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# Quantitative Phase Analysis of a Highly Textured Industrial Sample using a Rietveld Profile Analysis

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For the quantitative phase analysis on highly textured two-phase materials, samples with known weight fractions of zirconium and aluminum were prepared. Strong texture components prevailed in both zirconium and aluminum sheet. The diffraction patterns of samples were measured by the neutron and refined by the Rietveld method. The preferred orientation correction of diffraction patterns was carried out by means of recalculated pole figures from the ODF. The present Rietveld analysis of various samples with different weight fractions showed that the absolute error of the calculated weight fractions was less than 7.1 %.

KEYWORDS: Rietveld method, quantitative phase analysis, neutron diffraction, texture

## §1. Introduction

Strong preferred orientations often develop in most of multi-phase alloy materials produced by thermomechanical treatments. Because physical properties depend on the fraction of each phase, it is of importance to determine the volume fraction of phases in multi-phase alloy materials. The Rietveld profile refinement has been used with an ideal powder sample for the crystallographic characterization of materials including crystal structure and phase analysis.[1)] In order to apply the method to industrial materials, the preferred orientation factors should be properly corrected.[2,3)] In the textured materials, the integrated intensity of a peak in the diffraction pattern is directly proportional to the pole density of the corresponding inverse pole figure or recalulated pole figure, which can be obtained from the three-dimensional orientation distribution function(ODF) of the material.[4)]

In this work, the preferred orientation correction method by using the recalculated pole figures[5)] was introduced and the experimental study of its application to the Rietveld profile refinement for highly textured samples was carried out.

## §2. Experimental procedure

The as-received materials were a hot band of zirconium with 99.7 % purity and a hot band of the commercial aluminum alloy with 99.9 % purity. In order to obtain the strong cube-texture in the aluminum sheet, the aluminum hot band was first cold rolled to 90 % reduction and recrystallized at 300°C for 1h. Samples with known weight fractions of zirconium and aluminum were prepared by stacking both the zirconium hot band and the recrystallized aluminum sheet. The size of stacked specimens for the neutron diffraction was about 10mm × 10mm × 10mm.

Diffraction patterns were measured with the High Resolution Powder Diffractometer(HRPD) equipped with 32 detectors and complete pole figures of zirconium and aluminum were measured with the Four Circle Diffractometer(FCD) at HANARO in KAERI. The neutron wavelength was 1.84 Å and 0.99 Å for HRPD and FCD, respectively. The ODFs were analyzed by using the BEARTEX program,[6)] and the quantitative phase analysis was performed by using the FullProf program.[7)]

## §3. Results and Discussion

Figure 1(a) shows the diffraction pattern of the sample No.1 from Table 1. Circles and solid line represent observed and calculated pattern, lower dot line represents the difference. We can notice that the differences between observed pattern and calculated pattern are very high in Fig. 1(a). This means that there is strong preferred orientation of grains in both zirconium and aluminum.

To increase the statistic of measurement, samples were rotated about the z-axis parallel to the normal direction(ND), so that scattering vector was parallel to the x-y(or rolling direction(RD)-transverse direction(TD)) plane.

For the preferred orientation correction we measured a few complete pole figures of zirconium and aluminum. Measured and normalized pole figures were shown in Fig. 2. As shown in Table 1 and Fig. 2, it is noted that preferred orientation in the aluminum sheet was substantially stronger than that in the zirconium hot band, since the cold rolling and the subsequent annealing of the aluminum sheet gave rise to a quite pronounced cube texture $\{001\}\langle 100\rangle$. The preferred orientations in the zirconium hot band can be characterized by the $\{002\}$ and $\{110\}$ planes. Thus, in the zirconium hot band, the evolution of the $\{110\}//\mathrm{RD}$ preferred orientations with their basal plane $\{002\}$ lying 20° away from ND was observed as shown in Fig. 2(a).

After analyzing their ODFs using the WIMV(Williams-Imhof-Matthies-vinel)[8)] method, we recalculated the pole figures from the ODFs. And we averaged the pole densities of each recalculated pole figure with respect

