Bibliography
The energy dispersive X-ray diffraction method: annotated bibliography 1968-78

E. LAINE, I. LÄHTEENMÄKI
Department of Physical Sciences, University of Turku, Turku, Finland

The present summary is a brief survey of the energy dispersive X-ray diffraction (EDXRD) method and an appraisal of the current position. A comprehensive chronological list of annotated references on EDXRD is given, covering the period 1968 to 1978. An index of authors is appended.

1. Introduction

X-ray diffraction methods play an important role in structural studies of materials. The concept of obtaining structural information with the new method, X-ray energy dispersive diffraction (EDXRD) was originally introduced by Buras *et al.* [1] and Giessen and Gordon [2] in 1968, independently. This method is based on the good energy resolution of semiconductor detectors. A white, collimated X-ray beam is scattered by the sample through a fixed optimized angle 2θ and the energy distribution of the scattered photons is analysed by a semiconductor detector connected to a multichannel pulse height analyser. The measured energy distribution of the scattered X-rays shows distinct peaks – as in the angle dispersive diffraction (AXRD) method – providing the required information for structural studies of materials.

The EDXRD method has many advantages compared with the conventional ADXRD method. The major benefits of the EDXRD technique seem to be:

(1) The fixed scattering angle geometry. This makes the method especially suitable for *in situ* studies in special environments (e.g. under very low or high temperatures and/or pressures). When the EDXRD method is used, only one entrance and one exit window are needed.

(2) The reduction of the exposure time. This makes possible a rapid structure analysis and the method is wellsuited for studies of materials which are inherently unstable over short periods.

(3) The whole spectrum of radiation diffracted by the sample is obtained simultaneously. This feature makes the method especially applicable to texture studies and to structure studies in which the rate of structural change of the sample depends on crystallographic orientations. Furthermore, as the entire pattern is recorded at the same time, variations in sample environment from one diffraction peak to the next are eliminated.

The major difficulties with using the EDXRD technique are:

(1) The peaks are more than one order of magnitude wide than in ADXRD.

(2) The spectral intensity distribution of the incident white X-rays available from a conventional target is non-uniform and limited.

(3) The influence of polarization of the primary X-ray beam on integrated intensities [12, 108].

(4) The need for energy-dependent absorption corrections

(5) The presence of fluorescence peaks

(6) The limited input count rate. The total input count rate of all the photons entering the detector must not exceed some 5000 to 10 000 photons sec^{-1}, because of limitations imposed by the present state of the electronics.

Some of the above-mentioned difficulties can be diminished or removed by using the X-radiation produced by high energy synchrotrons. The synchrotron radiation, especially from a storage ring, seems to be an ideal X-ray source for EDXRD. The high intensity (about 10^3 times larger than that using a conventional X-ray tube) of synchrotron radiation, its smooth spectrum and well-defined polarization make it possible to obtain

0022–2461/80/020269–10$03.00/0

quantitative intensity measurements. Furthermore, it is possible to obtain data with very high values of $\sin \theta/\lambda$.

In conclusion it can be stated that the EDXRD method has, over a period of ten years, become a recognized and remarkable tool among conventional diffraction techniques. We believe that in the future EDXRD will be complementary to, rather than a substitute for, ADXRD methods.

In the bibliography we have collected information on basic and applied research on EDXRD during a decade of its development. The papers are arranged chronologically, and alphabetically within each year.

2. Bibliography 1968–78

1968

1. B. BURAS, J. CHWASZCZEWSKA, S. SZARRAS and Z. SZMID, "Fixed Angle Scattering (FAS) Method for X-ray Crystal Structure Analysis", *Inst. Nucl. Res. (Warsaw)*, Rep. No. 894/II/PS (1968) 10 pp.
Described the EDXRD method and gives a formula for integrated intensity. Applications were also considered. This and following paper [2] reported the first work on EDXRD.
2. B. C. GIESSEN and G. E. GORDON, "X-ray Diffraction: New High-Speed Technique Based on X-ray Spectrography", *Science* 159 (1968) 973.
Described and discussed the EDXRD method. Diffraction patterns of platinum sheet were reported. See also [1].

1969

3. P. J. FREUD and P. N. LAMORI, "Non-Dispersive High Pressure High Temperature X-ray Diffraction Analysis", *Trans. Amer. Cryst. Assoc.* 5 (1969) 155.
Described a high pressure apparatus and discussed the use of the EDXRD method in high pressure measurements. Measurements on iron and NaCl were made at about 2.5 GPa pressure.

1970

4. H. COLE, "Bragg's Law and Energy Sensitive Detectors", *J. Appl. Cryst.* 3 (1970) 405.
Considered theoretically Bragg's law and energy and angle dispersive diffraction methods. An expression for the power per unit length of diffracted cone for a flat powder sample is given in the case of EDXRD.
5. J. I. DREVER and R. W. FITZGERALD, "Fluorescence Elimination in X-ray Diffractometry with Solid State Detectors", *Mater. Res. Bull.* 5 (1970) 101.
Discussed the use of solid state detectors in angle dispersive diffractometry. The energy resolution of solid state detectors was sufficient to discriminate the fluorescence of sample and to eliminate the need for a $K\beta$ filter. The peak-to-background ratio was higher than in the case of the conventional counters.
6. B. C. GIESSEN and G. E. GORDON, "Recent Developments in Spectrometric Powder Diffractometry", *Norelco Rep.* 17 (1970) 19.
Reviewed problems in instrumentation and diffraction relations and discussed some applications.

