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Structural characterization of Nickel and Zinc Aluminate prepared by sol-gel technique

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Abstract. This work concerned on nanocrystalline $NiAl_2O_4$ and $ZnAl_2O_4$ having spinel structure prepared by Sol–gel technique. The structural and characterization properties for the obtained samples were examined using different measurements such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), finally, Field emission scanning electron microscope (FESEM).The Spinel-type for two prepared compound ($NiAl_2O_4$) and ($ZnAl_2O_4$) at different calcination temperature examined by XRD. Williamson-Hall Methods used to estimate crystallite size, Average distribution crystallite size of two compound were, 34.2 nm for $NiAl_2O_4$ and32.6 for $ZnAl_2O_4$, the increase in crystallite size affecting by increasing in calcination temperature for both compounds.

Keyword: spinel structure, nickel aluminate, zinc Aluminate

INTRODUCTION

The properties such as high strength, melting temperature and resistance, large fundamental band gap, and good electrical conductivity, in the other word. Thermal, mechanical and chemical stability of Nickel and Zinc aluminates gives different field of application at high temperature such as refractories, antithermal coating, catalysts, optical material, electronic ceramic, and in pigment products. The band gap of $ZnAl_2O_4$ is > 3.8 eV, $ZnAl_2O_4$ can be transparent to the UV part of spectra [1-3].
Sol-gel consider a simplest, safe and fast methods and consumes a little energy with a small particle size when preparing ceramic powders [4, 5].

Ultra-fine particles are preferable because of provide higher surface area. Moreover, highly stoichiometric position for starting material in preparing method. The aluminate play an important role for stabilized the cations in octahedral and tetrahedral sites [6].

Zinc and Nickel aluminate oxide having spinel structure, the general formula for spinal oxide is AB_2O_4, where (A) represents a divalent metal cation occupies a tetrahedral site and (B) is trivalent metal cations occupy the octahedral sites of a cubic lattice [7].
This paper concerned on uniform deformation Williamson-Hall analysis to estimate nanocrystalline and strain of Nickel and Zinc Aluminate preparation process by sol-gel technique and effect of calcination temperature for the final structural properties of these compounds.

MATERIALS AND METHODS

Stoichiometric composition of Zinc nitrate and aluminum nitrate used as a started materials to prepare $ZnAl_2O_4$ also nickel nitrate with aluminum nitrate used for prepare $NiAl_2O_4$ powder. Ethylene glycol and citric acid dissolved used to the ionic solution, the reaction between citric acid and ethylene glycol due to their carboxyl and hydroxyl group. Then, droppings ammonia to control on PH at 8 and using magnetic stirrer at 120˚C for 4 hrs, after that, it heated at 650° C using an electrical furnaces. The product was crashed in a gate mortar for 2 hrs, the obtained powder calcined at 1050 C for 2hr. The prepared powder were characterized using X-ray diffraction in a Shimadzu 6000 diffractometer 2θ gives at about
20-80°, using CuKα radiation (λ=1,5406Å) operate at 40 KV. Followed the crystallite size using Debby sheerer equation [8].

$$\beta_t = \frac{K\lambda}{D\cos\theta_{hkl}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

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the crystallite size (D), the shape factor (K), wavelength (λ) of X-ray, B_t is assumed that the line broadening (the peak width at half maximum height) of a Bragg reflection (hkl) planes with small crystallite size using formula (1), and (θ) is Bragg's angle and (t) is the effective crystallite size.
In Williamson-Hall method. The strain results due to distortion in prepared powder measured by follow Wilson equation [9, 10].

$$\varepsilon = \frac{\beta_{hkl}}{4\ tan\theta_{hkl}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

(ϵ) The microstrain. Assuming that the particle size and strain contributions to line broadening are independent of each other and both have a Cauchy like profile, the observed line breadth is simply the sum of the two which is called the Williamson-Hall equation [8].

$$\beta_{hkl} = \beta_t + \beta_\varepsilon = \left(\frac{K\lambda}{D\ cos\theta} + 4\varepsilon \tan\theta_{hkl}\right) \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

$$\beta_{hkl}\ cos\theta_{hkl} = \left(\frac{K\lambda}{D}\right) + (4\varepsilon\ sin\theta_{hkl}) \ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

The $\beta_{hkl} cos\theta_{hkl}$ values against $4sin\theta_{hkl}$ Plotted, where the slope of the line represented the microstrain ε, while, the crystallite size was calculated from intersection with the y-axis.
Grain size distribution measured used Field Emission scanning electron microscope (FESEM) model MIRA lll.

