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Recrystallization of High Purity Aluminum

Virgil H. Griswold Jr.

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RECRYSTALLIZATION OF HIGH PURITY ALUMINUM

by

Virgil H. Griswold, Jr.

A Thesis
Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science
in Metallurgical Engineering

MONTANA SCHOOL OF MINES
Butte, Montana
May 11, 1953
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RECRYSTALLIZATION OF HIGH PURITY ALUMINUM

MECHANISM OF RECRYSTALLIZATION

It is a known fact that, if a metal is first cold-worked and then heated to a sufficient temperature for an adequate length of time, the hardening properties due to cold-working disappear, and the metal will return more or less to its original state. Upon examination of the microstructure, we find that new grains have replaced the old grains through what is called recrystallization. The final size of the recrystallized grains depends upon three important variables—the degree of cold-work or deformation, annealing temperature, and annealing time.

The size of the grains produced is inversely proportional to the degree of deformation, and this deformation must exceed a certain definite amount, or no recrystallization will occur. The annealing temperature and annealing time show the opposite effect, i.e., the grain size increases with an increase in either of these factors.

During annealing, recrystallization is accomplished by the formation of nuclei from the most severely distorted volumes. These nuclei grow at the expense of their distorted neighbors until all the volume in question is occupied by new and undistorted crystals. At this point, the metal is said to be completely recrystallized. Complete recrystallization is a time-temperature function
and depends on the degree of deformation; that is, for a definite amount of deformation, the higher the temperature the shorter the time required to reach complete recrystallization. Coalescence, a process of continued grain growth after complete recrystallization, proceeds when the grains grow at the expense of their neighbors. Like recrystallization, coalescence is a time-temperature dependent function and the rate of growth increases with the increase in annealing temperature and increase in time at temperature.
PURPOSE OF INVESTIGATION

W. A. Anderson and R. F. Mehl, in attempting to explain recrystallization in terms of the rate of nucleation and the rate of growth, made both two and three-dimensional studies of recrystallized aluminum. In their two-dimensional work, these men used elongated percentages ranging from 1.9% to 10.6%; however, for their three-dimensional recrystallization, they used only 5, 10, and 15% elongations. The Anderson-Mehl investigation yielded orthodox isothermal curves showing the effects of temperature, degree of deformation, and original grain size on the rate of recrystallization.

This investigation will be limited to a study of three-dimensional recrystallization, and to small deformations approximately 5% being the maximum. The purpose of the investigation is to check the Anderson-Mehl data for three-dimensional recrystallization at small elongations. This will make it possible to see if this data can be extrapolated to show what grain size might be expected with small deformations, and, to prove that relatively same size grains, from equally deformed samples, can be produced at various temperatures by close control and calculation of the time required for recrystallization at these temperatures.
The materials used by Anderson and Mehl for three-dimensional recrystallization had the following analysis:

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>0.01%</td>
</tr>
<tr>
<td>Iron</td>
<td>0.03%</td>
</tr>
<tr>
<td>Copper</td>
<td>0.01%</td>
</tr>
<tr>
<td>Aluminum</td>
<td>99.95%</td>
</tr>
</tbody>
</table>
MATERIAL INVESTIGATED

A sample of high purity aluminum rod was secured from the Research Division of the Aluminum Company of America. In the as-forged condition, this bar was of the following analysis:

- Silicon 0.02%
- Iron 0.01%
- Copper 0.03%
- Magnesium 0.01%

As received, the sample was 3/4 in. rod approximately 3 ft long.
Throughout the laboratory work, the author has attempted to control his techniques as closely as possible to eliminate any additional variables. Because of limited laboratory equipment, in some cases the control of these variables was quite difficult; however, the author will note these variables and the degree to which they were controlled in the following paragraphs.

Construction of Tensile Bars

From the 3/4 in. rod as received, five tensile bars were constructed (Figure 1). It was first necessary to reduce the rod to 1/2 in. in diameter. The center portion, one inch in length, was then roughly machined to 1/4 in. in diameter with a 1/4 in. radius on each end. To insure a uniform diameter along the center portion, the bars were polished next with 1/0 and then 3/0 emery cloths. The final products were closely measured and checked with a micrometer for uniformity.

Initial Anneal

A holder for the tensile bars was constructed of a light refractory brick, which allowed the bars to rest on their ends but which prevented them from moving around. Such action not only insured the same annealing treatments to all the bars, but also prevented any deformation prior to the elongation procedures. The holder with bars was
Figure 1. Tensile Bar

Figure 2. Tensile Apparatus
placed in an electric furnace at 800°F for 15 minutes to obtain a homogeneous grain structure throughout. A small sample from the as-received rods was also given this treatment and, from this sample, the original grain size was obtained. After the prescribed time, the holder was removed, and the bars were cooled to room temperature. The initial grain size was determined to be 1.03 millimeter in diameter.

