FINAL REPORT
DOE GRANT DE-FG02-96ER45578

The Mechanisms of Grain Boundary Slip Transmission, Migration and Sliding

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INTRODUCTION

Grain boundaries are one of the most common and important microstructural features in engineering materials. They can be sites where precipitation occurs, sites where impurities may concentrate through segregation, sites for absorption of dislocations and vacancies, and, under the correct conditions, paths for easy failure both at low and high temperatures and a result of environmental attack. At elevated temperatures grain boundaries can also move through the material. This movement can eliminate cold worked structures which in turn softens the material. This movement can also produce an increase in grain size, which itself can have important effects on mechanical properties.

Because of the great importance of grain boundaries in engineering materials, there has been significant research activity focused on this microstructural constituent for decades. Much of the early work relied heavily on optical metallography and many of the basic principles that are used to describe grain boundaries today were obtained from these studies. For example, the basic laws describing grain boundary movement, the fact that boundaries tend to meet at triple points with dihedral angles of 120° , and the basic observation that twin boundaries tended not to be paths of corrosion and fracture were all made through observations with the optical microscope. In recent years there has been significant activity in the application of high resolution electron microscopy to the study of grain boundaries [1]. These studies have yielded some very important results. First, they have shown that the lattice structure was maintained basically right up to the interface. There was no real evidence of significant relaxation or disruption of the lattice on either side of the boundary. However, there have been some very significant limitations in these studies as well. Because of the inherent demands imposed by the imaging capabilities of even the best high resolution microscopes, one can still only examine very special orientations. These are generally so far from the random high angle grain boundaries that control material properties that it is difficult to know whether or not the structures that are obtained are relevant for the much needed qualitative picture that can be used to think about the engineering problems. Second, enough studies have now been performed which have examined the energy of grain boundaries, that it is clear that grain boundaries do not necessarily take on the lowest energy structure available to them based on the orientation of the two grains composing the boundary[2].

Another important concept that has arisen out of all of this work has been a general understanding that special and low angle grain boundaries are much less likely to be paths for intergranular fracture and corrosion than are random, high angle grain boundaries. This understanding has led in recent years to the field of grain boundary

engineering [3,4]. This engineering refers to thermo-mechanical treatments that can be applied to a material which will give rise to a high enough fraction of low angle and special grain boundaries so that it will be impossible for a crack to proceed through the material without frequently encountering some of these boundaries and thus making propagation much more difficult. The reason why such thermo-mechanical treatments produce the improvement that they do has not been well established.

Another important area of research has been the observation that grain boundaries are often sites for segregation and precipitation. These processes can produce significant local chemical changes in the grain boundaries and change their properties relative to the bulk. Thus it is because of segregation and precipitation that the boundaries often become the preferred paths for fracture and corrosion. Furthermore, it is quite likely that these processes change the structure of the grain boundary.

During the last eight years, we have worked on the general problems associated with grain boundaries in metals with DOE support. This final report summarizes the work that has been performed. At the start of this work, we took a much more atomistic approach to grain boundaries. However, as we performed this research it became clear that such approaches had the drawbacks listed above, and that we were not proceeding toward the more general understanding of grain boundaries that we have hoped to achieve. We then moved toward more macroscopic based experiments that we could use to understand the structure and motion of grain boundaries. From these we were able to begin deducing some of the most important results of this work and to provide information that can be used by others to understand the role of grain boundaries in materials. We thus present this report in a topical way and provide the experimental and theoretical underpinning that is needed at each point as we go forward.

GRAIN BOUNDARY STRUCTURE

The first topic we will consider is grain boundary structure. This was the initial area where we began our studies on this DOE proposal. In this work we examined grain boundaries made from aluminum. Initially we proposed to make the grain boundaries by growing bi-crystals. However, we found that it was not possible to make the boundaries in a controlled enough way to allow for examination by high resolution electron microscopy, since the microscope that we had available required that the grains composing the boundary have $\langle 110 \rangle$ normals in both grains in order to obtain atomic resolution. Therefore, we began to make the boundaries by a strain annealing method in which we deformed the material and then recrystallized it. The texture that developed was such that boundaries with $\langle 110 \rangle$ normals were produced.