RESULTS AND DISCUSSION

Figures (1 and 2) shows, the X-ray diffraction patterns of prepared powder at different temperature (650˚ C and 1050˚ C). Compared with spinal standard Joint Committee on Powder Diffraction standards (JCPDS) cards no. (96-900-7020) and (96-900-1435) for ($NiAl_2O_4$) and ($ZnAl_2O_4$) respectively. X-ray diagram examined for both compounds at different calcination temperature, it's clear that the face centered cubic Spinel-type for two compound ($NiAl_2O_4$) and ($ZnAl_2O_4$) can be formed using sol gel technique with space group Fd3m. In Fig 1. shows the ($NiAl_2O_4$) major diffraction peaks were seen at 31.61˚, 37.27˚, 43.30˚, 45.08˚, 59.37˚, 65.68˚, which can be assigned to diffraction from (202), (311), (222), (400), (333), (422), and (404), planes respectively. The highest peak located at about37.276˚, 37.075˚ for $ZnAl_2O_4$ compounds calcined 650˚ C and 1050˚ C respectively.
Figure 2 shows pure spinal ($ZnAl_2O_4$) with no impurity phase appearances. The highest peak located at about36.872˚, 36.833 ˚ for $ZnAl_2O_4$ compounds calcined 650˚ C and 1050˚ C respectively.

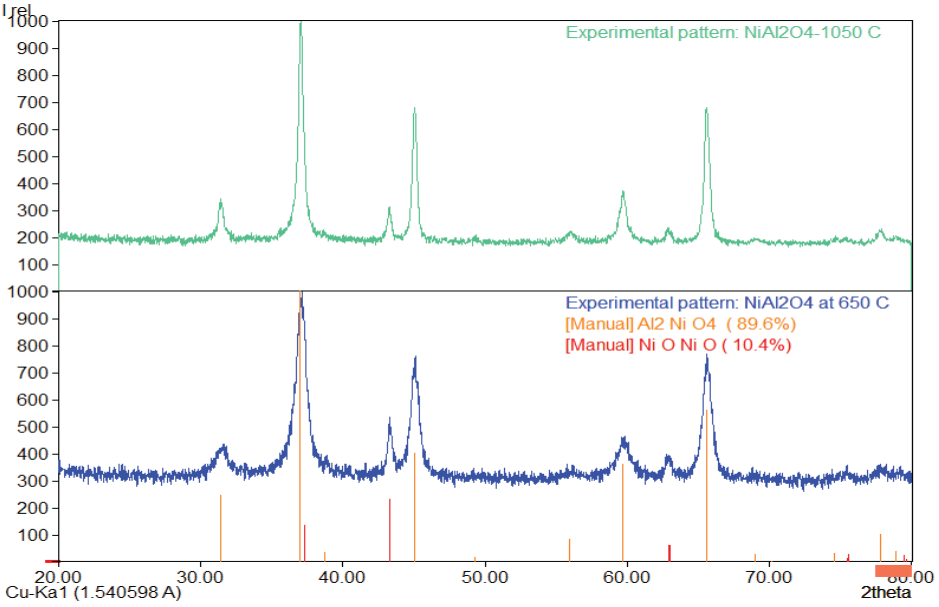

FIGURE 1. XRD for $NiAl_2O_4$ prepared powder with different calcination temperature (650˚ C and 1050˚ C).

