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# On Recrystallization in Metal Matrix Composites

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## ABSTRACT

The plane strain deformation of model continuous fibre composites such as Cu-W provides a vehicle for the study of the macroscopic effects of second phase particles on the strain distribution in the matrix and its possible effects on subsequent recrystallization behaviour. By using metallographic studies based both on optical gridding methods and low temperature recrystallization, the pattern of flow enforced by the fibres can be quantified and related to the spatial distribution of recrystallization events.

## INTRODUCTION

The effects of second phase particles, of order 1-5 μm, on deformation and subsequent recrystallization have been examined by a number of authors, particularly in the seminal work of Humphreys and co-workers [1]. These effects are concerned largely with the nature and extent of local strain gradients that occur around hard second phase particles and the local dislocation structures which give rise to particle stimulated nucleation. However, in composite materials containing either particulates or fibres which are in general greater in scale than the grain size of the polycrystalline matrix, a second effect can occur due to the influence of the particles on the macroscopic flow pattern of the matrix during deformation and the influence of this flow pattern on the spatial distribution of subsequent recrystallization events.

It is this latter aspect of deformation which has been considered in the present work by using a model system of 1.0 mm W wires embedded in a polycrystalline Cu matrix with an average grain size of 100 μm. The experiments performed on this system provide a comprehensive overview of the macroscopic deformation of composites at large plastic strains because they permit comparison with finite element method (FEM) calculations of well characterized imposed flow processes and the determination of the spatial distribution of texture both in the deformed and recrystallized conditions.

## EXPERIMENTAL

The composites were fabricated from 1 mm diameter commercial quality as-drawn tungsten wire and 99.99 % copper. The composite was prepared by liquid metal infiltration under vacuum in a modified crystal growing furnace. The tungsten fibres were held in place during the casting by graphite spacers. The distribution of fibres, the sample dimensions and orientation of deformation axes are shown in Fig. 1. In order to refine the as-cast structure in the copper matrix, the composite was deformed in channel die compression with the fibres perpendicular to the die walls. This enabled the deformation to be imposed in a controlled manner and reduced the possibility of void formation at the fibre-matrix interface. The composite was deformed 15 % and then rotated 90 degrees around the fibre axis and deformed a similar amount. This returned the sample to approximately its original dimensions. The samples were then annealed for 30 minutes at 500 °C. This deformation procedure was then repeated and the samples annealed at 400 °C for 30 minutes to further refine the grain structure. The resulting grain size was 50-200 μm (see Fig. 2) so that there were on average 5-10 grains between fibres. The samples were then metallographically prepared and a fine grid was place on the sample face perpendicular to the fibres by the method due to Attwood and Hazzledine [2]. The resulting grid spacing was 23 μm and is shown in Fig. 3a.

