Chapter 1
Introduction to Metallography

Metallography is essentially the science of the internal structure of metallic materials, broadly understood to include the examination and identification of grains, phases or interfaces and crystal orientations of a metal, as well as its non-metallic inclusions and voids, corrosion crusts and patinas. The technique traditionally relies on the sampling of an object to remove a representative sample, which is then mounted, ground, polished and etched for examination of the microstructure under the optical microscope. This principle procedural method is still valid, but there has been a drastic change over the decades from exclusively optically microscopical examination of microstructure to methods based on electron and ion beams or X-rays. In recent years, we have seen the emergence of new powerful techniques such as electron backscatter diffraction (EBSD), which are capable of identifying and characterizing quantitatively all microstructural components by automated high-speed pattern acquisition (see Chap. 3). Hence, metallography is not confined merely to microscopical examination but also comprises methods to reveal structure and mechanical properties of metals and alloys.

The increased resolution and importance of bulk structure accompanies a new concept of classification of structure into macro-, micro- and nanostructure, depending on the size ranges of structures and according to the measurement methods. The scale of macrostructure can be fairly large ranging from millimetres to the infinite, while microstructure is relevant for micro-devices having sizes typically of 1–1000 micrometres, while nanostructures have dimensions of about 1–100 nanometres [15]. Nanoscale magnification and three-dimensional microscopy have become common tools in modern metallography, and it is symptomatic that the chapter about “Microstructure” of the last edition of *Physical Metallurgy* that first published in 1965 does not refer to classical two-dimensional examination of surfaces any-more but to three-dimensional characterization by X-ray tomography or serial sectioning techniques and subsequent computer-simulated modelling of microstructure to replicate the microstructural features [53].

Nevertheless, “classical” metallography and the morphology of metals have been an important aspect of our understanding of metallic phases, bonding and the

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techniques that are used to manipulate properties and attributes such as hardness and colour. Many of these choices are culturally determined: they represent decisions made about the nature of materials and how they are to be used. Three of the metallic materials of antiquity have given their names to phases of societal development which we recognize as the Chalcolithic, the Bronze Age and the Iron Age, a lens through which we can view the past and discern the sometimes illusory nature of the concept of continual progress, of the aggregation of technological development as one generation builds upon the achievements of the former [5, 6], and the different paths which the development of metallurgy took in Africa and the New World; see, for example, Kusimba and Killick [27], Lechtman [28] and Scott [49].

The study of metals by means of simple optical devices dates back to the seventeenth century [50], but the science of metallography really began with the work of Henry Clifton Sorby (1826–1909), who made seminal advances in the field of the petrological examination of rocks and minerals and developed an incident-light microscope. Following that, Sorby realized that reflected light microscopy could be of essential application to the study of the microstructure of metals, particularly iron and steel [50]. The development and concepts of early metallography have been well-described by Cyril Stanley Smith in his book *A History of Metallography* (1960), which the reader interested in further historical information is referred to.

As applied to archaeological studies, metallography came into use in the first few decades of the twentieth century and has been of great importance to archaeologists, corrosion scientists, conservators, metallurgists and engineers ever since. The scientific examination of ancient metals had already started in the late eighteenth century with the determination of their chemical compositions (see Caley [3]) and was systematically extended during the 1930s, when instrumental analytical techniques such as optical emission spectroscopy became available [36]. Indeed metallography of ancient metals can be used to check on the results of analytical determinations, which may be affected by non-metallic inclusions, heterogeneity of the alloy or internal or external corrosion. The only way to gauge these effects on the analysis is by metallographic examination. Metallographic studies are an integral part of a comprehensive investigation of the elemental composition, isotopic data, surface binocular examination, X-ray macrostructure analysis and microstructure of the artefacts in question.

Pioneering works revealing the microstructure of metals had already been published by the early 1900s, and, in a few papers, the microstructures of ferrous and nonferrous objects from all over the world have been published and illustrated, albeit with quite (in)different quality (e.g. [1, 20, 24–26, 30, 45, 46]).

The principal difficulty in creating better research tools for metallography was the limitations on the kind of microscopy available and the quality of the illumination and photography in the early 1900s (see Sect. 3.3.1).

Comparatively few studies were published between the First and Second World Wars (e.g. [2, 4, 32, 33]), a dearth of young researchers meant that meagre advances occurred in conservation science or the metallurgy of ancient metals during this period. We recommend Édouard Salin's and Albert France-Lanord's [48] book *Le fer à l'époque mérovingienne*, which not only illustrates early medieval iron technology utilizing photographs, drawings, micrographs and radiographs but also documents the metallographical and analytical equipment of that time. This book also includes a discussion of the methodologies for the conservation and restoration of archaeological iron objects.

