X.R.D. QUANTITATIVE PHASES ANALYSIS OF TEXTURED MATERIALS BY USING A CURVED P.S.D.

J.Bessires, J.J.Heizmann, A.Eberhardt*
 Laboratoire de Mtallurgie Physique et Chimique.
 Universit de METZ Ile du Saulcy - 57045 METZ Cdex.

* Laboratoire de Physique et Mcanique des Matriaux.
 URA CNRS 1215 Ile du Saulcy - 57045 METZ Cdex.

INTRODUCTION

Quantitative phases analysis of polycrystalline materials needs isotropic samples. In the case of textured materials the sample is artificially made isotropic by different ways; so the analysis theoretical background of randomly oriented sample can be applied.

The $I(hkl)_i$ integrated intensity diffracted by (hkl) lattice planes of the i phase can be written:

$$I_{(hkl)_i} = \frac{K \cdot R_{hkl_i} \cdot C_i}{2\mu m} \quad 1$$

where C_i is the volumic fraction of the i phase in the mixture,

$R(hkl)_i$ represents the theoretical intensity diffracted by the $(hkl)_i$ planes randomly oriented,

μm is the linear absorption coefficient of the mixture,

K a constant depending on the wave length and the experimental conditions.

From this relation the comparison of the integrated intensities of diffraction lines gives the relative concentration of each phase:

$$\frac{C_i}{C_j} = \frac{\left(\frac{I}{R}\right)_i}{\left(\frac{I}{R}\right)_j} \quad 2$$

This approach is not valid for textured phases

This approach is not valid for textured phases because the integrated intensities are strongly depending on the texture.

To avoid the texture effect:

- some people analyse only the high multiplicity lattice planes. This method could be used for high symmetry structure and smooth texture (1),

- some others (2) analyse a great number of lines of each phase with a 2θ diffractometer. But for high symmetry structure with few lines this method may often miss important texture components.

- Recently some people measure the integrated intensity of $(hkl)_i$ pole figures (3). This is a good way to obtain an accurate value of the concentration. This method takes a long time if the goniometer movements are not fast enough.

We present a method which mixes the three previous methods. All the lines of each phase are analysed with a INEL Curved Position Sensitive Detector able to measure 120° of the 2θ spectrum and this for several particular positions of the sample defined by the an incidence angle ω of the X-Ray beam and a tilt angle ψ of the goniometer. In this way, we increase artificially the lines number which are analysed and the information comes from areas which are spread over the pole figure.

This method is checked with drawn rods of CuZn alloy with 40% Zn. In this alloy the α (F.C.C.) and the β (B.C.C.) phases can coexist with different concentrations (0 to 100%) depending on the annealing conditions. The analysed samples are strongly textured. The results of quantitative phases analysis obtained by:

- micrographic technique
- integrated intensity of pole figures
- Dickson method
- this new method

are compared.

EXPERIMENTAL   PROCEDURE

The samples are coming from a brass rod which is cut in two perpendicular directions (normal to the drawing axis and parallel to the drawing axis). We present only the results of the last direction.

MICROGRAPHIC   OBSERVATION.

The surface of the sample is polished then a selective attack allows to observe the grains belonging to each phase. On the picture(1) the white grains belong to the α phase, the others to the β phase.

The ratio between the black and white areas gives the relative concentration of each phase. The measurement of the a concentration made on several pictures located at different places on the sample shows that

- the grains of about $10\mu\text{m}$ large are oriented towards the rod axis,
- the phases repartition is homogeneous.

We obtains the following results: $C\alpha = 72\% \pm 3\%$

X.RAY EXAMINATION.

As the depth of penetration of X.Ray is about $10\mu\text{m}$ in brass, the X.rays examinations are superficial ones. The results obtenained with methods using X.R.D. results can be easily compared to the micrographic ones.

Integrated intensities of poles figure.

We have choosen for the α phase the pole figures 200, 220, 311 and for the β phase the 110, 200, 211 pole figures. We used a multipole figure goniometer i.e the $(220)\alpha$, $(211)\beta$ and $(311)\alpha$ pole figures have been measured simultaneously, as also for the three others.

We can notice that each simultaneous measurement take three hours. The integrate intensity of each pole figure is calculated. After intensities corrections (coming from the positions of the sample) and background subtraction. The two phases are strongly oriented (pictures 3 and 4), each phase have a fiber axis parallel to the drawing axis.