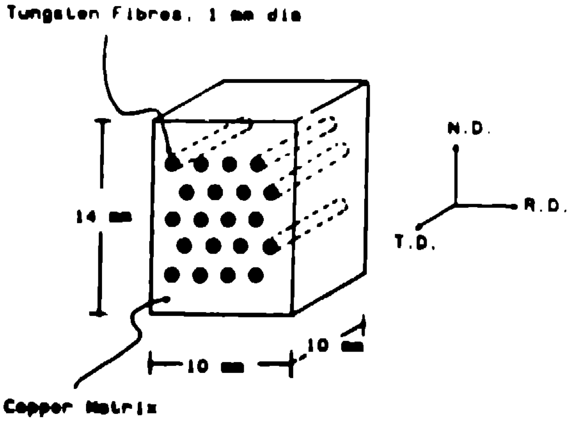


Figure 1. Schematic of sample

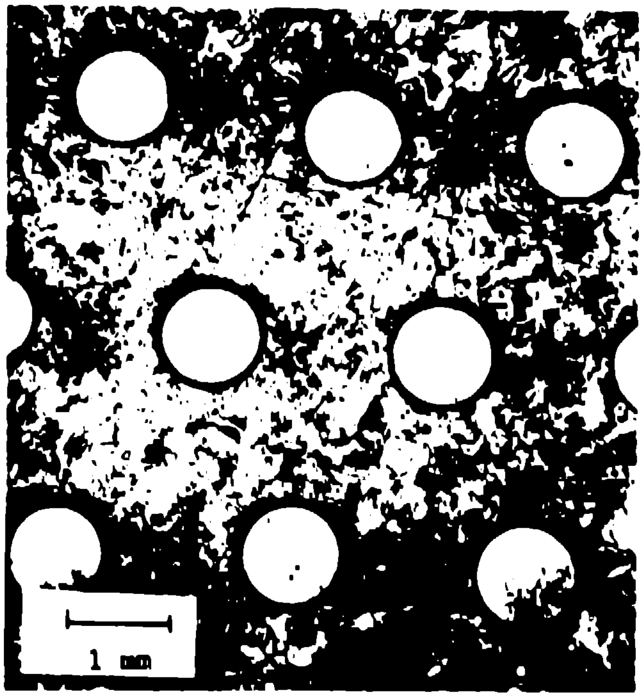


Figure 2. Initial microstructure

After measuring the initial grain size, the composites were deformed in a channel die. Teflon tape with Molykote lubricant sprayed on the side of the tape adjacent to the sample was used to minimize the effects of friction between the sample and die walls. As a result of the lubrication practices, it was observed that the samples deformed in a macroscopically homogeneous manner with the free faces remaining plane. The samples were deformed in 15 % increments to a total deformation of 30%. The teflon lubricant was replaced between strain increments. The sample was then sectioned for metallographic preparation and various sections annealed for different times at 300 °C. The metallographic preparation included grinding up to 4000 grit and light diamond (6 and 3 μm) and alumina (0.3 μm) polishes to reduce surface relief effects between the tungsten and copper. The matrix was etched in an ammonium persulphate solution (10 g ammonium persulphate , 100 ml distilled $H_2O$) for up to 6 minutes.

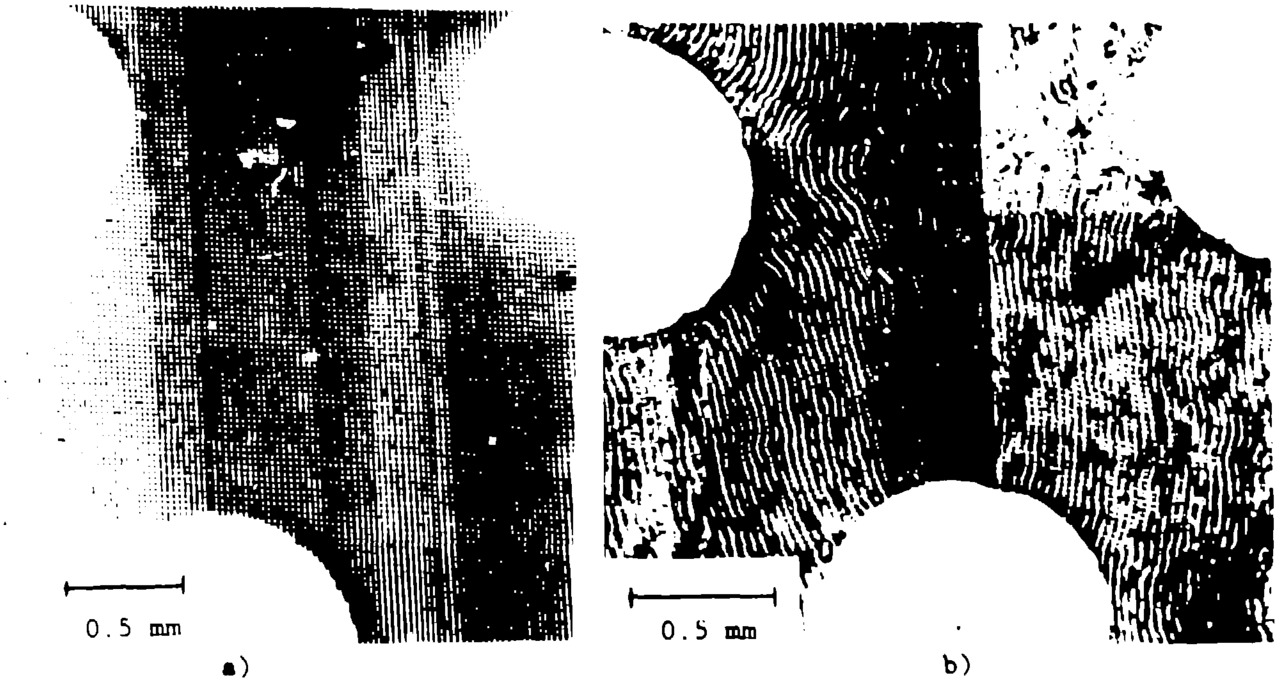


Figure 3. Back scattered electron image of a) initial grid
b) deformed grid (30 %)

