Measurements of spacing of sliding grain boundaries

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The spacing of grain boundaries at which grain boundary sliding (GBS) had occurred during superplastic (SP) deformation was determined by measuring the length of segments of marker lines inscribed on the pre-polished surface in Pb–62%Sn after superplastic deformation in shear. Statistical distribution of this segment length (L) was bimodal at low strain levels, but became unimodal at high strain levels. The concept of cooperative GBS, i.e. sliding of groups of grains as an entity, has been invoked to explain the evaluation of the L-distribution with strain. This investigation suggests that the real spacing of sliding grain boundaries should be taken into account for modelling of SP flow.

1. Introduction

Grain boundary sliding (GBS) is one of the major processes of deformation at elevated temperatures, particularly under superplastic conditions [1, 2]. Much of the work dealing with this phenomena has been centered on measurements of GBS in superplastic (SP) materials, for example, by using the fiducial marker lines technique [3, 4]. According to this technique, offsets of fine marker lines (inscribed on the polished surface before deformation) at grain boundaries are measured [3–7]. Deformation due to GBS, ε_{GBS}, is determined as [3]:

$$\varepsilon_{GBS} = \alpha \bar{U}_{GBS}/d \quad (1)$$

where $\bar{U}_{GBS}$ is an average marker lines offset and d is an average grain size. The value of the geometrical coefficient, α, is dependent on whether the inscribed marker lines are parallel or transverse to the deformation axis [7]. Simple averaging of GBS through all grain boundaries for determination of $\bar{U}_{GBS}$ assumes a priori homogeneous progress of GBS. In the case of such uniform GBS, average length of marker line segments L, is approximately equal to the average grain size, i.e. $L \approx d$. In general, GBS might occur in an inhomogeneous manner. For the case of the inhomogeneous GBS the spacing of sliding grain boundaries is $L > d$. It has been noted in some studies [8, 9] that marker lines are not broken up (i.e. displaced) at all grain boundaries. However, while accurate measurement of U_{GBS} values have been performed in various materials [3–7], the measurement of the spacing of grain boundaries at which GBS took place have not been measured thoroughly.

In this paper the spacing of sliding grain boundaries (the length of marker line segments between adjacent offsets) has been measured in superplastic Pb–62%Sn alloy, deformed in shear.

2. Experimental procedure and results

Details about the preparation of slotted specimens designed for single shear [10] and of deformation *in situ* in scanning electron microscope (SEM) are given elsewhere [11]. The experimental conditions, strain rate, $\dot{\varepsilon} = 4 \times 10^{-4}\,s^{-1}$ and temperature, $T = 300$ K, were close to optimum SP conditions [12, 13]. Marker lines were inscribed at the pre-polished surface with diamond paste, having a particle size of 1 μm, in directions perpendicular and parallel to the stress axis. Offsets of marker lines and length of segments in which marker lines are broken due to GBS (spacing of sliding grain boundaries), L, were measured from SEM micrographs by using an image analyser. The results of the measurements of marker lines offsets, in particular spatial distribution of marker lines offsets, are given in Ref. [11]. Here, the results of the measurements of the length of marker line segments are presented. The maximum screen resolution was 0.1 μm. The area analysed was 0.5×0.5 mm^2, which covered ca. 4×10^4 grains having an average size of ca. 2.5 μm.

Fig. 1 shows a part of the deformed portion of specimen at shear strain $\gamma = 1.1$. An insert in the upper right corner of Fig. 1 demonstrates a larger deformed area under low magnification. An insert in the lower left corner represents the specimen fixed in the device used for deformation in a SEM (at $\gamma = 2.1$). The segment AB represents the initial position of the edge of a slot; AB_1 shows the edge of the slot after strain by $\gamma = 1.1$. Macroscopical shear deformation occurred through the operation of shear surfaces, which appear as bright lines in Fig. 1 (indicated by arrow heads). Dark regions surrounded by shear surfaces are regions of a small deformation (indicated by thick arrows). Light regions are regions of large deformation (this is seen, for example, from the significant inclination of a macroscopical marker line CD, which

