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Comparison of transmission electron microscopy and optical microstructures in Al–1% Mg after plane strain compression

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Modelling the microstructural evolution of aluminium alloys during hot rolling operations is becoming increasingly dependent on accurate measurements of the deformed microstructure using transmission electron microscopy, scanning electron microscopy and light microscopy. In this paper, an experimental technique which directly compares the microstructure observed in thin foils with that observed using light microscopy is described.

1. Introduction

This work was carried out as part of a project to develop a physically based formulation for models of flow stress and microstructural evolution during single- and multi-pass thermomechanical processing (hot rolling) operations, and describes a technique which was used to determine accurately the longitudinal (or "rolling") direction (LD) in thin foils taken from deformed specimens of Al–1wt% Mg (1050 Al + 1wt% Mg). Specimens of the alloy were tested in plane strain compression (PSC), which is generally accepted as the best way to reproduce the main characteristics of high temperature rolling operations under controlled laboratory conditions [1]. PSC tests were carried out with controlled variations of strain, ε, strain rate, $\dot{\varepsilon}$, and temperature, T. The development of physically based models requires the measurement of subgrain size, δ, dislocation density inside subgrains, ρ_i, misorientation between subgrains, θ, and a detailed understanding of the evolution of these microstructural features with increasing strain [2, 3]. Knowledge of the true orientation of the foil with respect to the as-deformed specimen is necessary but is often difficult to interpret from the thin foil microstructure. In order to gain a more complete understanding of the microstructure and to determine the true orientation of the features observed in thin foils, it is necessary to relate the area of the microstructure observed by transmission electron microscopy (TEM) to that observed in the light microscope, from which the LD is determined. The current paper describes a technique for achieving this.

2. Experimental procedure

Plane strain compression (PSC) testing was carried out under controlled conditions of temperature and strain-rate. Details of the PSC technique are described elsewhere [4, 5]. The results described in this paper are from specimens deformed at a temperature of 305 °C and a strain-rate of 25 s^{-1}.

Fig. 1 is a schematic diagram illustrating the positions from which 3 mm diameter rods were spark eroded from the as-deformed specimens after strains of up to 1.4 (for $\varepsilon > 1.4$, a modified foil preparation technique was used [6, 7]). The rods were then carefully sectioned into discs which were ground to 100 μm thickness. Electropolishing was carried out with a Struers Tenupol-2 electropolisher using a solution of 10% perchloric acid in ethanol/analar methanol at −10 °C with a current of 0.10 A. The thin foils were then washed in methanol and dried before observation by TEM using a Jeol 200CX microscope operating at 200 kV.

The TEM microstructure of the PSC tested specimens consisted of an elongated subgrain structure within the deformed original grains. An area of each foil was selected for detailed analysis of the misorientation across the subgrain boundaries. The orientation of up to 120 adjacent subgrains was measured as described by Duly *et al.* [8] and this enabled the calculation of the misorientations of up to 230 boundaries between subgrains. Low magnification TEM micrographs of

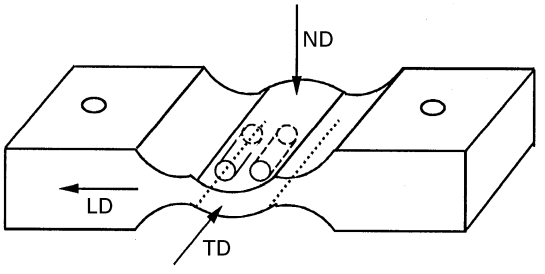

Figure 1 Schematic illustration of the deformed PSC specimen defining the positions from which thin foils were taken for TEM. The normal (ND), transverse (TD) and longitudinal (LD) directions are also indicated.

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the selected area were then taken so that a distinguishing feature (e.g., a precipitate or part of the thin edge of the foil) was included in the montage. After removal from the TEM, the foils were viewed under polarized light and Nomarski conditions using a Polyvar Met Widefield Photomicroscope.

