

# Determination of Dislocation Density and Correlation Length of Si, Ti, Au and Zno on Ge by Peak Profile

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## Research Article

**Keywords:** dislocation, Kragner method, correlation, peak profile, Ge

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# ***DETERMINATION OF DISLOCATION DENSITY AND CORRELATION LENGTH OF Si, Ti, Au and ZnO on Ge by PEAK PROFILE***

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## ***Abstract***

In this study, X-ray diffraction peaks of Si, Ti, Au and ZnO grown on Ge substrate with thickness of 500 nm by using sputtering method are analyzed to determine correlation length and dislocation density. It is seen that in most dense region of peaks, peak behaviour is in accordance with Gauss function. Right and left tails of peaks are in good accordance with q<sup>3</sup> law. For randomized dislocations, obeying q<sup>3</sup> law is typical and they can be monitored with w-scans by using open detectors. Whole profile is fitted with a limited dislocation dispersion. Edge dislocation density and correlation length are determined in the degree of 10<sup>10</sup>cm<sup>-2</sup> and 10<sup>3</sup> nm, respectively. In order to gain these values, semi-experimental equations in Kragner method are used. For making a good fit, fit iteration step is taken as 9x10<sup>6</sup>.

***Key words:*** dislocation, Kragner method, correlation, peak profile, Ge.

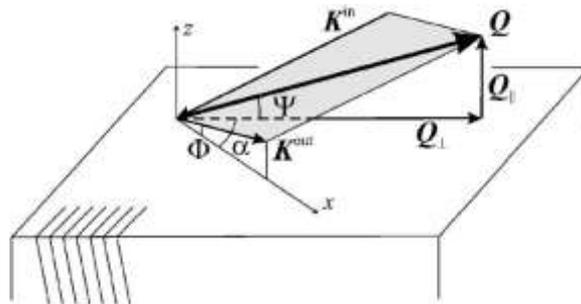
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## ***1. Introduction***

Structural defects effect optical and electric performance of opto-electronic devices in a bad way. In this situation optimisation of device is a good solution method[1]. The most important structural defect is dislocation. Increasing of dislocation density causes surface cracks in the structure. On the other hand, small grained and the same type poly-crystals may effect the structure in optical and electrical terms[2]. Ge is a good behaviour semiconductor, with 5.78 Å lattice constant and 0.67 eV band gap. It is common to use it as a substrate[3]. During growth of buffer layers on Ge substrate defect levels may change dependent on lattice mismatch. In limited situations as normal lattice planes, parallel to surface, broadening of X-ray reflections are effected with edge and screw type dislocations. This is related with layer tilt and twist commonly. This situation is caused by randomized dislocations formed by mis-oriented blocks and description of root mean square (RMS) values for defects is not sufficient[4]. Crystallite layer, for example Ge, Ti, Au and ZnO, Bragg reflections of symmetric peaks are too narrow.

Because, these structures are not influenced by edge dislocations. Also, edge dislocations produce defects in lattice planes parallel to surface, but they do not effect peak positions along layer normal. The highest value of edge dislocations is gained by diffraction patterns formed by lattice planes normal to surface. In this diffraction geometry X-ray reaches sample by sweeping the surface[4].

In order to determine the problem absolutely, it is good to give fundamentals of subject here. References [4, 5] are used to determine dislocation crystallite size by Kragner's method. Alternative geometry that can easily be performed in labrotary is asymmetric (screw) geometry. This geometry (incident and refracted waves have the same angle towards surface) is shown in Figure 1.



**Figure 1.** Curve geometry X-Ray pattern. Lattice planes of real reflection are described in left down side of figure. Incident wave  $K^{in}$  and diffracted wave  $K^{out}$  makes the same  $\Phi$  angle with sample surface. Scattering vector  $Q$  makes  $\Psi$  angle with sample surface [5].

Lattice planes normal to surface can be determined by measuring diffraction reflections with increasing tilt of lattice plane. Scanning curve can be gained by focusing wide tilts to odd reflections. Circular diffractometer is needed for symmetric geometry, because sample is tilted according to normal of surface. Asymmetric planes on circular diffractometer are less sensitive to edge dislocations and they have connection with lattice parts normal to surface[5]. Full width at half maximum (FWHM) of diffraction peak is not dependent on dislocation density but dependent on correlation among dislocations. FWHM is also dependent on natural orientations of scattering vector, dislocation path orientation and direction of Burgers vector[5, 6].

In this study, shape of peaks and especially tail regions of peaks are analyzed in whole diffraction profile. Dislocation densities are determined by using Kragner's q-3 law. In this method, Fourier transform is applied to correlation function numerically and dislocation densities, dislocation correlation lengths are determined[5].

## ***2. Experimental***

Sputtering is a common system to grow thin films on substrates in which atoms are detached from target material in vacuum medium. In this system, reactive ions such as Ar<sup>+</sup> are accelerated in high potential difference and target material is bombed with these ions. Molecules detached from target material are deposited on substrate. The best advantage in this system is that it permits deposition operation at low temperatures. With this advantage, any desired material can be deposited on the substrate.

Sputtering technique used for growth of Au, Si, Ti and ZnO structures on Ge substrate includes removal of atoms from target by applying high voltage to metal in high vacuum medium. Conductive material is used as anode and metal is used as cathode. Sample is coated in high vacuum. After forming an atmosphere of an inert gas such as Ar<sup>+</sup> in the tube with anode and cathode, there forms a plasma atmosphere at high voltage. At desired voltage value an electrical arc comes out. Sputtering systems are applied as radio frequency (RF), magnetic field, triode, direct current (DC) and ion beam sputtering.

In this study, Si, Au, Ti and ZnO are grown on high crystallized Ge with D.C sputtering method. In D.C sputtering method, coating material is inserted in cathode and substrate is inserted in anode. As Ar<sup>+</sup> is put in the coating medium and anode-cathode system is exposed to D.C voltage there forms the plasma atmosphere. As Ar<sup>+</sup> ions are accelerated and hit the target material, atoms are detached from target material. At the end of this operation surface of substrate is coated. In order to gain highest X-ray diffraction peak, Ge is coated with Au, Si, Ti and ZnO with thickness of 500 nm under 10<sup>-3</sup> mbar pressure.

