JOURNAL OF MATERIALS SCIENCE 21 (1986) 1688–1692

A determination of the texture of a directionally solidified sample of high-purity copper

EVAN GRANT
Department of Metallurgy and Materials Science, Cambridge University, Cambridge, UK
DORTE JUUL JENSEN
Metallurgy Department, Risø National Laboratory, Denmark
BRIAN RALPH
Institute of Materials, University College Cardiff, Cardiff, UK

A study making a combined use of neutron diffraction and selected-area electron channelling to determine the solidification texture in a high-purity copper sample is described. Good correlation between the techniques is shown with both demonstrating a strong [1 0 0] fibre texture in the directionally solidified rod.

1. Introduction

There is a continuing interest in the development of crystallographic texture during the thermo-mechanical processing of materials (e.g. [1–4]). Some considerable interest has also been expressed in the textures which are associated with solidification and casting (e.g. [5–8]). Here a distinction is made between planar growth, which in fcc metals leads to a pronounced ⟨1 1 1⟩ texture, and dendritic or columnar growth which favours the development of ⟨1 0 0⟩ components. A number of models have been suggested to account for these texture developments which accompany solidification (e.g. [8]).

Previous investigations of solidification textures have involved the use of X-ray diffraction. It is the purpose of the present short paper to suggest that additional information may be gleaned using a combination of neutron diffraction and electron channelling. These techniques have already been applied to investigations of textures and texture development during thermo-mechanical processing but have not previously been used in combination to study solidification textures.

Neutron diffraction has a number of significant advantages for such studies. Perhaps the principal one here is that large samples containing coarse grain distributions may be studied (e.g. [9, 10]) and the particular instrument used here allowed a full quarter pole figure to be measured and displayed in a time of about 10 min (e.g. [11, 12]).

It is possible to take electron channelling patterns in many forms of electron microscope while micrographs in a number of contrast modes can be taken from the same samples; this gives a locally specific picture of the texture with high crystallographic precision (e.g. [13–15]).

2. Experimental techniques

An as-directionally solidified sample of high-purity copper, approximately 1 cm diameter, supplied by Asarco was investigated. Light microscopy using polarized contrast was used to investigate the overall grain structure. Optimum contrast in this mode was achieved using polars which were not fully crossed.

The apparatus for determining textures using neutrons has been described previously [11]. Neutrons were accepted from the DR3 reactor at Risø and after a monochrometer arrangement which could be adjusted to give the neutron wavelengths required, they impinged on a bulk sample which was in turn mounted in a three-circle goniometer cradle. The orientation of the sample, via stepping motors, and the detector were then controlled using a PDP11 computer. Data processing to produce pole figures, etc., was also performed by this computer and the results displayed in colour on a Canon computer.

A JEOL 840 scanning electron microscope was used to produce the channelling patterns and microscope images. The imaging mode adopted used channelling contrast so that the change from imaging to selected-area channelling might be accomplished easily. Polaroid images were taken initially of the grain structure so that a record might be made of which selected-area channelling pattern came from which grain. To obtain channelling patterns does require very careful specimen surface preparation (here the surfaces were electropolished as the last stage of preparation) because the pattern is so sensitive to surface state (e.g. [13]). It is also a relatively time-consuming process to record sequential patterns from grain structures and accordingly the sample sizes investigated are quite small (here the two samples given are from 33 and from 28 grains). However, these prove to be enough to give a useful correlation with the neutron texture data.

