PART IB EXPERIMENTAL ENGINEERING

SUBJECT: **MATERIALS** **EXPERIMENT M2**

LOCATION: **MATERIALS TEACHING LABORATORY** **(SHORT)**

HEAT TREATMENT, MICROSTRUCTURE AND PROPERTIES OF CARBON STEELS

OBJECTIVES

1. To examine the microstructure of annealed mild (low carbon) steel and medium carbon steel. To observe changes in microstructure of medium carbon steel brought about by quenching and tempering heat treatments.
2. To use the Vickers Hardness machine and the Izod impact machine to measure hardness and relative toughness of normalised and heat-treated samples of steel. To assess how strongly these properties depend on the amount of carbon in the steel and on the type of heat treatment.
3. To relate the hardness and relative toughness of annealed and heat-treated steel samples to their corresponding microstructures.

Safety matters

1. **Gloves and goggles must be worn when removing specimens from the high temperature furnace. This operation must only be performed under the direct supervision of a demonstrator.**
2. **Specimens removed from the tempering furnace should be cooled in water immediately. Immerse the hook as well as the cage. NB: a hot specimen cooling on the bench might inadvertently be picked up, causing burns to the hand.**
3. **The special instructions given by the demonstrator for the safe operation of the Izod impact machine must be strictly followed.**

BACKGROUND

Metals and alloys for structural and mechanical engineering applications usually need to be hard, strong and tough. These properties depend partly on composition and partly on the microstructure, that is on the size and distribution of the phases and grains. For a metal, the finer we make the grain size (by controlling the casting, deformation and heat treatment processes) the greater the hardness, strength and toughness. But the mechanical properties depend even more strongly on the phases present, on a finer scale than the grains.

In alloys, the phases formed and the mechanical properties depend on composition in a rather complicated way. In its simplest form, steel is an alloy of iron containing a small amount of carbon, between about 0.1% and 1.0% by weight. Pure iron is tough but relatively soft, but if the carbon content is increased and the alloy cooled slowly (‘normalised’) the hardness and yield stress increase substantially, while the toughness falls away. However, if we take a sample of steel containing an intermediate amount of carbon, say 0.4%, heat it to a high temperature (850^{o}C) and then quench it in water, a dramatic increase in hardness occurs – with an equally impressive loss of toughness. This is because quenching changes the phases present.

Re-heating the hard but brittle quenched steel to a lower temperature (500^{o}C) is known as ‘tempering’. This reduces the hardness but restores most of the original toughness – due to a further change in phases present in the microstructure. The versatility of carbon steel as an engineering material stems from the ease with which its mechanical properties can be altered simply by adjusting the carbon content and heat treatment.

EQUIPMENT, STEELS & SPECIMENS

Equipment

Furnaces at 850°C; fluidised bed furnaces at 500°C; water baths for quenching. Vickers hardness machines with 30 kg loads; Izod machine with 163 Joule (120 ft.lb) hammer. Microscopes and prepared specimens.

Steels and Specimens

1. One "short" specimen of mild (low carbon) steel (M20) with a single 2.76 mm notch in the *normalised* condition (i.e. 100 minutes at 900°C and air cooled). This produces the equilibrium microstructure that characterises the normalised state. The composition is to specification BS070M20, containing 0.7% manganese and **0.2% carbon**.
2. One "short" specimen of medium carbon steel (M40) with a single 2.76 mm notch in the *normalised* condition (i.e. 60 minutes at 850°C and air cooled). The composition is to specification BS080M40, containing 0.8% manganese and **0.4% carbon**.
3. One "long" specimen of medium carbon steel M40 (**0.4% carbon**) with 2 notches. This specimen will be in the furnace at 850°C, ready for quenching in water.

EXPERIMENT

The experiment is divided into four parts as follows:

1. Water quenching a medium carbon steel (M40) specimen from 850°C to room temperature.
2. Performing hardness and Izod impact tests on a range of specimens.
3. Using an optical microscope to examine the microstructure of the same range of specimens.
4. Discussing the experimental findings with the demonstrator.

Two demonstrators will each take three groups through the experiment. One set will perform the experiment in the order 1, 2, 3, 4, and the other in the order 3, 1, 2, 4.

Brief explanatory notes on the microstructural changes taking place in the steel are given in Appendix 1. There is some repetition in the explanations to allow for groups performing Part 3 before Part 2.