1971

7. P. BANERJEE and P. CHARBIT, "Schnelle Röntgenbeugungsanalysen mit Hilfe eines Si(Li)-Halbleiterdetectors", *Siemens-Z.* 45 (1971) 549 (in German).
Described the EDXRD method, considered absorption in the sample and derived an expression for intensity. The method was used for determination of austenite content in austenite ferrite steel.
8. J. CHWASZCZEWSKA, S. SZARRAS, Z. SZMID and M. SZYMCZAK, "Application of Semiconductor Detectors in Crystal Structure Investigations", *Phys. Stat. Sol. (a)* 4 (1971) 619.
Reported on the EDXRD method and compared the calculated and observed positions of diffraction peaks for Al, Cu, Pt and Au foils and for a powdered Si sample. For the Si sample a comparison of the observed and calculated intensities was made. The resolution of the detector system was about 1 keV.
9. J. I. DREVER, "Early Diagenesis of Clay Minerals, Rio Ameca Basin, Mexico", *J. Sedim. Petro.* 41 (1971) 982.
Reported a clay mineral analysis in which the EDXRD method was used.
10. R. E. FERRELL, "Applicability of Energy Dispersive X-ray Powder Diffractometry to Determinative Mineralogy", *Amer. Mineral.* 56 (1971) 1822.
Demonstrated the applicability of EDXRD to the identification of single and multicomponent mineral powders. In the latter case the resolution of the detector was observed to limit the analysis.
11. E. LAINE and I. LÄHTEENMÄKI, "Structure and Microstructure Study on Splat-Cooled Cadmium", *J. Mater. Sci.* 6 (1971) 1418.
Reported preferred orientation and its recovery in splat-cooled cadmium. The intensity ratio of the 002 diffraction line and the $CdK\alpha$ fluorescence line were used as a measure of the preferred orientation.
12. V. W. SLIVINSKY, "Polarization of Bremsstrahlung from X-ray Tubes", *Amer. Phys. Soc. Bull.* 16 (1971) 546.
Reported the polarization of bremsstrahlung spectra from commercial X-ray tubes using high resolution Si(Li) and Ge(Li) detectors. The results showed that the X-rays at the high energy limit of the bremsstrahlung spectrum were strongly polarized in a direction parallel to the electron beam, whilst the lower energy X-rays exhibited a low but constant polarization in the same direction. The average polarization was found to increase as the voltage applied to the X-ray tube decreased. Abstract only.
13. M. M. UMANSKII and D. M. KHEIKER, "Equipment for X-ray Structural Investigations (Review)", *Instrum. & Exp. Tech. (USA)* 14 (1971) 665.

Reviewed modern equipments for X-ray structural analysis. EDXRD was also mentioned.

1972

14. L. M. ALBRITTON and J. L. MARGRAVE, "Polychromatic X-ray Diffraction; A Rapid and Versatile Technique for the Study of Solids under High Pressure and High Temperature", *High Temp.-High Press.* **4** (1972) 13.
Described apparatus and techniques for high pressure measurements with EDXRD. Reported high pressure investigations (up to 6 GPa) of Al and KCl at room temperature.
15. A. HEWITT-EMMETT, "The Application of the X-ray Diffractometer to Process Control in Cement Kilns", *Cement Technol.* 3 (1972) 58.
Described application of the EDXRD system (described in [21]) to process control in the cement industry.
16. H. A. KÄHKÖNEN, "A Semiconductor Detector in a Small Angle X-ray Diffraction Study", *J. Phys. E* 5 (1972) 652.
Described a small angle X-ray diffraction system in which a solid state detector was used.
17. E. LAINE, I. LÄHTEENMÄKI and M. KANTOLA, "Measuring of Diffraction Intensities by the Energy Dispersive Si(Li) Detector", *Acta Cryst.* **A28** (1972) S245.
Considered the accuracy of lattice parameter measurements and the agreement between the observed and calculated intensities of the peaks. Abstract only.
18. E. LAINE, I. LÄHTEENMÄKI and M. KANTOLA, "Adaptation of Solid State Detector in X-ray Powder Diffractometry", *X-Ray Spectrom.* **1** (1972) 93.
Described the EDXRD method and reported some experimental results obtained from Cu, Ni, Cd, Au, Zn and NaCl-powdered samples. The accuracy of lattice parameter and intensity measurements was discussed.
19. J. P. LAURIANT and P. PÉRIO, "Adaptation d'un Ensemble de Détection Si(Li) à un Diffractometre X", *J. Appl. Cryst.* **5** (1972) 117 (in French).
Reported the use of a Si(Li) semiconductor detector with a liquid nitrogen tank separate from the crystal. The cooling of the crystal was adjusted through a liquid nitrogen transfer line. Some experimental results were also discussed.
20. G. W. MARTIN and A. S. KLEIN, "A Complete Instrumental System for Energy Dispersive Diffractometry and Fluorescence Analysis", *Adv. X-ray Anal.* **15** (1972) 254.
Described a system for energy dispersive diffractometry and simultaneous fluorescence analysis. Testing experiments were also reported and the "best" 2θ angle discussed.
21. J. C. NUTTER, "A Non-dispersive On-stream X-ray Diffractometer for the Cement Industry", *Cement Technol.* 3 (1972) 55.
Described the theory and design of an EDXRD system for process control in cement kilns. See also [15].
22. A. OKAZAKI, "Use of Non-dispersive X-ray Diffraction for Critical Scattering Measurements", *Acta Cryst.* A28 (1972) S247.
Reported an EDXRD system for low temperature measurements. Abstract only.
23. C. J. SPARKS and D. A. GEDCKE, "Rapid Recording of Powder Diffraction Patterns with Si(Li) X-ray Energy Analysis System: W and Cu Targets and Error Analysis", *Adv. X-ray Anal.* **15** (1972) 240.
Described the EDXRD method, discussed errors caused by misalignment of the diffractometer and X-ray penetration into the sample together with errors in energy calibration of the multichannel analyser.