3. Results and discussion

The subgrain structure shown in Fig. 2a was analysed in a thin foil taken from a specimen deformed to a strain of 0.5. A schematic of the microstructure is shown in Fig. 2b. Key features of the deformed microstructure are lines of elongated subgrains which form

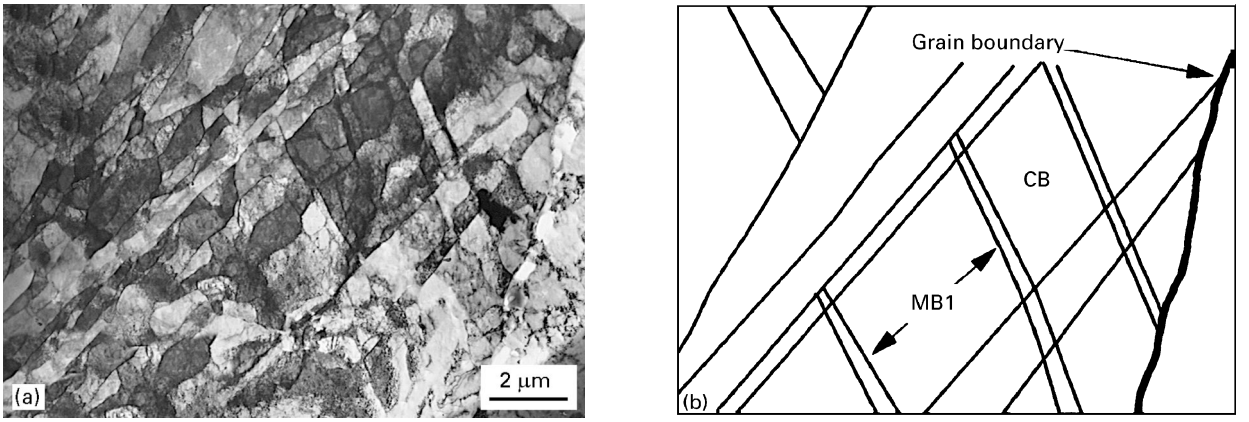
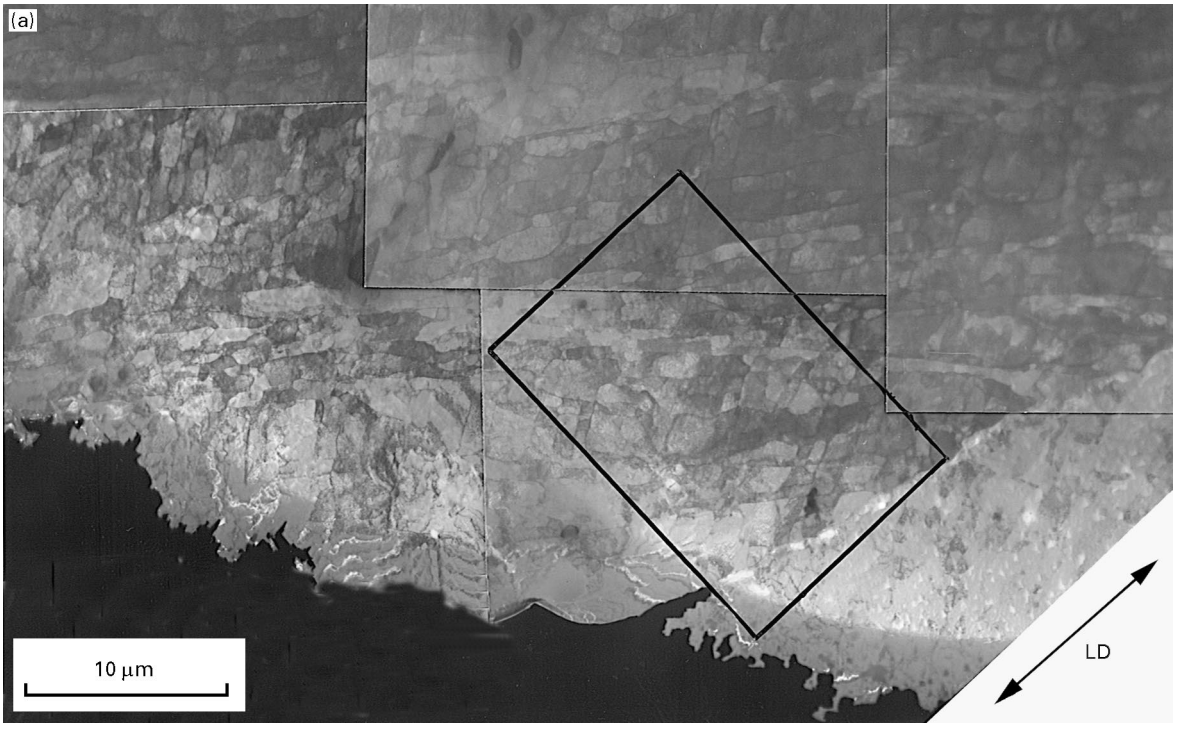


