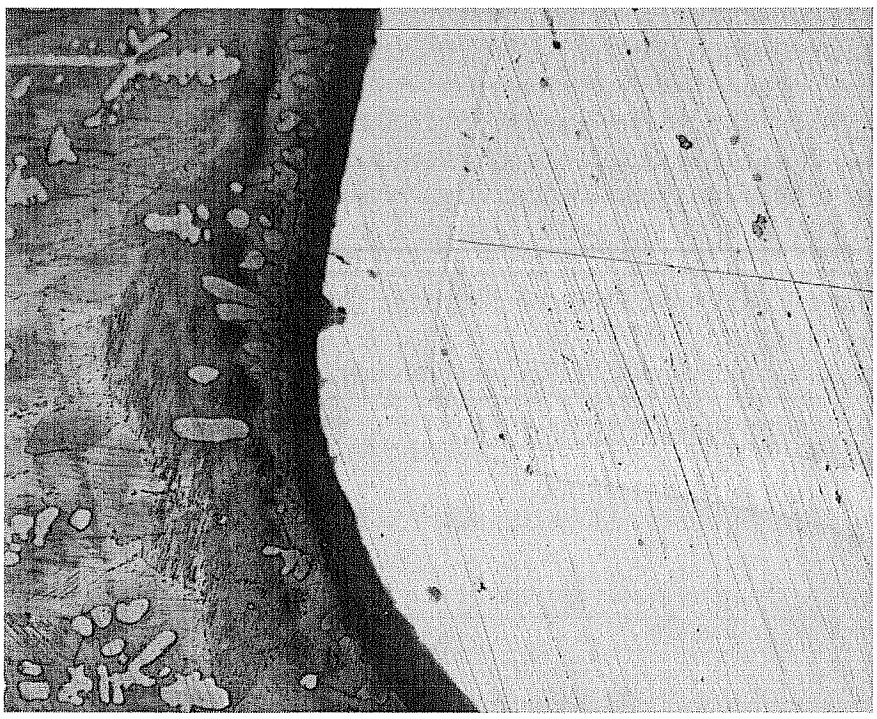
Avancerad materialdesign

MH2201

Practicals



2009

PM for the Practicals in Materials design

There are three compulsory practicals which are performed in groups of maximum three students

- Alloying by Diffusion
- Gas Carburization of Iron
- Isothermal Transformation

Reports:

Write each report after the instructions given in this PM. The reports should be written with great care and give a clear picture of what has been done and what the results are. Anyone should be able to read and understand the report without first reading the instructions for the practical. If information from literature is used the source must be referred to. If there is an abnormal discrepancy between your results and literature data or your results seem unreasonable you should discuss possible sources of error.

The reports must be handed in within two weeks from the date of the practical and will be examined by the teachers. Reports that do not fulfil the requirements will be given back to the students for revision. A revised report must be handed in at least three weeks before the date of the project seminar. Practicals which are not completed according to these rules will not be accepted. If a practical has not been accepted a second chance is given next year. If you are unable to attend a practical, due to illness or for some other reason you must contact the person responsible for the practical in question in order to decide how and when the practical can be performed. It must be emphasised that it is NOT possible to complete or perform a practical after the course is finished.

In order to finish the practical within five hours it is advisable:

- To read the instructions and connected pages in the literature in advance.
- To read the instructions and the connected pages in the book in advance
- That all the group members are in time and immediately contact the person responsible for the practical in question.
- That measurements or calculations to be made at the end of the practical are prepared during the experimental work.
- To divide the work.

Literature:
Some useful books to be found in the local library:

Diffusion	
Smithells, Metals Reference Book I-II	X 12- 13
TTT- and CCT-diagram	
Atlas zur Wärmebehandlung der Stähle	Z 22
U.S: Steel, Atlas of isothermal transformation diagram, supplement	Z 21
Etching Handbooks	
Berglund, Etshandbok	O 2
Physical properties , heat treatment etc.	
Metals Handbook	X 9-10
Steel catalogs	Z
Properties of the EN STEELS	Q 52

How to write the report

The report is a record over an investigation or/and an experiment, how it was performed, what the results are and it usually contains a discussion as well as major conclusions that can be drawn. The report should be written so that anyone can understand it and repeat the experiment. The report should contain the following headers.

Title

Authors and their adresses

Abstract
Only a few (5-10) lines - short summary of the whole report.

Introduction
This section should include a short presentation of the background and the purpose of the experiment. One should also describe the phenomena which will be studied and expected results.

Experimental methods
This section contains the materials used, the heat treatment, sample preparation, type of measurements, calculations and/or observations that have been made and how the data have been treated.

