Department of Materials Science

San Jose State College

# Experiment No. 1

# The Microscope

Submitted by

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Submitted to

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Materials Science 145

March 19, 1964

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Experiment 2:

**<u>Photographic Principles in Metallography</u>**

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a bit weak on the conclusion
otherwise excellent

Materials Science 145 Laboratory
San Jose State College
March 19, 1964

Met. E. 145
Metallography and Heat Treatment

EXPERIMENT NO. 2

Photographic Principles in Metallography

OBJECT: To become familiar with photographic principles in metallography leading to correct exposure and development of films and production of satisfactory prints for permanent record of metallurgical structures.

GENERAL INSTRUCTIONS:

Students should read and study chapter 4 in Principles of Metallographic Practice by Kehl before starting this experiment. It will be necessary for the student to make up certain photographic solutions to be used in this course. In so doing it is important that he follow all instructions carefully. Every student using the darkroom will be held strictly responsible for seeing that the room is clean and neat at the end of the laboratory period. All trays should be thoroughly rinsed out and dried, and table tops should be washed off with a clean sponge and dried.

Both metallographic plates and films may be available for photomicrographic work. Metallographic plates are in general more satisfactory, but at the same time they are more expensive and fragile. Therefore Ortho-x or Royal Ortho sheet film will ordinarily be used unless other instructions are given.

PROCEDURE:

Part A - Exposure and Development of Film

1.) The student will prepare solutions of photographic developer, shortstop and fixer and pour them into the proper compartments of the solution tank.

2.) Load a film holder (in darkness) with a sheet of film provided by the instructor.

3.) Set up prepared specimen from Exp. 1 on the stage of the Hils Metallograph. Bring a properly illuminated image (using a green filter) into focus at a magnification of 750X on the viewing screen.

4.) Prepare a trial-exposure negative on your sheet film using exposure times suggested by the instructor. This procedure is described in your text, pp. 135-137.

5.) Remove your test strip from the film holder and develop. It is important to control both temperature and time carefully. The following table shows how variations in solution temperature affect development time for various gamma contrasts with Ortho-X sheet film in DK 60a developer.

Development Time (Minutes)

| Temperature °F | Gamma 0.9 | Gamma 1.0 | Gamma 1.1 | Gamma 1.2 |
|---|---|---|---|---|
| 55 | 13 | 17 | 21 | 27 |
| 60 | 10 | 13 | 16 | 21.5 |
| 65 | 7.5 | 9.5 | 12 | 16 |
| 70 | 5.5 | 7 | 9 | 12 |
| 75 | 4.5 | 5.5 | 7 | 9 |

(These figures based on continuous agitation during development).





6.) Using the proper exposure time as calculated from the test strip, each student will expose and develop his negative.

Part B - Contact Printing

1.) Test strips of Velox F-1F-2F-3 and F-4 contact printing paper should be made. Printing times will be selected, by trial and error, which will give a print fully and correctly developed after 60 to 90 seconds in the diluted developing solution for each of these types of paper. All print strips should be identified with pencil on the reverse side, exposed, developed, washed with shortstop, fixed and dried.

2.) Properly exposed strips of the three types of printing paper should be compared for quality, and described in your report.

3.) Final prints will then be made with the most satisfactory type of printing paper. Include negatives and prints in your report with all exposure and processing data, and a description of procedures followed.

QUESTIONS:

1.) What factors control the degree of contrast obtainable in photomicrography? What actual conditions would you suggest to obtain a high contrast?

2.) To what might you attribute "fogging" of a negative and how may it be avoided?

3.) What factors affect the density of a negative?

4.) In photomicrogrphy (a) Is a long bellows draw essential? (b) What is the advantage of a long bellows draw? (c) What limit is there to the length of a bellows draw?

REFERENCES:

Kehl: Principles of Metallographic Laboratory Practice, Ch. 4.

QUESTIONS

give text of questions

1. The degree of contrast obtainable is a function of the film emulsion, the developer, and the printing paper used. An emulsion composed of very even grains, i.e., all grains very nearly the same size, will exhibit relatively high contrast characteristics. This is generally true for medium and slow emulsions (not very sensitive to light) as the grains involved are very small and would therefore tend to be more alike in size than an emulsion composed of large grains. Developers vary in their ability to produce contrast. Some developers produce a negative with a greater range between white and black areas than others, i.e., it tends to develop highly exposed grains, more than a conventional developer would and tends to develop very lightly exposed grains, less than the conventional would. Thus, the white-black range would be much greater. D-19, the developer used in this experiment, is considered a high contrast developer. Characteristics of D-19 and DK-60, a normal developer, are given in the Appendix. Increased development time often results in greater contrast, but this is a practice which must be used with experience and discretion. Contrast can also be varied by use of different grades of paper available. This is discussed in Principles and shown in Results.

2. "Fogging" is due to developer action on unexposed grains. It is often caused by the use of a developer which is too concentrated, developing at a solution temperature which is too



high, overdevelopment, and, rarely, by overly vigorous agitation. It may be avoided by simply avoiding the conditions listed above and properly following the instructions. The explanation given by the text (Kehl, p. 157) may have been adequate when the book was first published (probably around the turn of the 19th century), but modern developers are compounded so that the ratio of developer components is rarely the reason for a fogging effect.

3. The density of a negative is a function of the emulsion, the developing process, and the exposure. Density is a measure of the "optical density" or "light-stopping power" of a medium. Density is logarithm of incident light over transmitted light. The density of a completely transparent medium is 0; of a completely opaque medium, infinity.

The more silver that is formed on the negative, the more opaque it is, and therefore the denser it is. As exposure time is increased, density is usually increased, assuming constant development. If development time is increased, density usually increases. Density is a function of any process which results in the negative being more opaque than it would otherwise be.

