DR HR SHERCLIFF

ENGINEERING TRIPOS PART IB **JUNE 2005**
PAPER 3: MATERIALS

1. (a) Purpose of the Jominy end-quench test: to quantify the hardenability of a steel (i.e. the ability of the steel to form martensite during quenching).
Procedure:

- austenitise a cylindrical bar of the steel is austenitised;
- suspend vertically and quenched on its end with a jet of water – this gives a progressive fall in cooling rate with distance along the bar from the quenched end.
- measure and plot the hardness along the bar; examine microstructure for extent of martensite formation.

Martensite has high hardness, so higher hardenability steels show high hardness to larger Jominy distances (i.e. with lower cooling rates). Hardenability may also be defined by the distance over which a certain fraction of martensite (usually 50%) is exceeded.

(b) (i) $$T(t) = T_0 + (T_1 - T_0)\frac{4}{\pi}\left[\exp\left(-\frac{\pi^2 a t}{4l^2}\right)\right]$$

$$\frac{dT}{dt} = (T_1 - T_0)\frac{4}{\pi}\left[\exp\left(-\frac{\pi^2 a t}{4l^2}\right)\right]\left(-\frac{\pi^2 a}{4l^2}\right)$$

(Note: keeping the factor $4/\pi$ out, rather than combining with the last term, simplifies the following substitution; note also that $(T_1 - T_0) \neq A$).

Substitute back from original solution:

$$\frac{dT}{dt} = (T_1 - T_0)\left(\frac{T - T_0}{T_1 - T_0}\right)\left(-\frac{\pi^2 a}{4l^2}\right) = (T - T_0)\left(-\frac{\pi^2 a}{4l^2}\right) = -\frac{A\pi^2 a}{4l^2}$$

(ii) Reference temperature $T = (T_1 + T_0)/2$ and thus $B = 1/2$

Cooling rate in Jominy bar is

$$\frac{dT}{dt} \approx -\frac{AB^2}{\sqrt{\pi}}\left(\frac{4a}{x^2}\right)\exp\left(-B^2\right) = -\frac{A(1/4)}{\sqrt{\pi}}\left(\frac{4a}{x^2}\right)\exp\left(-1/4\right) = -\left(\frac{Aa}{\sqrt{\pi}\,x^2}\right)\exp\left(-1/4\right)$$

Equate the cooling rates for slab and Jominy bar:

$$\frac{A\pi^2 a}{4l^2} = \left(\frac{Aa}{\sqrt{\pi}\,x^2}\right)\exp\left(-1/4\right), \quad \text{hence } x^2 = \left(\frac{4l^2}{\pi^{5/2}}\right)\exp\left(-1/4\right)$$

For slab of thickness $2l = 24$ mm, $l = 12$ mm

Hence $$x^2 = \left(\frac{4(12)^2}{\pi^{5/2}}\right)\exp\left(-1/4\right) \Rightarrow x = 5.064\,\text{mm}$$

Alternatively, can substitute in $l = 12$ mm and $x = 5.1$ mm to show that both solutions give

$$\frac{dT}{dt} \approx -0.017\,Aa$$

(iii) From Jominy curves in databook, for $x = 5.1$mm (corresponding to centre of slab):
As quenched hardnesses: BS503M40: 540 kgf/mm^2; BS817M40: 600 kgf/mm^2
For tempered hardness of 300 kgf/mm^2:
BS503M40: temper at 575°C; BS817M40: temper at 650°C

(iv) On tempering: carbon precipitates out of supersaturated solid solution (i.e. the martensite) to form iron carbide (or alloy carbides in low alloy steels). The surrounding lattice reverts to ferrite. The precipitation hardening of the carbides is effective, but gives a lower hardness than the martensite (while simultaneously restoring useful toughness). Martensite is not acceptable in a weld due to its brittleness. Hardenable steels are therefore less weldable, since they are more likely to form martensite when subjected to the cooling rate produced by the welding process. Hence BS503M40 is the more weldable.

