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Introduction

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The desire for stronger steels probably goes back to the origins of the first transformations of a mixture of ore and charcoal into iron. This was done by the Chalybes and the Hittites in the South Caucasus. The Hittites were certainly the first to use iron in weaponry, as Hittite cuneiform tablets from the 18th-century BCE indicate the production of iron weapons. As for the Chalybes, they were the first metallurgists to produce steel at the beginning of the first millennium BCE: “the hard iron of the Chalybes” was much sought after for its hardness. The desire to have stronger steels increased during the Iron Age. It was discovered that heat treatment in carbonaceous residues followed by tempering allowed the manufacture of very resistant weapons and tools. It is necessary to remember that at that time, weapons were mainly cast in bronze, and whoever mastered the manufacture of more resistant weapons had a strategic advantage that allowed them to establish their domination.

1.1. Steels, a rich metallurgy

This ancient use of iron and steel was possible because of the large presence of iron ore in the earth’s crust and the specific characteristics of this alloy that make it both ductile and resistant. These properties come mainly from some specificities of steels and alloys based on the iron-carbon couple. First of all, iron has different structures depending on the temperature: ferrite or α iron, with a body-centered cubic structure, for temperatures below 912°C; austenite or γ iron, with a face-

New Advanced High Strength Steels,
coordinated by Mohamed GOUNÉ, Thierry IUNG, Jean-Hubert SCHMITT.

centered cubic structure, for temperatures between 912 and 1394°C; and finally, again the body-centered cubic structure phase between 1394 and 1538°C, the melting temperature of pure iron. These different phases are inherited by the steels, and the content of carbon and different alloying elements can influence the size of the domains of existence of these different phases and the critical temperatures. For example, while the solubility of carbon exceeds 2 wt% in austenite at 1154°C, it is extremely low in ferrite and of the order of a few parts per million (ppm) at room temperature. Moreover, the small size of carbon atoms compared to that of iron atoms leads to a solid solution of insertion, that is, carbon atoms are positioned within the network formed by iron atoms.

Finally, it can form a eutectoid compound, the pearlite, formed by alternating parallel lamellae of almost pure iron and iron carbide, the cementite, Fe₃C. This constituent is in fact a lamellar composite associating a deformable phase and a hard phase.

It is these microstructural features of steels that give them such different properties in terms of hardness, deformability, toughness, etc. The engineer has the full range of metallurgical mechanisms at his/her disposal by adjusting the chemical composition and the thermo-mechanical processes. By adjusting the carbon content, it is possible to develop different families of steels, from the softest, ferritic interstitial-free (IF) steels with a yield strength of just over 100 MPa, to pearlitic grades in which the strength increases as the spacing between the cementite lamellae is reduced. As the solubility of carbon is relatively low in ferrite, it is possible to obtain a fine hardening precipitation by adding titanium and niobium. In high-strength low alloy (HSLA) steels, the presence of carbonitrides of about 10 nm of size and the small grain size, whose growth is limited by the precipitates, lead to mechanical strengths of up to 800 MPa. Finally, by increasing the carbon content of the alloy, it is possible to develop a whole range of two-phase steels composed of a ferritic matrix and an increasingly large volume fraction of pearlite islands. The mechanical properties of these steels are directly related to the volume fraction of the hard phase.

In parallel, the existence of an austenitic phase at a higher temperature makes it possible to play on the cooling rates after hot deformation or heat treatment in order to obtain metastable phases. Martensite and bainite are thus hardened by an oversaturated carbon content and by numerous dislocations resulting from a non-equilibrium transformation. These hardening phases reach yield stresses above 1000 MPa. By combining the composition of the steel and the cooling kinetics from the austenitic phase, it is possible to obtain dual-phase (DP) steels where the martensite is a hard phase in a more deformable ferritic or bainitic matrix.

