EXPERIMENT 8
PRECIPITATION HARDENING IN 2024 ALUMINUM

Objective
To study the time and temperature variations in the hardness of Al-4% Cu alloy on isothermal aging.

Introduction
Materials can be hardened by inhibiting the motion of crystal defects called dislocations. In pure metals, the presence of defects (such as vacancies, interstitials, dislocations and grain boundaries) can enhance the strength. In single phase alloys, additional resistance to deformation may arise from the presence of foreign atoms. In two-phase alloys, additional stress is needed to enable the dislocation to intersect the second-phase particles. A finely dispersed precipitate may, therefore, strengthen the material. This phenomenon is termed precipitation hardening.

The thermodynamics of precipitation in 2024 Al can best be understood by referring to the binary phase diagram of Aluminum-Copper in the aluminum-rich region in Figure 8-1.

When the aluminum-copper alloy of less than 5 wt% copper is heated to a temperature just above the solvus line, only one phase (kappa, K) is thermodynamically stable. Other solid phases dissolve (disappear). This process is called solution treatment. The only requirement is that the specimen must be kept at this temperature for a long enough time. To solution treat a sample of 2024 Al (4 wt% Cu), the sample should be heated to 930°F (500°C) and held for 30 minutes.

When a solution treated sample is rapidly cooled (quenched) to below the solvus line (Figure 8-1), two phases are thermodynamically stable (kappa and theta). These phases are two
different solids, physically distinct, and separated by a phase boundary. The process is similar to precipitation of salt in supersaturated brine.

The process of precipitation is not instantaneous, as is often the case in liquid-solid precipitation. The process involves the formation of embryos of theta through thermal fluctuations and their subsequent growth, once they achieve stability. With time, more and more precipitates form. This process is called **aging**. Once the solution achieves an equilibrium composition given by the solvus line for the aging temperature, precipitation stops. For example, the precipitation of the copper-rich theta phase depletes the kappa phase of copper to approximately 1-1/2 wt% Cu at 715°F (380°C).

The distribution of precipitates affects the hardness and yield strength. The hardness and yield strength are greater when the precipitates are small and finely dispersed in the kappa matrix than when the precipitates are large and not finely scattered. Therefore, to gain hardness in 2024 Al, the specimen should be heat treated to produce a fine dispersion of small precipitates.

Unfortunately, there is a tendency when thermodynamic equilibrium is reached for large precipitates to grow and small precipitates to shrink. This will lower the surface to volume ratio of the precipitates, the surface energy, and therefore the energy of the system. As a result, at some point in the aging of 2024 Al, the precipitates begin to coarsen and (on average) the spacing between them gradually increases. At this point, the hardness and the yield point will begin to decrease with time of aging.

![Figure 8-2](image.png)

The process of aging is a function of temperature; the higher the temperature, the wider the spacing of the precipitates. They form initially on cooling from the solution treatment. Also, because coarsening is dependent upon the movement of copper atoms in kappa, the maximum point is generally reached sooner at a higher temperature than at a lower temperature, as shown in **Figure 8-2**.
In the present experiment, the precipitation hardening behavior of the Al-4%Cu alloy will be studied by measuring changes in hardness as a function of aging time.

Materials and Equipment
Five pieces of Al-4%Cu alloy (2024 Al)
Furnace for heat treating specimens at 500°C (930°F)
Pail of water
Hardness testers
Aging furnace at 190°C (370°F)

Procedures
1. Stamp the five 2024 Al specimens with an identifying mark.
2. Measure the hardness of all of the specimens using Rockwell B.
3. Place all five in a heat-treatment crucible and into a furnace for solution treatment at 500°C (930°F) for 30 minutes.
4. Natural Aging - Remove one specimen and drop into a pail of water. Once the specimen is cool, measure the Rockwell B hardness at intervals of approximately 30 min, 90 min, 1 day, and 1 week for this specimen. It will be necessary for one member to come back to the lab during the week when the lab is in session to measure the hardness.
5. Artificial Aging - Remove the remaining four specimens and drop them into a pail of water. Once the specimens are cool remove them from the quenching bucket. Measure the Rockwell B hardness. Next, transfer the specimens to a furnace set at 190°C (370°F). Remove one sample after 3 min, 10 min, 60 min and 90 min. Quench into water and measure the Rockwell B hardness. After one week, again measure the Rockwell B hardness of the 3- and 10-minute artificially aged specimens.

Glossary of Terms
Understanding the following terms will aid in understanding this experiment.

