PHASE TRANSFORMATIONS & HEAT TREATMENT

LABORATORY OBSERVATION BOOK

Name of the Student: .................................................................

Roll No: ....................................................................................

LABORATORY COORDINATOR(S)

A. LAVA KUMAR  
Assistant Professor

SUBHADRA SAHOO  
Assistant Professor

Department of Metallurgy & Materials Engineering

VEER SURENDRRA SAI UNIVERSITY OF TECHNOLOGY, BURLA

Odisha - 768018
FOREWORD

This lab manual is designed to suit the needs of the undergraduate metallurgist in acquainting with the heat treatment principles and transformations in materials. The metallographic techniques are stressed, as these practices are essential for a metallurgical engineer. In this observation book an attempt is made to clarify the concepts of Annealing, Normalizing, Hardening, Tempering and Hardenability. The students shall get clarity about microstructures of carbon steels, cast irons and non-ferrous alloys, in particular to differentiate between various phases present in metals and alloys. The student shall also be trained in grain size measurements, and quantitative metallography.

Instructor will indicate the experiments to be conducted and advise on the nature of the lab report and time of submission. However, a typical format for a lab report is included in this observation book. It is important that all the information necessary to complete the lab report is obtained before students leave the lab.

Laboratory Notebook Grading: 20% Neatness and Legible, 20% Experimental Description, 20% Observations and Comments, 20% Data Collection, 20% Data Analysis including plots, calculations, and conclusions drew.

Mr. A.Lava Kumar
Assistant Professor

Ms. Subhadra Sahoo
Assistant Professor
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SAFETY IN THE LABORATORY

All students must read and understand the information in this document with regard to laboratory safety and emergency procedures prior to the first laboratory session. Your personal laboratory safety depends mostly on you. Effort has been made to address situations that may pose a hazard in the lab but the information and instructions provided cannot be considered all-inclusive. Students must adhere to written and verbal safety instructions throughout the academic term.

Common Sense

Good common sense is needed for safety in a laboratory. It is expected that each student will work in a responsible manner and exercise good judgment and common sense. If at any time you are not sure how to handle a particular situation, ask your Teaching Assistant or Instructor for advice. DO NOT TOUCH ANYTHING WITH WHICH YOU ARE NOT COMPLETELY FAMILIAR!!! It is always better to ask questions than to risk harm to yourself or damage to the equipment.

Personal and General laboratory safety

1. Never eat, drink, or smoke while working in the laboratory. Read labels carefully.
2. Do not use any equipment unless you are trained and approved as a user by your supervisor.
3. Wear safety glasses or face shields when working with hazardous materials and/or Equipment. Wear gloves when using any hazardous or toxic agent.
4. Clothing: When handling dangerous substances, wear gloves, laboratory coats, and safety shield or glasses. Shorts and sandals should not be worn in the lab at any time. Shoes are required when working with the heat treatment furnaces.
5. If you have long hair or loose clothes, make sure it is tied back or confined.
6. Keep the work area clear of all materials except those needed for your work. Extra books, purses, etc. should be kept away from equipment, which requires air flow or ventilation to prevent overheating.
7. Disposal - Students are responsible for the proper disposal of used material if any in appropriate containers.
8. Equipment Failure - If a piece of equipment fails while being used, report it immediately to your lab assistant or tutor. Never try to fix the problem yourself because you could harm yourself and others.
9. If leaving a lab unattended, turn off all ignition sources and lock the doors. Clean up your work area before leaving. Wash hands before leaving the lab and before eating.

Cutting & Grinding

1. Power saws should not be operated without eye protection and an apron.
2. Do not push the specimen or materials against the cutting wheel directly with the fingers.
3. Long materials should be supported or clamped before cutting.
4. Hold the sample tightly.
5. Do not overheat the sample while grinding. Cool the sample in water during intermediate inspections.
**Heat Treating**

1. Use tongs to insert or remove the specimens from the furnace.
2. Use insulating gloves to open or close the doors to the furnaces.
3. Either cool the specimens immediately after removal from the furnace or place in a designated area for slow cooling. Hot specimens should not be left in the open where they may be accidentally touched.
4. The furnaces should be turned off when not in use.
5. If specimens are left in a furnace and the area is abandoned by the student, a sign must be left with a name and phone number and time for removal.
6. Quenching samples in oil can cause the oil to ignite. Be prepared to cover the container after immersion.
7. Use baskets or tongs for quenching in oil or water.

