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Neutron Diffraction Texture Analysis of 1 Vol.% Cu in Aluminium

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Neutron diffraction texture determination was carried out in two-phase composites, AlCu1%, prepared by powder metallurgical methods and extruded 84 and 89% respectively. The texture of the minor Cu-phase can be measured with a sufficient degree of accuracy within reasonable time. This texture is weaker than the texture of the Al-matrix phase corresponding to the lower internal deformation degree of the former one. It is estimated that the method can be extended to even lower volume fractions of the second phase.

KEY WORDS: Powder metallurgy, Aluminium-copper composite, Small volume fraction, Extrusion, Neutron diffraction, Inverse pole figures.

INTRODUCTION

Phase analysis and texture analysis in polyphase materials can be carried out by diffraction methods measuring relative intensities of Bragg-peaks. If one of the phases is present only in a small volume fraction then this method is limited by the relative accuracy with which intensity measurements of Bragg-peaks can be carried out. If the strongest Bragg-peak of a minor phase is in the order of magnitude of the background scattering then this method does not work. Hence, the lower detection limit of a minor phase depends on the peak to background ratio of the considered diffraction method. This is generally better in the focussing methods compared to

nonfocussing methods, it is better using photographic methods compared to diffractometer methods since a line on a photographic film can be easier distinguished from the background than a peak in an intensity plot and it is better in neutron diffraction compared with X-ray diffraction since all intensity correction factors in neutron diffraction are smaller than those in X-ray diffraction and can be calculated with a better accuracy (Bacon, 1975; Szpunar, 1976; Welch, 1986).

As a rule, the lower limit of texture analysis using X-ray diffraction is assumed to be at about 5 Vol.% of a minor phase in a polyphased material. In the present investigation it will be shown that, using neutron diffraction this lower limit is much smaller. The texture of 1 Vol.% copper in an Al-Cu composite could "easily" be measured and it turns out that this is not yet the lowest possible volume fraction.

EXPERIMENTAL PROCEDURE

Two Al-Cu composite samples were prepared by powder metallurgical methods. They were extruded 84% and 89% respectively. The characteristic data of the samples are given in Table 1. Because of the axial symmetry of the extrusion process the obtained textures of the copper and the aluminium phase are axially symmetric too, i.e. they are fibre textures.

The texture measurements were carried out at the research reactor FRG-1 at Geesthacht using the texture diffractometer TEX-1. A wavelength of $\lambda = 0.1618$ nm was obtained by an Al (111)

Table 1 Characteristic parameters of the analysed samples

Characteristics	Sample A	Sample B
aluminium	99%	99%
copper	1%	1%
sample size	cylinder ∅ 11.8 mm height 11.0 mm	cylinder ∅ 10.0 mm height 10.0 mm
deformation degree (extrusion)	84%	89%

Table 2 Bragg peaks of the composite sample in the available 2θ-range

Sample	*hkl*	2θ	Overlapped
Al	111	40.488	
	200	47.119	√
	220	68.852	
Cu	111	45.592	√
	200	53.161	

monochromator. A description in detail of the instrumental layout is given by a GKSS-Report (Brokmeier, Bunge, Brehler, Wagner, Wille, 1987). Due to the low absorption coefficient, samples of the dimensions given in Table 1 can be considered as being "spheres" such that no absorption correction is needed (Tobisch and Bunge, 1972). The limiting 2θ-angle of the diffractometer being 70°, the peaks given in Table 2 were within the available 2θ-range. Of these peaks, the Cu (111) peak was slightly overlapped within Al (200) peak as is to be seen from $\omega - 2\theta$-scans as shown in Figure 1.

Since the textures are fibre textures, only one-dimensional pole figures are to be measured. They are obtained by χ-scans of the Eulerian cradle corresponding to the pole figure angle α, i.e. the angle towards the rod axis. For each pole figure the Bragg-angle 2θ was chosen according to the peak-position of the 2θ-scan with an angular divergence of 1.1°. Additionally several 2θ-scans were measured in the 2θ-range of the Al (200) and Cu (111) in angular steps of $\Delta\alpha = 5°$ in order to better separate these two pole figures. The results are shown in Figure 2 for the two samples and three aluminium pole figures and two copper pole figures respectively. From these curves also the peak to background ratio can be estimated. The measuring times for the whole texture determination are shown in Table 3.

