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Microstructural aspects of non-homogeneity of grain-boundary sliding

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Direct observation in the scanning electron microscope of grain-boundary sliding (GBS) in Pb–62%Sn eutectic alloy, superplastically deformed in shear, showed non-uniformity of GBS. Such non-homogeneity of GBS reveals itself as sliding of large grain blocks with dimensions of tens of grain size and sliding of grain groups with dimensions of a few (four to eight) grain size. Sliding of large blocks of grains is a result of the sliding of grains as an entity along grain boundaries of former dendritic boundaries. The sliding of grain groups is due to the cooperative manner of GBS. Experimentally observed size of the grain groups can be explained from the view point of cooperative GBS, caused by glide of cellular dislocations.

1. Introduction

Grain-boundary sliding (GBS) is an important mechanism of superplastic (SP) flow [1]. It has been shown [2] that GBS occurs in a non-uniform manner. Generally, non-homogeneity of GBS can be caused either by structural non-uniformity or/and by characteristic features of the GBS process itself. The observed difference in GBS at random grain boundaries and at special grain boundaries [3] can be an example of non-homogeneity of GBS which is due to structural non-uniformity. Preferential sliding along grain boundaries, oriented at 45° with respect to the tensile axis, observed in optimal superplastic conditions [4], is an example of non-homogeneous GBS, caused by the non-uniformity of GBS itself.

These examples show non-homogeneity of GBS at the level of individual grains. Meanwhile, non-homogeneity of GBS can reveal itself also at coarser structural levels, i.e. at the level of grain groups and at the level of the entire deformed volume. Previous investigations of GBS were concentrated preferentially at individual grain boundaries, and non-uniformity of GBS at higher structural levels has not been a focus of the investigations.

In the present work, non-homogeneity of GBS, due to structural non-uniformity as well as that due to non-uniformity of GBS itself, were studied at the level of the entire deformed volume and at the level of grain groups in Pb–62%Sn eutectic alloy, superplastically deformed in shear.

2. Experimental procedure

The extruded Pb–62%Sn eutectic alloy rods were cold rolled to strips of 2 mm thickness (with total 80% reduction in area). Slotted sheet specimens designed for single shear [5] were cut with an electrical discharge machine so that the shear direction was parallel to the rolling direction. Prior to testing, the specimens were stored for 60 days to allow the structure to attain equilibrium at $-9\,°C$ (this temperature was low enough to prevent significant grain growth). One side of the specimen was mechanically polished, and longitudinal and transverse marker lines were inscribed on the polished surface with diamond paste having particle size of 1 μm.

The specimen was deformed inside a scanning electron microscope (SEM) with a constant speed of grip displacement, giving an initial strain rate, $\dot{\varepsilon} = 4.1 \times 10^{-4}\,s^{-1}$ at 300 K. These strain rates and temperatures are close to optimal superplastic conditions [6, 7]. Micrographs were taken from the same place of the deformed specimen at different strain level, using successively higher magnification. Shear strain, γ, has been calculated as a projection of segment AB (see Fig. 1b) in the direction of tension, divided by initial length of segment AB (Fig. 1a). After a shear strain of $\gamma = 2.1$, the specimen was repolished and deformed further to a shear strain of $\gamma = 3$. This was done in order to investigate the surface of specimens at higher strain level excluding the effect of the previous deformation. Optical microscopy was also used to examine the specimen surface after shear deformation at $\gamma = 2.1$ and 3.0.

3. Results

Fig. 1a and b show a schematic illustration of the gauge portion of the single shear slotted sheet specimen before deformation and after deformation, respectively. Initial microstructure is given in Fig. 1c. The gauge portion of the specimen after shear deformation at $\gamma = 1.7$ and 3, are shown in Fig. 1d and e, respectively. Initial grain structure in Fig. 1c looks quite uniform. The average grain size is ~ 3 μm. Meanwhile, the non-homogeneous manner of defor-

