02-Methods for Structure Determination and Analysis, Computing and Graphics

$$B = \frac{F(h,k,l) * F(h,k,l)^{*} - F(-h,-k,-l)*F(-h,-k,-l)^{*}}{2*\,[F(h,k,l) * F(h,k,l)^{*} + F(-h,-k,-l)*F(-h,-k,-l)^{*}]} \quad (1)$$

depends solely on the amount of the anharmonic force constant ß which is described in terms of an anharmonic one-particle potential expanded up to the third order (Dawson, Hurley & Maslen,1967)

$$V(x,y,z) = V_{oi} + \alpha_i/2 * (x^2+y^2+z^2) + \beta_i\, xyz + \ldots \quad (2)$$

(α_i describes the harmonic force constant). ß can be determined by measuring B near the K-absorption edge of any constituent. This experiment was performed for the (6,6,6) and (-6,-6,-6) reflection of GaAs between λ = 0.90 and 0.97Å near the arsenic K- edge. The HUBER four circle goniometer at the HASYLAB beamline D3 was used. The wavelength was tuned by a silicon (111) double crystal monochromator to an accuracy better than $\delta\lambda$ = 0.001 Å. The normalized scattering power was measured from <111> and <-1,-1,-1> cut single-crystal wafers (A and B surface) having nearly the same thickness of about 0,350 mm. Within the angular limits of the diffractometer ($2\Theta \leq 145°$) the (6,6,6) was measureable up to $\lambda \leq 1.0$ Å. The range between λ =0,9 Å and λ = 1.0 Å was chosen for measurement. For each wavelength ω-scans were performed for a range of ψ- values (PSI scan) in order to find regions free from Umweganregung. A nearly linear slope of I(6,6,6) and I(-6,-6,-6) was found between λ = 0.9 Å and about λ = 0.95 Å (Fig.1). Due to the influence of EXAFS oscillations the scattering power did not follow the expected behavior at larger wavelengths, thus the range above 0,95 Å was neglected in the further interpretation .The standard deviations of the measured I(6,6,6) and I(-6,-6,-6) are in the 1% region for each λ. In order to enhance the accuracy of the evaluated B(λ) the I(λ) for both reflections were approximated by straight lines. The evaluated B-values are determined using pairs of experimental I(h,k,l,λ) and the value from the fitted straight line. The anharmonicity constant was evaluated to ß = -1.75 ± 0.15 J / Å^3. Its accuracy is much better than that given in previous publications (Bilderback 1976). The anharmonicity of GaAs is almost the same as that of Ge (Roberto, Battermann & Keating,1974). This is not surprising taking the predominant covalent bonding character into account .

Bilderback, D.H. Thesis, Purdue University, West Lafayette 1976
Dawson B, Hurley, A.C. & Maslen V.W. , (1967) Proc.Roy.Soc.(London) **A298** 289-306
Roberto J.B.,Battermann B.W. & Keating D. (1974) Phys.Rev.**B9**,2590-2599

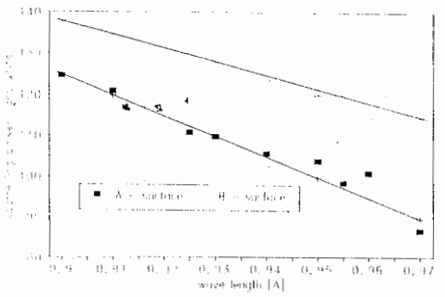

Fig.1: The intensities I(6,6,6) and I(-6,-6,-6) measured at regions free from Unweganregung between λ=0.9 Å and λ = 0.97 Å. The straight lines were obtained by regression of the measured values.

PS-02.02.07 THE SUPERSTRUCTURE OF $PbZrO_3$: INVESTIGATION AT Pb L_{III} AND Zr K ABSORPTION EDGES.
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It is demonstrated that a use of an effect of X-ray anomalous dispersion on superlattice diffraction is efficacious for the structure analysis. Intensity measurements on several superlattice diffractions from a $PbZrO_3$ single crystal have been made as a function of incident X-ray energy in the region of ±0.1 keV at Zr K and Pb L_{III} absorption edges at intervals of 0.008 keV. From the intensity, after the corrections for synchrotron orbit current, incident spectrum, Lorentz factor and absorption, $|F_{hk\ell}(\varepsilon)|^2$, the squared structure factor as a function of incident X-ray energy, is calculated. Observed changes in $|F_{hk\ell}(\varepsilon)|^2$ with ℓ even at the absorption edges directly indicate contributions of Pb and Zr atoms to the superstructure in the orthorhombic ab plane. This is consistent with the structure model in the literature. On the other hand, $|F_{hk\ell}(\varepsilon)|^2$ with ℓ odd is expected to be independent of the incident energy on the basis of the model; in other words, Pb and Zr atoms have no contribution to the superstructure along the c axis. On the contrary, a significant intensity change in $|F_{hk\ell}(\varepsilon)|^2$ at Zr K absorption edge is observed for the superlattice diffractions with ℓ odd; the results show the existence of displacement vector component of Zr atom along the c axis. The advantages of the present method are that elements to be examined can be selected by tuning incient X-ray energy, and that a relative intensity change due to a change in anomalous dispersion terms is quite large even if the superlattice diffraction is extremely weak.

PS-02.02.08 SOME NEW METHODS OF APPLYING MULTI-WAVELENGTH ANOMALOUS SCATTERING DATA.
By Fan Hai-fu, Institute of Physics, Beijing, China and M M Woolfson and Yao Jia-xing*, Department of Physics, University of York, UK.

Two analytical methods of using multi-wavelength anomalous scattering data are described. The first of these, called AGREE, explores a range of values of $|F|$, the contribution of the non-anomalous scattering, and finds that value which gives the greatest consistency of the anomalous scattering contribution for the different wavelengths, given all the magnitudes $|F^+|$ and $|F^-|$. The information enables both the positions of the anomalous scatterers to be found and also the angle, θ, between F and the real part of the non anomalous scattering - and thus the phase of F.

The second method, called ROTATE, assumes that the positions of the anomalous scatterers are known. Starting with six possible angles θ, uniformly occupying the range 0 to 2π and a value of $|F|$ which is the average of all the values of $|F^+|$ and $|F^-|$ the values of θ and $|F|$ are refined by a least-squares technique which takes into account the standard deviations of the observations. The values giving the least residuals are accepted.

Where there is only one type of anomalous scatterer then for different wavelengths the values of $|F^+|$ and $|F^-|$ should be related by having