**Chemical safety**

1. Treat every chemical as if it were hazardous.
2. Make sure all chemicals are clearly and currently labeled with the substance name, concentration, date, and name of the individual responsible.
3. Never return chemicals to reagent bottles. (Try for the correct amount and share any excess.)
4. Comply with fire regulations concerning storage quantities, types of approved containers and cabinets, proper labeling, etc. If uncertain about regulations, contact the building coordinator.
5. Use volatile and flammable compounds only in a fume hood. Procedures that produce aerosols should be performed in a hood to prevent inhalation of hazardous material.
6. Never allow a solvent to come in contact with your skin. Always use gloves. Never "smell" a solvent!! Read the label on the solvent bottle to identify its contents.
7. Dispose of waste and broken glassware in proper containers. Clean up spills immediately.
8. Do not store food in laboratories.

I __________________________ Scholar No ______________________ have read and understood the laboratory safety procedures and policies. I am responsible for following these procedures while in the laboratories.

____________________________
(Signature with date)
# PHASE TRANSFORMATIONS & HEAT TREATMENT LABORATORY

**EVEN SEMESTER 2015-16**

**LOGBOOK MARK SHEET**

Name:  
Scholar Number:  
Section:  
Group:  

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<th>S.No</th>
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**Total Marks obtained**

Unauthorized absences will be credited with a mark of 0/10. Your completed laboratory logbook (with this sheet firmly stuck in and fully completed) must be submitted to your instructor at the end of your each experiment. Your instructor will make a assessment and transmit the mark.

A. LAVA KUMAR  
Laboratory Coordinator

SUBHADRA SAHOO  
Laboratory Co-coordinator
EXPERIMENT 1

ANNEALING & NORMALIZING

OBJECTIVE

To study the microstructure, grain size and hardness of Annealed and Normalized plain carbon steel having \( \leq 0.2\%\text{C} \) and \( \leq 0.4\%\text{C} \), also to study the spheroidization annealing of \( \leq 1.2\%\text{C} \) steel.

EQUIPMENT / MATERIALS

Steel samples of \( 0.2\%\text{C}, \ 0.4\%\text{C} \) and \( 1.2\%\text{C} \), Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

The heat treatment includes heating and cooling operations or the sequence of two or more such operations applied to any material in order to modify its metallurgical structure and alter its physical, mechanical and chemical properties. Usually it consists of heating the material to some specific temperature, holding at this temperature for a definite period and cooling to room temperature or below with a definite rate. *Annealing, Normalizing, Hardening* and *Tempering* are the four widely used heat treatment processes that affect the structure and properties, and are assigned to meet the specific requirements from the semi-fabricated and finished components.

Steels being the most widely used materials in major engineering fabrications undergo various heat treatment cycles depending on the requirements.

Annealing refers to a wide group of heat treatment processes and is performed primarily for homogenization, recrystallization or relief of residual stress in typical cold worked or welded components. Depending upon the temperature conditions under which it is performed, annealing eliminates chemical or physical non-homogeneity produced of phase transformations. Few important variants of annealing are full annealing, isothermal annealing, spheroidization annealing, recrystallization annealing, and stress relief annealing.

*Full annealing*

Full annealing process consists of three steps. First step is heating the steel component to above \( A_3 \) (upper critical temperature for ferrite) temperature for hypo-eutectoid steels and above \( A_1 \) (lower critical temperature) temperature for hypereutectoid steels by 30-50 °C. The second step is holding the steel component at this temperature for a definite holding (soaking) period of at least 20 minutes per cm of the thick section to assure equalization of temperature throughout the cross-section of the component and complete austenization. Final step is to cool the hot steel component to room temperature slowly in the furnace, which is also called as *furnace cooling*. The *full annealing* is used to relieve the internal stresses induced due to cold working, welding,
etc, to reduce hardness and increase ductility, to refine the grain structure, to make the material homogenous in respect of chemical composition, to increase uniformity of phase distribution, and to increase machinability.