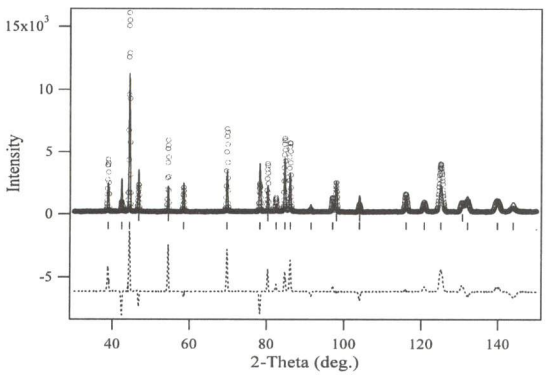
TableI. The results of the quantitative phase analysis for the samples of mixed Zr and Al

| Sample No. | Total weingt(g) | Al weight(%) | ODF: Zr | f(g) max Al | $\chi^{2*)}$ | Refinement $R_B$**):Zr | results $R_B$:Al | Al wt(%) | Error of Al weight (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 10.21 | 24.29 | | | 3.21 | 1.79 | 7.23 | 23.56(0.36) | 2.9 |
| 2 | 9.0 | 14.11 | 5.9 | 64.14 | 3.84 | 2.42 | 9.13 | 13.21(0.22) | 6.4 |
| 3 | 8.53 | 9.38 | | | 3.43 | 2.41 | 7.54 | 8.71(0.18) | 7.1 |
| 4 | 8.06 | 4.1 | | | 3.59 | 2.43 | 14.6 | 3.93(0.07) | 4.1 |

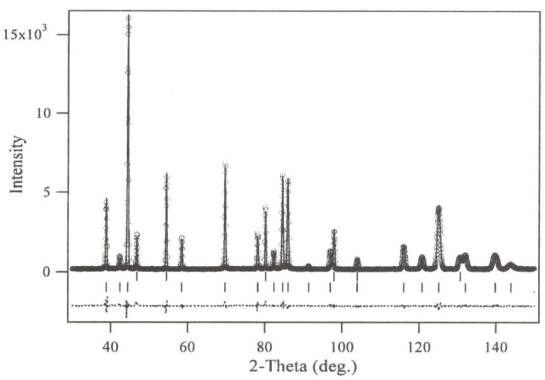
*) Conventional agreement index, the goodness of fit[7)]
**) Conventional agreement index, the Bragg R[7)]

to the azimuthal direction because the samples were rotated about ND in the diffraction measurement. These averaged pole densities of the recalculated pole figures corresponding to diffraction peaks were used as an input of the preferred orientation factors for the Rietveld analysis.

The result of refinement corrected with the preferred orientation factors was displayed in Fig.1(b). The Rietveld refinement without the preferred orientation correction in Fig.1(a) is proved to be unacceptable to analyze the highly textured material. In contrast, the diffraction pattern calculated by the Rietveld analysis

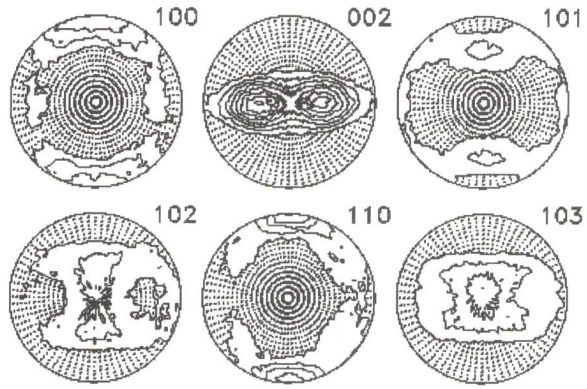



(a)Without the preferred orientation correction

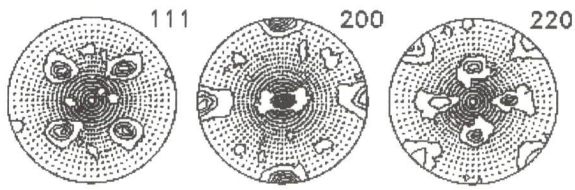



(b)With the preferred orientation correction

Fig. 1. The diffraction pattern of sample No.1 (a) without the preferred orientation correction, (b) with the preferred orientation correction: The weight percent of Al is 24.29. Circles and solid line represent observed and calculated pattern, lower dot line represents the difference.




(a)Pole figures of Zr




(b)Pole figures of Al

Fig. 2. The measured and normalized pole figure: (a) zirconium hot band, the maximum pole density is 4.63, (b) cold rolled and recrystallized aluminum sheet, maxmum pole density is 15.6.

with the preferred orientation correction is very close to the experimental result as shown in Fig. 1(b). It was also noted that the evaluation error was less than 3 % with fitting results of $R_B = 1.79$ for zirconium peaks, $R_B = 7.32$ for aluminum peaks and $\chi^2 = 3.21$. The results obtained from four samples with different weight fraction of zirconium and aluminum are summarized in Table I.

## §4. Summary

Refinements by the Rietveld profile analysis for the highly textured samples were successfully carried out by using the preferred orientation factors obtained from the recalculated pole figures, so that we could obtained the agreeable values of the weight percent for their component in all samples. These results show that the quantitative phase analysis would be possible for highly textured industrial samples.

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