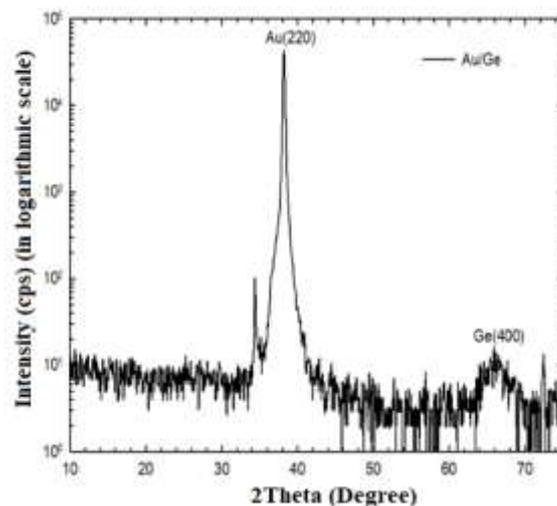
## ***3. Results&Discussion***

In diffraction peak profile analysis, limited moments of intensity distribution is calculated by using q<sup>-3</sup> asymptote. But, in randomized and limited dislocations viewing length can not be gained from asymptotic part of peak. Fit of whole peak profile maintains both dislocation distribution density and viewing length parameters. Numerical calculation of peak profile includes 1-D Fourier integral. By using this integral calculation can be made rapidly. Using q<sup>-3</sup> asymptote is a more reliable way to determine dislocation density because q<sup>-3</sup> asymptote is not effected among dislocations and it is in accordance with scattering in the region near to dislocation lines. Asymptotic part of intensity distribution does not include X-ray diffraction studies. Determination of dislocation density is related with FWHMs. If peak is effected by both bulk and defects, sum of Gaussian and Lorentz functions (Pseudo-Voigt) are fitted.

FWHM of Gaussian is used for determining dislocation density. Width of symmetric and asymmetric reflections are effected from dislocation densities and they change shape of the peak. It is likely that dislocation distribution is similar in similar film coating and growth. For films those have partly dislocations, width of given peak reflection is not only dependent on dislocation density, it is also dependent on limited randomized dislocation density and correlation length.

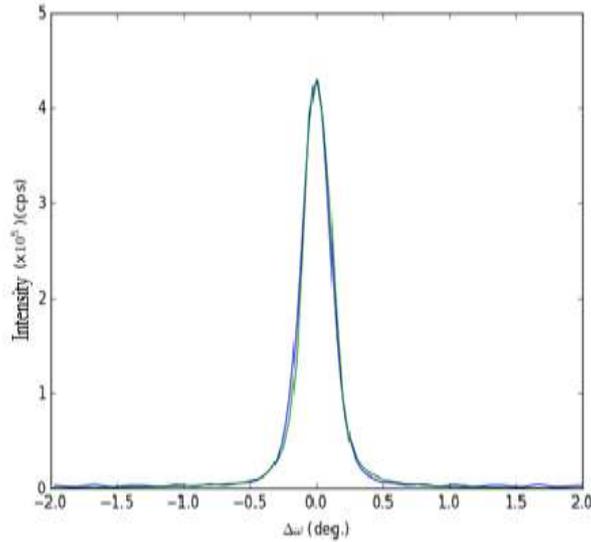
In this study, peak shape analysis is width of correlation length at the same time. Simple thought of peak width gives hand to determination of dislocation density. Peak shape analysis shows that X-ray diffraction profile is Gaussian at the center of peak. Peak tails obeys q-3 law in X-ray diffraction with randomized dislocations. When omega curves with analyser crystal obeys q-4 behaviour, omega curves are measured with an open detector detecting q-3 behaviour[5]. It is very convenient to study omega curves with an open detector both experimentally and theoretically because diffraction density is more and wider. Diffraction density is described by converting 1-D Fourier transform of peak profiles to correlation function. q-3 peak tails maintains determination of dislocation densities more accurately and they are denser with correlations among dislocations.

All diffraction profiles are fitted to main equation with suitable parameters. These parameters are dislocation density ( $\rho$ ), viewing length and crystallite size (L). Next term is in accordance with Burgers vector equal to zero and average crystallite volume[5]. In order to compare dislocation density value, inverse of Scherrer equation is used and this can be seen in literature frequently[40].



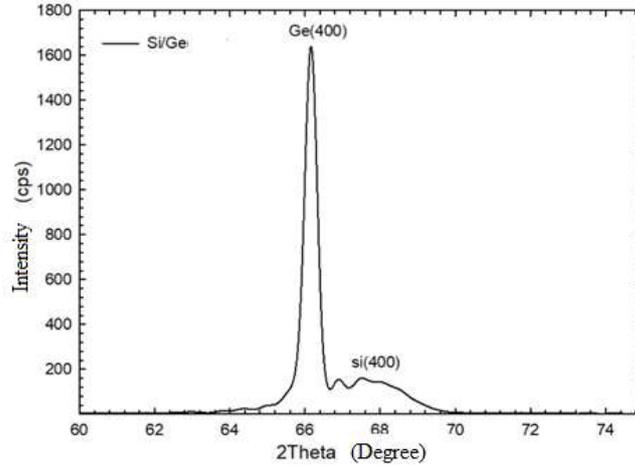
**Figure 2.** 2 theta scan of Au/Ge(substrate)

In Figure 2 2 theta versus intensity plot can be seen . Germanium peak is at 66.017 degree. (400) peak is corresponding to PDF-4-545 XRD database. Lattice constant is  $a = 5.657 \text{ \AA}$ . Structure is face centered cubic. Gold peak center is at 66.017 degree and it corresponds to (400) peak PDF-4-784 XRD database. Lattice constant is  $a=4.079 \text{ \AA}$ . This structure is also face centered cubic.



