

Practical 2P4

Casting

Safety

<i>Hazards</i>	<i>Control measures</i>
Handling molten metal	• Wear protective clothing consisting of wool trousers & jacket, visor, gauntlets, and splash boots
Equipment at high temperatures	• Only use furnaces when wearing the protective clothing
Manual grinding of samples in cold water leading to numb fingers	• Take care not to grind the ends of fingers
Use of etchants containing HF	• Etchants only to be used by the trained Technicians

Safety rules:

- Listen and follow instructions carefully. If you do not understand the instructions, then ask before you perform the tasks.
- **A Technician or Teaching Assistant must be present for all furnace and casting work.**
 - The furnaces are at high temperature and any contact with them, or hot crucibles or metal can result in a severe burn. Only use the furnaces when wearing the protective clothing.
 - It is possible that a clay mould may crack during casting and the molten metal escape. It is therefore important that the clay crucibles and the casting area are positioned such that should this happen there is no possibility for any materials to be set alight by the molten alloy or any chance of contact with personnel.
 - The acids used in making the etch are dangerous. HF in particular is a very severe poisonous acid. An HF burn is **extreme** and occurs by rapid movement of HF along cell boundaries, causing severe damage. Washing is largely ineffective. In the event of an HF burn a special gel should be applied. Check this gel is at hand. Etchants should be used in a fume cupboard wearing a lab coat, gloves and glasses. **The etchants containing HF are going to be applied by trained technicians only, the students may observe the process.**

What you should learn from this practical Science

This practical illustrates some of the points from the lecture course on Phase Transformations. It will help you understand:

1. Cast microstructure
2. Casting defects
3. The relationship between microstructure and a phase diagram.

Practical Skills

1. Melting and casting methods
2. Metallography of Al based alloys

Overview

Commercial purity Al and Al-Cu 20/80 (by weight) hardener are mixed together to produce an Al-4.5wt%Cu alloy. The Al and hardener are melted in a furnace, cast into two moulds: a steel permanent mould and a pre-heated clay mould, and allowed to cool and solidify. The thermal histories of the solidifying castings are recorded using a datalogger and thermocouples.

The cast ingots are sectioned longitudinally and prepared by grinding and polishing for macro-etching to show the overall cast microstructure. In each case, the macro/micro structure is recorded, and any defects such as porosity, cracking, shrinkage, pores, inclusions, etc. noted. The dendrite arm spacing is estimated and **related** to the cooling rate.

Safety

- Protective clothing for furnace work consists of wool trousers & jacket, visor, gauntlets and splash boots.
- The etchant, Keller's reagent, contains HF and will be made up and applied by a technician only.

Experimental details

Melting and Casting

One melt of Al-4.5wt%Cu, weight of ~400g, is to be cast.

1. Weigh the correct quantities of pure Al and Al-Cu 20/80 hardener for the alloy.
2. Put the components into a clay bonded graphite crucible in the furnace room – ensure that the metal rattles in the crucible.
3. One of the demonstrators (technician or teaching assistant) will put your loaded crucible into the pre-heated furnace.

Safety Note: The furnaces are at high temperature and any contact with them, or hot crucibles or metal can result in a very severe burn. Only use the furnaces when wearing the protective clothing.

1. Check the furnaces are set at 750°C and ensure the demonstrator places the crucible into the furnace using the long handled tongs.
2. The demonstrator will remove the crucible from the furnace after 1hour and stir with a graphite rod. Crucible is returned to furnace.
3. Ensure that the demonstrator has placed the clay moulds in the pre-heating furnace to heat them to 200°C.

Safety Note: This is potentially dangerous. The demonstrator **must** wear protective clothing.

4. The demonstrator will assemble the metal permanent mould on the heat resistant mats in the casting area and ensure the mould halves are securely joined. They will then place the clay mould in the casting area. Note that the clay mould is pre-heated to 200°C – do not touch! They will place the thermocouple in the top of the mould. Ensure that the temperature is correctly displayed on the datalogger and that temperature as a function of time can be recorded at regular intervals of typically 1 second.

Safety Note: It is not impossible that the clay moulds may crack during casting and the molten alloy escape. It is important therefore that the clay crucibles and the casting area are positioned so that should this happen there is no possibility for any flammable materials to be set alight by the molten alloy or any chance of contact with personnel.

5. The student in your group who will pour the castings must be wearing all of the protective equipment. At least one demonstrator must be present and wearing full protective equipment. Remove the crucible, checking the alloy is fully melted by stirring again with the graphite rod. Pour the liquid alloy into each mould with the casting tongs. Care should be taken not to pour too quickly to avoid splashing. A steady, single pour is preferred. Do not overfill the moulds. Record the temperature from before the alloy is poured until the casting has solidified (the eutectic temperature is 548°C and so recording of temperature should continue until ~ 500°C).

Safety Note: The casting operation is dangerous. You **must** wear protective clothing. A demonstrator or technician **must** be present for this procedure.

6. Leave the ingots to cool. Enter or manipulate the thermal history data into a suitable software package to produce a plot of temperature against time for both types of mould.