QUESTIONS

4. A long bellows draw is essential if the objective-eyepiece lens combination produces insufficient magnification. The magnification is proportional to the projection distance (see Experiment 1) and, therefore, the greater the bellows draw, the greater the resultant magnification.

The primary advantage of a long bellows draw is the resultant magnification it provides. Also, a system with a bellows can be used relatively easily as a camera system by merely installing a shutter.

The limit to the bellows draw is that beyond a certain point, empty magnification would result. Useful magnification is proportional to the resolving power of the lens and a function of the numerical aperture (see Experiment 1, Principles). Also, if the bellows draw becomes excessive, the light source may not be intense enough to provide film exposures of a reasonable length of time.

# Experiment 3:

# Structure of Commercially Pure Metals and Solid Solution Alloys

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March 19, 1964

## QUESTIONS

1. Discuss any evidence of impurity constituents seen in the microstructure. Make an effort to identify such impurities when observed.

This question was adequately discussed in the Results section of the paper.

2. What would you say were essential differences between wrought and case single phase metals?

Wrought structures are hot or cold worked and, therefore, will have slightly distorted grains. This will give rise to an increase in hardness and decrease in ductility over the cast structures. Any slag remaining in the metal will, on working, be drawn out into fibers and can be easily recognized.

3. What means might one use to make a cast pure metal stronger?

Assuming the metal was absolutely pure, i.e., there were no impurities to change properties, the only way to increase its strength is to cold work it or decrease the grain size by rapid cooling of the melt.

4. What is the difference, if any, between coring and the formation of dendrites?

Coring is the result of non-equilibrium cooling and occurs across the dendrite or grain. Coring is explained in full in the Principles section of this report. Dendrites will form even if equilibrium cooling occurs as they are the result of preferential growth in a given direction.

## CONCLUSION

It was found that although the laboratory experience was challenging and useful, at least as muched was learned from researching and doing the write up of the experiment.

Although the exact history of the specimens was not known, the method of casting, rate of cooling, degree of cold working, etc., one would tend to get the impression that these factors would have an effect on the microstructure. It would have been instructive, perhaps, if samples of exactly the same composition but varying in treatment had been compared.

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San Jose State College

Department of Materials Science

Experiment 4

Intermetallic Compound, Eutectic and

Peritectic Formation

| Submitted by | Submitted to |
| --- | --- |
| Dennis Olsen - Date | Prof. Larsen-Badse |
| Joe Andrade | Materials Science 145 |
| Neil Beckley | March 19, 1964 |
| Charles Hovey | |

# INTRODUCTION

The objective of this experiment is to seek out the intermetallic compounds and the formations of peritectic and eutectic reactions through microstructure analysis. The analysis of the microstructure will be based on theoretical principles, and any deviation from these principles will be discussed in the summary.

# PRINCIPLES

## Introduction

A discussion of the theoretical principles will follow. A brief look at the basic reactions will be made. Then each phase diagram pertinent to this experiment will be discussed in detail, especially in the areas where the samples are located. In this more detailed look at each alloy, results will be predicted for each sample.

## Definition of Terms

Terms used in discussion of phase diagrams will be defined here to avoid interruption of the text that follows.

1. The liquidus is the boundary between the liquid phase and a two phase region containing a solid phase and the diluted liquid phase.
2. The solidus is the boundary between the phase region containing a liquid phase and a solid phase and the phase region of that solid phase.

# EXERCISES

Q.1. Describe all changes which would occur in a 40% Sb-60% Sn alloy cooling under equilibrium conditions from the liquid state to room temperature.

A.1. This is the cooling path CC' of Fig. 6. Cooling from the melt, we will encounter a given rate of cooling until the liquidus is crossed at 400 C., whereupon the cooling rate changes as the solid phase begins to freeze. This rate remains constant until the peritectic isotherm of 325 C. is reached. Now the peritectic phase is formed from the reaction between the melt and the phase and for equilibrium, the phase will be completely reacted. While this reaction is taking place, the temperature remains constant. When all of the has been reacted and the reaction ceases, a new cooling rate sets in for the liquid phase and the newly formed phase. As it continues to cool, continues to increase by freezing out of the liquid phase. When the peritectic isotherm 246 C is reached, the remaining liquid phase and a portion of the phase react to form the peritectic Sn. Again, this is an isothermal reaction. Below the peritectic isotherm, after the reaction's completion, the peritectic phase exists at equilibrium in solid solution.

Q. 2. Identify all phases present in the prepared specimens.

A. 2.

| | |
|---|---|
| Sb-5%Sn chill cast | Matrix--Sb |
| | Grains-- phase |
| Sb-5%Sn slow-cooled | Matrix--Sb |
| | Grains--corphase |
| | Core-- phase |
| Sb-50%Sn slow-cooled | Matrix-- phase |
| | Grains-- phase |
| | Core--Sb |
| Sb-50%Sn chill cast | Matrix-- phase |
| | Grains-- phase |
| | Core--unknown |
| Sb-60%Sn chill cast | Matrix-- phase |
| | Grains-- phase |
| | Core--unknown |
| Sb-80%Sn slow-cooled | Matrix-- phase |
| | Grains--Sn peritectic |
| | Core-- phase |
| Sb-80%Sn chill cast | Matrix-- phase |
| | Grains--Sn peritectic |
| | Core-- phase |
| * Sb-35%Zn sand cast | Matrix--Sb plus ZnSb |
| | Grains--Sb |
| *Sb-20%Zn sand cast | Matrix--SnSB |
| | Grains--SnSb plus Sb |
| Sb-80%Zn chill cast | Matrix--Zn plus |
| | Grains-- phase |
| | Core--$Zn_3Sb_2$ |
| Sb-80%Zn slow-cooled | Matrix--Zn plus |
| | Grains-- phase |
| | Core--$Zn_3Sb_2$ |

* Composition in accordance with photograph in specimen labeling but contrary to the compositions arrived at in the conclusions.

