Objective

The objective of this investigation is to learn how and why certain alloys (primarily Aluminum alloys) can be strengthened by Precipitation Hardening heat treatment processes.

Abstract

The strength and hardness of some metal alloys may be enhanced by the formation of extremely small uniformly dispersed particles of a second phase within the original phase matrix; this must be accomplished by appropriate heat treatment. The process is called Precipitation Hardening or Age Hardening which involves three distinct steps: Solution Treatment to minimize segregation in the alloy, Quenching to create a supersaturated solid solution and Aging to facilitate the formation of coherent precipitates which strengthen the alloy by interfering with dislocation movement. Both the Natural Aging and Artificial Aging processes will be investigated by measuring the hardness of 2024 aluminum specimens at various time intervals into the aging process following the quench. Phenomena related to precipitation hardening, such as Guinier-Preston zones, overaging, coherent participates, etc., will be discussed.

Background

The strength and hardness of some metal alloys may be enhanced by the formation of extremely small uniformly dispersed second-phase particles within the original phase matrix in a process known as "Precipitation (or Age) Hardening". The precipitate particles act as obstacles to dislocation movement and thereby strengthen the heat-treated alloys. Many aluminum based alloys, copper-tin, certain steels, nickel-based super-alloys and titanium alloys can be strengthened by age hardening processes. In order for an alloy system to be able to be precipitation-strengthened, there must be a terminal solid solution that has a decreasing solid solubility as the temperature decreases. The Al-Cu phase diagram shown in Figure 1 shows this type of decrease along the solvus between the α and $\alpha + \theta$ regions. Consider a 96wt%Al – 4wt%Cu alloy which is chosen since there is a large degrease in the solid solubility of solid solution α in decreasing the temperature from 550°C to 75°C. The precipitation-hardening process involves three basic steps: Solution Treatment, Quenching and Aging.
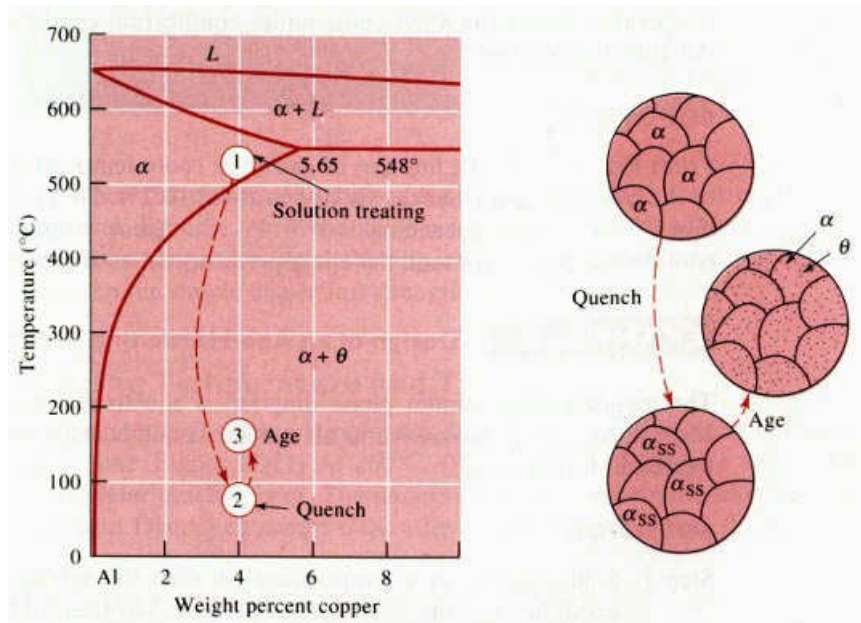

Figure 1 - The aluminum rich end of the Al-Cu phase diagram showing the three steps in the age-hardening heat treatment and the microstructures that are produced. [Askeland, 1994]

Solution Treatment, or Solutionizing, is the first step in the precipitation-hardening process where the alloy is heated above the solvus temperature and soaked there until a homogeneous solid solution (α) is produced. The θ precipitates are dissolved in this step and any segregation present in the original alloy is reduced.

