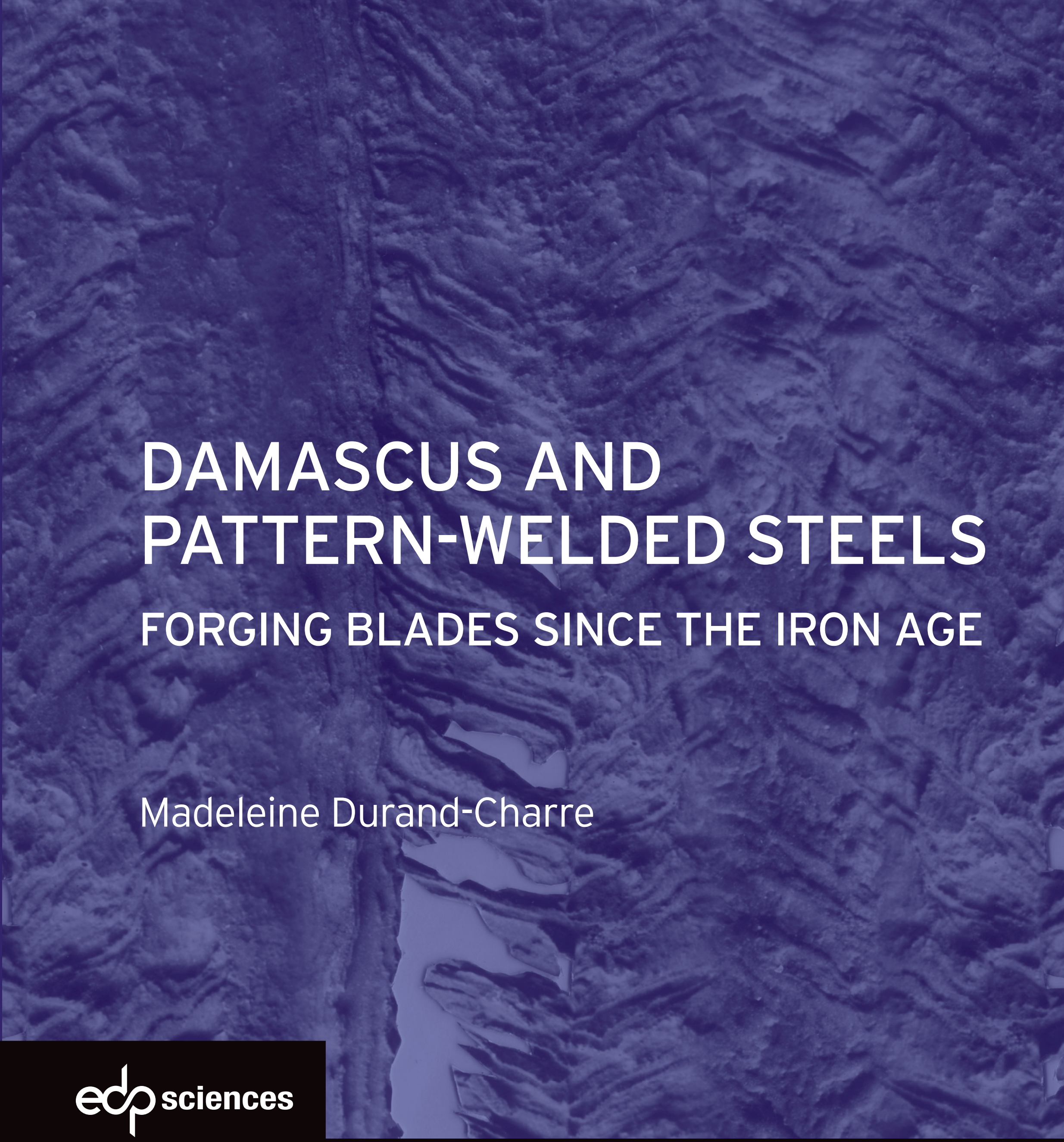

DAMASCUS AND PATTERN-WELDED STEELS

FORGING BLADES SINCE THE IRON AGE

Madeleine Durand-Charre

edp sciences

metallurgy | **materials**

DAMASCUS AND PATTERN-WELDED STEELS

Forging blades since the iron age

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This new book is an updated translation from French of "Les aciers damassés. Du fer primitif aux aciers modernes" (Presses des Mines, 2007).