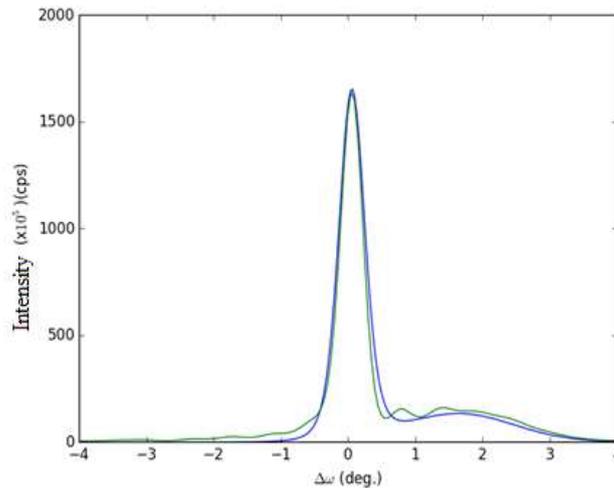
**Figure 3.** Profile fit curve of Au layer grown on Ge in (220) XRD reflection plane.

Figure 3 shows w scan of Au. XRD scanning curve (green color) can be fitted by intensity equation. As the result of fit in figure,  $\rho_e b_e^2 = A/f = 6.52 \times 10^{-5}$  is gained for Au grown on Ge. Here  $b_e = 0.40786 \text{ nm}$  is the length of Burgers vector for edge type dislocation. For Au thin film, dislocation density is  $\rho_e = 3.919 \times 10^{10} \text{ cm}^{-2}$ . By the help of simple Scherrer equation, determined value is very near to  $5.78 \times 10^{10}$  value for (400) Miller plane. Average length between edge type dislocations is  $r_d = 1/\sqrt{\rho_e} = 50.5 \text{ nm}$ . From figure 3  $L/b_e = B/\rho = 842$  is gained. This result implies characteristic dislocation correlation length as  $L = 343$ . Dimensionless parameter characterising dislocation correlations is found as  $M = L/r_d = 6.5$  for this sample.



**Figure 4.** 2 theta XRD scan of Si/Ge (substrate).

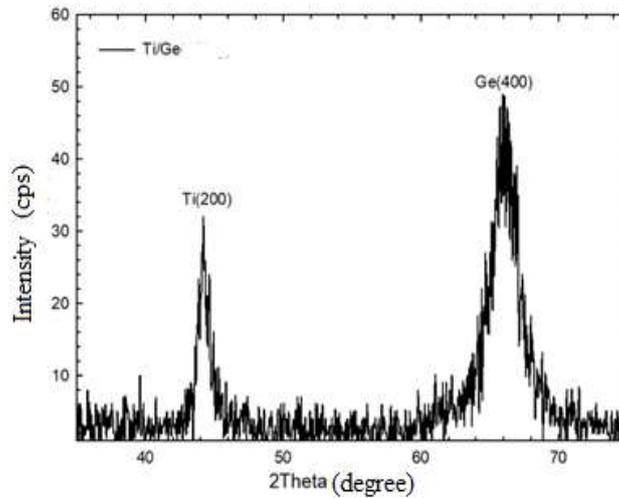
Figure 4 shows 2 theta versus intensity curve. Peak center for Germanium is determined as 66.017 degree. This value corresponds to (400) peak in PDF-4-545 XRD database. Lattice parameter is determined as  $a=5.657 \text{ \AA}$ . Structure has face centered cubic property. Si peak center value is 67.9 degree and this value corresponds to (400) peak in PDF-75-589 XRD database. Lattice parameter is determined as  $a=5.043 \text{ \AA}$ . Unit cell of the structure has face centered cubic property. As can be seen in Figure 4 crystal structure of Si has tilt towards (004) and lattice strain because of low FWHM. In this situation peak shift is a little angle together with offsets.



**Figure 5.** Profile fit curve in (400) plane for Si grown on Ge.

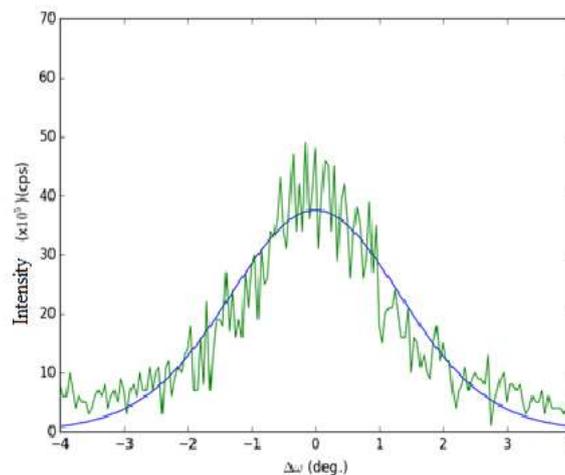
Figure 5 shows omega scan versus intensity of Si grown on Ge. XRD scanning curve (green colour) is fitted with intensity equation. By using Figure 5, Si on Ge with  $\rho_e b_e^2 = A/f = 9.4 \times 10^{-4}$  value is gained. Here  $b_e = 0,5658 \text{ nm}$  is the length of Burgers vector for edge type dislocation.

Dislocation density for Si thin film is found as  $\rho_e=4.03 \times 10^{10}$  cm. This value is very near to  $6.55 \times 10^{10}$  value gained from simple Scherrer equation for (400). Average length between edge type dislocations is found as  $r_d=1/\sqrt{\rho_e} = 49.75$  nm. By using figure 5  $L/b_e=B/\rho=1923$  value is gained. This result implements characteristic dislocation correlation lengths as  $L=1031$ . For this sample dimensionless parameter characterising dislocation correlations is gained as  $M=L/r_d=1,85$ .



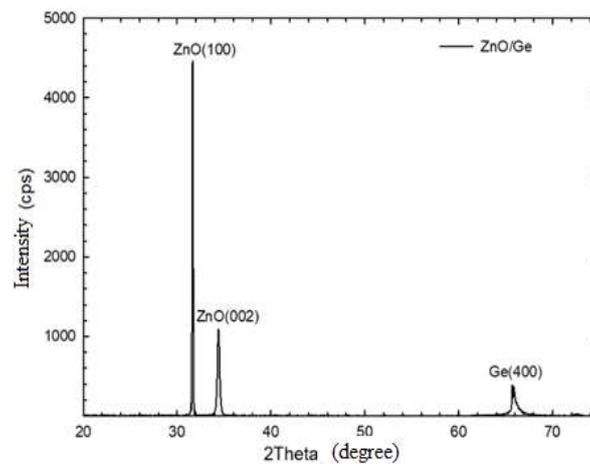
**Figure 6.** 2theta scan for Ti/Ge (substrate)

Figure 6 shows 2xtheta versus intensity for Ti/Ge structure. Peak center value for Ti is determined as 44.025 degree and it corresponds to PDF-88-2321 XRD database value. Lattice parameter is determined as  $a=4.0600$  Å. Unit cell of this structure has face centered cubic property. (200) plane peak has small intensity for Ti, this situation shows that Ti layer is grown as mono type poly crystal.



