**University of Ulm**
**Institute for Micro- and Nanomaterials**

# Lab
# „Materials Science“

**Summer Term 2007**

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F1: Microstructure I
lab on June, 21st 2007

# 1 Questions

## 1.1 What is the goal of metallographic sample preparation?

The goal of metallographic sample preparation is to obtain a sample, which is representative for the examined material. You should be able to take a look at all interesting structures of the sample, e.g. grains or grain boundaries.

## 1.2 What definition of microstructure can you give?

A microstructure is composed of chemically and physically homogenous microregions which are seperated by interfaces and are called phases. That means, if looking to the microstructure of some material, you can see the grains (chemically and physically homogenous regions) and the grain boundarys as well as lattice defects - these are also part of the microstructure.

## 1.3 Discuss the Fe-C diagram given below. Identify the regions of pure $\alpha$ and pure $\gamma$ phases. What reaction occures during cooling at 0.8 wt% C alloy below 7272°C? Name and describe the microstructure formed during this reaction. What happens during cooling of an alloy with lower carbon content (e.g. 0.25 weight% C)? Describe the microstructure formed for this composition.

In the figure below (fig. 1) you can see the regions of pure $\alpha$-phases (marked green) and of pure $\gamma$-phases (marked blue).

If we take a deeper look at the processes which happen at 0.8 weight% C (red line in fig. 1) we can see, that by cooling the material first turns into austenite ($\gamma$-phase) at about 1350°C (the formation of austenite begins at about 1480°C). For quite a long temperature range the FeC stays in the austenite-phase. At temperatures below 727°C (eutectid temperature) the austenite turns into cementite and pearlite. The pure cementite phase is built only in a short temperature range (about 740°C and 727°C), therefore we would expect to see only a few cementite grains at the end.

If we cool down an alloy with a lower carbon content (e.g 0.25 weight%, orange curve in fig. 1), we can see that first the material turns into some $\delta$-phase at about 1530°C and afterward becomes austenite (at nearly 1500°C). The austenite phase longs until we reach a temperature of about 820°C. Here the austenite turns into ferrite. The pearlite phase is