Examiner's comments

(a) "Hardenability" was not mentioned by some candidates, or "hardness" used instead (incorrectly).
(b) The temperature halfway between T_1 and T_0 was often taken as $(T_1 - T_0)/2$ rather than $(T_1 + T_0)/2$. Many used 12mm for the Jominy distance when reading the curves in the Databook, thereby missing the whole point of the analysis, which is to find the equivalent Jominy distance for the same cooling rate as takes place in the slab. This was even done in (iii) by candidates who had correctly done the analysis in (ii). In the last part, some candidates described other microstructural "changes", e.g. with position along the Jominy bar).

2. (a) Typical heat treatment schedule for Al-Mg-Si (6000 series) alloy:

- solution heat treatment in single phase region of phase diagram (approx. 500-550°C):
  alloying elements dissolve completely;
- quench to room temperature:
  retain alloying elements in supersaturated solid solution;
- artificially age at intermediate temperature (approx. 180-200°C):
  precipitation of hardening phases, depleting the remaining matrix of excess solute (initially GP zones, evolving towards equilibrium Mg_2Si via other metastable phases such as β");
  during ageing, hardening precipitates progressively coarsen and lose crystal coherency at the interface between precipitate and matrix – this causes the yield strength to rise to an ageing peak.

Forging is done at 300°C to reduce the forging loads by reducing the yield stress and the tendency for work hardening. The first stage of heat treatment requires full dissolution above the solvus boundary – typically 500°C – so the forging temperature is too low.

(b) Non-heat-treatable alloys must use solid solution hardening and work hardening. No ageing treatment is applied after forging, as these alloys do not produce the necessary metastable hardening precipitates. To achieve comparable strength to the heat-treated case, work hardening must be exploited (as solid solution hardening is relatively weak). Hence forging will be conducted at a lower temperature than 300°C (possibly room temperature).

(c) (i) Arc welding subjects the component to temperatures up to the melting point, which leads to significant softening in the surrounding solid heat-affected zone (HAZ):

Heat-treatable alloys: re-solution and over-ageing of hardening precipitates (with some post-weld strength recovery by natural ageing at room temperature);

Non-heat-treatable alloys: recrystallisation of the work hardened microstructure, with no subsequent strength recovery.

(ii) Prolonged exposure to 100°C:

Heat-treatable alloys: slow over-ageing of hardening precipitates, giving progressive strength loss;

Non-heat-treatable alloys: recrystallisation unlikely at such a modest temperature, so work hardening should be retained, as should solid solution strength (as difficult to form precipitates in these alloys).

(d) From process attribute charts in Databook:
Material compatibility – no restriction on shaping process for Al alloys
Mass = 0.05 kg: bit small for forging, so may be expensive or difficult
Min. thickness = 6 mm: no problem
Tolerance <= 0.1 mm: would require machining after forging
Economic batch size = 50,000: beyond normal economic range, so may be expensive.

Forging is viable (with machining for tolerance) but is likely to be expensive. Consider alternative shaping processes:
Mass: machining, powder methods, investment casting OK, die casting close, so assume OK
Thickness: all processes OK
Tolerance: machining, investment casting OK, others require machining
Economic batch size: powder methods, die casting

So powder methods or die casting, followed by machining, are both viable. Investment casting and machining very unlikely to be economic. Die casting is the more routine process for metals than powder processing, so likely to choose this.

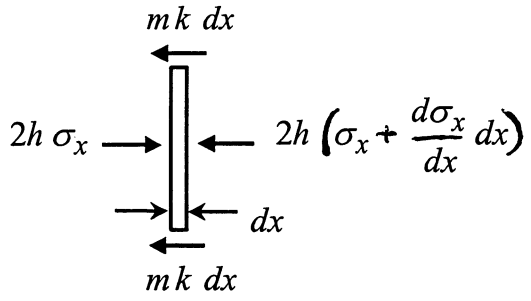
Changing to die casting would require changing to cast Al alloy instead of a wrought alloy. Cast Al microstructures generally have coarser grain structures with eutectic regions containing Si, and some porosity. Toughness generally lower, but strength could be comparable if a heat-treatable casting alloy is chosen.
(For powder, some porosity would also result, but properties as good as wrought alloy).