Books by the same author:

"The microstructure of superalloys" (Gordon and Breach, 1997)

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Introduction

Trying to understand what a damask steel is, I discovered a fascinating subject, rich in multiple facets which initially appears simple: this is laminated steel, a composite material artistically exploited. In fact, the name is confusing when considering the words refering to Damascus such as damask, damascene and Damascus steels have different meanings and refer to very different materials whose common feature is just a wavy pattern.

During the first millennium BC the Celtic smiths acquired control of hot iron working, in particular for the production of strong sword blades. The latter were built by forge welding more or less carburized pieces of smelted iron. Damask steel appears as a pattern-welded composite resulting from this know-how several centuries later.

Meanwhile, in Eastern countries a high carbon steel was developped. This legendary steel displayed a moire pattern after specific forging. It was known by several names, Indian steel, Damascus steel or wootz etc. and remained rather mysterious until the 17th century. The history of the debate comparing the different kinds of steels is evoked in chapter 4.

Since this period, modern sophisticated tools became available to observe the specimens. Many researchers, in particular the Verhoeven team, investigated the microstructure of ancient blades, thus enabling clarification of how they were forged. This is the point of view that I plan to develop in detail.

What readers are likely to be interested by this subject?

- Metallurgists, I thought about my colleagues because I regretted, after having taught the structural metallurgy during many years, not having opened a very small window, a few minutes in my timetable to explain to the students how the metallurgy of iron has developed.

- Archaeologists, the curators of a museum and all the researchers confronted with the problems of expertises of the vestiges. They can, therefore, be interested by a detailed approach to the micrographic aspects.

- Blacksmiths who have a good technical approach to metallurgy supported by sound experimental know-how. They participate on the Internet in numerous discussion forums. As an occasional observer, I noted some points for which a fundamental approach could be useful.

- Collectors and amateurs who appreciate to be informed of the making of their valuable artistic blades.

However, when writing this book it appeared difficult to me to address such different readers, to avoid specialist's vocabulary and find a common language. That is why the text has been divided into two parts. A first descriptive part which presents the developments in the technology of forging in various places and various periods, and a more fundamental second part with the aim of explaining the scientific mechanisms and present the most recent findings for the formation of particular microstructures such as the bands or alignments responsible for the moire structure.

However, steels are a material with multiple and complicated transformations; this is true even for steels of basic cutlery industry. The explanation of a mechanism requires a detailed description in order to respect a strict argumentation. To ensure a sound basis to the discussion in its scientific context, the concepts of phase, grain, segregation and the particular features of high carbon steels are revisited in chapter 8. This edition is supplemented with a few new examples, with tables summarizing the findings and an index. Ultimately, this book does not propose practical solutions to the blacksmiths but rather a microscopic vision of their metal in order to support a better understanding of the formation of the microstructure.

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Aknowledgements

To acheive this work I ventured a little imprudently out of my strict scientific discipline. Also it was necessary for me to ask for the assistance of specialists in other disciplines extremely far away from mine. I express my gratitude to all those who believed in this project and who allowed the use of documents or samples, or prepared specific photographs.

I will quote in particular MM Paul Merluzzo, Louis Bonamour, Eric Perrin and Ernst Kläy. Many archaeologists, researchers, curators of a museum, transmitted documents or information to me: Mmes Christine Bouclet-Riquier, Véronique Despine, Anna Feuerbach, M.C. Lebascle, Aurélie R. v.Bieberstein, Bernadette Schnitzler, C. Vigouroux, et MM Gilles Desplanque, M. Ferry, Leon Kapp, J. Parisot, Jean Renaud, O. Renaudeau, J. P. Sage, Philippe Schaffnit, Pierre Thomas, Eric Verdel, John Verhoeven and Yoshindo Yoshihara.

