Precipitation hardening

Objective

The objective of this laboratory is for you to explore the kinetics and strengthening of Al by precipitation hardening.

Preparation

Read sections 10.1-10.5 of Meyers and Chawla.

Equipment and samples

- First week: Differential scanning calorimeters; ovens for heat treating Al alloys. Second week: mechanical testing facilities in Talbot.

- Discs of Al 2024 alloy for DSC studies; bars of Al 2024 for tensile testing and hardness testing.

- Computer, plotting software.

Introduction

Pure fcc metals such as Al have low yield strengths because the stress required to move a dislocation is small. One method of strengthening an alloy is through precipitation reactions. Precipitation hardening is the process of hardening or strengthening of an alloy by precipitating finely dispersed precipitates of the solute in a supersaturated matrix. This process involves the following three basic steps:

1. The first step is the solution heat treatment or homogenization. Figure 1 illustrates this phenomenon. During this step, an alloy of composition X_1 is heated to a temperature T_1, between the solvus and solidus temperatures and soaked there until all of the solute dissolves into the α phase and a uniform solid-solution structure is produced.

2. The second step is quenching. Quenching is simply cooling the sample rapidly to a lower temperature, T_3, usually room temperature, and the cooling medium is usually water at room temperature. During quenching, the solute is not immediately able to diffuse out of a phase and the alloy is said to be supersaturated. The rationale behind the quenching process is to preserve the uniform solid solution structure of the alloy below the homogenization temperature.

3. The final step in the precipitation hardening process is aging. It is the process of precipitating incoherent precipitates from a supersaturated solid solution. When aging occurs at room temperature, it is called natural aging. Aging above room temperature is called artificial aging.
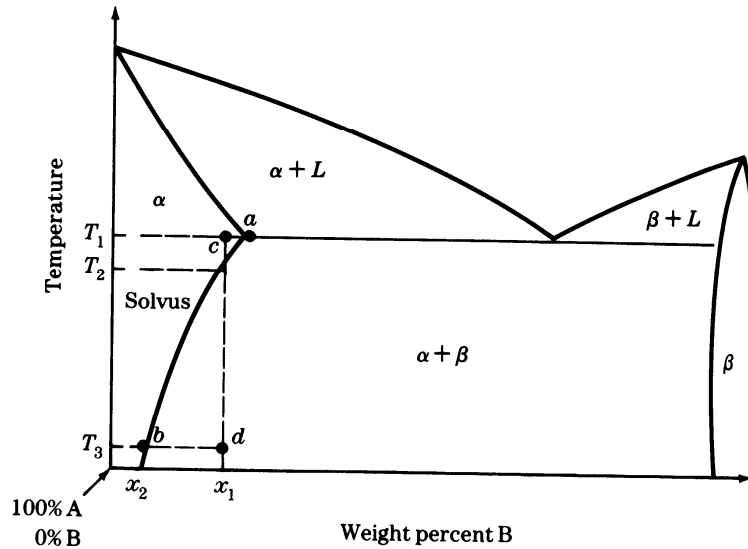
Figure 1. Binary phase diagram for two metals A and B having a terminal solid solution   which has a decreasing solid solubility of B in A with decreasing temperature [1].

The phase diagram for Al-Cu is illustrated in Fig. 2 By solution annealing an Al alloy (2024) containing ≈ 4.4 Wt% Cu, and then quenching, i.e. cooling so fast that little diffusion is possible, the Cu can remain dissolved in Al, but it is not in equilibrium. It was mentioned above that solute hardening is not an effective hardening mechanism, and so the quenched alloy must be slowly annealed to precipitate incoherent Al_2Cu (θ phase) particles. Since the number density of precipitates controls hardness, not the size of the individual particles, the trick is to nucleate as many particles as possible but keep them from growing. If the anneal is too long or at too high a temperature, some precipitates will grow while others shrink and disappear, lowering the number of precipitates and the strength. This condition is called overaging.
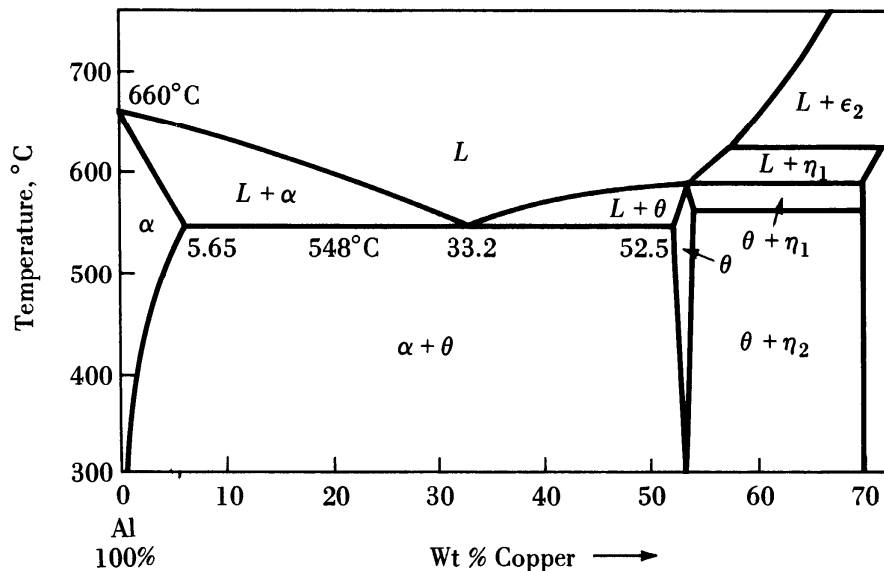
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Figure 2. Phase diagram for Al-Cu [1].