Theory
Relevant theories are presented.

Results
Here the measured, observed and/or calculated results are presented.

Discussion and conclusions
Under this section the results are discussed. Are they unexpected? Do they violate established physical principles or theories? Do they support one hypothesis rather than another one? State your conclusions.

(Acknowledgments, optional)

References
Example:
K.Kuo, J. Iron Steel Inst.,Vol.181, 1955, pp.134-137.

Alloying by Diffusion

The purpose of this practical is to illustrate how phase diagram data may be used to predict the occurance of intermetallic phases in contact with a melt. Prepare yourself by solving the problem below.

Hot dip galvanization is a common method to improve the corrosion resistance of iron and steel products. The objects to be protected are dipped in liquid zink and a zink-rich layer that provides galvanic protection is formed on the surface. Apart from the ordinary solid Zn-phase a number of intermetallic phases form and since some of the intermetallic phases are quite brittle this may cause problems if the shape of the protected object is changed after galvanizing. It could therefore be interesting to know how to avoid the intermetallic phases.

For practical reasons you will instead of the Fe-Zn system which would be relevant for the case above, investigate the Cu-Zn system.

Problem

Consider the Cu-Zn phase diagram. What phases may appear at different temperatures? Sketch a reasonable concentration profile in a Cu specimen that has been immersed in liquid Zn for a while at 500°C.

Experimental Details

The assistant will provide copper, zink and three glass tubes. The experimental set-up consists of a furnace and a designed specimen holder.

Experimental Procedure

1. Heat the furnace to approximately 500°C.

2. Put zink in the tubes and enter the tubes into the furnace. Wait approximately 15 minutes to level the temperature and allow for the zink to melt.

3. Without removing the tubes from the furnace, enter the pieces of copper, one in each tube. Heat treat for 20, 40 and 80 minutes. Remove the tubes from the furnace one by one and let the specimen cool in the designed holder.

4. When the first specimen has cooled for a couple of minutes, rinse the tube in water (to make sure that it is actually cold) and remove the specimen from the tube using e.g. a sledge hammer and protective goggles. Then grind off the zink until the copper piece appears, mount it in bakelite and grind and polish the specimen.

5. Etch the specimen using nital and investigate the microstructure. Compare with the Cu-Zn phase diagram.

6. Repeat the above steps for the other specimens.

7. Measure the thicknesses of the layers as a function of time. (What time dependence do you expect?) Which conclusions may be drawn regarding the relative size of the diffusion coefficients in the different phases?

8. Ask the assistant for a preprepared specimen. This specimen has been heat treated at another temperature. What is the main difference between the specimens prepared by you and the assistant's specimen? Try to find out in which temperature regime the assistant's specimen was heat treated.

Gas Carburization of Steel

The purpose of this practical is to illustrate case hardening and how thermodynamical and kinetical considerations may be used to estimate the process parameters to obtain a case with desired properties.

Case hardening is a common method to improve hardness and wear resistance of steels. During the process the carbon and/or nitrogen content in the near-surface region is increased and a hardenable rim is formed. Carburized materials combine a hard surface with a tough interior and are often competitive when compared with homogeneous carbon steels. Only the case is transformed to martensite which reduces the risk of cracking during hardening. As bulk material, cheap low-carbon steels may be used and since hardening is carried out after shaping, machining is made easier. Typical applications are gear wheels and lock shackles.

Carburization is always performed in the austenite region since the solubility of carbon in ferrite is much lower. There are three main categories of carburization methods: pack, liquid and gas carburization. Today, the most popular method is the latter, in which the material is heat-treated in a carbon containing atmosphere. Pack carburization is achieved by packing into a solid mixture of carbonates and charcoal or coke but is hard to control and not well adapted to industrial production. Liquid carburization is performed in liquid cyanide baths, which imposes environmental limitations on its use.

The mechanical properties of the case are governed by its composition and thickness which in turn depend on the carburization temperature and time. In order to obtain a case with certain properties one may either adopt the trial-and-error approach or try more sophisticated methods, i.e. solve the diffusion problem for this situation.

Process modelling

During carburization carbon diffuses through the surface and into the material. In order to estimate the local carbon concentration one needs the concentration of carbon at the surface and a suitable solution to the diffusion problem. Lacking more sophisticated data than the Fe-C phase diagram one may estimate the surface composition as the composition of austenite in equilibrium with graphite at the temperature under consideration. The diffusion equation can by solved analytically in the current case if one assumes a constant diffusion coefficient. The concentration in the material can then be described with the erf-solution

$$c = A + B\,erf\left(\frac{z}{\sqrt{2Dt}}\right) \qquad (1)$$

where z is the distance to the interface. The parameters A and B may be adjusted to agree with the surface and initial concentrations.