Q.3. Were there any differences between the 50-50 Sb-Sn alloys slow-cooled and chill cast. If so, how would you account for such differences?

A.1. There were differences--for one thing in the grain size. The grain size is definitely dependent on the rate of cooling. Also noted is that the coring agent was larger in comparison to the grain size for chill cooled. The increased rate of cooling did not give the Sb phase time to react with the melt to form the phase as was the case of the slow-cooled.

Q.4. Estimate the relative amounts of the respective phases in specimen no. 4. How does this compare with theoretical proportions? Account for any differences.

A.4. As stated before in the conclusions, I believe specimen no. 4 to be actually specimen no. 5. Therefore, to approximate the relative amounts of Sb-20%Zn, I will make the calculations of relative amounts from specimen no. 5. The approximated relative amount of the eutectic phase is 85%, and the remaining 15% is given up to the solid phase Sb. Theoretically, however, the specimen should with equilibrium cooling result in a 100% eutectic phase. To account for this 15% Sb phase, we can postulate two methods through which this could come about. The first would simply be an error in the original composition: placing the specimen's composition to the left of the eutectic's composition, approximately Sb-17-18%Sn. The second and more likely occurrence is that during the cooling process, which is non-equilibrium, the Sb began freezing out as though it were a composition as described above. Another possible discrepancy could possibly arise out of the magnification of the photomicrograph. The high magnification may have over-emphasized the amounts of Sb grains present in the eutectic matrix. If so, the discrepancy must be blamed on inexperience and lack of forethought by the calculator.

Q.5. Discuss characteristics of intermetallic compounds in general.

A.5. In general, the intermediate phase which freezes directly from the melt is called intermetallic compound. In most cases, the width of the phase is extremely narrow, and in the more limiting cases, it can only be described by one line of composition. Crystals formed by these compounds sometimes have extremely complex crystalline structures and exhibit hardness and brittleness. As described earlier in this report, these intermetallic compounds will cause discontinuities in the physical behavior of the rest of the alloy system, i.e., electrical conductivities, magnetic susceptibilities, and thermo-electric powers.[4]

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[4] Mantell, pp. 221-222.

Experiment 5:

Cold Working and Annealing of Brass

Joe Andrade, Dataman
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Charles Hovey
Dennis Olsen

Try to wring all information and tabulate conclusions out of your data even if you made a few mistakes in lab. "Principles" [illegible] cut down without suffering.

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Materials Science 145 Laboratory
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May 22, 1964

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Appendix

## INTRODUCTION

The primary object of this experiment was to test the students' ingenuity and initiative by requiring them to develop a "research-type experiment" from an article in the literature. The article selected by the instructor is "Recrystallization and Grain Growth in Alpha Brass," by S.L. Channon and H.L. Walker (<u>ASM Trans</u>, V. 45 1953, pp. 200-220).

After many careful readings of the paper, it was decided to check Conclusions 1 and 2, two conclusions in a series listed by the authors at the end of their paper. This was to be accomplished by selecting several values of per cent deformation, annealing temperature, and annealing times such that the necessary grain size data could be obtained. Channon and Walker's Table IV (p. 205) was the basis for this selection. Deformations of 0, 30, and 75 per cent were selected as hopefully reasonable representative samples. Annealing temperatures of 250, 300, 350, and 400 C. were selected at times of 15, 30, 60, 90, and 120 minutes. The reasons for these selections and the procedure involved will be discussed later.

The "research-type experiment" (which is a contradiction in terms with reference to Channon's article) was performed as outlined in the hypothetical "green sheet" (which is usually white) and the results are reported in this paper.

Mat. Sci 145 <u>Experiment "Green" Sheet</u>

Experiment 5:

<u>Cold Working and Annealing of Brass</u>

<u>Object</u>:

To study the effects of various degrees of cold-working on the microstructure and hardness properties of cartridge brass; to study the effects of annealing temperature and time on the microstructure and hardness property of cold-worked brass.

<u>Material</u>:

Four three-inch bars of cartridge brass (70% copper--30% zinc), $\frac{1}{2}$ x $\frac{1}{4}$ inch.

<u>Procedure</u>:

Anneal the samples at a temperature of 500 C. for one hour. Cold roll two of the bars to about 30% deformation; cold roll one of the bars to about 75% deformation. Cut all four samples into 1-1/2 inch lengths and carefully label all specimens. Anneal the 30% deformation specimens at temperatures of 300, 350, and 400 C. for 15, 30, 60, 90, and 120 minutes (15 samples). Anneal the 75% deformation specimens at temperatures of 250, 300, and 350 C. for the same times (15 samples). Be sure to keep at least one unannealed specimen of each deformation. Take hardness readings of all specimens.

Polish and etch and examine all microstructures. Prepare metallographic samples of several specimens both on the surface and in their interior and compare the microstructures.

<u>Questions</u>:

1. What is the recrystallization temperature of the brass at the various deformations (30 and 75)? How does the experimental values compare with those in the literature?

2. Is there any apparent relationship between the recrystallized grain size and the annealing temperature?

3. What is "critical deformation"? Why wasn't it observed in this experiment?

<u>References</u>:

Mehl, "Recrystallization," <u>Metals Handbook</u>, 1948, pp. 259-63.
Channon and Walker, "Recrystallization in Alpha Brass," <u>ASM Transactions</u>, Vol. 45, pp. 200-220.