**Iron-carbon phase equilibrium diagram**

**Spheroidise annealing**

Spheroidise annealing is one of the variant of the annealing process that produces typical microstructure consisting of the globules (spheroid) of cementite or carbides in the matrix of ferrite. The following methods are used for spheroidise annealing.

- **Holding at just below \( A_1 \)**
  Holding the steel component at just below the lower critical temperature \( (A_1) \) transforms the pearlite to globular cementite particles. But this process is very slow and requires more time for obtaining spheroidised structure.

- **Thermal cycling around \( A_1 \)**
  In this method, the thermal cycling in the narrow temperature range around \( A_1 \) transforms cementite lamellae from pearlite to spheroidal. *Figure* depicts a typical heat treatment cycle to
produce spheroidised structure. During heating above $A_1$, cementite or carbides try to dissolve and during cooling they try to re-form. This repeated action spheroidises the carbide particles. Spheroidised structures are softer than the fully annealed structures and have excellent machinability. This heat treatment is utilized to high carbon and air hardened alloy steels to soften them and to increase machinability, and to reduce the decarburization while hardening of thin sections such as safety razor blades and needles.

![A typical heat treatment cycle to produce spheroidised structure](image)

**Normalizing**

Normalizing process consists of three steps. The *first step* involves heating the steel component above the $A_{3\text{cm}}$ temperature for hypoeutectoid steels and above $A$(upper critical temperature for cementite) temperature for hypereutectoid steels by $30^0 \text{C}$ to $50^0 \text{C} \ (Figure \ 4.7.5)$. The *second step* involves holding the steel component long enough at this temperature for homogeneous austenization. The final step involves cooling the hot steel component to room temperature in still air. Due to air cooling, normalized components show slightly different structure and properties than annealed components.

The properties of normalized components are not much different from those of annealed components. However, normalizing takes less time and is more convenient and economical than annealing and hence is a more common heat treatment in industries. Normalizing is used for high-carbon (hypereutectoid) steels to eliminate the cementite network that may develop upon slow cooling in the temperature range from point $A_{\text{cm}}$ to point $A_1$. Normalizing is also used to relieve internal stresses induced by heat treating, welding, casting, forging, forming, or machining. Normalizing also improves the ductility without reducing the hardness and strength.
Schematic representation of annealing operation

PROCEDURE
OBSERVATIONS

Draw Fe-Fe$_3$C Phase Diagram and identify the phases
Draw TTT Diagram for Hypo-Eutectoid, Eutectoid, Hyper-eutectoid
Draw CCT Diagram for Hypo-eutectoid, Eutectoid and Hyper-Eutectoid
Observe the microstructure and write down the phases present

**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*

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**Magnification used** ____________

The phases present in the microstructure and the approximate % of major phases are

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**Etchant used** ________________  
**Hardness** ________________

*From the observation of microstructure & Hardness of the given sample*

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**Etchant used**

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**Hardness**

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*From the observation of microstructure & Hardness of the given sample*

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Answer the following Questions

1. If one of the specimens of the same kind of steel is normalized and the other is annealed, which will show more strength and why?

2. Out of the normalized and annealed samples, which one is more machinable and why?

3. How does the hardness vary with cooling rate?

4. How do your microstructures compare with standard microstructures.
5. What temperature will be used for annealing and normalizing the mild steel specimen with 0.25%C?

6. What is the nature of the microstructure in spheroidized high carbon steel, normalized high carbon steel, annealed high carbon steel?

7. The microstructure of an iron-carbon alloy consists of proeutectoid ferrite and pearlite; the mass fractions of these two microconstituents are 0.174 and 0.826, respectively. Determine the concentration of carbon in this alloy.

