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Investigation of the Micro-Structural Parameters of Iron Alloys

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Abstract
The aim of the present work is to demonstrate the practical use of the "single line method for analysis" for determination of the micro-structural parameters of the solid solutions, like the domain size-D and the micro-strains of the lattice – ε. It demonstrates the practical use of the "single line method for analysis" for determination of the micro-structural parameters, developed on "Excel" program and complemented with the program "Origin" capable of processing the experimental results.

Keywords: micro-structural parameters, X-Ray analysis, single line method.

1. Introduction

The main micro-structural parameters of the solid solutions on iron base are: domain size-D (crystalline size), micro-strains of the lattice-ε and the concentration of the stacking faults -α. The last one is of significance only for some highly alloyed alloys with low stacking fault energy and only for some particular temperature and concentration intervals.
The aim of the present work is to demonstrate the practical use of the "single line method for analysis" for determination of the micro-structural parameters of the solid solutions on iron base, developed on "Excel" program and complemented with the program "Origin" capable of processing the experimental results [8-15].

2. Principles of the "single line method" for analysis

Basic principal of all known methods for determination of the micro-structural parameters of the solid solutions lies in investigation of their influence on the diffraction lines broadening [1-10].
This is the method of homologous pairs, in which the widths of two conjugated diffraction lines are examined [1-2].
Another technique for studying the microstructural parameters is the width of two and three reflection lines. It examines the widths of two or three of the major X-ray lines [3-7].
The principle of single line analysis is, on the basis of certain assumptions, that it is sufficient to study a diffraction line at low angles of reflection with maximum intensity at which its doublet does not have a noticeable effect on the width of the experimentally obtained line.
To avoid inaccuracy connected to the interpolation of the tabular data or the usage of different graphical methods the authors are worked out empirical formulas which provide a high accuracy of the experimental results processing.
The "single line method" for analysis is based on using the Voigt function for analyzing the integral width of a diffraction line. This method allows a quick and accurate determination of the micro-structural parameters as the domain size-D and the micro-strains-ε to be done.
The base principals of the "single line" analysis can be formulated as follow [8-14]:

- Investigation of the low angle diffraction lines with maximum intensity for which the broadening caused by presence of a doublet is negligible small.
- It is assumed that neither the Gauss function nor that of Cauchy (Lorenz) can completely describes the profile of the diffraction lines.
- The Pseudo-Voigt function, which normally is used for describing the neutron diffraction distribution, can be said to be a combination of Gauss and Cauchy (Lorenz) functions. If $I(x)=I(o).\exp\{-\pi.x^2/\beta_g^2\}$ is the Gauss function describing the profile of the intensity of the diffraction line I(x) in case of maximum intensity I(o), and $I(x)=I(o)/\{\beta_c^2/\pi^2+x^2\}$ describes the same profile with Cauchy (Lorenz) function then the integral breadth of the profile using Pseudo-Voigt function- β can be given by the dependence connecting the breadths of the maximums described by Gauss- β_g and by Cauchy- β_c.
  $I(x)=I(o).\{\beta/\beta_c\}.Re[erfi(\pi^{1/2}.x/\beta_g+ik)]$
  k- characteristic integral breadth of the Pseudo-Voigt function
  $k=\beta_c/\pi^{1/2}.\beta_g$ и $\beta^2=\beta_c.\beta+\beta_g$

On base of the assumptions made above Keijser, Landford and Mittemeijer [8] give direct relation between the micro-structural parameters, the breadths of the approximated lines β_c and β_g, λ and the diffraction angle θ of the respective line.

$$D=\lambda/\beta_c(f).\cos\theta,\ \varepsilon=\beta_g(f)/4.\tan\theta,$$

where $\beta_g(f)$ and $\beta_c(f)$ are the physical breadths of the line of the Gauss's and the Cauchy's component, determined after correction of the experimental and the instrumental breadths of the line.

The micro-structural parameters of the solid solution of the considered alloys were determined by using some special calculating features included in the program "Excel" [11].

3. Data analysis of the alloys

For processing the experimental data, the programs Origin 5.0 Pprofessional" and "Microsoft Excel" were used. Some procedures must be followed during the processing of the data for the diffraction lines, like:

- The Voigt function can be applied only for Kα1 radiation i.e. the diffraction maximum must be symmetric.
- $K_{\alpha 2}$ can be eliminated using a focusing monochromator.
- If K_α radiation is used the $K_{\alpha 2}$-duplet should be analytically or graphically separated from the investigated profile.