Met. 214.5
Metallography and Heat Treatment

EXPERIMENT NO. 5
COLD WORKING & ANNEALING OF BRASS

Introduction:

The majority of the experiments in this course have as their primary objectives the understanding of certain fundamental metallurgical principles, and the development of basic competencies in metallographic techniques. Because of limitations of time, and the fact that most students are having their first introduction to these techniques it has been necessary to devise experiments in which the students closely follow a set of directions prepared by the instructor. It is felt, however, that it would be useful as a test in ingenuity and initiative to have the groups develop one research-type experiment from information available in the literature. This would ordinarily take more time than is available in the semester, so one specific published paper will be used as the basis for this experiment.

Object:

The object of this exercise will be to devise an experiment after a careful reading of the following paper:

"Recrystallization and Grain Growth of Brass"
S.L. Channon and H.L. Walker Trans. ASM V. 45 1953, pp. 200-220

The experiment should fulfill the following requirements:

1. It should be possible to complete it in four or five laboratory periods.
2. It should only utilize materials and supplies available in the lab.
3. The data taken should permit a check of one, or preferably two, of the conclusions appearing on page 217.

Your report to the instructor will be similar to the other reports, except that it will include an experiment green sheet of your own devising.

Intro
Object
Discussion
Procedure
Exercise
Questions
References

Department of Materials Science

San Jose State College

Experiment No. 6

Principles of Age Hardening

| | |
|---|---|
| Submitted by | Submitted to |
| Charles Hovey - Data | Professor Larsen-Badse |
| Neil Beckley | Materials Science 145 |
| Dennis Olsen | April 23, 1964 |
| Joe Andrade | |

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# INTRODUCTION

The primary objective of this experiment is to study the effects of time and temperature on age hardening and to correlate these changes with changes in microstructure. However, an additional exercise in this experiment is to identify the microconstitutents in a cast specimen; this has proved to be most intriguing and tedious, but the results here are most intesesting and it has been decided, on the part of this writer, to treat this as a second section of the report. This report, then, will be divided into two sections with a small change in format of the results to facilitate this presentation.

Section I will deal with the phenomenon of age hardening in aluminum, the principles of which are now well understood (to use a phrase quite loosely) and applied to other metals. This section will also include a section on coringg in alloys since it is very common, significant,,and applicable to either section I or II of this report as it produces observable micro-constitutents that play an important role in determining the properties of an alloy.

Section II will, as mentioned, present the results of the exercise to identify the microconstitutents. Because it would be impossible to understand the schema by which these results have been obtained without some knowledge of the principles involved, a section on principles has been included.



Experiment No. 6

Principles of Age Hardening

**Object:** To study the effects of time and temperature upon the hardening characteristics of an age-hardening aluminum alloy, 2024 - S, and to correlate these effects with microstructural changes.

**Material:**
All specimens cut from a bar of annealed 2024S aluminum alloy. One as-cast specimen will be prepared by the group conducting the experiment. The remaining specimens for solution heat-treatment will have been thoroughly soaked at 915°F. prior to the beginning of the laboratory period. They will be water-quenched and aged according to the schedule shown below. It is suggested that the procedure and timing for subsequent heat-treatment be carefully planned before proceeding with the experiment.

**Procedure:**

1. Specimens will be water-quenched and aged as follows:

| Number of Specimens | Aging Temperature | Times (Minutes) |
|---|---|---|
| 2 | Room Temperature | 5, 10, 20, 30, 60, 90, 150 |
| 7 | 212°F | 5, 10, 20, 30, 60, 90, 150 |
| 8 | 400°F | 5, 10, 20, 30, 60, 90, 150 (2) |
| 8 | 575°F | 5, 10, 20, 30, 60, 90, 150 (2) |

2. The aging treatments at the respective temperatures should be scheduled in the various furnaces to be done simultaneously. Boiling water may be used for the treatment of 212°F. It should be arranged in advance to complete all laboratory work, with the exception of microstructural examination, in one laboratory period.

3. Take Rockwell F hardness readings, immediately after quenching, on one of the two specimens to be aged at room temperature. This may
9 be used as representative of a sample of the alloy in the "as-quenched" (solution heat-treated) condition. This specimen is then further prepared as indicated in Procedure 6. Rockwell F hardness readings will be taken on all the other specimens at the times indicated. For the specimens aged above room temperature, remove one specimen at each time interval, and cool in water to room temperature before taking a reading.

5. Prepare the cast specimen by placing a piece of bar stock of suitable size in a small refractory crucible. Melt the charge in a furnace set at 1250°F. and allow the melt to solidify slowly in the crucible. From this ingot prepare a suitable metallographic specimen to identify the microsconstituents present.

6. Immediately after water-quenching, prepare one of the waterquenched specimens for microexamination and reproduce the microstructure. Also reproduce microstructures of the specimens heated $2\frac{1}{2}$ hours at 400°F and 575°F. Attempt to identify microconstituents.

Notes: Schemes for identification of the various microstructural constituents in aluminum alloys are found in several of the references listed below. The students should make up the necessary etchants and store them in properly labelled bottles.
Insofar as possible, it would be desirable to avoid using the etchants containing hydrofluoric acid. If it is necessary to use one of these etchants, DO NOT LET EVEN A TRACE OF HF NEAR MICROSCOPE OPTICS. The specimens should be washed and dried with extreme thoroughness after etching. Cracked mounts or specimens must not be used.

Exercises:

1. Plot a curve for each aging temperature (all on one graph), showing the hardness as a function of aging time.
2. Account in detail for the different types of curves obtained by aging at the four different temperatures.
3. Which temperature should you recommend as the proper aging temperature of this alloy? Why?
4. What is overaging?
5. What microconstituents were identifiable in the cast specimen? In the wrought heat-treated specimens?