Deformation: Apparatus and Method

Because elongation seems to be the simplest method for insuring uniform deformation, this procedure was chosen. Two very fine holes, approximately 3/4 in. apart, were inscribed on the center portion of each bar with the points of a drafting compass. With these holes as references, the initial distance between them was determined microscopically (Table I). A bar, so marked, was then ready for testing.

By the use of a standard drill press and a lathe chuck, a tensile machine was constructed (Figure 2). The drill-press chuck acted as the upper support for the tensile bar and the lathe chuck fastened to the lower end of the bar. Inserted and threaded through the adjusting pin of the lathe chuck was a short piece of a piano wire which supported a weight bucket.

One end of the bar to be tested was first securely fastened in the lathe chuck while the other was fixed in the drill-press chuck. Into the weight bucket, test lead
was slowly poured until the desired elongation was reached. Periodic checks on the reference holes were made by inserting a drafting compass, measured to the approximate final length. For accurate determination of their final length, each bar was again measured microscopically using a Filar eyepiece. Calculations of the per cent elongation were made with the use of the following equations, and the results were entered in Table I.

\[ \Delta L = L_f - L_i \]

\[ \% \text{ elongation} = \frac{\Delta L}{L_i} \times 100 \]

Where:
- \( L_f \) = final length or reading
- \( L_i \) = initial length or reading
- \( \Delta L \) = change in length or reading

Table I

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>.234&quot;</td>
<td>3/4&quot;</td>
<td>20.4</td>
<td>20.6</td>
<td>91#</td>
<td>0.98</td>
</tr>
<tr>
<td>2</td>
<td>.234&quot;</td>
<td>3/4&quot;</td>
<td>20.3</td>
<td>20.8</td>
<td>162#</td>
<td>2.46</td>
</tr>
<tr>
<td>3</td>
<td>.230&quot;</td>
<td>3/4&quot;</td>
<td>20.3</td>
<td>20.9</td>
<td>158#</td>
<td>2.96</td>
</tr>
<tr>
<td>4</td>
<td>.230&quot;</td>
<td>3/4&quot;</td>
<td>20.3</td>
<td>21.2</td>
<td>170#</td>
<td>4.44</td>
</tr>
<tr>
<td>5</td>
<td>.227&quot;</td>
<td>3/4&quot;</td>
<td>20.3</td>
<td>21.6</td>
<td>173#</td>
<td>6.40</td>
</tr>
</tbody>
</table>

Annealing Calculations

To accomplish complete recrystallization without grain growth, the metallurgist must carefully select his annealing temperatures and correlate the annealing time to these
temperatures. As it has been stated before, the rate of coalescence or grain growth increases rapidly with both temperature and time. The author, by the use of data published by W. A. Anderson and R. F. Mehl, was readily able to make the necessary calculations. Anderson and Mehl determined the time required to reach complete recrystallization of high purity aluminum at various temperatures. In their work, these men used a form of the Arrhenius equation to relate nucleation and growth to recrystallization.

Arrhenius Equation:

\[ R = Ae^{-\frac{Q}{RT}} \]

Where:

- \( R \) = rate of reaction
- \( A \) = constant
- \( e \) = base of natural logarithms
- \( Q \) = activation energy
- \( R \) = gas constant
- \( T \) = absolute temperature

When this equation is expanded with respect to time, it will take the following form:

\[ \log \frac{t_2}{t_1} = \frac{Q}{2.303R} \left( \frac{T_2 - T_1}{T_2T_1} \right) \]

Where:

- \( t_1 \) = time required to complete recrystallization at \( T_1 \)
- \( t_2 \) = time required to complete recrystallization at \( T_2 \)
\[ T_1 = \text{given temperature for } t_1 \]
\[ T_2 = \text{given temperature for } t_2 \]

Q, in this case a value similar to an activation energy for the recrystallization of aluminum, varies inversely with the degree of deformation. To calculate the values of Q for the elongations used in this investigation, the author used data determined by Anderson and Mehl, and plotted the curve shown in Figure 3. Annealing time, as we have said before, also varies with per cent elongation at a constant temperature; thus, it was necessary to again take the results of Anderson and Mehl's work and plot a log-time versus elongation curve (Figure 4). With a constant temperature of 350°C and time for 5, 10, and 15, per cent elongations given, the curve was plotted and extrapolated to the elongations less than 5%. The data given by Anderson and Mehl are shown in Table II and the results by the author are shown in Table III.