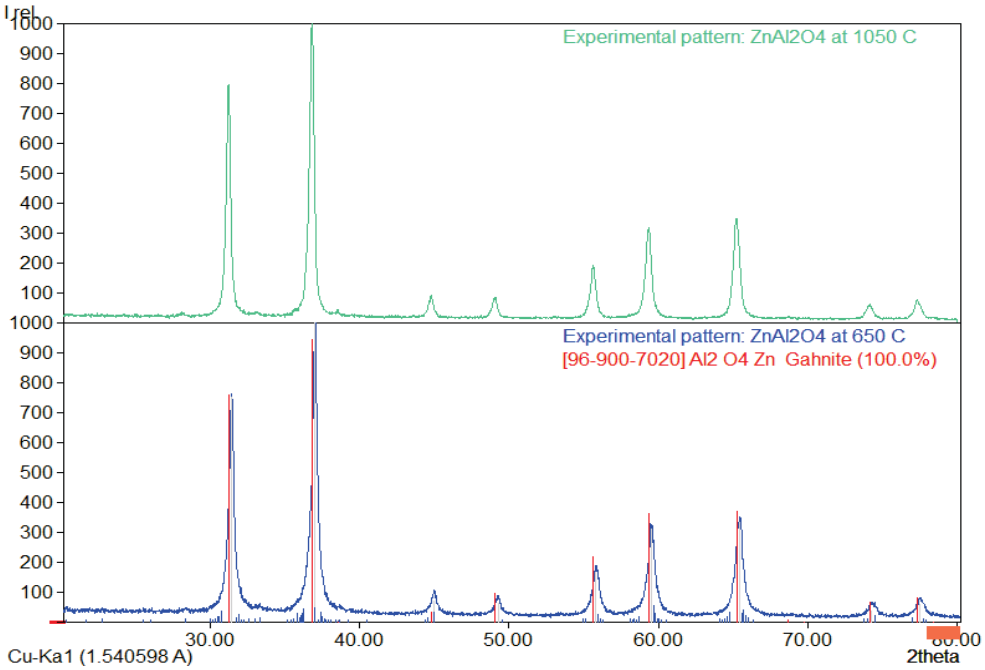

FIGURE 2.XRD for $ZnAl_2O_4$ prepared powder with different calcination temperature (650˚ C and 1050˚ C).

In this study, Uniform deformation Williamson-Hall analysis of nanocrystalline was done assuming for the two prepared compounds. The strain supposed to be uniform in all the directions. The slope of line gives the microstrain ε and the crystallite size calculated from intersection with the vertical axis. β_{hkl} Value used here is the instrumental corrected values using Williamson-Hall analysis to estimate the lattice strain in nanometer range. The average crystallite sizes measured from the graphs shown in figures 3(a, b) for $NiAl_2O_4$ and (c, d) for $ZnAl_2O_4$ at 650 C˚ and1050C˚ respectively. 34.2nm and 32.6nm was crystallite size for $NiAl_2O_4$ and $ZnAl_2O_4$ respectively. The values of the crystallite size affecting by calcination temperature. It's clear from Figures 3(b, d) that, the increases in crystallite size with calcination temperature increasing for both compounds.