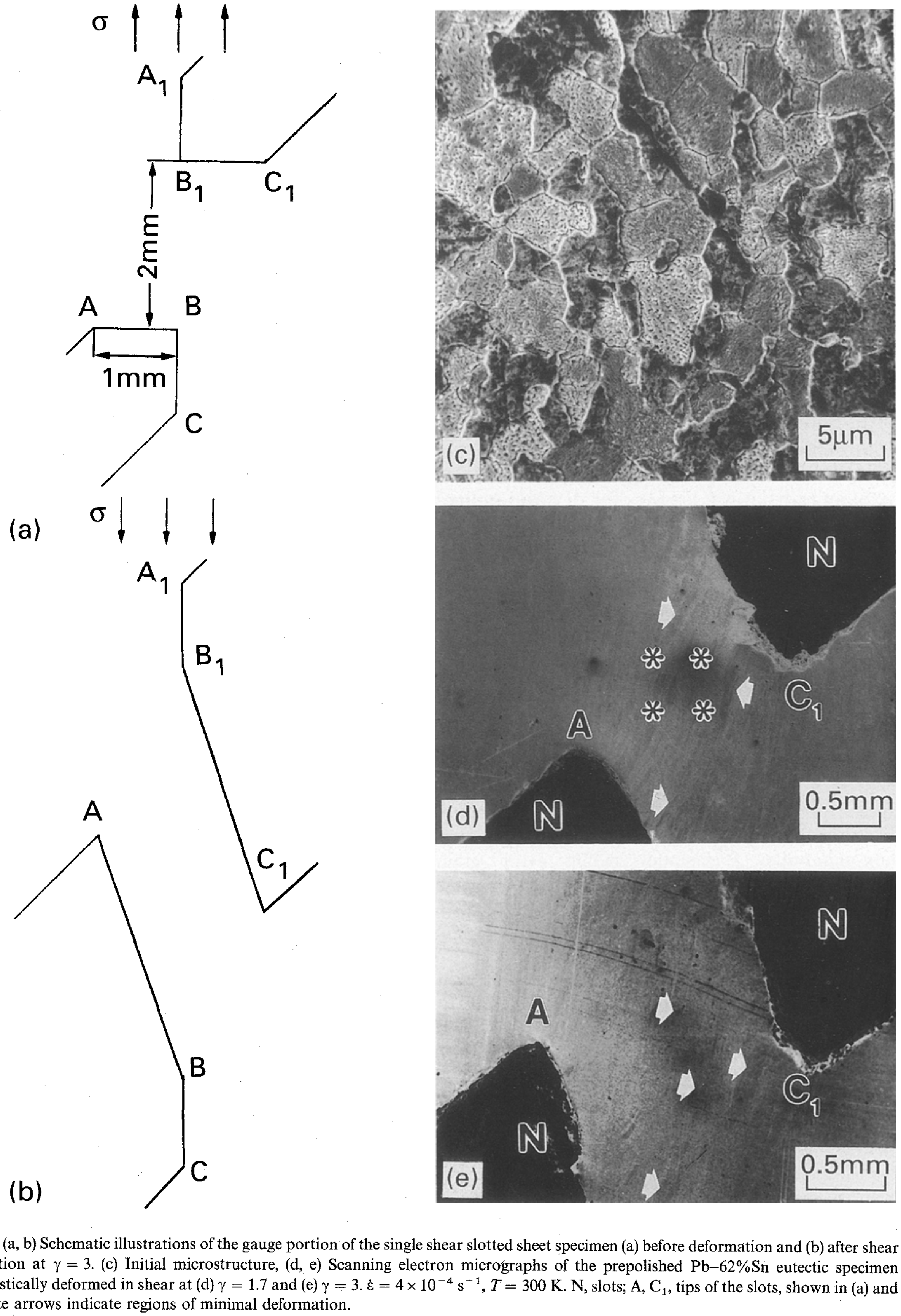


Figure 1 (a, b) Schematic illustrations of the gauge portion of the single shear slotted sheet specimen (a) before deformation and (b) after shear deformation at $\gamma = 3$. (c) Initial microstructure, (d, e) Scanning electron micrographs of the prepolished Pb–62%Sn eutectic specimen superplastically deformed in shear at (d) $\gamma = 1.7$ and (e) $\gamma = 3$. $\dot{\varepsilon} = 4 \times 10^{-4}\ \mathrm{s}^{-1}$, $T = 300$ K. N, slots; A, C_1, tips of the slots, shown in (a) and (b). White arrows indicate regions of minimal deformation.