With both Q and t₁ known for any per cent elongation at a particular temperature, one can readily calculate t₂ for any chosen temperature. The author, having chosen to work between the time limits of approximately 30 seconds and 24 hours, decided on the following annealing temperatures: 350°C, 400°C, and 450°C. The time required for complete recrystallization at these temperatures and for each elongation is also shown in Table III. An example of this type of calculation for 0.98% elongation at 400°C is shown.
Figure 3.

Anderson-Mehl Data
For Activation Energies
○ Calculated
○ Observed

Per Cent Elongation

Activation Energy (Q) in Kcal
Figure 4.

Anderson-Mehl Data
For 350°C Ann.
### Table II

From Anderson-Mehl Data

<table>
<thead>
<tr>
<th>Per Cent Elong.</th>
<th>Q in cal</th>
<th>Ann. Time At 350°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Observed</td>
<td>Calculated</td>
</tr>
<tr>
<td>5.0</td>
<td>-64,500</td>
<td>-64,100</td>
</tr>
<tr>
<td>10.0</td>
<td>-59,600</td>
<td>-58,000</td>
</tr>
<tr>
<td>15.0</td>
<td>-52,100</td>
<td>-54,000</td>
</tr>
</tbody>
</table>

### Table III

<table>
<thead>
<tr>
<th>Per Cent Elong.</th>
<th>Q in cal</th>
<th>Calc. Ann. Times in min at 350°C</th>
<th>400°C</th>
<th>450°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>350°C</td>
<td>400°C</td>
<td>450°C</td>
</tr>
<tr>
<td>0.98</td>
<td>-68,700</td>
<td>1450 min</td>
<td>24 min</td>
<td>40 sec</td>
</tr>
<tr>
<td>2.46</td>
<td>-67,200</td>
<td>980 min</td>
<td>17 min</td>
<td>32 sec</td>
</tr>
<tr>
<td>2.96</td>
<td>-66,500</td>
<td>860 min</td>
<td>16 min</td>
<td>31 sec</td>
</tr>
<tr>
<td>4.44</td>
<td>-64,800</td>
<td>590 min</td>
<td>12 min</td>
<td>25 sec</td>
</tr>
<tr>
<td>6.40</td>
<td>-62,700</td>
<td>360 min</td>
<td>8 min</td>
<td>20 sec</td>
</tr>
</tbody>
</table>
\[ t_1 = 1450 \text{ min} = \text{required annealing time at 350°C} \]
\[ T_1 = \text{annealing temperature of 350°C or 623K} \]
\[ T_2 = \text{annealing temperature of 400°C or 673K} \]
\[ Q = -68,700 \text{ cal per deg. per mole} \]
\[
\log \frac{t_2}{1450} = \frac{-68,700}{2.303 \times 1.987} \frac{(673 - 623)}{(673 \times 623)}
\]
\[ \log \frac{t_2}{1450} = -1.791 \]
\[ \log t_2 = -1.791 \neq \log 1450 \]
\[ \log t_2 = 1.370 \]
\[ t_2 = 24 \text{ min at 400°C} \]

**Annealing Procedures**

Having been elongated to desired percentages, the center portion of each tensile bar was cut into three samples which in turn were annealed at the above temperatures for the calculated times. A small wooden block, through which a 1/4 in. hole had been bored, was fitted around the center portion of each bar, and the cutting action was accomplished by sawing through both block and bar. Such action was done to prevent twisting or any further deformation prior to annealing. Figures 5 and 6 show the electric furnaces used for 350°C and 400°C anneals, respectively. All samples in these furnaces were laid on a fine-meshed screen to prevent temperature gradation. When removed from the furnaces, the samples were uniformly cooled by laying them on a screen placed over a scorifying dish. Figure 7 shows the electric furnace used for the 450°C annealing treatment.
Fig. 5. Furnace at 350°C  
Fig. 6. Furnace at 400°C  
Fig. 7. Furnace & Lead Bath at 450°C
This furnace contains a lead bath. Because the annealing time for each elongation at this temperature is measured in seconds, the lead-bath treatment was used.

**Etching and Metallographic Procedures**

A galvanic macro-etching technique, developed by Dr. I. S. Servi, was first tried. However, a simple etching technique recommended by C. S. Barrett and L. H. Levenson, proved to be more satisfactory and quicker. The Barrett reagent consists of 9 parts HCl, 3 parts HNO₃, 2 parts HF, and 5 parts H₂O. The author wishes to mention, at this time, that careful control over the immersion depth must be exercised if the same degree of etch on all samples is to be obtained in equal time. The reaction is slower near the surface of the solution and increases near the bottom. The time required for these samples was from 30 to 35 seconds.

