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# Determination of stress from HR-XRD and Raman for GaN/AlInN/AlN/Sapphire HEMTs

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**Keywords**

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**Abstract**

In this study, structural properties of GaN/AlInN/AlN/sapphire high electron mobility transistors (HEMTs), grown by metal organic chemical vapor deposition technique, are investigated. High resolution X-ray diffraction technique (HR-XRD) and Raman mesurements are made to determine stress values and stress type for GaN layers dependent on Al content. It is seen that stress values gained from these two techniques are approximately at the same level. It is noticed that there is tensile stress in all three samples according to Raman shift measurements. Also strain values are calculated by using full width at half maximum (FWHM) values in HR-XRD pattern.

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## 1. INTRODUCTION

Silicon (Si) and III-V group semiconductors are not so convenient for designing and producing optoelectronic devices operating at blue region of electromagnetic spectra. For example GaAs based electronic devices can not be used at high temperatures. Group III nitrites are applicable for these kind of applications. Band gap of group III nitrites are wide and have direct transition. Forbidden energy band gap of InN, GaN and AlN are 0.7 eV, 3.4 eV and 6.2 eV respectively (Vurgaftman et al., 2003). GaN, InN and AlN are some elements of group III nitrites which have hexagonal crystal structure (Bayal et al., 2023). AlN, GaN, proved themselves in application of high electron mobility transistors (HEMTs), because of difference between In and N atoms in dimension (Li et al., 2006).

X-ray diffraction may be used for determining micro-structural properties of samples such as crystallite size, strain and stress. XRD is a harmless method for determining such properties. w-2 theta method is used frequently for determining such properties.

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Raman spectroscopy is applied in order to characterize semiconductor materials in solid state Physics. There is great interest on optical properties of low dimension systems. Linear Raman spectroscopy can be used to determine phonon energy and dependency of them to micro structural dimension in nano scale. Raman spectroscopy investigates acoustic and optic phonons.

AlInN thin films became promising materials with technological developments and took attention of researchers (Huang et al., 2009). Modification of band gap of InN to a narrower value maintained new applications for III-nitrite systems and increased variety on obsenities for these new applications (Supekar et al., 2018). III-group nitirites are proved as excellent material systems for opto electronic devices. This material system became center of attention for photo voltaic solar cell systems. (Zhang et al., 2003). AlInN semiconductors are respected as perfect candidates for solar cell applications (Wu et al., 2008). This situation has relation with consistincy of adjustment of forbidden band gap between 0.7 eV and 3.4 eV (Harish et al., 2021). Production of renewable energy systems by using these type of semiconductors plays central role in decreasing fossil fuel consumption for coming decades (Miyata et al., 1997).

In this study characterization of samples is made also by HR-XRD method. XRD is a harmless method for materials. Stress values for GaN layers of samples are calculated not only by XRD but also Raman spectroscopy. Results gained from both methods are compared with each other. This study may play a key role for future studies for comparison of stress values from XRD and Raman techniques

## 2. MATERIAL AND METHOD

Theree sample are formed with succesful growth of AlN, AlInN and GaN layers were grown on (00.1) oriented sapphire substrate by MOCVD method. Trimethylgallium (TMGa), Trimethylindium (TMIn), trimethylalliminium (TMAl) and ammonia ($NH_3$) sources were used together with Gallium (Ga), Alliminium (Al) and Nitrogen (N). Hydrogen ($H_2$) was used as the carrier gas. Before growing the epitaxial film, the sapphire substrate was annealed at 1100 $^oC$ for 10 minutes to remove surface impurities. AlInN HEMT structures are named samples A, B, and C, respectively. In Table 1 schematic diagrams of samples can be seen. There is sapphire substrate, AlN nucleation layer and AlN buffer layer in sample A. During the growth of this sample, first a 15 nm thick AlN nucleation layer was grown at a low temperature of 840 $^oC$. Then, the reactor temperature was increased to 1150 $^oC$ and a 500 nm thick buffer layer was grown. There is sapphire substrate and AlN, GaN buffer layers in sample B. For sample C, first, 15-20 nm thick Al nucleation layer was grown at a low temperature of 840 $^oC$. Then, the reactor temperature was increased to 1150 $^oC$ and a 600 nm thick AlN buffer layer was grown. As a final step, the growth process is stopped for 2 min to ensure the growth conditions for GaN. The GaN buffer layer is formed with a thickness of 2 μm at a growth rate of 2 μm/h at 1070 $^oC$.

