MSE 528 - PRECIPITATION HARDENING IN 7075 ALUMINUM ALLOY

Objective

To study the time and temperature variations in the hardness and electrical conductivity of Al-Zn-Mg-Cu high strength alloy on isothermal aging.

Introduction

Materials can be hardened by inhibiting the motion of crystal defects called dislocations. 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 or age hardening.

The thermodynamics of precipitation in 7075 Al-alloy can best be understood by referring to the binary phase diagram of Aluminum-Zinc in the aluminum-rich region in Figure 8-1.



Figure 8-1: Schematic phase diagram for an age hardenable alloy.

When the aluminum-zinc alloy of less than 5-6 wt% zinc is heated to temperature just above the solvus line, only one phase is thermodynamically stable. Other solid phases dissolve. This process is called solid-solution heat-treatment. The only requirement is that the specimen must be kept at this temperature for a long enough time. This means to solid-solution heat-treat a sample of 7075 Al (6 wt% Zn) the sample should be heated to 890 $^{\circ}$ F (480 $^{\circ}$ C) and held for 30 minutes.

When a solid-solutionized sample is rapidly cooled to below the solvus line in Figure 8-1, two phases are thermodynamically stable (alpha and beta). 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.



Figure 8-2: Comparison of phases present in Al-Mg-Zn alloy, solution treated, aged at 120 C (250F), dashed lines, and 175 C (350 F) (solid lines)

The process of precipitation is not instantaneous as is often the case in liquid-solid precipitation. The process involves the formation of embryos of 2^{nd} phase 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 Zn-Mg-rich phase depletes the alpha phase (matrix) at approximately at 720°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 matrix than when the precipitates are large and not finely scattered. Thus, to gain hardness the specimen should be heat treated to produce a fine dispersion of small precipitates (coherent).

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 7075 Alalloy, 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.

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 solid-solution heat treatment temperature. Also, because coarsening is dependent upon the movement of atoms (diffusion), the maximum point is generally reached sooner at a higher temperature than at a lower temperature, as shown in Figure 8-3.



Figure 8-3: Effects of temperature on age hardening process.

Aluminum-Zinc-Magnesium and Aluminum-Zinc-Magnesium-Copper Alloys. The aging of rapidly quenched aluminum-zinc-magnesium alloys from room temperature to relatively low aging temperatures is accompanied by the generation of GP zones having an approximately spherical shape. With increasing aging time, GP zones increase in size, and the strength of the alloy increases. Extended aging at temperatures above room temperature transforms the GP zones in alloys with relatively high zincmagnesium ratios into the transition precipitate known as η' or M', the precursor of the equilibrium MgZn₂, η or M phase precipitate. The basal planes of the hexagonal η' precipitates are partially coherent with the {111} matrix planes but the interface between the matrix and the *c* direction of the precipitate is incoherent. Aging times and temperatures that develop the highest strengths, characteristic of the T6 temper, produce zones having an average diameter of 2 to 3.5 nm (20 to 35 A) along with some amount of η' . The nature of the zones is still uncertain, although they undoubtedly have high concentrations of zinc atoms and probably magnesium atoms as well.

Several investigators have observed that the transition phase η' forms over a considerable range of compositions that are in the Al + T field, as well as those in the Al + η field under equilibrium conditions. With increased time or higher temperature, the η' converts to (MgZn₂) or, in cases-where T is the equilibrium phase, is replaced by T (Mg₃Zn₃Al₂). Evidence exists for a transition form of T in alloys with lower zinc-magnesium ratios, at times and temperatures that produce over aging. The precipitation sequence depends on composition, but that of rapidly quenched material aged at elevated temperatures may be represented as:

$$SS \rightarrow GP \text{ zones [spherical]} \bigvee^{\eta' \rightarrow \eta}_{T' \rightarrow T}$$

In this schematic, GP zones nucleate homogeneously, and the various precipitates develop sequentially within the matrix. However, the presence of high-angle grain boundaries, subgrain boundaries, and lattice dislocations alters the free energy such that significant heterogeneous nucleation may occur either during quenching or aging above a temperature known as the GP zone solvus temperature. Above this temperature, the semicoherent transition precipitates nucleate and grow directly on dislocations and subgrain boundaries, and the incoherent equilibrium precipitates nucleate and grow directly on high-angle boundaries. These heterogeneously nucleated precipitates do not contribute to strength and, hence, decrease attainable strength by decreasing the amount of solute available for homogeneous nucleation.