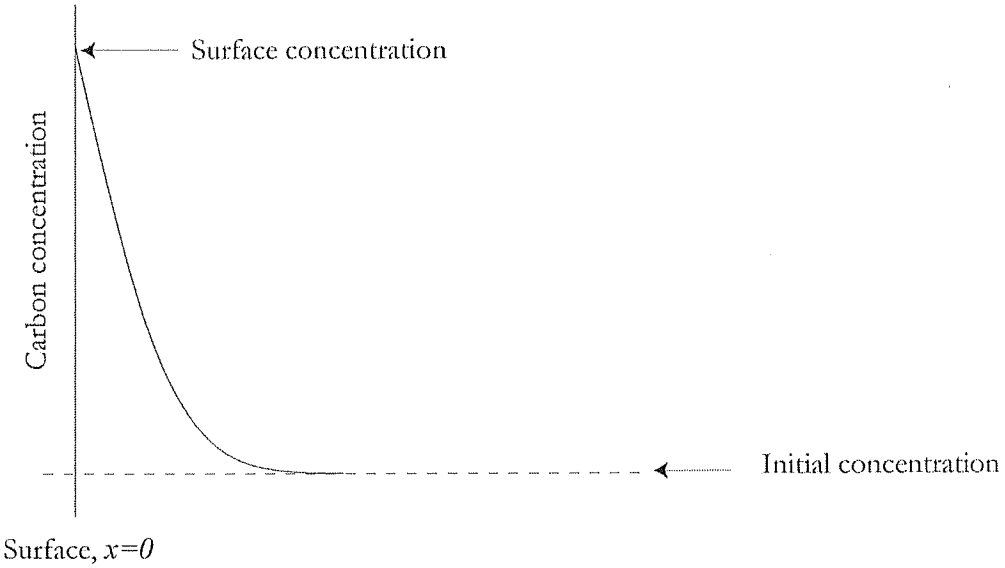


Figure 1. The approximate carbon distribution profiles after carburization.

When the specimen is quenched and the temperature of the austenite drops below the so-called M_s temperature the transformation to martensite starts. M_s depends on the composition of the material and decreases if the carbon content is increased. Carbon also affects the hardness of the martensite, as illustrated by the appended diagram.

Problem

Estimate the hardness as a function of distance from the surface for hardened specimens carburized at 870°C for 15, 30 and 60 minutes. Use the erf-solution and the Fe-C phase diagram or Dictra to solve the diffusion problem. Suppose that all austenite is transformed to martensite and that the initial carbon concentration is zero.