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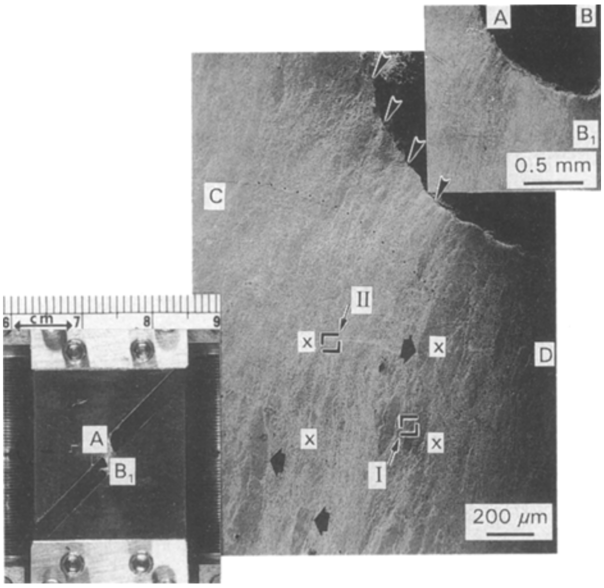

Figure 1 The pre-polished surface of Pb–62%Sn, superplastically deformed in shear by $\gamma = 1.1$, $T = 300$ K and $\dot{\varepsilon} = 10^{-4}$ S^{-1}. The insert in the right upper corner shows the deformed portion of the specimen under low magnification. AB and AB_1 show the initial position and the final position of the edge of a slot (the black region), respectively. Letters C and D mark a macroscopical marker line (traced by a dotted line). Arrow heads point to surfaces of shear; thick arrows point to regions of a small deformation.

was initially straight, in light regions). The trace of the marker line CD is emphasized by the dotted contour line drawn parallel to the marker line CD. Figs 2a and c and 3 show the details of marker lines offsets in a region of a small-deformation (region I in Fig. 1) and in a region of large deformation (region II in Fig. 1), respectively. Significant offsets of marker lines at grain boundaries indicate grain boundary sliding as one of a dominant mechanism for progress of the shear process [11]. It is important to note that sliding occurs through cooperation of grains in certain groups which slide as an entity. The fact that marker line offsets are observed only at certain grain boundary surfaces (for example, shown by arrow heads in Fig. 2a) clearly indicates sliding of grain groups. GBS results in breaking up of marker lines in segments of various lengths in the regions of both large and small deformation.

Fig. 4 demonstrates statistical distribution of length of marker line segments, L, in the region designated by crosses in Fig. 1, which contain regions of both large and small deformation. Statistical distribution of L-value is bimodal at low strain. Two peaks in the L-distribution reflects existence of regions of large deformation and regions of small deformation. The L-distribution becomes unimodal at high strain level. The mean L-value decreases with strain. Fig. 5 illustrates the evolution of L-distribution with strain in a region of small deformation (region I in Fig. 1). Schematics of the offsetting of some marker lines, visible in Fig. 2a to c, are given in the right upper corner in Fig. 5a to c. The curved lines show schematically surfaces of shear (as an example, small arrows in the schematic illustration given in Fig. 5a point to the same shear surfaces which are indicated by arrow heads in Fig. 2a). The asymmetry of L-distribution as