1973

24. D. CARPENTER and J. THATCHER, "Evaluation of the Energy Dispersive Detector as a Detector–Filter System for the X-ray Diffractometer", *Adv. X-ray Anal.* **16** (1973) 322.
Compared the solid state detector with conventional scintillation and proportional detectors in angle dispersive diffractometry.
25. H. S. DEBEN and B. BROYDE, "Compositional and Texture Analysis of Tantalum Thin Films by Energy Dispersive X-ray Analysis", *Appl. Spectrosc. (USA)* 27 (1973) 99.
Quantitative measurements, using EDXRD, of concentrations are given for the phases present in undoped tantalum thin films.
26. T. FUKAMACHI, S. HOSOYA and O. TERASAKI, "The Precision of Interplanar Distances Measured by an Energy Dispersive Diffractometer", *J. Appl. Cryst.* **6** (1973) 117.
Discussed and estimated the precision of measured lattice spacings by EDXRD. It is shown that a precision of about 0.01% is attainable without much difficulty by the use of a suitable Soller-slit system and a suitable scattering angle. This was demonstrated on an Al powder sample.
27. T. FUKAMACHI, S. TOGAWA and S. HOSOYA, "Escape Peaks Caused by a Ge(Li) Detector in an Energy Dispersive Diffractometer", *J. Appl. Cryst.* **6** (1973) 297.
Reported the appearance and identification of escape peaks in an EDXRD spectrum.
28. S. HOSOYA and T. FUKAMACHI, "Rapid Determination of Polarity Sense by an Energy Dispersive Diffractometer", *J. Appl. Cryst.* **6** (1973) 396.
Determined the polarity sense of the non-centrosymmetric GaP crystal with EDXRD by choosing a suitable scattering angle. By EDXRD the polarity sense can be determined rapidly and unambiguously with high reliability and this can be used to distinguish between an enantiomorphous pair. This method will, moreover, be very useful both in the study of anomalous scattering itself and in its various applications to crystal structure analysis.
29. R. JENKINS, "Combination of the Energy Dispersion Spectrometer with the Powder Diffractometer", *Norelco Rep.* **20** (1973) 22.
Described the combination of an energy dispersive spectrometer and a diffractometer. Some comments on practical use were also made.
30. E. LAINE, "X-ray Diffraction Studies of Structures in Some Metals and Alloys", *Ann. Univ. Turku.* **160A**

(1973) 11 pp.
Thesis. Reported a summary of several investigations including also papers [11], [17] and [18].

31. E. LAINE and I. LÄHTEENMÄKI, "A Rapid Energy Dispersive X-ray Diffraction Method for Structure Analysis", *Kemian Teollisuus* **30** (1973) 381.
Described the principle of the EDXRD method. Comparison of measured peak positions and intensities obtained using various exposure times was made with those calculated theoretically. It was found that the accuracy of lattice parameter did not depend essentially on the exposure times ($\geqslant 10$ sec). Also, a good agreement was obtained between the observed and calculated intensities of the peaks, when the exposure times were longer than 1 min.

32. E. LAINE and L. TUKIA, "Isotope-Excited X-Ray Fluorescense Analysis of Binary Alloys Using Energy Dispersion", *X-Ray Spectrom.* **2** (1973) 115.
Reported on a radioisotope-excited X-ray fluorescence method for the analysis of binary alloys. The method was applied to Cu–Sb alloys. The results were checked by EDXRD.

33. W. LIN, "A Rapid Fluorescence and Energy Powder Pattern Analysis System", *Adv. X-Ray Anal.* **16** (1973) 298.
Reported a computer-coupled EDXRD system capable of performing rapid elemental analysis and phase identification employing fluorescence and energy powder pattern analyses. Software was developed for qualitative or/and quantitative elemental analysis.

34. J. M. PROBER, J. M. SCHULTZ and S. I. SANDLER, "Liquid Structure Analysis by Energy-scanning X-ray Diffraction", *Nature Phys. Sci.* **243** (1973) 32.
Reported preliminary results of quantitative liquid structure analysis using EDXRD. The radial distribution of Hg atoms in a melt at room temperature was determined. See also [51].

35. A. J. C. WILSON, "Note on the Aberrations of a Fixed-Angle Energy-Dispersive Powder Diffractometer", *J. Appl. Cryst.* **6** (1973) 230.
Described geometrical and physical aberrations affecting the positions and breadths of diffraction maxima in EDXRD and ADXRD methods. The main differences were investigated and the accuracy of spacing measurements was discussed.

1974

36. B. BURAS and L. GERWARD, "Relations between Integrated Intensities in Crystal Diffraction Methods for X-rays and Neutrons", Report no. 7 (1974) of Laboratory of Applied Physics III, Technical University of Denmark, 18 pp.
It is shown that there are simple relations between the formulae for the integrated intensities of the different X-ray diffraction methods, and that the intensity formulae for all the methods can be simply generated, provided that one of them is derived conventionally.

37. B. BURAS, J. STAUN OLSEN, A. LINDEGAARD ANDERSEN, L. GERWARD and B. SELSMARK, "Evidence of Escape Peaks Caused by a Si (Li) Detector in Energy-Dispersive Diffraction Spectra", *J. Appl. Cryst.* **7** (1974) 296.
Reported the appearance and identification of silicon Kα escape peaks in EDXRD spectra.

38. I. HEILMANN, J. M. KNUDSEN, N. B. OLSEN, B. BURAS and J. STAUN OLSEN, "Studies of Thermal Decomposition of $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$", *Sol. Stat. Comm.* **15** (1974) 1481.
Reported studies of the thermal decomposition products of Mohr's salt $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$. In this study the Mössbauer effect, ADXRD, EDXRD, infra-red spectroscopy, gravimetric and thermal differential methods are used.

39. E. LAINE, I. LÄHTEENMÄKI and M. HÄMÄLÄINEN, "Si(Li) Semiconductor Detector in Angle and Energy Dispersive X-ray Diffractometry", *J. Phys. E* **7** (1974) 951.
Described an X-ray diffractometer system which can be used in angle dispersive, energy dispersive and combined angle and energy dispersive diffraction measurements. The appropriateness and the advantages of these methods under different experimental conditions are discussed.

40. J. SZPUNAR, M. OJANEN and E. LAINE, "Application of the Energy Dispersive X-Ray Diffraction Method to Texture Measurements", *Z. Metallkde* **65** (1974) 221.
Described the application of EDXRD to texture measurements. The method described permits the direct recording of information necessary for inverse pole figure determination. Inverse pole figure measurements of cold rolled steel were carried out.