Examiner's comments

(a,b,c) Many had not understood that the "solution" prior to quenching is solid, and talked about melting then quenching. Descriptions of ageing were too detailed (i.e. reproducing previous Tripos questions, not the brief summary asked for in this question, worth only a few marks). Some poor use of technical terminology, e.g. using "grains" and "precipitates" as more or less inter-changeable. The distinction between "heat-treatable" and "non-heat-treatable" had clearly passed by many candidates.
(d) Candidates mostly showed encouragingly good judgement, and correctly allowed some leeway with the Databook charts (noting that processes beyond the range might be more expensive, but still possible). Many opted to stretch the economic batch size rather than a technical attribute, and chose machining or investment casting (which were accepted). Sheet forming was often given as a good alternative to forging without comment on the differences in component geometry. Many were clearly unable to accurately locate values on the log scales (e.g. thought 50,000 was less than 10^4). A few interesting (?) process variants were proposed: "strain the casting", "machine then cast", "work harden after shaping".

3. (a) Benefits of a process model for forging (any four of the following acceptable):
- calculation of forging loads
- sensitivity to process variables (e.g. billet temperature, friction) and alloy composition
- low cost prototyping, without interrupting production
- process understanding, by visualisation of deformation and temperature distributions
- relationship with microstructure and mechanical properties
- improved die design to avoid defects
- input to failure analysis
- assist development of real-time control systems

(b) (i) For unit depth normal to the cross-section, horizontal forces on element:



Resolving horizontally, taking compressive stresses as positive:

$$2h\,\frac{\mathrm{d}\sigma_x}{\mathrm{d}x} + 2mk\,dx = 0, \quad \Rightarrow \quad \frac{\mathrm{d}\sigma_x}{\mathrm{d}x} = -\frac{mk}{h}$$

(ii) Assuming the shear stress is sufficiently small compared to the vertical stress $p(x)$ and the horizontal stress σ_x, then these may be treated as principal stresses. In plane strain the out-of-plane stress is between the other two, so $p(x)$ and σ_x are related by the Tresca yield criterion. Also, assume σ_y is constant (no work hardening at high temperature).

$$p(x) - \sigma_x = \sigma_y, \quad \text{so} \quad \frac{dp}{dx} = \frac{d\sigma_x}{dx} = -\frac{mk}{h}$$

Integrating with boundary condition: $x = w$, $\sigma_x = 0$ and thus $p(w) = \sigma_y$ and substituting $k = \sigma_y / 2$:

$$p(x) = \sigma_y\left(1 + \frac{m}{2}\left(\frac{w-x}{h}\right)\right)$$

(iii) Pressure varies linearly: at $x = w$, $p(w) = \sigma_y$ and at $x = 0$, $p(0) = \sigma_y\left(1+\frac{mw}{2h}\right)$

Hence average pressure $p_{ave} = \sigma_y\left(1+\frac{mw}{4h}\right)$

Total load per unit length of billet, P = width × average pressure = $\sigma_y\, w\left(1+\frac{mw}{4h}\right)$

(Can also find the same result by integrating the pressure distribution from 0 to w).

The transverse restraining force is found from the horizontal stress σ_x at $x = 0$. From the Tresca yield criterion, $p(0) - \sigma_x(0) = \sigma_y$, hence $\sigma_x(0) = \sigma_y \dfrac{m\,w}{2h}$.

Hence the transverse restraining force per unit length is $F = 2h \times \sigma_y \dfrac{m\,w}{2h} = \sigma_y\, m\, w$

(Alternative method: horizontal equilibrium for whole billet: $F = 2w \times m\,k = \sigma_y\, m\, w$).

Examiner's comments

(a) Several candidates described the benefits of forging itself, not of *modelling* forging.
(b) Analysis well done, with most recognising that the problem was simply half of the open-die forging in the lectures and examples paper. Many didn't comment on neglecting the shear stress in applying Tresca. A significant number didn't recognise that F was found from the stress at $x = 0$ and tried another integration.

4. (a) (i) Distillation of $SiHCl_3$ and reaction with hydrogen is used to produce electronic grade Si (from SiO_2) with a resulting impurity level less than 1 in 10^{11}.
(ii) Czochralski solidification is the process of forming a large single crystal of Si from a seed crystal withdrawn very slowly from a bath of molten Si.
(iii) Oxidation if Si is used to provide insulation between different conducting or semiconducting regions, or as part of a device (e.g. gate region in a MOS transistor).