8. Give the nature of pearlite and cementite grains in spheroidized steel?
9. α-ferrite and δ-ferrite, both contain BCC structure. Why the solubility limit varies from α-ferrite to δ-ferrite? Explain. BCC is open structure, Although it is stable at higher temperatures why?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 2

HARDENING & TEMPERING

OBJECTIVE

To study the microstructure, grain size and hardness of Hardened and Tempered plain carbon steel having \( \leq 0.2\% \text{C} \), \( \leq 0.4\% \text{C} \), \( \leq 1.2\% \text{C} \) steel.

EQUIPMENT / MATERIALS

Steel samples of 0.2\%C, 0.4\%C and 1.2\%C, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

Different techniques to improve the hardness of the steels are conventional hardening, and tempering.

Conventional hardening

Conventional hardening process consists of four steps. The first step involves heating the steel to above \( A_3 \) temperature for hypoeutectoid steels and above \( A_1 \) temperature for hypereutectoid steels by 50 \( ^\circ \text{C} \). The second step involves holding the steel components for sufficient soaking time for homogeneous austenization. The third step involves cooling of hot steel components at a rate just exceeding the critical cooling rate of the steel to room temperature or below room temperature. The final step involves the tempering of the martensite to achieve the desired hardness. Detailed explanation about tempering is given in the subsequent sections. In this conventional hardening process, the austenite transforms to martensite. This martensite structure improves the hardness.

Following are a few salient features in conventional hardening of steel.

1. Proper quenching medium should be used such that the component gets cooled at a rate just exceeding the critical cooling rate of that steel.
2. Alloy steels have less critical cooling rate and hence some of the alloy steels can be hardened by simple air cooling.
3. High carbon steels have slightly more critical cooling rate and has to be hardened by oil quenching.
4. Medium carbon steels have still higher critical cooling rates and hence water or brine quenching is necessary.

Figure 1 depicts the conventional hardening process which involves quenching and tempering. During quenching outer surface is cooled quicker than the center. Thinner parts are cooled faster than the parts with greater cross-sectional areas. In other words the transformation of the austenite is proceeding at different rates. Hence there is a limit to the overall size of the part in this hardening process.
**Tempering**

The hardened steel is not readily suitable for engineering applications. It possesses following three drawbacks.

- Martensite obtained after hardening is extremely brittle and will result in failure of engineering components by cracking.
- Formation of martensite from austenite by quenching produces high internal stresses in the hardened steel.
- Structures obtained after hardening consists of martensite and retained austenite. Both these phases are metastable and will change to stable phases with time which subsequently results in change in dimensions and properties of the steel in service.

Tempering helps in reduce these problems. Tempering is achieved by heating hardened steel to a temperature below $A_1$, which is in the range of 100°C to 680°C, hold the component at this temperature for a soaking period of 1 to 2 hours (can be increases up to 4 hours for large sections and alloy steels), and subsequently cooling back to room temperature.

The tempering temperature is decided based on the type of steel. Highly alloyed tool steels are tempered in the range of 500°C - 600°C. Low alloy construction steels are tempered above 400°C to get a good combination of strength and ductility. Spring steels are tempered between 300°C and 400°C to get the desired properties. *Figure 2* depicts the influence of tempering temperature on the properties of steel. It is observed that the increase in the tempering temperature decreases the hardness and internal stresses while increases the toughness.
PROCEDURE
OBSERVATIONS

Observe the microstructure and write down the phases present

Sample No:

*Draw the microstructure in the circle given below and write down the phases*

Magnification used

The phases present in the microstructure and the approximate % of major phases are

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Etchant used

Hardness

From the observation of microstructure & Hardness of the given sample

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**Etchant used**

**Hardness**

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*From the observation of microstructure & Hardness of the given sample*

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Answer the Following Questions

1. How do your tempered structures compare with standard microstructures of tempered martensite and spheroidized steel?

2. Give the physical and mechanical changes that occur during the above phase transformation

3. What is the main purpose of tempering steel?

4. Why is tempering preceded by converting the steel into martensite phase?
5. Which steel will be more tougher, tempered high carbon steel or tempered low carbon steel?

6. Which of the two steel (a) tempered low carbon steel (b) annealed low carbon steel will be more tougher and why?

7. How does hardness vary with tempering temperature? How do you explain this variation?
8. Why it is necessary to be really quick in transferring the specimen from the austeniting furnace to the water bath during quenching?