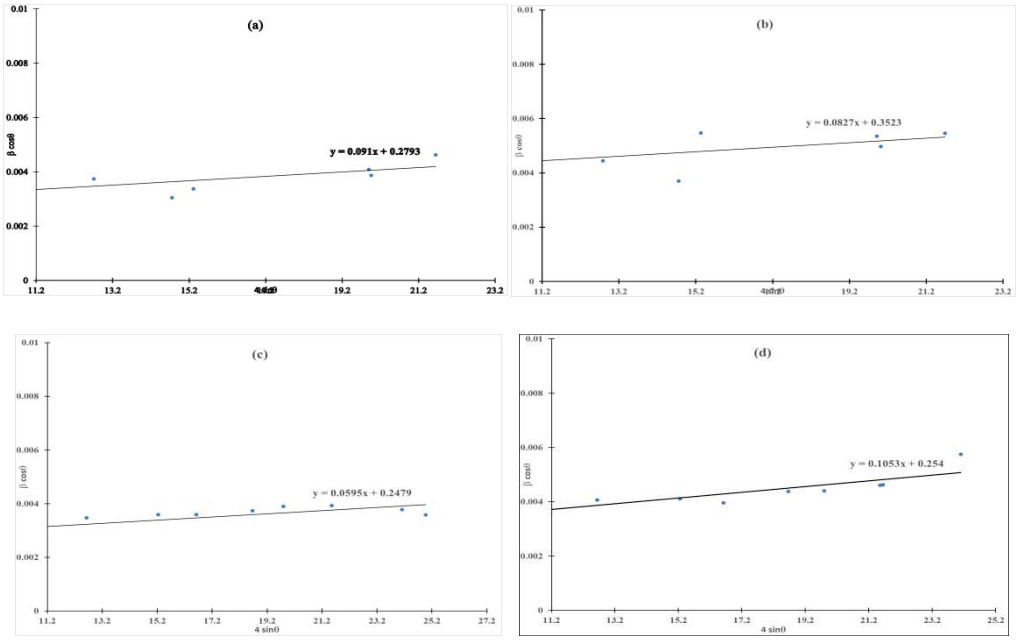

FIGURE 3. Uniform Williamson-Hall plot of nanocrystalline for Aluminate compound at different temperatures; (a) $NiAl_2O_4$ at 650 °C, (b) $NiAl_2O_4$ at 1050 °C, (c) $ZnAl_2O_4$ at 650 °C, (d) $ZnAl_2O_4$ at 1050 °C

FTIR spectra of the spinel aluminate compounds ($NiAl_2O_4$) and ($ZnAl_2O_4$) were measured in the wavenumber region of 400-4000 cm^{-1} and calcined temperature at 650°C and 1050° C respectively. In figure (4(c, d)) for $ZnAl_2O_4$ show that the regions of 3600, the wide absorption bands centered at 3435 indicate to Alcohol stretching OH group which is contributed of slight water content in the crystallites. Also the bands at 1622 cm^{-1} are still present with a little splitting and this is due to the formation of metal hydroxides. Three sharp absorption bands visible in the range between 400cm^{-1} to 760 cm^{-1} arise from the stretching vibrations of octahedral and tetrahedral bonds in the spinel structure. The peak at 2349 cm^{-1} can be attributed to the oxygen–oxygen bonds in the fcc crystal lattice as shown in figure (4). No other impurity phase was detected by FTIR spectra.