(b) (i) Pre-deposition is the process of diffusing dopant into a thin surface region of the silicon, by maintaining a concentration of the dopant at the surface at high temperature. In the drive-in stage, the source of dopant is removed, and the dopant diffuses a much greater distance into the silicon, to give a low, approximately uniform concentration.

(ii) Diffusion constant: $D = D_o \exp(-Q/RT)$

Characteristic diffusion distance: $x = \sqrt{Dt}$

Pre-deposition diffusion distance $x_1 = \sqrt{D_1 t_1}$ and drive-in distance $x_2 = \sqrt{D_2 t_2}$

$$\left(\frac{x_2}{x_1}\right)^2 = (10)^2 = \frac{D_2 t_2}{D_1 t_1} = \frac{D_o \exp(-Q/RT_2)}{D_o \exp(-Q/RT_1)}\left(\frac{t_2}{t_1}\right) = \exp\left[\left(-\frac{Q}{R}\right)\left(\frac{1}{T_2} - \frac{1}{T_1}\right)\right] \times \left(\frac{t_2}{t_1}\right)$$

$$\text{Hence } (10)^2 = \exp\left[\left(-\frac{333000}{8.314}\right)\left(\frac{1}{T_2} - \frac{1}{(1273)}\right)\right] \times \left(\frac{60}{10}\right) \quad \Rightarrow T_2 = 1398\,K = 1125\,^oC$$

(Can also find value of D for 1000°C, then use time and distance ratios to find D for drive-in temperature, and hence the temperature).

(c) $y^2 + Ay = Bt$ with A = 0.09 μm, B = 7.5×10^{-18} $m^2\,s^{-1}$ = 7.5×10^{-6} $\mu m^2\,s^{-1}$ (at 1100 °C).

(i) $y = 150\,nm = 0.15\,\mu m$

Hence $t = \dfrac{(0.15)^2 + 0.09\times0.15}{7.5\times10^{-6}} = 4800\,s = 80\,\text{min}\,utes$

(ii) $t = 90\,\text{min} = 5400\,s$

$y^2 + 0.09y = 7.5\times10^{-6}\times5400$

$y^2 + 0.09y - 0.0405 = 0$

Hence $y = \dfrac{-0.09 + \sqrt{0.09^2 + 4\times0.0405}}{2} = 0.161\,\mu m = 161\,nm$

(iii) Time to form first half of 161nm:

$t = \dfrac{(0.161/2)^2 + 0.09\times0.161/2}{7.5\times10^{-6}} = 1830\,s = 30.5\,\text{min}\,utes$

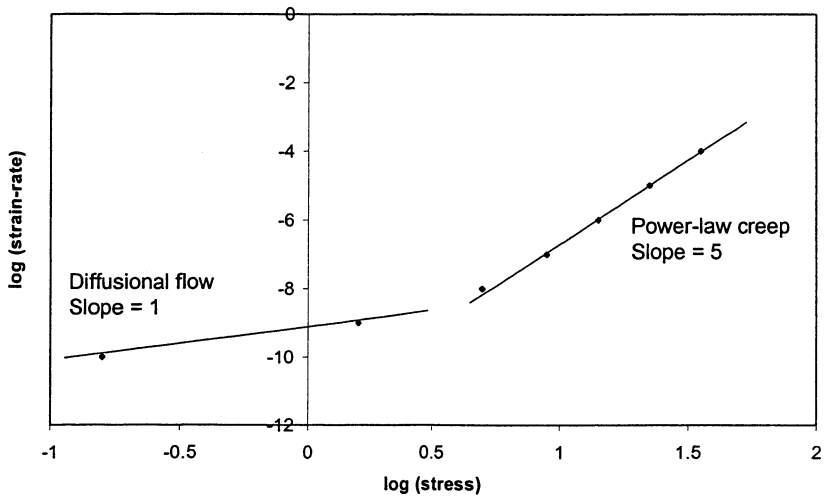
Hence time to form second half = 90 – 30.5 = 59.5 minutes
Ratio = 30.5/59.5 = 0.513.

Examiner's comments

No particular problems with routine process descriptions and straightforward, familiar calculations.

