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Case hardening development review (2001-2020)

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ABSTRACT

Purpose: The purpose of the work is a complex review of methods applied industrially as case hardening. The paper contains an overview of scientific and development works on surface case hardening methods, especially carburising and quenching, described in the literature from 2001-2020.

Design/methodology/approach: State-of-the-art was reviewed by a critical review of the world literature published in 2001-2022, including theoretical work, scientific research, and industry reports. An additional examination of the state of the art was conducted in terms of patent works.

Findings: The period of 2001-2020 was a time of intensive work on the modernisation of case hardening techniques to improve the repeatability and uniformity of the produced layers and minimise deformations after hardening. Developing computing technologies have played a large part in this progress. New technologies have also been developed.

Research limitations/implications: The review of papers and patent databases was limited to databases providing English-language content options.

Practical implications: Case hardening is a crucial stage of steel heat treatment in almost every industrial branch: mechanical, tool, automotive, railway, and aviation.

Originality/value: A synthetic review of case hardening methods was presented, particularly carburizing and quenching methods; it also analysed the possibilities and directions of their development.

Keywords: Heat treatment, Hardening, Quenching, Distortions, Carburizing, Simulation

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Heat and thermo-chemical treatment is an important area of global industry, constituting a critical production stage in almost every market sector: mechanical, tool, automotive, railway, and aviation (Fig. 1). The global heat-treating market was valued at USD 100.73 billion in 2021 and is expected to expand at a compound annual growth rate

(CAGR) of 3.4% from 2022 to 2030 [1]. The rapid growth of the electric vehicle industry and the growing demand for metallurgical alterations to suit specific applications is expected to boost market growth in the coming years. Hardening processes, particularly *case hardening*, dominate the world of heat treatment [2-7].

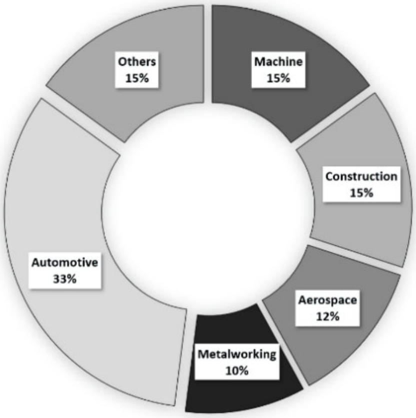

Fig. 1. Global heat-treating market share, by application, according to Grand View Research, 2021 [1]

The surface hardening process (including *case hardening*) is a process in which only the surface layer of the object is subject to modification of the structure and properties. Its market and application significance have been poorly described in the literature of the last twenty years because, in science, it is perceived as a set of conventional technologies. Therefore, reports on the importance and impact of surface hardening come mainly from the industrial literature [1, 8-20] and from the analysis of the patent status in patent databases of various countries (patents selection of the last twenty years related to case hardening is presented in Tab. 1).

2. Case hardening process

The *case hardening* process most often consists of the process of saturating the surface layer with an element capable of creating interstitial hardening in the crystal lattice of the iron alloy (carbon, nitrogen, boron) and heat treatment (quenching). However, the quenching may occur before (in conjunction with nitriding or boriding) or after the saturation step (in conjunction with carburising).

2.1. Carburising

Carburizing is heating the material in a carbon-bearing medium's environment at the temperature of the existence of homogeneous austenite for the time needed for carbon diffusion to occur in the material and the necessary thickness of the layer to be formed. Although gas carburising is mainly used in industrial conditions [8], carburising carried out in a reduced pressure environment has a significant advantage over carburising in an endothermic atmosphere because it protects the material against oxidation at the grain boundaries, ensures excellent surface quality, and allows to shorten the duration of the process. In the 21st century, many researchers point to the technological advantages of vacuum carburising over atmospheric carburising [21-24]. As a result, in the last twenty years, the vacuum carburising market has increased from 3% to 15%, and publications on low-pressure carburising prevail in the literature [5, 25-37].