mation is seen from Fig. 1d and e. Deformation occurs in the region, designated A B_1 C_1 B in Fig. 1b. There is evidence of shear surfaces, along which the shear process proceeds, and undeformed regions between them. Traces of shear surfaces on the prepolished specimen surface appear as curved lines (having a bright contrast) between the notches (black region in Fig. 1d and e, labelled N). Some dark regions (shown by white arrows in Fig. 1d and e) between shear surfaces are seen on the specimen surface both after shear deformation at $\gamma = 1.7$ and 3 (with repolishing at $\gamma = 2.1$). The contrast from these dark regions is close to the contrast of the undeformed portion of the specimen, thereby attesting to the lack of significant deformation in these regions. Fig. 2a and b show the region, squared by stars in Fig. 1d, under higher magnifications. One can see a non-regular shape of the regions with minimal deformation in Fig. 2a. The relative area of these regions (in Fig. 2a) is about 50%. Grain structure, which reveals itself owing to sliding of grains and grain deformation, and breaking up of marker lines, are seen from Fig. 2b. Offsets of the marker lines at grain boundaries are observed in the light regions in Fig. 2b, and appear so due to the bright contrast at the sliding surfaces of grain boundaries. Offset of the marker lines in the right-hand side picture in the montage in Fig. 2b is so frequent and numerous that it is hard to see clearly the marker lines. Meanwhile, segments of the marker lines are clearly seen in regions of small deformation (for example,

