Ageing Heat Treatment of Aluminum Alloys

Content

1 Introduction ............................................................................................................................................. 2
2 Age Hardening ...................................................................................................................................... 3
3 Age Hardening of Aluminum Alloys ................................................................................................. 5
4 Procedure ............................................................................................................................................... 7
Appendix A: Aging Characteristics of Aluminum Alloys ...................................................................... 8
Appendix B: Heat Treatments for Aluminum Alloys ............................................................................. 9

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1 Adapted from: Callister, W.; Engineering and Science of Materials, Wiley and Sons, 1998; Askeland, D., La Ciencia e Ingeniería de Materiales, Iberoamerica, 1990; Mangono, P.L., Ciencia de Materiales: Selección y Diseño, Prentice Hall, 2001; and other sources.
1 Introduction

The accidental discovery of precipitation hardening of aluminum alloys was discovered in Germany by Prof. Alfred Wilm, when hardness tests were repeated on a Duralumin sample, an aluminum-copper alloy, after it had been sitting on a laboratory bench for a while. When the test was repeated, a much higher hardness was observed. Researches, therefore, explained that the material had aged. The first name given to the phenomenon was that of aging hardening. Studies on this phenomenon showed that this also occurred in other alloy systems, and that the reason for hardening is the precipitation of a secondary phase from supersaturated solid solutions. Therefore, the correct name of the phenomenon should by “hardening by precipitation”, although it is still known in industry as “age hardening”. Further exploration of the phenomenon should, during the decade of the 1960’s, that the presence of the right sized precipitates actually disrupts dislocation flow. Therefore, the phenomenon should be called “precipitation strengthening” or “strengthening by precipitation”. The actual scientific name is “dispersion strengthening by secondary phase particles”. In any case, the normal procedure to account for the properties change is carried out via the hardness test, reason why the generally accepted name is “age hardening”.

The fundamental requirements for an alloy to be strengthened by the dispersion of secondary phase particles are the following (see Figure 1):

a) A large maximum solid solubility at high temperature; i.e. that the alloy presents increasing solid solubility as the temperature increases.

b) The alloy composition must be in between the maximum and the minimum solubility.

c) That on quenching, the solid solution becomes supersaturated. This implies that the TTT curve of the alloy presents a feasible critical cooling rate to avoid diffusional transformations.

Since the quenched alloy (at room temperature) contains more solute in solution than indicated by thermodynamic equilibrium, it is a supersaturated, unstable solution that tends to precipitate the excess solute into a dispersion of secondary phase particles when heated slightly.

Figure 1: Typical phase diagram of age hardening alloys.
2 Age Hardening

2.1 Strengthening by a Dispersion of Secondary Particle

A dispersion of secondary phase particles strengthens (and hardens) an alloy by hindering the flow of dislocations in various ways:

- Small particles produce coherency strain field:
  - As long as the particles are small they seek to maintain a close relationship with the lattice of the matrix, and this causes strain in the matrix that acts just as the strain fields of dislocations or other solutes.

- Large incoherent particles:
  - As the particles grow, they lose coherency; and therefore, the strain field disappears.
  - If the particles are "hard", dislocations have to find ways to to surround them.
    - Bowing
    - Climbing
    - Cross Slip
  - If the particles are "soft" dislocations tend to cut through them.
    - Particles cutting also known as chemical hardening.

2.2 The age hardening process

Heat treatment of nonferrous alloys is generally performed at temperatures below the reaction temperature\(^2\). As seen in Figure 2, age hardening temperatures are lower than annealing temperatures, and well below the reaction temperature. In fact, the age hardening temperature has to be below the solvus temperature of the alloy in order to completely exploit the precipitation effect.

Based on the requirements established in the above section, the heat treatment process to achieve strengthening by the dispersion of secondary phase particles ("age hardening") consists of the following 3 stages, illustrated in Figure 3:

1. Solution Treatment: The term "solution" indicates that the alloy is heated to a temperature at which all the material becomes a single solid solution. This stage is also known as the homogenization treatment (see Figure 4 for the A6160 alloy).
2. Quenching: To form a supersaturated solution the cooling rate must be greater than the critical cooling rate. This is most critical, otherwise the amount of solute in the supersaturated solution will decrease and the hardening effect will be significantly reduced.
3. Ageing: The hardening of the alloy is achieved by precipitating the excess solute into its equilibrium phase in the form of a transient, metastable and coherent precipitate. The hardening effect is due to the strain induced by the coherent precipitate. When the precipitation is done at room temperature, it is a natural aging; when it is done at higher temperatures, it is called artificial aging.

\(^2\) In many cases nonferrous alloys, on the diluted side, are eutectic in nature.
Figure 2: Typical temperature ranges for nonferrous alloy heat treatment processes.

Figure 3: Schematic illustration of the age hardening heat treatment for nonferrous alloys.