9. How does the hardness of specimen tempered at 700°C compare with that of the furnace cooled specimen? A Steel given such a tempering treatment is preferred to a furnace cooled specimen for good machinability. Can you explain this on the basis of the difference in microstructure?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 3

RECOVERY, RECRYSTALLIZATION & GRAIN GROWTH

OBJECTIVE

To study the effect of recovery, recrystallization and grain growth (annealing) in a plastically deformed metal on its microstructure and mechanical properties.

EQUIPMENT / MATERIALS

Al-alloy, Steel samples of known history, Rolling mill, Precision micrometer, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

A small percentage of the energy expanded in plastically deforming a material remains stored in the metal as an increase in internal energy. This increment in internal energy is associated with lattice defects such as vacancies, interstitials, dislocations, and stacking faults, generating during the deformation. The distribution of dislocations through the bulk of the metal is very inhomogeneous and consists of relatively strain free cells in which the dislocation density is low, separated by boundary regions of high dislocation density in which the dislocations are arranged in tightly packed tangles. There may be many of these cells in each grain. If the deformation occurs at a sufficiently low temperature, many dislocation tangles will be retained in the material. The processes of recovery and recrystallization can occur in such materials during further working or on heating. These are thermally activated mechanisms, usually depending upon atomic diffusion, which repair the structural damage caused by mechanical deformation.

Recovery involves changes in the number and distribution of point defects and dislocations. At low temperatures this is generally caused by the clustering of point defects like vacancies and interstitials, and migration of point defects to dislocations, grain boundaries, and external surfaces. At sufficiently high temperature, the dislocations may eventually grain appreciable mobility and move by both glide and climb to relieve the internal strains. The tangled dislocations in the walls of the cells which formed during deformation, rearrange themselves. The dislocations climb out of their slip planes with the aid of vacancies, and some dislocations of opposite sign annihilate each other. The cell walls become more clearly defined and are called sub-boundaries. Throughout this process the dislocation density in the interior of the cells decreases. During the later stages of recovery, the cells increase slightly in size. Small changes in hardness, which are sometimes observed during recovery, can be due to decrease in dislocation and point defect density and to growth of subgrains.

If increased thermal activation is available (i.e., if the temperature is raised), further decrease in the free energy of the material can be brought about by the formation of new strain free grains.
This process is termed recrystallization. These grains initially form in regions where the local degree of deformation is highest. Such sites include grain boundaries, sub boundaries, deformation bands, twin intersections, and free surfaces. If inclusions or precipitates are present, they may also act as preferential sites for the nucleation of new grains. These new grains grow at the expense of the deformed structure until the whole matrix has been consumed. The process occurs either by migration of original grain boundaries or by subgrain growth. The recrystallized matrix has much lower dislocation density than the deformed material. The driving force for boundary migration is the decrease in free energy resulting from the decrease in the density of imperfections. Above a minimum critical strain the rate of formation of recrystallization nuclei increases sharply with increasing strain.

The lowest temperature at which stress free grains appear in the structure of a previously plastically deformed metal is termed the recrystallization temperature. This depends upon the grain size, the severity of plastic deformation, and the presence of solute atoms or second phase particles.

If a recrystallized material is further annealed at the same temperature, or at a higher temperature, grain growth usually occurs. Boundaries between annealed grains migrate and larger grains grow by consuming smaller ones, which disappear. Grain growth depends upon the fact that the grain boundary energy of the material is reduced due to the decrease in grain boundary area for a given volume of the material. In the absence of complicating factors, e.g., second phase particles, this energy reduction leads to a relationship governing grain growth which can be expressed as

\[ D_t - D_o = K t^n \]

Where \( D_t \) is the average grain diameter after time \( t \)
\( D_o \) is the initial grain diameter
\( K \) is constant

The value of \( n \) is experimentally observed to be \( \leq \frac{1}{2} \)

The presence of precipitates or inclusions drastically reduces the grain growth. The particles have a pinning effect on the grain boundaries.