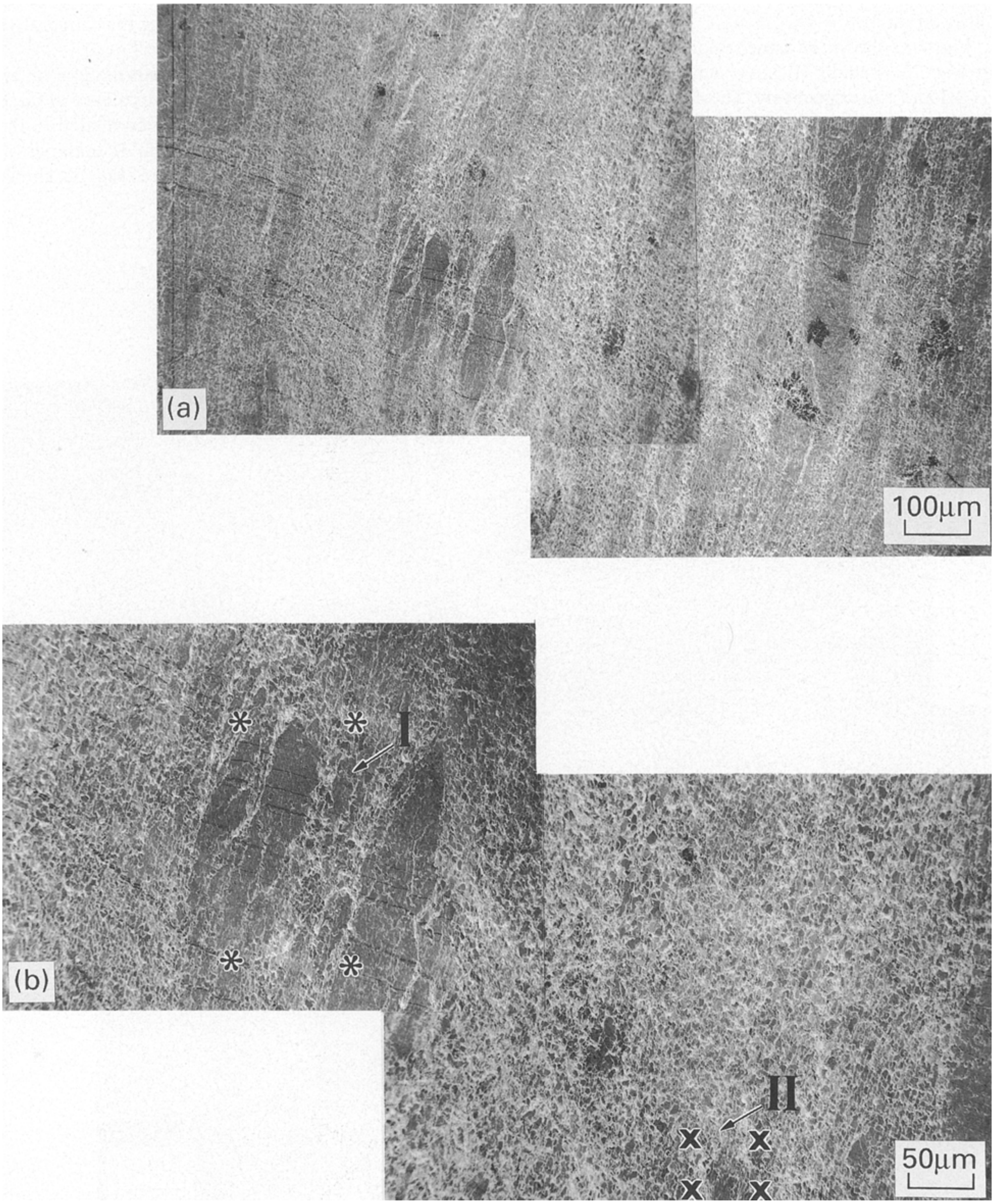


Figure 2 Scanning electron micrographs of the region of the prepolished surface of Pb–62%Sn eutectic alloy (designated by stars in Fig. 1d) after shear deformation at (a) $\gamma = 1.6$ and (b) $\gamma = 1.7$.

Region I in Fig. 2b). The fact that there is no significant breaking up of the marker lines inside regions of small deformation and that the contrast at grain boundaries is not so bright in comparison with grain boundaries inside the light regions, attests to the absence of significant GBS inside these regions. Together with this, clearly evident and significant offset of marker lines is observed at the grain boundaries, which provide the contours of the grain blocks of small deformation. These facts are more clearly seen from the larger montage of scanning electron micrographs (Fig. 3), taken at higher magnification from the region, labelled by stars in Fig. 2b (Region I) after shear strain at $\gamma = 1.1$.

Fig. 4a–c shows the same region, labelled by crosses in Fig. 2b (Region II) after shear deformation at $\gamma = 1.3$ and 1.5, respectively. The dimensions of Region II are much smaller in comparison to dimensions of Region I. The block of grains, shown by arrows along its contour (Fig. 4a) was sliding as an entity, which is apparent from the nature of the breaking up of marker lines.

Mutual displacement of this block of grains, designated U_b, is a result of shear at coplanar grain boundary surfaces (arrowed in Fig. 4b). The amount of the overall offset increases with deformation due to sequential shear along the grain-boundary surfaces that have been active, and as a result of initiation of shear along new surfaces inside this grain block. Operation of new shear surfaces causes the breaking up of marker lines inside this grain block. The length of the new segments, L_g, varies from four to eight grain sizes. The offsets of parallel marker lines are observed in the same direction, thereby indicating the cooperative manner of GBS.

Examination of relief due to deformation in an optical microscope showed that the existence of large grain-block sliding as an entity, can be related to the occurrence of GBS along the grain boundaries of former dendritic cast microstructure. Fig. 5a shows