3. Results and discussion

3.1. Light microscopy

Fig. 1 gives typical results obtained for the transverse section of the directionally solidified rod (a) and for the longitudinal section (b) of the same sample. The

0022–2461/86 $03.00 + .12

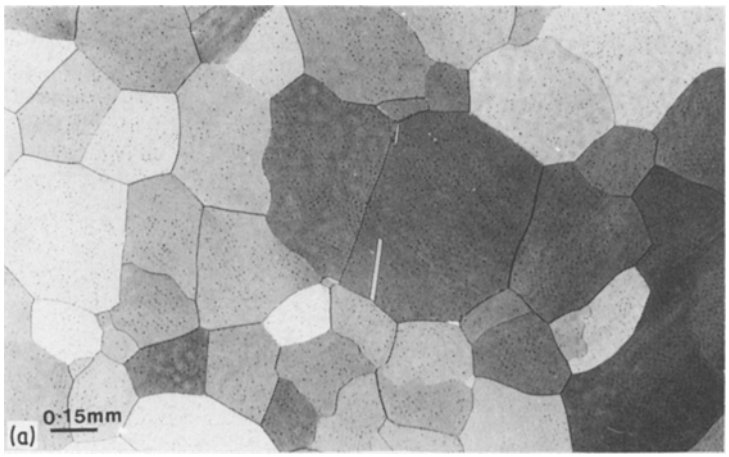
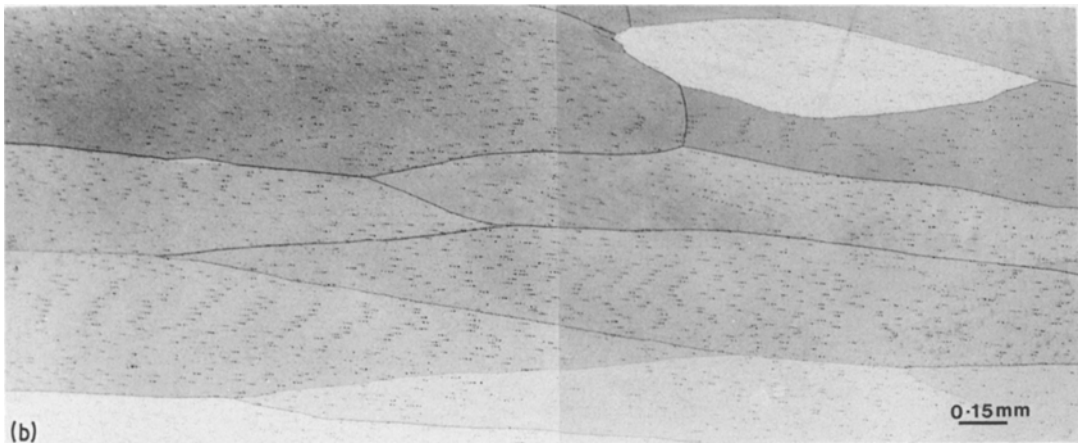


Figure 1 Light micrographs using polarizing contrast of the transverse section (a) and longitudinal section (b) of an as-directionally solidified high purity copper sample.