***Table 1.*** *Growth conditions of samples A, B and C*

| Sample A | thickness | Sample B | thickness | Sample C | thickness |
|---|---|---|---|---|---|
| GaN cap layer | ≈2 nm. | GaN cap layer | ≈4 nm. | GaN cap layer | ≈4 nm. |
| AlInN inter layer | ≈8 nm. | AlInN inter layer | ≈14 nm. | AlInN inter layer | ≈14 nm. |
| AlN inter layer | ≈1 nm. | AlN inter layer | ≈1 nm. | AlN inter layer | ≈1 nm. |
| Ud-GaN layer (5) | ≈90 nm. | Ud-GaN layer (5) | ≈90 nm. | Ud-GaN layer (5) | ≈90 nm. |
| Ud-GaN layer (4) | ≈180 nm. | Ud-GaN layer (4) | ≈180 nm. | Ud-GaN layer (4) | ≈180 nm. |
| Ud-GaN layer (3) | ≈90 nm. | Ud-GaN layer (3) | ≈90 nm. | Ud-GaN layer (3) | ≈90 nm. |
| Ud-GaN layer (2) | ≈600 nm. | Ud-GaN layer (2) | ≈600 nm. | Ud-GaN layer (2) | ≈600 nm. |
| Ud-GaN layer (1) | ≈200 nm. | Ud-GaN layer (1) | ≈200 nm. | Ud-GaN layer (1) | ≈200 nm. |
| HT-AlN Buffer layer | ≈320 nm. | HT-AlN Buffer layer | ≈320 nm. | HT-AlN Buffer layer | ≈320 nm. |
| AlN nucleation layer | ≈15 nm. | AlN nucleation layer | ≈15 nm. | AlN nucleation layer | ≈15 nm. |
| Sapphire substrate | ≈330 µm. | Sapphire substrate | ≈330 µm. | Sapphire substrate | ≈330 µm. |

## 3. RESULTS AND DISCUSSION

In Figure 1, 2 theta versus intensity plot is given for symmetric planes. Diffraction peaks for GaN layer are found as 34.640, 73.02, 126.27 degrees for (002), (004) and (006) planes respectively. Diffraction peaks for AlN layer are 36.332, 76.505 and 136.559 for the same planes respectively. FWHM values for GaN layers are 0.59621, 0.906832 and 0.667506 for samples A, B and C respectively.

