

Laboratory and computing methods

MICROCOMPUTER SYSTEM FOR QUALITATIVE AND QUANTITATIVE POWDER DIFFRACTION PHASE ANALYSIS

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An efficient identification system RIFRAN 89 for rapid qualitative and quantitative diffraction phase analysis of polycrystalline materials was developed on the basis of studies [1—4] published earlier. The system is represented by a set of interactive computer programs written in the TURBOBASIC language, and implemented on the 8-bit ATARI 130 XE home computer.

The identifying procedure for the analysis is based on comparing the diffractograms of standards with those of the unknown sample, utilizing statistical criteria and chemical analysis for qualitative analysis, and the method of mathematical optimizing for quantitative analysis.

INTRODUCTION

Determination of phase composition on the basis of diffractograms of the analyzed substance by comparing with a reference data base of standard diffractograms is generally a complex matter, particularly owing to the large number of standards involved. A highly efficient searching in extensive collections of reference diffractograms is obviously ensured by computers. In connection with the recent expansion in the field of efficient and readily controllable computer technology one can also see its rapidly increasing utilization in phase analysis. Among the factors supporting this trend there is above all the availability of relatively cheap and well controllable microcomputers with comparatively extensive operating and capacity capabilities. The precision and completeness of the analyses then depends only on the strategy and algorithms of the programs chosen, as well as on the technical means available. The respective procedures have already been repeatedly published for large computers, minicomputers, as well as microcomputers, e.g. [8—14]. In this country, these efforts are represented by identification systems [4, 15] for a "large" computer and by [1—3] as a variant for portable programmable calculators.

The RIFRAN 89 identification system described in the present paper was implemented on the 8-bit ATARI 130 XE home computer, furnished with the ATARI 1050 disk drive. The software of the system is written in the TURBOBASIC XL language, which is adequately rapid for the operations required. In such an arrangement, there is 34 kB of main memory jointly with 64 kB of the

RAM disk for additional data files. Each floppy disk provides 256 kB for arbitrary files.

In view of the relatively large scope of the complete identification system, it has been segmented into individual programs that are called from the program run, similarly to the data files. In spite of this the system is flexible and comparatively fast.

QUALITATIVE ANALYSIS

The method employed

The qualitative analysis by the RIFRAN 89 system is based on comparing the patterns of the reference database one after the other with a diffractogram of the sample being measured. As the high operating speed of the computers allows the diffractograms to be compared in great detail, all of the peaks are being compared, and not only some of the most intensive ones, as is the case of most "manual" identification systems. The whole philosophy of the search has been described in detail, e.g. in [16], so that only brief information is given here.

The basic idea is to represent the sample being measured, as well as the standards, by means of factors. To determine the phase composition of an unknown substance one has to express its diffractogram \mathbf{x} by superposition of diffractograms y_1, y_2, \dots, y_k of known substances, standards. This means to find non-negative numbers c_1, c_2, \dots, c_k for which

$$\sum_{\gamma=1}^k c_{\gamma} y_{\gamma} = 1 \tag{1}$$

and which conform, as best as possible, to the equation

$$\mathbf{x} = \sum_{\gamma=1}^k c_{\gamma} \mathbf{Y}_{\gamma} \tag{2}$$

thus minimizing the residuum

$$\begin{aligned} f(c_1, c_2, \dots, c_k) = & \left| \mathbf{x} - \sum_{\gamma=1}^k c_{\gamma} \mathbf{Y}_{\gamma} \right|^2 = \left| \mathbf{x} \right|^2 - 2 \sum_{\gamma=1}^k c_{\gamma} \mathbf{x} \mathbf{Y}_{\gamma} + \\ & + \sum_{\gamma=1}^k \sum_{m=1}^k c_{\gamma} c_m \mathbf{Y}_{\gamma} \mathbf{Y}_m. \end{aligned} \tag{3}$$