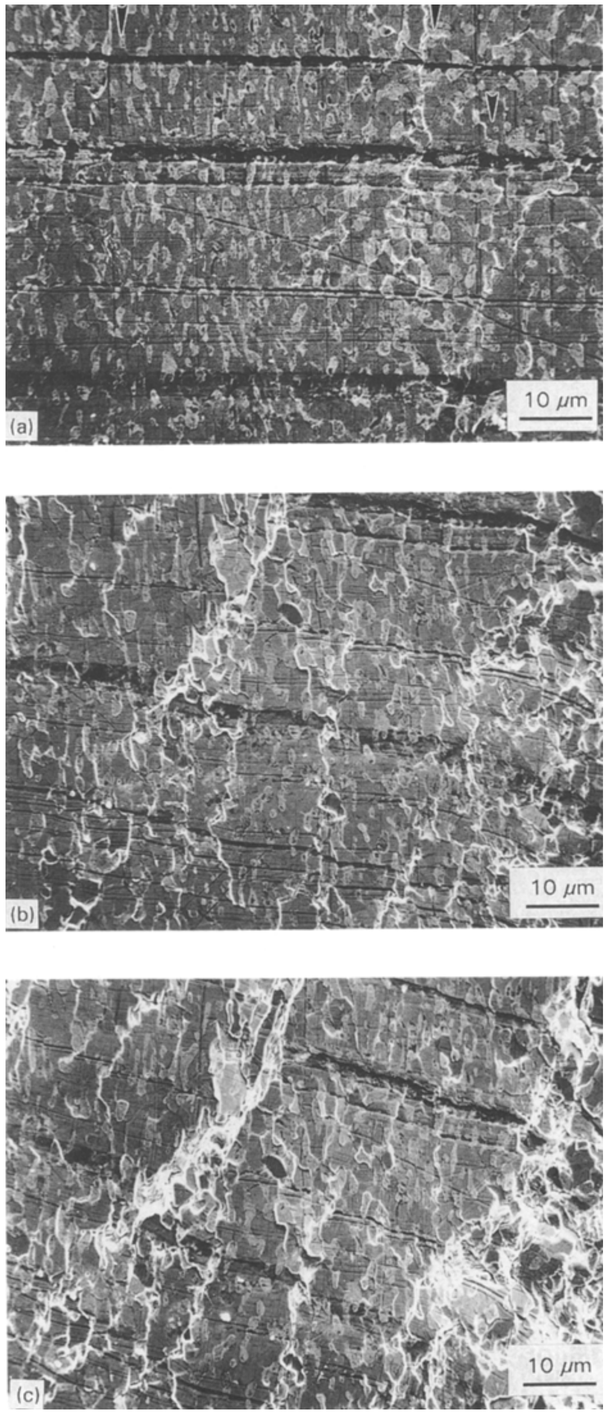

Figure 2 SEM micrographs taken from region I in Fig. 1 at successively higher strain levels: (a) $\gamma = 0.4$; (b) $\gamma = 0.8$; (c) $\gamma = 1.4$ of Pb–62%Sn at $T = 300$ K. Arrow heads in (a) point to grain boundary surfaces at which GBS took place.

well as the L-value decreases as strain increases. This suggests that deformation is becoming more uniform, i.e. larger amount of grain boundary surfaces are involved in GBS process. Fig. 6 shows L-distribution in the region of large deformation (region II, Fig. 1) at strain level $\gamma = 1.1$. The schematic illustration of some marker line offsets, visible in Fig. 3b, is given in the right upper corner in Fig. 6. The curved lines show some surfaces at which significant offsets of marker lines are observed. Mean length of marker line segments, $\bar{L}$ is approximately equal to average grain intersect, i.e. $\bar{L} \approx d \approx 2.5$ μm. Meanwhile, the exist-