Figure 1 shows a low angle grain boundary that we produced in this way. The three panels represent the different locations along the length of the boundary. The misorientation across the grain boundary is 7° . One notes that the image in one grain is not as clear as the image in the other. The reason for this difference is that there is a slight twist component to the grain boundary and this twist causes the image to blur. Inspection of the boundary brings out several important aspects of its structure. The first is that the plane of the boundary is not straight but has a zigzag shape. The image would suggest that the boundary is faceted, but this appearance turns out to result from contrast in the image rather than faceting of the boundary plane. The boundary is composed of

dislocations. These are indicated in the figure by a line, and the “S” indicates the termination of the dislocation. In a grain boundary of this type, it is possible to have either Lomer or 60° dislocations, and one can use the image of the dislocation to determine which is present. An analysis of these dislocations indicates that they are all 60° dislocations, but that two different orientations of these 60° dislocations are present. The spacing between each pair of dislocation is similar but not precisely identical.

It is important to compare these results to both theoretical and other experimental studies. Figure 2 shows a simulation of this boundary which was performed as part of this study in which the atoms interact via an embedded atom potential and the structure is relaxed using lattice statics methods. The simulated structure is more perfect than the experimentally observed one, but the essential feature of 60° dislocations is reproduced. In the simulation the spacing of the dislocations is very regular and a definite faceting of the boundary plane is observed. The fact that these were not found in the actual boundary probably results from the fact that the boundary in the specimen evolved from a cold rolled structure whereas the simulated structure evolved from a symmetric arrangement of atoms. The good agreement between the simulated structure and the experimental structure gives confidence in the simulation methods that are described in more detail below.

There have been many other studies of grain boundary structure in aluminum. Mills and Stadelmann [1] examined low angle grain boundaries in aluminum and found that Lomer dislocations were present in boundaries with misorientations greater than 3° . The difference in their result and ours probably arises from the fact that their boundaries were produced by creep. Medlin *et al.* [2] found that the structure of grain boundaries also depended on the entire three dimensional structure of the sample. Thus near the intersection with another boundary, the boundary under examination had a higher energy structure than it did well away from this intersection. With more powerful microscopes it has become possible to examine asymmetric boundaries and to analyze them in terms of the structural unit model [5].

In summary, these studies make several important points. The first is that one can use the general concepts of dislocation models of low angle grain boundaries and the structural unit model of high angle grain boundaries to describe boundaries produced by normal processing routes. The results also show that grain boundary structure depends on the processing route and the full three-dimensional of the boundaries. One cannot use the energy of the boundary as a single criterion to predict whether or not a given structure will be observed. Rather, one must assume that a variety of structures are possible for a given boundary and that the particular structure that is observed is the result of the processing of the material

As part of the underpinning modeling investigation in this program we also used boundaries modeled with embedded atom method potentials for aluminum to determine basic information about grain boundary structure. Forty-four $\langle 110 \rangle$ symmetric grain boundaries were investigated with misorientations ranging between 0 and 50.48° . By considering a large number of possible rigid body translations in the grain boundary plane it was found that there were many different structures available to the grain boundary. The large number of initial configurations that one can use make it very difficult to compute the structure with a global energy minimum. The competing structures for a given misorientation have their energies closely spaced with energy

differences of no more than 5%. Hence any model intended to predict the minimum energy structure should be sophisticated enough to pick out these small differences in energies. It was also found that the structural unit model did not necessarily predict the structure with minimum energy and that the breakdown of this model is particularly important for high Σ boundaries. (The Σ value represents the amount of atomic matching across a grain boundary. A value of 1 represents perfect matching and an increase in the Σ values indicates less and less matching.)

CREEP IN ALUMINUM

Grain boundaries are often paths for fracture in engineering materials. At low temperature this occurrence can usually be associated with a change in the local chemistry of the material around the boundary. Although we examined this problem to a small degree as part of this work, much more attention was paid to high temperature failure that results from creep. In particular we were concerned with the motion of grain boundaries and the evolution of stress at the boundaries. We performed this part of the study in collaboration with David Davidson of Southwest Research Institute and made use of the DISMAP system that he has developed. With this system one can follow creep processes in the scanning electron microscope and also obtain information about the strain that develops in the sample by using an image analysis program. We now present the results of this study.