Figure 4: The Al-Mg$_2$Si Phase Diagram.
3 Age Hardening of Aluminum Alloys

The age hardening heat treatment of aluminum alloys is generally a somewhat longer procedures at low temperature. The hardening and/or strengthening effect of the precipitation process can be better explained by the curves in Figure 5. These “aging curves” show the changes in basic mechanical properties over time when heated at constant temperature to promote secondary phase particle precipitation. Note that peak hardness and strength is reached faster at higher temperatures, but the maxima is always lower than that for transformation at lower temperatures. This effect can be easily associated with average particle size and distribution.

![Figure 5a](image1.png)  
Figure 5a: Effect of ageing on the tensile strength of an A6160 aluminum alloy.

![Figure 5b](image2.png)  
Figure 5b: Effect of ageing on the yield strength of an A6160 aluminum alloy.

![Figure 5c](image3.png)  
Figure 5c: Effect of ageing on the ductility of an A6160 aluminum alloy.

![Figure 5d](image4.png)  
Figure 5d: Effect of ageing on the hardness of an A6160 aluminum alloy.

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Particle size control is a very important feature of the ageing heat treatment process. Figure 6 shows the microstructure of a cast A356 alloy solution heat-treated for 6 h at 540°C. Note that the process has not dissolved the Mg$_2$Si particles, which are elongated and have an average diameter well below 5 μm.

![Figure 6: Microstructure of the alloy A356 solution heat-treated 6 h at 540°C.](image)
(a) Optical micrograph of a polished section.
(b & c) SEM images showing silicon particles exposed after a deep-etching procedure.

However, when hot worked, age hardenable aluminum alloy have a much refined recrystallized microstructure which on solution heat-treatment and ageing produce a much finer distribution of equiaxed secondary phase particles which range in average diameter for 0.1 to 2 μm. The combined effect of the distribution of particle sizes typically shows a double-peak behavior as depicted in Figure 7. Obviously, the maximum strengthening effect will be reached at the point where particles are evenly distributed and big enough to obstruct dislocation flow. This point is reached when particle-matrix coherency begins to break down.

![Figure 7: Combined strengthening effects in age hardenable aluminum alloys as a function of precipitated particle size.](image)


4 Ageing Heat Treatment Procedure

1) Preparation:
   a. Set the first box furnace to 585° C
   b. Set the second box furnace to 230° C.
   c. The instructor will explain the general procedure for the session.
   d. Mark each sample as not to confuse them.
   e. Take the initial hardness of each of the 8 samples (at least 3 replicas).

2) Solution treatment:
   a. Place all samples at 585° C for 30 minutes.
   b. Quench all samples into water.
   c. Turn the first box furnace back to 260° C
   d. Take the hardness of each sample (at least 3 replicas).

3) Ageing treatment:
   a. Place sample 1 - 5 at 230° C.
   b. Place sample 6 - 8 at 260° C.
   c. Extract samples and quench into water according to the following schedule:

<table>
<thead>
<tr>
<th>Sample #</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time (min)</td>
</tr>
<tr>
<td>----------</td>
</tr>
<tr>
<td>5</td>
</tr>
<tr>
<td>10</td>
</tr>
<tr>
<td>20</td>
</tr>
<tr>
<td>30</td>
</tr>
<tr>
<td>40</td>
</tr>
<tr>
<td>50</td>
</tr>
</tbody>
</table>

4) Take the hardness of each sample (at least 3 replicas).
5) Plot the results into the provided chart.
6) Analyze and discuss your hardness results contrasting with the provided metallographic images and stress-strain curves of an Al-6061 heat treated as per the experiment just finished.
Appendix A: Aging Characteristics of Aluminum Alloys

If the precipitation process does not produce the coherent transition stages, no matter how much precipitation occurs, there will be no increase in strength or hardness. The alloys that present precipitations without hardening are described as non-heat-treatable. The binary Al-Si and Al-Mg alloys present a considerable degree of precipitation when heat treated, but the changes observed in the mechanical properties are relatively insignificant. Therefore, these alloys are non-heat-treatable.

The main commercial heat-treatable aluminum alloy systems rely on precipitation intermetallic phases, and are the following:

- Al-Ci system with hardening by CuAl₂ (2XXX)
- Al-Mg-Si system with hardening by Mg₂Si (6XXX)
- Al-Mg-Zn system with hardening by MgZn₂ (7XXX)
- Al-Li² system with hardening by Al₃Li (8xxx)

The commonly available aluminum alloys are described in Table A1.

After a solution treatment the alloy is in its softest condition. This temper condition offers the optimum opportunity for any forming operations to shape the material into complex components. However, during solution treatment, warpage and distortion can be induced. Therefore, in practice, this process is almost immediately followed by cold working, shaping, or straightening. Furthermore, cold working an alloy in the solution condition increases the dislocation density, and with it, the number of potential heterogeneous nucleation sites for the following ageing process.

Table A1: Commonly Available Aluminum Alloys\(^7\).