5 (a) Power law creep involves the climb of dislocations past obstacles such as precipitates. The atoms on the obstructed part of the dislocation diffuse away along the dislocation core and through the bulk. Core diffusion dominates at lower temperatures, as this is the easier path (lower activation energy); at higher temperatures atoms can overcome the higher activation energy for bulk diffusion, and this mechanism dominates.
Diffusion creep involves the transfer of atoms from grain boundaries parallel to an applied tensile stress to grain boundaries perpendicular to the stress, changing the grain shape and giving progressive strain. Diffusion is along the grain boundaries or through the bulk – again the easier path of lower activation energy (grain boundaries) dominates at lower temperatures, and bulk diffusion at higher temperatures.

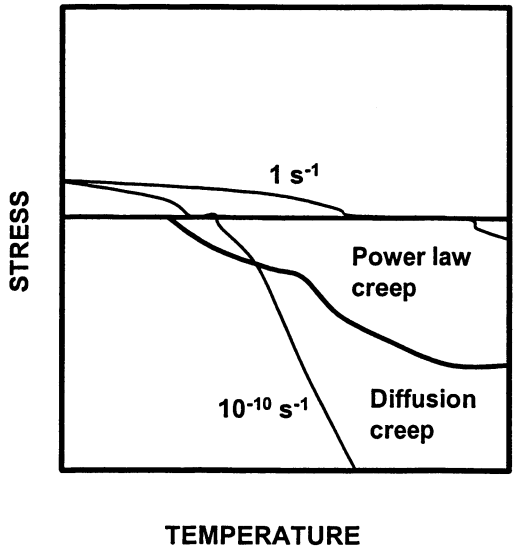
(b) For vertical line through 800°C, estimate log(stress) for each strain-rate contour, then plot log(strain-rate) against log(stress). From $\dot{\varepsilon} = A\sigma^n \exp(-Q/RT)$, at constant T, $\dot{\varepsilon} = B\sigma^n$, so slope is n.



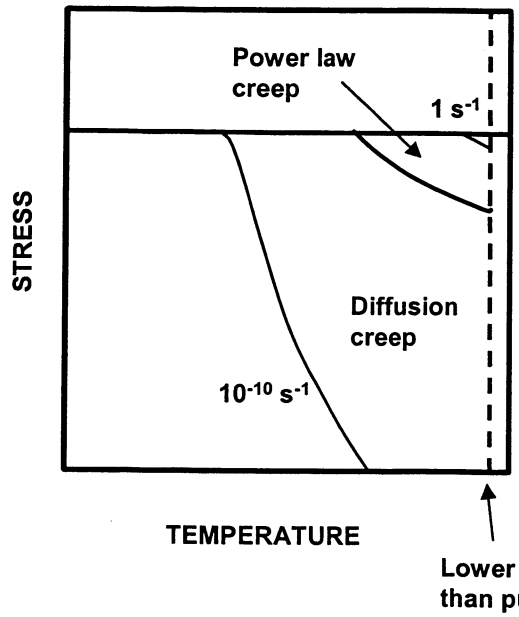
From $\dot{\varepsilon} = A\sigma^n \exp(-Q/RT)$, at constant stress: $\dot{\varepsilon} = C\exp(-Q/RT)$
Taking natural logs: $\ln \dot{\varepsilon} = \ln C - Q/RT$
Along a horizontal line (constant stress), read off temperature (and convert to Kelvin) for each strain-rate contour. Plot $\ln \dot{\varepsilon}$ against $1/T$ (Kelvin^{-1}), slope is $-Q/R$.

(c) Stress = 2 MPa, T = 600°C lies in the diffusion creep regime; operating regime is in the power-law creep regime. Different mechanism – extrapolation of low stress/low temperature data will *under-estimate* actual strain-rate, so this is not safe in design. (e.g. see graph above – extrapolation of diffusional creep line to higher stress falls well below power-law data).