Quenching is the second step where the solid α is rapidly cooled forming a supersaturated solid solution of α_{ss} which contains excess copper and is not an equilibrium structure. The atoms do not have time to diffuse to potential nucleation sites and thus θ precipitates do not form.

Aging is the third step where the supersaturated α, α_{SS}, is heated below the solvus temperature to produce a finely dispersed precipitate. Atoms diffuse only short distances at this aging temperature. Because the supersaturated α is not stable, the extra copper atoms diffuse to numerous nucleation sites and precipitates grow. The formation of a finely dispersed precipitate in the alloy is the objective of the precipitation-hardening process. The fine precipitates in the alloy impede dislocation movement by forcing the dislocations to either cut through the precipitated particles or go around them. By restricting dislocation movement during deformation, the alloy is strengthened.

In the age hardening of Al-Cu alloys, four structures of the precipitates can be recognized: (1) GP-1 Zones, (2) GP-2 Zones (also called θ''), (3) the θ' Phase, and (4) the θ -(CuAl$_2$) Phase. A large number of very small solute-rich clusters form during the early stages of the aging process that are completely coherent, that is they form on the FCC lattice of the matrix (Figure 2). Since the atomic dimensions of the solute-rich regions will differ somewhat from that of the lattice, strains occur in the lattice regions around the clusters. These regions that are distorted by the clusters are referred to as Guinier-Preston zones, or

(a)   (b)
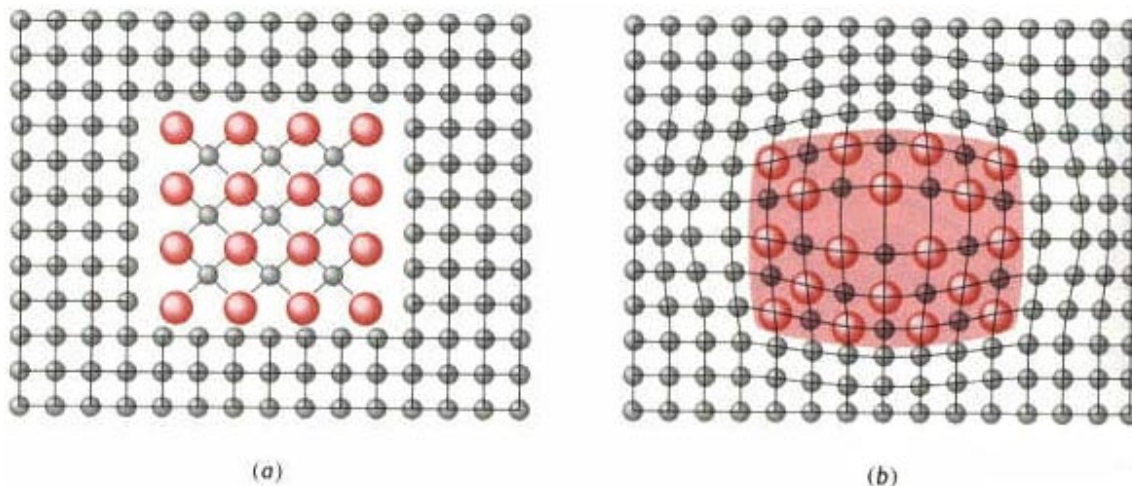
GP zones.

Figure 2 - (a) A noncoherent precipitate which has no relationship with the crystal structure of the surrounding matrix. (b) A coherent precipitate that is clearly related to the surrounding matrix's crystal structure. [Askeland, 1994].