The addition of certain alloying elements such as nickel or manganese considerably increases the size of the austenitic domain to such an extent that, for certain chemical compositions, the steels can retain a face-centered cubic structure at room temperature. This is the case for austenitic stainless steels where the addition of more than 11% by weight of chromium provides surface protection, while the further addition of 8–10% nickel stabilizes the austenite. Thus, grade 18-10 (18% Cr and 10% Ni) has been the most developed grade for cutlery and household items. The cost of nickel and its variability have led to a search for other alloying elements that can stabilize austenite, which has the advantage of a high strain hardening rate leading to a potential elongation greater than 50% in tension. Steels with a high manganese content (of the order of 22% by weight) and carbon (around 0.6% by weight) have represented the second generation of advanced high strength steels, the TWIn-Induced Plasticity (TWIP) steels.

Finally, more recently, steels have been developed which combine a ferrite-bainite matrix and metastable austenite islands, that is, that can be transformed into martensite under the effect of stress or strain: TRansformation-Induced Plasticity (TRIP) steels. These steels present a new mode of hardening insofar as the fraction of the second hardening phase increases during deformation. This mechanism makes it possible to combine high mechanical strength values – above 1200 MPa – with a tensile elongation of more than 15%. Precise control of thermomechanical cycles, in particular step cooling, enables the industrialization of these third-advanced high-strength grades, which are useful for lightening structures and increasing their safety.

It is clear from these few examples, which are not exhaustive, that the richness and particularities of steel metallurgy have enabled a development that continues to this day.

1.2. Steel, a dense history

Until the 17th century, steels did not evolve much and the main mode of hardening remained quenching, with no study on the hardening mechanisms. One of the first models of hardening is attributed to René Descartes in 1639. He introduced the concept of “fire particles” at high temperatures, “air particles” during slow cooling and “water particles” during rapid cooling. The hardening of steel would thus result from the replacement of the “fire particles” by the smaller “water particles”. In 1671, Jacques Rohault used Descartes’ theory to explain that a “sudden” cooling prevents the particles from returning to their original position. They then appear frozen, leading to a denser and “stronger” steel. The work of René Antoine Ferchault de Réaumur, published in 1722 and entitled *L’Art de convertir le*

ferforgé en acier et l'art d'adoucir le fer fondu, marked a turning point. Based on the observation of the metal structure revealed by the fracture surfaces, he proposed an explanation of the steel hardening mechanism based on “molecular transformations” produced by heat. In the molecules, he distinguished between “iron particles” and “sulfide and salt particles” and explained that steel becomes harder because the “sulfide and salt particles” cannot be removed and remain fixed in the steel during rapid cooling. However, Réaumur was unable to define the nature of the “sulfide and salt particles”. They were successively renamed “phlogiston”, “plumbago” and “carbon” in 1800. All of this academic work gave a strong impetus to a better understanding of steels and to their industrialization. The industry of “cemented steels” developed throughout Europe and America during the 18th century and the first half of the 19th century. It met the needs of the mechanical industry, which required parts with a high surface hardness. Hardened steels were also highly sought after in the military field because of their hardness, as Gaspard Monge’s treatise *Description de l'art de fabriquer des canons* published in 1794 attests. However, until the beginning of the 19th century, the design of steels was only based on empirical elements.

In 1868, in a paper to the Russian Technical Society entitled *The Structure of Steel*, Chernov, an engineer at the Obukhov steel mills in St. Petersburg, demonstrated the existence of critical temperatures for the transformation of steel in the solid state. He defined the critical temperature from which it is necessary to heat a steel if one wants to harden it by rapid cooling. This was an important step forward, as Chernov’s work allowed Johan Brinell to describe, in 1885, the mechanism of hardening by cooling, as a process either to conserve “hardening carbon” as “hardening carbon” (for high cooling rates) or to convert “hardening carbon” into “cemented carbon” (for low cooling rates). The “cemented carbon” was identified in 1881 and 1888 by Abel and Muller as the compound Fe_3C . Floris Osmond gave it the name *cementite* a few years later. Moreover, Osmond’s work is of major importance. In 1885 and 1887, he established the importance of the different states of carbon and showed the existence of two allotropic varieties of iron: α iron and β iron. He attributed the hardening properties to the ability of the steel to retain the stable high-temperature phase β at room temperature, which he called “hardenite”. However, the high-temperature β -phase and the low-temperature β -phase cannot have the same composition and structure, as one is non-magnetic and the other magnetic. To solve this contradiction, Albert Sauveur proposed to look at the high temperature phase as a solid solution and not as a defined compound, this solid solution being able to decompose during cooling into carbides dispersed in a magnetic ferritic matrix. This was an important step, because the hardening of steel was no longer seen as a process of retention of the high-temperature phase β , but as the decomposition of the latter.