(d) (i) Pure Ni, grain size = 0.1mm (as in question)



(ii) MAR-M200 with same grain size (0.1mm)

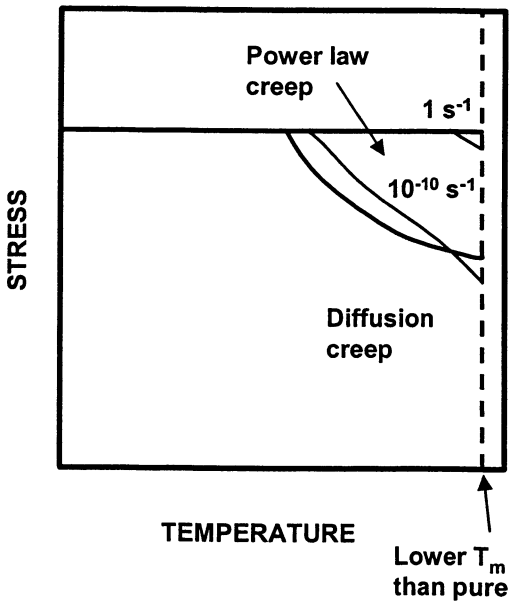


Note: main effect of alloying is to introduce precipitates, and greatly reduce the power-law creep rate.
High strain-rates and PLC regime move to much higher stresses.

Diffusion creep is not affected – so very low strain-rates essentially same as for pure Ni.

(There is a slight reduction in melting point for the alloy compared to pure Ni – not expected in exam answer).

(iii) MAR-M200 with 1000 times greater grain size.



Note: main effect of increasing grain size is to greatly reduce the diffusion creep rate.
Low strain-rates move to much higher stresses (and now fall in PLC regime).

Power law creep is not affected – so very high strain-rates unchanged.

Examiner's comments

This question depended largely on a good understanding of deformation mechanism maps, and the ability to interpret them. The main shortcoming was to explain how the form of the map changes with grain size.

6. (a) (i) θ in Al-Cu is at approx. 54 wt% Cu

Atomic weights: Al = 26.98, Cu = 63.54

Moles Al = 46/26.98; Moles Cu = 54/63.54

Ratio, moles Cu/moles Al = (54/63.54)/ (46/26.98) = 0.498 (approx. 0.5) ∴ $CuAl_2$

(ii) ε in Cu-Sn is at approx. 38 wt% Sn

Atomic weights: Cu = 63.54, Sn = 118.69

Moles Cu = 62/63.54; Moles Sn = 38/118.69

Ratio, moles Sn/moles Cu = (38/118.69)/ (62/63.54) = 0.328 (approx. 0.333) ∴ $SnCu_3$

(iii) Mullite in Silica-Alumina is at approx. 72 wt% Al_2O_3

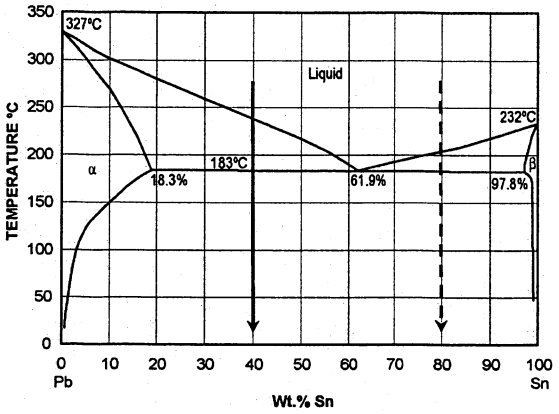
Molecular wts: Al_2O_3 = (26.98x2) + (16.00x3) = 101.96, SiO_2 = 28.09 + (16.00x2) = 60.09

Moles SiO_2 = 28/60.09; Moles Al_2O_3 = 72/101.96

Ratio, moles Al_2O_3/moles SiO_2 = (72/101.96)/ (28/60.09) = 1.52 (approx. 1.5)

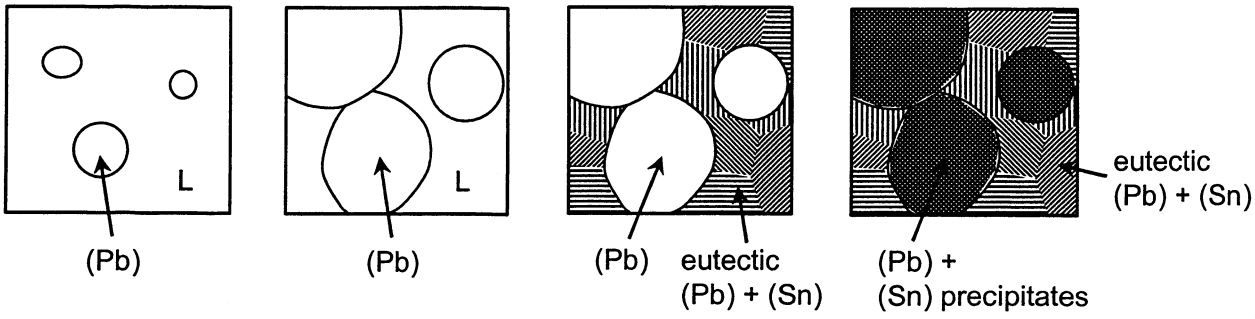
∴ $(Al_2O_3)_3 (SiO_2)_2$

(b)