Grain sizes obtained required photographing at low magnifications. For this reason then, the samples were first photographed at 5.5x; however, the pictures at this magnification proved to be too small for accurate calculation of grain sizes. Therefore, the samples were re-photographed at 10x.
RESULTS OBTAINED

The grain size of each sample was calculated directly from the photographs at 10x. First, the number of grains per square inch was determined by drawing a square inch on the face of the photographs and multiplying the number of grains counted by ten. The diameter was calculated in inches and then reduced to millimeters. The results of the investigation are clearly shown in Table IV and by the photographs on the following pages.

Table IV

<table>
<thead>
<tr>
<th>Per Cent Elongation</th>
<th>Grain Size mm Dia.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>800°F</td>
</tr>
<tr>
<td>As rec’d</td>
<td>1.03</td>
</tr>
<tr>
<td>0.98</td>
<td>1.23</td>
</tr>
<tr>
<td>2.46</td>
<td>1.36</td>
</tr>
<tr>
<td>2.96</td>
<td>1.33</td>
</tr>
<tr>
<td>4.44</td>
<td>1.25</td>
</tr>
<tr>
<td>6.40</td>
<td>1.25</td>
</tr>
</tbody>
</table>
All Samples Annealed
At 350°C Except
As Received Which
Was Annealed
At 800°F

As Rec’d  Time 15 min
0.98%  Time 1450 min

2.46%  Time 980 min
2.96%  Time 860 min

4.44%  Time 590 min
6.40%  Time 360 min
All Samples Annealed
At 400°C

0.98% Time 24 min

2.46% Time 17 min

2.96% Time 16 min

4.44% Time 12 min

6.40% Time 8 min
ALL SAMPLES ANNEALED AT 450°C

0.98% Time 40 sec

2.46% Time 32 sec

2.96% Time 31 sec

4.44% Time 25 sec

6.40% Time 20 sec
DISCUSSION

In a somewhat similar investigation, L. W. Eastwood, R. W. James, and R. F. Bell\(^4\) found that the grain sizes for 1 to 5 per cent ranged from 0.61 to 1.83 millimeters in diameter at the temperature used in this investigation. However, the Eastwood-James-Bell investigation did not emphasize the control of recrystallization; that is, in some cases the specimens had not recrystallized, others had partially recrystallized, and still others may have had considerable amounts of grain growth after complete recrystallization. With the grain sizes in Table IV compared on this basis, the results of this investigation substantiate the fact that the Anderson-Mehl data can be effectively used to predict the sizes of recrystallized grains of aluminum at small deformations.

For the 2.46, the 2.96, and the 4.44 per cent elongations, the data proves that same size grains of equally deformed samples, can be produced at various temperatures by merely correlating time and temperature. Although, the 0.98 and the 6.40 per cent elongations show a great deal of discrepancy between the corresponding grain size, the author feels this discrepancy is due to defects of the tensile bars used. These results substantiate the second fact that the Anderson-Mehl data can be used to relate annealing time to temperature, thus producing equal grain size from equally deformed samples at any temperature.
CONCLUSIONS

From the research performed on high purity aluminum, the following conclusions have been drawn:

1. The Anderson-Mehl data can be extrapolated to low deformation values and will yield accurate results; therefore, the activation energies and the time required for complete recrystallization, given in their report and used in this investigation, are correct.

2. By using the Anderson-Mehl data on three dimensional recrystallization of aluminum, one can predict the grain size to be expected with small deformations. The smaller the deformation, the larger the final grain size will be on complete recrystallization without grain growth.

3. Furthermore, by using the activation energies and the time-temperature values determined by these men, one may also control the grain size of equally deformed specimens in order to produce the same size grains at any chosen annealing temperature.

The results of this investigation may be used as the basis for future research, along this line, which will only go to further substantiate the above conclusions.
RECOMMENDATIONS

If further research on recrystallization or any other problem involving elongations is attempted, the author recommends that a regular tensile machine or a different type of an apparatus be used other than the one used by him. The weight bucket of the apparatus used here continuously turns on the piano wire and may produce undesirable effects. A recommended apparatus might well be constructed on a smaller scale. Such a machine would consist of a stationary lathe chuck fastened to a vertical metal plate, and an adjustable lathe chuck with a turn-buckle nut fastened to a vertical plate some distance from the first. Proper keys and keyways must be provided for the adjustable chuck to prevent twisting. A microscope may then be mounted directly over the tensile bar, and the elongations can be watched constantly as the bar is elongated by turning the nut. Proceeding along this line, one can more closely control the desired elongations.

An interesting future problem along this line might be a study of the grain structure at various time intervals to prove that no grain growth will occur during the calculated time and temperature used by this author. Other research might be in determining the effects of initial grain size on the final grain sizes, or the determination of the germination point.
BIBLIOGRAPHY


ACKNOWLEDGEMENT

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