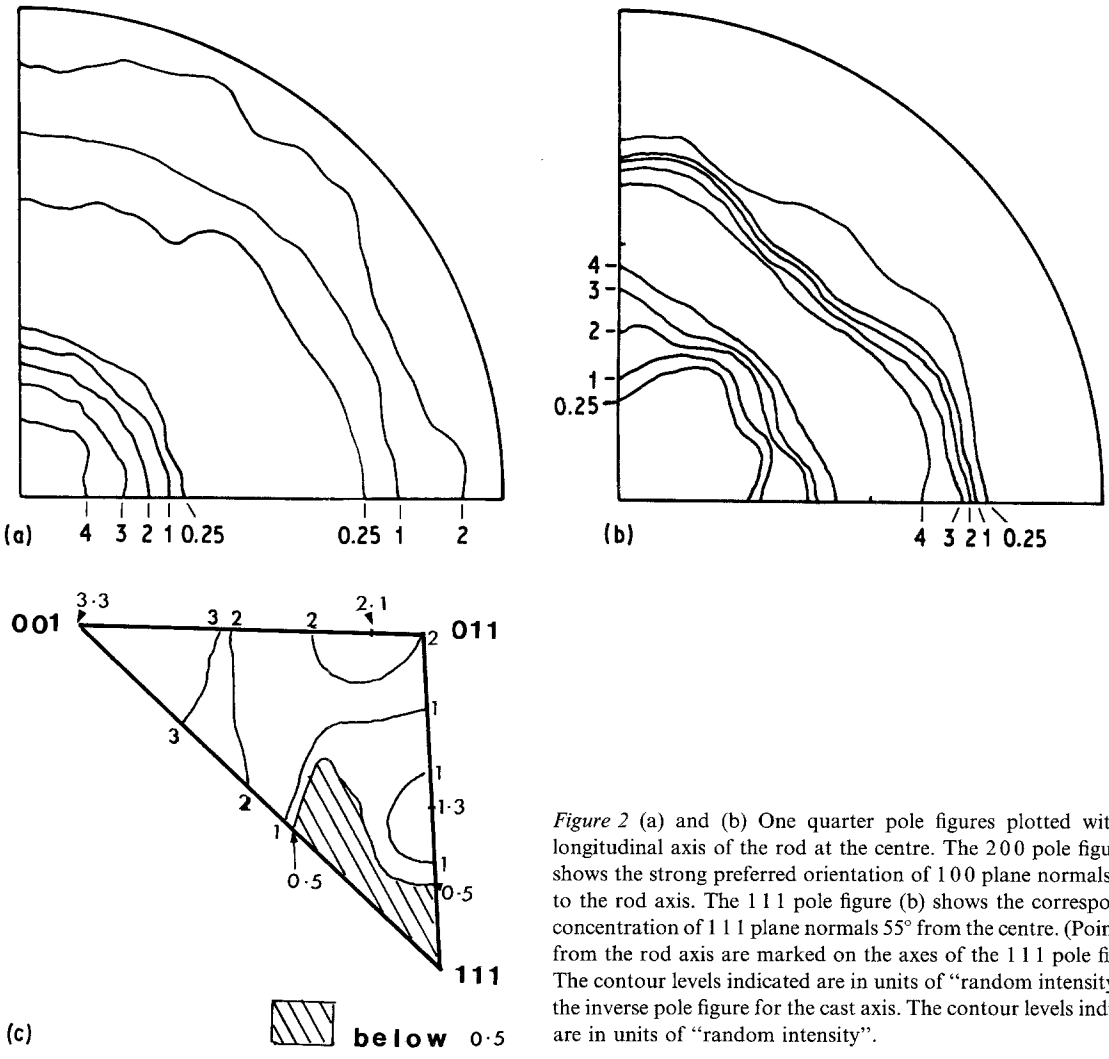


Figure 2 (a) and (b) One quarter pole figures plotted with the longitudinal axis of the rod at the centre. The 2 0 0 pole figure (a) shows the strong preferred orientation of 1 0 0 plane normals close to the rod axis. The 1 1 1 pole figure (b) shows the corresponding concentration of 1 1 1 plane normals 55° from the centre. (Points 55° from the rod axis are marked on the axes of the 1 1 1 pole figure.) The contour levels indicated are in units of "random intensity". (c) the inverse pole figure for the cast axis. The contour levels indicated are in units of "random intensity".