GP-1 zones grow rapidly up to a certain size, typical of the temperature of aging, and then growth practically stops. At room temperature this size is 3-7 nm thick and at 70-130°C the diameter is of the order of 10-15 nm. Hardening and a decrease in ductility accompany the formation and growth of the GP-1 zones; these effects have been attributed to the distortion of the lattice which inhibits the movement of dislocations. The GP-1 zones are eventually replaced by GP-2 zones during the aging process. GP-2 zones ("θ''" phase) are larger than the GP-1 zones and their number is correspondingly smaller since the amount of solvent in the zones does not change. The size range for the θ'' phase is 10-100 nm diameter, and 1-4 nm thick. Optimal mechanical properties are achieved by growing the θ'' phase as large as possible without beginning to from the intermediate θ' phase.

With continued aging, the intermediate 'θ' phase will begin to form causing recrystallization, softening, and a decrease in strength; this is known as over-aging. The θ' phase has a tetragonal structure with a different lattice parameter from the matrix; no coherency strains exist, but each particle is surrounded by a ring of dislocations. The size of the θ' phase depends on time and temperature; size ranges from 10 to 600 nm diameter with a thickness of 10-15 nm.

Eventually the θ' phase is replaced by θ-(CuAl$_2$) which has the same structure and composition as the θ phase found by solidification.

Not all structures form at all aging temperatures. Below 80°C aging seldom progresses past the GP-1 stage; only above 220°C does the θ' phase appear within a reasonable time interval, and temperatures above 280°C are needed for the θ phase to appear. Conversely, at higher temperatures the early stages do not appear and the GP-1 zones do not appear above 180°C; the θ'' phase will not form above 230°C. Maximum hardness is reached sooner at higher temperatures and the maximum hardness reached generally decreases as the aging temperature is raised for precipitation-hardening.
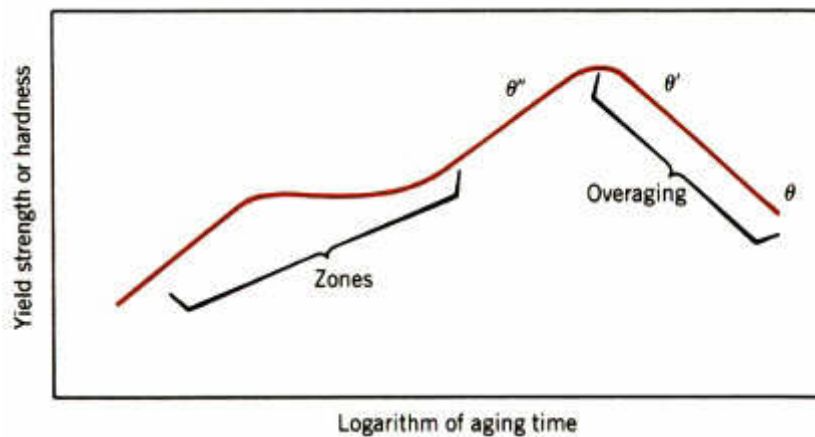
Figure 3 – Schematic diagram showing tensile strength and hardness as a function of the logarithm of aging time at constant temperature during the precipitation hardening. [Callister, 1991]

Activation Energy, Q, refers to the amount of energy required to cause an atom to move (diffuse) where

$$Q = 1.987 \left[(T_1)(T_2) / (T_2 - T_1) \right] \left[2.303 \log_{10} (t_1 / t_2) \right] (cal / mole)$$

T_1 = Room Temperature

T_2 = Artificial Aging Temperature

t_1 and t_2 = Aging Times

R = Gas Constant = 1.987 cal/mol°K

Example: Two pieces of the same aluminum alloy were Precipitation Hardened; one by Natural Aging (25°C) and one by Artificial Aging (180°C). If it took 10 minutes for the Natural Aged specimen to reach a RHB of 43 and it took only 3 minutes for the Artificial Aged specimen to reach a RHB of 43, determine the Activation Energy, Q, for the aluminum alloy.

Procedure

MATERIALS: Aluminum Alloy 2017 (Al - 4wt%Cu – 0.5wt%Mn – 0.5wt%Mg)

1. Solution treat four Al 2017 alloy specimens for 12 hours (overnight) at 550°C; two of the specimens should be cut into 6 equally sized pieces. (Lab Instructor).