As it turns out, Cu in aluminum do not simply precipitate out according to the phase diagram, but rather it forms metastable precipitate phase, before finally forming the equilibrium phase, such as Al_2Cu. For Al-Cu, the first phase to form is a fully coherent precipitate called a GP (Guinier Preston) zone. This is followed by intermediate θ'' and θ' phases before the stable θ (Al_2Cu) phase forms. As these reactions occur, the hardness changes, at first increasing as the precipitates nucleate and grow (or age), and changing from coherent to incoherent phases. Eventually the strength decreases as the incoherent precipitates grow in size but decrease in number (i.e., overage), see Fig. 3.
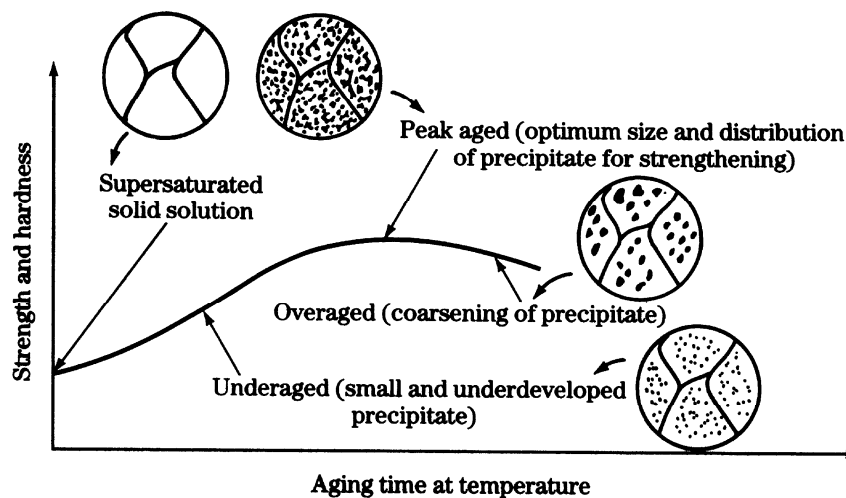

Figure 3. Microstructural development with aging time [1].

The 2024 alloy is unstable at room temperature after solution heat treatment and may reach substantial hardness levels with "natural aging".

Session 1: Scanning calorimetry to determine the kinetics of precipitation of Al 2024 alloy

- In this session of the lab, you will analyze precipitation kinetics in a 2024 series aluminum alloy. Samples for differential scanning calorimetry have been prepared for you at various annealing times and temperatures. Each group will work with one temperature, observing the changes in the DSC data with increasing annealing times. You can then look at data acquired by other groups data in the class to see the effect that temperature has on the kinetics of this reaction.

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- Use a DSC measurement of quenched 2024 Al to measure the total enthalpy associated with the precipitation of 2024 Al.

- Use DSC to measure the fraction of Cu that has precipitated at as a function of annealing time.

- You will be given tensile bars of 2024 to perform your own age hardening on in the lab. Each student will have one sample and will pick an aging time. Three furnaces will be set up with quench tanks next to them. After your sample has been aged for the desired time, you will pull it out and drop it in the quenching water. It will then be marked and stored for use in the Talbot Mechanical Testing Lab the following week.

Session 2: Yield strength and hardness of Al 2024 alloy

- John Williams and the TA will describe the procedures you will follow at the Talbot Mechanical testing lab.

Instrument procedures: DSC

1. Log on to the computer station using your NetID and password.
2. Under Start -> All Programs -> PYRIS Software, launch Pyris Manager.
3. Click

4. In the Method Editor, enter Sample ID, Operator ID, and any comments. Enter the sample mass. Under the File Name section, choose a desired location (Desktop works best) and a name for your data file.

5. Under the Initial State tab, change the Initial Temp to 25°C. Y-initial should be 0.

6. In the Program tab, heat from 25 to 400°C at a rate of 25°C/min.
7. Remove the inner and outer lids of the DSC using tweezers. Make sure a reference pan is present in the furnace. Add the sample pan, making sure it is centered on the small circular platform. Replace the inner and outer lids.

8. If everything is set the way you want it, click to start the run.
9. After the curve has been generated, click on Calculate -> Peak Area and set the outer bounds on either side of the peak you wish to analyze.

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10. Wait until the DSC has cooled down to 25°C before changing samples.

When you are done using the DSC for the day, exit out of Pyris Manager and tell the TA. Do not turn off the DSC, intercooler, or gas regulator. Transfer all of your ASCII files and any screen shots or other files either onto a USB memory stick or via email, netfiles, etc. Take your sample out of the DSC and place it in the provided sample trash bin.

References:

1. W.F. Smith, *Foundations of Materials Science and Engineering*, McGraw-Hill, Inc., 2nd Ed, New York, 1993.

2. This laboratory handout was originally prepared for ME-231. The help of P. Hurath, J. Williams, T. Mackin and B. Coverdill is gratefully acknowledged.

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