Figure 2 (a) TEM micrograph taken from a specimen deformed at 305 °C, 25 s^{-1} to a strain of 0.5 showing the subgrain structure. (b) Schematic of (a) showing first generation microbands, MB1, and cell blocks, CB.



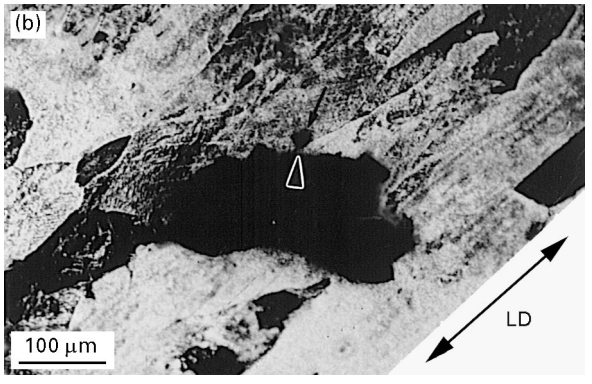


Figure 3 (a) TEM montage taken at lower magnification than in Fig. 2a in which the area analysed in detail is outlined. (b) Optical micrograph of the thin foil taken under polarized light conditions which reveals the grain structure from which LD is determined. Microbands are revealed as faint lines at $\pm 35^{\circ}$ to LD. The area analysed in detail is contaminated by a thin carbon film on the surface of the foil (arrowed).

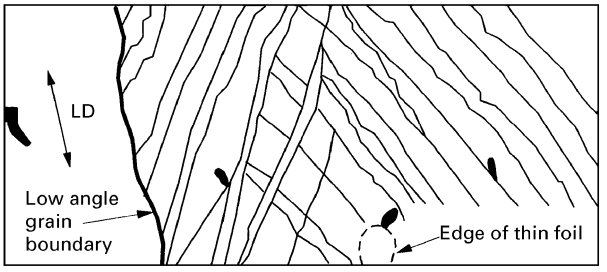
first generation microbands, MB1's, that run through the grains to form cell blocks, CB [9, 10]. The average width of the bands was measured to be 1.2 μm. These features and the misorientations measured between them are described in detail by Duly *et al.* [8]. It was important to establish accurately the orientation of the microbands with respect to LD.

Fig. 3a is a TEM montage which was taken at a lower magnification and includes the area imaged in Fig. 2a. Easily recognizable features, such as part of a grain boundary and the edge of the thin area, are included in the montage. Fig. 3b is an optical micrograph of the thin foil. Preparation of the foil using an ethanol based solution has produced a surface in which there is a difference in contrast between the deformed original grains.