The first set of results to consider are those obtained in the in situ straining stage in the SEM. These results all refer to a specimen that was tested first at 250°C and then at 300°C as the test progressed. In this experiment one triple point in the large grain aluminum sample was focused upon. A micrograph of its initial state is shown in Figure 3a. Note that the three grains are identified as A, B, and C. Micrographs of the specimen taken at various stages during the test are shown in Figures 3b-d. Figure 3b shows the sample after 2 hours and 17 minutes of testing at 250°C. Note that the grain boundary between grains B and C has migrated. When the specimen was heated to 300°C, creep processes were much more rapid. Figure 3c shows the specimen after 5 minutes of testing at this temperature and Figure 3d shows the specimen after 30 minutes of testing. Migration has now occurred along all three boundaries, and there appears to be a separation between grains A and C.

These figures also show other features that we found as part of this study. First we note that there is clear evidence of migration. Closer examination of such migration in other samples indicated that the migration occurred in steps. We also found that there was little change in the orientation within the grains composing the triple point as creep occurred. Finally, we found in another sample that the deformation at the triple point was sufficient to cause complete recrystallization. Figure 4 shows an example of this type of event.

The strain measurements were made on the specimen for the part of the test that was carried out at 250°C. One way to describe the strain is through the use of Mohr's circle of strain taken at various points on the specimen. These plots are shown in Figure 5 for specimens crept for different times. In these plots the diameter of the circle is proportional to the magnitude of the maximum shear strain. If the diameter line drawn on the circle is parallel to the horizontal, the strain is axial. If it is vertical, the strain is pure

shear. The arc associated with the diameter drawn on the circle indicates the deviation from a tensile strain. If the line is nearly horizontal and there is only a small arc, the strain is predominantly tensile. If there is an arc that indicates an angle close to 180° , the strain is primarily compressive.

The evolution of strain in these specimens is now described. Throughout Figure 5 the approximate positions of the grain boundaries are indicated. The scale relating the diameter of the circle to the actual strain is given at the bottom of Figures 5a-e. Figure 5a is for a specimen tested for 14 minutes. The first point to note is that the magnitude of strain in Figure 5a is much less than that in Figures 5b-e. The bar in Figure 5a represents only 2% strain whereas in Figure 5b-e it represents 10% strain. Note also that the strain varies significantly between tensile, compressive, and shear.

After 44 minutes of testing, the overall strain level is still small, as shown in Figure 5b, but after 57 minutes the strain is larger, more uniform, and appears to be evolving toward a tensile strain in all areas of the specimen. However, the strain is much less in grain A than in grains B and C. Figure 5d shows that after 1 hour and 30 minutes the strain has increased significantly in grain A, and also the strain appears to be highest around the grain boundaries. After 2 hours and 17 minutes the strain is more evenly spread throughout the grains, as shown in Figure 5e.

Several general remarks can be made concerning the above results. (i.) After 0.75 to 1 hours the strain is primarily tensile and remains that way throughout the test. (ii.) The strain is never high around the triple point. (iii.) There is never any abrupt change in the strain in crossing a grain boundary (iv.) The strain appears to be evolving with time. It builds up and then relaxes in various places across the specimen.

Another way to plot these results is to use the effective strain at various points on the surface. Values for this strain are shown in Figure 6 for the same testing times as those used to plot Mohr's circle diagrams in Figure 5. In Figure 6a the grain boundary structure has been superimposed so that regions of strain can be identified with the three grains indicated in Figure 1a. After 14 minutes of creep testing, the overall strain is low and rather uniform throughout the specimen. After 44 minutes of testing, the strain has increased in grains B and C in the direction of the tensile stress, but grain A shows little evidence of strain. The strain continues to build up in this way during the first 1.4 hours of loading. Strain has developed in grain A but it is below that in grains B and C. Finally, after 2 hours and 17 minutes of testing, there is relaxation of the strain in grains B and C, presumably through dislocation processes, although the strain at this stage remains at the same level in grain A. Again the strain remains very low around the triple point.

GRAIN BOUNDARY MIGRATION

One of the most important phenomena that relate to grain boundaries is their motion. We have already observed in the study above that grain boundaries move under stress during the creep process. We also know from many previous studies that grain boundaries move as a result of stored energy in the crystal and that that movement can lead to recrystallization. As a major part of our study we examined the movement of grain boundaries in deformed and annealed samples in order to understand the controlling factors that determine how grain boundaries move and their ultimate structure in a

material. In order to accomplish this goal we used aluminum bi-crystals that were deformed at room temperature and then annealed. The samples were oriented so that the initial bi-crystal in the sample had an $\langle 011 \rangle$ direction parallel either to the tensile axis or perpendicular to the tensile axis. The particular orientations were chosen so that upon deformation one grain would be strained more than another. If our results had followed the classical mechanisms that have been proposed for grain boundary motion, then we would have expected the boundary to move into the grain that had the higher strain and thus sweep across the sample. However, we found that the motion of the boundary was much less predictable and much more complex than this. In two papers which are in preparation and which will be sent to DOE upon completion, we analyze all of the boundaries that were formed. However, for this final report we simply present the results of three experiments which typify the type of motion that we observed.