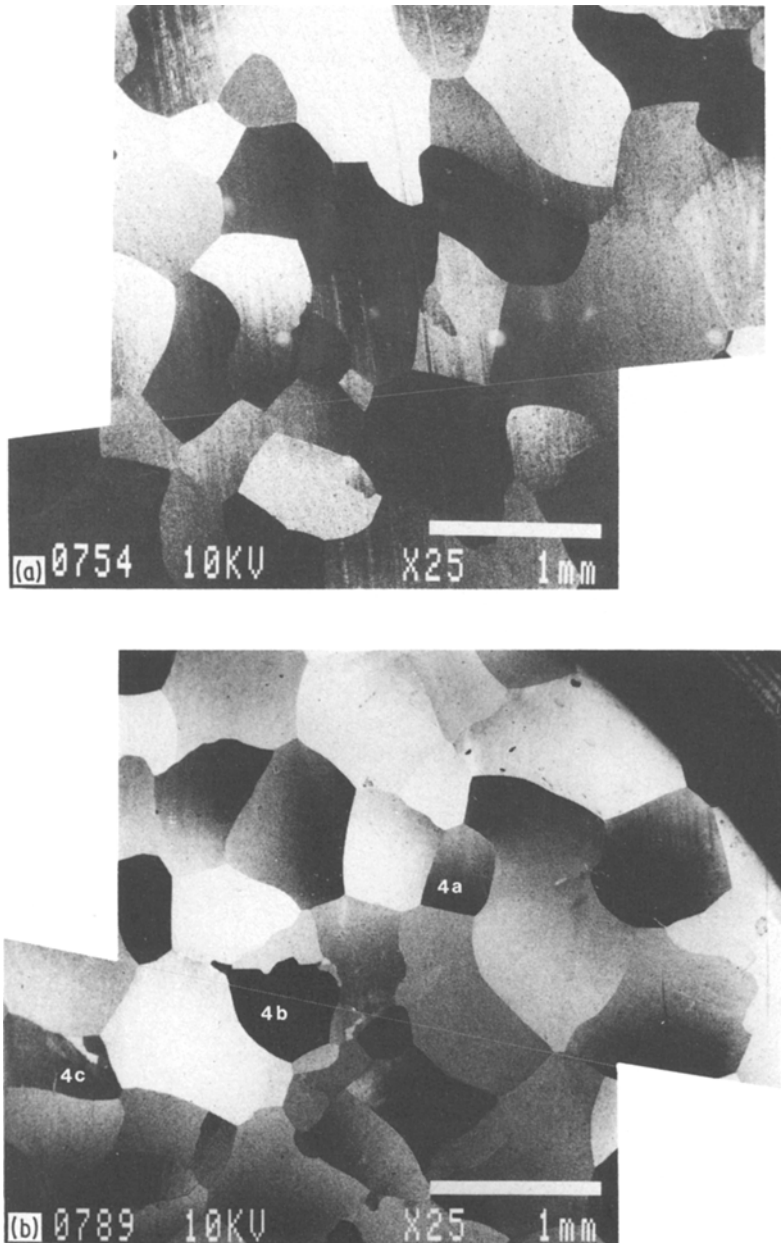


Figure 3 Channelling contrast scanning electron micrographs of transverse specimen sections. Most of the grains in each of these fields of view were crystallographically analysed using the selected-area channelling techniques (see text). The grains from which the channelling patterns in Fig. 4 were taken are marked appropriately on (b) and Figs 4a to c.

grains are seen to be columnar in nature with an aspect ratio of around 20.