41. A. J. C. WILSON, "Synergistic Indentification by Energy-Dispersive Diffractometry", *J. Appl. Cryst.* 7 (1974) 93.
Letter to the Editor.

42. A. P. VOSKAMP, "High-Speed Retained Austenite Analysis with an Energy Dispersive X-ray Diffraction Technique", *Adv. X-ray Anal.* **17** (1974) 124.
Discussed the applicability of EDXRD for the determination of retained austenite. Experiments have been carried out using both the conventional ADXRD and the EDXRD technique. The results obtained were compared and the error possibilities discussed. Calculations for intensity and intensity correction factors were also reported.

1975

43. O. ALSTRUP, L. GERWARD, B. SELSMARK, B. BURAS and J. STAUN OLSEN, "Polarization of X-rays from a Thick Target and its Influence on Integrated Intensities of Bragg Reflections", *Acta Cryst.* **A31** (1975) S234.
Reported preliminary qualitative results from polarization of X-ray tube spectrum. Abstract only. See also [108].

44. P. BANERJEE and P. CHARBIT, "Si(Li)-Halbleiterdetector zur AlF_3-Überschussbestimmung im Aluminiumelectrolyseofen", *Siemens-Z.* **49** (1975) 54. (in German)
Reported a possible method to analyse the $Na_5Al_3F_{14}$ – and CaF_2 – content of outlet specimens from an aluminium oven. From these the AlF_3 remaining can

be calculated.

45. B. BURAS, J. STAUN OLSEN, L. GERWARD, B. SELSMARK and A. LINDEGAARD ANDERSEN, "Energy-Dispersive Spectroscopic Methods Applied to X-ray Diffraction in Single Crystals", *Acta Cryst.* **A31** (1975) 327.
Described and investigated two single-crystal energy-dispersive crystallographic methods. Formulae for integrated intensities were derived for mosaic and perfect single crystals. Experimental results and a comparison between measured and calculated integrated intensities for a perfect germanium crystal were given. Special features and possible applications of the methods were discussed.

46. T. FUKAMACHI and S. HOSOYA, "The Measurement of Anomalous Scattering Factors near the GaK Absorption Edge in GaP", *Acta Cryst.* **A31** (1975) 215.
Reported a study in which the energy dependence of anomalous scattering factors ($\Delta f'$ and $\Delta f''$ values) of Ga was measured with EDXRD around the GaK edge. The values of $\Delta f''$ were determined by measurement of the absorption coefficient. The values of $\Delta f'$ were then obtained from the precisely measured ratio of Friedel-pair reflections from a (1 1 1) single-crystal plate of polar GaP.

47. K. INOUE, A. NAKAUE and Y. YAGI, "Measurement of Melting Points of Pb up to 90 kbar by Energy Dispersive X-ray Diffraction Technique", *Rev. Phys. Chem. Jap.* **1975 (SI)** (1975) 757.
Reported a cubic anvil press apparatus, with which one can obtain energy dispersive powder diffraction patterns at pressures up to 13 GPa and temperatures to 1500° C. The melting points of Pb were determined up to 9 GPa (based upon the NaCl pressure scale).

48. J. H. JOHNSTON and J. F. DUNCAN, "Manganese Ion Site Distribution Studies in Tourmaline by Anomalous X-ray Scattering Methods", *J. Appl. Cryst.* 8 (1975) 469.
Reported a determination of Mn^{3+} site distribution in a manganese-containing tsilaisite tourmaline sample. For this, anomalous X-ray scattering techniques, in conjunction with the EDXRD procedures, were used.

49. V. K. MAHAJAN, P. T. CHANG and J. L. MARGRAVE, "A Study of Phase Transitions in Lead Difluoride by Use of Polychromatic X-ray Diffraction", *High Temp.-High Press.* 7 (1975) 325.
Reported a study in which the EDXRD method was used to produce patterns from α-PbF_2 and β-PbF_2. The results obtained at room temperature and at 314° C, at 1 atm., and at room temperature at 550 MPa, were in good agreement with literature data.

50. S. G. METCALF and W. I. WINTERS, "An X-ray Diffraction Technique for Radioactive Powders", *Appl. Spectr.* 29 (1975) 519.
Reported an ADXRD system, which used a Si(Li) detector. Radioactive powder samples were measured using this system.

51. J.M. PROBER and J. M. SCHULTZ, "Liquid-Structure Analysis by Energy-Scanning X-ray Diffraction: Mercury" *J. Appl. Cryst.* 8 (1975) 405.
Reported quantitative liquid-structure analysis using EDXRD. The measurement of the primary beam spectrum, the nature of the absorption and the dispersion corrections, details of the polarization correction, the ranges of the atomic scattering factor and of the incoherent scattering term were discussed. The interference spectrum of liquid mercury was measured. See also [34].

52. J. M. SCHULTZ, "A New X-ray Diffraction-Fluorescence Method for the Study of Films: Preliminary Results for Cu_xS on CdS", *J. Appl. Cryst.* 8 (1975) 333.
Reported a structure study of an ion-exchanged layer of Cu_xS on CdS by EDXRD yielding diffraction and fluorescence results simultaneously. A reversible phase transition near 100° C was observed.

53. J. M. SCHULTZ and T. C. LONG, "Energy-Scanning Small-Angle X-ray Scattering: Polyethylene", *J. Mater. Sci.* **10** (1975) 567.
Demonstrated energy-scanning small-angle X-ray scattering; where λ-, rather than θ-space was scanned. Patterns in times as low as 100 sec were observed. Good correlation between θ- and λ-scanning was shown.

54. K. SYASSEN and W. B. HOLZAPFEL, "Energy-Dispersive X-ray Diffractometry in High Pressure Research", *Europhys. Conf. Abstr.* **1A** (1975) 75.
Reported an EDXRD system with a diamond-anvil high pressure device. Preliminary high pressure measurements were performed on samples of NaCl and Cs.

55. R. UNO and J. ISHIGAKI, "Determination of the Structure Factors by the White X-ray Diffraction from a Powder Sample of GaP", *Acta Cryst.* **A31** (1975) S236.
Reported preliminary results of study [56]. Abstract only.