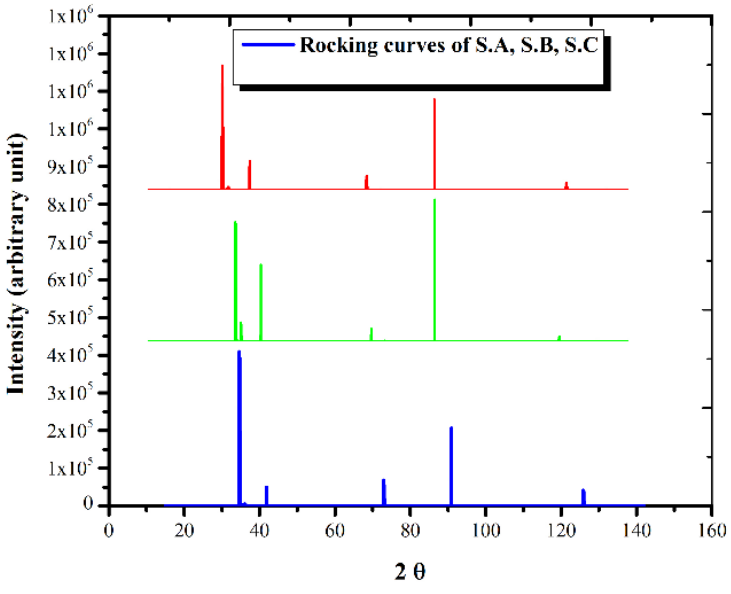


***Figure 1****. XRD pattern for samples A, B and C*

Small FWHM value implies high crystallity, low mosaic defects and a smooth structure. Stress calculation can be made by the help of strain. Relationship between strain and stress are given in Equation (1).

$$\varepsilon = \frac{\sigma(1-\nu)}{E} \tag{1}$$

In Equation (1) $\varepsilon$ is strain, $\sigma$ is stress, $\nu$ is Poisson ratio and E is Yong modulus. $\varepsilon$ (strain) can be determined by the slope of $\sin\theta$ versus $\beta\cos\theta$ plot. This plot can be seen in Figure 2. Strain and stress values for GaN layers of Samples A, B and C can be seen in Table 2.

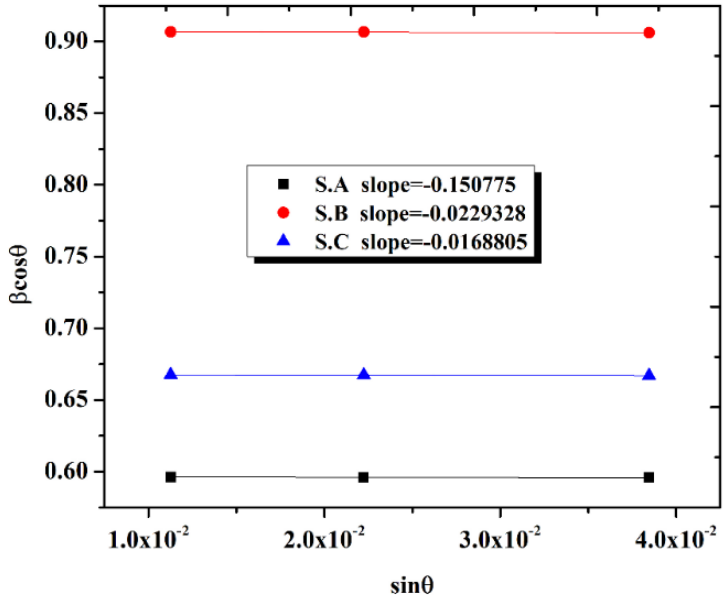


***Figure 2***. *Plot for determination of Strain*

When a material is exposed to photons, if there is no absorption, elastic or ineleastic scattering may occur. Here scattering may be defined as variation in direction of photon. In elastic scattering case, incident and scattered photons have the same wavelength. This situation is called as Rayleigh scattering. On the other hand, if incident and scattered photons have different wavelengths (inelastic scattering), this situation is called as Raman scattering. Here, the difference between vibration energy levels of photon and interacted molecule is equal to more or less energy quantity formed after scattering. By the help of this principle, measurement of difference between incident and scattered photons by spectroscopy and data on vibration energy levels of interacted molecules are discovered by C. V. Raman who gained Nobel prize. Measured difference is called as Raman shift. Basic working principle is as follows: sample is exposed to laser beam. Before photons reach to sample they pass through a transperant filter. When they reach to sample they interact with molecules of sample and make inelastic scattering. The scattered photons are selected according to their wavelengths and reach to the detector. In detector these photons are gathered and sent to registering device. After this step data reaches to computer. Computer calculates Raman shift automatically by the help of software saved. Raman shift versus intensity of incident light beam plot is formed at the same time with measurement by the computer. Here the expert making measurement can choose the region on the sample that will be exposed to laser beam.