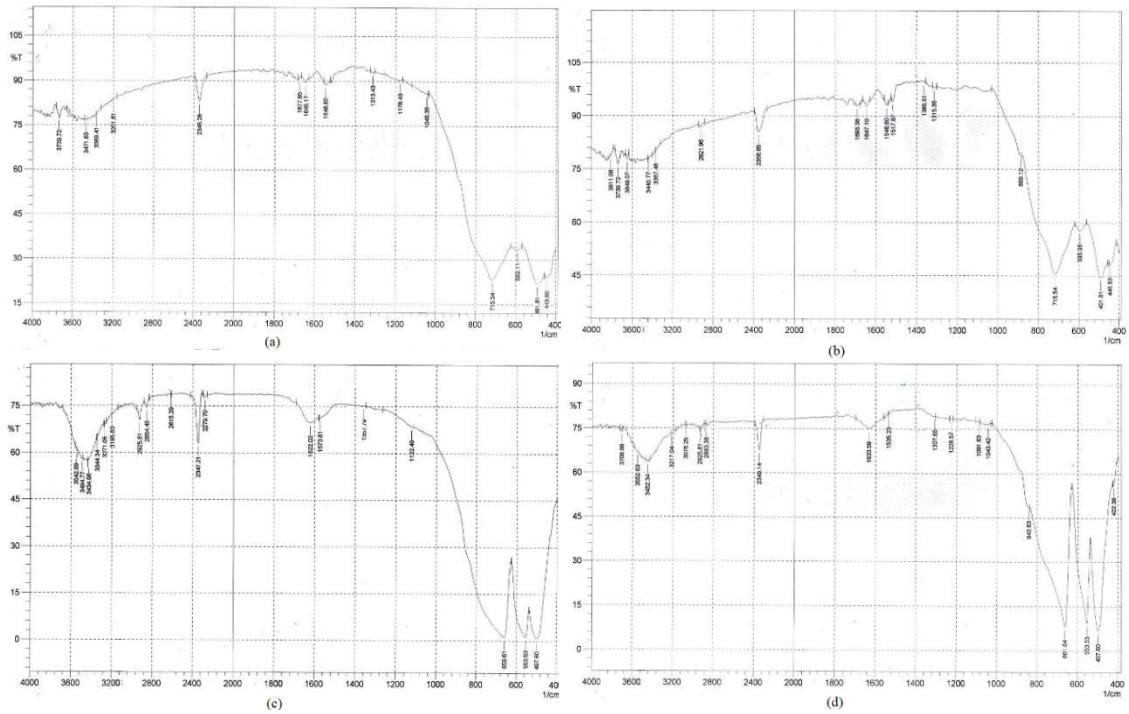

FIGURE 4. Infrared spectrum of Nickel and Zinc Aluminate at different temperature
(a) $NiAl_2O_4$ at 650 °C, (b) $NiAl_2O_4$ at 1050 °C, (c) $ZnAl_2O_4$ at 650 °C, (d) $ZnAl_2O_4$ at 1050 °C

The FESEM for ($NiAl_2O_4$) and ($ZnAl_2O_4$) prepared compounds at 650 ˚C shown figure 5(a, c), it can be seen from this figure that the prepared powder consisted regular shaped nanometric grains had different size distributions. Agglomerates and growth exhibit an irregular morphology for both aluminate compounds ($NiAl_2O_4$) and ($ZnAl_2O_4$), its affected by increasing in calcination temperature, as shown in figure 5(b,d) .

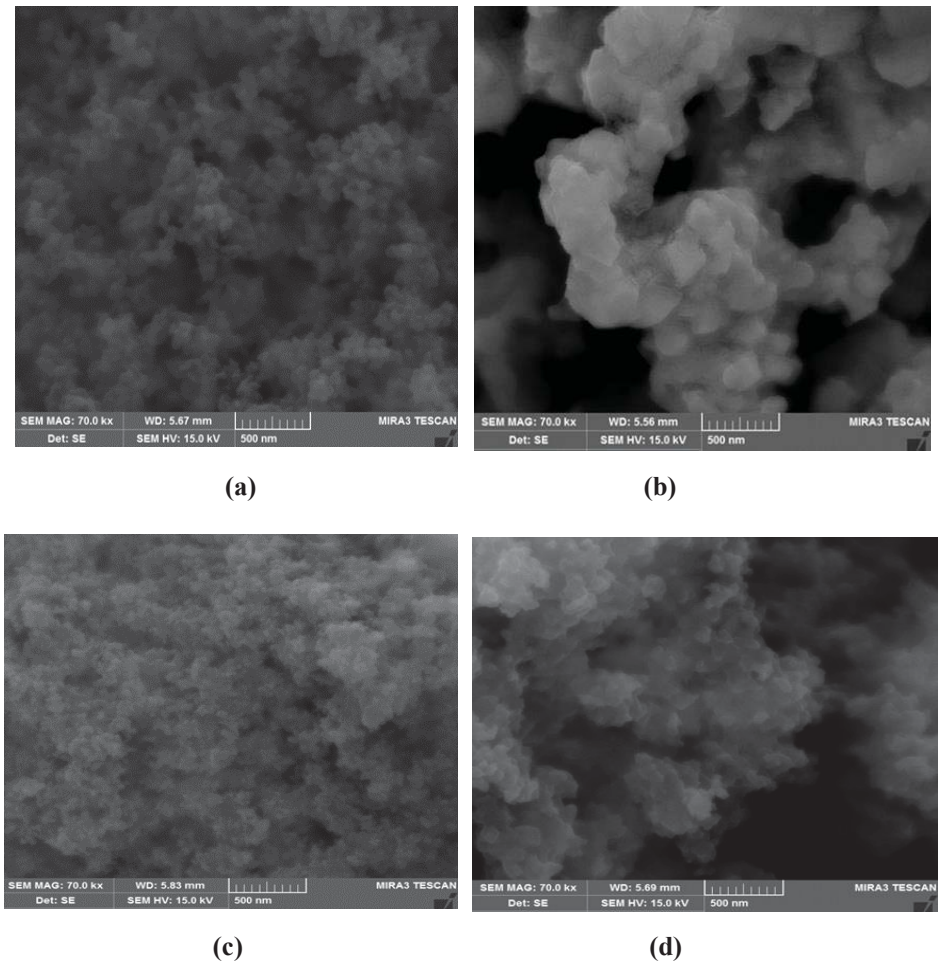

FIGURE 5. FESEM micrograph for the prepared compound by sol gel technique for $NiAl_2O_4$, $ZnAl_2O_4$ at different temperature (a, c) at 650 °C, (b, d) at 1050 °C.

