

## Part IB Paper 3: MATERIALS

Examples Paper 3 : Materials Processing –  
Controlling Microstructure and Properties

ISSUED ON

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*Straightforward questions are marked †*  
*Tripos standard questions are marked \**

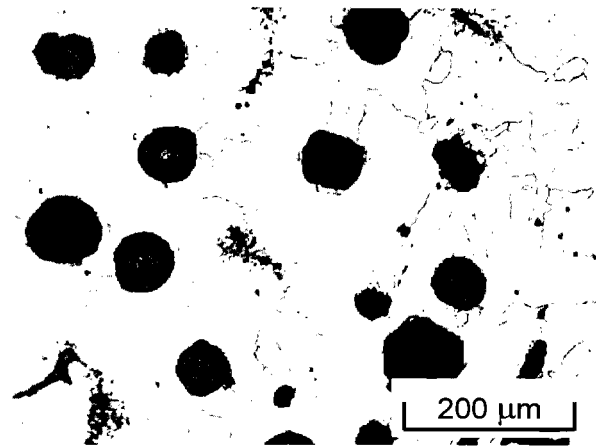
Solidification and Casting

† 1. Figure 1 shows two micrographs of a ferritic cast iron. In the as-cast condition, graphite flakes form in a matrix of ferrite. A small alloying addition, combined with heat treatment after casting, causes spheroidal graphite to form. In tension the as-cast material fails by brittle fracture with a failure strength = 210 MPa, and its fracture toughness = 8 MPa√m. Use this data to estimate the inherent flaw size responsible for brittle failure. What is the likely origin of these flaws. How do you expect the properties of the spheroidal cast iron to compare?

[NB: Refer to IA notes and the Materials Databook for a reminder of the definition of fracture toughness.]



Ferritic cast iron – as-cast



Spheroidal cast iron

Figure 1

2. (a) Question 12 of examples paper 2 analyses the homogenisation of an impurity in a continuous casting. Sketch the grain structure through the cross-section of a large metal casting, showing the shape and size of the grains. By considering *segregation* during solidification (concentration gradients between the first and last solid to form), explain the initial impurity distribution. How can the casting process be modified to minimise the homogenisation stage?

(b) Zinc alloy toy cars are shaped by *die casting*: molten metal is injected at high pressure into a cooled mould. What features of this casting process help improve the strength of the toy by influencing the microstructure?

Deformation and Annealing

† 3. Distinguish between *recovery* and *recrystallisation* in an alloy which has been hot or cold worked. Describe briefly how deformation may be used to control the grain size in metals.

### Heat Treatment of Metal Alloys

4. Samples of an Al-4.5% Cu alloy were heated to 530°C for 1 hour and then quenched in water at 20°C. The yield strength of the samples after this treatment was 230 MPa. The quenched samples were then aged for different times at 145°C. Room temperature yield strengths varied with ageing time as follows:

Time at 145°C (hours)	Yield strength (MPa)
1	250
10	370
10 <sup>2</sup>	420
10 <sup>3</sup>	315
10 <sup>4</sup>	205

(a) Sketch the ageing curve. Summarise the changes in microstructure brought about by the quenching and ageing treatments, and hence explain the shape of the ageing curve.

(b) What characteristics of the Al-Cu phase diagram are being exploited in this heat treatment?

(c) An Al-4.5% Mg was heat treated in the same way. Explain why the yield strength remained unchanged after quenching and ageing, even though the phase diagram is similar for this alloy.

(d) Further samples of Al-4.5% Cu were also held at 530 °C for 1 hour, and then heat treated as follows:

- (i) quenched to 20°C, and aged at that temperature;
- (ii) quenched to 20°C, and then aged at 120°C;
- (iii) slow cooled to 20°C, and aged at 120°C.

Explain in each case how the microstructure evolution will differ from that in the initial heat treatment. Sketch the corresponding evolution of yield strength with ageing time, including the curve sketched in (a).

[The Al-Cu phase diagram is given in the Materials Data Book.]

5. (a) Refer to the TTT diagram for BS503M40 steel in the Materials Databook. Note that this steel is *hypo-eutectoid* – the carbon concentration is below the eutectoid value. Identify the carbide line on the TTT diagram, and describe what it represents. Samples of this steel are austenitised at 845°C and then subjected to the following heat treatments. Identify the microstructures produced.

- (i) quench to room temperature in less than 1 second;
- (ii) quench to 600°C, hold for 2 minutes, quench to room temperature;
- (iii) quench to 600°C, hold for 20 seconds, quench to room temperature;
- (iv) quench to 400°C, hold for 2 minutes, quench to room temperature;
- (v) quench to 400°C, hold for 20 seconds, quench to room temperature.