My project began with my professional retirement when I had time available but I did not have any more the daily contact with my former laboratory at the Polytechnic Institute of Grenoble (INPG). Micrographs coming from my work of research, almost the memories of a photo album, were not sufficient. I am particularly indebted to my former colleagues and friends Annie Antoni, Florence Robault, Catherine Tassin and Muriel Véron for fruitful discussions and their assistance to supplement some examinations, analyses or micrographs.

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Avril 2014
Madeleine Durand-Charre

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First part :

The Blacksmith’s steel spanning four millennia

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1 *Primitive iron*

1.1 Iron before the Iron Age

The metallurgy of iron appeared in most ancient societies subsequent to that of gold and that of copper. The most significant appearance was situated in Anatolia in the Hittites, between 1400 and 1700 BC, thus determines the beginning of the Iron Age. The oldest discoveries date back to the prehistoric period around 5000 years BC in Iraq (Samara), in Iran (Tepe Sialk) and in Egypt (El Gerseh). More recently, discoveries in the period known as the bronze age (3000–1600 BC) are all situated on a wide border East and Southeast of the Mediterranean Basin in Mesopotamia, Turkey, Egypt and Cyprus (more details can be found in [1]).

Furthermore, the presence of objects made from iron does not necessarily imply the ability to extract the metal from its ores, since iron also exists in native and particularly meteoritic forms, although the sources are by no means abundant.

Primitive iron

The earliest iron used by man was generally meteoritic in origin. It is the presence of nickel which distinguishes it from the other categories of iron. Modern characterization techniques enabled detecting that nickel is present in most objects of the prehistoric period and in those from the early and middle bronze ages. The iron found as metallic meteorites, called also siderites (Figure 1.1.1), were worked in the same way as stone in order to form tools. In Greenland, three meteorites among the largest ever found (one weighed 36 tonnes) had been used for generations by Eskimos.

In Central and South America, the Aztecs, Mayas and Incas used meteoritic iron well before knowing its metallurgy. They considered it

Figure 1.1.1 :
Polished section of a metallic meteorite from a multiple fall, known under the name of Gibbeon (Namibia). To notice the Widmanstätten structure consisting in long needles imbricated along three directions.
Courtesy ENS Lyon, Fr.

as extremely precious and restricted its use to jewellery and religious objects. In Egypt, the blade of a magnificent ceremonial dagger found in Tutankhamen's tomb (1350 BC) was identified as being made from meteoritic iron. It was one of a pair of objects, the other being gold.

Meteoritic iron is an alloy which generally contains a few percent of nickel, with amounts ranging from 5 to 26%, together with small amounts of cobalt (up to 1%) and traces of sulfur, phosphorus and carbon. Metallic meteorites are relatively malleable. They are one of the three major classes of meteorites, corresponding to metallic, stoney and mixed structures [2]. They are generally believed to be fragments of planets that have disintegrated, the metallic meteorites emanating from deep inner layers. The crystalline phases present in metallic meteorites have names specific to this field of study. For low nickel concentrations, the body-centered cubic crystal structure is known as kamacite (α ferrite in steels, § 8.1), whereas the face-centered cubic structure found in high nickel meteorites is called tænite (γ austenite in steels). This structure consists of plate-like ferrite and was observed for the first time in 1808 in a meteorite by Aloïs von Beckh Widmanstätten (Figure 1.1.1). The plates are oriented in directions which form an octahedron. The origin of this structure in meteorites has been suggested to be associated with the existence of a solid state reaction at very high pressures [3]. However, for certain meteorites the microstructure is so coarse, with broad plates several millimeters, that a solid state transformation could seems unlikely [4].

Telluric iron may be found in the native state in basalt or other rocks as small grains or nodules. It often contains a high content of nickel, up to 70%. This iron, rarer than the meteoritic iron, was sometimes found in precious objects. Ural native iron contains 50 % of platinum.