56. R. UNO and J. ISHIGAKI, "Determination of Structure Factors by White X-ray Diffraction from a Powder Sample of GaP", *J. Appl. Cryst.* 8 (1975) 578.
Reported a determination of the energy spectrum of incident white X-rays, measurement of the energy dependence of the relative absorption coefficient, correction for the contribution from thermal diffuse scattering, and correction for the anomalous dispersion.

1976

57. J. BORDAS, I. H. MUNRO and A. M. GLAZER, "Small-Angle Scattering Experiments on Biological Materials Using Synchrotron Radiation", *Nature* **262** (1976) 541.
Reported an energy dispersive small-angle diffraction system, which used synchrotron radiation. Preliminary experiments on rat-tail tendon were also reported.

58. B. BURAS, J. STAUN OLSEN and L. GERWARD, "X-ray Energy-Dispersive Powder Diffractometry Using Synchrotron Radiation", *Nucl. Instr. Meth.* **135** (1976) 193.
Reported and discussed results of a test using synchrotron radiation in an EDXRD system.

59. A. FRIANT. "Applications des Spectrométres Si(Li) Dispersifs en Énergie", *Onde Electr.* **56** (1976) 69 (in

French).
Reviewed energy dispersive X-ray spectrometers, included X-ray analysis by X-ray fluorescence or charged particle excitation, and application of Si(Li) spectrometers in nuclear physics and X-ray diffraction.

60. T. FUKAMACHI, S. HOSOYA and M. OKUNUKI, "X-ray Intensity Measurements on Large Crystals by Energy-Dispersive Diffractometry. I. Energy Dependences of Diffraction Intensities near the Absorption Edge", *Acta Cryst.* **A32** (1976) 104.
Reported measurements of intensity variations of X-ray diffracted from a nearly perfect GaAs plate, in symmetrical Laue and Bragg cases, in the energy region near the AsK absorption edge with small energy intervals, by the use of EDXRD. The first paper of a series of three. See also [61] and [75].

61. T. FUKAMACHI, S. HOSOYA and M. OKUNUKI, "X-ray Intensity Measurements on Large Crystals by Energy-Dispersive Diffractometry. II. Energy Dependences of the Friedel Pair Intensities and Their Ratio near the Absorption Edge", *Acta Cryst.* **A32** (1976) 245.
Considered theoretically the intensity ratio of Friedel pair reflections in a perfect crystal as well as in a mosaic crystal, both in the symmetrical Bragg case and in the symmetrical Laue case. The experimental measurements were carried out on a nearly perfect GaAs crystal plate in the energy range near the AsK absorption edge by the use of EDXRD. See also [60] and [75].

62. L. GERWARD, S. LEHN and G. CHRISTIANSEN, "Quantitative Determination of Preferred Orientation by Energy Dispersive X-ray Diffraction", *Texture* **2** (1976) 95.
Reported the use of EDXRD for quantitative determination of preferred orientations in polycrystalline specimens and demonstrated the adaptability of the method to *in situ* studies.

63. L. GERWARD, S. MØRUP and H. TOPSØE, "Particle Size and Strain Broadening in Energy-Dispersive X-ray Powder Patterns", *J. Appl. Phys.* **47** (1976) 822.
Reported the development of EDXRD for a rapid analysis of broadening of diffraction lines in powder patterns. Experimental results were given for magnetite powders, with sizes in the range 5 to 20 nm, and compared with those from standard ADXRD and electron microscopy.

64. P. JUGELT and S. KOCH, "Einsatz der Energiedispersiven Pulverdiffraktometrie zur Phasenanalyse", *Wiss. Z. Tech. Univ. Dres.* **25** (1976) 391 (in German).
Reported the principle of the EDXRD method and its applications for quantitative phase analysis.

65. A. KOTLICKI, N. BOYE OLSEN and J. STAUN OLSEN, "Mössbauer and X-ray Study of Proton Radiation Effects in Biotite", *Rad. Effects* **28** (1976) 1.
Reported Mössbauer and X-ray investigations of 3 MeV proton irradiation effects in biotite.

66. J. C. MALAURENT and J. DIXMIER, "X-ray Diffractometry in Situ of Evaporated Thin Films: Application to Amorphous Tellurium", *Thin Solid Films* **36** (1976) L1.
Described an EDXRD system for studying the structure of evaporated thin films *in situ*.

67. M. MANTLER and W. PARRISH, "Energy Dispersive X-ray Diffractometry", *Adv. X-ray Anal.* **20** (1976) 171.
Described the principles, methods, instrumentation and results of EDXRD, and a computer method of profile fitting, to obtain corrected intensities and peak energies from isolated and overlapping reflections.

68. P. E. D. MORGAN, "Low Temperature Synthetic Studies of Beta – Aluminas", *Mater. Res. Bull.* **11** (1976) 233.
Reported a phase transition study, where the ADXRD method with an energy dispersive solid state detector was used.

69. T. NAKAJIMA, T. FUKAMACHI, O. TERASAKI and S. HOSOYA, "The Detection of Small Differences in Lattice Constant at Low Temperature by an Energy-Dispersive X-ray Diffractometer", *J. Appl. Cryst.* **9** (1976) 286.
Described a cryostat for the EDXRD systems so that the specimen can be measured between 1.48 K and room temperature. A typical example was described of detecting the difference in lattice constant between ^{92}Mo and ^{100}Mo. See also [81].

70. E. F. SKELTON, "Measurement of the Temperature Dependence of Debye-Waller Factors by Energy-Dispersive Methods: Application to $NbC_{0.98}$", *Acta Cryst.* **A32** (1976) 467.
Reported an EDXRD method for evaluating the temperature dependence of Debye–Waller factors, or equivalently the mean-square atomic displacements (MSD). A procedure was employed to determine the temperature dependence of MSD of $NbC_{0.98}$ over the temperature range from 296 to 600 K. Results were compared with values computed from a double-shell lattice–dynamic force model and with the thermal expansivity of $NbC_{0.98}$. See also [71].