During grain growth a curved grain boundary usually migrates towards its center of curvature. This is in the opposite direction to that usually observed in recrystallization. When grain boundaries in a single phase meet at angles other than \( 120^\circ \), the grains included by the more acute angle will be consumed, and all angles approach \( 120^\circ \) during the grain growth. When grain shapes in actual specimens are studied metallographically it is often seen that the approach is not very close.

Since dislocation density increases on plastic deformation, dislocation-dislocation interactions occur more readily in deformed materials. The motion of dislocations, therefore, requires larger
applied stress in specimens which have higher dislocation density. Thus cold work increases the hardness of a material. Since recovery and recrystallization involve a reduction in density of imperfections, these processes are generally accompanied by a decrease in hardness of the material. In polycrystalline materials, the grain boundaries also offer resistance to the motion of dislocation. The size of the grains can thus markedly affect the mechanical properties and grain growth softens the material. Hall and Petch have found that

$$\sigma_y = \sigma_i + K d^{-1/2}$$

Where $\sigma_y$ is the lower yield stress

$\sigma_i$ and $K$ are constants, characteristics of the materials and

$d$ is the average grain diameter

The effect of annealing a plastically deformed metal on its grain size, internal stress, and strength is shown in figure below.
PROCEDURE
OBSERVATIONS

Observe the microstructure and write down the phases present

Sample No:

Draw the microstructure in the circle given below and write down the phases

Magnification used ____________

The phases present in the microstructure and the approximate % of major phases are

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Etchant used ________________ Hardness ________________

From the observation of microstructure & Hardness of the given sample

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34
Observe the microstructure and write down the phases present.

Sample No:

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Etchant used _______________  Hardness _______________

From the observation of microstructure & Hardness of the given sample

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Observe the microstructure and write down the phases present

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Etchant used ______________ Hardness ______________

From the observation of microstructure & Hardness of the given sample

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Answer the Following Questions

1. Discuss the effect of annealing temperature on hardness and grain size of specimens.

2. What is the effect of annealing time on the hardness of specimens annealed at 400°C? Explain

3. Does recovery influence the nucleation of new strain free grains?
4. Is the rate of grain boundary migration influenced if recovery and recrystallization processes occur simultaneously?

5. Explain mechanism of recrystallization

6. What factors raise the recrystallization temperature of a metal or alloy? Discuss their effect.
7. When will the grain growth stop in a single phase polycrystalline material? Will the grain size continue to increase until the material is a single crystal?

8. What is the effect of grain growth on hardness and ductility?

9. Most of the materials that we encounter in our daily lives are polycrystalline. If the grain boundary energy does increase the free energy of the system, why do they not disappear leaving behind a single crystal?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 4

PRECIPITATION HARDENING

OBJECTIVE

To study the effect of precipitation hardening treatment on Al-4% Cu alloy on Isothermal ageing.

EQUIPMENT / MATERIALS

Samples of Al-4%Cu alloy, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

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 2014 Al can best be understood by referring to the binary phase diagram of Aluminum-Copper in the aluminum-rich region in Figure 1.

![Figure 1. Binary Phase Diagram for Al-Cu](image-url)
When the aluminum-copper alloy of less than 5 wt% copper is heated to a temperature just above the solvus line, only one phase (alpha, α) 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 2014 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 1), two phases are thermodynamically stable (alpha 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 2014 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 2014 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.

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.
PROCEDURE
OBSERVATIONS

1. Record the date in following table

   Solution treatment temperature:
   Hardness of Quenched specimen:
   Aging temperature:

<table>
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<tr>
<th>Ageing Time</th>
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2. Plot Hardness vs ageing time curve
Answer the Following Questions

1. Discuss the variation in hardness as a function of ageing time.

2. How will the peak hardness vary with ageing temperature?

3. How will the hardness vs ageing time curve shift for an alloy with 5 percent copper?
4. Aluminum alloy rivets can be deformed easily when put in place but age-harden at room temperature. How could one salvage a batch of rivets which had age-hardened before they had been used?

5. What methods other than hardness measurement could be used to study the precipitation phenomenon? Discuss any of them.