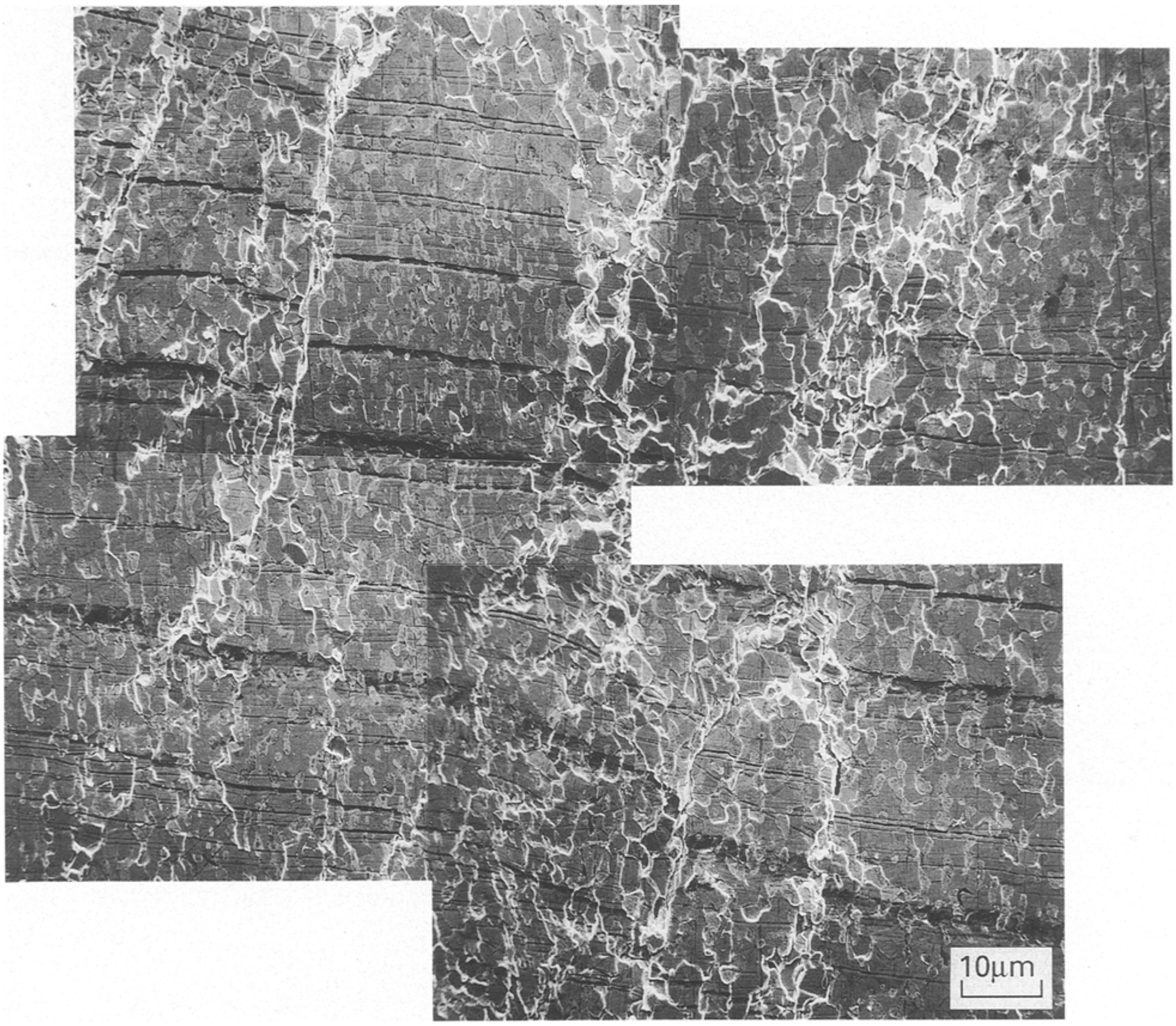
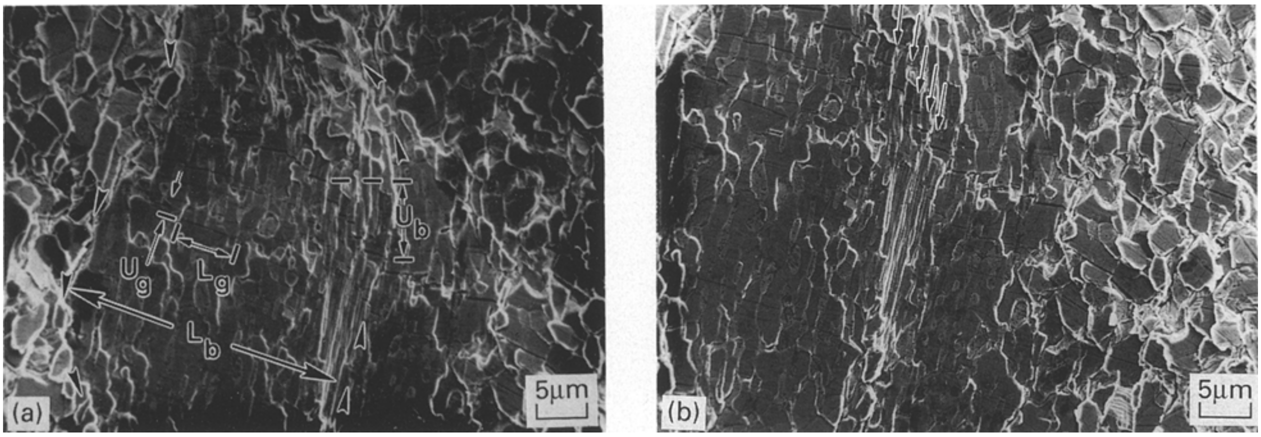


Figure 3 Montage of scanning electron micrographs of Region I (Fig. 2b) of the prepolished surface of Pb–62%Sn eutectic alloy after shear deformation at $\gamma = 1.1$. Offset of marker lines is seen along the borders of grain blocks, sliding as an entity.