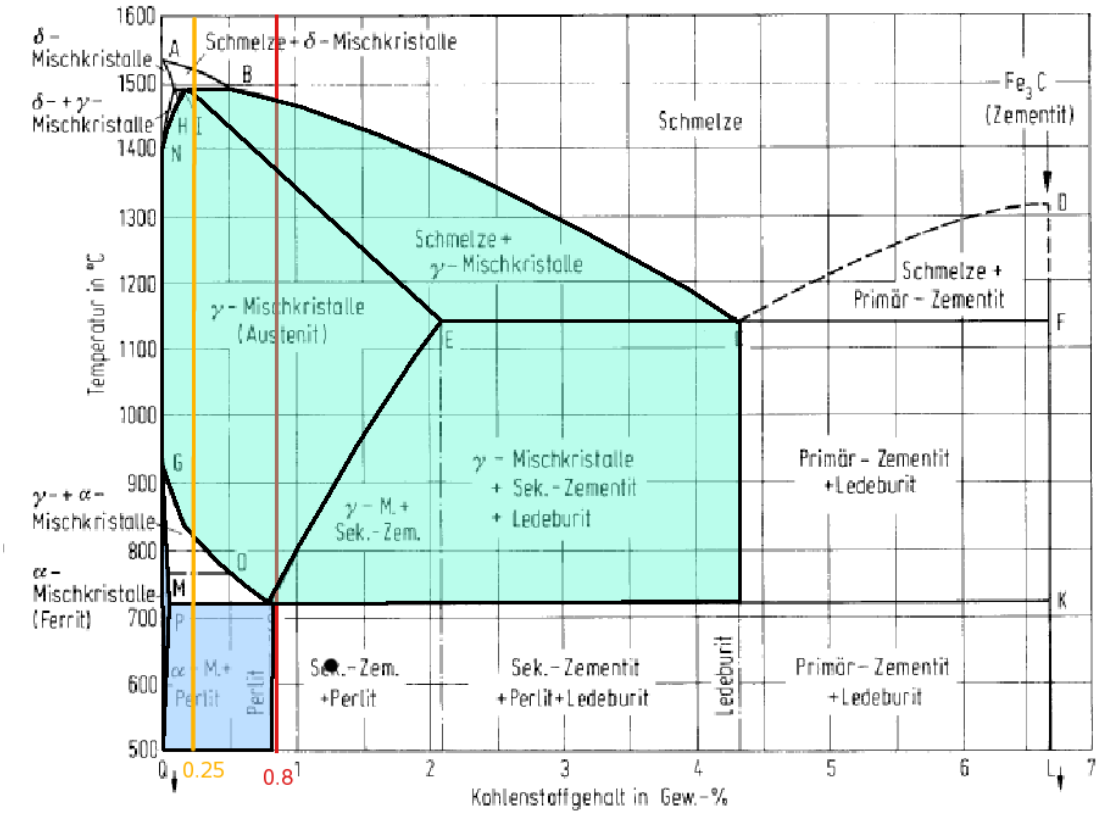


Fig. 1: Fe-C-Phase Diagramm

reached at about 720°C. As the temperature range of the pearlite phase is quite big (nearly 100°C), we would expect a microstructure being half out of pearlite and half out of ferrite.

## 1.4 Discuss the time-temperature-transformation diagram of steel as shown below. Which phase is obtained by very rapid cooling ($< 1$ s) and what are its properties? What happens after quenching (fast cooling to 600°C and isothermal annealing at that temperature (phases, microstructure)? Explain why the kinetic of the transformations are different for both cases.

As you can see in fig. 2 we obtain austenite and martensite if we cool down the melted material very rapid (blue region in the figure 2). If we cool down the material to 600°C and anneal it isothermal at this temperature, we obtain first austenite which begins to turn into pearlite after a bit more than one second. After 10 seconds there no austenite remains and

the whole material has turned into pearlite (see green curve in fig. 2).

This behaviour is due to the fact, that with fast cooling the C-atoms stay as interstitial in the $\alpha$-mixed-chrystal; this leads to tension in the lattice and so this martensit is harder than the other FeC-phases. By annealing the probe at 600°C the C-atoms can diffuse through the lattice and pearlite forms, which is not so hard as martensite.