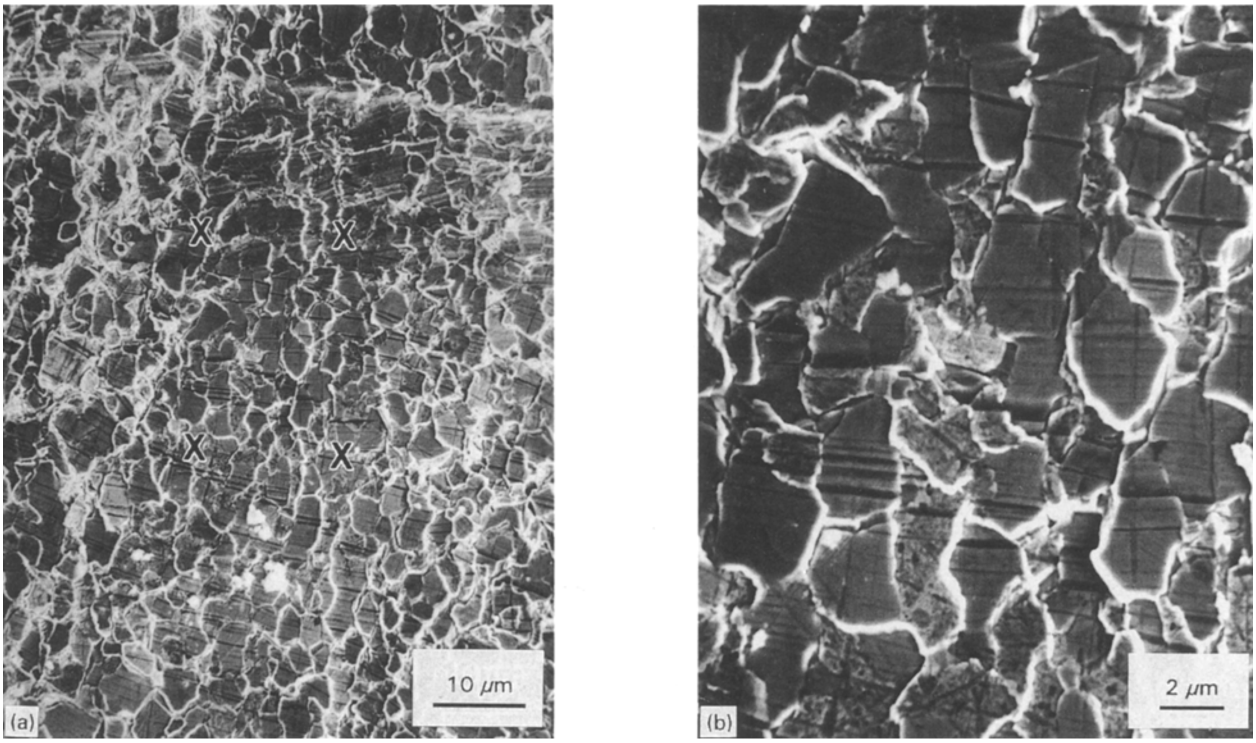

Figure 3 SEM micrographs taken from the region II in Fig. 1. (b) Represents the region marked by crosses in (a) at a higher magnification. Pb–62%Sn, $T = 300$ K and $\gamma = 1.1$.

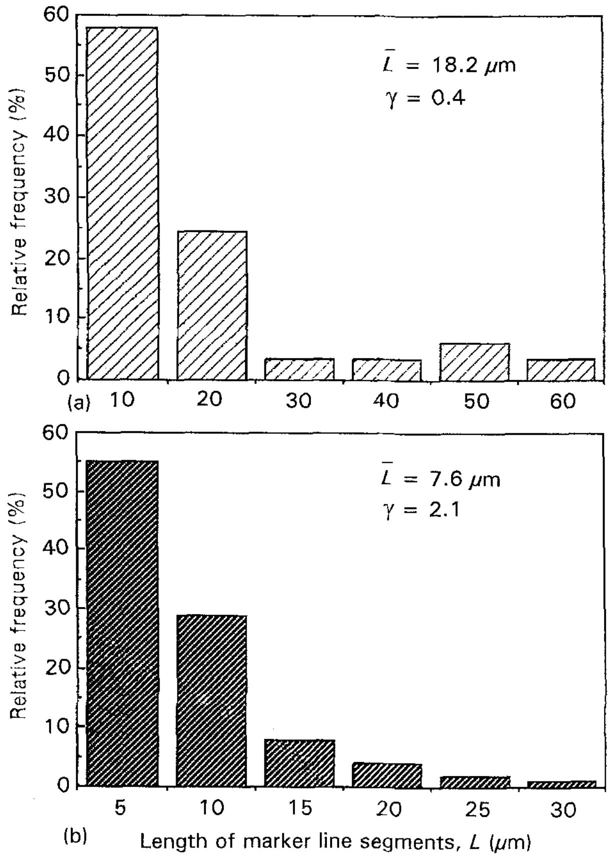

Figure 4 Statistical distribution of the length of marker lines segments at the strain levels $\gamma = 0.4$ (a) and $\gamma = 2.1$ (b).

ence of $L > \bar{d}$ indicates sliding had not taken place at all grain boundaries.

3. Discussion

The length of marker line segment reflects the dimension of sliding grain groups. For the case when sliding occurs at all grain boundaries, $\bar{L} = \bar{d}$. Generally, spacing of surfaces of sliding grain boundaries, as demonstrated in these experiments, might be more than grain size $L \geqslant d$. Taking into account the possibility of nonhomogeneous GBS, Equation 1 can be presented in the following form:

$$\varepsilon_{GBS} = \alpha \bar{U}^{a}_{GBS}/\bar{L} \qquad (2)$$

where $\bar{U}^{a}_{GBS}$ is a mean value of actual offsets of marker lines, not equal to zero.