1. Water quenching a medium carbon steel (M40) specimen from 850°C

Wearing the gloves and goggles provided, and under the supervision of the demonstrator, water quench the double-notched M40 specimen. The specimens in the furnace have been held at 850°C for more than 25 minutes (see Note 1, Appendix 1).

2. Performing hardness and lzod tests

1. Perform an Izod test (see Note 2, Appendix 1) on the water-quenched specimen as instructed by the demonstrator. Have one notched length only, projecting above the vice. ***Observe the safety instructions during testing***. Enter all measurements on the Results pages.
2. Suspend the larger of the two broken pieces of the quenched specimen in the fluidised bed at 500°C and leave for about 20 minutes to temper (see Note 3, Appendix 1).
3. Use the Vickers hardness machine to measure the hardness of the normalised M20 and normalised M40 specimens, and also measure the hardness of the smaller broken piece of the quenched M40 specimen. Enter the values on the Results pages.

4. Measure the Izod values for the normalised M20 and M40 specimens. Enter the values on the Results pages.
5. Measure first the hardness, and then the Izod values, of the tempered M40 specimen. This specimen should be cooled in water on removal from the tempering furnace (do not forget to immerse the hook as well as the cage). The cooling now has no effect on the microstructure – it is performed here as a safety measure and to reduce the waiting time. Enter the values on the Results pages.

3. Optical microscopy of etched steel specimens

Each student has 4 microscope specimens in differently coloured mounts, each corresponding to one of those whose mechanical properties are measured during the experiment. Each specimen has been polished and then etched in Nital (nitric acid/alcohol solution) to show the microstructure. *Examples of typical microstructures are shown on the Results pages.*

- **Red mount – M20 normalised**: mainly *ferrite* (light grains) with some *pearlite* (dark regions).
- **Green mount – M40 normalised**: less *ferrite* and more *pearlite* (roughly 50:50)
- **Blue mount – M40 quenched from 850°C**: *martensite* (fine needle-shaped light grains).
- **Black mount – M40 quenched from 850°C and tempered at 500°C**: *tempered martensite* (similar to as-quenched, but grains appear darker).

Do not touch the metal surfaces directly. Work through the specimens entering your answers on the **Results pages.** The demonstrator will instruct you on the use of the microscope, and help you to interpret the microstructures. A glossary of the names of phases and microstructures in plain carbon steel is given in Appendix 2.

RED: M20 normalised

The microstructure consists mainly of light grains of ferrite – the equilibrium crystal structure of pure iron at room temperature, with a little carbon (<0.01%) in solid solution. Most of the carbon (0.2%) is in the dark-etching, two-phase regions of pearlite. These consist of fine-scale alternating layers of ferrite (light phase) and iron carbide, Fe_3C (dark phase) – see the micrographs on the lab posters and later in the handout. Iron carbide is much harder than ferrite, so the pearlite is harder, but it is also less tough. **Annotate the normalised M20 microstructure on the Results pages.**

GREEN: M40 normalised

Comparing this steel with M20, the major difference is the greater proportion of pearlite in the microstructure, due to the increase in carbon content from 0.2% to 0.4% (almost all the carbon is in the iron carbide in the pearlite). Hardness increases and toughness decreases with increasing amounts of pearlite. **Annotate the normalised M40 microstructure on the Results pages**.

BLUE: M40 water quenched from 850°C

The effect of heating M40 steel to 850°C is to change its structure from body-centred cubic (bcc) ferrite and pearlite to single-phase face-centred cubic (fcc) austenite. The austenite contains all the carbon in solid solution (individual C atoms in the interstitial spaces between the iron atoms). On quenching, the austenite is entirely replaced by a distorted bcc structure called martensite, with the carbon remaining in supersaturated solid solution. Martensite appears as light-coloured fine needle-like grains – the carbon in solution is not affected by the etch. The bcc lattice is distorted by the carbon atoms being forced into supersaturated solid solution. This distortion increases the hardness and yield strength, but reduces the toughness to a very low value. **Annotate the M40 microstructures (before and after quenching) on the Results pages.**