In this study, Raman spectroscopy measurement of samples are made with WITec alpha 300R device. All data are gained at room temperature. GaN layer has hegzogonal crystal structure and belongs to $C_{6v}^4$ space group. According to group theory, GaN layer which has Hegzogonal crystal structure includes six active Raman mode. These are $1A_1(TO)+1A_1(LO)+1E_1(TO)+1E_1(LO)+2E_2$.

Raman shift and wavenumber versus intensity plots for samples grown on sapphire substrate are shown in Figure 3. These samples are grown with different Al compositions. Raman shift value for %87.4, %86.6 and %86.4 Al compositions(Bilgili et al., 2021) are found around 557 $cm^{-1}$. These peaks are related with vibration of $E_2$ (high) optic phonon mode of GaN thin films which have hegzagonal crystal structure(Harima et al., 2002). This value is smaller than values found in literature(Chen et al., 2001). Raman intensity is higher for samples A and B. This result shows us effect of Al compositions on optic phonon modes of GaN thin films. In summary, different Al compositions cause different Raman intensity but same Raman shift values. This result implies, by changing Al compositions one can control optic phonon modes.

Mechanical properties of samples grown has important effect on fragility. Examination of these properties become strongly important in device applications. Raman spectra gives important data on important mechanical properties of samples such as residual stress. For GaN thin film, stress free Raman shift peak value is determined as 567.2 $cm^{-1}$ belonging to $E_2$ (high) optic phonon mode in literature (Christopher et al., 2015). If Raman shift value, belonging to $E_2$(high) optic phonon mode of GaN thin film is bigger than this value, that is bigger wavenumber, implies blue region shift. In this situation, GaN thin film has compressive stress. If the material has less Raman shift value than mentioned value, this situation is called as red region shift. In this case there is tensile stress in material.

Residual stress of GaN thin films can be calculated with equation (2) which is found by Wang et al (Jian et al., 2016).

$$\boldsymbol{\alpha = \frac{\Delta W}{K}} \tag{2}$$

Here, σ is residual stress, Δw, deviation of Raman shift peak value from stress free peak value and k is Raman stress coefficient (4.3 $cm^{-1}(GPa)^{-1}$). Residual stress values for samples grown on sapphire substrate are given in table (1).

In different Al compositions Raman shift values for GaN thin films shift to red region for samples A, B and C. As a result of this study GaN thin films shows tensile stress property. Raman shift values are determined as 557.549, 557.676 and 557.752 $cm^{-1}$, for samples A, B and C, respectively. There is difference in Raman intensities also. This may be because of polarizations of GaN molecules in samples. As can be seen in table 2. When Al content decrease, residual stress value also decrease.