From the stationarity condition

$$\frac{\partial f}{\partial c_1} = \frac{\partial f}{\partial c_2} = \dots = \frac{\partial f}{\partial c_k} = 0$$

one obtains the fundamental system of equations

$$\begin{aligned} \mathbf{x} \mathbf{Y}_1 &= c_1 \mathbf{Y}_1 \mathbf{Y}_1 + c_2 \mathbf{Y}_2 \mathbf{Y}_1 + \dots + c_k \mathbf{Y}_k \mathbf{Y}_1 \\ \mathbf{x} \mathbf{Y}_2 &= c_1 \mathbf{Y}_1 \mathbf{Y}_2 + c_2 \mathbf{Y}_2 \mathbf{Y}_2 + \dots + c_k \mathbf{Y}_k \mathbf{Y}_2 \\ \mathbf{x} \mathbf{Y}_k &= c_1 \mathbf{Y}_1 \mathbf{Y}_k + c_2 \mathbf{Y}_2 \mathbf{Y}_k + \dots + c_k \mathbf{Y}_k \mathbf{Y}_k \end{aligned} \tag{4}$$

by means of which the phase composition is determined. In these expressions, the symbol

$$\mathbf{xy} = \sum_{n=1}^n x_n y_n \quad (5)$$

designates the scalar product of the pattern vectors

$$\mathbf{x} = \{x_1, x_2, \dots, x_n\} \text{ and } \mathbf{y} = \{y_1, y_2, \dots, y_n\}$$

and the standard of the pattern vector

$$|\mathbf{x}| = \sqrt{\mathbf{xx}} = \sqrt{\sum_{n=1}^n x_n^2} \quad (6)$$

The number k is the number of phases which must be necessarily taken into account in the identification. The number is mostly very large and the solution of the system (2) is unstable. Identification is therefore affected by firstly narrowing the complete set of standards to smaller sets of k substances, whose diffractograms are as similar as possible to that of the substance being measured and only then the optimizing program is carried out for the substances selected.

Standard database

The database of standard diffraction patterns for qualitative analysis is segmented on floppy disks into several files according to the standard types and the user's requirements. One set is able to accommodate about 250 to 300 standards in view of the operational memory capacity of the microcomputer employed. The individual standards are described by the following identifiers:

- MINERAL** — specifies the mineralogical or chemical name of the substance jointly with the quality of the data
- SYSTEM** — crystallographic system of the standard
- FORMULA** — chemical formula of the substance
- CODE** — as the database of standards for qualitative analysis is composed of data taken over from various sources, use was made either of existing identification codes, or those were determined so as to be useful for seeking the original data in the literature, e.g.
 A 25983 (ASTM 25983) [17]
 B 71.80 (Brindley and Brown, page 71, 1980) [18]
 M 310 (Micheyev, table No. 310) [19] and the like.

The standard is further described by four characteristic peaks, according to which it is classified into a narrowed set of potential candidates for identification. The data on the standard is concluded by the other values of peak positions and their relative intensities. Algorithm and program description.

The program menu offers three possibilities: Preparation and editing of the standard database, identification proper and database surveying. Following the selection, the respective program is introduced into the main memory from the floppy disk.

Working and editing the standard database

For data preparation, the file is opened by an arbitrary name under which it will henceforth exist. Then follows setting of input values of wavelengths, identifiers, the characteristic and all other peaks in the form of d -values and their relative

intensities. All the data can be edited, and after checking the file is stored in the memory. The program is returned into the menu and the cycle is repeated. The setting of the new standards is concluded by closing the respective file.

On recalling the file content from the menu, the program will display the contents of the chosen file in the form of mineralogical names and identifying code in the sequence recorded. This serves above all for checking the file and its supplementing.

After the check, the respective file is copied from the memory under the same name onto the floppy disk at a command from the menu.

Search of the standard database

Having loaded the program in the main memory of the microcomputer, the input data for analysis are set: wavelength of radiation employed, the name of the sample being measured using at the most 32 ASCII characters, and selection of the data setting either as values "d" or as " 2θ ". Further setting is carried out on the basis of selection into the narrow file, i.e. the number of characteristic peaks of the four possible ones being in agreement. Next comes the definition range of Bragg angles 2θ , i.e. the experimental window, in which the analysis is to be performed. The usual range set is 10 to 90 degrees 2θ .

Last parameter to be set is a list of at the most 10 chemical elements, which are not contained in the sample being studied, in the form chemical formulas, separated by a dash and terminated with zero.