71. E. F. SKELTON, "A New and Improved Method for Measuring the Temperature Dependence of Debye-Waller Factors with the Use of Energy Dispersive Analyses", *Rep. NRL Progr.* 1976 (1976) 1–11. See [70].

72. J. SZPUNAR, "Texture and Neutron Diffraction", *At. Energy Rev.* **14** (1976) 199.
Reviewed mainly texture and neutron diffraction. Also the application of EDXRD to texture studies was reported.

1977

73. J. BORDAS, A. M. GLAZER, C. J. HOWARD and A. J. BOURDILLON, "Energy-Dispersive Diffraction from Polycrystalline Materials Using Synchrotron Radiation, *Phil. Mag.* **35** (1977) 311.
Described EDXRD techniques using synchrotron radiation from the Daresbury synchrotron, NINA. By using a silicon powder standard the measurement of Debye–Waller factors and structure factors of NaCl and KCl has been achieved with good precision.

74. B. BURAS, J. STAUN OLSEN, L. GERWARD, G. WILL and E. HINZE, "X-ray Energy-Dispersive Diffractometry Using Synchrotron Radiation", *J.*

Appl. Cryst. **10** (1977) 431.
Discussed special features of EDXRD using synchrotron radiation based on experiments performed with the Deutsches Elektronen-Synchrotron, DESY. Studies of phase transformations and chemical reactions at elevated temperatures and at high pressures were reported. Studies of time dependent phenomena using pulsed external fields were also discussed.

75. T. FUKAMACHI, S. HOSOYA, T. KAWAMURA and M. OKUNUKI, "X-Ray Intensity Measurements on Large Crystals by Energy-Dispersive Diffractometry. III. Fine Structures of Integrated Intensities and Anomalous Scattering Factors near the K Absorption Edges in GaAs", *Acta Cryst.* **A33** (1977) 54.
Reported investigations of the anomalous scattering factor $f' + \mathrm{i}f''$ of GaAs very near the K absorption edges. With values obtained for f'' and f', the curves of integrated reflection power near the edges have been calculated by use of formulae presented in Parts I and II (papers [60] and [61]). The curves thus obtained agreed reasonably with the measured ones.

76. D. F. GRANT, E. J. LISHER and R. H. MITCHELL, "Equipping a Four-Circle Single-Crystal Diffractometer with an Interchangeable Si(Li) Solid-State Detector", *J. Appl. Cryst.* **10** (1977) 1970.
Reported an ADXRD system where a four-circle diffractometer was fitted with a Si(Li) solid state detector (SSD) which could be interchanged with a scintillation detector. The TDS intensities from single crystals of aluminium and vanadium were measured at a large number of points between the reciprocal-lattice points.

77. H. HELLER, J. SLAKHORST and T. VERBRAAK, "The Development of Rolling and Recrystallization Textures in High Purity Al", *Z. Metallkde* **68** (1977) 31.
Reported texture studies where the EDXRD method was used for measurements of the intensity of X-ray reflections.

78. E. LAINE, J. KIVILÄ and I. LÄHTEENMÄKI, "A Study of Preferred Orientation by Energy-Dispersive X-ray Diffraction", *Texture* **2** (1977) 243.
Reported an investigation of the influence of preferred orientation on integrated X-ray intensities in a powder specimen using the EDXRD method. The measurements were carried out using the Schulz technique using a defocusing correction. Experimental results were given for three Al powder specimens.

79. M. NAGAO and S. KUSUMOTO, "Poly-Chromatic X-ray Stress Measurement", *J. Soc. Mater. Sci. Jap.* **26** (1977) 576 (in Japanese).
Described and tested experimentally an EDXRD system for stress measurements. See also [80].

80. M. NAGAO, S. KUSUMOTO and Y. ITO, "Non-Destructive Sub-Surface Distribution Measurement of Physical Quantities. An Application of Poly-Chromatic X-ray Techniques", *J. Soc. Mater. Sci. Jap.* **26** (1977) 18 (in Japanese).
Reported a study of the usefulness of the EDXRD system described in [79] by measuring the peak intensity distribution from the fatigue fracture surface and determining the plastic zone size, the real surface X-ray peak intensity, and the peak intensity gradient.

81. T. NAKAJIMA, T. FUKAMACHI, O. TERASAKI and S. HOSOYA, "Experimental Confirmation of the Isotopic Volume Effect in Superconducting Molybdenum by Means of Energy-Dispersive X-ray Diffraction at Low Temperatures", *J. Low Temp. Phys.* **27** (1977) 245.
Reported measurements carried out by the EDXRD system described in [69]. Differences in the lattice constants of Mo-100 and Mo-92 have been measured at temperatures of 290, 85.3 and 4.31 K. The isotopic volume effect was discussed thermodynamically and estimated from experimental results.

82. E. F. SKELTON, "A Simplified Calibration Procedure for Energy-Sensitive X-ray Detectors", *J. Appl. Cryst.* **10** (1977) 127.
Reported a rapid and safe method for the calibration of an energy-sensitive X-ray detector. The procedure requires only a single measurement of multiorder Bragg reflections and is limited in accuracy by the energy resolution of the detector.

83. R. UNO, A. AHTEE and T. PAAKKARI, "Determination of the Structure Factors of a LiF Powder Sample by the Energy Dispersive X-ray Diffraction", Rep. Ser. Phys., Univ. of Helsinki, No. 125 (1977).
Determined the structure factors of a LiF powder sample using EDXRD. The structure factors generally agree within the limit of 5% error with those obtained by conventional ADXRD.

84. A. H. VENKATESH and K. R. RAO, "A simple White Beam Neutron Diffraction Technique", *Pramāna* **8** (1977) 184.
Reviewed white beam neutron diffraction by the time-of-flight (TOF) technique. The EDXRD method was also mentioned. The principles of TOF and EDXRD methods are same.

1978

85. G. L. AYERS, T. C. HUANG and W. PARRISH, "High-Speed X-ray Analysis", *J. Appl. Cryst.* **11** (1978) 229.
Reported a profile-fitting method to analyse data, collected at speeds of up to 1° (2θ) per second with a computer-controlled diffractometer. The method was compared with EDXRD and with the use of a position-sensitive proportional-counter system.