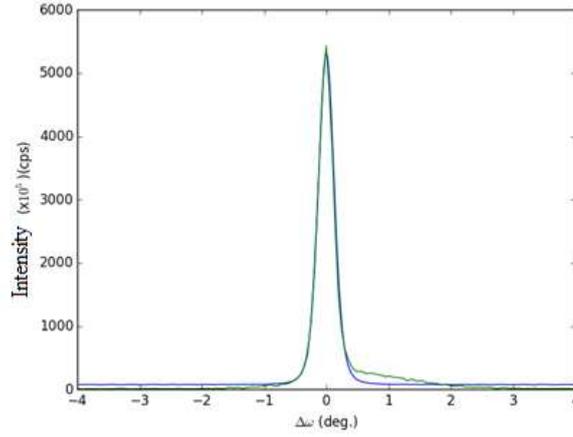
**Figure 7.** Profile fit curve for Ti grown on Ge for (200) plane.

Figure 7. shows omega versus intensity plot for Ti grown on Ge. XRD scanning curve (green color) is fitted with intensity equation. As the result of fit we gain  $\rho_e b_e^2 = A/f = 2.5 \times 10^{-3}$  for Ti on Ge. Here  $b_e = 0.32$  nm is the length of Burgers vector for edge dislocations. Dislocation density for Ti is found as  $\rho_e = 6.1 \times 10^{10}$  cm. This value is very near to dislocation value,  $1.57 \times 10^{10}$ , gained from simple Scherrer equation. This calculation is mad efor (200) Miller plane. Average distance between edge dislocations is found as  $r_d = 1/\sqrt{\rho_e} = 9.299$  nm. By using Figure 7  $L/b_e = B/\rho = 1898$  value is gained. If L is calculated from this equation, it is found as 889 nm. L is the characteristic length of dislocation correlations. Dimensionless parameter characterising dislocation correlations is found as  $M = L/r_d = 1.85$ .



**Figure 8.** 2xtheta versus intensity plot for ZnO on Ge

Peak center of ZnO is 44.025 degree and this value corresponds to PDF-2321 XRD database value. Lattice constant is found as  $a = 4.0600$  Å. Unit cell of structure has face centered cubic property. ZnO peak centers are determined as 31.736 degree for (100) and 34.378 degree for (002) planes. They correspond to PDF-89-1397 XRD database value. Lattice coefficients are determined as  $a = 3.253$  Å and  $c = 5.213$  Å. Unit cell of ZnO structure has hexagonal property. Because ZnO structure has good crystal quality it has sharp peaks.

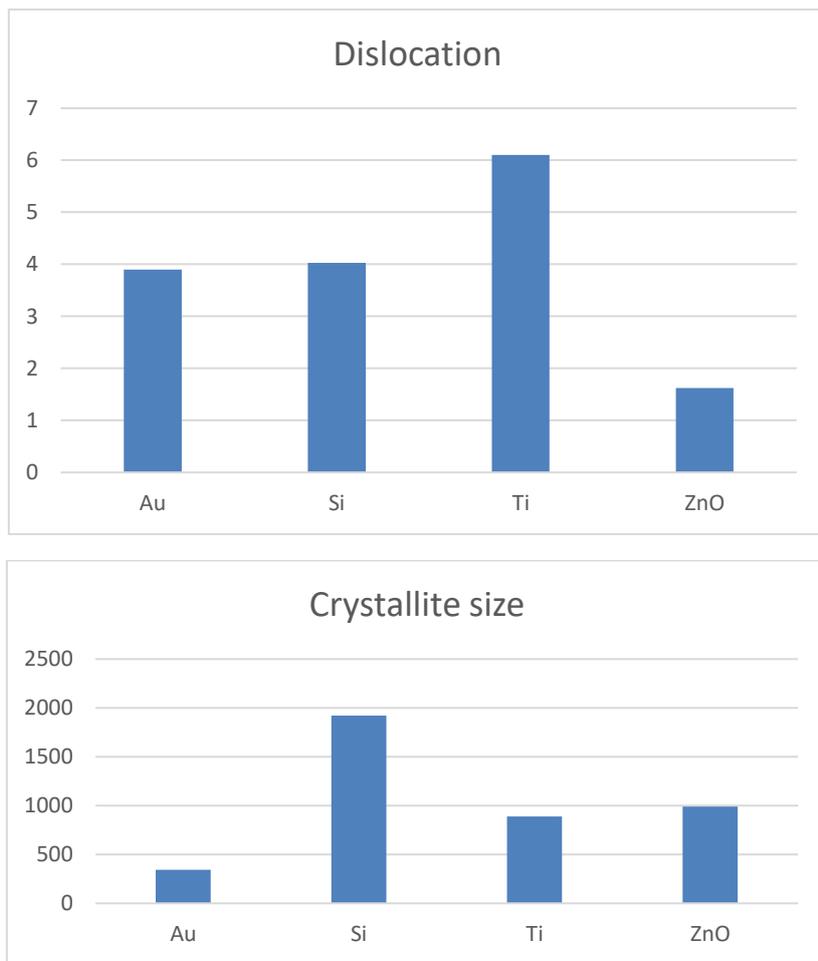


**Figure 9.** Profile fit curve for ZnO grown on Ge for (002) plane.

Figure 9 shows omega versus intensity scanning curve for ZnO grown on Ge. XRD scanning curve (green color) is fitted with intensity equation. As the result of fit in Figure 9  $\rho_e b_e^2 = A/f = 4,42 \times 10^{-5}$  equation is gained for ZnO on Ge. Here  $b_e = 0.5241$  nm value is length of Burgers vector for edge type dislocation. Dislocation density for ZnO is determined as  $\rho_e = 1,62 \times 10^{10}$  cm. This value is very near to dislocation value,  $4,47 \times 10^{10}$ , gained from simple Scherrer equation. Average distance between edge type dislocations is found as  $r_d = 1/\sqrt{\rho_e} = 78$  nm. By using figure 9,  $L/b_e = B/\rho = 1898$  equation is gained. From this equation characteristic dislocation length value can be found as  $L = 989$ .