CONCLUSION

Sol gel technique successfully high homogenous poly crystalline with small crystallite size for spinel aluminate compound, in other word, XRD showed the presence of pure spinel phase in all samples ($NiAl_2O_4$) and ($ZnAl_2O_4$). Uniform Williamson-Hall analyses to estimate the crystallite size and strain. Effect of calcination temperature for the two prepared compound increase with increasing temperature. The FSEM results corresponds well with the Williamson-Hall result.

REFERENCES

1. S. K. Sampath and J. F. Cordaro, ‘‘Optical Properties of Zinc Aluminate, Zinc gallate, and Zinc Alumino gallate Spinels,’’ J. Am. Ceram. Soc., 81 [3] 649–54 (1998). https://ceramics.onlinelibrary.wiley.com/doi/abs/10.1111/j.1151-2916.1998.tb02385.x.

2. H. Kawazoe and K. Ueda, ‘‘Transparent Conducting Oxides Based on the Spinel Structure,’’ J. Am. Ceram. Soc., 82 [12] 3330–6 (1999). https://ceramics.onlinelibrary.wiley.com/doi/abs/10.1111/j.1151-2916.1999.tb02247.x.
3. K. Christine Stella and A. Samson Nesaraj, ‘‘Effect of Fuels on the Combustion Synthesis of $NiAL_2O_4$ Spinel Particles’ Iranian Journal of Materials Science & Engineering,7[2]043709, 36-44pp (2010). https://scholar.google.com/scholar?sxsrf=ACYBGNR3ufWs8enaGlepESj9dyfNLAd-tg:1579899566038&um=1&ie=UTF8&lr&q=related:mnj5qtpDpIEcEM:scholar.google.com/.

4. John D. Wright, Nico A.J.M. Sommerdijk, “Sol-Gel Materials: Chemistry and Applications”, CRC press web sites at www.crcpress.com (2001).
5. I.M. Abdulmajeed, F. A. Chyad, M. M. Abbas, H. A. Kareem, “Fabrication and Characterization of Ultrafine Crystalline MgO and ZnO Powders”, IJIRSET, 2[10], 5101-5106(2013). http://www.rroij.com/open-access/fabrication-and-characterization-of-ultrafinecrystalline-mgo-and-zno-powders-.php?aid=47427.
6. Cooley, R.F., and Reed, J.S. “Equilibrium cation distributions in $NiAl_2O_4$, $CuAl_2O_4$ and $ZnAl_2O_4$”, Journal of American Ceramic Society, 55, 395-398, (1972). https://ceramics.onlinelibrary.wiley.com/doi/abs/10.1111/j.1151-2916.1972.tb11320.x.
7. Z. Z. Chen, E. W. Shi, Y. Q. Zheng, B. Xiao, J. Y. Zhuang. "Hydrothermal synthesis of nanosized $CoAl_2O_4$ on $ZnAl_2O_4$ seed crystallites." Journal of the American Ceramic Society”, 86 1058, (2003). https://www.tib.eu/en/search/id/tema%3ATEMA20030802782/Hydrothermal synthesis-of-nanosized-CoAl2O4-and/.

8. M. H. K. AL-Mamoori, D. K. Mahdi, S. M. Al-Shrefi “Synthesis and spectroscopic study of CdS nanoparticles using hydrothermal method”, Technologies and Materials for Renewable Energy, Environment and Sustainability AIP Conf. Proc 1968, 030011-1–030011-8 (2018). https://ui.adsabs.harvard.edu/abs/2018AIPC.1968c0011A/abstract.
9. V. Biju, Neena Sugathan, V. Vrinda, S. L. Salini, Estimation of lattice strain in nanocrystalline silver from X-ray diffraction line broadening”, J Mater Sci, 43:1175–1179(2008). https://link.springer.com/article/10.1007%2Fs10853-007-2300-8.
10. VD Mote, Y Purushotham, BN Dole, “Williamson-Hall analysis in estimation of lattice strain in nanometer-sized ZnO particles”, J. of Theoretical and Applied Physics, 6[6], pp. 2-8(2012). https://cyberleninka.org/article/n/471552.