In all of this work we made extensive use of orientation imaging in the scanning electron microscopy. This technique, more correctly known as Electron Backscattering Diffraction (EBSD) in the scanning electron microscope (SEM), allows detailed crystallographic information to be obtained in the scanning electron microscope through analysis of Kikuchi patterns obtained from the backscattered electrons. We used software produced by HKL, Inc. for our analysis which gave us information about grain orientation, grain boundary structures, and maps displaying particular features of the structure. In this study we make particular use of inverse pole figures. These locate within the standard stereographic triangle the orientation in a particular grain. In particular, one chooses three axes on the sample and plots the direction in the grain that is parallel to this direction. In the results below we will always take the y-direction as parallel to the tensile axis, the z-direction as perpendicular to the sample surface and the x-direction in the plane of the sample but perpendicular to y and z. One will also note that for each grain there will be a cluster of points. These result from the fact that the data are obtained by taking many points within a single grain and plotting all of them. Thus as the sample changes its orientation by rotation or bending, one picks up a spread in the data.

Example A: Rapid Motion Leading to a Single Triple Point in the Sample

One type of result that we observed was where the original grain boundary was rapidly eliminated from the sample leaving one or two triple points in the sample. The first time that we observed such behavior the sample had completely eliminated the original grain boundary and produced a single triple point after an initial deformation of 20% followed by annealing at 500°C for 30 minutes. Therefore we repeated the experiment with much shorter anneals to examine the results more carefully. Again the samples was deformed 20% and then annealed at 500°C. Figure xx shows the initial boundary and the boundary after aging for three minutes. Beneath the images of the boundary are the inverse pole figures. The grain marked “1” in Figure 7a was strained more than the grain marked “2.” From those figures we can see that the microstructure of the bi-crystal did not change greatly during the first three minutes of annealing except that some small residual grains were annealed out of the sample, which can be clearly observed from the EBSD maps. Also, after annealing the angular spread of the two main grains was less.

Annealing for an additional ten minutes completely transformed the structure. We found that several smaller grains appeared at the end of the less-strained grain while most of the original specimen was covered by a single crystal. Two triple junctions were formed and they were connected via one grain boundary. These grains are shown in Figure 8. We can see from the corresponding inverse pole figures that large grain that covered most of the original bi-crystal, which is the brown-colored grain in the right of this figure, has an orientation different from either starting grain. Thus the original structure has been replaced. We also see that of the five boundaries shown in Figure 8 that two are special boundaries and three are high angle random grain boundaries. The two special boundaries are $\Sigma 3$ and $\Sigma 17b$. Annealing for an additional 35 minutes did not change the orientation of the grains but did change their relative lengths. In particular, the $\Sigma 17b$ boundary has increased in length relative to the random high angle grain boundaries. Thus we have extended the length of this special boundary relative to the sum of the lengths of the two random high angle grain boundaries. The change can be seen quite clearly in Figure 9 where the trace of the original boundary can still be seen. Further annealing made no change in the shape of the triple junctions, which implies that the system is stable.

Figure 10 is a schematic picture of Figures 8 and 9 showing the equilibrium state of the two triple junctions. Suppose boundary $\Sigma 17b$ has an energy per unit length of $\gamma_{\Sigma 17b}$, boundary $46.8^\circ/\langle 210 \rangle$ has energy per unit length of $\gamma_{\langle 210 \rangle}$ and boundary $21.2^\circ/\langle 321 \rangle$ has energy per unit length of $\gamma_{\langle 321 \rangle}$. According to the geometric stability condition for a triple point we have

$$\gamma_{\langle 210 \rangle} / \sin \alpha_3 = \gamma_{\langle 321 \rangle} / \sin \alpha_2 = \gamma_{\Sigma 17b} / \sin \alpha_1 \quad (1)$$