The name *terrestrial iron* is given to iron extracted from ores ; it is normally free from nickel. Iron of this type has been found in objects in Egypt, in the Temple valley and Cheops' pyramid at Giza (2500 BC) and at Abydos (2200 BC). However, the number of such objects is small and their authenticity is doubtful, due to their poor state of conservation.

The oldest iron not of meteoritic or native origin is found as small decorative inlays in gold jewellery or tiny cult objects. It has been suggested that this iron is a by-product of the gold production process.

Magnetite is frequently present in the gold-bearing sands in Nubia and could have been reduced during the smelting operation as pasty iron floating in the slag above the molten gold. Another possibility is that iron oxides were deliberately associated with other oxides used as fluxes for the manufacture of bronze. The iron dating from this period was described as *accidental* iron [5].

Whether the production of iron by the reduction of ores was discovered at an early stage, before 2000 BC is a subject of controversy. All the more, the presence of non-meteoritic iron objects is not always associated with evidence of local mining activities. For example, in Egypt, where iron ore deposits are abundant, there is no sign of their exploitation. The argument is that it is probably due to the absence of forests capable of supplying the charcoal necessary for reduction.

Recent investigations with modern means of analysis have evidenced that a bead from the prehistoric Gerzeh cemetery, approximately 3300 BC, which was considered as the earliest example of exploitation of iron in Egypt, was in fact made out of cold-worked meteoritic iron [6].

It must be remarked that several millennia elapsed between the first reliable identifications of iron artifacts and the start of what can be genuinely termed the iron age. Several explanations can be suggested. The most obvious one is the inherent difficulty of extracting iron from its ores. The processes used for gold and copper are not applicable, and in particular, much higher temperatures are required.

It is difficult to forget what we know about iron to imagine how this new material was considered. In fact, the iron obtained by the most primitive processes of reduction of the ore should not have been regarded as an interesting material. When iron is reduced, it is pure and highly malleable, thus usable only for ornaments. It was rare, and therefore very precious and its value could exceed several tens of times that of gold.

1.2 Early iron making techniques

Iron ores

After aluminum, iron is the second most abundant metal in the Earth's crust. The major iron ores are essentially oxides (magnetite Fe_2O_3, hematite Fe_3O_4 and limonite), carbonates (siderite) and sulphides (pyrite). The preparation by washing and crushing of the ore is the same one as that practiced for the other ores. Many ore deposits occur in the eastern Mediterranean basin and can often be readily recognized due to the associated rust-red coloration of the earth. Indeed, they were often exploited as pigments, giving the yellows, ochres, browns and reds used by the Egyptians. Evidence of early mining activities is visible in deposits in Syria and Cappadocia, which appear to have been the first to be exploited on a large scale. Metallurgical culture is extremely ancient throughout the fertile crescent, facilitated by the presence of numerous rich ore deposits. The Assyrians seem to have practiced the reduction of iron ore as early as the 19^{th} century BC.

Some ores were famous, probably due to the natural presence of alloying elements such as manganese (Siegerland in Germany), nickel (Greek or Corsican ores) or phosphorus (Lorraine ore) [7-8].

Iron smelting

In the earliest iron processes, washed and crushed ore was heated with charcoal in a primitive furnace, often consisting of little more than a hole in the ground. The temperature attained was insufficient to achieve melting and the oxide was reduced by the carbon in the solid state, leading to a spongy agglomerate called a bloom. Many primitive furnaces were built in such a way as to optimize natural drought (Sri Lanka). Furthermore, the use of rudimentary bellows made from animal hide was probably adopted at an early stage. The furnace can be controlled by the injection of air and, depending on the setting, local carburization of the iron can be acheived. This carburization occurs at high temperature by contact of the iron with a CO atmosphere near the charcoal. Traces of cast iron found amongst the slag in ancient smelting centers indicates that the temperatures attained were sufficiently high to induce partial melting. However, such cast iron was probably initially obtained accidentally and considered as a worthless by-product, since it was hard, brittle and unworkable.