### **Conclusion**

In this study, profile fit of Au, Si, Ti and ZnO thin films grown on Ge substrate by sputtering method is made after XRD scans. As the result of fit, dislocation density and correlation lengths are determined. Comparison of these results can be made with Figure 10. According to these results dislocation densities are found almost at the same level ( $\sim 10^{10}$ ). Here the biggest dislocation density value belongs to Ti and biggest crystal length belongs to Si.



**Figure 10.** Comparison of dislocation density and correlation lengths for Au, Si, Ti and ZnO layers grown on Ge.

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## ***References***

- [1] Durukan, Ilknur Kars; Ozturk, Mustafa Kemal; Corekci, Suleyman; et al. *Journal Of Nanoelectronics And Optoelectronics*, 12(2) , 109 -117 ( 2017)
- [2] Sertel, T.; Ozen, Y.; Cetin, S. S.; et al., *journal Of Materials Science-Materials In electronics* Volume: 29 Issue: 3 Pages: 1939-1946
- [3] M. Yıldırım, H. Durmuş, Selçuk Ü. *Fen Ed Fak Fen Dergisi*, 30, 93-96 (2007).
- [4] M A Moram and M E Vickers 2009 *Rep. Prog. Phys.* 72 036502
- [5] V. M. Kaganer, O. Brandt, A. Trampert, and K. H. Ploog, ' X-ray diffraction peak profiles from threading dislocations in GaN epitaxial films', *Physical Review B* 72, 045423 (2005)
- [6] Hammond C 2001 *The Basics of Crystallography and Diffraction* 2nd edn (Oxford: Oxford University Press)

# Figures

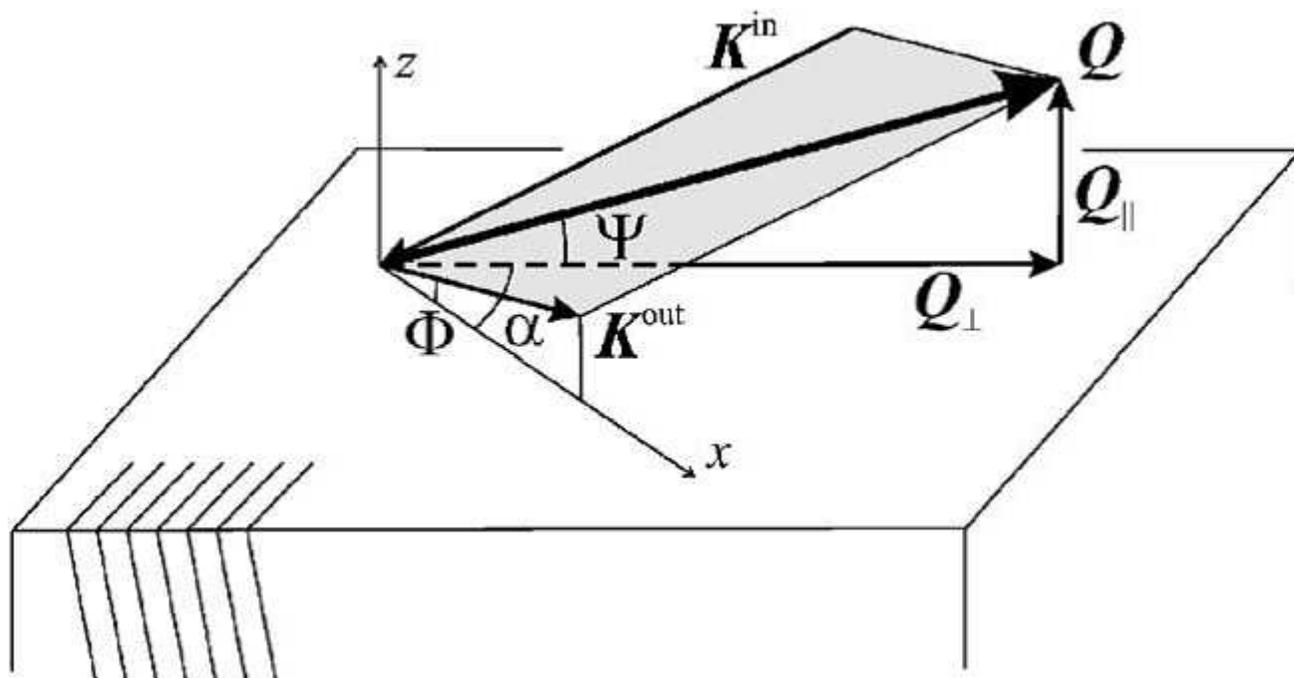
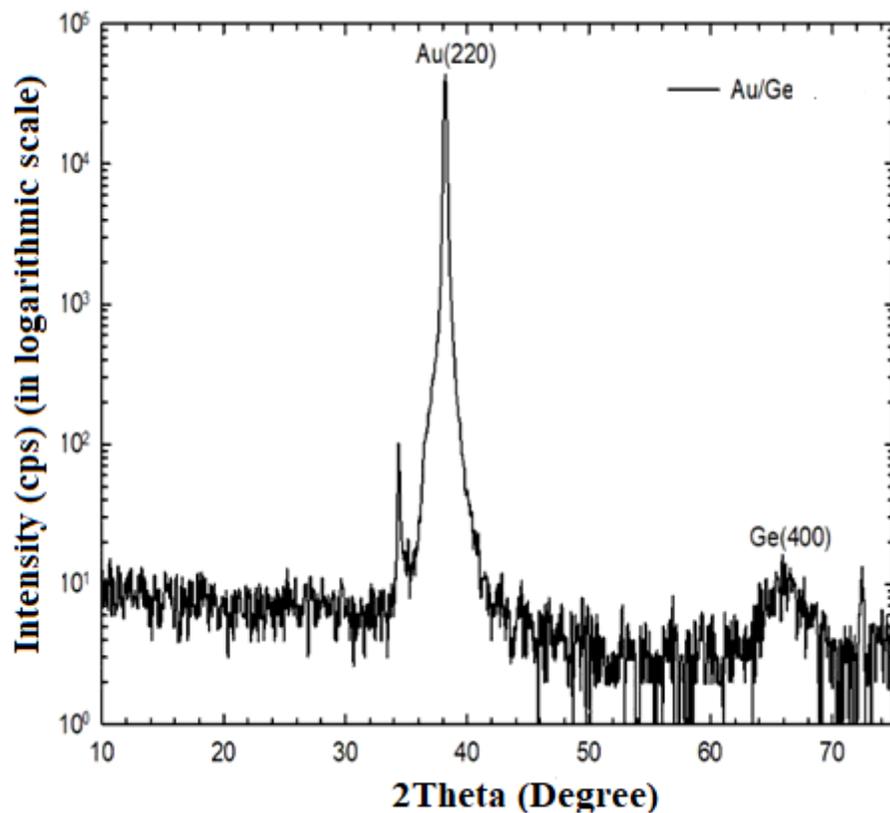


