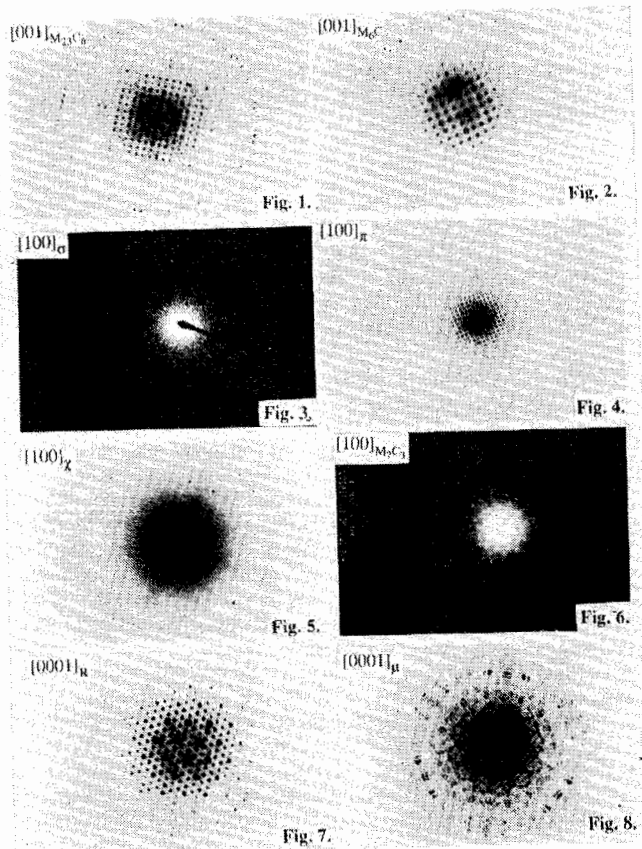displacements between ZOLZ and FOLZ for σ-phase and π-phase. Morever, the radii of the FOLZ rings in Figs 3, 4 and 5 reveal that c=4.54 Å and a=6.47 Å and a=8.8037 Å for σ-phase, π-phase and χ-phase respectively.

M_7C_3 has the space group Pnma with a= 4.526 Å, b=7.010 Å and c= 12.142 Å. Its pseudo-hexagonal [100] axis pattern in Fig.6 shows similar spacings in comparison with the pattern from the [0001] zone of R-phase (R$\bar{3}$, a=10.903 Å and c=19.342 Å) in Fig.7. However, the radii of the FOLZ rings are very different, which give a=4.526 Å for M_7C_3 and c=19.345 Å for R-phase respectively.

Because of the very long c-axis of μ-phase (R$\bar{3}$m, a=4.754 Å and c=25.71 Å) the pattern of [0001] has a distinctive feature: small radius for the FOLZ ring and several higher order Laue zones may appear (Fig.8) .

Figs.1-8. CBED patterns. **Figs.1-5.** The [001] zone axes of $M_{23}C_6$, M_6C, σ-, π- and χ-phase respectively. **Figs.6-7.** The [100] and [0001] zone axes of M_7C_3 and R-phase respectively. **Fig.8.** The [0001] zone axis of μ-phase.

[001]$_{M_{23}C_6}$ [001]$_{M_6C}$ Fig. 1.   Fig. 2.

[100]$_\sigma$ [100]$_\pi$ Fig. 3.   Fig. 4.

[100]$_\chi$ [100]$_{M_7C_3}$ Fig. 5.   Fig. 6.

[0001]$_R$ [0001]$_\mu$ Fig. 7.   Fig. 8.

PS-20.01.10 STUDY OF Z PHASE IN MB25 Mg ALLOY. By Z.P. Luo* and S.Q. Zhang, Institue of Aeronautical Materials, Beijing 10095, P.R. of China.

The microstructures of Mg-Zn-Y system alloys were studied by Padezhnova et al. (1982). By means of x-ray diffraction (XRD) analysis, a Z phase was identified in this system alloys, but its crystallographic structure was not determined out. The present paper gives the results of study on the structure of the so-called Z phase in literature.