The LD direction is accurately determined in the optical micrograph from the direction of elongation of the grains. The distinguishing features included in the TEM montage allow the alignment of the optical and TEM images (the area of interest in the present case is also marked by a thin carbon film on the surface of the foil (Fig. 3b) which built up during the detailed TEM analysis of the microstructure [8]). The first generation microbands analysed in the TEM (Fig. 2b) occur at $+35^\circ$ and -35° to LD. Careful optical examination of the grain of interest revealed evidence of the microbands as faint lines at $\pm 35^\circ$ to LD, the average spacing (1–1.5 μm) corresponded to the microband width measured by TEM. The microbands were observed to cross the full grain in one direction but only cross a limited region in the other direction (at 70° to the first), in agreement with observations made in the TEM. Similar evidence of microbands within 5° of $\pm 35^\circ$ to LD was found in other grains in the thin foil.

The TEM montage shown in Fig. 4a was taken from a specimen deformed to a strain of 1.0 and electropolished using an analar methanol based solution. This area is drawn schematically in Fig. 4b which highlights microbands occurring in two directions in this grain at 65–70° apart. A grain boundary and part of the thin edge of the foil are also indicated in the schematic. No detail within the deformed grains was revealed on observing the foil under polarized light, however, the features of interest were revealed using Nomarski interference conditions (Fig. 4c). Microbands within 5° of $\pm 35^\circ$ to LD are evident in Fig. 4c in thicker regions of the same deformed grain. Unfortunately the edge of the thin foil was not flat and thus a micrograph showing the exact area analysed using TEM was not obtainable. Despite this, it was possible to reveal the microbands in this area by carefully adjusting the focus.

The observation that microbands occur in deformed grains in many aluminium alloys at characteristic angles to LD is well established [8–13]. The more conventional method by which these features are observed is in specimens which have been polished, etched using Barker's reagent and observed under crossed polars, as shown in Fig. 5 for a specimen deformed to a strain of 2.5. With increasing strain from 0.5 to 2.5 the microbands in Al–1wt% Mg retain



(b)

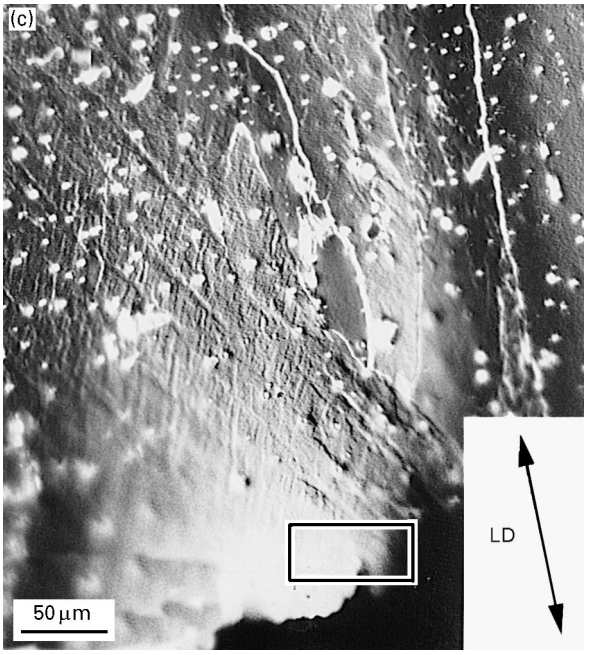


Figure 4 (a) TEM micrograph taken from a specimen deformed at 305 °C, 25 s^{-1} to a strain of 1.0 showing the subgrain structure. (b) Schematic of (a) highlighting microbands which occur in two directions. (c) Optical micrograph taken under Nomarski conditions from which LD is determined and the area shown in (a) and (b) is highlighted. The microbands are revealed more clearly in thicker regions of the thin foil.

a characteristic angle (within 5° of $\pm 35^\circ$) to LD, which implies that the dislocations which make up the boundaries are mobile and that the features are related to the specimen geometry and deformation conditions rather than to the crystallographic orientation of the grains in which they occur [8, 9]. The results of the technique described in this paper have provided an unambiguous link, which proves that the features observed in the TEM and optical microscopes are the same.