Then follows the setting of the position values of the peaks being observed, as in this case the search is based solely on comparing the peak positions and on criterial tests. The setting is terminated by setting the zero values.

Having selected the chosen file of standards and recording them into the main memory, one sets the tolerance interval to which a given diffraction of the substance being measured the respective standard is attributed. One has to take into account the measure of inaccuracy of its determination, called the error window, i.e. around each diffraction of the specimen the tolerance interval having the size $d \pm \text{WINDOW} \cdot 10^{-3} d^2$ is constructed [21].

Agreement of one to four peaks (characteristic ones) according to the user's choice is the basic criterion according to which the standard is classified into the narrow file. The method has proved successful in previous versions of the RIFRAN system [1—3] and is fully satisfactory for this selection. After that the "negative" chemical elements set [8] are compared with the FORMULA identifier of each standard. Having established agreement between the negative elements and standards, a standard is not immediately removed from further analysis, but is no longer subjected to additional criterial analysis, and in place of its results a warning of chemical incorrectness of the standard found is displayed. The standards with correct chemical composition proceed to the subsequent test. This makes use of two comparing criteria, as already described [2, 3], namely the ratio of the number of identical peaks in the sample being measured and in the respective standard, to the total possible number of peaks in the standard in the given experimental window, and as the other criterion, the ratio of the sum of intensities of adjoined peaks in the standard to the total sum of intensities of the standard in a given experimental window. A synthetic criterion is calculated from the two criteria by their mutual multiplication, and the standards are sorted according to the decreasing value of the product.

Having created a new set of potential candidates, the program offers the possibility of providing the results in either graphical or tabular form. In view of the relatively limited displaying capabilities of the ATARI microcomputer (320 × 192 dots) one screen allows three standards to be displayed jointly with the diffractograms of the sample being measured in the form of line patterns, placed one above the other including their description. The graphical output allows arbitrary standards to be displayed in either decadic or logarithmic scale according to the user's choice in the selected experimental window of angles 2θ .

The output of results in tabular form can be displayed on the screen, or printed by the attached matrix printer. In the table, the sorted standards are printed in descending sequence jointly with the results of criterial tests. In the case of chemical incorrectness, a warning of chemical disagreement is printed in place of the results of the criterial test. The warnings are placed at the end of the list.

As soon as the listing of results is concluded, the table of statistical assignment of the individual diffractions is printed. The first column contains the values of all the set positions of peaks for the sample being measured, the next column states the number of assignments to the respective diffraction, and the following column lists the standard to which the diffraction has been assigned.

This output is of considerable significance, as it points out completeness of the analysis and the possible disassignment of some peaks, e.g. because the file employed does not contain an appropriate standard, or because the WINDOW parameter has been unsuitably employed, or because the substance in question shows a high degree of non-stoichiometry, in the extreme case indicating a maladjusted goniometer, etc.

Following this output, the analysis can proceed by using the next file of standards until the cycle is concluded.

It should be borne in mind, however, that no program of this type, including the RIFRAN 89 system, is absolute and capable of ensuring analyses without exception. Man must always have the last word, so that the results have to be checked manually on the basis of the listing obtained. To avoid tedious looking up of the individual cards or data of standard diffractograms from various resources, the RIFRAN 89 system offers the possibility of scanning the database within the framework of the master menu.

Scanning the database of standards

After soliciting the name of the file which is to be surveyed, the program will request the keyword (use can also be made of any arbitrary numerical value, which makes it possible e.g. to scan cards in which the peak being seeked is contained), or even only its fragments, on the basis of which the screen will display the complete file with all identifiers. Each additional card that has the same keyword will appear after next depression of the key.

Having concluded the scanning, it is possible to set a new keyword and to recheck, or return to the master menu at any moment. This concludes the entire cycle of qualitative analysis.

The program works in a dialogue mode and even untrained operators are capable of using it after a short period of time on the basis of information provided by the display.

The counting of one file containing about 100 standards and selection according to characteristic diffractions takes about 40 seconds, and comparison for one

selected standard takes about 5 seconds. The entire analysis takes about 2 to 3 minutes and the whole cycle including all inputs and outputs takes no longer than 10 minutes.