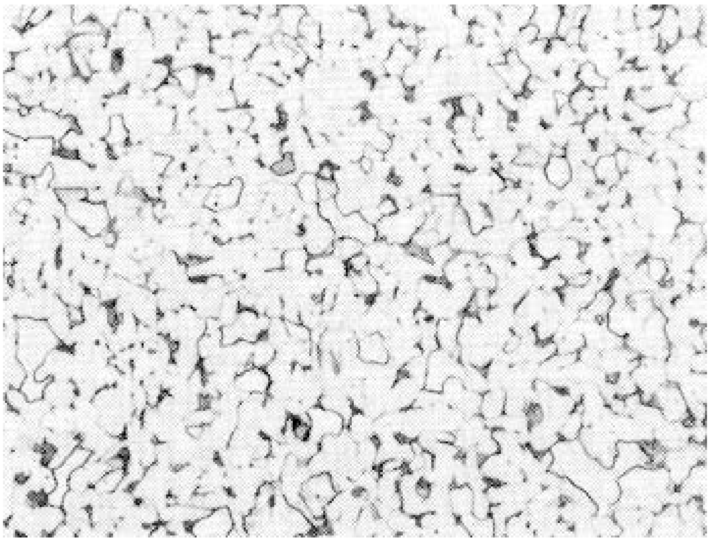
BLACK: M40 water quenched from 850°C and tempered at 500°C

The effect of *tempering* on the appearance of martensite is to change it from its as-quenched light appearance to a much darker structure. The grain structure of the martensite has not changed – the darkening is due to the precipitation of iron carbide within the grains, which the etch can pick out (though individual precipitates are too small to resolve here). Precipitation removes carbon atoms from the supersaturated solid solution, which reverts gradually to normal bcc ferrite. The fine-scale iron carbide precipitates harden the ferrite grains (compared to pure ferrite), but the hardness is lower than that of martensite (but with the toughness being restored). **Annotate the microstructure of tempered M40 on the Results pages.**

4. Discussion of the experimental findings (with the demonstrator)

Complete the property chart (end of Results pages) using your hardness and impact energy data, answer the two questions below the chart, and discuss with the demonstrator.

RESULTS



RED MOUNT: M20
(mag. × 100)

The light phase is:

……………….……….…….….….….…….…

The dark (second) phase region is:

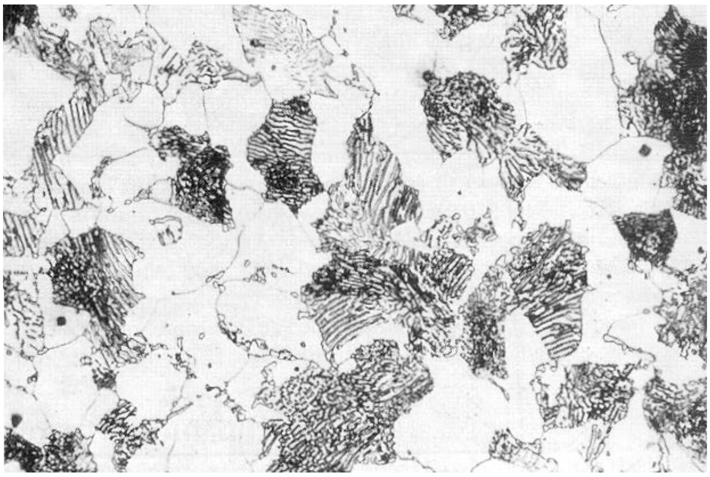
………………….………….….….…….....…

The phases in the dark 2-phase regions (grains) are:

…………………………..….….….….…...

Hardness H (kgf/mm^2): ………………………

Izod impact energy (Joules): ……………………



GREEN MOUNT: M40
(mag. × 200)

(Higher magnification than M20 above)

Are the phases the same as in M20?

……………

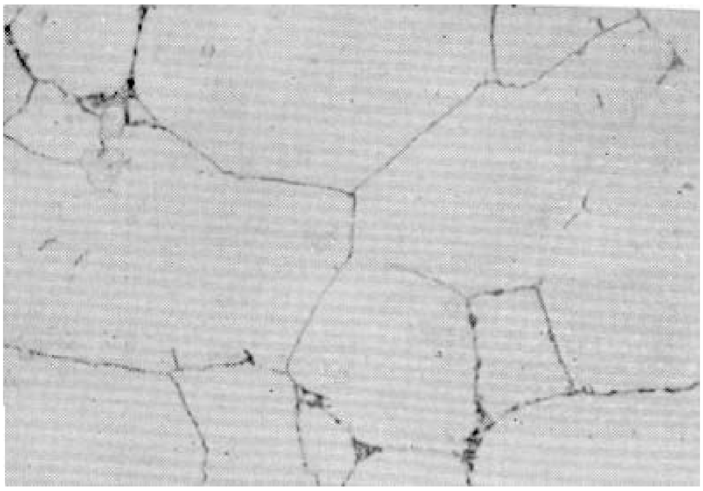
What is the major difference between the microstructures of M20 and M40?