The experimental material is as-cast MB25 alloy with compositions of 5.56~5.78 wt-%Zn, 0.47~0.6 wt-%Zr, 0.89~1.72 wt-%Y and balanced Mg. By means of
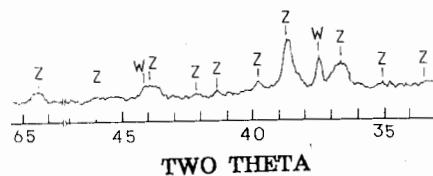
electrolysis (Zhang, 1990), the extracted powder is obtained from the alloy and examined by XRD. The figure below shows the XRD pattern which contains the so-called W and Z phase (Padezhnova et al., 1982) peaks. However, detailed examinations by conventional selected area diffraction (SAD), convergent beam diffraction (CBD) and energy dispersive spectrum (EDS) analysis show that the icosahedral quasicrystal (QC), MgZn$_2$-type Laves phase, W phase and an ordered structure of W phase (W' phase) are present. It is clearly that the distinct XRD peaks from the Z phase in the XRD pattern are in fact coincident with those peaks from the icosahedral QC and MgZn$_2$-type Laves phase, as listed in the table below. The XRD peaks from the QC in rapidly solidified Mg-Al-Zn alloy (Rajasekhara, 1986) are given out for comparison. It is necessary to point out that the QC in the Mg-Zn-Y system has never been reported before.

Table Indexing of the XRD Peaks from the Z Phase

Exper.		indexing		Z phase		QC–MgAlZn	
d(nm)	I/I$_o$	QC	Laves	d(nm)	I/I$_o$	d(nm)	I/I$_o$
0.2455	53	(100000)		0.245	50	0.2423	51
0.2328	100	(110000)		0.234	100	0.2292	100
0.2267	24		(021)	0.225	12		
0.2182	13		(004)	0.217	7		
0.2061	32	(111101)		0.206	33	0.2032	23
0.198	13		(113)	0.1985	16		
0.1449	21	(101000)		0.1442	30	0.1428	19

Padezhnova E M et al. (1979). Akademiia Nauk SSSR, Izvestiia, Metally, No.4, 204–208 (in Russian)
Zhang S Q (1990). Acta Metall. Sinica, **3**, 110–115
Rajasekharan T et al. (1986), Nature, **322**, 528–530

TWO THETA

PS-20.01.11 THE STRUCTURAL REARRANGEMENT ASSOCIATED WITH LITHIUM INSERTION INTO V6O13. By C. Lampe-Önnerud * and J.O. Thomas, Uppsala University, Institute of Chemistry, Box 531, S-751 21 Uppsala, Sweden.

In spite of the fact that V6O13 is one of the most common cathode materials used in modern battery design, the structural mechanisms of lithium insertion are still not properly understood. Conventional wisdom says that phases LixV6O13, where x=1, 4 and 8, are created successively on lithium insertion. It is known, however, that difficulties can arise with respect to battery reliability and reproducibility. These may well result from difficulties incurred in obtaining

phase-pure V6O13. Our efforts here focus specifically on this problem, and subsequently on achieving controlled *chemical* lithium insertion. Phase-pure V6O13 has also been incorporated into thin-film polymer batteries, which were then cycled to facilitate *in situ* X-ray diffraction studies (transmission mode) of the corresponding *electrochemical* lithium insertion process. Structural rearrangements involved in both insertion processes are analyzed carefully and compared; observations are also made regarding the reversibility of these processes.

20.02 – Multiphase Analysis

DS–20.02.01 RECENT DEVELOPMENTS IN THE COMPUTER SEARCH/MATCHING OF MULTI-PHASE MATERIALS. By R. Jenkins, International Centre for Diffraction Data, Swarthmore, PA, U.S.A.

While most contemporary methods of qualitative analysis of multi-phase materials are still based on the classic Search/Match/Identify process developed by Hanawalt, Rinn and Frevel in the 1930's, during the past 10 years or so the Personal Computer has made a dramatic impact on the *ways* in which classical procedures are being implemented.