Experimental details

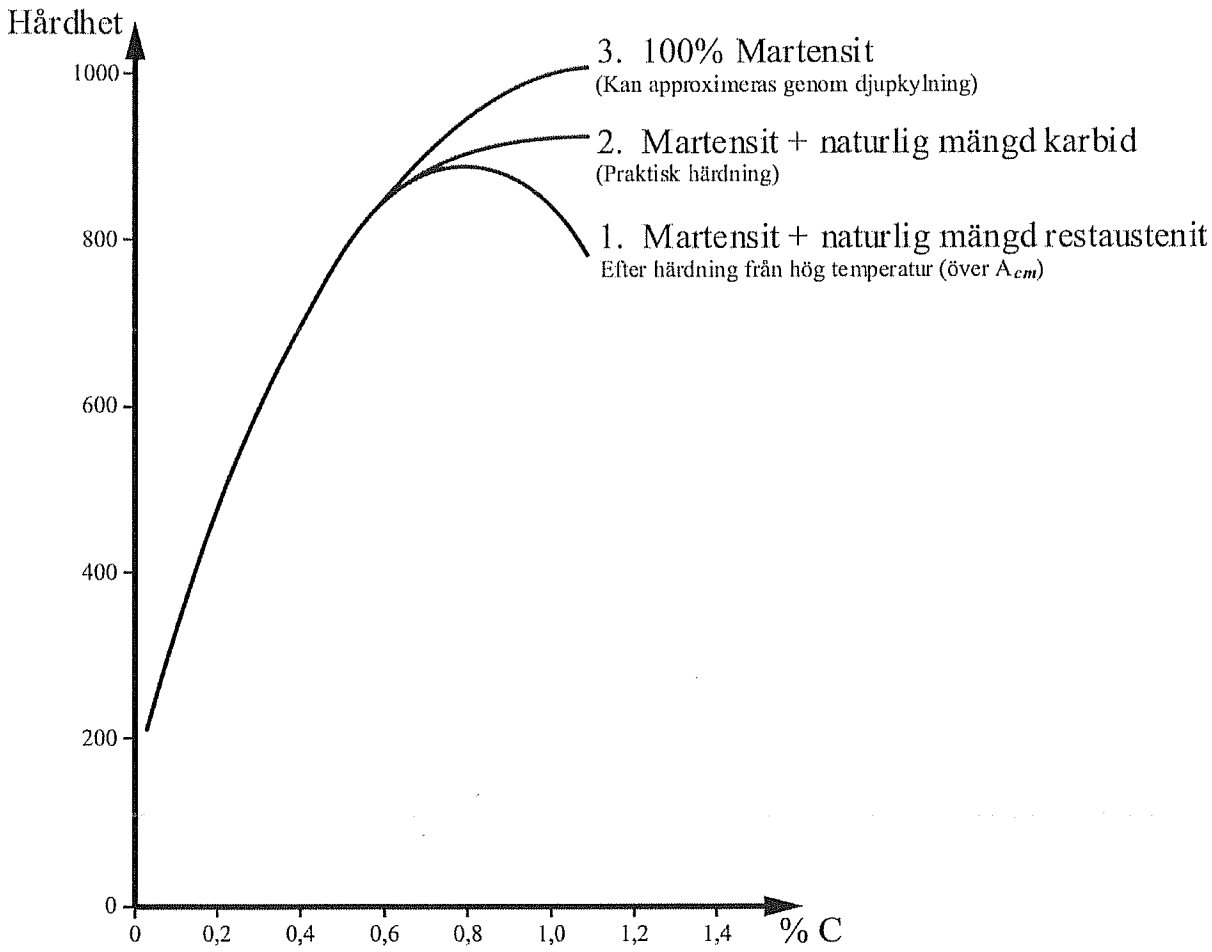
The assistant will supply three pieces of low carbon steel (<0.05%C) which are to be carburized. The experimental set-up consists of a tube furnace which contains an atmosphere which is either inert (nitrogen) or carburization (nitrogen + hydrocarbon). The gas flow is regulated by knobs which will be demonstrated by the assistant.

Experimental procedure

- Heat the furnace to approximately 870°C. Flush the furnace with nitrogen gas.
- Enter the specimens into the furnace, flush the furnace first with nitrogen only and then with the nitrogen + hydrocarbon mixture. Carburize the specimens for 15, 30 and 60 minutes respectively and quench them into water.

If you managed to carburize the specimens they are now considerably harder than before. Try to cut them using a hacksaw. You will probably not manage. Instead, grind away the hardened layer on one side of each specimen and mount them in bakelite. Make sure that the specimens are mounted in such a way that the hardened layer can be investigated. Grind the specimens using grinding paper but do not polish them.

- Measure the micro hardness from the edges into the middle of the specimens. Compare the experimental hardness curves with the calculated ones. Usually the hardness is found to drop close to the edge. Why?
- Grind the specimens again to remove the indentation marks.
- Examine the microstructure.


Hårdhet
1000
800
600
400
200
0
0
0,2
0,4
0,6
0,8
1,0
1,2
1,4
% C
3. 100% Martensit
(Kan approximeras genom djupkylning)
2. Martensit + naturlig mängd karbid
(Praktisk härdning)
1. Martensit + naturlig mängd restaustenit
Efter härdning från hög temperatur (över A_{cm})

Isothermal Transformation

Preparation

Read about the KJMA-equation in chapter 6 in the book "Mikro och nanostrukturer".

The purpose of this practical is to study the isothermal transformation of the material close to the pearlite nose.

Material

Steel with the composition 0.35%C, 0.8%Mn, 1.1%Cr and 0.2%Mo.

Experimental Procedure

1. Make sure that you are acquainted with the equipment. Do not hesitate to ask the assistant if you feel uncertain about some parts of the laboratory work. Remember to open the damper above the lead bath.

2. See attached TTT-diagram for an alloy which is close in composition to the above. Choose austenitizing temperature and adjust the temperature on the furnace.

3. Choose a heat treatment temperature first and adjust the temperature of the lead bath. Then agree on a number of heat treatment times.

4. Mount each sample on a separate welding wire using a thin wire, see Figure 1. Make sure that the samples move easily and minimize the risk for wires and samples clinging to each other.