<table>
<thead>
<tr>
<th>Wrought alloys:</th>
<th>Casting alloys:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1xxx</td>
<td>1xx.x.</td>
</tr>
<tr>
<td>2xxx</td>
<td>2xx.x.</td>
</tr>
<tr>
<td>3xxx</td>
<td>3xx.x.</td>
</tr>
<tr>
<td>4xxx</td>
<td>4xx.x.</td>
</tr>
<tr>
<td>5xxx</td>
<td>5xx.x.</td>
</tr>
<tr>
<td>6xxx</td>
<td>7xx.x.</td>
</tr>
<tr>
<td>7xxx</td>
<td>8xx.x.</td>
</tr>
<tr>
<td>8xxx</td>
<td>9xx.x.</td>
</tr>
<tr>
<td>9xxx</td>
<td>Not currently used</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Commercially pure Al (&gt;99% Al)</th>
<th>Commercially pure Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Not age-hardenable</td>
<td>Not age-hardenable</td>
</tr>
<tr>
<td>Age-hardenable</td>
<td>Age-hardenable</td>
</tr>
<tr>
<td>Age-hardenable if magnesium is present</td>
<td>Some are age-hardenable</td>
</tr>
<tr>
<td>Not age-hardenable</td>
<td>Not age-hardenable</td>
</tr>
<tr>
<td>Age-hardenable</td>
<td>Age-hardenable</td>
</tr>
<tr>
<td>Age-hardenable</td>
<td>Age-hardenable</td>
</tr>
<tr>
<td>Age-hardenable</td>
<td>Age-hardenable</td>
</tr>
<tr>
<td>Age-hardenable</td>
<td>Age-hardenable</td>
</tr>
</tbody>
</table>

\(7\) The Al-Li system is interesting because it hardens and increases the modulus of elasticity, as it decrease in density.

Appendix B: Heat Treatments for Aluminum Alloys

The different thermomechanical treatments, also known as tempers, for non-ferrous alloys, and particularly for Aluminum alloys, are designated by a letter specifying the type and a number indicating the degree to which the treatment is carried out.

These thermomechanical treatments, which can be used on cast and hot worked alloys, include the following 5 types:

- **F**: standing for “as fabricated”, that is a nonspecific metallurgical condition.
- **O**: is the fully annealed condition.
- **H**: considers all the cold worked conditions (these tempers apply only to previously hot worked materials).
- **W**: is the solution-heat-treated condition for age-hardenable alloys.
- **T**: stands for “temper” en includes all the aged conditions in which the alloys can be delivered.

All ageing processes are designated “T”. In designations T1 through T5 and T10 the material is cooled from the fabrication temperature and then aged either naturally or artificially. For the T6 through T9 tempers the alloy is first solution-treated (W) and then artificially aged. Table B1 shows the complete temper designations available.

### Table B1: Thermomechanical treatments used on Aluminum Alloys

<table>
<thead>
<tr>
<th>Letter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>As-fabricated (hot-worked, forged, cast, etc.)</td>
</tr>
<tr>
<td>O</td>
<td>Annealed (in the softest possible condition)</td>
</tr>
<tr>
<td>H</td>
<td>Cold-worked</td>
</tr>
<tr>
<td></td>
<td>H1x—cold-worked only. (x refers to the amount of cold work and strengthening.)</td>
</tr>
<tr>
<td></td>
<td>H12—cold work that gives a tensile strength midway between the O and H14 tempers.</td>
</tr>
<tr>
<td></td>
<td>H14—cold work that gives a tensile strength midway between the O and H18 tempers.</td>
</tr>
<tr>
<td></td>
<td>H16—cold work that gives a tensile strength midway between the H14 and H18 tempers.</td>
</tr>
<tr>
<td></td>
<td>H18—cold work that gives about 75% reduction.</td>
</tr>
<tr>
<td></td>
<td>H19—cold work that gives a tensile strength greater than 2000 psi of that obtained by the H18 temper.</td>
</tr>
<tr>
<td></td>
<td>H2x—cold-worked and partly annealed.</td>
</tr>
<tr>
<td></td>
<td>H3x—cold-worked and stabilized at a low temperature to prevent age hardening of the structure.</td>
</tr>
<tr>
<td>W</td>
<td>Solution-treated</td>
</tr>
<tr>
<td>T</td>
<td>Age-hardened</td>
</tr>
<tr>
<td></td>
<td>T1—cooled from the fabrication temperature and naturally aged.</td>
</tr>
<tr>
<td></td>
<td>T2—cooled from the fabrication temperature, cold-worked, and naturally aged.</td>
</tr>
<tr>
<td></td>
<td>T3—solution-treated, cold-worked, and naturally aged.</td>
</tr>
<tr>
<td></td>
<td>T4—solution-treated and naturally aged.</td>
</tr>
<tr>
<td></td>
<td>T5—cooled from the fabrication temperature and artificially aged.</td>
</tr>
<tr>
<td></td>
<td>T6—solution-treated and artificially aged.</td>
</tr>
<tr>
<td></td>
<td>T7—solution-treated and stabilized by overaging.</td>
</tr>
<tr>
<td></td>
<td>T8—solution-treated, cold-worked, and artificially aged.</td>
</tr>
<tr>
<td></td>
<td>T9—solution-treated, artificially aged, and cold-worked.</td>
</tr>
<tr>
<td></td>
<td>T10—cooled from the fabrication temperature, cold-worked, and artificially aged.</td>
</tr>
</tbody>
</table>

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