The development of iron smelting was particularly facilitated in areas where ore deposits were associated with ready supplies of charcoal and refractory materials for furnace construction.

The smelting stage produces a spongy agglomerate called a bloom, consisting of well reduced iron particles, residual oxide inclusions and areas with carburized iron particles. The bloom has to be repeatedly heated and hammered to expel residual slag inclusions, forming a more compact mass. The addition of sand during reheating contributes to the removal of oxide inclusions by the formation of a fusible envelope of fayalite. The iron obtained in this way was fairly pure, since oxides of other metallic elements such as Si or Al could not be reduced in these conditions. Its carbon content is low, it is therefore malleable.

Pure iron can be carburized by simple welding in contact with more carburized iron agglomerates during repeated hot hammering. If the metal working is intense, the agglomerates are crushed, mixed and carbon diffuses between the iron grains, it results in a homogenization of its concentration.

The soft iron can also be carburized by a further treatment known as cementation whereby carbon difffuses into the metal. At the temperatures attained, the depth of carbon penetration was no more than about a millimeter (see Table 8.1.2). Thus, this process can be carried out only locally to strengthen superficial areas, or sharp edges, or on iron divided into pellets or thin strips. Prakash underlines that the Indian blacksmiths had acquired a great knowledge of the effects of carbon in iron and knew how to control the production of more or less carburized iron [9].

Crucible steel in East Asia

The fusion of steel in a crucible is an invention of the first millennium BC. The production of *crucible steel* goes back to 300 BC according to the history of the wootz written by Srinivasan and Ranganathan at the University of Bangalore in India, [10-11]. This date is based on the discovery of high-carbon steel on a production site of this period (Kodumanal). This vestige is the oldest known, it is presented as a relatively reliable proof of the more or less complete fusion of the steel.

The same authors also quote a document according to which, Indian king Pôros, defeated by Alexander Great, offered to his conqueror 30 pounds of Indian iron in 326 BC. Several indices let suppose that it was most probably crucible steel. However this assumption remains rather controversial.

Recent excavations revealed several sites of production in Central Asia, among them Merv in Turkmenistan and Achsiket in Uzbekistan. The oasis of Merv located on the silk route was an important production center in the 8th et 9th centuries. Anna Feuerbach, who took part in excavations, presents in several publications an example of exploitation of vestiges led in a rigorous scientific way [12-15]. The sites of Achsiket are dated between the 9th and 14th centuries.

The iron as reduced from its ore was melted in a crucible in a distinct subsequent operation. Steel was prepared by small charges, each consisting mainly of iron, charcoal of bamboo and leaves of specific plants. The iron used was probably in the form of an iron sponge, it still contained oxides particles which while melting form a protective slag at the surface of the ingot. The whole was then hermetically sealed in clay crucibles. A batch of some twenty crucibles was heated for a long time at temperatures up to 1200 °C, and then let cool. Liquid iron was carburized by reaction with charcoal enabling a partial melting [12-13].

The iron mass found in the bottom of the crucible was a small ingot called *cake*, typically weighing up to 2 kg [9]. Iron prepared in this way differed from other irons by its high carbon content, up to about 1.5 %. Indeed, it is a high carbon steel. Trace elements such as vanadium and titanium, possibly from the bamboo charcoal or other plants employed, probably contributed to the exceptional properties of this steel (§ 10.1).

The metallurgical know-how of India was highly appraised as early as the Pre-Islamic period [11]. The method of preparation by fusion, initially exploited in the southern part of the Indian sub-continent including Sri-Lanka, slowly propagated originally towards Asia and then towards the Middle-East, Iran, Turkey and even as far as Russia. Later, this steel will be known under the name of *wootz*, a name which undoubtedly comes from the English deformation of the ancient Indian word Ukko and which was stabilized at the beginning of the 19th century [16-17]. Unfortunately vestiges of this time are very rare. According to Srinivasan and Ranganathan [11], one of the reasons is perhaps that the Indians had, at least during the historic period, no funerary tradition of tombs like many other civilizations.