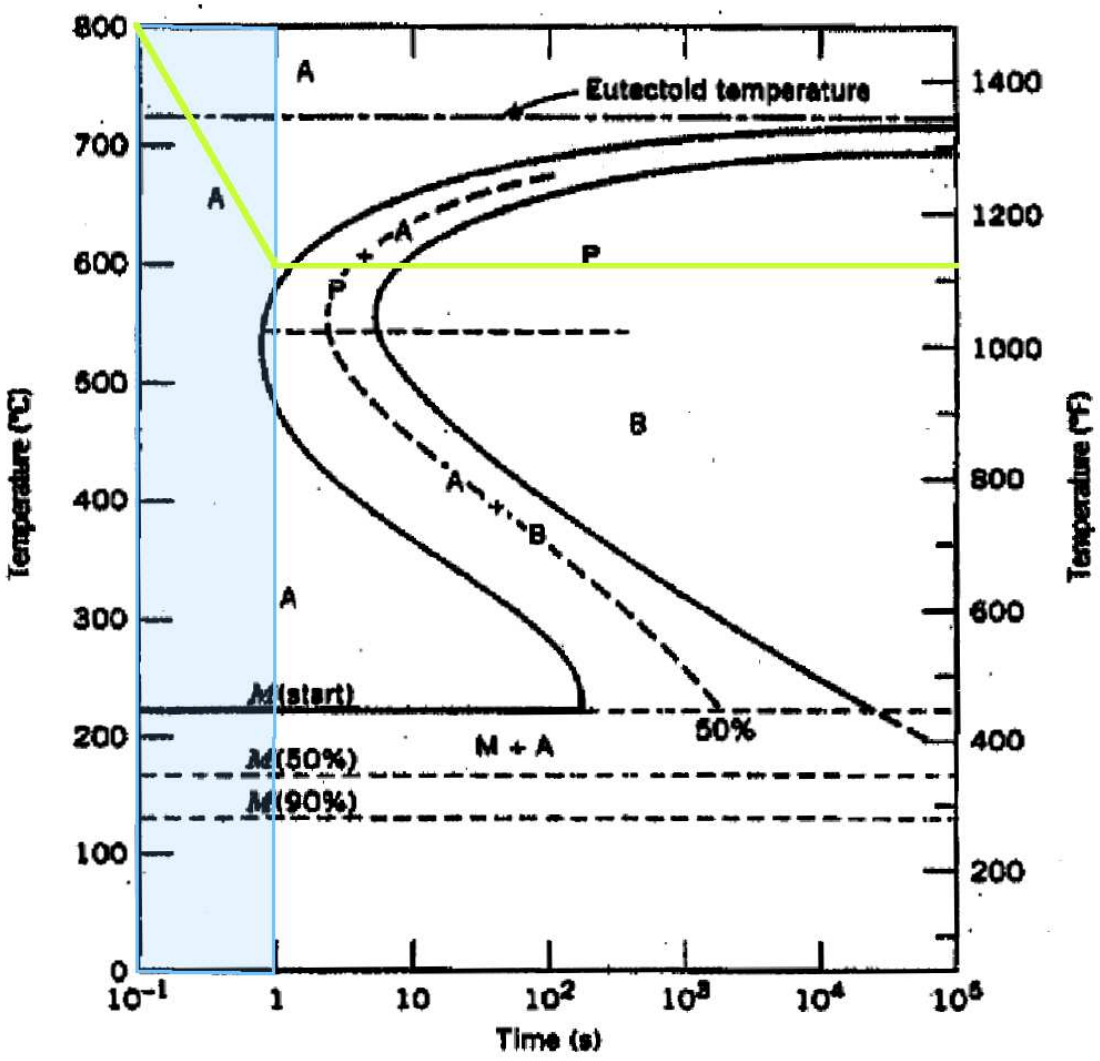


Fig. 2: Time-Temperature-Diagramm

## 1.5 Describe the usual sequence of steps of the metallographic specimen preparation.

Preparing of a specimen for metallographic examination is done as follows:

1. **Sampling**

   First a specimen has to be cut out of a greater sample.

2. **Mounting**

   Then this specimen has to be cold or hot mounted.

3. **Grinding**

   After mounting the specimen has to be grinded abrasively.

4. **Polishing**

   Next thing is to polish the specimen. Like grinding this is normally done in different steps, getting finer each time.

5. **Etching**

   To make the different phases visible, the specimen in the end has to be etched with a fitting etching solution.

6. **Examination**

   After the specimen is prepared, it can be examined under a optical microscope for example.

## 1.6 Which are the general requirements to a metallographic specimen

A metallographic specimen has to fulfill the following requirements:

- the specimen must be representative
- all structural elements must be retained
- the surface must be without scratches and deformations
- no foreign matter must be introduced in the specimen surface
- the specimen must be plain and highly reflective
- all preparations must be 100% reproducible

### 1.7 Why should we thoroughly clean (wash) the specimen after each grinding/polishing step?

We have to wash the specimen thoroughly after each step to wash off the residues of the abrasives and lubricants and the chips of the specimen.

### 1.8 How can we choose the etching solution and etching conditions fot the certain alloy?

Eiter we know which etching solution whe have to use e.g. from literature. If we don't know how long to etch, we can try this out by etching some seconds, look at the probe under an optical microscope, and etch further or not.

In general we can state, that the etching solution has to be suitable for the material.

## 2 Carrying out the Experiment

In this lab we examined two probes, one was a high-carbon steel with 0.6 weight% C and the other was a low-carbon steel with 0.1 weight% C.

The high carbon steel probe has been already cut, mounted, grinded, polished and etched, so we only had to prepare the second probe.

The first thing we did was to cut of a small peace of the low carbon steel probe. After this was done, we mounted it with epoxy (cold mounting).

While this probe was hardened, we took another already mounted probe and polished it again, because it has been etched already. The polishing was done in two steps with two different surface (the second was finer [0.02 $\mu$m] than the first [0.1 $\mu$ m]). After each step we thouroughly washed the probe, to clean it from residues.

After the probe has been polished we took a look at the unetched specimens under the optical microscope.

In the figures 3 and 4 we can see the result for both probes, the high- and the low-carbon steel. As we can see, before etching there is no difference between the things we can see from the two probes.

The next step was to etch the probes with a 3% solution of $HNO_3$ in ethanol for 40 seconds.

Etching reveals the microstructure of the specimen, because the different phases react chemically in a different way with the etching solution.