Microstructural Examination

1. The technician will ensure the moulds and ingots are cool enough to handle.
2. They will remove the ingots by disassembling the moulds. Note any surface features.
3. They will cut the ingots in half length-ways using the cut-off machine.
4. Grind one half of each ingot on successively finer grades of SiC paper for macroetching. Use plenty of water as a lubricant and gradually reduce the pressure as the paper grade becomes finer.

Safety Note: Take care not to grind the ends of your fingers! The cold water often numbs the fact you are steadily grinding your fingertips. Once removed from the water, this is very painful!

5. Polish the cross-sections with $6\mu\text{m}$ and the $1\mu\text{m}$ diamond polishing fluid. Again, use plenty of lubricant. Keep the ingots clean and avoid cross contamination of polishing fluid. Check your progress in removing scratches with the optical microscope.

6. Give the ingots a final polish with colloidal silica – the demonstrators will explain how to do this. Wash in lots of cold water. A small amount of detergent can be used with the thumb to help remove polishing debris. If your ingot has pores, it is important to make sure all polishing residue is removed as this will leak back over the surface. A demonstrator will place your sample in the ultrasonic bath for a final clean.
7. Mount the specimen flat on a glass slide using plasticine and the hand press, using a folded tissue to prevent scratching of the polished surface by the press. Use the optical microscope to examine the microstructure at a series of magnifications. Capture the images, noting:
 - The macro-shape of the ingot including the presence or otherwise of a shrinkage “pipe”, surface finish and internal defects.
 - How the as-cast microstructure does or does not conform to textbook pictures – if not, why not?
 - Any finer scale features such as interdendritic/cellular segregation (eutectic phases?) and differences in grain morphology such as dendritic, cellular or equiaxed grains.
 - Any differences between the two mould types? If so, why?
8. Etching of the ingots is carried out by the **trained technicians only** by swabbing them with Keller’s etch in the fume cupboard for 5 seconds, initially. If necessary, this is repeated until the grain boundaries can be seen. Al-Cu alloys etch readily and it is important to take care not to over-etch. In the event of over etching, ingots should be re-polished using colloidal silica. Keller’s reagent, 0.5HF-1.5HCl-2.5HNO₃-95.5H₂O, may only be made up and used by a technician.

Safety note: The acids used in making the etch are dangerous. HF in particular is a very severe and poisonous acid. A HF burn is **extreme** and occurs by rapid movement of HF along cell boundaries, causing severe damage. Washing is largely ineffective. In the event of an HF burn a special cream should be applied. Check this cream is at hand. Etchants should be used in a fume cupboard wearing a lab coat, gloves and glasses. The etchants containing HF **are going to be applied by trained technicians, the students may observe the process.**
Students must not distract the technicians during this process.

9. Re-examine the ingots and check to see if any features are now clearer after etching.
10. Estimate the dendrite arm/cell spacing. Ideally in a number of locations over the ingot cross-section. The dendrite arm spacing is expected to relate to the local cooling rate experienced by the alloy, which will vary from centre to edge and top to bottom. The secondary dendrite arms (the “branches”, rather than the “trunk”) are particularly sensitive to cooling rate.

Timetable

Day 1: Weighing of alloys, melting and casting.
Spend the rest of the afternoon researching what you will expect to see in your microstructures, and any differences you expect between the two casting. You should document your predictions in your lab notebook. It is sufficient to note that e.g. “a particular effect” is due to “xxx”; i.e. it is not necessary to write out a full explanation. You may want to

note down any references you use. You may want to include sketches or diagrams.

Days 2&3: Sectioning, grinding, polishing and microscopy.

What should be included in your lab notebook

1. Predictions

What you are expecting to see in your cast microstructures and a brief comment as to why you are expecting to see this.

2. Results

Only include those images which show significant features which you would intend to discuss if you were doing a full write-up. Try to bring out microstructural differences from edge to centre, or as a function of the alloy composition. A series of figures with only minimal text for labelling is sufficient. Give an indication of the magnification / scale of the microstructure.

Try and distinguish between columnar, dendritic or equiaxed grains, and eutectic phases.

Label the different morphologies and phases in the same convention as the Al-Cu phase diagram.

Show different types of casting defects.

Show how the dendrite arm spacing varies from place to place.

3. Discussion

Write brief comments on the effect of mould type on microstructure and defects. What had you expected to see; and what did you see?

Illustrate your observations with key images, which are correctly labeled and scaled. Refer to the Al-Cu phase diagram. Why does eutectic form at grain or dendrite/cell boundaries? Why do we get dendrites?

How do the different types of defect arise? Does the distribution of defects depend on position in the ingot?

Why should the dendrite arm spacing be a function of the local cooling rate? Is there any reason to suppose the dendrite arm spacing should depend upon composition? **Relate** any differences in microstructure and dendrite arm spacing to the measured cooling rate during solidification.

4. Conclusions

A brief list of conclusions.

Suggested Reading (*italics = available as e-book*)

Casting, J Campbell. *Excellent mix of theory and practice.*

Solidification Processing, M C Flemmings. Classic undergraduate textbook.

Fundamentals of Solidification, W Kurz & D J Fisher. More advanced mathematical treatment.

Phase Transformations in Metals and Alloys, D A Porter & K E Easterling.