The entire cycle of qualitative analysis is immeasurably faster and more comfortable than the previous versions of the RIFRAN system [1—3].

These version have been restricted above all by the use of EMG 666/B calculator which did not allow for direct displaying on the graphical unit, and by the minimum capacity of the main storage [8 kB]. In contrast to this, the RIFRAN 89 version being described here makes use of a superior programming language, allows the results and intermediate results to be checked instantaneously on the monitor, and provides their graphic displaying. The much higher capacity of the main storage, moreover in connection with the floppy disk unit, permitted the evaluation criteria to be expanded significantly, including the statistics of assigning the individual peak, scanning of the standard database, flexible supplementing of the database, etc. In addition to this, the microcomputer employed is much more commonly used than the EMG 666 calculator and in addition to this, following a minor adjustment, the program can be transcribed into the software of other related microcomputers; such transcription of the the EMG instruction code was very difficult.

QUANTITATIVE ANALYSIS

A part of the RIFRAN 89 system, designed for quantitative phase analysis under the name MICRO Q, is a mildly modified and into TURBO-BASIC transcribed version of the KVAN program of the XQPA system [4]; the adjustments were made to meet the requirements of the system. As the method has already been described, e.g. [4, 20, 21, 22], we shall deal only with the basic principles.

The method employed

The basic idea is again to represent the sample being measured, and the respective calculated standards, as vectors. The phase analysis has the purpose to find non-negative numbers c_1, c_2, \dots, c_k , that minimize the modulus of the residual intensity vector according to equation (3). Standardization of c_i yields the numbers v_i , representing optimum estimates of the volume shares of standards y_i in the sample being measured.

Equation (3) represents the fact that agreement between a diffraction pattern measured and the suggested model of a mixture phases is never perfect owing to the real structure of the sample and to experimental errors. The least square method is used in seeking a model most similar to the diffraction pattern being studied. On substituting the vectors x or y_i with their coordinates on the assumption that only the j -th peak of i -th standard contributes to the j -th peak of sample x_j (i.e., the peaks of different phases do not show superposition), equation (3) will ideally acquire the form

$$x_j = c_i \cdot y_{ij} \quad (7)$$

and represents the basic equation of the quantitative phase analysis as a modification of the basic relationship suggested by Klug and Alexander [23].

Direct solution of the problem of minimizing the residuum is numerically unviable owing to the large number of standards in the file, and in addition to this,

it is impracticable for a sample to contain more than 10 identifiable phases. This is why the file of standards has to be narrowed on in the first step of the analysis. Generally, this is effected on the basis of the chemical characteristics and by comparing the interplanar distances and diffraction intensities of the standard with those of the peak intensities of the standard and the sample being measured. The reduction of the set of standards to a narrower set is achieved by means of tests using optional parameters P1 through P8, where the individual parameters represent the following:

- P 1 — the standard is rejected if it contains less than P 1 peaks.
- P 2 — the standard is rejected if it contains less than P 2 percent of peaks.
- P 3 — the standard is rejected if the sought estimate of representation in percent in the mixture is less than P 3 percent.
- P 4 — the so-called error window, representing the tolerance interval around each sample peak, as mentioned above.
- P 5 — among the peaks of the standard, only those with calculated intensity higher than P 5 are taken into account.
- P 6 — the top unit of the range of interplanar distances d for the sample being measured.
- P 7 — the bottom limit of the range of interplanar distance d in the sample being measured.
- P 8 — approves or rejects the preselected standards.

In the case of RIFRAN 89 system, the set is narrowed down in its part QUALITY and in quantitative analysis, use is made of standards set from outside by the user on the basis of prior experience (parameter P 8). Experience has shown that the procedure makes this analysis substantially faster, more accurate and effective.

After that, the criterial function of the measure of probability that the standard is contained in the sample is calculated for the given standard:

$$R = \frac{100 m}{M} \left[1 - \frac{1}{m} \sum_{k=1}^m \left| \frac{1}{d_{st, k}} - \frac{1}{d_{s, k}} \right| \right] \cdot \left[1 - \frac{\sum_{k=1}^m \max(0, I_{st, k} - I_{s, k})}{\sum_{k=1}^m I_{st, k}} \right] \quad (8)$$

where m is the number of peaks of the sample, M is the total number of peaks of the standards within the limits P 6 to P 7, d_{st} , I_{st} are interplanar distances, or intensities of diffractions of the standard situated over the interval expressed by parameter P 4, d_{vz} , I_{vz} are interplanar distances or peak intensities of the sample.