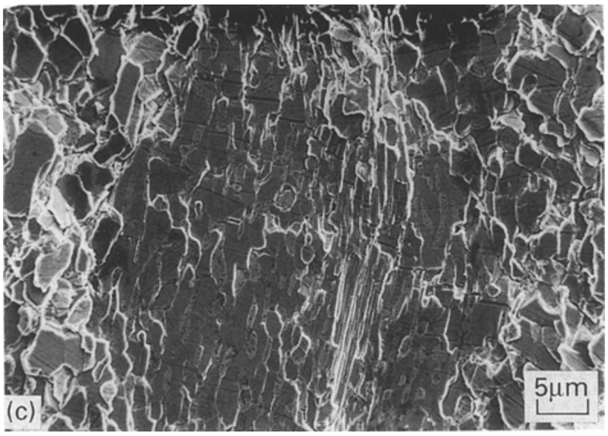


Figure 4 Scanning electron micrographs of the same region (Region II in Fig. 2b) of the prepolished surface of Pb–62%Sn eutectic alloy after shear deformation at (a) $\gamma = 1.3$, (b) $\gamma = 1.4$ and (c) $\gamma = 1.5$. See text for designations.

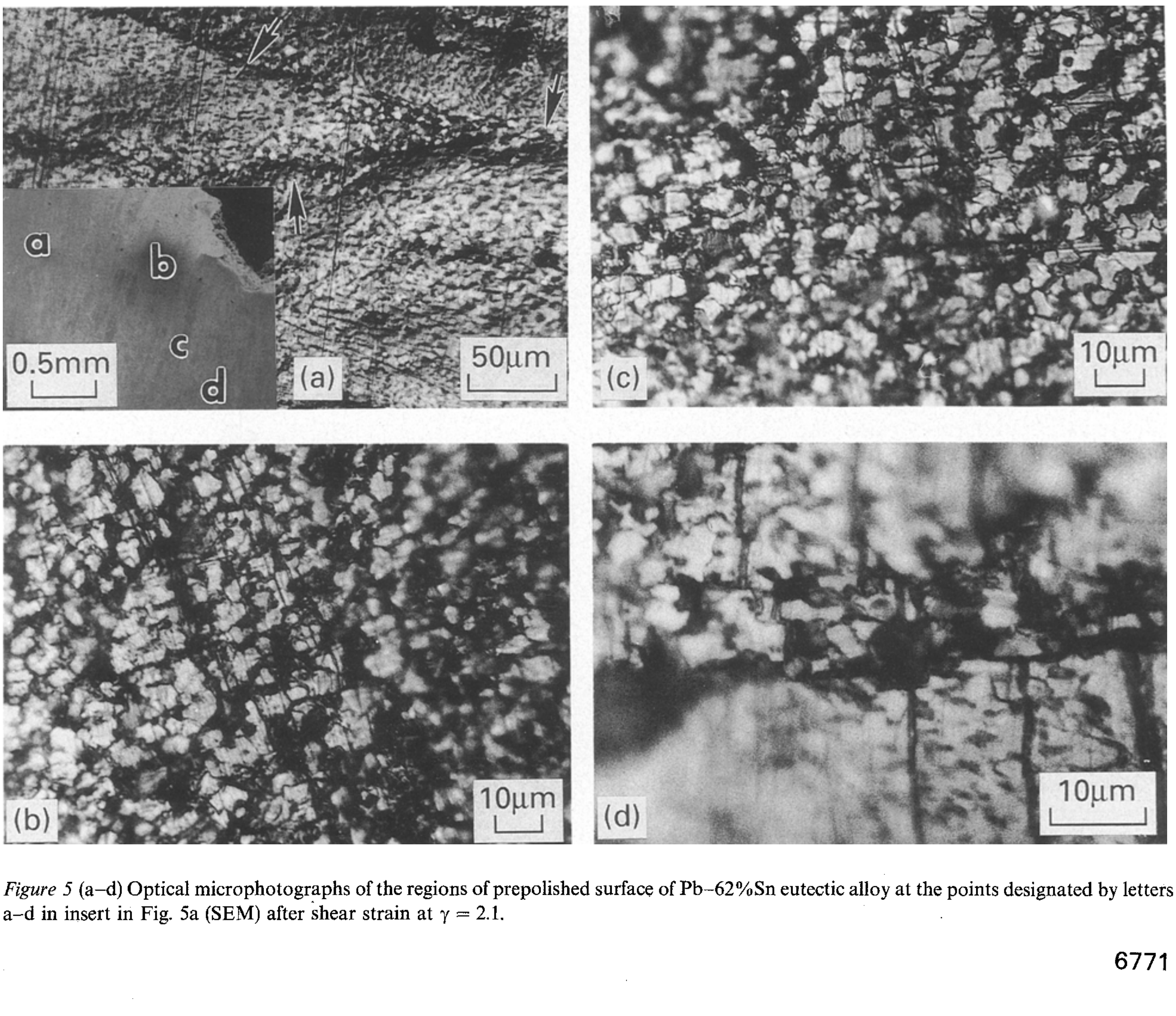


Figure 5 (a–d) Optical microphotographs of the regions of prepolished surface of Pb–62%Sn eutectic alloy at the points designated by letters a–d in insert in Fig. 5a (SEM) after shear strain at $\gamma = 2.1$.

sliding activities along two such former dendritic boundaries (arrowed in Fig. 5a). The insert in the left lower corner in Fig. 5a shows the scanning electron micrograph of the specimen gauge at $\gamma = 2.1$ and the sites from which the optical microphotographs were taken, designated a–d in Fig. 5. Occurrence of GBS along former dendritic boundaries is more clearly seen in a portion of the sample, having been deformed lightly (Fig. 5a) or from Fig. 5d, taken at grain boundaries, along which the large grain block slid. Operation of shear surfaces inside such large grain blocks divides them into smaller grain groups as illustrated by Fig. 5c and d, taken from the specimen gauge.

4. Discussion

The experiments reported here showed non-homogeneity of GBS. There is evidence of large grain blocks, having dimension (L_b in Fig. 4) of tens of grain diameters, sliding as an entity. These blocks eventually are divided into smaller grain groups, having dimensions (L_g in Fig. 4) of several grain diameters due to initiation of shear surfaces within the blocks when strain increases. The reasons for the occurrence of SP deformation in such a non-homogenous manner, can be visualized as reported below.