Age hardening. A special dispersion-strengthening heat treatment. By solution treatment, quenching and aging, a coherent precipitate forms that provides a substantial strengthening effect by acting as obstacles to dislocation movement.

Artificial aging. Reheating a solution-treated and quenched alloy to a temperature below the solvus to provide the thermal energy required for a precipitate to form.

Coherent precipitate. A precipitate whose crystal structure and atomic arrangement have a continuous relationship with the matrix from which the precipitate formed. The coherent precipitate provides excellent disruption of the atomic arrangement in the matrix and provides excellent strengthening.

Dislocation. A line imperfection in the lattice of a crystalline material. Movement of dislocations helps explain how materials deform. Interference with the movement of dislocations helps explain how materials are strengthened.

Grain boundary. A surface defect representing the boundary between two grains. The lattice has a different orientation on either side of the grain boundary.
**Interstitialcy.** Atom occupying an interstitial site not normally occupied by an atom in the perfect crystal structure or an extra atom inserted into the perfect crystal such that two atoms occupy positions close to a singly occupied atomic site in the perfect structure.

**Natural aging.** When a coherent precipitate forms from a solution-treated and quenched age hardenable alloy at room temperature, providing optimum strengthening.

**Solid solution.** A solid phase that contains a mixture of more than one element, with the elements combining to give a uniform composition everywhere.

**Solution treatment.** The first step in the age-hardening heat treatment. The alloy is heated above the solvus temperature to dissolve any second phase and to produce a homogeneous single-phase structure.

**Solvus.** A solubility line that separates a single solid phase region from a two solid phase region in the phase diagram.

**Strain energy.** The energy required to permit a precipitate to fit into the surrounding matrix during nucleation and growth of the precipitate.

**Supersaturated solid solution.** The solid solution formed when a material is rapidly cooled from a high-temperature single-phase region to a low-temperature two-phase region without the second phase precipitating. Because the quenched phase contains more alloying element than the solubility limit, it is supersaturated in that element.

**Vacancy.** A vacancy is created when an atom is missing from a lattice point.

**Write Up**
1. Prepare a memo report.
2. **Plot the hardness vs. time of aging for each aging temperature, using Excel.**
3. How does aging temperature affect the time and hardness?
4. What happens to the 190°C (370°F) 3 and 10 minute specimens after one week? Why?
5. If you were going to use 2024 Al in an application at a temperature of 190°C (370°F), what problems could be encountered?
6. Discuss errors in this experiment and their sources.

**References**
3. Smith, Science and Engineering Materials, Chapter 8
4. Flinn & Trojan, Engineering Materials and Their Applications, Chapters 4 and 5
Memorandum Format Used

<table>
<thead>
<tr>
<th></th>
<th>Poor</th>
<th>Fair</th>
<th>Average</th>
<th>Good</th>
<th>Excellent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spelling, grammar, &amp; punctuation correct</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
</tbody>
</table>

**Report includes:**

<table>
<thead>
<tr>
<th>Report includes</th>
<th>Poor</th>
<th>Fair</th>
<th>Average</th>
<th>Good</th>
<th>Excellent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Graph hardness vs. time of aging for specified aging temperatures. (Final result will be 1 natural and 1 artificial curve overlayed; take log of time for X-axis, use Excel).</td>
<td>5</td>
<td>10</td>
<td>15</td>
<td>20</td>
<td>25</td>
</tr>
<tr>
<td>Discuss how aging temperature affects the aging time and hardness.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Discuss what happens to the 190°C, 3 and 10 minute specimens after one week.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Discuss reason for changes in the 190°C, 3 and 10 minute specimens after one week.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Discuss the use of 2024 Al in an application at a temperature of 190°C (370°F).</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Discuss errors in this experiment and their sources.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Include table of results and data collected</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Column Subtotals</th>
<th>Poor</th>
<th>Fair</th>
<th>Average</th>
<th>Good</th>
<th>Excellent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5</td>
<td>10</td>
<td>15</td>
<td>20</td>
<td>25</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Overall level of effort apparent</th>
<th>Poor</th>
<th>Fair</th>
<th>Average</th>
<th>Good</th>
<th>Excellent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quality of graphs</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Quality of Abstract</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Quality of work description</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Quality of conclusions</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Column Subtotals</th>
<th>Poor</th>
<th>Fair</th>
<th>Average</th>
<th>Good</th>
<th>Excellent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
</tbody>
</table>