Both Equations 1 and 2 give the same value of ε_{GBS}. However, generally $L \geqslant d$ and $\bar{U}^{a}_{GBS} \geqslant \bar{U}_{GBS}$. As a result, the rate of GBS determined from $\bar{U}^{a}_{GBS}$, $\dot{U}^{a}_{GBS} = \bar{U}^{a}_{GBS}/\Delta t$ and from $\bar{U}_{GBS}$, $\dot{U}_{GBS} = \bar{U}_{GBS}/\Delta t$ (Δt being a time interval of deformation) may not be the same: $\dot{U}^{a}_{GBS} \geqslant \dot{U}_{GBS}$. Averaging of U_{GBS} through all grain boundaries leads to the underestimation of the actual GBS rate. This fact needs to be considered when predictions of theoretical models of SP, which assume GBS as a rate-controlling mechanism, are compared with the results from experiments. Furthermore, L can be a function of stress, σ, temperature, T, and grain size, d: $L = L(\sigma, T, d)$. A constitutive equation for this case can be written (using the standard designations) as [14]:

$$\dot{\varepsilon} = \dot{\varepsilon}^{a}_{GBS} \cdot \frac{d}{L} = \frac{Ad}{L(\sigma, T, d)} \cdot \frac{D}{kT} Gb \left(\frac{b}{d}\right)^{p} \left(\frac{\sigma}{G}\right)^{n} \times \exp(-Q/RT) \qquad (3)$$

where $\dot{\varepsilon}^{a}_{GBS}$ is the actual strain rate due to GBS: $\dot{\varepsilon}^{a}_{GBS} = \dot{U}^{a}_{GBS}/d$. p, n and Q are parameters characteristic of the microscopical mechanism of GBS. Theoretical models of GBS give values of these parameters from

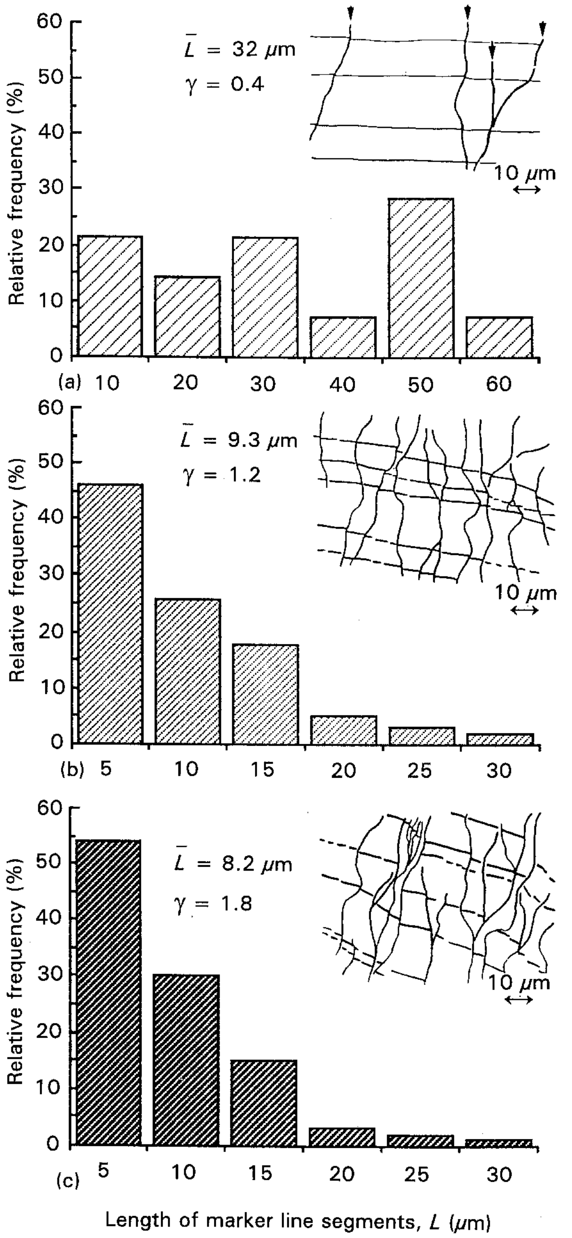

Figure 5 Statistical distribution of the length of marker lines segments in the region of a small deformation (region I in Fig. 1) at successively higher strain levels: (a) $\gamma = 0.4$; (b) $\gamma = 1.2$; (c) $\gamma = 1.8$. Schematic illustrations in the right upper corner show some marker lines offset at shear surfaces (depicted by curved lines) visible in Fig. 2a to c.