References:


1. Kehl, Principles of Metallographic Laboratory Practice, pp. 424-26.
2. Mondolfo, Metallography of Aluminum Alloys
3. -------, Metals Handbook, ASM
4. Keller and Bossert, Revealing the Microstructure of 24S Alloy, Alcoa Research Lab. Technical Paper, No. 8, 1942.
5. Keller and Wilcox, Identification of Constituents of Aluminum Alloys, Alcoa Tech. Paper, No. 7, 1942.
6. Keller and Brown, The Heat Treatment of 24S and Alcl-d 24S Alloy Products, Alcoa Tech. Paper No. 9, 1943.
7. Barker, L.J., "Revealing the Grain Structure of Common Aluminum Alloy Metallographic Specimens", Trans. A.S.M., 1950, pp. 347.


Experiment 7:

# Effect of Gradient Quench on Microstructure of Steels


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Neil Beckley
Charles Hovey


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San Jose State College
April 23, 1964

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## INTRODUCTION

> Iron accommodates itself to all our wants, our desires, and even our caprices; it is equally serviceable to the arts, sciences, to agriculture, and war; the same ore furnishes the sword, the ploughshare...the spring of a watch or a carriage, the chisel, the chain, the compass, the cannon, and the bomb. It is a medicine of much virtue, and the only metal friendly to the human frame. (Ref. 1)

Iron may not deserve all of the laurels it received above, but the iron-carbon alloy system is certainly deserving of most of them. Steel is the workhorse of present day civilization. Future archeologists might well call our times "The Age of Steel."

This experiment deals with a very small portion of the iron-carbon spectrum, but a portion which is very important in science and industry today. The "official" object of the experiment is to "...study the various direct-transformation structures of a medium-carbon steel produced by different rates of cooling from the austenitic state."

In order to perform this optimistic study, the essentials of the iron-carbon phase diagram, the time-temperature-transformation diagram, the hardenability concept, and the Jominy test must all be studied.

The "Principles" section of the report will include short discussions of all topics necessary to the proper completion of the experiment. The general discussions will be limited to those topics specifically included in the experiment. Alloy steels will not be discussed and neither will high-carbon steels. It is assumed that the discussions in all of the previous experiments are available and reference may be made to them.

> Gold is for the mistress--silver for the maid--
> Copper for the craftsman, cunning at his trade,
> "Good!" said the Baron, sitting in his hall,
> "But Iron--Cold Iron--is Master of them all."
> (Ref. 2)

41 A

Met. E. 145 Experiment No. 7

Effect of Gradient Quench on Microstructure of Steels → grain growth

Object:

To study the various direct-transformation structures of a medium-carbon steel produced by different rates of cooling from the austenitic state.

Material: Take 6 specimens : Run 3 for hardness at 1/16 interval

One Jominy specimen of A.I.S.I. 1040 steel, end-quenched from 1550°F. 45 min.

Procedure:

Carefully polish a longitudinal flat surface on the specimen using the best recommended practices for steel. A length of about two inches from the quenched end should be covered. It may be necessary to split the section into two or three segments for convenient handling, but great care should be taken not to overheat the specimen in the cutting-off operation.

Thoroughly examine the polished and etched surface and reproduce significant microstructures. Where microstructures agree on the 3 samples, reproduce only one. If they disagree – reproduce all at that point.

Exercises:

1. Give an interpretation of the microstructure of the bar in the light of the TTT curve for the steel. Kehl p 307, 309 – for cooling rates (draw cooling lines on TTT curve.)
2. Estimate the A.S.T.M. grain size from the observed microstructures. What specific structural changes have made the grain size estimate possible? 5-6 [illegible] grain size
3. What is a "split transformation"? Was there any evidence of a split transformation in the structures examined? Yes, Fig 5
4. How would you suppose that increasing the carbon content of this bar to, say, 0.80% carbon, would change the observed microstructures, assuming an identical heat treatment? 2 [illegible] phase diagram
5. What is the significance of austenitic grain size, and why is it important?
6. What is the relation of the A.S.T.M. grain size observed in Question 2 to austenitic grain size? [illegible]

So ASTM no. should be lower p. 171, Smith

References:


Kehl, Principles of Metallographic Laboratory Practice, pp. 266, 303-310
Smith, Alloy Series in Physical Metallurgy
Bullens-Battelle, Steel and Its Heat Treatment, vol. 1


esp pp: 272-345

study pp 11-32

pp 161-255 (Chap 7)

4. (Cont):

If the carbon content were 0.8%, the bar would be composed of eutectoid carbon steel. In this case there would be no pro-eutectoid ferrite structure. The microstructure would be pearlite or martensite or a mixture of the two. Bainite would not form due to the "overhanging" nature of the CT diagram. Please see Figs. 9 and 11.

5. What is the significance of the austenitic grain size, and why is it important?

The austenite grain size refers to the size of the original austenite grains before transformation. It is closely related to the size of the pearlite and ferrite grains which form from it, as discussed in Question 2. It is important because it has a very large effect on the properties of the steel. The coarser the austenite grains, the less grain boundary area for nucleation of precipitation to occur, therefore there is a longer time period before transformation begins -- which makes for a high hardenability steel. Also, the coarser the grains, the less the toughness strength because the grains tend to prevent slip movement, thereby creating a more brittle structure.

6. What is the relation of the A.S.T.M. grain size observed in Question 2 to austenitic grain size?

This question was answered with Question 2.

QUESTIONS

1. Give an interpretation of the microstructure of the bar in the light of the TTT curve for the steel.

Please see Conclusions.

2. Estimate the A.S.T.M. grain size from the observed microstructures. What specific structural changes have made the grain size estimate possible?