Modern search/matching procedures can conveniently be broken down into three categories - Manual Methods, Computer-Aided Methods, and Fully Automated Methods. Even where purely manual search/match methods are used, the computer based diffractometer systems still give the potential benefit of high quality d/I data, especially where automated angular and intensity calibration is employed. In the case of computer-aided and fully automated techniques, the additional use of newer data processing techniques including profile fitting, α_2 stripping, etc., have all conspired to provide the diffractionist with accurate, artifact-free data, with which to start the search/ matching process. Finally, the ability to search index files containing physical and experimental data, beyond the traditional d/I list, and the availability of comprehensive sub-file selections, has resulted in robust boolean search algorithms, which give the user great flexibility in setting up search strategies.

As to the implementation of the search/match approach, the use of fast, modern PC's, with integrated CD-ROM storage and powerful windows-based operating systems, has allowed the software developer a degree of flexibility, undreamed of a decade ago.

This paper will discuss recent progress in the use of some of these personal-computer based, data collection, processing and analysis programs, with special emphasis on their use in the analysis of multi-phase materials.

DS–20.02.02   Search / Match Using Full Trace Scans: A Strong Improvement over Using d/I Files

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A table of d/I values in many cases represents, quite adequately, the ideal powder diffraction pattern of a single phase reference material. However, reducing the diffraction pattern of a complex mixture to such a list is more difficult and more questionable for several reasons:

- Determining "real" peaks through any "peak search" algorithm or "profile fitting" technique becomes practically impossible for weak lines of the measured diffractogram. This in turn, can preclude the identification of minor phases.

- Clusters of lines are most likely to be impossible to decompose due to (an inevitable) lack of information on the number, positions, and widths of non-resolved lines.

- The information on line-widths is lost - whereas in fact this is also an attribute of the respective phase.

The DIFFRAC AT search/match uses the full trace scan with subtracted background. Therefore it takes full advantage of all available information - particularly very weak lines, line clusters and the line widths.

Examples will be considered to demonstrate how important these features are for successful and reliable phase identifications.

Compared to traditional d/I tables, the DIFFRAC AT search/match uses much more input data which could be expected to have a severe penalty in speed of the search/match. In fact, due to very highly optimized code, a search employing the current whole ICDD data base (sets 1 - 42: 61,993 reference patterns) takes less than 15 seconds when performed on a i486/50-based PC.

DS–20.02.03 QUANTITATIVE POWDER DIFFRACTION PHASE ANALYSIS - RECENT DEVELOPMENT AND CURRENT STATUS. By J. Fiala[1] and H. Toraya[2], [1]Central Research Institute SKODA, Czech Republic, [2]Ceramics Research Laboratory, Nagoya Institute of Technology, Japan.

Methods for quantitative phase analysis (QPA) by using X-ray powder diffraction technique are reviewed, and their precision and accuracy are discussed in this paper. A development of experimental design shifted the analysis strategy from few-line measurement to many-lines and even to whole-pattern measurements. Preparation techniques, which can suppress detrimental effects of texture, particle size, inhomogeneity and amorphization, have been developed for powder samples while a serious problem remains for bulk samples. Standard reference materials of known phase composition have been produced. Chemical constraints are used to help with phase abundance analysis. Large scale systematic measurements were undertaken (inter-laboratory tests, reference intensity ratios determinations), and they form a necessary base for a quantitative assessment of the reliability of QPA. The main problem of QPA, which consists in the fact that diffraction intensities are influenced not only by phase composition of the mixture under analysis but also by real structure (and variable crystal structure) of its individual components and of polycrystalline aggregate as a whole, is addressed by simultaneous determination of phase composition, real structure and crystal structure of all constituents. However, correlations existing between the individual parameters enhance uncertainty of their estimates obtained by such a multivariate refinement. Factorial analysis of diffraction patterns of different fractions from the mixture analyzed provides intensity factors which are automatically corrected for the influence of real structure. Some forty years ago, accuracy of the QPA was estimated to (1 - 10)% absolute. The present day rather optimistic estimate, 2% absolute, seems to be limited to favorable cases of powder mixtures of phases, in which the real structure does not affect their diffraction appreciably.

DS–20.02.04 QUANTITATIVE ANALYSIS OF MULTIPHASE MATERIALS USING THE RIETVELD METHOD. By Julius Schneider, Institut für Kristallographie, Universität München, Theresienstraße 41, D-8000 München 2, Germany.

Use of the Rietveld method in quantitative analysis of multiphase mixtures has recently found increasing attention. As compared to conventional discrete-peak methods the consideration of the entire