…………………………………………....…

……………………………………………

Hardness H (kgf/mm^2): ………………………

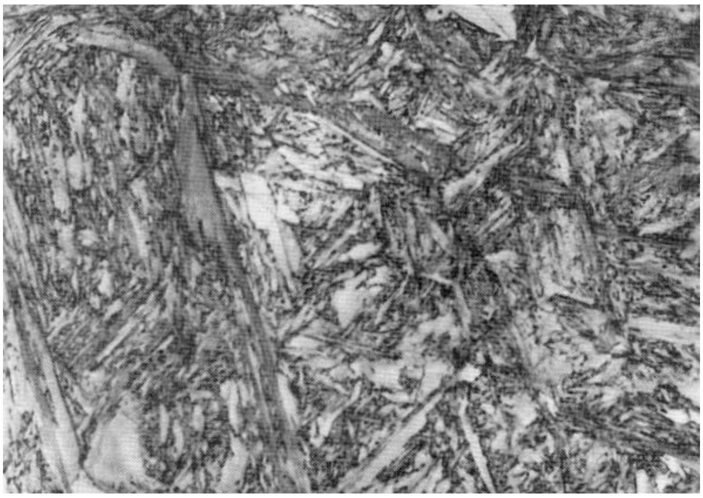
Izod impact energy (Joules): ……………………



M40 heated to 850 ^{o}C (mag. × 400)

The single phase <u>at 850 ^{o}C</u> is:

…………………..………………



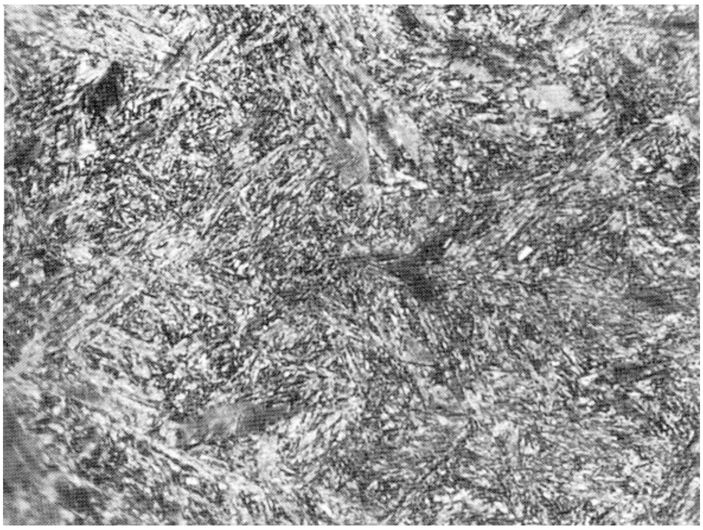
BLUE MOUNT: M40 water quenched from 850 ^{o}C (mag. × 1600)

The single phase needle-like structure consists of fine crystals of

…………………………………………

Hardness H (kgf/mm^2): ………………………

Izod impact energy (Joules): ……………………



BLACK MOUNT: M40 quenched and tempered at 500 ^{o}C (mag. × 800)

Microstructure is the same as quenched M40 above, but the fine crystals appear dark. Why is this?

………………………………

What phase or phases are present?