The narrower set of standards enters the optimizing process proceeding according to equation (3). As the vectors y_i are independent, the necessary and satisfactory condition for an absolute minimum of equation (3) is the validity of the system of equations

$$\frac{\partial f}{\partial c_i} = 0 \quad i = 1, 2, \dots, n \quad (n \leq 10) \quad (9)$$

so that

$$\sum_{j=1}^n c_j(\mathbf{Y}_i, \mathbf{Y}_j) = (\mathbf{x}, y_i) \quad i = 1, 2, \dots, n. \quad (10)$$

There is always a solution c_1, c_2, \dots, c_n , and the solution is unambiguous.

As a result of the real structure, unsuitable selection of parameters or incorrect scale of experimental data it may happen that some c_i will attain negative values, which is physically impermissible. This effect is synonymous with that when the user tries to substitute additional standards into an "occupied" diffraction pattern of the sample being measured. Practice shows that standards, for which the volume share is negative, do not occur in the sample. An algorithm of the analysis then uses the method of dimensional reduction: if $c_i \leq 0$, i -th line and the column in the vector on the right-hand side of the i -th coordinate is eliminated from the matrix on the left-hand side of the system of equations (10). The matrix reduced in this way reenters the solution of the system of equations (10) and the procedure is repeated until all $c_i > 0$. Standardization then yields the resultant values of volume shares of the individual phases in the sample being measured given in percent by volume.

The residuum in the form

$$\text{Res} = \sqrt{\left| \frac{f(c_1, c_2, \dots, c_x)}{f(0, \dots, 0)} \right|} \cdot 100 \quad (11)$$

serves as an estimate of the analytical correctness.

Database of standards

Quantitative analysis requires the peak intensities of the standards to be classified in an absolute scale, common for all standards, as a relative scale would be useless. These absolute intensities are associated solely with the crystal structure of the standard and with the X-radiation employed for the diffraction of the i -th phase

$$I_{i, hkl}^{abs} = |F_{i, hkl}|^2 m_{i, hkl} LP_{i, hkl} \cdot \frac{1}{V_i^2} \quad (12)$$

where the structural factor has the form

$$F_{hkl} = \sum_{p=1}^r f_p \exp\left(-B_p \frac{\sin \Theta_{hkl}}{\lambda}\right) \cdot \exp[-2\pi i(hx_p + ky_p + lz_p)] \quad (13)$$

- where f_p is the atom form factor of the p -th atom of the elementary cell,
 B_p is the isotropic temperature factor of the p -th atom in the elementary cell,
 Θ_{hkl} is a Bragg angle of diffraction with indices hkl
 λ is the wavelength of the radiation employed,
 x_p, y_p, z_p are the coordinates of the p -th atom of the elementary unit,
 m_{hkl} is the multiplicity of diffraction hkl
 LP_{hkl} is Lorentz polarization factor of diffraction hkl
 V is the volume of the elementary cell.

This form is intended for Bragg-Brentan's parafocusing arrangement of the goniometer for a "thick" sample, i.e. one having a thickness of at least 1 mm. It is

further assumed that the sample has been prepared so as to allow the effects of microabsorption, thermal diffusion scatter on the diffraction intensity, the unevenness of the sample surface, and primary extinction to be neglected. Additional corrective terms have to be introduced into equation [12] if the requirements cannot be met.

The database of the standards for the MIKRO Q program is stored on a floppy disk. The table of a standard contains its standard, by means of which the respective standards are called, the name of the standard, the number of diffractions and the values of their positions in the form of d values, and their absolute intensities "Iabs".

The standards were obtained in various ways. By calculations [24] using the programs LAZY PULVERIX [6] or DIFK [5]. Additional standards were obtained from database [17] which already specifies the scaling factor related to corundum, by means of which substances with intensities in the absolute scale were obtained. The last method of determining standard diffraction patterns was their direct measurement [7].