6. What are the stages of precipitate formation in precipitation strengthened alloys?
7. What is the essential condition for age hardening?

8. What is the structural difference between GP zones and $\theta$?

9. What’s the difference between a Coherent and a Non-Coherent Precipitate?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 5

HARDENABILITY

OBJECTIVE

To study hardness and microstructure as a function as a function of quench rate, and investigate the hardenability of steels by Jominy test.

EQUIPMENT / MATERIALS

Sample of medium or high carbon steel (the steel must have more than 0.35%C), or alloy steel, Quenching tank, Electric furnace, Special tongs, Rockwell hardness tester, metallographic polishing equipment, Metallurgical microscope.

THEORY

In a practical sense it is not possible to heat-treat all parts to the same degree. The difference is due to the thickness or volume effect. Basically, when a part is quenched in water or some other fluid, the heat must be conducted out through the surface. This leads to a temperature gradient $\frac{dt}{dx}$ between the surface and the center of the part being heat-treated. The temperature gradient varies with time. The temperature gradient is less steep between the center and the edge at later times. Therefore, the temperature of the center lags in time behind the temperature of the surface. If we were to plot a time profile of the center and the edge temperatures as shown in Figure 1, the time to reach a given temperature $T_2$ is definitely longer in the center than at the edge. This means that cooling rate varies as a function of depth. The greater the depth the slower the cooling rate. The situation with respect to the cooling rate can lead to a different hardness in the center than at the edge. The edge could transform to martensite and the center to pearlite or bainite.

![Figure 1: Cooling curves for the surface and center of a quenched specimen](image)

In selecting a steel, the ability to cool the center depends upon the thickness of the part. The thicker the part, the slower the cooling rate at the center. For a given thickness, one must select a
steel that can be hardened in the center if that is desired. The cooling rate in this case is fixed. The center part of steel can be hardened by shifting the time-temperature transformation diagram through alloying. Figure 2 shows that alloying elements added to plain carbon steel can shift the nose of the TTT curve to longer times and raise the $M_s$ temperature. This means a slower cooling rate can be used to reach the martensitic state. A slower cooling rate means a thicker part can be heat-treated.

![TTT diagram for two different steels](image)

**Figure 2 : TTT diagram for two different steels**

To obtain standardized data on the hardness of steels as functions of cooling rates, the Jominy End Quench test was developed. In the test, water is sprayed on one end of a bar of steel while it is hot. This leads to a one dimensional heat transfer cooling. Except near the surface of the bar the temperature is controlled by heat flow along the length of the bar (like thickness in the part). Moving axially away from the quenched end of the bar, the temperature and the rate of change of temperature are changing. The temperature is higher and the cooling rate is lower. If surface hardness is measured as a function of distance from the end, a hardness profile can be obtained which applies to any part made from the same steel, as shown in Figure 3.
PROCEDURE
**OBSERVATIONS**

Sample No:

*Draw the microstructure in the circle given below and write down the phases*

Draw the microstructure in the circle below and write down the phases.

**Magnification used** ____________

**The phases present in the microstructure and the approximate % of major phases are**

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**Etchant used** ____________

*From the observation of microstructure & Hardness of the given sample*

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1. Measure hardness as a function of distance from the rapidly quenched end. Make a hardness reading every $\frac{1}{8}$ in. in the most rapidly quenched one inch, every $\frac{1}{4}$ in.

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<th>Distance (inches)</th>
<th>Hardness Measurements</th>
<th>Hardness ( )</th>
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Answer the Following Questions

1. How do your hardness measurements correlate with your microstructural observations?

2. Give five applications for quench-hardened steels.

3. What will be the difference in hardness profile of a steel part quenched in water and in oil at the same temperature?
4. Steel gears are being quenched by dropping into a tank of room temperature oil. A competitor makes gears of the same size and shape, apparently from the same raw material, but his gears last longer than yours. What can you do to improve the quality of your product and cut down wear? What is the danger in this course of action if you overdo it?

5. What is the ideal critical diameter and can it be determined with a Jominy test?

6. How is the role of carbon and various alloy elements on the hardenability of steels?
7. What effect does surface scale have on hardness measurements?