After the etching is finished we looked again at the specimen under the optical microscope.

What we could see then is showed in the figures 5 and 6.

The results were as we expected: in the low carbon steel we can see a lot of grains out of ferrite and only few pearlite (dark regions). If we would have had a greater resolution at our optical microscope we would have been able to see the lamellar structure of the pearlite.

In the high carbon steel the pearlite regions are much more (the probe is nearly at the eutectid point) than the ferrite grains. We now had a greater resolution and can see the lamellar structure of the pearlite.

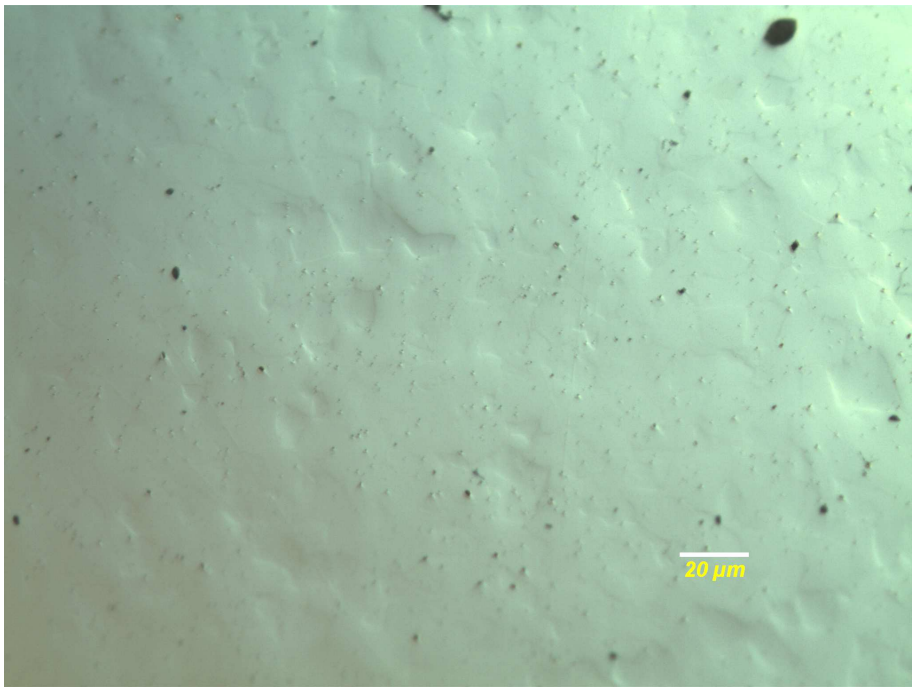


Fig. 3: Low Carbon Steel before Etching

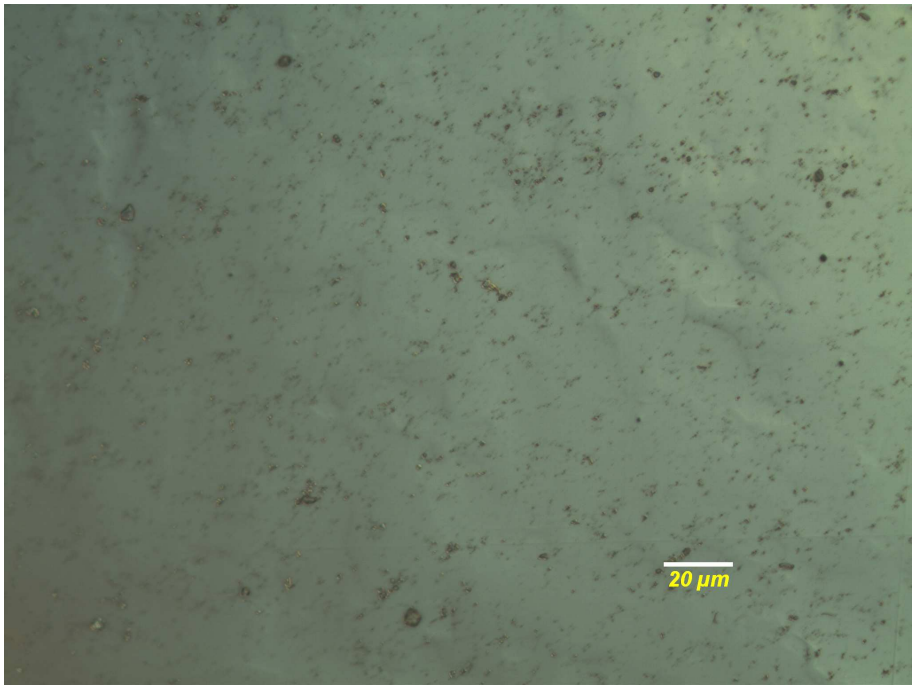


Fig. 4: High Carbon Steel before Etching

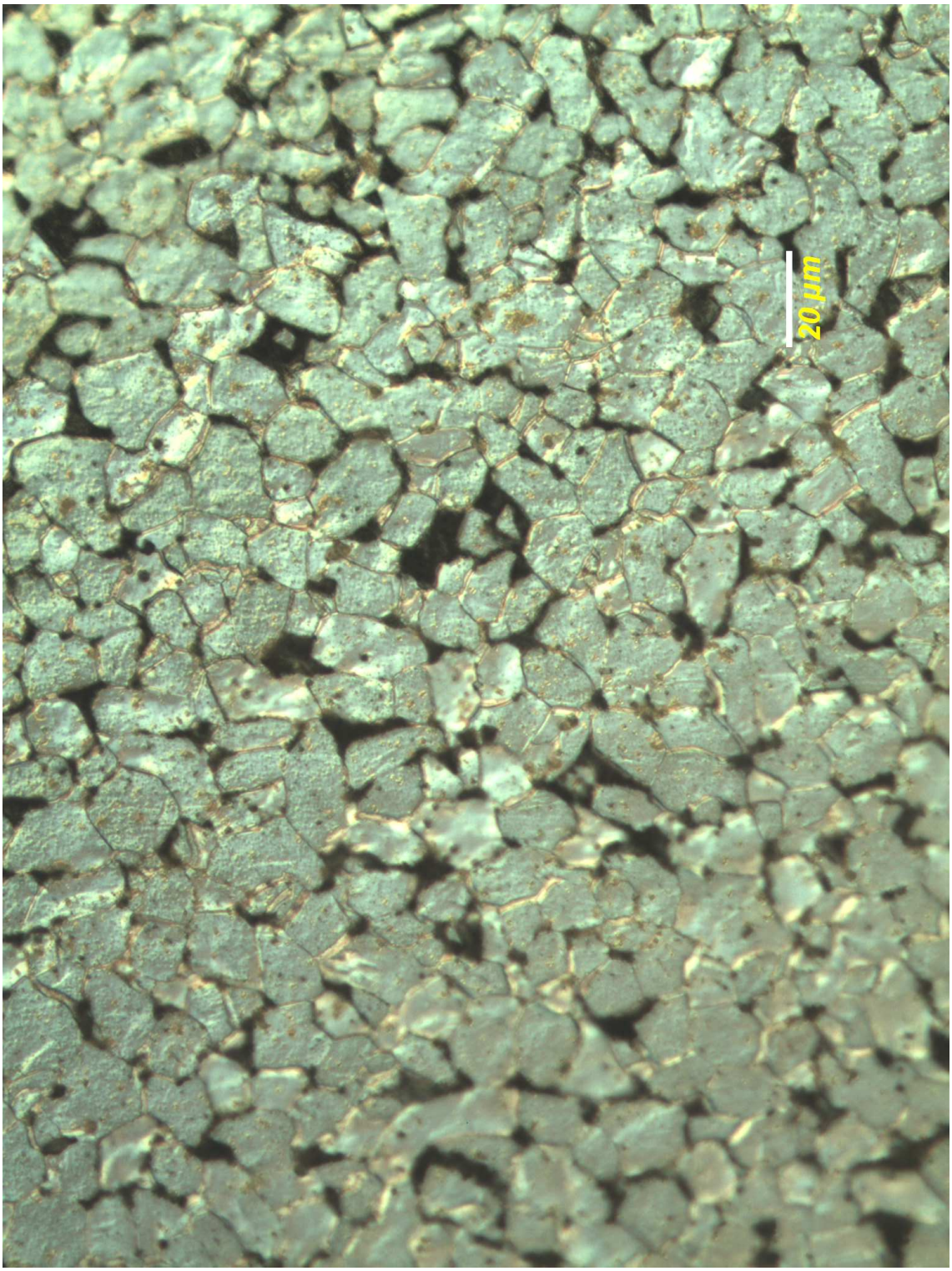


Fig. 5: Low Carbon Steel after Etching

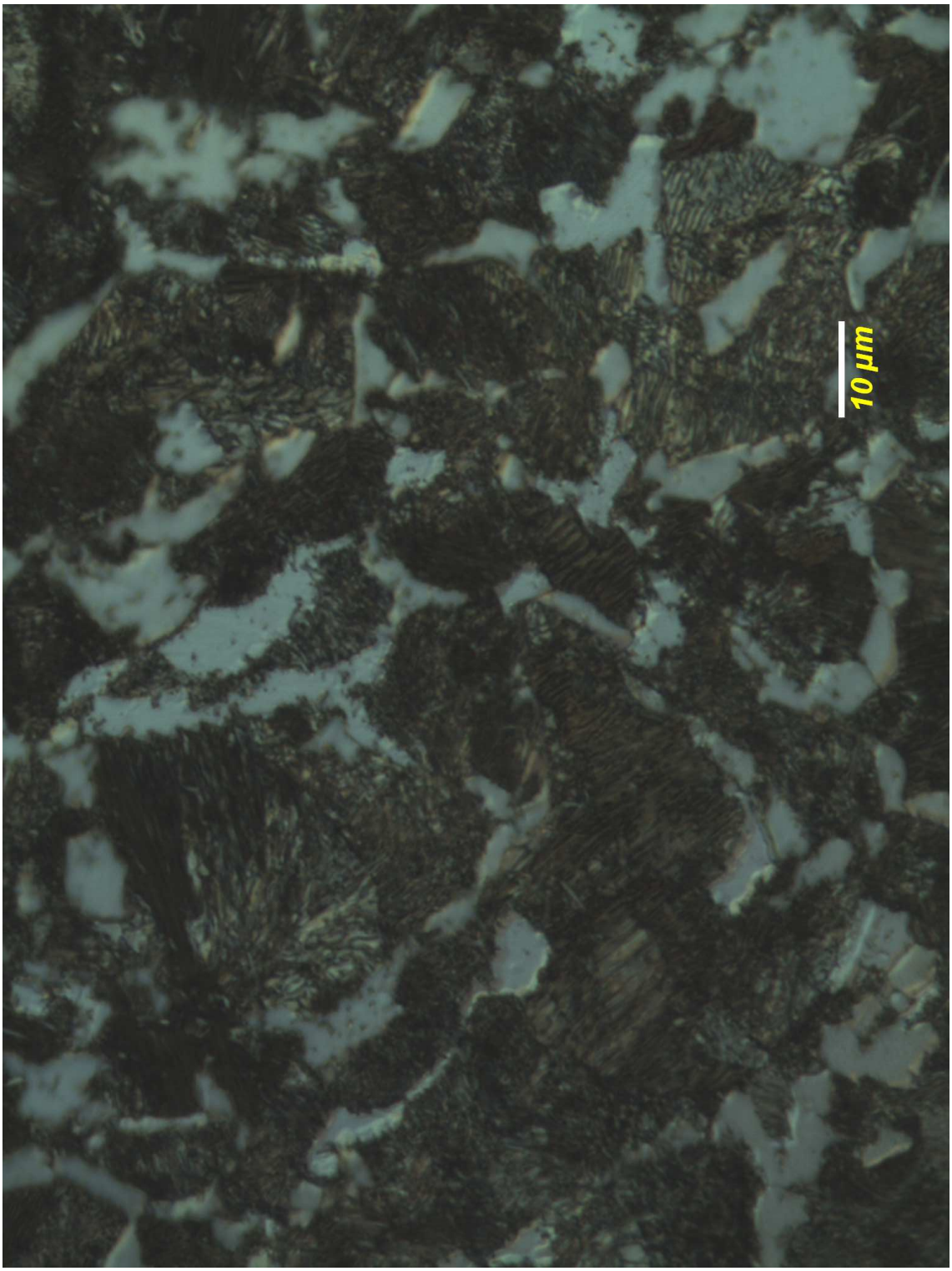


Fig. 6: High Carbon Steel after Etching