This condition in most cases creates an experimental difficulty because it requires special equipment or using of methods that Introduce greater errors during the data processing. Moreover, it is impossible to eliminate completely the influence of the $K_{\alpha 2}$- radiation especially on the greater angles of diffraction.

To avoid the above problem the calculations are done on diffraction lines with lower angles and with maximum intensities. On these lines the broadening caused by the presence of a duplet is neglectfully small.

The potential of the method is demonstrated with the processing of the experimental data for the armko-Fe on Fig.1.

The XRD analysis was carried out using standard apparatus "Philips- Micro 111" with Brag-Brentano geometry. The radiation used was – $Co_{k\alpha}$. Diffraction spectra were recorded for all the investigated specimens with step of 0,02°/min. Constant time for measuring were applied. In order to increase the accuracy, the holding time on each point was made 10 seconds.

For practical approximation of the experimental data program “Origin 5.0 Professional” was used.

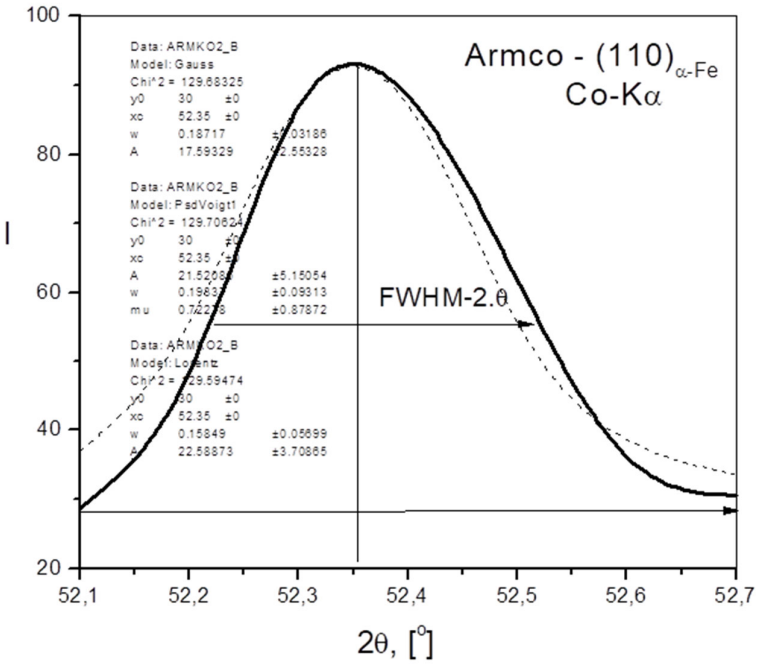


Fig.1. Non-linear approximation of the profile of the diffraction line using Pseudo-Voigt peak – armko-Fe

The approximation was carried out in the following order:
1. The diffraction line was drawn in an appropriate large scale as a narrow interval of angles around the maximum was chosen and the option “B-spline was applied for smoothing out the profile of the peak.
2. Using the Pseudo-Voigt peak function a non-linear approximation of the profile of the diffraction line was made. The values from the experimental curve, y_o and x_c were used and multiple interpolations were made until the values of the parameters of the curve were stabilized – Fig. 2 a), b), c).
3. The results were automatically plotted via the option – Action/Results/Paste parameters to plot and Plot Details/B-Spline – Fig. 2 d).
4. From plotting, we get the three dimensions – 2Θ(º2θ), 2ω (º2θ) (FWHM), the β(integral breadth), which we fill in the first three columns of Exel – Table 1, developed by I.T. Waluer [11]. This automatically obtains the values for D (domain size) and ε (strain) based on the program. Analogous calculations of the Arm-iron integral width after full annealing were used as a reference for obtaining the instrumental width of the line.

Table 1. Experimental data for the micro-structural parameters of “Armco-iron” calculated in “Excel”.