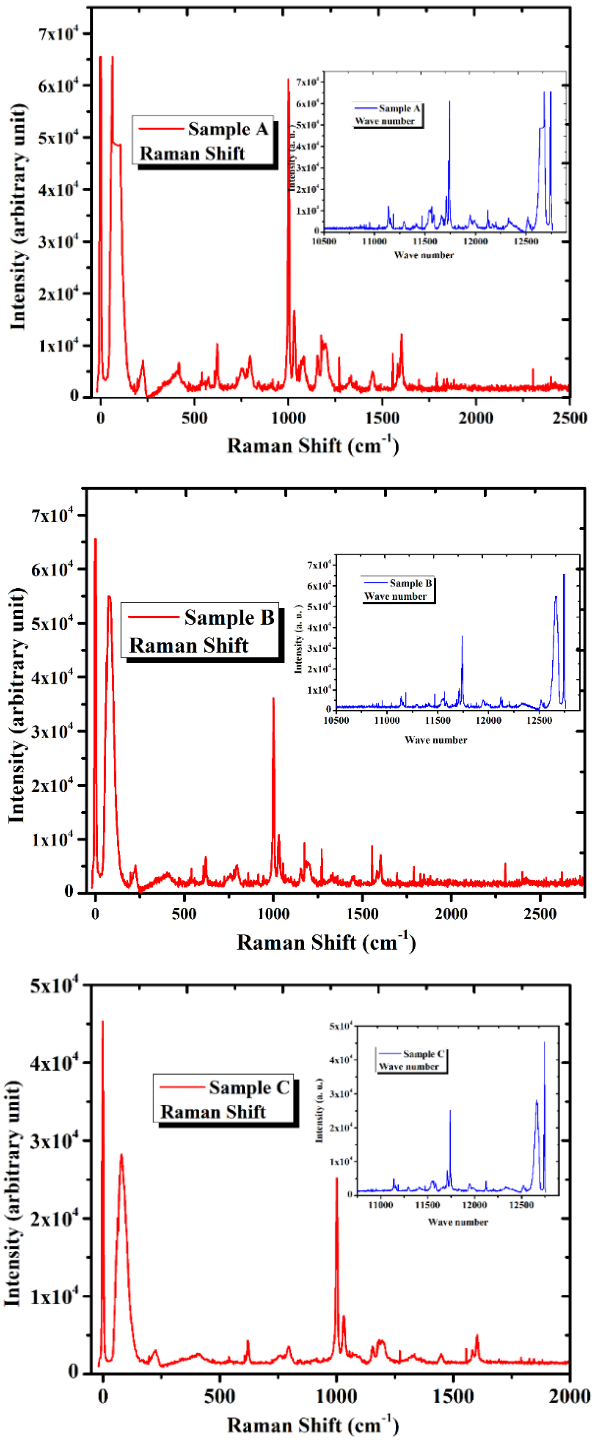


***Figure 3.*** *Raman shift and wavenumber plots for samples A, B and C*

***Table 2.*** *Raman and XRD analysis results for samples A, B and C*

| Al content | Strain(XRD) | Stress(XRD) | Stress(Raman) | Stress type |
|---|---|---|---|---|
| %87.4 | -0.015 | 2.6137 | 2.2442 | Tensile |
| %86.6 | -0.023 | 3.0854 | 2.2148 | Tensile |
| %86.4 | -0.017 | 2.0860 | 2.1972 | Tensile |

## 4. CONCLUSION

In this study, some structural features of GaN/AlInN/AlN/ sapphire HEMTs are examined. HR-XRD and Raman techniques are employed to determine strain and stress values of GaN layer. Strain values are obtained as -0.015, -0.023 and -0.017 for samples A, B and C respectively. One of difference between samples is Al content. Al contents are found for these samples in a previous study. Also stress type is determined from Raman shift. It is seen that stress calculated from Raman shift are directly proportional with Al content. However it can not be said that this situation is valid for stress values obtained from HR-XRD results. They are not proprtional with Al content. This may be because of FWHM values used in calculation of stress. Low and correct FWHM values imply good crystal quality. In this respect, crystal quality for Sample B is poor and stress value from XRD is high for this sample. All results are presented in Table 1. This study is rare because application of HR-XRD and Raman techniques at the same time for samples is rare. This study may play a key role for future studies for other researchers.

## AUTHOR CONTRIBUTIONS

Conceptualization, Ö.B.; methodology, software, A.K.B.; visualization, investigation, M.K.Ö.; supervision, N.K.; software, validation, writing, E.H.; reviewing and editing, Y.Ö. Authorship should be limited to those who contributed significantly to the article.

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## CONFLICT OF INTEREST

During the publication process, there was no conflict of interest between the authors arising from the findings, conclusions or opinions and statements stated in the publication, which could create direct or indirect financial interest or bias against academic competition.