8. You have three steels. Select the most appropriate steel to achieve the necessary levels of mechanical properties, residual stress and distortion in a 1mm diameter wood-working drill. Why? 1% C, 0.4% Si, 1% Mn, 5% Cr, 1% Mo and 0.4% C, 0.4% Mn, 0.3% Si, and 0.5% C, 4% Cr, 6% Mo

9. As grain size increases, effect boron on hardenability decreases. Why?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 6

EQUILIBRIUM DIAGRAMS BY DIRECT COOLING CURVES

OBJECTIVE

To measure cooling curve of a Pb-Sn alloy using thermocouple and draw the equilibrium diagrams with these cooling curves

EQUIPMENT / MATERIALS

Fireclay crucibles, Chromel-Alumel thermocouple with a protective-insulating sheath, a vertical induction furnace, Potentiometer for reading thermocouple, stirring rod and samples with different compositions.

THEORY
EXPERIMENT
RESULTS

Draw the Pb-Sn phase diagram with corresponding microstructures.
Answer the Following Questions

1. What is cooling curve? Draw the cooling curves for Homogeneous and Heterogeneous nucleation processes.

2. What is the difference between Direct and Inverse cooling rates?

3. What is the expected difference in cooling curves for Pb-10%Sn and Pb-40%Sn alloys? Explain

4. What type of binary system is represented by the Pb-Sn diagram?
5. Would you expect a difference in the arrest points obtained by heating curves and cooling curves?

6. From the Pb-Sn phase diagram, estimate the composition of eutectic alloy and draw the microstructure.

7. List out the different type of thermocouples available in the market?
8. Using the following data, calculate the volume fraction of the beta phase and eutectic at the eutectic temperature, for an alloy of composition 75 wt% Ag. Assume equilibrium conditions. At eutectic temp: eutectic composition = 71.9 wt% Ag, maximum solid solubility of Cu in Ag = 8.8 wt% Cu, density Ag = 10 490 kg/m$^3$, density Cu = 8 920 kg/m$^3$

9. Under what conditions could the compositions of the phases present differ from that predicted in the phase diagram?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 7

DIFFERENTIAL THERMAL ANALYZER (DTA)

OBJECTIVE

To determine the transition temperature and heat of transition of given sample using DTA

EQUIPMENT / MATERIALS

Differential thermal analyzer, potentiometer for temperature measurement, micro-voltmeter for differential EMF measurement, Pb-Sn eutectic alloy.

THEORY
RESULTS
1. What is the transition temperature?

2. Describe briefly how the DTA technique is performed and sketch a typical DTA plot for the melting of a pure metal.

3. What are the advantages and disadvantages of the DTA?
4. Describe briefly how DSC differs from DTA and sketch a typical DSC plot for the melting of a pure metal.

5. What are the advantages and disadvantages of the DSC?

6. What is the effect on the shape of the free-energy curve for a solution if its interaction parameter is positive?
7. In terms of interatomic bonding, what does a negative interaction parameter represent?

8. What are the factors that govern the choice of chromel-alumel or Pt-Pt 10% Rh wires for differential thermocouple?

9. What is meant by reference standard? How one can select reference standard for different materials?

10. What is the significance of this experiment? How is it related to your course of study?
EXPERIMENT 8

CASE STUDY

1. Post weld treatment
2. Surface Hardening of steels
3. Zone refining in electronic materials
4. Development of Bainitic steels
5. Heat treatment of Titanium & alloys
6. Heat treatment of Superalloys
7. Shape memory alloys
8. Development of Peak aging condition in Al-alloys
9. Dual Phase steels
10. Bearing steels
11. Gears
12. Spring steels

Select any topic above given, and study the heat treatment cycles for above materials with required properties and applications.

REPORT FORMAT

OBJECTIVE

COMPOSITION

ROLE OF ALLOYING ELEMENTS

HEAT TREATMENT CYCLES

PROPERTIES

Microstructural characteristics

Mechanical properties

APPLICATIONS

PRESENT STATUS

REFERENCES