## RESULTS AND DISCUSSION

Specimens were gridded and compressed 30% in channel die compression. The form of the surface grids before and after deformation is shown in Fig. 3. The strains were measured by considering the location of the corners of the grids and the changes in location with deformation [3,4]. Errors arise both due to the finite width of the grids and the assumption that no displacements occur perpendicular to the plane of the grid. The measured Von Mises equivalent strain distribution is illustrated in Fig. 4. A finite element method simulation based on ABAQUS was used to calculate the distribution of Von Mises stress and strain for comparison with the experimental data. By inputing an initial flow stress of the copper and tungsten, 20 MPa and 3000 MPa respectively, and a piece-wise linear plastic flow curve for the copper based on pure copper experimental data, the Von Mises stress and strain distributions shown in Fig. 5 are obtained. The comparison with the experimental data in Fig. 4 is very good.

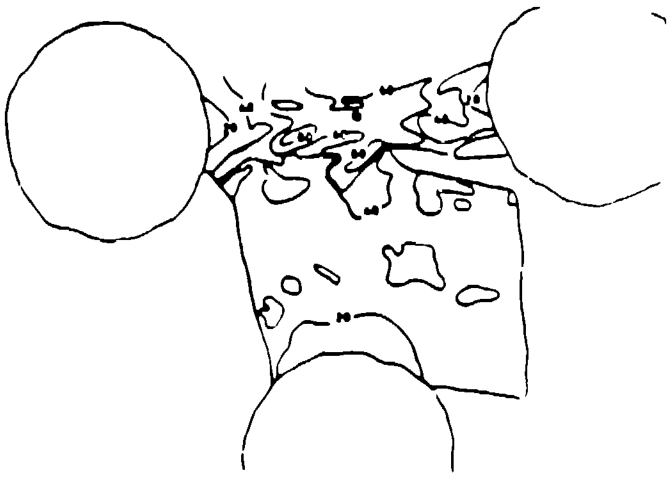
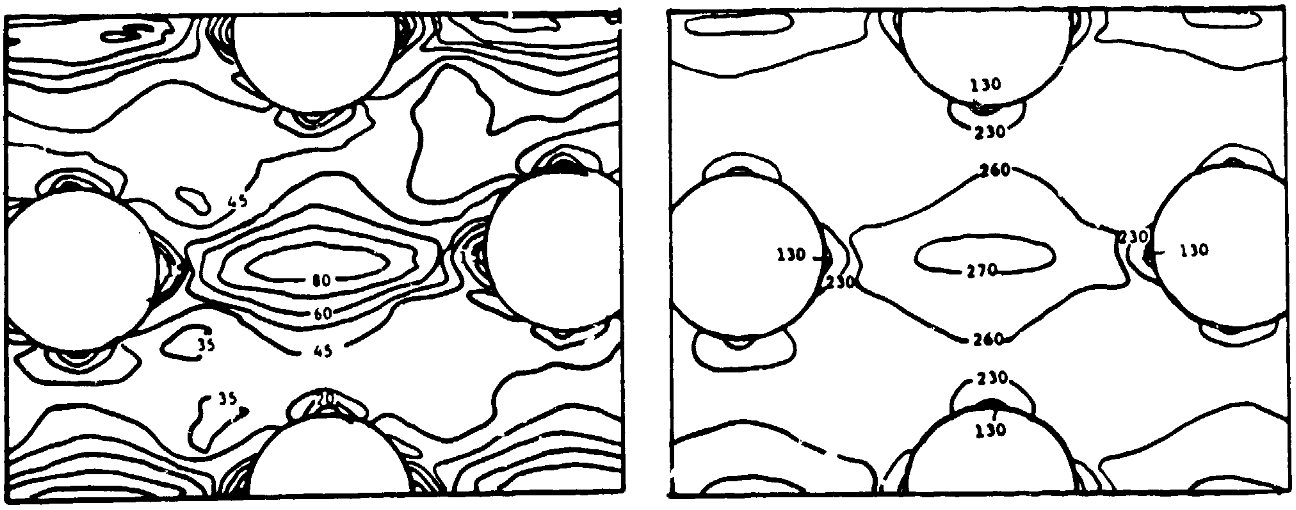


Figure 4. Experimental Von Mises strain distribution

Following deformation, recrystallization studies were carried out at 300 °C for various periods of time in order to assess the spatial distribution of the recrystallization events. In discussing the recrystallization sequence, consideration was given to the role



a) b)

Figure 5. FEM results for 30% deformation a) Von Mises Strain
b) Von Mises Stress (MPa)

of initial grain size on recrystallization. The previous work of Clarebrough et al. [5] and Hutchinson [6] indicates that in fine grained materials the process of recrystallization is accelerated. Thus, a series of metallographic observations were undertaken to ascertain that the influence of variations in grain size in the initial undeformed sample was much less than the effects of the strain distribution enforced by the tungsten fibres.