RESULTS

As is seen from Figure 2, the volume fraction of 1% Cu in 99% Al corresponding to total amounts of 106 or 66 mg Cu respectively was sufficient to determine two pole figures of the minor phase. The

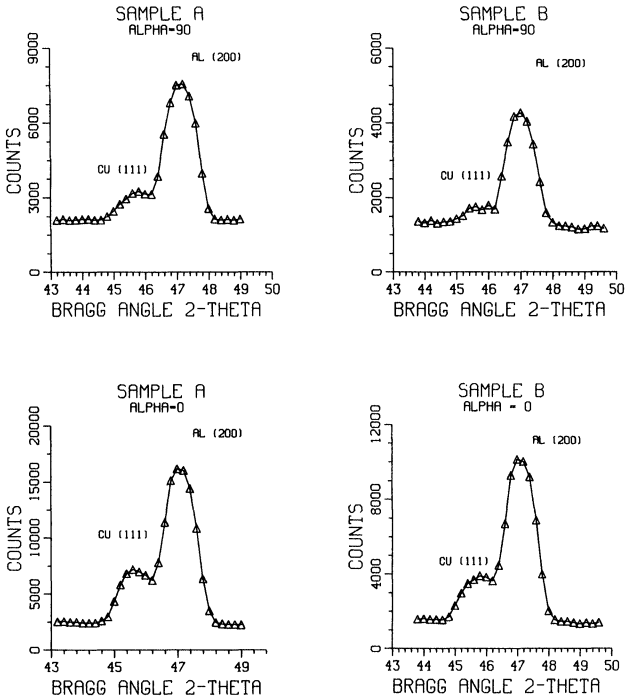

Figure 1 2θ-scan in the range of the overlapping Bragg-peaks Al (200) and Cu (111).

total deformation degrees of the two samples were 84% and 89% respectively. This is also the deformation degree of the softer Al-matrix phase, whereas the harder Cu-phase was much less deformed (Brokmeier, Böcker and Bunge, 1988). The estimated internal deformation degree of this phase was only 45% and 55% respectively. Correspondingly, the texture of the Cu-phase was much weaker than the texture of the Al-phase.

Orientation distribution functions (ODF) were calculated from the pole figures using three and two pole figures respectively and series expansions up to $l = 22$ and $l = 20$ for the two phases (e.g. Bunge, 1982; Dahms, 1987). The results are shown in Figure 3. Both textures are double fibre textures $\langle 111 \rangle$ and $\langle 200 \rangle$ with peak intensities given also in Figure 3.

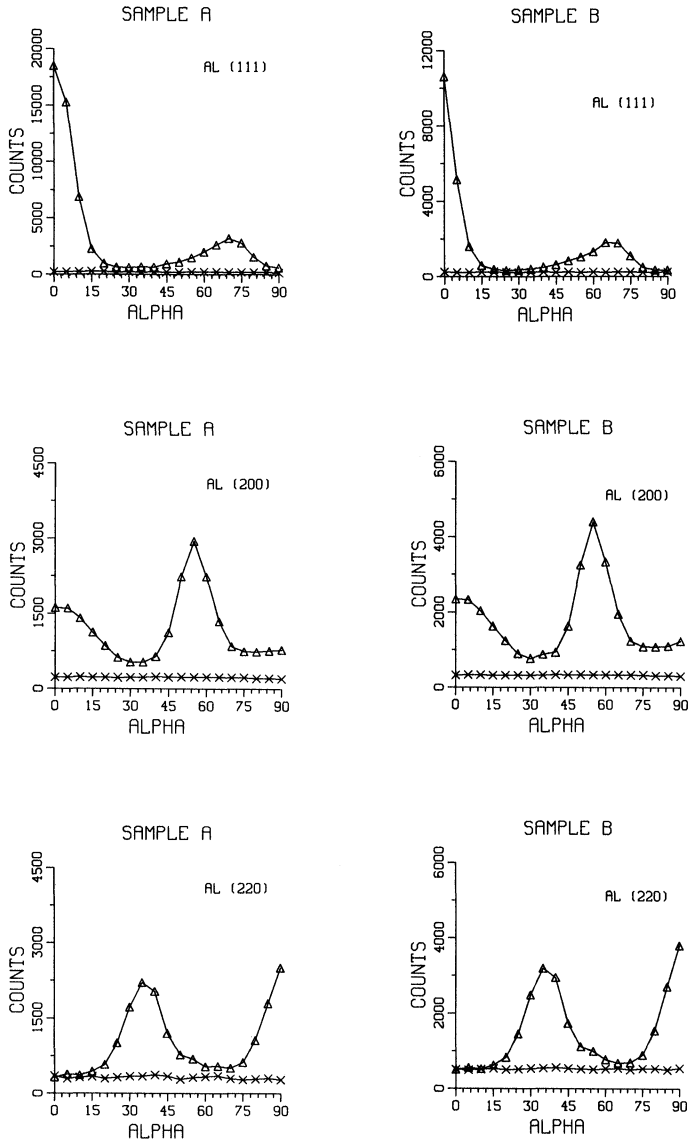

Figure 2 Experimental pole figures of the Al- and Cu-phase in two Al-Cu composites.

