Charge, Spin & Momentum Density III
New Information on Crystals from Electron Diffraction

MS09.03.01 QUANTITATIVE CONVERGENT BEAM ELECTRON DIFFRACTION APPLIED TO BONDING STUDIES. R. Høier, Department of Physics, Norwegian University of Science and Technology, N-7034 Trondheim, Norway.

Methods for quantifying convergent beam electron diffraction patterns have been developed considerably over the last years. By multiparameter least square fitting of observed and calculated patterns we may today determine phases in non-centrosymmetric crystals and magnitudes of individual structure factors with an accuracy down to 0.1 degree and 0.1 %, respectively. The methods used to achieve this high accuracy are demanding both numerically and experimentally. Various strategies are being tested out, based on non-systematic, systematic row or zone axis diffraction, so far all of them on materials with relatively small unit cells.

Methodologically one of the main challenges is related to the numerical computations where the time consuming part is associated with solving a large non-Hermitic eigenvalue problem. This is done repeatedly a large number of times and the standard algorithm in use is very unfavorable for vectorization. We have optimized the diagonalization by determining efficient beam selection criteria. It is also found that the computational efficiency may be strongly increased by using parallel computers. Among the crystal parameters the Debye-Waller factor is still a problem. It is in general anisotropic and atom dependent and difficult to refine in the numerical procedures in use. This fact is in particular clear when experimentally determined bonding distributions are compared with excising computed ones that assume zero thermal vibrations.

Bonding has been investigated in the intermetallic material TiAl, undoped and doped with different other atom types. This material is interesting for high temperature applications, but has an unfortunate brittle-ductile transition at moderate temperatures. It has been found experimentally that the transition temperature is modified by the doping, and e.g. Mn is found to be beneficial in this respect. To understand this effect electron bond distributions of TiAl alloyed with Mn and other atoms have been investigated experimentally by convergent beam diffraction, electron energy loss fine-structure studies and theoretically by LAPW charge density calculations. Influence of dopant type is determined and experimentally consistent differences are seen for Mn, Cr and V.

MS09.03.02 DEBYE-WALLER FACTORS AND SUB-LATTICE ORDERING IN TiAl. S. Swaminathan, S. Jayanti, I. P. Jones*, D. M. Maher**, A. W. Johnson***, H. L. Fraser, Dept. of Matls. Sci. and Eng., The Ohio State University, OH, *School of Metallurgy and Materials, University of Birmingham, UK, **Dept. of Matls. Sci. and Eng., North Carolina State University, NC, ***Center for Microscopy, University of Western Australia, Australia.

The Debye-Waller factors of Ti and Al sites of Ti-^{54}Al alloy have been determined by four circle single crystal x-ray diffraction method. The analysis of x-ray diffraction data shows an ordered substitution of excess Al atoms (i.e. those over the stoichiometric composition) on the Ti sites. For the off-stoichiometric alloy studied, the least squares analysis yielded unequal Debye-Waller factors for the two crystallographically equivalent sites on the Ti-sublattice. This result is consistent with the ordering of excess Al atoms on Ti sites. On going research plan involves, data collection and analysis of x-ray diffraction intensities from a series of TiAl single crystals of different compositions. The results of this work and the effect of sublattice ordering on structure factors measured by convergent beam electron diffraction method will be presented.

MS09.03.03 ACCURATE STRUCTURE FACTOR MEASUREMENTS BY CONVERGENT BEAM ELECTRON DIFFRACTION. M. Saunders, P. A. Midgley, T. D. Walsh and R. Vincent, H. H. Wills Physics Laboratory, University of Bristol, Tyndall Avenue, Bristol, UK.

Quantitative Convergent Beam Electron Diffraction (CBED) is now emerging as the most accurate generally applicable method of low-order structure factor measurement for inorganic materials [1,2]. Using an energy-filter attached to a conventional Transmission Electron Microscope (TEM) it is possible to acquire diffraction patterns arising from electrons that have undergone predominantly elastic scattering. The theory of electron diffraction has now developed to the stage where accurate simulations of elastic scattering can be performed. The new techniques of quantitative CBED rely on adjusting the simulation until a best fit is obtained between the theory and experiment. The variable parameters include the low-order structure factors we wish to measure, the sample thickness and various scaling constants. Using silicon as a test case we have demonstrated [1] that quantitative CBED is as accurate as the best X-ray Pendellösung measurements [3]. A description of the zone-axis pattern matching technique developed by Bird and Saunders [4] will be given and the results for the silicon test case will be compared to the best X-ray and theoretical values. More recent results showing the application of the technique to other materials such as Ge, diamond, III-V semiconductors and metals such as Ni and Cu will also be discussed.

[1] M. Saunders, et al. (1995) Ultramicroscopy 60, 311.
[2] J.C.H. Spence and J.M. Zuo (1992) Electron Microdiffraction, Plenum, New York.
[3] Z.W. Lu, et al. (1993) Phys. Rev. B47(15), 9385 and references therein.
[4] D.M. Bird and M. Saunders (1992) Ultramicroscopy, 45, 241.

MS09.03.04 DETERMINATION OF BOND CHARGE DENSITY AND TEMPERATURE FACTORS FOR NiAl BY CONVERGENT BEAM ELECTRON DIFFRACTION. A. L. Weickenmeier, W. Nüchter, J. Mayer, Max-Planck-Institut für Metallforschung, Seestrasse 92, 70174 Stuttgart, Germany

Quantitative convergent beam electron diffraction (CBED) is increasingly appreciated as a very powerful tool to determine bond charge densities and temperature factors (TF) of very small (spot size 20 nm or less) areas of crystalline specimens. Using the Bloch wave formalism, CBED patterns are computed with extremely high accuracy as a function of the TFs and structure factors (SF, Fourier coefficients of the crystal potential). To derive the TFs and SFs the simulations are fitted to experimental patterns. Once a sufficient number of SFs has been obtained the potential is synthesized and by means of Poisson's equation converted to the total charge density. Using the measured TFs for the given temperature the total charge density of a corresponding crystal made from neutral atoms is constructed and subtracted yielding the bond charge density. Since the bond charge density is only a small fraction of the total charge density (of order 1 percent) maximum accuracy in experiment and data analysis is required.

The material we investigated is the ordered intermetallic phase NiAl, which is of technical interest as a high performance material at elevated temperatures. Since the room temperature brittleness is usually attributed to a partially covalent bonding, an experimental determination of the bond charge density will yield important information on the mechanical behavior.

With our energy filtering Zeis EM912 Omega transmission electron microscope equipped with a cooled YAG scintillator CCD-camera we have examined stochiometric NiAl at liquid nitrogen temperature. Specimens were prepared in different crystallographic orientations and investigated under various incident beam directions and for different thicknesses. Patterns were taken showing