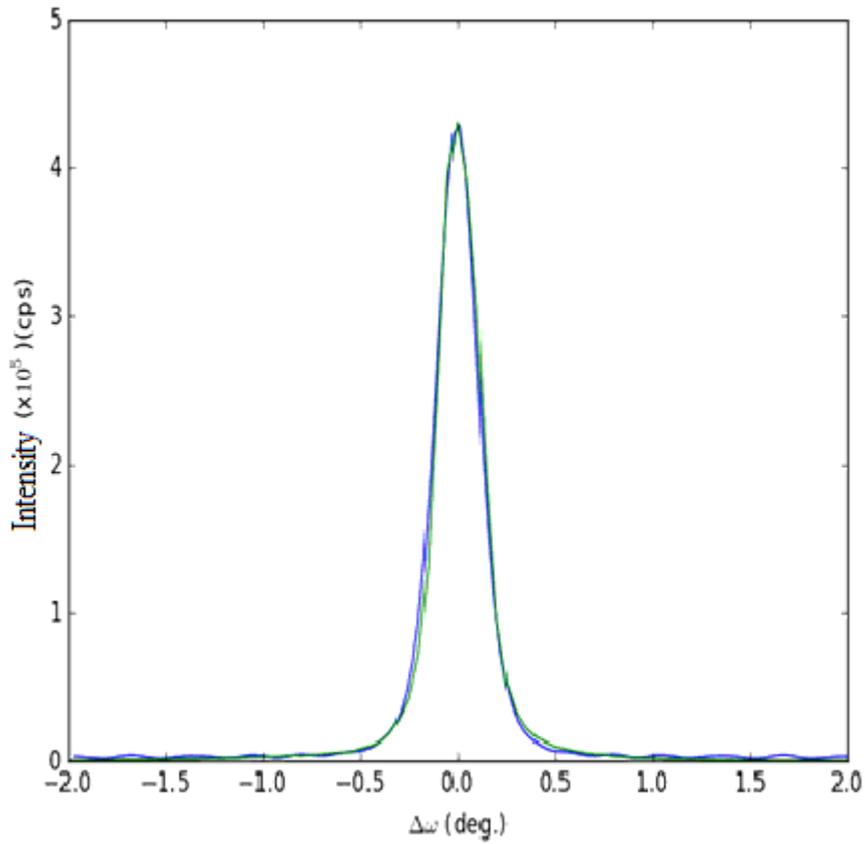
Figure 1

Curve geometry X-Ray pattern. Lattice planes of real reflection are described in left down side of figure. Incident wave  $K^{\text{in}}$  and diffracted wave  $K^{\text{out}}$  makes the same  $\Phi$  angle with sample surface. Scattering vector  $Q$  makes  $\Psi$  angle with sample surface [5].



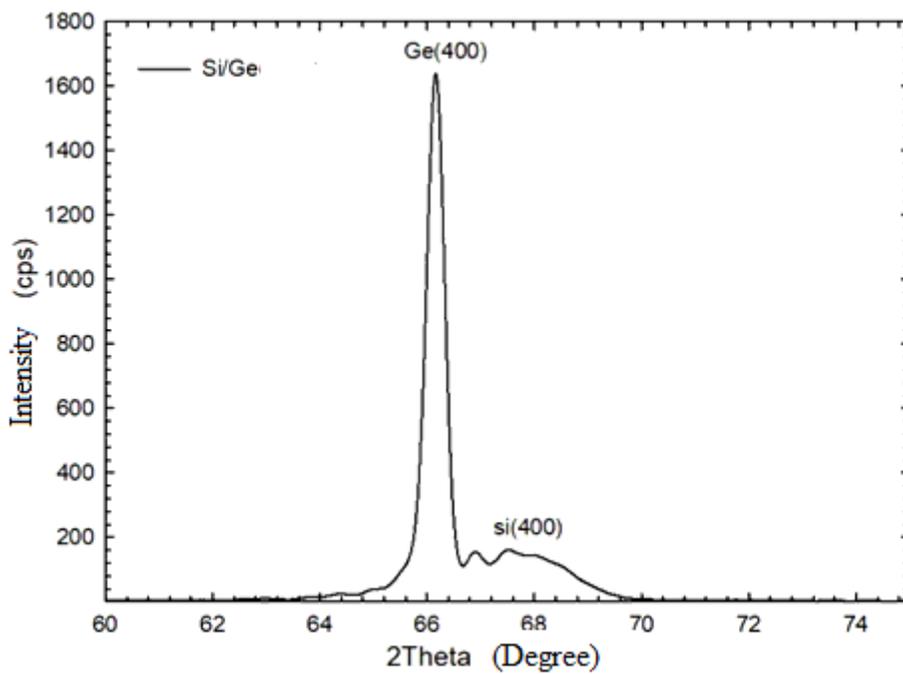
**Figure 2**

2 theta scan of Au/Ge(substrate)