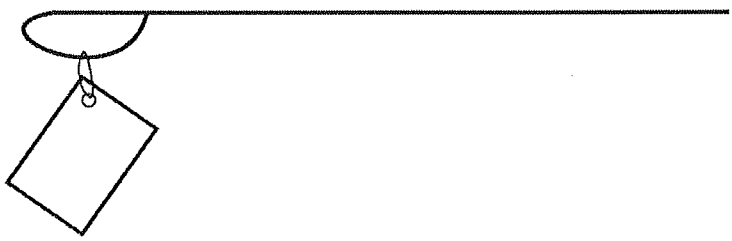


Figure 1. Mounting of sample.

5. Turn on and adjust the nitrogen gas flow through the austenitizing furnace and enter all the samples at the same time.

6. When the austenitizing is completed it is important to move all the samples at the same time as fast as possible to the lead bath. (Note that the sample holders, welding wires, could easily bend if not moved carefully enough or if the coal is not kept out of the way.)

7. Take the samples out one at the time and quench them in salt water. Make sure to keep the different samples apart.

8. Mount a few samples together, then grind and polish. Use nitric acid and ethanol ("Nital") as etching agent and begin with short etching times (seconds).

9. Each one in the group then makes a visual estimation of the amount pearlite transformed in each sample. Then make estimation with an ocular grid using an "intersection - pearlite - coincidence - counting - method".

10. Describe the rate of transformation with the generalized KJMA - equation by fitting a K and an n constant.

11. Turn off all equipment. Add some more coal on top of the lead if needed, return pliers etc. and finally CLEAN UP around the equipment.

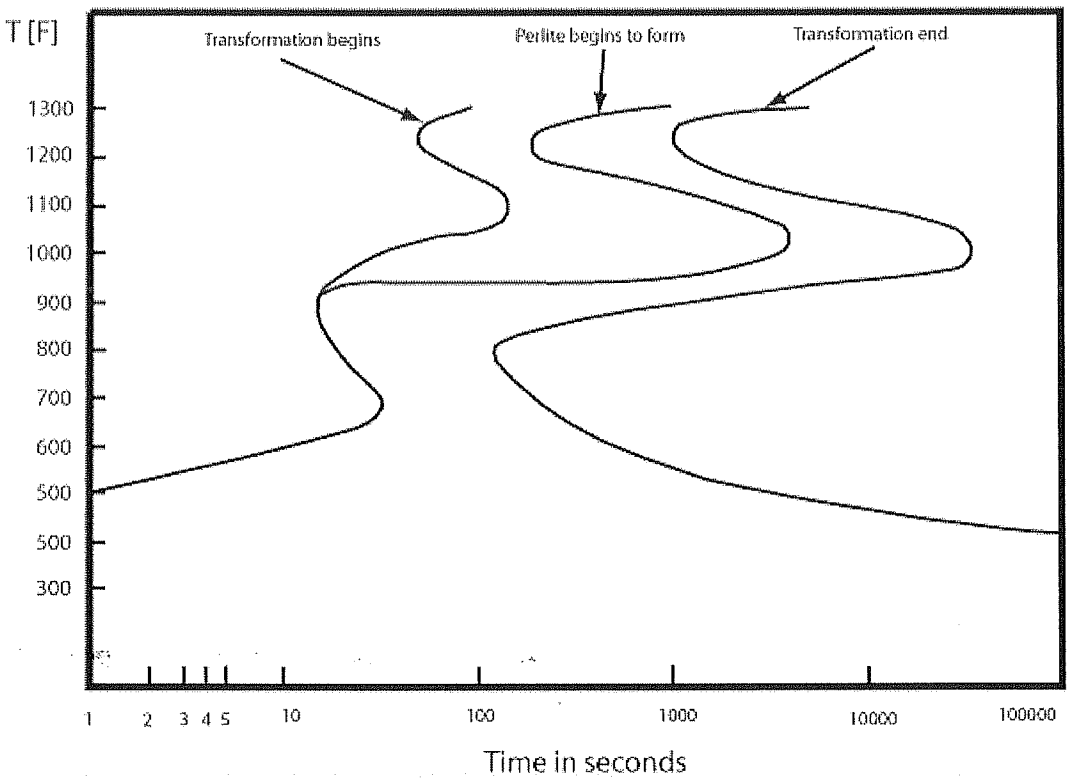
Summary of the aim:

I. Find the time when pearlite first appear and compare with the appended TTT-diagram.

II. Find a description for the transformation using KJMA's generalized equation.

III. Last but not least, focus on the possible errors and uncertainties during the work.

SAE 4140



C - 0.38
Mn - 0.82
P - 0.018
S - 0.022
Si - 0.23
Ni - 0.29
Cr - 1.02
Mo - 0.20