We measure the three angles to be $\alpha_1 = 93^\circ$, $\alpha_2 = 136^\circ$ and $\alpha_3 = 131^\circ$. From this equation we can calculate that

$$\gamma_{\langle 321 \rangle} = 0.6956 \gamma_{\Sigma 17b} \quad (2)$$

and

$$\gamma_{\langle 210 \rangle} = 0.7557 \gamma_{\Sigma 17b} \quad (3)$$

Equations 2 and 3 show that although $\Sigma 17b$ is a special coincidence site lattice boundary, its energy is still larger than the other two random high-angle grain boundaries. This result could occur because a simple identification of the boundary as $\Sigma 17b$ does not define the boundary plane and this plane can have a significant effect on the boundary energy.

If we look at Figure 10 and measure the values of α_1' , α_2' and α_3' which have dihedral angles of the same triple junction in the earlier staging of annealing we will find that they have almost the same values as α_1 , α_2 , and α_3 . This is an important observation because it implies that the dihedral angles of this triple junction did not change even though the sample of the grains have been altered. But note that the geometrical stability condition only depends on the dihedral angles between the junction components, so we can draw the conclusion that the geometrical stability condition of the triple junction had

already been reached after 13 minutes of annealing. However, from the energetic point of view, the whole system still did not reach equilibrium at this stage.

Similarly we can measure in Figure 10 that $\beta_1=163^\circ$, $\beta_2=116^\circ$ and $\beta_3=83^\circ$. Suppose that $\Sigma 3$ boundary has the boundary energy $\gamma_{\Sigma 3}$ and boundary $49.5^\circ/\langle 421 \rangle$ has the boundary energy $\gamma_{\langle 421 \rangle}$. Using equation 1 we have

$$\gamma_{\Sigma 3} = 0.3253 \gamma_{\Sigma 17b} \quad (4)$$

and

$$\gamma_{\langle 421 \rangle} = 1.0989 \gamma_{\Sigma 17b} \quad (5)$$

These equations combined together can tell us the relationship between the different boundary energies, which is shown in Figure 11. We can clearly see that only the $\Sigma 3$ boundary has a significantly lower boundary energy.

Example B: Sample with Recrystallization and Grain Growth

The sample used for this experiment was strained to 16% and then annealed in steps at 500°C for 5 to 47 minutes. Unlike the previous example, the more highly strained grain immediately divided into many smaller grains while the less-strain grain remained unchanged during the first 15 minutes of annealing. We used this sample as a chance to study the details of grain growth. Figure 12 and Figure 13 show the map of grains and grain boundary evolution, respectively, during the annealing process. The times were 5, 15, 23, 31 and 47 minutes.

In Figure 13 the grains denoted by black lines are random, high angle grain boundaries and those denoted by red are $\Sigma 3$ boundaries. Other colored grain boundaries are special boundaries with Σ less than 29 but not $\Sigma 3$. The reason that we do not consider special boundaries with Σ values larger than 29 is because in those boundaries the extent of coincidence between the two lattices is so low that it would be better to treat them as random high angle boundaries. The original boundary is denoted by an arrow. We see that in the right hand grain there are initially many small included grains that are eliminated after 15 minutes of annealing. We also see that the original grain boundary is still visible after 5 and 15 minutes of annealing, although after 23 minutes it has disappeared. In the left hand grain we see that many grain boundaries have formed after five minutes of annealing. We note that many of them are high angle grain boundaries, but there are a significant number of $\Sigma 3$ boundaries as well. It also appears that almost all of the special boundaries are $\Sigma 3$ and that other low Σ boundaries are much less frequently observed.

The annealing treatment that took the total time from 5 to 15 minutes clearly caused grain growth in the area that had originally been the left hand grain. Again almost all the special boundaries were $\Sigma 3$. After this point we were able to catalog all of the grain boundaries in the sample and track them. Figures 14 shows the histograms of the percentage of total boundary number plotted as a function of misorientation after 15, 23, 31, and 47 minutes of annealing. One can see that $\Sigma 3$ boundaries were the only boundaries that appeared with significant frequency and that they represent approximately 20.5% of the boundaries present. With continued annealing for a total

time of 23 minutes, one can observe that there has been significant grain growth, and grains have now formed in both of the original grains. Finally, we note that with continued annealing for 31 and 47 minutes there is continued grain growth.