Thus the period during which this process was used extends from 300 BC to 1856 AD. This last date corresponds to the beginning of the modern age of iron in 1856 with the invention of the Bessemer process to manufacture molten steel on an industrial scale, which cannot be compared with the manufacturing of the Indian steel cakes. The fusion of steel was invented by Benjamin Hunstman in Sheffield in 1740, approximately two millennia after crucible steel.

Cast iron in China

Iron production by smelting appears to have been known in China from about 1000 years BC. However, China was the first country to use cast iron *(i.e.* molten) in around the 6^{th} and 5^{th} centuries BC [1, 18-21].

Among the vestiges which support this discovery are cast iron cauldrons dating from 512 BC and cast iron molds from the end of the first millennium BC. It has been suggested that these developments were made easier by the presence of phosphorus-rich ores, since phosphorus lowers the melting point of the carbon-iron alloy. Furthermore, technical know-how in related fields was more advanced in China than in other parts of the world. For example, in the case of pottery, the Chinese mastered the manufacture of both red pottery, baked in oxidizing atmospheres, and black and egg-shell pottery baked in reducing environments. Their furnaces were ingeniously designed and made from high quality clay refractories, and bellows were in regular use in the 4^{th} century BC. Their superiority was maintained by improvements such as the introduction of piston bellows in the 2^{nd} century BC, and the replacement of charcoal by coal in the 3^{rd} century BC, nearly two thousand years before Europe.

Several original techniques were developed to transform white cast iron into iron and steel by decarburizing heat treatment and also under the combined decarburizing action of forging and heating. Under the Han dynasty, in the 2^{nd} century BC, cast iron was decarburized to transform it into ductile iron. Early in the 5^{th} century AD, an original iron carburizing technique was developed, consisting in immersing rough iron in cast iron and then subjecting the coated product to a series of forging and folding cycles to produce a homogeneous medium carbon metal. Even more surprising is the recent discovery of cast iron objects dating from the Han and Wei dynasties (206 BC to 225 AD) containing graphite nodules similar to modern spheroidal graphite iron, invented in... 1948! Chemical analysis revealed none of the nucleation agents used today for spheroidization. It has been suggested that an appropriate Mn/S ratio enables graphitization of cementite to occur in the solid state with a nodular morphology [22-23].

Iron making in Africa

In Equatorial Africa, neolithic practices were directly followed by an iron age, with no intermediate use of copper or bronze. The analysis and dating of many small artifacts indicates that the metallurgy of iron in this area goes back to at least the 3^{rd} millennium BC, and possibly even to the 4^{th} [24]. In Gabon, furnaces dug into the soil have been carbon dated to the 7^{th} century BC, based on charcoal residues found in the vicinity. However, the lack of spatial coincidence does not provide unambiguous proof. Furthermore, iron objects remain relatively rare, due to the difficulty of conservation in the prevailing moist climate and acidic soils.

It has been suggested that liquid iron was obtained at an early stage in prehistoric furnaces found near Lake Victoria in Tanzania, high temperatures being attained by the injection of preheated air. However, what is probably more important is that, in the region concerned, the iron ore is extremely rich in phosphorus, while the local vegetable matter mixed with the ore is also rich in phosphorus, facilitating melting.

From Asia Minor to Europe

Around 1500 BC iron smelting was practiced in the Caucasus region Northeast of Turkey. The metallurgical know-how propagated slowly. Towards 600 BC, the Etruscans and the Catalans practiced an advanced metallurgy. And in 300 BC, the Celtic culture spread as far as Ireland. The metallurgical culture expanded over all of Europe. The following well documented books can be used as initiation to the history of iron specifically to this period : [18, 25-29].

The competition between bronze and iron swords lasted for several centuries. The Romans, who had conquered territories in Spain containing rich deposits of copper ores, used bronze swords. They did not see the advan-

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