…………………………………………

Hardness H (kgf/mm^2): ………………………

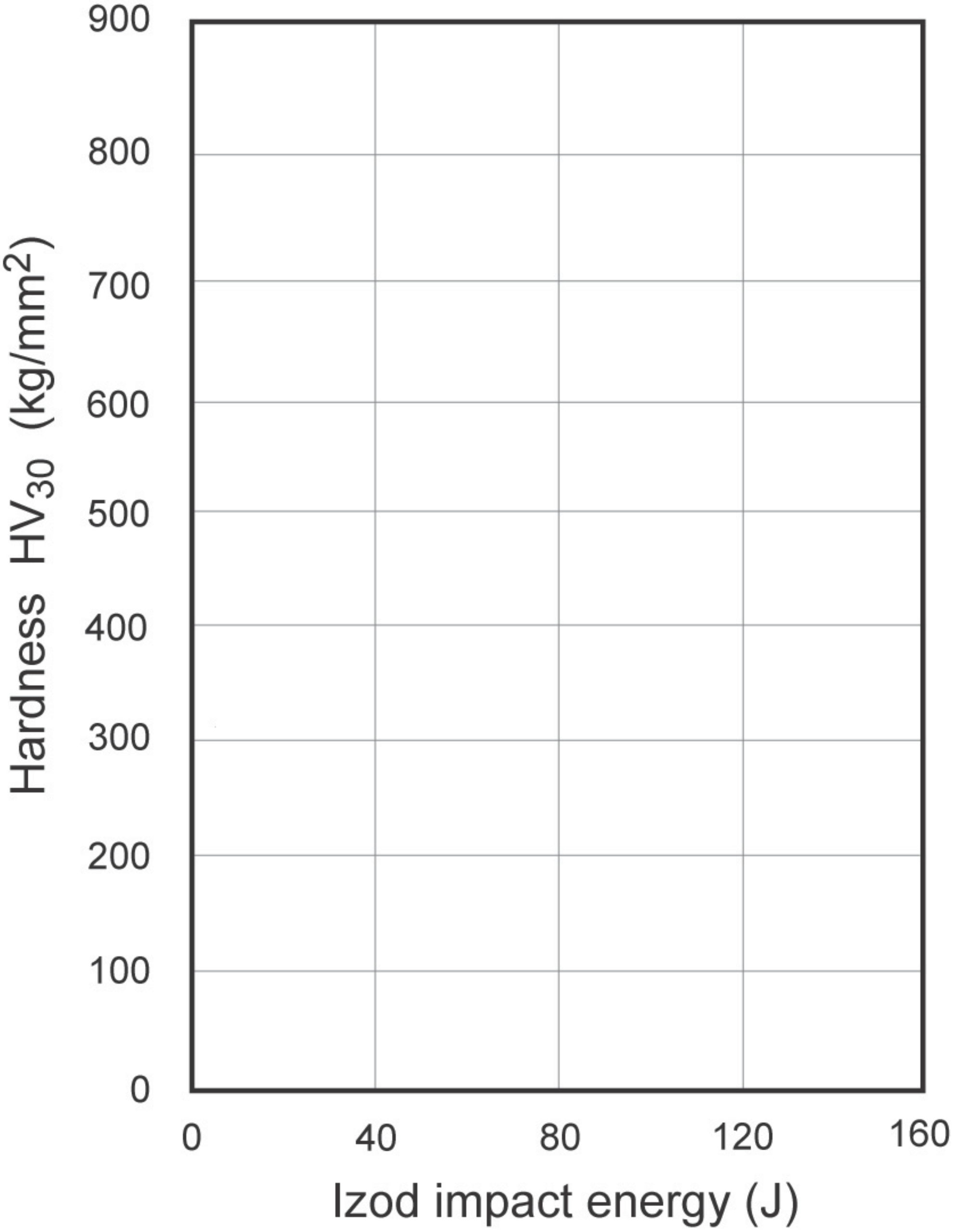
Izod impact energy (Joules): ……………………

PROPERTY CHART

Complete the property chart showing the values of Hardness and Impact energy for the 4 different steels and heat-treatments (note that this is analogous to a yield stress – toughness property chart).

Show each steel/heat treatment as a small “property bubble” for clarity (in practice there will be a spread of values, say ± 5 J for Izod energy, and ± 20 for hardness).

Note the key result: compare the hardness and impact energy of normalised M40 with those of the quenched and tempered M40.