During the late 1950s and early 1960s, the situation began to improve and has continued to go from strength to strength since that period. For archaeological metals, some of the authors in English who began to have an impact were Herbert H. Coghlan [7–14]), Ronald F. Tylecote [58–60] and Cyril Stanley Smith [50–52]. CS Smith was an exceptionally creative metallurgist who developed theoretical models and methods for deriving the three-dimensional shapes of the crystalline structures of metals from the two-dimensional microscope images, but he is also one of the pre-eminent metallurgists of ancient metals, whose research forms a body of work brought together in the seminal volume published in 1981, which deserves to be better known.

Important writers in the metallographic field of ancient metals emerged in many countries from the 1950 to 1960 period onwards, with seminal contributions by Jerzy Piaskowski [37–39] in Poland, Carlo Panseri and Massimo Leoni [34, 35] in Italy, Albert France-Lanord [21–23] and Eduard Salin [47] in France, Robert Thomsen [56, 57] in Denmark and Erik Tholander [54, 55] in Sweden, Joachim Emmerling [16–19] in Germany and of course Radomìr Pleiner [40–44]) in the former Czechoslovakia, who published several volumes concerning ancient iron technology.

Advances in optical techniques and the application of the scanning electron microscope (SEM) and electron probe microanalyser (EPMA) have continued to make metallography more accessible to a wider array of researchers across the globe and now form part of many investigations of ancient metals (Chap. 3). Metallography continues to be an important method for the understanding of how metals were smelted, cast, worked, plated, alloyed or corroded. Metal objects may be plated or coated with other metals, which limits the extent to which non-destructive analysis can be used to provide a definitive answer to the question of their identity. Precious metal alloys such as gold-silver, silver-copper and gold-copper alloys are often finished by surface enrichment or depletion gilding or some other form of surface treatment or coating (Chap. 6). In such cases, metallography has to be used to gain further information and can determine, in conjunction with analyses of the polished cross-section, the nature of the surface coating. The primary distinction to be made in a great number of initial studies is whether the metal or alloy has been cast to shape, or whether it has been worked and annealed to shape, and the extent of corrosion has important implications for authenticity studies of ancient metals.

Metallography can help to inform us about the past use of metallic materials as well as aspects of their extraction, the use of sulphide or oxide ores in the smelting of copper, and to answer quite sophisticated questions concerning the fabrication of ancient iron, steel and cast irons. As the methodology is also applied to waste products, furnace linings, technical ceramics and metallurgical slag, the latter often includes tiny prills of metallic materials along with the silicate minerals that constitute the slag.

Numerous problems relating to the authentic nature of ancient metallic artefacts, particularly silver and copper alloys, as well as iron and steel artefacts, can be addressed by metallography. In cases of authenticity questions, it is often possible to adduce enough information from the metallographic section to avoid the need for further studies, particularly in the case of ancient bronzes, where the extent of patina penetration and the type of patina may be crucial and sufficient evidence may exist to condemn the artefact as a modern reproduction. Some of these achievements are illustrated through the metals and alloy discussed in this book, which we hope will be a reliable guide for the understanding of microstructures of ancient metals, as well as an introduction to principal practices of metallography.

There are numerous standard textbooks available in many languages concerning metallography in the wider context; to the English reader, we refer to further work detailed in Chap. 3, and these textbooks should be consulted for further reading. Many of them have become obsolete because of substantial changes in materials, preparation methodology and especially digital imaging but still give valuable information about materials and practices used for previous metallographic investigations. In modern metallography large samples may be ample and often easily replaced, but samples of ancient metals are of important historical value, are often unique and should be stored safely.

Some textbooks deal exclusively with preparation procedures or selected applications, while others are concerned with etching principles, or they introduce basic concepts and practical application of quantitative methods for characterizing the microstructure. Some textbooks, such as the *ASM Handbooks* (Lyman [29]; Mills [31; Vander Voort [61]), offer theoretical metallographic background information and individual practical application, both of which are essential for the examination and interpretation of metallographic structures.

Nevertheless, there are major differences between the constitutions and the examination of modern metals and ancient artefacts, which will be discussed in this book in greater detail so that the reader can form an appreciation of the excitement and curiosity that ancient metals can arouse as well as inform us of some of the achievements of cultures long since dead.

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