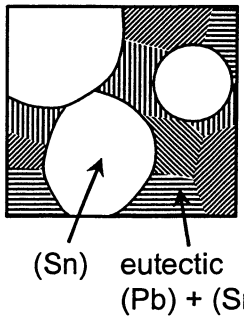
Microstructure evolution on cooling, for 40 wt% Sn:

- solidification starts at 235 °C, with nucleation of primary Pb-rich grains.
- grains grow on cooling until just above the eutectic temperature (183 °C) structure is roughly 50% liquid (eutectic composition 61.9% Sn), 50% Pb-rich solid (composition 18.3% Sn)
- on cooling the remaining liquid forms a eutectic structure of Pb-rich solid and Sn-rich solid (rough proportion 50:50, from tie-line between 18.3, 61.9 and 97.8 wt% Sn)
- on further cooling, Pb-rich solid becomes supersaturated, as equilibrium composition falls; in the primary Pb-rich grains, precipitation of fine-scale Sn-rich solid occurs; in the eutectic region, the proportions and compositions of the phases adjust slightly by inter-diffusion to retain equilibrium (so no real change in appearance).

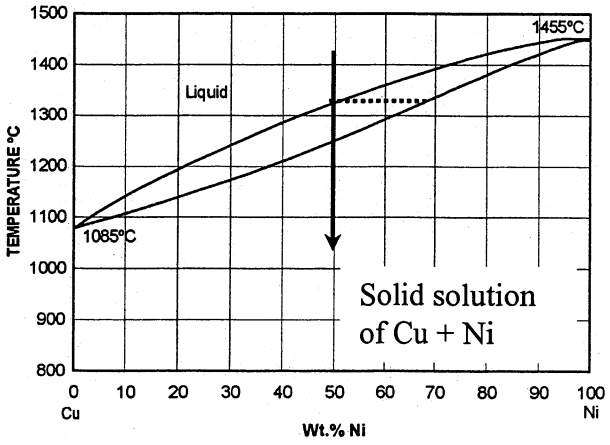


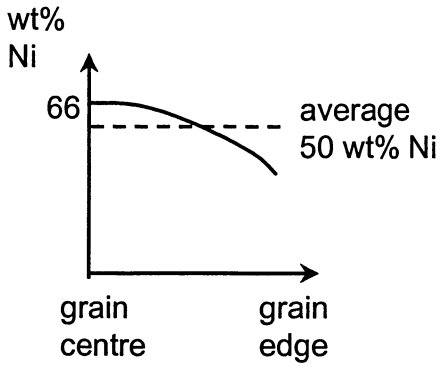
For 80 wt% Sn:
- first solid to form is Sn-rich solid
- at eutetectic, proportions of liquid to eutectic similar, and eutectic structure identical
- on further cooling, as Sn-rich solid contains little Pb and there is little change in solubility with temperature, there will be no precipitation within the Sn-rich grains.

Final structure:



(c)





On solidification, first solid to form is solid solution containing 66% Ni (from tie-line). As solid grows, Ni content of liquid falls, and Ni content of solid forming also falls.

For slow cooling, composition of solid has time to adjust to uniform equilibrium composition, eventually giving 50% Ni solid solution.

As cooling is moderately fast, the concentration gradient across each grain is retained – this is called "segregation".

The average composition must be the composition of the alloy, 50 wt% Ni.

Examiner's comments

The most popular question, attempted by 90% of candidates (the remaining 5 problems all being attempted by about 60% of candidates). In spite of this, candidates lacked the skill to make good sketches of microstructures, and explanations of how these structures came about were vague. Determination of the chemical formulae of compounds in most cases scored highly.

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