Decreasing the quench rate has another consequence besides allowing solute atoms an opportunity to nucleate heterogeneously. Slow quenching permits vacancies to migrate to free surfaces and become annihilated. Decreasing the number of vacancies decreases the temperature at which GP zones nucleate homogeneously. Therefore, a particular aging temperature may allow only homogeneous nucleation to occur in rapidly quenched materials, but may allow heterogeneous nucleation to predominate in slowly quenched material. Under the latter conditions, the precipitate distribution is extremely coarse, so strength developed is particularly low. Some of the loss in strength from slow quenching in this case can be minimized by decreasing the aging temperature to maximize homogeneous nucleation.

When an aged aluminum-zinc-magnesium alloy is exposed to a temperature higher than that to which it has previously been exposed, some GP zones dissolve while others grow. Whether a GP zone dissolves or grows depends on its size and on the exposure temperature. When the zone size is large enough, most of the zones transform to transition precipitates even above the GP zone solvus temperature. This phenomenon is the basis for the two-step aging treatments that most often recommended for these alloys.

The addition of up to 1% copper to the aluminum-zinc-magnesium alloys does not appear to alter the basic precipitation mechanism. In this range, the strengthening effects of copper are modest and attributed primarily to solid solution. Higher copper contents (2% or higher) afford greater precipitation hardening, with some contribution of copper atoms to zone formation, as indicated by an increased temperature range of zone stability. Crystallographic arguments indicate that copper and aluminum atoms substitute for zinc in the MgZn₂ transition and equilibrium precipitates. In the quaternary aluminum-zinc-magnesium-copper system, the phases MgZn₂ and MgAlCu form an isomorphous series in which an aluminum atom and a copper atom substitute for two zinc atoms. Moreover, electropotential measurements and x-ray analyses indicate that copper atoms enter into the η ` phase during aging temperatures above about 150 °C (300 °F). These observations are significant because aging aluminum-zinc-magnesium-copper alloys containing above about 1% copper above this temperature substantially increases their resistance to stress-corrosion cracking. Little effect is shown on the stress corrosion of alloys containing lower amounts of copper.

In the present experiment, the precipitation hardening behavior of the Al-6%Zn-2%Mg-2%Cu alloy will be studied by measuring changes in hardness and electrical conductivity as a function of aging time.

Materials and Equipment Five pieces of Al-Zn-Mg-Cu alloy (7075 Al) Furnace for heat treating specimens at **480°C (895°F)** Pail of water Hardness testers Aging furnace at **160°C (320°F)**

<u>Procedures</u> Obtain six specimens of 7075 Al. Stamp the specimens (using hammer) with an identifying mark. Measure the hardness and electrical conductivity of all of the specimens using Rockwell B, and IACS%. Place all six samples in the crucible inside the furnace for solid-solution heat-treatment at **480°C** (**895°F**)

Artificial Aging

for 30 minutes.

Remove the specimens and drop them into a pail of water. Once the specimens are cool, remove them from the quenching bucket. Wait 10 minutes, then measure the Rockwell B hardness and electrical conductivity. Next, transfer the specimens to a furnace set at 160°C (320°F). Remove one sample each after 3 min, 10 min, 30 min, 60 min and 90 min. Quench in water and measure the Rockwell B hardness

and electrical conductivity. Make sure to measure the Rockwell B hardness and electrical conductivity on the sixth sample (**natural aging** for comparison with the above results).

Write Up

- 1. Prepare a memo report.
- 2. Plot the hardness vs. time of aging (convert X-axis to log scale AFTER graphing data in Excel; right mouse click on X-axis). Convert aging time to seconds from minutes, then plot.
- 3. Plot the electrical conductivity vs. time of aging
- 4. If you were going to use 7075 Al-alloy in an application at a temperature of 160°C (320°F), what problems could be encountered? Discuss over aging.

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 impeding dislocation movement.

Artificial aging - Reheating a solution-treated and quenched alloy to a temperature below the solvus in order 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 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.

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.

Solid-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 2 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 form 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.