In 1897, Le Chatelier presented the theory developed jointly with Osmond and concluded that above 900°C, steel is composed of a homogeneous non-magnetic solid solution of carbon in γ iron. On slow cooling, this solution behaves like an aqueous solution containing a eutectoid with 0.8% carbon, named pearlite. By rapid cooling, the formation of pearlite is avoided and a magnetic solid solution is formed. This solid solution will be named martensite in honor of the metallographer Adolf Martens and γ iron austenite by Osmond. Meanwhile, a first Fe-C phase diagram was proposed by Roberts Austen in 1895. By applying the phase rule explained by Gibbs in 1878, Roozeboom proposed, in a 1900 work entitled *Iron and steel from the point of view of the phase doctrine*, an Fe-C diagram very similar to the one used today. By the end of the 19th century, the mechanisms of hardening of tempered steels could be considered to have been elucidated.

At the same time, large-scale steelworks based on the Bessemer–Thomas or Siemens–Martin process were operating efficiently, alloying metals were available in large quantities, and demand was growing in fields as varied as armaments, railway construction, machine tools and energy. All the conditions for the transformation of the steel industry and the development of steel in the 20th century were met. As early as 1880, steel supplanted puddled iron. Interest in so-called special alloy steels quickly piqued, as the addition of alloying elements gave the steels remarkable properties, which were mainly used in the manufacture of shells, cannons and armor. From 1880 onwards, the steelworks concerned began to develop increasingly resistant special steels, fueled by a self-perpetuating process: the strength and toughness of the shells increased as the hardness of the armor plates increased and vice versa. Chrome steel shells were produced as early as 1882, and nickel steel armor as early as 1891. In terms of the development of alloy steels, we can mention the patent filed in 1882 by Sir Robert Hadfield for a revolutionary steel with 1.2% carbon and 12% manganese, the patent filed by the Creusot steelworks for the manufacture of ferrochromes and nickel stainless steels, an exhaustive description of which can be found in Léon Guillet’s work published in 1902 and entitled *Étude micrographique et mécanique des aciers au nickel*. In addition, the Americans Taylor and Maunsell White discovered self-hardening high-speed tool steels composed of chromium and tungsten in 1899, for which industrial production began in the United States in 1910.

The discovery of X-ray diffraction by Max von Laue in 1912 and the construction of the first transmission electron microscope (TEM) in 1932 by Knoll and Ruska accompanied the development of steel. This period marks the transition from empirical to scientific metallurgy. The concept of dislocation was introduced in 1934 by Orowan, Taylor and Polanyi. The role played by dislocations on plastic deformation, precipitation hardening, solid solution hardening and grain size

hardening was understood well before their direct observation in TEM by Hirsch and Whelan in 1952. The first criterion of rupture stress based on the theory of elasticity was published by Griffith in 1921 under the title *The Phenomena of Rupture and Flow in Solids*. This work, taken up by Irwin in 1948 and Orowan in 1949, was consolidated and gave the basis for modern fracture mechanics.

A better knowledge of the relationship between the microstructure and the properties of steels, industrial development and the needs of the post-war period led to the development of steels whose mechanical properties were constantly improved. At the end of the First World War, governments in Europe became aware that the steel sector was strategic. The automotive sector became the lifeblood of national trade and industry in the 1920s. In Europe, from 1930 onwards, steel was produced not only in ingots but also as rolled products, sheets, beams, rails, tubes, wires, wheels, fishplates and axles.