It is quite instructive to look at the details of the boundaries that were annihilated and that formed during the annealing sequence. Since $\Sigma 3$ boundaries were by far the most common special boundaries, we will divide the boundaries into being either $\Sigma 3$ or other boundaries. Table I lists information concerning these processes.

The following important points can be drawn from these results. First we should note that the number of $\Sigma 3$ boundaries is much greater than what would be predicted to occur in a random polycrystal. Warrington and Boon [6] calculated from the geometrical point of view the probability of a random boundary that would fall within a reasonable deviation about each CSL boundary up to $\Sigma 25$. According to their calculation the percentage of $\Sigma 3$ boundaries within a random distribution would only be 1.53%. The second point is that although there is some scatter in the results it would appear that the percentage of $\Sigma 3$ boundaries did not change that greatly during grain growth and that the number annihilated by grain growth is, if anything, higher than the representative fraction of boundaries present, as can be seen from the Table. In addition it would appear the few $\Sigma 3$ boundaries are created during the growth process. Thus we conclude that the grain growth process alone does not enhance the presence of these special boundaries and significant changes in the number of boundaries would occur only through repeated cold work and annealing.

Another comment about our results is the characteristics of the $\Sigma 3$ boundaries. Most of the $\Sigma 3$ boundaries shown in Figure 13 did not show the normal shape of annealing twins. Since aluminum is a material with a high stacking fault energy, the probability of creating annealing twins is much lower than in a low stacking-fault energy materials such as copper and nickel. However, we have no effective way to know if the $\Sigma 3$ boundaries in our specimen are annealing twins or not because the HKL software does not provide a method to find the boundary plane. Thus we can only treat these $\Sigma 3$ boundaries in our specimen as special boundaries with a $60^\circ/\langle 111 \rangle$ angle/axis pair and low boundary energy.

Table I
Statistics on $\Sigma 3$ Grain Boundaries During Annealing

Annealing Time (min.)	$\Sigma 3$ Boundaries Present	Other Boundaries Present	$\Sigma 3$ Boundaries Annihilated	Other Boundaries Annihilated	$\Sigma 3$ Boundaries Created	Other Boundaries Created
15	17 (20.5% of total)	66				
23	8 (13.5% of total)	51	7	32	3	19
31	12 (18.7% of total)	52	1	2	0	1
47	7 (15.9% of total)	37	5	15	0	3

However, one way in which we can make a qualitative estimate of the energy of these $\Sigma 3$ boundaries is to examine the dihedral angles associated with them. In order to do this we consider the following subset of triple junctions in the this sample. These include the triple junctions stable at the end of the entire annealing process, those formed during the last annealing period, and those annihilated during the last annealing period. Figure 3.15 shows two histograms of the dihedral angles. Although there is some overlap between the two histograms we can see that the histogram that forms the $\Sigma 3$ boundaries is centered at a higher value and has some of the highest values measured for these samples. Thus we conclude that these $\Sigma 3$ boundaries are probably of lower energy than the random boundaries found throughout the sample.

Example 3: Controlled Grain Boundary Migration

In some samples we were able to follow the motion of a single grain boundary move across the sample. We now give an example of this.

Figure 16 shows the maps of the entire bi-crystal sample. During the first period of aging the original boundary did not move. However, a new grain boundary that had a misorientation of $35^\circ/\langle 310 \rangle$ was formed near the left hand part of the map shown in the Figure. With continued aging this boundary moved across the sample transforming the orientation of the grain on the right to the one on the left. During this migration the original boundary did not move. After 55 minutes of annealing, the migrating grain boundary reached the original boundary and they reacted to form a new boundary with an angle/axis pair of $42^\circ/\langle 210 \rangle$. Further annealing for another 30 minutes made the new grain boundary move into the less-strained grain. The entire migration process is shown in the Figure.

From studies such as this we could also obtain grain boundary velocities. We found that the actual values of the velocity varied between 1.9 and 6.4 μ/sec . These values provide fundamental information about the migration rates of grain boundaries in materials.