The respective files were used independently, although they can be compiled for mutual rescaling.

The algorithm and description of the program

After keying in the program designation the selected file of standards is transferred into the microcomputer memory. The criteria P1 through P7 are set as already described, followed by the sample name and the values of "d" and "Iabs" measured, in the most 51 values. Having checked the data, the numbers of standards considered for calculation are set, at the most, 10. The data setting is followed by summing, calculation of the matrix and parallel listing of the results of criterial analysis. Having calculated the share by volume of the individual standards in the sample being measured, the program displays a table containing the numbers of standards, their names, the percent in volume calculated, and the standard deviations. After the first approximation and automatic rejection of unsuitable standards, there follows another cycle, after which the calculation is usually concluded.

It has been repeatedly proved [4, 20] that the method exhibits a high reliability and accuracy. Compared to the corresponding versions in the FORTRAN language [4], the present version is somewhat slower but nevertheless fast enough. The overall time of analysis, including the setting of input data, takes at the most 10 minutes, including the printing of results on the matrix printer connected.

DISCUSSION

Both the qualitative and the quantitative parts of the RIFRAN 89 system are utilized independently, being not organically interconnected through the database of standards. This is given by the incompatibility of the standard databases. Those for the quantitative analysis can be employed in qualitative analysis, but the number and choice of standards stored in the files is so far unsatisfactory. This is the only reason why two databases are used. Direct application of standards is a difficult problem above all for reasons of a lack of precise data on the crystal structure of a given substance; on the other hand, perfect specimens

of the mineral suitable for measuring a standard diffraction pattern are not always available. A comparatively advantageous way would be addition of databases [17] with specified scaling factors related to corundum; in this manner it would be possible to create a database common for both qualitative and quantitative phase analyses.

The RIFRAN 89 system does not claim to be capable of measuring all possible types of samples. On the contrary it appears to be highly suitable and useful for specialized laboratories dealing with a comparatively narrow range of materials, namely a database prepared by the user should contain about 300 standards. Under such conditions the results of qualitative analysis are very satisfactory and only rarely differ from actual analysis. Moreover, the statistical survey of the diffraction assignment readily contributes to making the analysis more accurate and to rendering the standard database more complete.

The results of quantitative analysis only confirm the reliability and suitability of this method. Of course, it is necessary to test very carefully all newly calculated standards and to gain experience with the use of the "instrument" factor. From the standpoint of standards, it is best to use actual measured ones, whose data were obtained on one and the same instrument used in the measuring of experimental samples.

CONCLUSION

On the basis of experience gained so far, the RIFRAN 89 system for automatic qualitative and quantitative phase analysis can be regarded as a suitable and practical tool in particular for specialized X-ray diffraction laboratories. The system makes the analytic work significantly faster and more efficient by allowing the use of routine procedures which, although demanding of the standpoint of measuring, calibration and evaluation, provide more accurate and reliable results.

References

- [1] Škrobian M., Havlík T., Havlík M.: *Hutnické listy* 5, 341 (1985).
- [2] Škrobian M., Havlík T., Havlík M.: *Powder Diffraction* 1, 3 235 (1986).
- [3] Havlík T., Škrobian M., Havlík M.: *Chemical Papers* 42, 2 121 (1988).
- [4] Weiss Z., Krajiček J., Smrček L., Fiala J.: *J. Appl. Cryst.* 16, 493 (1983).
- [5] Weiss Z., Krajiček J.: *Modelling of X-ray Diffraction Spectra* /in Czech/, Research Report VUÚ SP-4, Ostrava-Radvanice (1979).
- [6] Yvon K., Jeitschko W., Parthé E.: *J. Appl. Cryst.* 10, 73 (1977).
- [7] Šanc L., Špaček J., Kavalir J.: *Automation of X-ray Diffraction Phase Analysis* (in Czech), Research Report RFR 85-20, Kutná hora (1986).
- [8] Johnson G. G., Vand V.: *Ind. and Eng. Chemistry* 59, [8] 19 (1967).
- [9] Frevel L. K., Adams C. E., Ruhberg L. R.: *J. Appl. Cryst.* 9, 199 (1976).
- [10] Snyder R.: *Adv. X-Ray Anal.* 24, 83 (1981).
- [11] Goehner P., Garbaskas M.: *X-Ray Spectrometry* 13, [4] 172 (1984).
- [12] Schreiner W. N., Surdukowski G., Jenkins R.: *J. Appl. Cryst.* 15, 513 (1982).
- [13] Lindsey J. S., Cristensen C. P., Henslee W. W.: *Adv. X-Ray Anal.* 22, 151 (1979).
- [14] Hare T. M., Russ J. C., Lanzo E.: *Adv. X-Ray Anal.* 25, 237 (1982).
- [15] Fiala J., Fialová J.: *Chemické listy* 79, 48 (1985).
- [16] Fiala J.: *Silikáty* 31, 173 (1987).
- [17] *Powder Diffraction Set 25*. The Joint Committee for Powd. Diffr. Standards, Swathmore, Pennsylvania 1975.
- [18] Brindley G. W., Brown G.: *Crystal Structure of Clay Minerals*. Mineralogical Society, London 1980.