## REFERENCES

Bayal, Ö., Balcı, E., Bılgılı, A. K., Öztürk, M., Özçelik, S., Özbay, E. (2023). Analysis of Dislocation Density for GaN Based HEMTs in Screw Mod. *Gazi University Journal of Science Part A: Engineering and Innovation*, *10*(2), 131-139. https://doi.org/10.54287/gujsa.1215224

Bilgili, A. K., Hekin, E., Öztürk, M. K., Özçelik, S., & Özbay, E. (2021). Mosaic defect and AFM study on GaN/AlInN/AlN/Sapphire HEMT structures. *Politeknik Dergisi*, *25*(4), 1613-1619. http://doi.org/10.2339/politeknik.787700

Chen, W., Inagava, Y., Omatsu, T., Tateda, M., Takeuchi, N., Usuki, Y. (2001). Diode-pumped, self-stimulating, passively Q-switched Nd3+: PbWO4 Raman laser. *Optics communications*, *194*(4-6), 401-407. http://doi.org/10.1016/S0030-4018(01)01148-8

Christopher, M., Kafle, K., Belias, D., Park, Y., Glick, R., Haigler, C., Kim, S. (2015). Comprehensive analysis of cellulose content, crystallinity, and lateral packing in Gossypium hirsutum and Gossypium barbadense cotton fibers using sum frequency generation, infrared and Raman spectroscopy, and X-ray diffraction. *Cellulose*, *22*, 971-989. http://doi.org/10.1007/s10570-014-0535-5

Harima, H. (2002). Properties of GaN and related compounds studied by means of Raman scattering. *Journal of Physics: Condensed Matter*, *14*, 38. http://doi.org/10.1088/0953-8984/14/38/201

Harish, D. V. N. (2021). Investigation of thermal residual stresses during laser ablation of tantalum carbide coated graphite substrates using micro-Raman spectroscopy and COMSOL multiphysics. *Ceramics International*, *47*(3), 3498-3513. https://doi.org/10.1080/23311916.2024.2398650

Huang, W., Zhu, X., Wua, D., He, C., Hu, X., Duan, C. (2009). Structural modification of rhodamine-based sensors toward highly selective mercury detection in mixed organic/aqueous media. *Dalton Transactions*, *47*, 10457-10465. https://doi.org/10.1039/B914490K

Jiang, Y. C., Ju, Gao., & Wang, L. (2016). Raman fingerprint for semi-metal WTe2 evolving from bulk to monolayer. *Scientific Reports*, *6*(1), 19624. http://doi.org/10.7907/vh7k-4w84

Li, Y., Xiang, J., Qian, F., Gradecak, S., Wu, Y., Yan, H., Blom, D. A., & Lieber, C. M. (2006). Dopant-free GaN/AlN/AlGaN radial nanowire heterostructures as high electron mobility transistors. *Nano Letters*, *6*(7), 1468-1473. https://doi.org/10.1021/nl060849z

Miyata, N., Watanabe, S., & Okamura, S. (1997). Infrared and Raman study of H-terminated Si(100) surfaces produced by etching solutions. *Applied Surface Science*, *117-118*, (26-31). https://doi.org/10.1016/S0169-4332(97)80046-3

Supekar, D., Brown, J., Alan, G., Juliet, G., Bright, V. (2018). Real-time detection of reverse-osmosis membrane scaling via Raman spectroscopy. *Industrial & Engineering Chemistry Research*, *57*(47), 16021-16026. http://doi.org/10.1021/acs.iecr.8b01272

Vurgaftman, I., Meyer, J. R. (2003). Band parameters for nitrogen-containing semiconductors. *Journal of Applied Physics*, *94*(6), 3675-3696. https://doi.org/10.1063/1.1600519

Wu, D. Y., Li, J. F., Ren, B., Tian, Z. O. (2008). Electrochemical surface-enhanced Raman spectroscopy of nanostructures. *Chemical Society Reviews*, *37*(5), 1025-1041. http://doi.org/10.1039/b707872

Zhang, Ye., Jia, H., Wang, R., Chen, C., Luo, X., Yu, D. (2003). Low-temperature growth and Raman scattering study of vertically aligned ZnO nanowires on Si substrate. *Applied Physics Letters*, *83*(22), 4631-4633. https://doi.org/10.1063/1.1760594