4.1. Existence of large grain blocks

There is structural non-homogeneity due to the existence of former dendritic boundaries, arising from the erstwhile cast microstructure, even though initial microstructure of the specimen appears apparently homogeneous (Fig. 1c). Indeed, grain boundaries associated with the former dendrites differ from the other grain boundaries. Their chemical composition differs due to dendritic coring and the higher concentration of impurities. Furthermore, there is also a difference in the structure of these two types of boundaries. The dendritic boundaries are often of random type, while there is a higher proportion of special boundaries inside the grain blocks [8]. Subsequent plastic deformation during thermomechanical treatment can increase the amount of random boundaries (in relation to special boundaries) inside the grain block [9]. However, such an increase will still leave the dendritic boundaries possessing a larger proportion of random boundaries. It is likely that due to these differences, the boundaries of the former dendrites are more non-equilibrium in nature than the other boundaries. Because non-equilibrium grain boundaries are more prone to sliding [10], GBS is initiated at the former dendritic boundaries.

Thermomechanical treatment (rolling at room temperature) creates a recrystallized microstructure; however, such mechanical treatment cannot significantly change the chemical composition. This results in movement of all grains, surrounded by the former dendrites grain boundaries as a block. The total amount of GBS along former dendritic boundaries is significant (see U_b in Fig. 4a).