CONCLUSIONS

This report provides a summary of the work that we have performed on grain boundaries as part of this DOE grant. Some of the most important conclusions are the following:

1. For a given misorientation, there are many different grain boundary structures of similar energy that can be adopted by the grain boundary. The boundary probably uses these different structures as it undergoes such processes as recrystallization and fracture. It seems fruitless to try to consider a single low energy structure since the particular structure the boundary may adopt will depend on the processing schedule that created the boundary and the overall three-dimensional network of the grain boundaries.
2. During creep processes, migration plays an important role and can lead to stress reduction and to recrystallization.
3. The motion of grain boundaries is very complicated phenomenon and very difficult to control even under what would be considered to be ideal circumstances. Grain boundaries do not necessarily move into regions of highest deformation to eliminate strain.

4. A simple measure of dihedral angles gives significant information about the energetics of grain boundaries.
5. The velocity of grain boundary migration in aluminum is much higher than often considered.

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Figure 1: High resolution transmission electron micrograph of a 7° tilt boundary in aluminum. The three panels show different regions along the grain boundary; the lines on the micrographs indicate the location of 60° dislocations and S shows the termination of the dislocation line.

Figure 2: A lattice statics simulation of grain boundary shown in Figure 1. Again the 60° dislocations present in this boundary are indicated by lines.

Figure 3: Micrographs showing evolution of structure around a triple point in aluminum during creep. The stress on the sample was 3.3 MPa. (a.) before testing; (b.) after testing for 2 h and 17 min at 250°C; (c.) after testing as for Figure 3b plus 5 min. at 300°C; (d.) after testing as for Figure 1b plus 30 minutes at 300°C.

Figure 4: Scanning electron micrograph of a triple point around which grain boundary migration and recrystallization have occurred.

Figure 5: Mohr's circles of strain plotted for different areas on the specimen surface at given times during the creep test. Areas of analysis are the same as those in Figure 3 and the temperature of the test was 250°C. (a.) 14 min. (b.) 44 min. (c.) 57 min. (d.) 1 h and 30 min. (e.) 2 h and 17 min.

Figure 6: Effective strain plotted for different areas on specimen surface at given times during a creep test. Areas of analysis are the same as those in Figure 3 and the temperature of the test was 250°C. (a.) 14 min. (b.) 44 min. (c.) 57 min. (d.) 1 h and 30 min. (e.) 2 h and 17 min.

Figure 7: Grain boundary maps and inverse pole figures for a sample before annealing and after annealing at 500°C for three minutes. The boundary down the center of the micrographs is the original grain boundary in the bicrystal. (a.) Original sample. (b.) Boundary after annealing for three minutes. (c.) Inverse pole figure for the original grain boundary. (d.) Inverse pole figure of the grains after 3 minutes of annealing. Note that the basic orientation is unchanged.

Figure 8: Grain boundary maps and inverse pole figures for the sample shown in Figure 7 but after annealing for a total of 13 minutes and 48 minutes. (a.) Sample after annealing for 13 minutes. (b.) Sample after annealing for 48 minutes. (c.) Inverse pole figure for the sample after annealing for 13 minutes. (d.) Inverse pole figure for the sample after annealing for 48 minutes. Notice that the grain orientation is unchanged but the lengths of the boundaries have changed.

Figure 9: SEM image of the triple junction after annealing for 48 minutes. Note that the image shows the original and final location of the triple point.

Figure 10: Schematic picture of the triple points shown in Figure 8. The dashed lines mark the position of the triple junction after 13 minutes of annealing while the solid lines mark the position of the triple junction 48 minutes after annealing.

Figure 11: Boundary energy relationship for all five boundaries shown in Figure 8.

Figure 12: The grain boundary maps of the sample annealed for 5, 15, 23, 13, and 47 minutes. Time increases going from the top to the bottom figure.

Figure 13: Maps showing the grain boundary orientation for samples annealed for 5, 15, 23, 31, and 47 minutes. Black lines indicate random high angle grain boundaries, red lines indicate $\Sigma 3$ boundaries, and other colored lines indicate special boundaries with $5 < \Sigma < 29$.

(a) Grain boundary distribution after 15 minutes annealing.

(b) Grain boundary distribution after 23 minutes annealing.

(c) Grain boundary distribution after 31 minutes annealing.

(d) Grain boundary distribution after 47 minutes annealing.

Figure 14: - Grain boundary distribution after 15, 23, 31 and 47 minutes of annealing. The boundaries with 60° misorientation are $\Sigma 3$ boundaries.

Figure 15: The dihedral angle for random boundaries and $\Sigma 3$ boundaries formed during annealing.

Figure 16: Grain boundary maps of a specimen after 10, 20, 30, 55, 85 minutes of annealing.

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