The $C\alpha$ concentration is calculated by using the relation 2 in which I_i and I_j are respectively integrated intensities of a pair of pole figures of the i and j phases. The $C\alpha$ values are listed in the table 1 and depend on the pair of pole figure choice.

ratio	$\frac{(220)_\alpha}{(211)_\beta}$	$\frac{(311)_\alpha}{(211)_\beta}$	$\frac{(200)_\alpha}{(200)_\beta}$	$\frac{(200)_\alpha}{(110)_\beta}$	$\frac{(220)_\alpha}{(200)_\beta}$	$\frac{(220)_\alpha}{(110)_\beta}$	$\frac{(311)_\alpha}{(200)_\beta}$	$\frac{(311)_\alpha}{(110)_\beta}$	$\frac{(200)_\alpha}{(211)_\beta}$	$\overline{C_\alpha}$
C_α	76.3	68.8	67.6	72.5	70.6	75.2	62.2	67.5	73.7	70.5

table 1

The mean value is $C\alpha = 70,5\% \pm 6\%$

By using all the pole figures and the Dickson relation in which the intensity of each line is replaced by the integrated intensity of each pole figure:

$$C_i = \frac{\frac{1}{n_i} \times \sum \left(\frac{I}{R} \right)_i}{\frac{1}{n_i} \times \sum \left(\frac{I}{R} \right)_i + \frac{1}{n_j} \times \sum \left(\frac{I}{R} \right)_j} \quad 3$$

where n_i and n_j are respectively the number of pole figure of each phase, we obtain a more precise value of the concentration: $C\alpha = 72\% \pm 4\%$

X.R.D. lines intensities analysis.

In the range of 2θ lines diagram, 9 lines appear, 5 belonging to the α phase and 4 to the β phase (picture 4). The recording of a 2θ spectrum takes 5mn. with the C.P.S. detector. The background is substracted and the integrated intensity of each line is calculated, then this intensity is corrected because the lines are not in the Bragg-Brentano conditions.

During the intensities measurement, the sample rotates around the normal of its plane to increase the diffraction probability as it is generally done with a 2θ diffractometer. The analysis of the 2θ spectrum gives a value of the $C\alpha$ concentration by using the method of Dickson (in this case 9 lines).

If with only one spectrum, we get a good value of the concentration (because all the main components of the texture have been taken into account) a second experiment done with another orientation of the sample should give the same value of $C\alpha$ even if the 2θ spectrum is different.

For this we had calculated several $C\alpha$ concentrations coming from different positions spread over in space. By analysing each spectrum of 9 lines coming from 12 different experiments, the values of $C\alpha$ are ranged between 64% and 84%. This shows there is not enough lines on a spectrum to represent all the texture components.

In fact if the number of lines is infinite all the texture components are taken into account because to each

line corresponds a set of orientations. By changing the sample position other sets of orientations are detected, it is as if we have other lines. For example by coupling of two different spectrums it as if we have 18 lines. The results are given in the table 2.

number of lines	9	18	36	90
C_{α}	74 ± 10	75 ± 7	72.5 ± 3.5	74 ± 1.5
time in mn.	5	10	20	50

table 2

We can observe that we reach a limit value of C_{α} after 90 lines:
 $C_{\alpha} = 74\% \pm 1,5\%$

CONCLUSION.

The different results are presented in the following table 3

methods	micrographic	Pole figure	Polefigure set	9 lines	36 lines	90 lines
C_{α} en %	72 ± 3	70.5 ± 6	72 ± 4	74 ± 10	72.5 ± 3.5	74 ± 1.5
time in mn.		180	360	5	20	50

table 3

All the methods give results in agreement with different precision a pair of pole figures or only a 2 θ spectrum (9 lines) give poor results. We can notice that the multispectrum analysis method is a fast method which gives good results.

BIBLIOGRAPHY.

1 DURNIN J. and RIDAL K.A.-*J. Iron St. Inst.* (1968) 206
2 DICKSON J. - *J. Appl. Cryst.* **2** (1969) 216
3 FILLIT R. and BRUYATS H. *X.R. Coll. Siemens. Grenoble* (1985)

Pict.2: 200 pole figure of α phase

Pict.3: 110 pole figure of β phase

Pict.4: 2 θ spectrum of the α and β phase ($\omega=40$, $\psi=0$)

Pict.1: α and β phases in a brass rod