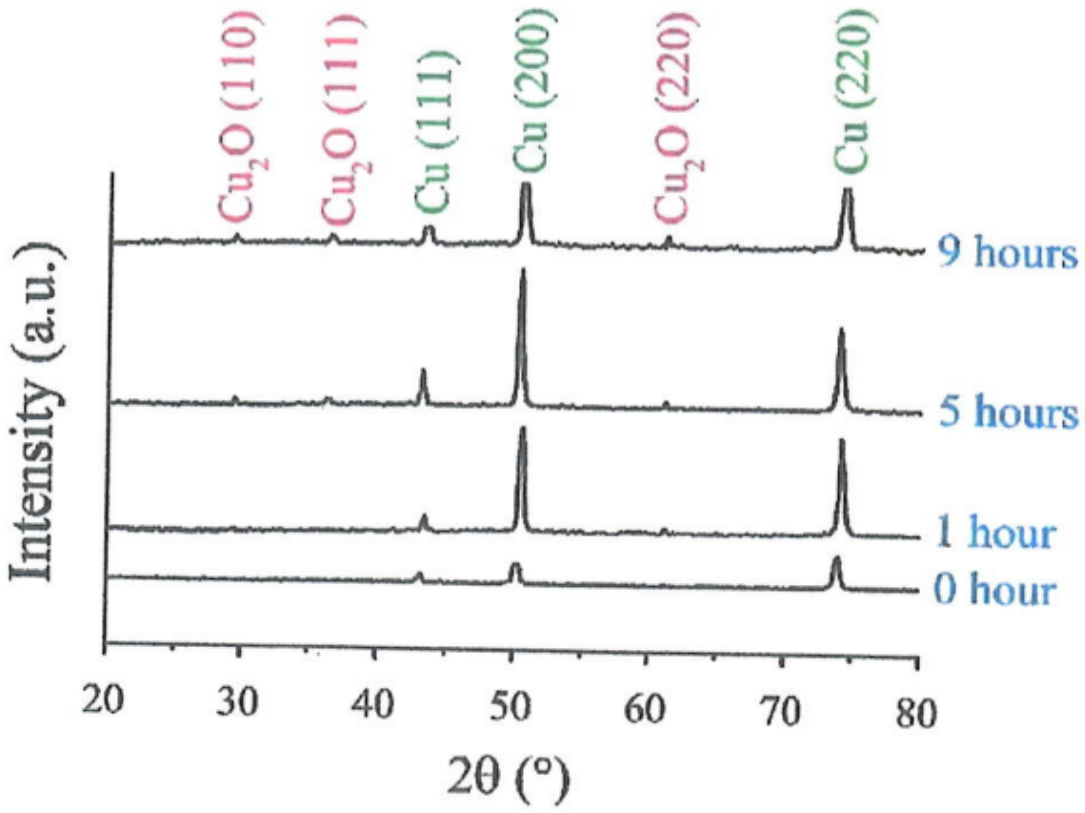

Figure 2.

Figure 3-1: XRD profiles for the dominant oxide peaks of Cu samples for various hot water treatment time [10].

Figure 3-2 shows thr realtion between on y-axis and on x-axis and the calculatet slope represent the stanin () according to equation 2-3.

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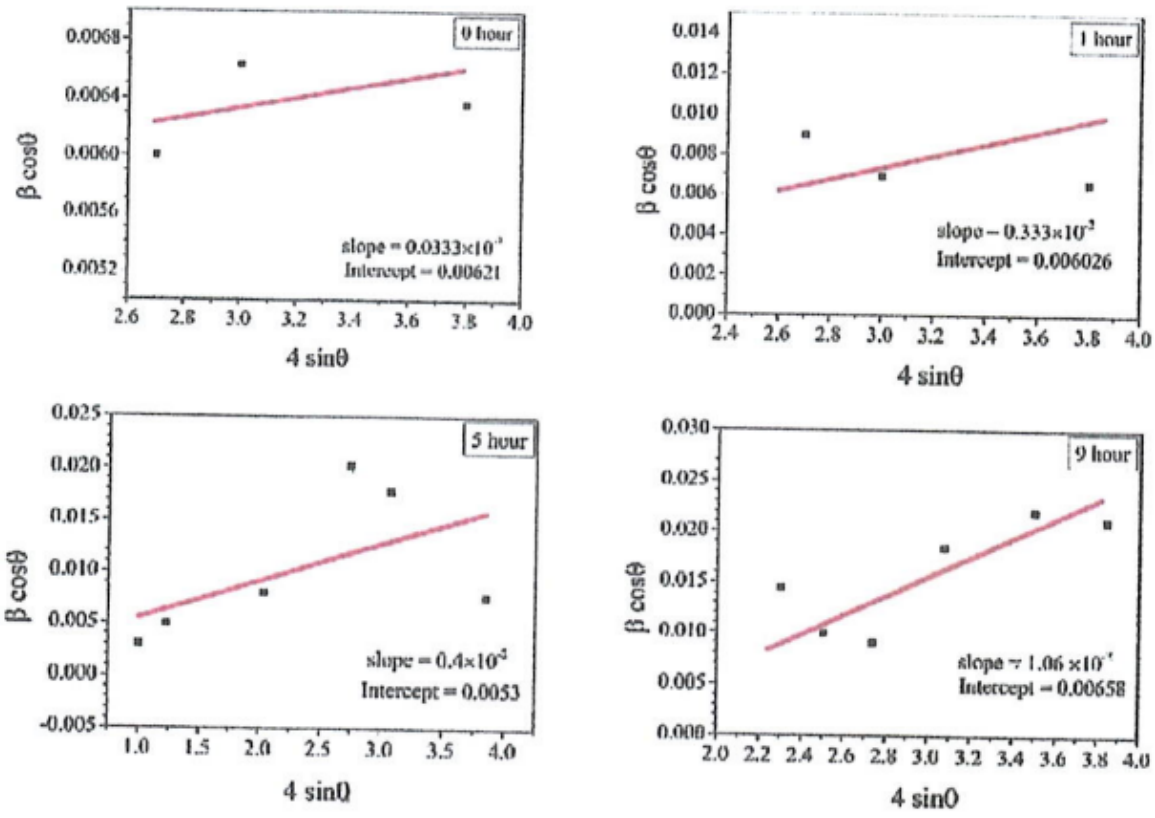