After annealing for 30 minutes at 300 °C, the pattern of recrystallization is shown in Fig. 6a. It can be seen that there are many small, equiaxed grains in the regions of highest Von Mises strain between the particles: we take these to be recrystallized. Under the particular geometry used these produce bands between the particles perpendicular to the compression direction. A more detailed examination of the distribution of recrystallization events shown in Fig. 6b shows that recrystallization also starts at the edge of the small zone of constrained deformation close to the particles. It is interesting to note that the zones of high shear strain and large rigid body rotations on the diagonal between the fibres are less effective initiating recrystallizatation events.

In rationalizing the experimental observations it is useful to consider that the recrystallization process must depend both on the accumulation of stored energy and of local lattice misorientations necessary to initiate recrystallization. A crude approximation for the distribution of stored energy can be made by considering that the total stored energy due to dislocations is proportional to the square of the flow stress. Thus, contours of the square of the Von Mises stress can be plotted as shown in Fig. 7 and compared with the distribution of initial recrystallization events (Fig. 6a).

This comparison indicates an important difference between the macroscopic composites considered in the current work and the effects of 1 μm size particles involved in particle stimulated nucleation. Recrystallization does not occur at the large macroscopic particles but occurs in the region of amplified Von Mises strain which occurs as a consequence of the pattern of flow imposed by the particles [7]. It should also be noted that the recrystalliztion is accelerated in specific areas of the flow pattern, i.e. in regions of maximum Von Mises strain.