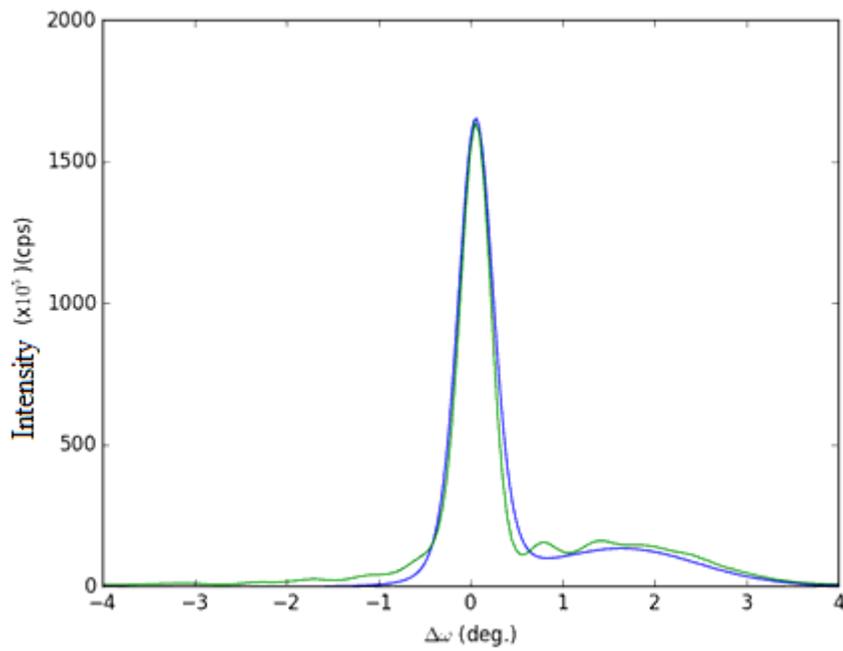
**Figure 3**

Profile fit curve of Au layer grown on Ge in (220) XRD reflection plane.



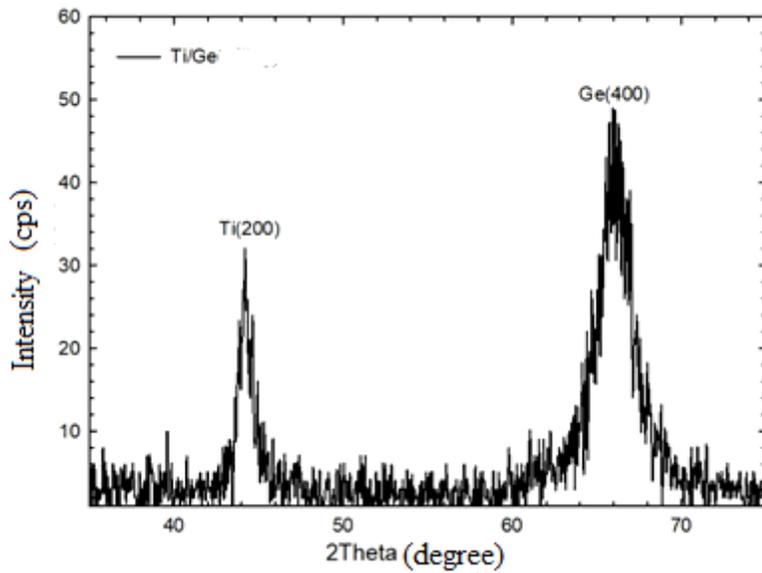
**Figure 4**

2 theta XRD scan of Si/Ge (substrate).



**Figure 5**

Profile fit curve in (400) plane for Si grown on Ge.



**Figure 6**

2theta scan for Ti/Ge (substrate)

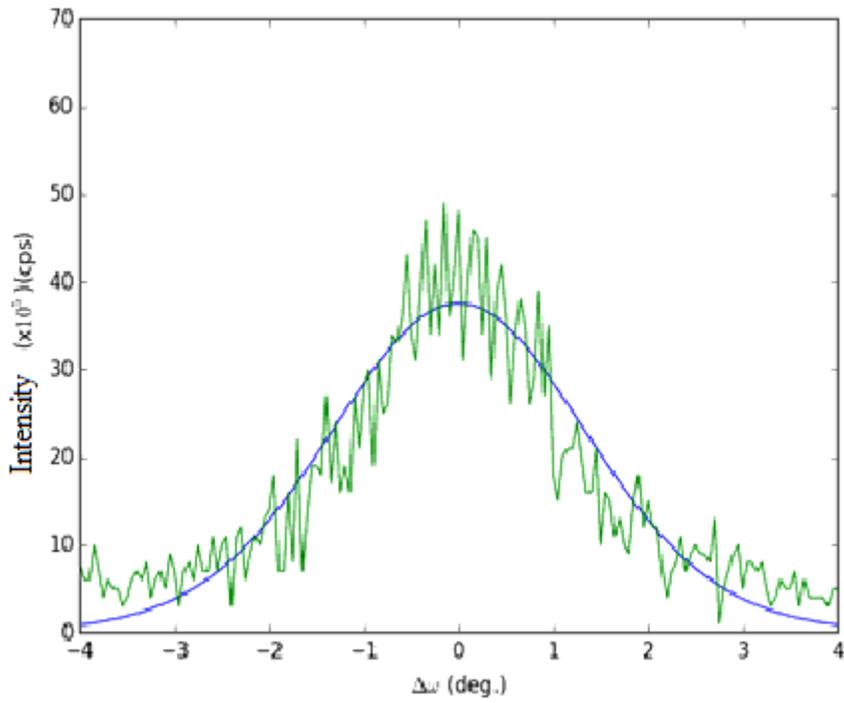


Figure 7

Profile fit curve for Ti grown on Ge for (200) plane.