2. Rapidly quench all of the specimens in ice water.

3. Immediately following quenching, place one of the Charpy specimens and all of the small cut pieces into an oven that has been preheated to 180°C.

4. Immediately following quenching, take one Rockwell Hardness (HRB) measurements on the Natural Aging specimen; make sure the reading is taken away from the notched region of the specimen.

5. Measure the hardness of the Natural Aging specimen over the course of cooling according to the provided data sheet (T4 Treatment -> aging at room temperature) taking one HRB hardness reading after 0, 2, 4, 6, 8, 10, 15, 20, 30, 45, 60, 75, and 90 minutes. After 90 minutes, conduct a Charpy Impact test on the specimen. Complete the Natural Aging Data Sheet provided.

6. Using the small piece samples, measure the hardness due to Artificial Aging over the course of the process (T6 Treatment -> artificial aging) taking one HRB hardness reading after 0, 1, 2, 4, 6,

8, 10, 12, 15, 20, 30, 45, and 60 minutes.   After 60 minutes, conduct a Charpy Impact test on the Artificial Aging Charpy specimen. Complete the Artificial Aging Data Sheet provided.

Natural Aging (Room Temperature)

TIME (MIN)	HARDNESS(HR$_B$)
0	
2	
4	
6	
8	
10	
15	
20	
30	
45	
60	
75	
90	

Artificial Aging (180°C)

TIME (MIN)	HARDNESS (HR$_B$)
0	
1	
2	
4	
6	
8	
10	
15	
20	
30	
45	
60	

Lab Requirements

Follow the procedure outlined in the "Procedure" section of this module then complete the following:

1. Construct a graph showing the hardness data obtained versus time for the two aging processes investigated; include both plots in a single graph.

2. For Artificial Aging, describe the precipitation hardening process in terms of the regions obtained on the Artificial Aging plot including brief discussions about the microstructures which should be present in each region.

3. Discuss which aging process shows the highest hardening effect? Explain why.

4. Based on the test results, suggest how one could maximize the strength of an Al 2017 alloy at room temperature? What effect would you expect maximizing the alloy's strength would have on its' Fracture Toughness?

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5. Compare the measured Impact Energies for the two specimens and discuss the results.

6. How could one maximize the amount of Impact Energy absorbed by an Al 2017 alloy at room temperature?

7. Calculate the Activation Energy, Q, for the Al 2017 material being investigated; discuss the results.

Homework

1. What is Age Hardening and how does it differ from Quench Hardening?

2. Briefly describe the three steps involved in the Precipitation Hardening process.

3. What are Guinier-Preston Zones?

4. What's the difference between a Coherent and a Non-Coherent Precipitate?

5. Why would the formation of the θ' phase generally be undesirable, and the formation of θ-(CuAl2) be even less desirable, when precipitation hardening an alloy?

6. What metals does a 2024-T4 alloy contain? Briefly define/explain what the T4 represents?

7. What metals does a 6061-T6 alloy contain? Briefly define/explain what the T6 represents?

8. Consider the following three alloys from the Al-Cu system (see Al-Cu Phase Diagram): Pure Aluminum, 3wt%Cu-97wt%Al, and 8wt%Cu-92wt%Al.

   • Which of these alloys is the best candidate for Age Hardening? Why?

   • Which of these alloys is the worst candidate for Age Hardening? Why?

Data Sheet: Precipitation Hardening – Natural Aging

Specimen Description:	
Date & Time:	
Hardness Load Scale and Indenter Type:	

Time (Min)	Rockwell Hardness Measurement
0	
2	
4	
6	
8	
10	
12	
15	
20	
30	
45	
60	
75	
90	

Data Collected by: Name & Signatures	

Data Sheet: Precipitation Hardening – Artificial Aging (180°C)

Specimen Description:
Date & Time:
Hardness Load Scale and Indenter Type:

Time (Min)	Rockwell Hardness Measurement
0	
1	
2	
4	
6	
8	
10	
12	
15	
20	
30	
45	
60	

Data Collected by: Name & Signatures	