Use the property chart to select a steel and heat-treatment suitable for:

(a) woodworking chisel: ……………………………………………………………………………

(b) teeth of a gear wheel: …………………………………………………………………………

A.E. Markaki / G.J. McShane / H.R. Shercliff, September 2015

Appendix 1: *Explanatory Notes*

1. At 850^{o}C steel has a face-centred cubic (fcc) structure with carbon dissolved in an interstitial solid solution. This microstructure is single phase *austenite*; the heat-treatment is called ‘austenitising’. On quenching, austenite is converted to a new phase, *martensite*, which has a distorted bcc crystal structure, with high hardness (yield stress), but a very low toughness.
2. The *Izod Test* provides empirical data on the comparative toughness of notched metals under impact bending conditions. The fracture energy of a standard notched specimen is measured in the range 0–163 Joules (0–120 ft.lb), often over a range of temperatures. For some service conditions, minimum acceptable Izod values can be specified, e.g. 20–25 Joules would represent the lower limit of usefulness, but for structural applications the limit would be around 60 Joules. The Izod test is most useful for comparing the changes in toughness for different alloys and heat treatments. It does *not* provide true design data on unstable crack propagation – for this purpose a valid fracture test must be used to find values of the fracture toughness, K_{IC}.
3. During *tempering*, the carbon atoms which caused the distortion when in supersaturated solid solution in the iron (quenched martensite) now precipitate out forming the compound iron carbide. In this way martensite converts to undistorted body-centred cubic (bcc) ferrite. The martensite grains become ferrite grains hardened by a fine iron carbide precipitate. These changes restore toughness substantially with a loss in hardness compared to martensite (but a hardness above that of the normalised condition).
4. Normalised M20 and normalised M40 steels consist of soft grains of ferrite, which is almost pure iron with a little carbon (0.01%) in solution, and harder regions of pearlite, which is a layered arrangement of alternating phases, ferrite and iron carbide. It is the iron carbide that gives pearlite its hardness. The amount of pearlite increases linearly with carbon content from 0% at 0.01 wt%C to 100% at about 0.8% carbon. Normalised M40 is therefore significantly harder than M20 and has a lower toughness. Pearlite is an example of a ‘eutectoid’ microstructure (see *Teach Yourself Phase Diagrams*).
5. Normalising consists of heating to between 800^{o}C and 900^{o}C (e.g. to soften the metal after cold working) then cooling in air (much slower cooling rate than quenching in water). This cooling rate leads to the equilibrium phases, and controls the scale of the alternating layers in the pearlite.

Appendix 2: Glossary of phases and microstructures in plain carbon steels (0 1% -0.8% carbon)

Ferrite is a soft and ductile single phase. The iron atoms are arranged in a body-centred cubic (bcc) lattice with a small amount of carbon (<0.01%) in solid solution. The carbon atoms fit in some of the interstitial spaces between the iron atoms. Ferrite is stable at temperatures below 723^{o}C.

Austenite is a high temperature single phases, with all the carbon in solid solution. It is a stable form above 723^{o}C, and at this temperature it is soft and ductile. The iron atoms are arranged in a face-centred cubic (fcc) lattice and the carbon atoms occupy some of the interstitial spaces – much more carbon can dissolve in austenite than in ferrite.

Iron carbide is a single phase intermetallic compound of iron and carbon (Fe_3C) that is stable below 723^{o}C. It is a hard, brittle phase, controlling the hardness and toughness of pearlite and tempered martensite.

Pearlite is a *mixture of two phases*, ferrite and iron carbide, arranged in fine alternating layers which are just resolvable using your microscopes. Pearlite is much harder but not as tough as ferrite. The amount of pearlite increases linearly with the amount of carbon, from zero at 0.01% carbon to 100% at 0.80% carbon by weight. Pearlite is stable up to 723^{o}C (when the iron carbide dissolves).

Martensite is a non-equilibrium (metastable) single phase formed by quenching austenite from above about 800^{o}C. Instead of forming bcc ferrite, a distorted form of this phase is produced which has a pseudo-body-centred tetragonal structure. The distortion is caused by the carbon atoms locked into a supersaturated solid solution. The crystals of martensite are extremely fine and difficult to resolve with your microscope. It is very hard and brittle. Martensite decomposes on tempering.

Tempered martensite is less hard than quenched martensite but much tougher. It is formed by tempering quenched martensite in the temperature range 200^{o}C to 600^{o}C. This allows atoms of carbon to precipitate as iron carbide and the distorted martensite to revert to bcc ferrite – i.e. it contains *two phases*. There is no change in the as-quenched grains during tempering. Iron carbide precipitates on a fine scale within the grains of what is now precipitation hardened ferrite.