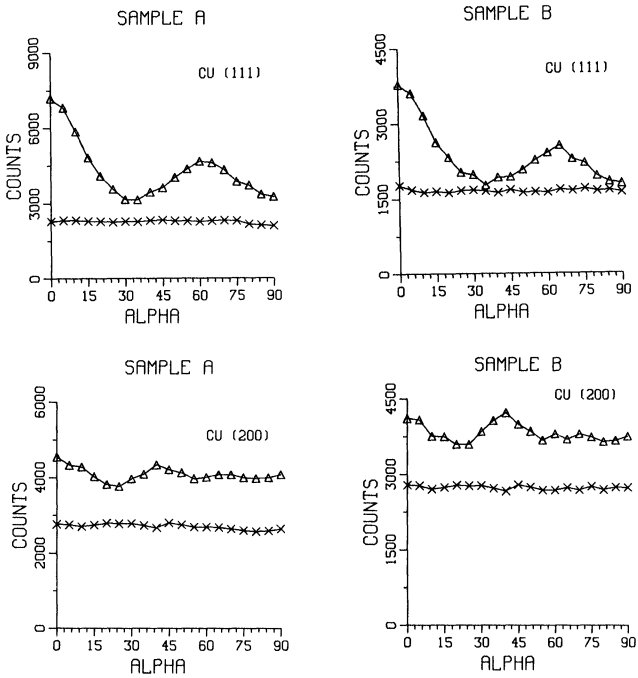

Figure 2 (*Continued*)

CONCLUSION

It has been demonstrated that texture determinations of phases of small volume fractions can be carried out with a sufficient degree of accuracy using neutron diffraction. By critically analysing the obtained data, it can be concluded that even lower volume fractions can be reached if the experimental conditions are optimised (if

Table 3 Measuring times for different scans

Characteristics	Sample A	Sample B
complete analysis	18.4 h	27.1 h
only aluminium	2.4 h	3.4 h
Cu 111	7.5 h	7.5 h
Cu 200	8.5 h	16.2 h

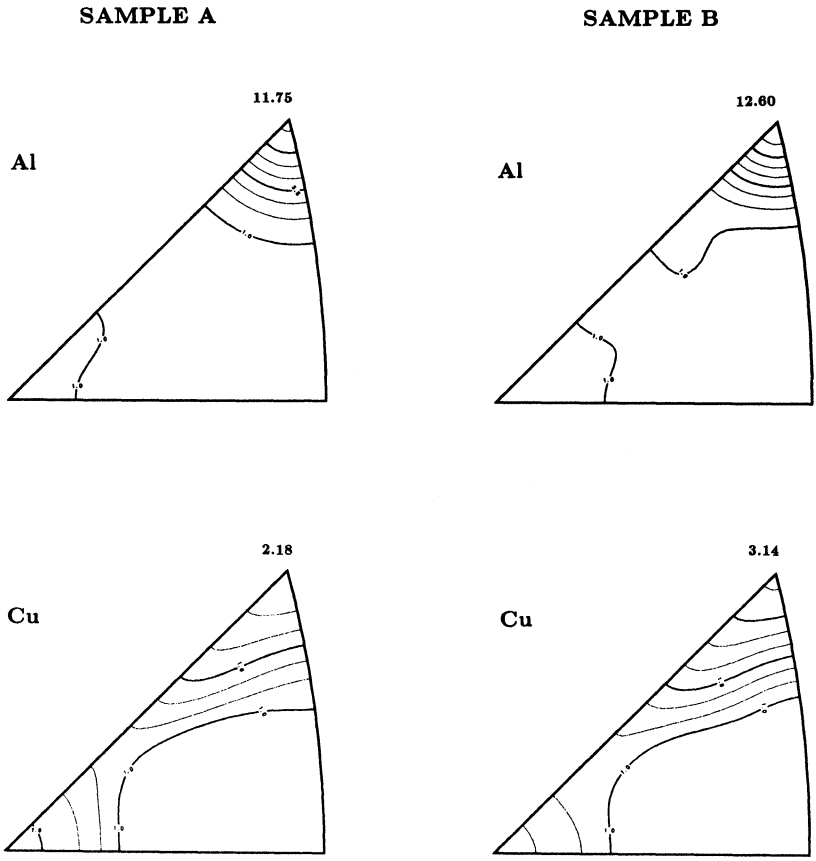

Figure 3 Inverse pole figure calculated from pole figures (Figure 2).

necessary on the expense of still longer measuring times). It was estimated that in the system Al-Cu texture measurements of 0.1% Cu should be possible. (Preliminary experimental results obtained in 0.1 Vol.% samples seem to corroborate this estimation.) This opens up the possibility of texture studies in phases occurring in small volume fractions such as for example small precipitates in matrix materials which are virtually not possible by X-ray diffraction. This will be interesting with respect to the precipitation

process itself as well as to the deformation of precipitation hardened materials.

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