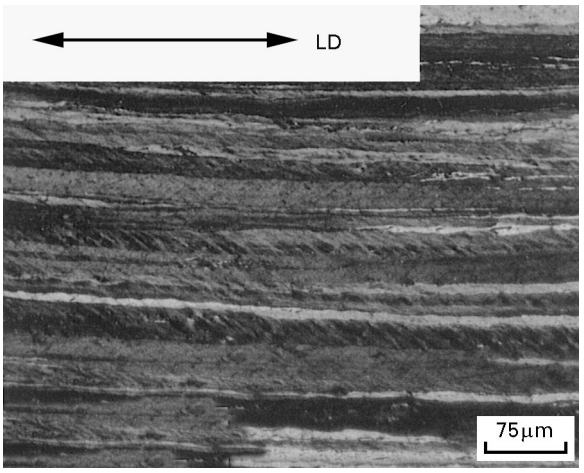


Figure 5 Optical micrograph taken from a specimen deformed to a strain of 2.5 showing microbands which occur in deformed grains within 5° of ±35° to LD.

4. Conclusions

(1) A technique has been developed by which the LD direction may be determined accurately in thin foils of Al–1wt% Mg.

(2) Some contrast between deformed grains was observed using polarized light in foils which had been prepared using an ethanol based solution. In foils which had been electropolished using 10% perchloric acid in analar methanol, the grain structure was revealed using Nomarski interference.

(3) The features remain at the same characteristic angles to LD with increasing strain and are thus determined by the specimen geometry and deformation conditions rather than by the crystallographic orientation of the grain.

Acknowledgements

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References

1. C. M. SELLARS, in Constitutive relationships for metal deformation, Institute of Materials Meeting, London, (1995).
2. G. BAXTER, T. FURU, J. A. WHITEMAN and C. M. SELLARS, in Proceedings 5th Int. Conf. on Aluminium Alloys (ICAA5), Grenoble, France, July 1996, edited by J. H. Driver, B. Dubost, F. Durand, R. Fougeres, P. Guyot, P. Sainfort and M. Suery (Transtec Publications Ltd, Zurich, Switzerland, 1996) part 1, p. 459.
3. T. FURU, H. R. SHERCLIFF, C. M. SELLARS and M. F. ASHBY, *ibid*, part 1, p. 453.
4. C. M. SELLARS, J. HUMPHREYS, E. NES and D. JUUL JENSEN, in Proceedings of the 15th Risø International Symposium on Materials Science, edited by N. Hansen, D. Juul Jensen, Y. L. Liu and B. Ralph (Risø National Laboratory, Roskilde, Denmark, 1994) p. 109.
5. H. SHI, A. J. McLAREN, C. M. SELLARS, R. SHAHANI and R. BOLINGBROKE, *Mater. Sci. Technol.* (in press).
6. P. J. GOODHEW, "Thin foil preparation for electron microscopy", (Elsevier, Oxford, 1985) p. 139.
7. G. J. BAXTER, PhD thesis, Sheffield University, Sheffield (1994).
8. D. DULY, G. J. BAXTER, H. R. SHERCLIFF, J. A. WHITEMAN, C. M. SELLARS and M. F. ASHBY, *Acta Mater.* **44** (1996) 2947.
9. T. LEFFERS and D. JUUL JENSEN, *Textures and Microstructures* **14–18** (1991) 933.
10. B. BAY, N. HANSEN, D. A. HUGHES and D. KUHLMANN-WILSDORF, *Acta Metall. Mater.* **40** (1992) 205.
11. A. KORBEL, J. D. EMBURY, M. HATHERLY, P. L. MARTIN and H. W. ERBSLOH, *Acta Metall.* **34** (1986) 1999.
12. B. BAY, N. HANSEN and D. KUHLMANN-WILSDORF, *Mater. Sci. Engng* **A113** (1989) 385.
13. G. I. ROSEN, D. JUUL JENSEN, D. A. HUGHES and N. HANSEN, *Acta Metall. Mater.* **43** (1995) 2563.

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