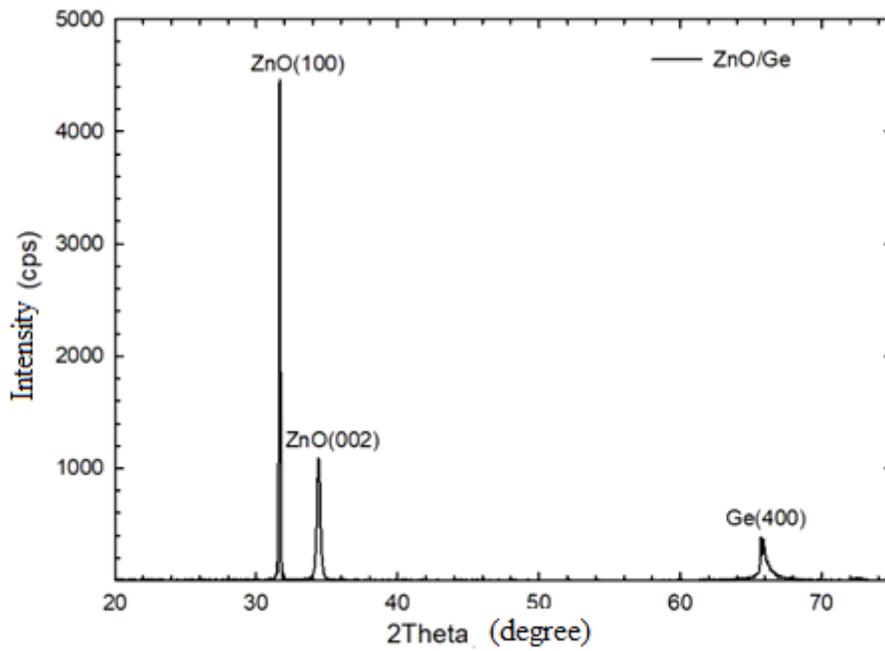


Figure 8

2xtheta versus intensity plot for ZnO on Ge

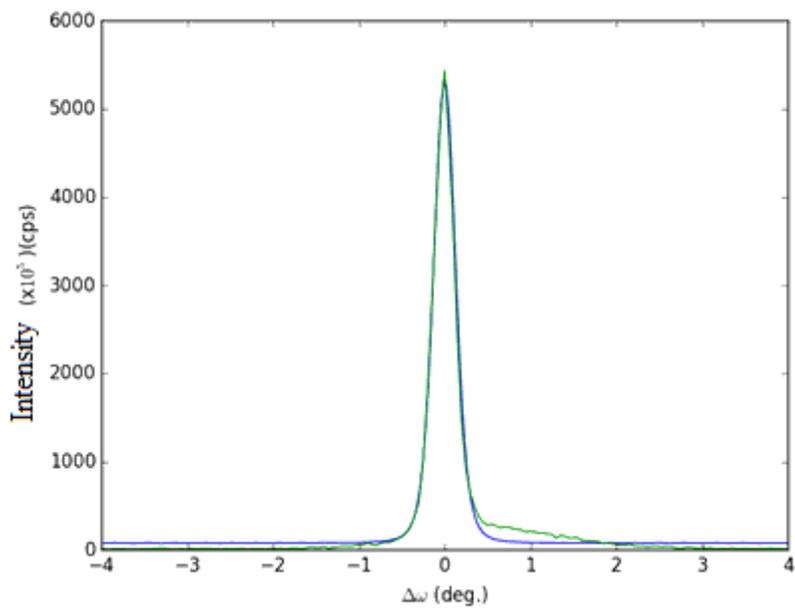


Figure 9

Profile fit curve for ZnO grown on Ge for (002) plane.

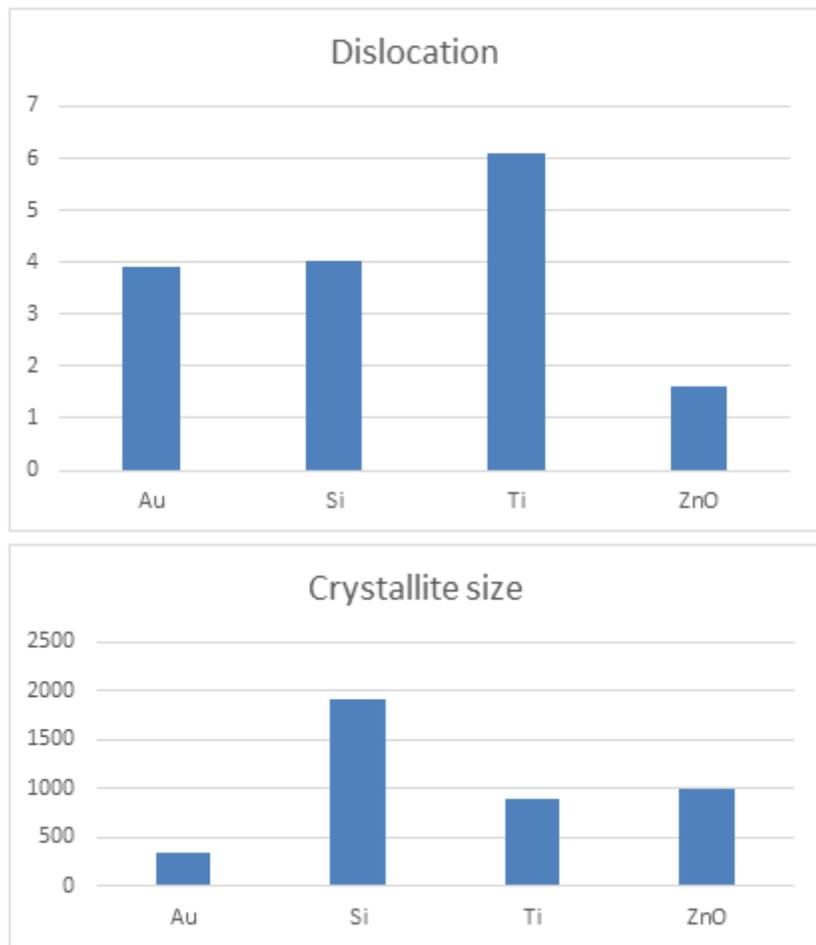


Figure 10

Comparison of dislocation density and correlation lengths for Au, Si, Ti and ZnO layers grown on Ge.