(b) Use the TTT diagram for BS817M40 steel (also hypo-eutectoid) in the Materials Databook to select a quench-hold-quench heat treatment which produces the following micro- structures, in the shortest times:

- (i) 40% ferrite, 60% pearlite

- (ii) 10% ferrite, 40% pearlite, 50% martensite
- (iii) 50% bainite, 50% martensite

6. (a) Define what is meant by *hardenability*. "Hardenability" and "weldability" in carbon and low alloy steels are considered to be opposites. Why? Describe briefly the effect of alloying on the "hardenability" and "weldability" of steels.

A steel shaft 40 mm in diameter is to be hardened by austenitising followed by quenching into cold oil. The centre of the bar must be 100% martensite. The table below gives the cooling rate at the centre of an oil quenched bar as a function of bar diameter.

Bar diameter (mm)	Cooling rate (K s <sup>-1</sup> )
500	0.17
100	2.5
20	50
5	667

It is proposed to make the shaft from a Ni-Cr-Mo low-alloy steel. The critical cooling rates (CCR) of Ni-Cr-Mo steels for formation of martensite are given quite well by the empirical equation

$$\log_{10} (\text{CCR in K s}^{-1}) = 4.3 - 3.27 C - \frac{(\text{Mn} + \text{Cr} + \text{Mo} + \text{Ni})}{1.6}$$

where the symbol given for each element denotes its weight percentage.

(b) Plot a suitable graph to find the cooling rate at the centre of the given bar. Then select the best steel for this application from those listed in the table below, justifying your choice.

(c) In practical heat treatment, why should the cooling rate be kept as slow as possible while achieving the required microstructure?

Steel	Weight percentages					Cost (£/kg)
	C	Mn	Cr	Mo	Ni	
A	0.30	0.80	0.50	0.20	0.55	0.50
B	0.40	0.60	1.20	0.30	1.50	0.90
C	0.36	0.70	1.50	0.25	1.50	1.25
D	0.41	0.85	0.50	0.25	0.55	0.60
E	0.40	0.60	0.65	0.55	2.55	1.00

### Diffusion in Materials Processing

7. A thick steel plate is taken from a furnace at an initial uniform high temperature  $T_1$ . The plate is plunged into a water bath at room temperature  $T_0$ , in order to form martensite. The bath is large and heat transfer is efficient, so it may be assumed that the surface temperature instantaneously changes to  $T = T_0$  and remains constant for all times  $t > 0$ .

(a) (i) On axes of temperature ( $T$ ) against depth into the plate ( $x$ ), sketch the temperature profile of the plate and water bath at time  $t = 0$ . Sketch how you would expect this profile to

evolve with time during the early stages of cooling, when a thick plate can be regarded as semi-infinite.

(ii) The temperature below the surface  $T(x, t)$  is governed by the (one-dimensional) heat flow equation:

$$\frac{\partial T}{\partial t} = a \frac{\partial^2 T}{\partial x^2}$$

where  $a$  is the thermal diffusivity of the steel. By considering the boundary conditions in this quenching problem, *write down* the solution for  $T(x, t)$  in the early stages of cooling in terms of the error function.

(b) For  $y < 0.7$ , to a good approximation:  $\text{erf}(y) = y$ . Use this approximation to derive a simple expression for the depth at which the temperature  $T$  reaches a value halfway between  $T_1$  and  $T_0$  in a time  $t$ .

(c) Summarise the analogy between transient heat flow and diffusion, and identify a corresponding expression for a characteristic diffusion distance in time  $t$ , defined as for heat flow in (b).

The thermal diffusivity of steel at  $1000^\circ\text{C}$  is  $9 \times 10^{-6} \text{ m}^2\text{s}^{-1}$ . Use the data below to find the diffusion coefficient  $D$  for carbon at this temperature. Hence estimate how much faster heat "diffuses" a distance  $x$  in steel compared to the diffusion of carbon at  $1000^\circ\text{C}$ .

$D$  is the diffusion coefficient,  $D = D_0 \exp(-Q/RT)$ . For carbon diffusion in austenite:

$D_0 = 9 \times 10^{-6} \text{ m}^2\text{s}^{-1}$ , and the activation energy  $Q = 125 \text{ kJ/mol}$ .  $R$  is the universal gas constant and  $T$  is the absolute temperature.

8. A carbon steel component containing 0.2 wt% C is to be case-carburised at a temperature of  $1000^\circ\text{C}$ . Use the Fe-C phase diagram to explain why the maximum concentration of carbon at the surface is 1.6 wt%.