4.2. Existence of grain groups

Shear at grain boundaries, inside the former dendritic block divides the large grain blocks, which used to have macroscopical dimensions, into smaller grain groups (Fig. 4). The number of such shear surfaces increases with strain (the length of segments of marker lines, L_g, decreases with deformation (Fig. 4), which can be the result of strain hardening of active shear surfaces. It can be suggested from the offset of parallel marker lines at grain boundaries, through which shear surfaces propagate (with bright contrast in Figs 2–4), that there is a long-range cooperation in GBS. Morral and Ashby [11] analysed such cooperative grain-boundary sliding in terms of movement of cellular dislocations, considering mostly, topological aspects. Recently [12], the concept of such dislocations has been extended to the case of deformation-induced cellular dislocations and in application to the case of two-phase materials [13]. It has been suggested [12, 13] that grain-boundary sliding along a shear surface proceeds as a result of sequential shear of grains. In the context of incomplete shear, the boundary delineating the separation between the shifted and undisturbed part of the polycrystal can generally be considered as an edge cellular dislocation (Fig. 6). Glide movement of such a cellular dislocation shifts an extra row of grains (from point A to point B in Fig. 6) resulting in relative shear of a group of grains. There is stress field around the cellular dislocation because of the compression of grains, situated near the extra row of grains (extra plane of grains in three dimensions). The calculation of the elastic stress field around a cellular dislocation (especially in two-phase materials, considered here), has not yet been carried out nor has an investigation of the interaction between cellular dislocations. If the distance at which elastic stress decreases rapidly is assumed to be equal to two or four grain sizes, it is possible to explain the experimentally observed dimension of grain groups, L_g, to be equal to four to eight grain diameters.

The spacing of active shear surfaces of grain groups, L_g, decreases with deformation (repolishing at $\gamma = 2.1$ allowed us to exclude measurement ambiguities,

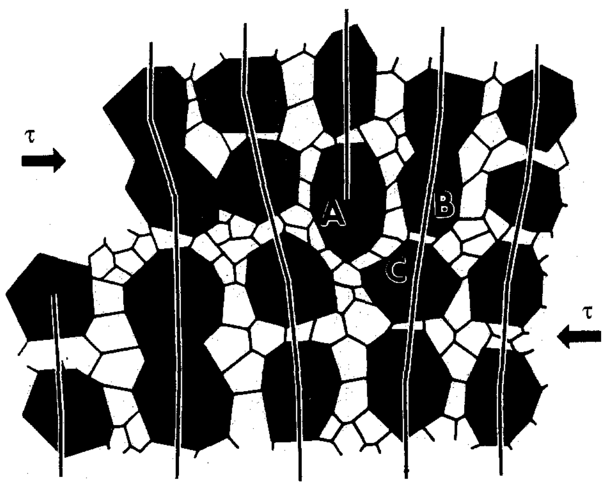


Figure 6 Schematic illustration of cellular dislocation in a two phase material.

which are due to the existence of offset of marker lines, occurring at previous strain levels). It should be noted that the accommodation of grain-boundary sliding inside grain groups, providing compatibility of their movement, also results in offset of marker lines within grain groups. Both the decrease of L_g with deformation, and accommodation GBS inside the sliding grain groups, result in the observation of segments of marker lines with a length of less than four grain diameters (which reflects the size of the grain group). Thus, the non-homogeneity of GBS reveals itself at the level of the entire deformed volume (as sliding blocks of grains) and at the level of grain groups in addition to one observed at the level of individual grains [2–4].

This investigation also indicates that a phenomenon exists in which grain-boundary structure inherited from a former, i.e. "ghost", microstructure can affect superplastic flow [14].

5. Conclusions

1. Grain-boundary sliding (GBS) in Pb–62%Sn eutectic alloy superplastically deformed in shear occurs in a non-uniform manner. Non-homogeneity of GBS reveals itself as sliding of large grain blocks of macroscopical dimensions (of tens of grain sizes) at the level of the entire deformed volume and as sliding of grain groups with dimensions of several grain sizes (four to eight) at the level of the grain groups.

2. Sliding of large blocks of grains is shown as a result of inherited microstructural nonuniformity. Grains slide as an entity along grain boundaries of former dendrites which, because of their non-equilibrium nature, are more prone to sliding in comparison to the other grain boundaries.

3. Sliding of grain groups is due to the cooperative manner of GBS. Grain groups slide along shear surfaces, formed by segments of sliding grain boundaries. The experimentally observed size of grain groups can be explained from the point of view of cooperative GBS, caused by glide of cellular dislocations.

Acknowledgement

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