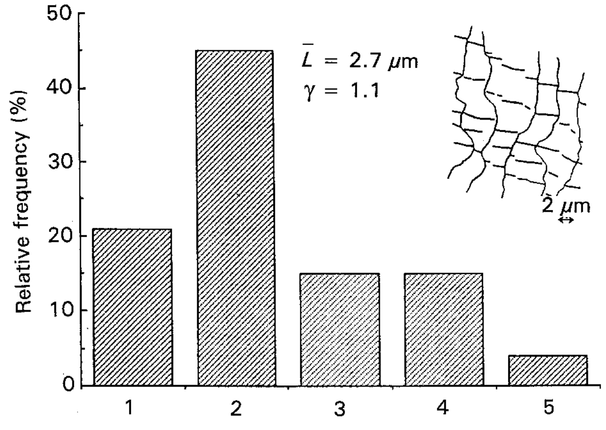

Figure 6 Statistical distribution of the length of marker lines segments in the region of an active deformation (region II in Fig. 1) at strain level $\gamma = 1.1$. The schematic illustration in the right upper corner show some marker lines visible in Fig. 3b offset at shear surfaces (depicted by curved lines).

an analysis of atomic mechanism of GBS. The dependence of $L = L(\sigma, T, d)$ characterizes deformation progress at the mesoscopic scale [15] (at the level of grain groups). Macroscopic strain rate, $\dot{\varepsilon}$, depends on deformation features both at the microscopic scale and at the mesoscopic scale.

The present experiments in superplastic Pb–62%Sn alloy, deformed in shear, showed bimodal L-distribution at low strain levels. The values of two modes of L, corresponding to two peaks of relative frequency, are approximately equal to 6 grain diameters and to 20 diameters. This indicates sliding of large blocks of grains (with dimensions of tens of grain size) and sliding grain groups (with dimensions of several grain sizes). L-distribution becomes unimodal at higher strain levels; L-value decreases when strain increases. This fact reveals that new grain boundary surfaces become involved in the progress of the shear at higher strain levels. Structural inhomogeneity has been assumed [16] to be responsible for sliding of large grain blocks. Indeed, grain boundaries associated with the former dendritic regions seem more prone to slide because of differences in their chemical composition and atomic structure [16, 17]. This results in movement of all grains surrounded by the former dendritic grain boundaries as a block. Sliding of grain groups of dimensions of several grain size might be explained as a consequence of the cooperative nature of GBS [18–20].

According to the concept of cooperative grain boundary sliding (CGBS), grains slide as an entity along shear surfaces formed by segments of sliding grain boundaries. This process is similar to the process of coarse slip in intragranular (dislocation) creep [21]. Similar to the case of the intragranular slip, where spacing of active shear surfaces is more than an atomic size, spacing of intergranular slip surfaces (surfaces of CGBS), $L > d$. It is possible to assume from the principle of similitude in analogy with dislocation slip [22] that $L \sim \sigma^{-1}$. Based on this assumption, the phenomenology of SP behaviour, in particular the strain rate dependence on σ and d (Equation 3) can be explained [23]. This indicates the importance of measurements of spacing of CGBS surfaces in order to assess the dependence $L = L(\sigma, T, d)$, as has been done earlier in the investigations of coarse slip [21]. Note that Burton [24] and Langdon [25, 26] discussed dislocation slip and SP flow from a unique point of view and suggested that optimum SP occurred when subgrain size was equal to grain size. In general, the spacing of active CGBS surfaces, which is determined by the size of sliding grain group, can be more than a grain diameter.

4. Conclusions

1. Average spacing of grain boundaries, $\bar{L}$, at which grain boundary sliding (GBS) takes place in the course of superplastic deformation in shear in Pb–62%Sn, is more than a grain diameter.

2. Statistical distribution of distances between sliding grains, L, is bimodal at low strain level as a result of cooperative sliding of large grain blocks (with

dimensions of tens of grain size) and of grain-groups (with dimensions of several of grain size).

L-distribution becomes unimodal at high-strain level, which can be explained by the operation of new surfaces of cooperative GBS.

3. The real spacing of sliding grain boundaries needs to be incorporated in future, and hopefully, better models for SP flow.

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