The A.S.T.M. grain size of the last two photographs in the Results section was about 24 grains per inch, or a grain size of 5 to 6.

In the cooling of hypoeutectoid steels, ferrite first appears at the austenite grain boundaries. The ferrite grains then grow at the expense of the austenite. Finally, when the eutectoid temperature is reached, All of the austenite goes to pearlite. The pearlite grains are then substantially smaller than the austenite grains. In the structure of Figs. 24, 25 the ferrite-pearlite ratio appears to be about 50-50. Thus the original austenite grains were probably twice as large as the pearlite grains. This would mean an austenitic grain size of from 3 to 4.

3. What is a "split transformation"? Please see page 21. Was there any evidence of a split transformation in the structures examined? Yes, please see Fig. 22.

4. How would you suppose that increasing the carbon content of this bar to, say, 0.8% carbon, would change the observed microstructures, assuming an identical heat treatment?

EXPERIMENT NO. 8

# PARTIAL ISOTHERMAL TRANSFORMATION OF AUSTENITE

Materials Science 145

GROUP I

Neil Beckley (data man)
Joe Andrade
Dennis Olsen
Charles Hovey

## INTRODUCTION

The purpose of this experiment is to observe the microstructure and correlate it with some of the physical properties, as a steel transforms isothermally from austenite to pearlite or bainite.

The Principles Section will also cover some of the fixed variebles, such as alloying elements and different heat-treating methods. The results will then be compared with transformation diagrams obtained from the literature.

EXPERIMENT No. 9

# METALLOGRAPHY AND HEAT TREATMENT OF TITANIUM ALLOYS

**Object:** To become familiar with effects of heat treatment on the structure and properties of a commercial titanium alloy.

**General Discussion:**

The metallography of titanium and its alloys has a major difference from aluminum and magnesium in that pure titanium can exist in two allotropic forms: (a) body-centered cubic beta above 1625 F. and (b) hexagonal-close-packed alpha below this temperature. Titanium alloys may contain alpha stabilizers such as aluminum, carbon, oxygen and nitrogen; and/or beta stabilizers such as chromium, manganese, iron, molybdenum or vanadium. There may be a number of different phases present depending upon the alloy composition and heat treatment. Thus a low alloy or commercially pure titanium slowly cooled from the beta region will be primarily alpha at room temperature. If an alloy containing a considerable amount of beta-stabilizinh element is slowly cooled from the beta region, much of the beta may persist on cooling and be present as equiaxed grains along with alpha at room temperature; or if rapidly cooled to a non-equilibrium state certain other decomposition products may appear.

A knowledge of composition and prior heat treatment is important in identifying the numerous phases in titanium alloys which may include, in addition to alpha, beta and beta decomposition products, such phases as the carbides and hydrides of titanium.

Considerable care is required in the metallographic preparation of titanium and its alloys. Mechanical work introduced in grinding and cutting may give rise to slip lines and twins in the structure. If care is taken in grinding, conventional polishing techniques using alumina are acceptable, but one or more repolishing and re-etching cycles are desirable. Micro-etching is done with concentrations of HF or $HNO_3$-HF up to about 5% in either water, alcohol, glycerine or combinations of these. (See reference No. 3 concerning precautions against use of titanium in contact with fuming $HNO_3$.) Techniques vary but HF alone tends to darken alpha and leave beta clear. The technology of titanium is so new that little specific information concerning it will be found in textbooks. However, many articles appear in current periodicals, some of the more important of these being listed at the end of these pages. The student is asked to familiarize himself with these articles before beginning this experiment.

**Material:**

The specimen material consists of 3/8" diameter round titanium alloy bar stock having the following composition and mechanical properties:

| Grade | Alloy Content (%) | Ultimate Tensile Str. (psi) | Elong. % | R.A. % |
|---|---|---|---|---|
| C130AM (annealed) | C 0.07, N 0.02, MN 3.9, AL 4.2, H 0.0053 | 156,500 | 16.9 | 40.3 |



**Procedure:**

1. Examine and describe (or reproduce) microstructure of as-received material, and check Rockwell "C" hardness.
   NOTE: In describing multiphase structures estimate the relative amount of each phase present. Microhardness surveys may sometimes be of help in distinguishing phases.
2. Soak five small specimens for one hour at 1450 F. Air cool one of these specimens from the soaking temperature and water quench the remaining four.
3. Age three of the water-quenched specimens at 900 F for 1, 3 and 10 hours respectively.
4. Check Rockwell "C" hardness for all of the above specimens and describe the microstructure.

**Exercises:**

1. Discuss the formation of the observed microstructures in terms of the phase diagram for this particular type of alloy, and your knowledge of possible non-equilibrium transformations. Were you able to observe alpha prime in any of your structures? Explain.
2. Account for any differences in structure observed between center and outside of specimens.
3. Sketch a curve showing the effect of aging time on hardness, from 0 to 5 hours aging time. Discuss hardness variations in terms of phase changes.
4. What differences in the as-quenched structure would you have expected if the soaking time had been 1830 F? Why?
5. If you were attempting to make a titanium alloy having good elevated temperature (about 1000 F) mechanical properties, what type of chemical composition and structure do you think would be most desirable?

**References:**

1. S. Abkowitz and D. Evers, "Two Promising New Titanium Alloys". Metal Progress, Sept. 1957, p. 97.
2. Everhart, J., "Titanium", Materials in Design Engineering Manual No. 142, Oct. 1957, p.149
3. Gilbert, L.L. and Funk, C.W., "Explosion of Titanium and Fuming Nitric Acid," Metal Progress, Nov. 1956, p.93.
4. Jaffe, I.D., "Heat Treatment of Titanium Alloys", Metal Progress, March 1955, p. 104.