Figure 3.

Figure 3-2: The W-H Analysis Cu samples for various hot water treatment time.

Table 1 reveal calculated crystal size of samples for various hot water treatment time where the intercepts of the plots in figure 3-2 represent the value kλ/D in equation 2-5, where *D* can be obtained which is represent the crystal size by 1-2 Williamson- Hall (W-H) method. The calculate crystal size by Scherer method also included in Table 3-1 for comparison.

Treatment time	D_(W-H) (nm) Williamson-Half method	D_sh (nm)[9] Scherer method
0	22	21
1	23	23
5	26	27
7	21	21

Table 1. *calculate crystal size*

Temperature can affect the mechanical properties of copper, where the dynamic softening mechanisms play a dominant role in the deformation of copper [11].

Conclusion

1- From the calculation of strain, we can observe that increasing treatment time increases the strain of the thin films,

2- The calculated crystal size was in good agreement with the one measured by reference [10].

Recommendation

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Determine other relevant physical parameters such as stress and energy density.

References

1. N. Schäfer, A. J. Wilkinson, T. Schmid, et al., "Microstrain distribution mapping on CulnSe2 thin films by means of electron backscatter diffraction, X-ray diffraction, and Raman microspectroscopy," Ultramicroscopy, vol. 169, pp. 89-97, Oct. 2016.
2. G. K. Williamson and W. H. Hall, "X-ray line broadening from filed aluminium and wolfram," Acta Metallurgica, vol. 1, no. 1, pp. 22-31, Jan. 1953.
3. V. D. Mote, Y. Purushotham, and B. N. Dole, "Williamson-Hall analysis in estimation of lattice strain in nanometer-sized ZnO particles," Journal of Theoretical and Applied Physics, 2012.
4. Y. T. Prabhu, K. Venkateswara Rao, V. Sesha Sai Kumar, and B. Siva Kumari, "X-ray Analysis of Fe doped ZnO Nanoparticles by Williamson-Hall and Size-Strain Plot Methods," International Journal of Engineering and Advanced Technology (IJEAT), vol. 2, no. 4, pp. 876-879, Apr. 2013.
5. P. M. Brandstetter, S. Van Petegem, and H. Van Swygenhoven, "Williamson-Hall anisotropy in nanocrystalline metals X-ray diffraction experiments and atomistic simulations," Acta Materialia, vol. 56, pp. 165-176, 2008.
6. A. Khorsand Zak, W. H. Abd. Majid, M. E. Abrishami, and R. Yousefi, "X-ray analysis of ZnO nanoparticles by Williamson-Hall and size-strain plot methods," Solid State Sciences, vol. 13, pp. 251-256, 2011.
7. Y. T. Prabhu, K. Venkateswara Rao, V. Sesha Sai Kumar, and B. Siva Kumari, "X-Ray Analysis by Williamson-Hall and Size-Strain Plot Methods of ZnO nanoparticles with fuel variation," Journal of Nano Science and Engineering, vol. 4, pp. 21-28, 2014.
8. G. S. Thool, A. K. Singh, R. S. Singh, A. Gupta, and M. A. B. H. Susan, "Facile synthesis of flat crystal ZnO thin films by solution growth method: A microstructural investigation," Journal of Saudi Chemical Society, vol. 18, no. 5, pp. 712-721, Nov. 2014.
9. H. Sarma and K. C. Sarma, "X-ray peak broadening analysis of ZnO nanoparticles derived by precipitation method," International Journal of Scientific and Research Publications, vol. 4, no. 3, pp. 1-5, Mar. 2014.
10. M. F. Abdullah, "Formation And Characterization Of Cupper Oxide Nanostructures Prepared By Hot Deionized Water," Thesis, College of Science, University of Kirkuk, 2022.
11. S. Liu, A. Wang, and J. Xie, "Effect Of Deformation Temperature, Strain Rate And Strain On The Strain Hardening Exponent Of Copper/Aluminum Laminated Composites," Advanced Composites Letters, vol. 27, no. 4, pp. 91-98, 2018.

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