The 1970s marked a break in the development model for steels due to the combination of several factors. On the one hand, the rise in energy prices due to the oil crises of 1973 and 1979 had significant repercussions on energy consumption, production, transport and recycling costs; on the other hand, the first European standards relating to the emission of polluting and harmful particles by motor vehicles appeared as early as 1970. Finally, there was an increasing international awareness of environmental issues. In 1970, the Meadows report warned of the depletion of raw material resources and the Stockholm Conference in 1972 put ecological issues on the international agenda for the first time. Since then, these trends have become more pronounced: energy costs are higher, emission standards are increasingly stringent and governmental bodies are imposing financial penalties for CO₂ emissions. In this particular context, the lightening of steel structures has become an important issue for the steel industry. In an attempt to meet this challenge, the strategy chosen has been based mainly on reducing the thickness of the products developed. However, it requires the development of increasingly resistant steels, mainly for reasons of rigidity and/or impact resistance. In fact, at iso-energy absorption, any relative reduction in thickness must be compensated by a greater relative increase in mechanical strength. This paradigm shift is the basis for the development of advanced high-strength steels.

1.3. Steels, a continuous development

This trend, observed in many sectors such as automotive, packaging, construction and energy, requires the search for new compromises between mechanical strength and processing and usage properties.

We can mention the effort to lighten packaging steels. Over the last 20 years, the thicknesses of these steels have been reduced by an average of 33%. For example, the average thickness of a can has been reduced from 0.20 mm in 1986 to 0.13 mm today.

The automotive sector has also followed this trend. In the 1990s, the need for lighter weight led to the massive use of HSLA steels micro-alloyed with niobium and titanium. Their strength was around 750 MPa. Now, ultra-high strength steels (1500 MPa) are widely used. A macroscopic analysis shows that these steels currently represent more than 15% of the steels used in vehicles. The strength target for vehicles is now 2000 MPa. Different steel families have been developed to meet this high-strength requirement. Multiphase steels of the dual phase or TRIP type have good forming properties. They are mainly used for cold forming of complex parts. The strength levels reach 1200 MPa, with developments toward 1500 MPa. In the case of hot forged or stamped parts, bainitic or martensitic grades are mainly used. They cover the range from 1000 to 2000 MPa.

In the energy market, the evolution of in-use temperatures (an increase in temperatures in the context of thermal power plants, or a decrease in the context of fluids such as natural gas or liquefied hydrogen) requires that high-strength steels improve their properties such as high temperature resistance or cold fracture resistance (toughness). Examples are the bainitic or martensitic grades used for gas transport pipes or power plants. These grades are developed for their excellent fracture toughness. Special attention is given to their weldability, which may require a high reduction of carbon content. For use at very low temperatures (e.g. natural gas or hydrogen), steels with a high nickel content (7–9% wt) are suggested.

The automotive sector is a good example of the development of ultra-high strength steels. Since the 1990s, their commercial use has developed to meet the challenges of safety and weight reduction. Sales of these steels¹, with a maximum strength of over 450 MPa and up to 2000 MPa, represented 1.3 million tons in 2010 in Europe. With a double-digit annual growth rate, more than 4 million tons of ultra-high strength steels were sold in Europe in 2018. Considering that Europe accounts for about 20% of global automotive production, it is reasonable to estimate that the global figure of 20 million tons will be exceeded during the 2020s.

¹ From a commercial point of view, very high strength steels include multiphase steels (excluding HSLA), DP, TRIP, CP, ferrite/bainite, bainitic grades and martensitic steels. In 2018, the production of TWIP steels represents only a marginal percentage of the production of very high strength steels.

The years to come will open up new perspectives for the development of steels. It will be necessary to respond to environmental concerns (reduction of CO₂ emissions and increase in the use of recycled steel in steel production). As a material of choice in construction, steel will contribute to sustainability objectives in this field. New emerging manufacturing processes, such as additive manufacturing, also offer new opportunities for this material. Thus, the performance and progress of steels, validated by decades of experience, have excellent development prospects.

This book, dedicated mainly to high strength steels, is based on the trade-offs between mechanical strength and certain properties such as strain hardening, anisotropy, damage and fracture, fatigue resistance and endurance, corrosion and oxidation resistance, crash resistance, cut edge resistance, hydrogen resistance and weldability. Based on a review of the physical mechanisms underlying the various properties sought, this book should provide a better understanding of the metallurgical developments in terms of microstructure, chemical composition and elaboration processes that have been necessary for the emergence of these new generations of steels. Thus, mainly intended for students and young engineers, this book also aims to propose an approach to development and innovation that may prove to be a useful guide for their future needs as materials specialists.