5. Jaffe, L.D. "Choosing a Titanium Alloy", Metal Progress, Mar.'55, p.104

6. Kaufmann, D.W.; Finlay, W.L. and Gentz, W.W. :"How to Heat Treat Titanium", Materials and Methods, June 1956 , p. 127

7. Reynolds, J.E., Ogden, H.R. and Jaffee, R.I.. "A Study of the Air Contamination of three Titanium Alloys", Trans. A.S.M. Vol. XLVIII, 1956, p. 280.

8. Rostoker and Domagla, "Metallography of Tempering of Alpha-Prime in Titanium Alloys", Trans. A.S.M., Vol. XLVIII, 1956, p. 762.

9. Osadchnk, R.; Koster and Kahtes, "Metallographic Structures in Commercial Titanium", Metal Progress, Nov. 1953, p. 93

San Jose State College

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Experiment 10

Pyrometric Principles

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Submitted to:
Prof. Larsen-Badse
Materials Science 145
March 26, 1964

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# INTRODUCTION

The laws and principles of pyrometry will be stated and discussed. Then a brief coverage of the present methods of standardization and their classifications will be made, and the method used in the experiment will be compared. A distinction between "accuracy" and "precision" will be made to clarify the terminology. The step by step procedure of the thermocouple calibration will be explained. Next a comparison of the temperature of an object will be made between readings of the existing installation and the optical pyrometer. The results and conclusions of the experiment will be correlated with the principles.

QUESTIONS

Q. 2. If you wished to check the accuracy of the temperature indicating pointer on a recording pyrometer controller, and you had a potentiometer and two copper lead wires available to you, what do you think might be one way to do it?

A. 2. Since the current in the circuit follows Ohm's law, the following equation expresses the relationship of the emf of the thermocouple to the potential difference across the terminals of the instrument.

$$V = \frac{Rm}{Rm \quad Rx} E$$

where:

$V =$ potential difference

$E =$ thermocouple emf

$Rm =$ Resistance of millivoltmeter

$Rx =$ Resistance of leads and thermocopple

From the equation, the calibrated Rx can be determined, and this can be compared to a Rx value measured using the potentiometer.

Q. 3. What is the effect of a small increase in lead wire resistance, on the true emf of a couple as indicated by a potentiometer? Explain.

A. 3. There would be no effect since the potentiometer is measuring the emf of the thermocouple without any current flow, i.e., the emf is in an open circuit; therefore, a change in the resistance is not seen by the potentiometer.

Q. 4. How may compensation for a non-zero cold junction be made in standardizing?

A. 4. The compensating reference junction is found on most industrial potentiometers. It is a precision variable resister scaled in millivolts. The observer sets the dial to read a prescribed value according to the reference junction and thermocouple being used.

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Metallography and Heat Treatment

Experiment No. 10

PYROMETRIC PRINCIPLES

**Object:** To become familiar with a method for thermocouple standardization and to check the accuracy of an existing pyrometric installation.

**General Discussion:**

Thermoelectric couples may be standardized by several methods. Two of the most common of these are (a) basal standardization against the freezing points of pure metals, and (b) comparison with standard couples which have been standardized by the U.S. Bureau of Standards (or with secondary standards which have been compared with primary standards).

a) Freezing point method - Melting (or freezing) points of certain metals, some of which are listed in the following table, are commonly used for standardization of couples by the crucible method.

| Metal | M.P. °C. | Metal | M.P. °C. |
|---|---|---|---|
| tin | 231.9 – 450°F | aluminum | 660.0 |
| cadmium | 320.9 | silver | 960.5 |
| lead | 327.3 – 621°F | gold | 1063.0 |
| zinc | 419.4 – 786°F | copper | 1083.0 |
| antimony | 630.0 1166F | nickel | 1452.0 |

790°C

In all cases the metals or salts used must be of high purity. Standard metal samples may be obtained from the U.S. Bureau of Standards. For ordinary work the usual C.P. reagent metals are sufficiently pure. The method of calibrating consists of 1) measuring the e.m.f.'s obtained with the cold junction at 0°C. and the hot junction at several known fixed temperatures in the desired range, 2) evaluating with the aid of these data the constants of an empirical formula selected to represent them and, 3) computing a table or graphing a figure to show directly temperature differences corresponding to an observed thermal e.m.f. A formula sometimes used in such calibrations is of the type:

$$E = a\Delta T + b(T)^2 + c(T)^3$$

of which E is the thermal e.m.f. and T, the difference between the hot and cold junctions. This equation is usually only applicable to a restricted range. Thermocouple calibrations are usually plotted as difference curves to show the difference in temperature to be added to or subtracted from the temperature indicated by the couple, using standard thermocouple calibration tables.

**Procedure:**

(a) Sufficient of the metal chosen is placed in the crucible to fill it about 3/4 full, and a layer of powdered or flaked graphite is placed on top. (why?) The crucible is placed in the furnace and the metal heated. When one of the metals has become molten insert a refractory thermocouple protecting tube and then insert the couple. (Be careful not to heat far above the melting point.) The couple is attached to the indicating instrument. For most accurate work an ice bath reference junction is used.

Turn the furnace power off and take emf readings at intervals of 20 seconds. At the freezing point of the metal the millivolt reading will be constant for a considerable period of time, and this reading will give a point on the calibration curve.

The thermocouple protecting tube may be left in the metal for a few degrees below the freezing point without breaking. If it cannot then be removed, the metal should be remelted immediately.

Most pure metals do not undercool more than 0.1°C. or 0.2°C. although antimony tends to undercool much more than this. If antimony is used as one of the experimental metals, stirring may be required to reduce undercooling. Other molten metals should not be stirred because pockets of graphite may be formed.