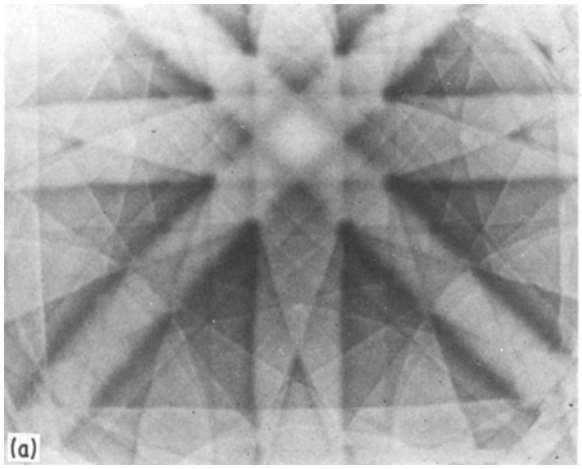
3.2. Neutron diffraction

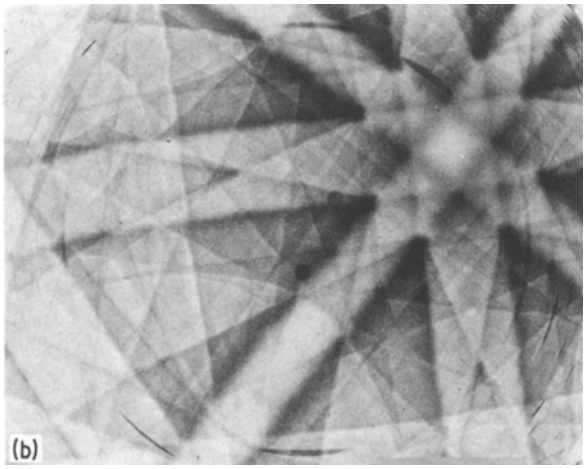
Two complete quarter pole figures, (1 1 1) and (2 0 0), were measured by neutron diffraction. Both figures showed full rotational symmetry about the cast/fibre axis (see Fig. 2a). The three-dimensional orientation distribution functions (ODF) were calculated using the series expansion method [4]. The series were truncated at I max $= 22$ and only terms of even rank were considered. Based on the ODF data, an inverse pole figure (Fig. 2c) for the cast/fibre axis was calculated, giving information about the volume density of crystals having the $[h\,k\,l]$ direction parallel to this axis. Since the pole figures show rotational symmetry, this with the inverse pole figure data gives a very full picture of the texture of this sample. The inverse pole figure (Fig. 2c) demonstrates a relatively sharp [1 0 0] texture. Based on the previous data on solidification textures (e.g. [5–7]) it would appear that the growth mode which gave rise to this columnar grain structure was dendritic since this is the texture pattern normally associated with dendrites.

3.3. Scanning electron microscopy

Fig. 3 gives typical channelling images from transverse specimens of this material. The area shown in Fig. 3a includes a region where 33 neighbouring grains were crystallographically analysed by the selected-area channelling technique. Fig. 3b shows a similar area; in this case 28 neighbouring grains were subjected to crystallographic analysis using the channelling technique. The positions from which the three selected-area channelling patterns given in Fig. 4 were obtained are marked on this figure.

Fig. 5 gives the orientation of the surface normals of the grains inserted on to unit stereographic triangles. Fig. 5a gives the data relevant to Fig. 3a while Fig. 5b gives the same thing for Fig. 3b. The data from both Figs. 5a and b has been combined into the plot given as Fig. 5c. Clearly only a limited amount of the information available from the selected-area channelling patterns has been used in these simple plots (i.e.





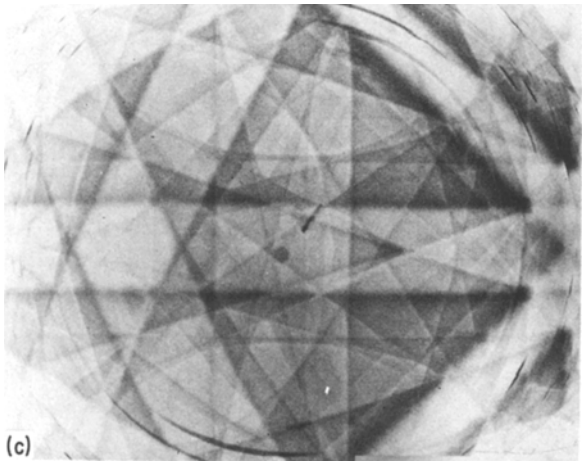
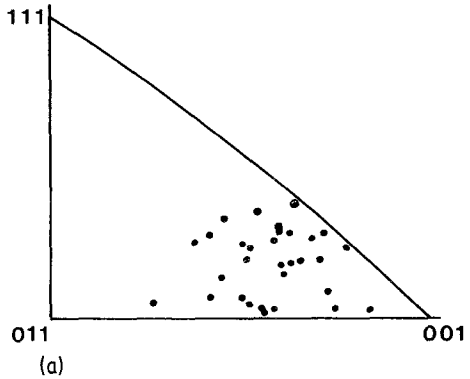
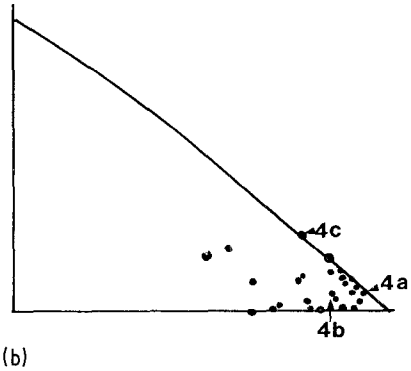


Figure 4 Selected-area channelling patterns from three of the grains imaged in Fig. 3b. (a) and (b) are from grains relatively close to [1 0 0] surface normal while (c) is from a grain rather further from [1 0 0] in orientation. These orientations are marked on to the unit stereographic triangle given as Fig. 5b.