86. J. BORDAS and J. T. RANDALL, "Small-Angle Scattering and Diffraction Experiments in Biology and Physics Employing Synchrotron Radiation and Energy-Dispersive Techniques", *J. Appl. Cryst.* **11** (1978) 434.
Reported small-angle scattering and diffraction experiments using the continuum of X-rays provided by synchrotron radiation and the energy dispersive technique. An analysis of the characteristics of the method, backed with experimental results obtained from fibres and proteins in solution, was performed.

87. J. BORDAS, J. WOODHEAD-GALLOWAY and D. W. L. HUKINS, "Energy Dispersive X-ray Diffraction by Collagen Fibrils in Costal Cartilage Using Synchrotron Radiation", *Bioc. Biob. R.* **84** (1978) 627.

Reported a low-angle X-ray diffraction study where synchrotron radiation and EDXRD techniques were used. From recorded diffraction patterns the isotropic distribution of collagen fibrils was calculated.
88. A. J. BOURDILLON, A. M. GLAZER, M. HIDAKA and J. BORDAS, "High-Resolution Energy-Dispersive Diffraction Using Synchrotron Radiation", *J. Appl. Cryst.* **11** (1978) 684.
Described a high-resolution EDXRD system, which uses a synchrotron source and a scanning channel-cut silicon crystal. The technique was demonstrated with a powdered sample of $BaTiO_3$.
89. B. BURAS, N. NIIMURA and J. STAUN OLSEN, "Optimum Resolution in X-ray Energy-Dispersive Diffractometry", *J. Appl. Cryst.* **11** (1978) 137.
Discussed the resolution problem of EDXRD. It was shown that for a given characteristic of the solid-state detector system and given range of interplanar spacings, an optimum scattering angle can be easily found for any divergence of the incident and scattered beams.
90. B. BURAS, J. STAUN OLSEN and L. GERWARD, "White Beam, X-ray, Energy-Dispersive Diffractometry Using Synchrotron radiation", *Nucl. Instr. Meth.* **152** (1978) 293.
Discussed special features of EDXRD, using synchrotron radiation, on the basis of experiments.
91. C. S.. G. COUSINS, L. GERWARD and J. STAUN OLSEN, "Multiple Diffraction in Crystals Studied by an X-ray Energy-Dispersive Method", *Phys. Stat. Sol. (a)* **48** (1978) 113.
Reported a study where the EDXRD method was developed for the study of multiple diffraction effects in single crystals. Experimental "Umweganregung" patterns were given for the 2 2 2 reflection in silicon, where as an example a six-beam case was observed.
92. T. EGAMI, "Structural Relaxation in Amorphous $Fe_{40}Ni_{40}P_{14}B_6$ Studied by Energy-Dispersive X-ray Diffraction", *J. Mater. Sci.* **13** (1978) 2587.
Reported a study of atomic structure and structural relaxation in amorphous $Fe_{40}Ni_{40}P_{14}B_6$ alloy using EDXRD. It was demonstrated that the structure of the amorphous alloy can be determined self-consistently with high accuracy by this method.
93. T. EGAMI and S. D. DAHLGREN, "Low-Field Magnetic Properties of Sputter Deposited Amorphous $Fe_{80}B_{20}$", *J. Appl. Phys.* **49** (1978) 1703.
Reported a study of amorphous $Fe_{80}B_{20}$ alloy obtained by sputtering onto a copper substrate cooled by liquid nitrogen. At room temperature the sample on the copper substrate was under tension because of the differential thermal expansion.
94. T. EGAMI and T. ICHIKAWA, "Kinetics of Structural Relaxation in Amorphous Alloy Observed by X-ray Diffraction", *Mater. Sci. Eng.* **32** (1978) 293.
Described a study of the kinetics of structural relaxation in amorphous $Fe_{40}Ni_{40}P_{14}B_6$.
95. T. FUKAMACHI, Y. NAKANO, S. HOSOYA and O. SHIMOMURA, "Applications of a Two-Circle SSD Diffractometer in the Angle-Dispersive Mode", *J. Appl. Cryst.* **11** (1978) 688.
Reported the construction of a standard two-circle diffractometer with Ge(Li) solid state detector controlled by a microcomputer. A single angle-dispersive scan with this system can give diffraction patterns with Kα, Kβ and fluorescence radiation simultaneously. Powder or amorphous samples of small volume can be more easily studied with this system: typical examples reported include crystalline InSb and amorphous GaSb, both in a diamond-anvil high-pressure cell.
96. M. GASGNIER and J. C. MALAURENT, "Etudes de Couches Minces d'Ytterbium par Diffraction X *in situ*", *J. Appl. Cryst.* **11** (1978) 141 (in French).
Reported an EDXRD analysis *in situ* performed on ytterbium metal thin films condensed on amorphous substrates. The coexistence of the hcp (2 H) and fcc (3 C) phases was studied in the temperature range 77 to 340 K. The deposits were made at different temperatures and annealed and quenched. Electron microscopy and diffraction observations were made on single crystals.
97. V. P. GIMANOV, D. A. GOGANOV, A. P. KOSYANKOV, A. A. VAZINA, A. M. MATYUSHIN, V. M. SHELESTOV and G. N. KULIPANOV, "Application of Transistor Spectrometer C3PM-1(M) in Small-Angle Diffractometry of Biological Objects", *Biofiz.* **23** (1978) 393 (in Russian).
Described the application of EDXRD to biological materials such as collagen and frog muscle. Synchrotron radiation of the storage ring VEPP-3 was used as a source of polychromatic radiation.
98. A. M. GLAZER, M. HIDAKA and J. BORDAS, "Energy-Dispersive Powder Profile Refinement using Synchrotron Radiation", *J. Appl. Cryst.* **11** (1978) 165.
Reported the results of powder-profile refinement using a solid state detector with synchrotron radiation. The rapidity and precision of the method shows that it will be possible in the near future to find structural parameters of a powdered material in few seconds, or minutes, thus allowing simultaneous structure refinement with changing sample environment.
99. A. M. GLAZER, M. HIDAKA, J. BORDAS, B. BURAS, J. STAUN OLSDEN and L. GERWARD, "Energy-Dispersive Methods with Synchrotron Radiation", *Acta Cryst.* **A34** (1978) S331.
Reviewed the use of solid state detectors with synchrotron radiation and discussed results obtained on the synchrotrons NINA and DESY and on the storage ring DORIS. Abstract only.
100. E. HINZE, G. WILL, B. BURAS, J. STAUN OLSEN and L. GERWARD, "Energy Dispersion as a Method for X-ray Diffraction under High Pressure in a Diamond Anvil Cell", *Acta Cryst.* **A34** (1978) S343.
Reported *in situ* experiments under high pressure using the EDXRD method. Abstract only.
101. R. JENKINS, "Interdependence of X-ray Diffraction and X-ray Fluorescence Data", *Adv. X-ray Anal.* **21** (1978) 7.
Discussed various X-ray methods for phase analysis, the quality of diffraction data and use of elemental information. A combined X-ray diffraction and X-ray fluorescence system for routine phase analysis was also