(b) A general check of an existing installation will be made by a comparison method. A standard indicator and standard or calibrated couple are used to check the actual furnace temperature and observations are made of the temperature indicated by the installed pyrometer.

**Exercises:**

1. Submit with your report the three cooling curves and a calibration for the thermocouple. (Affix a tag to the couple giving the date and result of the calibration). Give the results of your check on the existing installation. *Plot the actual millivolts vs. the true melting point of the metal in degrees.

Illustrate set-ups and show wiring diagrams.

**Answer The Questions:**

2. If you wished to check the accuracy of the temperature indicating pointer on a recording pyrometer controller, and you had a potentiometer and two copper lead wires available to you, what do you think might be one way to do it?

3. What is the effect of a small increase in lead wire resistance, on the true emf of a couple as indicated by a potentiometer? Explain.

4. How may compensation for a non-zero cold junction be made in standardizing?

**References:**


1. Kehl: Principles of Metallographic Laboratory Practice, Ch. 8.
2. ASM: Metals Handbook, p. 174.
3. Amer. Inst. of Physics: Temperature, Its Measurement and Control, pp. 265-278, pp. 284-313 (Library)

## The Optical Pyrometer

Objective: To study the characteristics of an optical pyrometer.

Procedure: The temperature of a piece of fire-brick will be taken by optical pyrometer and by the calibrated thermocouple. The thermocouple will be located as close as possible to the surface read by the pyrometer. Temperature of the furnace as indicated by the meter located on the control panel should also be recorded.

To determine the temperature corresponding to that being measured by the thermocouple, focus the instrument on the object in the furnace by means of the knurled ring at the front of the telescope. Bring the filament to the point where it appears to cross the object.

Press the switch on the telescope and with this held closed, rotate the large knob on the box until the filament of the lamp blends with ( has the same brilliance as) the image of the hot object. Next, looking away from the telescope, keep the switch on the telescope closed and press the small knob in. While holding it in rotate it until the galvanometer pointer balances at zero on the scale. The object of doing this is to standardize the instrument. Then read the value of temperature (on the proper scale) which is under the hair line index over the scale. The proper scale to use is determined by the position of the index on the front of the telescope. If the "L" is opposite the fixed line, use the lower scale, and if the "H" is opposite the fixed line use the higher scale. Each student should take the reading through the optical pyrometer and at the time of reading another is to read the potentiometer .

Next take a glass plate between the pyrometer and the furnace, and proceed as above. Compare and comment on the results.

DEPARTMENT OF MATERIALS SCIENCE

SAN JOSE STATE COLLEGE

# EXPERIMENT No. 11

# TENSILE PROPERTIES OF HEAT TREATED ALLOY STEELS

Submitted by

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Submitted to

Professor Larsen- Badse

Materials Science 145

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## INTRODUCTION

Since the times of the ancient armorer that whirled a sword three times before he plunged it into an unfortunate slave, the art of heat treating has obviously progressed. Excepting the effects of alloying elements, the discussion that follows on the tensile properties of heat treated alloys is directed to the use of previously determined data involving tempering temperatures and resulting tensile stregnths. In much the same way we are like the armorer but we are prepared to deal with much more than swords.

## PRINCIPLES

As the object of this experiment is to heat treat a certain metal so that it will have specified physical properties, an adequate approach to the logic of such a procedure can be found in a study of the iron-carbon compounds and their mechanisms of formation. Then, by knowing these properties,andknowledge of their thermodynamics or the way they react to heat treatment will yield the manner by which they are obtained. Basically, then, the overall philosophy of heat treatment is manifested by theknowledge of the compounds and the way they react to heat treatment.

Properties of the iron-carbon compounds

As seen in the iron-carbon phase diagram (Fig. 1) and the time, temperature, transformation curve (Fig. 2), this experiment will deal with six major compounds: ferrite, austenite, cementite, pearlite, martensite, and banite.

Mat.E.145
Parkman

Experiment No. 11

## TENSILE PROPERTIES OF HEAT TREATED ALLOY STEELS

Object: To become familiar with methods of achieving specified tensile properties for alloy steels.

Material: Machined tensile specimens of A.I.S.I.4130 and A.I.S.I. 4140 steels will be available.

Procedure: It will be required to heat treat a tensile specimen of one of the above types to give mechanical properties within specified limits listed below:

| | AISI 4130 | AISI 4140 |
|---|---|---|
| T.S. - | 130,000 psi (± 3000 psi) | 140,000 psi (± 3000 psi) |
| % Elongation | 17% min. | 16% min. |

Determine from published data the required procedure and make a number of decisions such as (a) preliminary treatment, if any, (b) time and temperature for austenitizing, (c) quench medium, (d) temperature, time and subsequent cooling for austenitizing, etc.

Since only one specimen will be issued to each group, it is important that the steel should not be tested in the tensile machine until the group makes fairly sure, by any means available to them, that the required physical properties have been achieved.

The following data should be obtained:
- (a) tensile strength
- (b) yeild strength by offset method
- (c) % elongation
- (d) % reduction of area
- (e) fracture appearance
- (f) Rockwell hardness
- (g) grain size
- (h) micro

Exercises: Do you feel that the mechanical properties which you obtained could have been achieved by any different type of heat treatment sequence? Explain.

What effect might changes in the following factors have upon your results?
- (a) Difference in grain size
- (b) Difference in section size
- (c) Difference in quenching medium
- (d) Difference in post-temper cooling

Refrer References:
Bullens-Battelle, Steel and Its Heat Treatment, Vol. 1
A.S.M., Metals Handbook
U.S. Steel, Carilloy Steels
Climax Molybdenum Company, Molybdenum in Steel