2Θ (º2θ *e*)	2ω (º2θ) (FWHM)	β(integral breadth)	ϕ(2ω/β)	βc/β	βg/β	βc(h)	βg(h)	βc(f)	βg(f)	D domain size	ε(strain)
52,28	**0,229**	**0,289**	0,793	0,524	0,630	0,0026	0,00318	**0,00104**	0,00295	190,63	0,0015

λ_{Co} =0,178.10 $^{-10}$ [m]

subscript c refers to Cauchy component
subscript g refers to Gaussian component

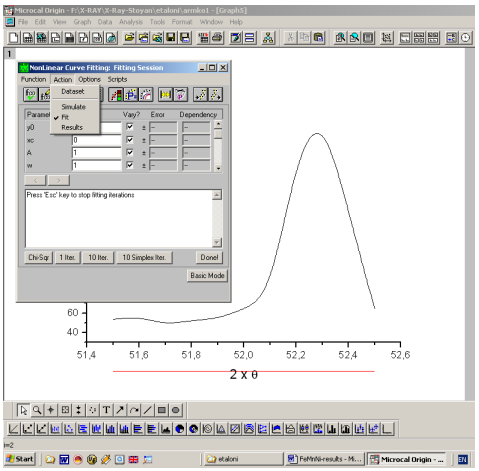
β (instr)
Coshi 0,0016063
β (instr)
Gausi 0,0011872

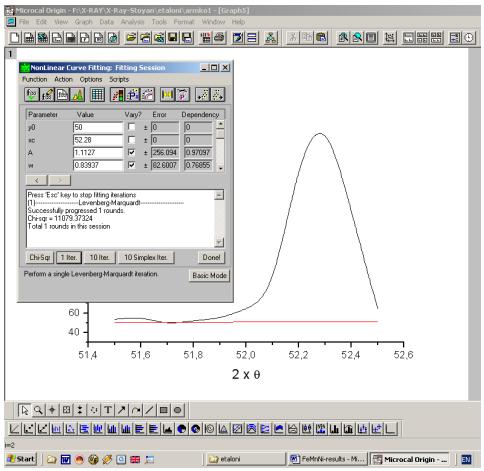
4. Conclusion

The results show the potential of the so called “single line method” for analysis of the micro-structural parameters of the solid solution of iron-based alloys. The method is developed on base of the program “Microsoft Excel” and complemented with the abilities for processing and plotting the experimental data with the program ‘Origin 5.0 Professional”.

a) Plot details

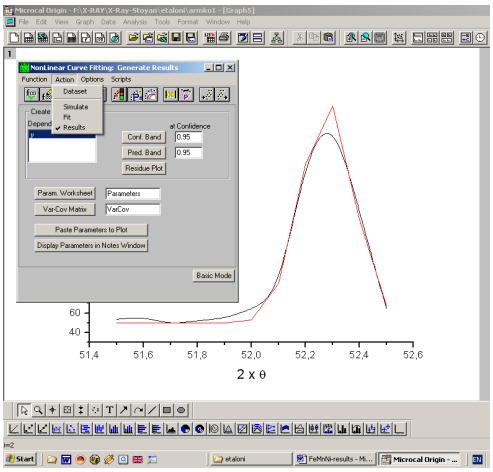
b)Non Linear Curve Fitting – Select Function

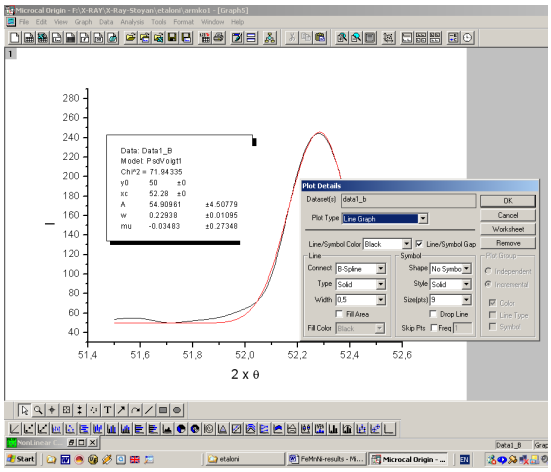




c) Non Linear Curve Fitting – Fitting Session

d) Non Linear Curve Fitting Session – Fitting Dataset





e) Non Linear Curve Fitting Session- Generate Resultats from the approximation

f) Plotting the results from the approximation B-Spline

Fig.2. Non-linear approximation of the profile of the diffraction line using Pseudo-Voigt peak function, plotting the results from the approximation

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