Metallographic examination

Metallography requires the removal of small samples, which are then mounted in a resin or bakelite block, polished and etched in dilute acid, before examination under a metallurgical microscope. This reveals the crystal structure of the metal, from which an assessment of the type of alloy and its mechanical and heat treatment history can be made. Metallography thus provides a good measure of the quality of the metal and its suitability for a particular application. Scott (1991) provides a good introduction to the structure of metals, metallography and the phase diagrams which help explain the microstructures it reveals.

The structure of many ancient alloys is, typically, heterogeneous. Ferrous alloys were never heated to melting temperature and therefore retain quantities of slag as well as an uneven distribution of alloying elements, particularly carbon and phosphorus.

Non-ferrous alloys may show segregation of certain elements towards the centre or surface of the artefact. All alloys can show depletion of elements at the surface following oxidation, either during manufacture or after post-depositional corrosion.

Because of this heterogeneity, bulk chemical analyses, especially of the surface, may be misleading. Metallography, especially when combined with micro-analysis, investigates rather than ignores the microstructure of the metal and provides a greater insight into the technological and post depositional history of the artefact.

Ferrous alloys
It is with iron alloys that metallography can extract the most detailed information. The following notes show the sort of features which can be determined.

Slag content:

Examination of the unetched sample allows the slag inclusions to be measured, both by volume, and by shape. The shape of the inclusions show the way the artefact has been wrought.

Alloy identification:

Prior to the introduction of the blast furnace three main ferrous alloys were in use.
Ferritic iron: The grains of ferrite appear white and contain no impurity elements; it is a soft, ductile metal.
Phosphoric iron: Indicated by grain enlargement and 'ghosting'. Phosphorus contents in the region of 0.1-0.2% are sufficient to give a significantly harder, tougher alloy. *Steel:* The dark etching regions, are known as pearlite (and contain 0.8 % carbon). Steel is not only harder and tougher than iron but allows further hardening by heating and quenching.

Heat treatment:

An acicular structure, martensite, results from the rapid cooling *(quenching)* of steel without subsequent tempering. This is a very hard material but with a tendency to brittleness.
Tempered martensite appears less distinct and although not as hard provides a much tougher metal.
Metals worked at low or ambient temperatures and not subsequently annealed show deformed, elongated grains. This *cold working* increases the hardness of both iron and steel.

Composite Artefacts:

To capitalise on the hardness of steel and the ductility (and cheapness) of iron the two alloys were welded together to form composite artefacts. Such structures are frequently found in edged tools and weapons. A weld is generally seen as a sharp division between two metal types (although carbon may diffuse across a boundary). Scale and silicate inclusions may also be trapped at the interface.

Techniques for combining different alloys may have important cultural implications. For example, in many Saxon knife blades a steel edge was butt welded to an iron back, whilst Anglo-Scandinavian smiths favoured 'sandwiching' the steel between two low carbon sides.

Carburization was an alternative means of creating a hardenable surface. The iron was heated with a carbon-rich material in a reducing atmosphere so carbon diffused into the surface. Unfortunately this thin layer may be lost to corrosion in all but the best preserved artefacts.

Non-ferrous alloys

Identification of phases present allows the nature of the alloy to be determined. For instance, in leaded copper alloys the lead is immiscible and will be visible as dark grey areas at the grain (crystal) boundaries.

The shape of the metal crystals will show how the object was produced. Cast alloys generally have the characteristic dendritic structure, but this is broken down by subsequent annealing which produces equiaxed crystals. Cold working is indicated by deformation of these crystals and subsequent annealing produces a twinned crystal structure.

Fabrication techniques

Whilst ancient iron could be fire welded by heating and hammering, most non-ferrous alloys required additional liquid metal to bond the parts together. This may be of similar composition to the object's parts or an alloy chosen because is has a lower melting point (or melting range) than the material to be joined.

Surface treatments such as gilding, silvering and tinning were all used in antiquity. Metallography can identify the method used to apply the coating by examination of compounds formed between the two dissimilar metals.

Alternative approaches

Occasionally it is possible to polish a small area of the surface of a well preserved artefact, and examine it without the removal of a specimen. This will be of more limited value, because it does not allow the examination of a complete section, however it may be the only method considered acceptable.

Associated methods: Both micro-hardness and analysis in the SEM or microprobe (see Datasheet 12) are extremely useful in extending studies of Metallographic sections because they operate on a scale that allows individual phases to be examined. A micro-hardness tester measures the indentation produced by a diamond under a set load and provides a direct measure of the hardness of the metal.

Justification: Most opposition to metallography arises because of the need to 'destructively' remove samples. This must always be balanced against the potential evidence which could be obtained by the technique.

Metallography can be a very informative investigative technique but programmes need to be well thought out to ensure that valid conclusions can be reached.

Reference

Scott, D A (1991) *Metallography and microstructure of ancient and historic metals.* Getty Conservation Institute.

David Starley
Ancient Monuments Laboratory
English Heritage

April 1995