Fig. 5). It is perfectly possible to use such channelling patterns to determine each of the boundary misorientations in the field of view (e.g. [16]). However, the simple interpretation given here confirms the results from the neutron texture experiment, that is that there is a relatively strong [1 0 0] fibre texture.





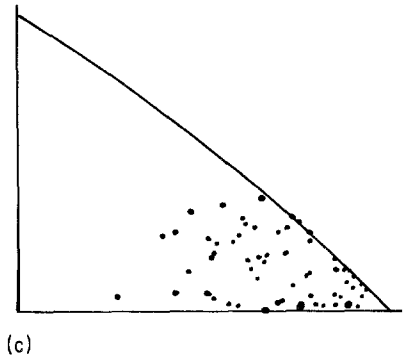


Figure 5 Plots of the surface grain normals for the transverse sections in Fig. 3 inserted into unit stereographic triangles. (a) The orientation data from Fig. 3a; (b) has a similar relationship to Fig. 3b. (c) The combined data from the two plots. The surface grain normals for three grains whose channelling patterns are given as Figs. 4a to c and which are marked on Fig. 3b are arrowed on (b).

Acknowledgements

Financial support from the Risø National Laboratory is gratefully acknowledged, as is the interest shown and guidance given by Dr Niels Hansen.

References

1. I. L. DILAMORE and W. T. ROBERTS, *Met. Rev.* **10** (1965) 271.
2. P. GORDON and R. A. VANDERMEER, "Recrystallization, Grain Growth and Textures" (American Society for Metals, Metals Park, Ohio, 1966).
3. M. HATHERLEY and W. B. HUTCHINSON, "An Introduction to Textures in Metals" (Institution of Metallurgists, London, 1979).
4. H. J. BUNGE and C. ESLING (eds), "Quantitative Texture Analysis" (Deutsch Gesselschaft für Metallkunde, Oberursel, 1982).
5. F. WEINBERG and B. CHALMERS, *Can. J. Phys.* **29** (1951) 382.
6. D. WALTON and B. CHALMERS, *Trans. Met. Soc. AIME* **215** (1959) 447.
7. A. HELLAWELL and P. M. HERBERT, *Proc. R. Soc.* **A269** (1962) 560.
8. G. A. CHADWICK and W. A. MILLER, *Met. Sci. J.* **1** (1967) 132.

9. G. E. BACON, "Neutron Diffraction", 3rd Edn (Clarendon Press, Oxford, 1975).
10. J. SZPUNAR, *Atomic Energy Rev.* **14** (1976) 199.
11. D. JUUL JENSEN and J. K. KJEMS, *Textures and Microstructures* **5** (1983) 239.
12. E. GRANT, D. JUUL JENSEN, B. RAPLH and N. HANSEN, Proceedings of the 7th International Conference on "Textures of Materials", edited by C. M. Brackman *et al.* (Netherland Society for Materials Science, Amsterdam, 1984) p. 239.
13. D. C. JOY, *Microsc.* **103** (1975) 1.
14. F. J. HUMPHREYS, in "Microstructural Characterization", edited by M. Hessel Andersen *et al.* (Risø Press, Denmark, 1984) p. 35.
15. D. J. DINGLEY, in "Scanning Electron Microscopy", edited by O. Johari, in press.
16. S. M. PAYNE and B. RAPLH, work in preparation.

Received 3 June
and accepted 8 July 1985