described.
102. L. KAIHOLA, E. LAINE and E. TIILIKKA, "Observation of X-ray Interferences on Thin Films by Energy Dispersive Method", *Phys. Scripta* **18** (1978) 7.
Reported on the possibility of using conventional X-ray apparatus with an energy dispersive solid state detector to study the interference of continuous X-ray spectra on thin copper films. X-ray interference obtained by this method was compared with angular resolved interference.
103. S. KOCH and P. JUGELT, "Einsatzmöglichkeiten der energiedispersiven Pulverdiffractometrie zur Phasenanalyse metallurgisher und geologisher Proben", *Isotopenpraxis* **14** (1978) 261 (in German).
Reported a study where the EDXRD method was used for determination of α- and γ-phases in steel. Also the applicability of the EDXRD method to geological samples and glass was studied.
104. E. S. U. LAINE, "A High-Speed Determination of the Volume Fraction of Ferrite in Austenitic Stainless Steel by EDXRD", *J. Phys. F* **8** (1978) 1343.
Reported a study in which the EDXRD method was applied to the determination of the ferrite content in austenitic stainless steel. The method was adapted for measurements of thermal expansion and phase fractions of austenitic stainless steel at temperatures from 17 to 700° C.
105. E. LAINE, I. LÄHTEENMÄKI and I. LEHTORANTA, "An X-ray Study of Splat-Quenched Sn–Bi and Cu–Sb Alloys by Energy-Dispersive Diffraction", *J. Mater. Sci.* **13** (1978) 108.
Reported a study where the solubility limit produced by splat quenching was shifted in Sb–Bi alloys and decomposition of the supersaturated α-phase was investigated in the temperature range −40 to 0° C. Splat-quenched Cu–Sb alloys with different compositions were also studied.
106. M.-B. LIU, D. M. GRUEN, A. R. KRAUSS, A. H. REIS Jr and S. W. PETERSON, "Ion Nitriding of Titanium and Zirconium by a d.c.-Glow Discharge Method", *High Temp. Sci.* **10** (1978) 53.
Reported a study of nitrided surfaces of titanium and zirconium. The golden-yellow nitrided surfaces were explored by EDXRD and electron diffraction techniques. The kinetics of the formation of surface nitride layers was discussed in terms of a reaction between nitrogen–hydrogen molecular ions and the substrate.
107. J. SCHELTEN and R. W. HENDRICKS, "Recent Developments in X-ray and Neutron Small-Angle Scattering Instrumentation and Data Analysis", *J. Appl. Cryst.* **11** (1978) 297.
Reviewed the developments in instrumentation and data analysis in general that have occurred in the field of small angle X-ray and neutron scattering since 1973.
108. J. STAUN OLSEN, B. BURAS, T. JENSEN, O. ALSTRUP, L. GERWARD and B. SELSMARK, "Influence of Polarization of the Incident Beam on Integrated Intensities in X-ray Energy-Dispersive Diffractometry", *Acta Cryst.* **A34** (1978) 84.
Reported the influence of polarization on the integrated intensities. This was considerable at high energies and at a scattering angle of around 90°. At energies below one half of the high-energy limit, the influence of polarization was negligible for all scattering angles. For a scattering angle equal to 20°, the influence of polarization is less than 2.5% for the powder over the whole energy range.
109. Y. MURATA and K. NISHIKAWA, "The Construction of an Energy-Dispersive X-ray Diffractometer for Liquids and Its Application to CCl_4", *Bull. Chem. Soc. Jap.* **51** (1978) 411.
Described an EDXRD system for determination of liquid structures, and the procedure for data analysis to obtain the results for liquid CCl_4. It was observed that liquid CCl_4 has a long-range correlation at room temperature.
110. A. NAKAUE, "Studies on the Pressure-Temperature Phase Diagram of Nd, Sm, Gd and Dy", *J. Less-Common Met.* **60** (1978) 47.
Reported a study of the pressure–temperature phase diagrams of Nd, Sm, Gd and Dy by EDXRD at pressures up to 90 kbar and at temperatures up to 700° C, using a cubic anvil-type press. The lattice parameters of Nd, Sm and Dy were also measured by EDXRD at high pressures and at 300° C.
111. J. SZPUNAR and L. GERWARD, "Texture Measurements Using the Energy Dispersive X-ray Diffraction Method", *Acta Cryst.* **A34** (1978) S385.
Reported the application of the EDXRD method to simultaneous measurements of several pole figures in rolled duplex alpha–beta brass. Abstract only.
112. R. UNO and J. ISHIGAKI, "Determination of Structure Factors of Powder Samples by the Energy Dispersive X-ray Diffraction", *Acta Cryst.* **A34** (1978) S335.
Reported measurements of the intensity spectrum of the incident beam for determination of structure factors. Calculations were made for GaP and GaSb. See also [55]. Abstract only.

Author Index 1968–78