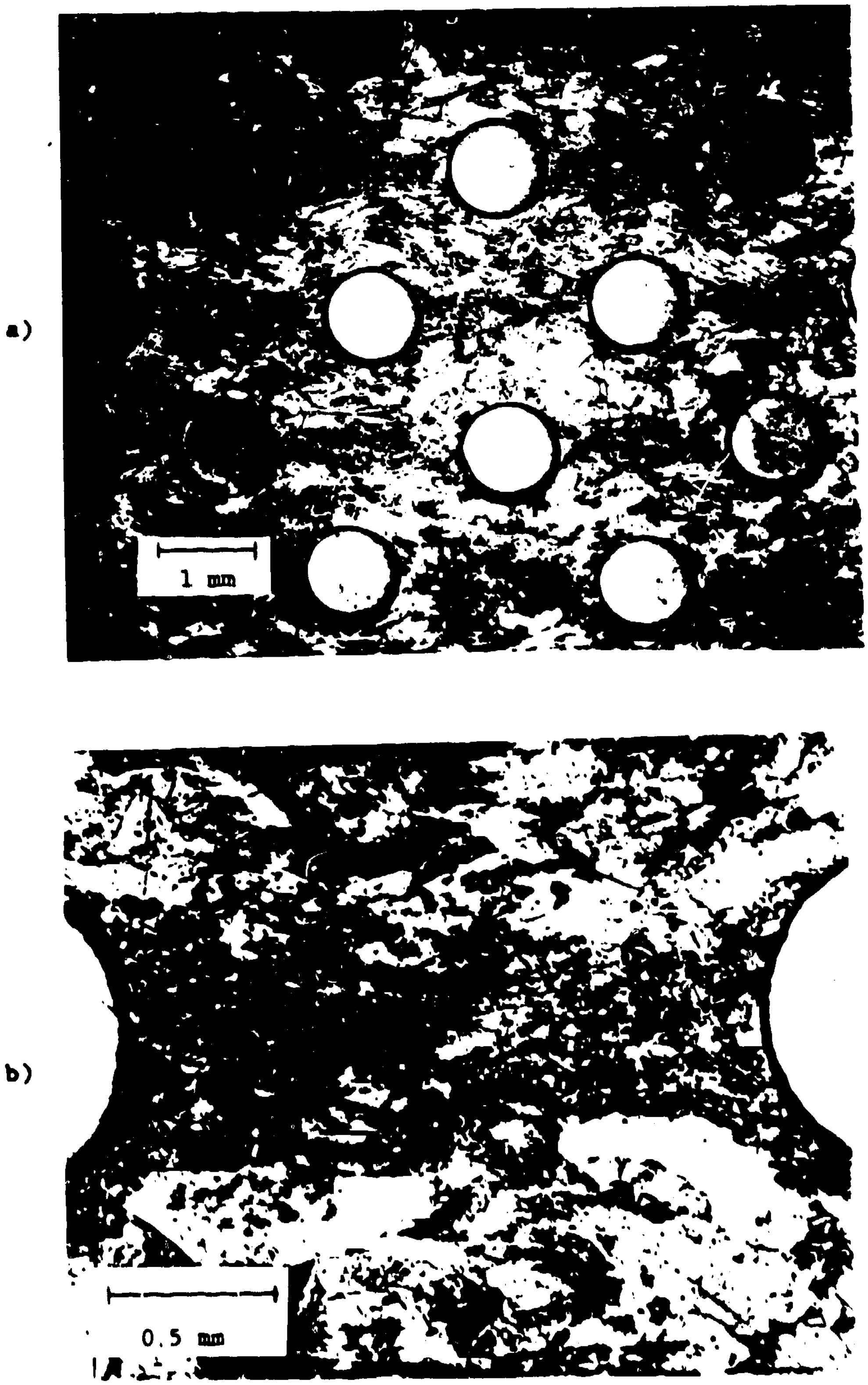


Figure 6. Recrystallization pattern after annealing @ 300 °C for 30 min. a) low magnification b) high magnification

## CONCLUSIONS

The present work indicates the value of model systems for the study of strain distributions and the subsequent spatial distribution of recrystallization events in metal matrix composites. The method outlined allows the pattern of flow to be related to the spatial variation of texture and recrystallization. The studies indicate the importance not only of local strain gradients at particles but of the direct influence of large particles on the pattern of deformation and the spatial variation of stored energy on recrystallization in metal matrix composites.

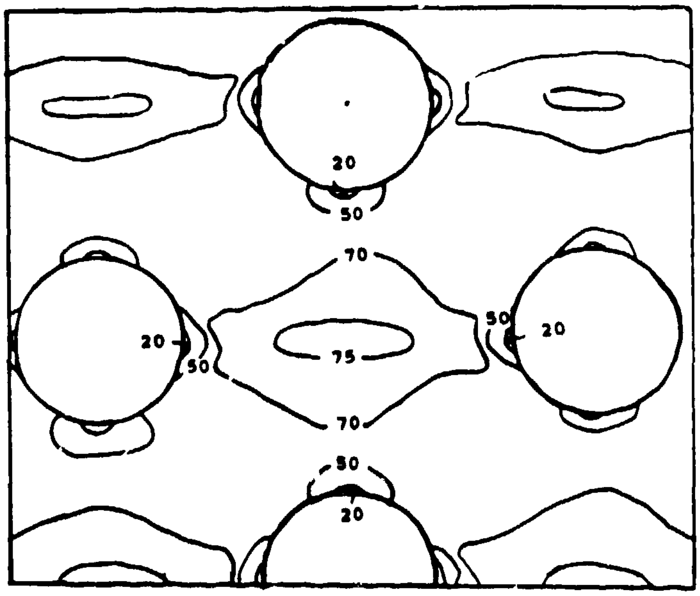


**Figure 7.** The square of the Von Mises Stress (relative units) assumed to be proportional to stored energy

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## REFERENCES

1) F.J. Humphreys and K. Kalu. Acta Met., 38, 917, (1990).

2) D.G. Attwood and P.M. Hazzledine. Metallography, 9, 483, (1976).

3) R. Sowerby, E. Chu and J.L. Duncan. J. Strain Anal., 17, 95, (1982).

4) P.R. Dawson, Personal Communication.

5) L.M. Clarebrough, M.E. Hargreaves and M.H. Loretto. Acta Met., 6, 725, (1958).

6) B. Hutchinson, S. Jonsson and L. Ryde. Scripta Met , 23, 671, (1989).

7) W.J. Poole, U.F. Kocks, R.E. Bolmaro and J.D. Embury. Proc. 12th Riso Int. Symp. on Materials Sci., ed. N. Hansen et al., Denmark, 587, (1991).