Carbon diffusion into the component follows the governing 1-D partial differential equation (Fick's 2<sup>nd</sup> Law):

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2}$$

where  $C(x, t)$  is the concentration at time  $t$  at a depth  $x$  below the surface. The diffusion coefficient  $D$  and data for carbon diffusion in austenite are defined in Q7.

The boundary conditions are:  $C(x, 0) = C_0$  and  $C(0, t) = C_s$ , where  $C_0$  is the initial (uniform) carbon content of the steel, and  $C_s$  is the constant surface carbon concentration. By analogy with the heat flow analysis in Q7, show that the solution for the concentration is:

$$C(x, t) = (C_s - C_0) \left\{ 1 - \text{erf} \left( \frac{x}{2\sqrt{Dt}} \right) \right\} + C_0$$

Sketch the variation of carbon concentration  $C$  against dimensionless distance below the surface  $x / 2\sqrt{Dt}$ . Hence find the time required for carburisation to a case depth of 0.3mm, if the depth of the case is taken to be the value of  $x$  for which  $C = 0.5 \text{ wt \% carbon}$ .

[Definition and values for the "error function"  $\text{erf}(y)$  are in the Materials Databook].

- \* 9. State the boundary conditions for Fick's Second Law which apply to 'pre-deposition' diffusion and 'drive-in' diffusion in semiconductor processing. For what purpose would pre-deposition be followed by a drive-in stage?

In the fabrication of a silicon semiconductor device, boron is diffused into silicon which is uniformly doped with phosphorus at a concentration of  $10^{17}$  atoms  $\text{cm}^{-3}$ . The pre-deposition stage lasts for 10 minutes at a temperature of  $1000^\circ\text{C}$ , with the concentration of boron at the surface maintained at  $10^{20}$  atoms  $\text{cm}^{-3}$ .

This is followed by drive-in diffusion for one hour at  $1100^\circ\text{C}$ .

Calculate the depth at which the concentration of boron atoms is equal to that of phosphorus atoms (i.e. the  $p$ - $n$  junction depth).

[For the diffusion of boron in silicon,  $D_0 = 0.037 \text{ cm}^2 \text{ s}^{-1}$ , and the activation energy  $E = 3.46 \text{ eV}$ . You will need to use the expressions for the integral of the error function solution and for the drive-in solution from the lecture handout.]

### Polymer Processing

10. For the following polymer products, outline how microstructure control can improve the properties. In each case nominate a suitable shaping process, and describe how the required microstructure is achieved.

- (a) climbing rope
- (b) supermarket carrier bag
- (c) car headlamp cover

### Materials Processing – synoptic questions

11. For each of the following processes, identify: (a) the microstructural changes occurring; (b) whether a phase transformation is taking place; (c) the driving force; (d) details of any diffusion of atoms. [It is recommended that you present your answers as a table].

- (i) solidification of a Pb-Sn alloy of eutectic composition;
- (ii) recrystallisation of Al-Mn-Mg alloy after hot rolling;
- (iii) cooling a medium carbon steel from  $850^\circ\text{C}$  to  $20^\circ\text{C}$  at a cooling rate above the critical cooling rate;
- (iv) tempering of a carbon steel at  $500^\circ\text{C}$ , immediately after re-heating to that temperature;
- (v) tempering of a carbon steel at  $500^\circ\text{C}$ , during a prolonged hold at temperature.

12. Summarise some of the major reasons for alloying metals. Explain briefly the role of the following elements in the alloys given:

- (a) Mg and Si in wrought heat-treatable aluminium alloy
- (b) Si in A356 aluminium casting alloy
- (c) Ni in low alloy steels
- (d) W in tool steels

### Answers

6. Steel B.

7. (a)  $T(x,t) = T_0 + (T_1 - T_0) \operatorname{erf}\left(\frac{x}{2\sqrt{at}}\right)$

(b)  $x = \sqrt{at}$

(c)  $x = \sqrt{Dt}$ ;  $\approx 10^5$  times faster

8. 7.2 minutes.

9. 0.22  $\mu\text{m}$ .

### Relevant IB Tripos Questions from previous years

2004 Q1, 5; 2005 Q1, 2(a-c), 4(b); 2006 Q2, 3, 6; 2007 Q4, 5; 2008 Q1, 2; 2009 Q2(d), 4, 5, 6, 2010 Q2, 5; 2011 Q3, 4, 6; 2012 Q3, 6

[NB: Until 2007, the course contained more detail on processing of polymers, semiconductors and ceramics, and also covered glasses. Past Tripos questions on these topics usually ask for more detail than would now be expected, and some of the content is no longer covered.]

GJ McShane  
HR Shercliff  
MPF Sutcliffe  
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