- [19] Michéyev V. I.: *X-ray Determination of Minerals* (in Russian). Gosud. nauch.-tech. izd. lit. Moscow 1957.
- [20] Fiala J.: *Hutnické listy* 6, 435 (1977).
- [21] Moravcová H., Fiala J.: *Věstník ústř. úst. geol.* 55, [4] 237 (1980).
- [22] Fiala J.: *Analytical Chemistry* 52, 1300 (1980).
- [23] Klug H. P., Alexander L. E.: *X-ray Diffraction Procedure*. J. Wiley and Sons, New York 1963
- [24] Fiala J., Fialová J.: *X-Ray Analysis of Substances in Metallurgy* (in Czech), Report TIZ 1352 UVZU Škoda Plzeň (1985).

MIKROPOČÍTAČOVÝ SYSTÉM PRE KVALITATÍVNU A KVANTITATÍVNU PRÁŠKOVÚ DIFRAKČNÚ FÁZOVÚ ANALÝZU

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V prezentovanej práci je popísaný malý, efektívny a flexibilný mikropočítačový systém pre automatickú difrakčnú kvalitatívnu a kvantitatívnu analýzu polykrystalických materiálov. Systém je tvorený sadou interaktívnych programov, napísaných v jazyku TURBOBASIC a implementovaných na domáci 8-bitový počítač ATARI 130 XE, vybavený disketovou jednotkou ATARI 1050.

Štandardnú databázu pre kvalitatívnu analýzu tvorí súbor štandardov, v ktorej každý je popísaný maximálne 55 difrakciami v hodnotách „d“ a „Irel“.

Pre kvantitatívnu analýzu je štandardná databáza vytvorená z vypočítaných [5, 6] alebo nameraných [7] difraktoqramov, reprezentovaných hodnotami „d“ a „Iabs“.

Identifikačná procedúra využíva porovnávanie difraktoqramov štandardov neznámej vzorky, využívajúc štatistické kritériá, chemickú analýzu a metódy matematickej optimalizácie.

МИКРОВЫЧИСЛЕННАЯ СИСТЕМА ДЛЯ КАЧЕСТВЕННОГО И КОЛИЧЕСТВЕННОГО ПОРОШКОВОГО ДИФРАКЦИОННОГО ФАЗОВОГО АНАЛИЗОВ

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В предлагаемой работе описывается небольшая, эффективная и гибкая микровычислительная система, предназначенная для автоматического дифракционного качественного и количественного анализа поликристаллических материалов. Система состоит из набора интерактивных программ в языке TURBOBASIC, используемых отечественным 8-битовым вычислителем ATARI 130 XE, оснащением дискетной единицей ATARI 1050.

Стандартную базу для качественного анализа представляет набор стандартов и каждый из них описывается максимально 55 дифракциями в величинах „d“, и „Irel“.

Для количественного анализа предназначена база, состоящая из вычисленных [5, 6] или измеренных [7] дифрактограмм, представленных величинами „d“ и „Iabs“.

Идентификационная процедура основывается на сопоставлении диаграмм стандартов и неизвестной пробы с использованием статистических критериев, химического анализа и метода математической оптимизации.