2.2. Nitriding

Nitriding is the second type of diffusion saturation process with far-reaching applications in *case hardening*. During this process, the surface layer of a metallic object (iron and its alloys, titanium) is enriched by diffusion with nitrogen. The most crucial property of nitrided layers is their high hardness, which determines high resistance to sliding, abrasive and erosive wear. In another variation of nitriding, the goal of the process is to achieve surface corrosion resistance. The emergence of nitriding methods in the first half of the 20th century was a response to the shortcomings of the then-carburizing methods. The nitriding process developed by A. Fry was introduced into industrial practice in the early years after World War I. It was successful in the aviation, textile, railway, automotive, and machine industries [38-41]. Compared to carburising, nitriding was carried out at a temperature about 400°C lower than the carburising temperature. The steel, after nitriding, had a hardness of about 300-400 HV, with no deformations during processing.

The alloy is hardened at process temperature and does not require a subsequent quenching process. In addition, in industrial practice, there is a belief that nitriding is much simpler than carburising in terms of implementation. The advantages of nitriding were already described in the German literature in the 1920s; among them, the high resistance of the nitrided layers to wear, abrasion, corrosion, and high surface compressive strength were emphasised. At that time, the long process time (up to several dozen hours) was perceived as a disadvantage [41]. Alternatives to nitriding carried out in gaseous conditions quickly appeared, i.e., salt nitriding and ion nitriding. Although nitriding in salts is not developed in the 21st century for ecological reasons, ion nitriding is still being developed [42-47]. Since the 1960s, low-pressure nitriding has also been developed as the concept of low-pressure nitriding has seen the potential for better control of the nitrogen stream into steel. In 1966~ A. Smirnov and J. Kulesov presented an equilibrium diagram of ammonia dissociation degree – pressure – solid phases of the iron-nitrogen system and showed that lowering the pressure below atmospheric pressure shifts the equilibrium towards the ’ and , phases, containing more nitrogen than the internal impregnation layer [48]. Work on this concept is continued today and has given rise to low-pressure nitriding technologies, such as the FineLPN technology [49,50]. In addition, in the field of gas nitriding, in the early 2000s, gas nitriding with the economical use of process gas was developed, which is a variant of atmospheric nitriding, strongly focused on the economic and ecological aspects of its use [51-54].

In conclusion, nitriding still plays a vital role in many industrial applications in the 21st century. With the derivative process of nitrocarburizing, it is often used in producing aircraft parts, bearings, automotive components, textile machines, and turbine systems, i.e., in making machine elements and tools with heavily loaded friction nodes and cyclic loads. Variations of nitriding used in practice are mainly gas nitriding under atmospheric pressure [55-61], ion nitriding [62-64], laser nitriding [65], low-pressure nitriding [49,50, 66-68], and obsolescent fluidized bed nitriding [69-71] and bathing (salt nitriding). In the context of research conducted in the 21st century, mainly focused on reducing costs (consumption of operating gases or time) are controlled nitriding, nitriding with the economical use of ammonia, and nitriding under reduced pressure [58,59].

2.3. Boriding

Initially, the term boriding was defined as the enrichment of the material's surface with boron through thermo-chemical treatment. Using thermal energy and chemical reactions, boron atoms were adsorbed on the surface, introduced into the base material lattice, and then diffused towards the core, forming borides with the substrate atoms. However, the intensive development of boron techniques meant that the term was extended to other physical methods where boron diffusion was less critical [72,73]. Currently, boriding is used to increase the hardness, resistance to abrasive wear, and corrosion in aggressive environments to increase heat resistance. This process consists of the diffusion of boron atoms into the crystal lattice of the substrate material, which causes a hard interstitial boron compound to form on the surface. Boriding plays an essential role in modern production technologies and is mainly carried out in two ways: by liquid electrolysis or gas boriding [73,74]. There are also works on the method of laser boriding [73,75,76]. Boriding allows obtaining a uniform hard layer over the entire depth from the surface to the end of the diffusion layer. The hardness obtained is often higher than with any other surface hardening process. The combination of high hardness and low coefficient of friction improves wear, abrasion, and surface fatigue properties. Other benefits of boron deposition are the retention of high hardness at elevated temperatures, corrosion resistance in acidic environments, and reduced lubricant consumption.

2.4. Quenching

Quenching is a heat treatment that occurs in *case hardening* together with carburizing, nitriding or boriding. It consists in heating the steel to a temperature of about 30-50°C higher than Ac_3, heating the elements in it for the time necessary for the austenitic transformation to occur, and then quickly cooling in the quenching medium. The hardening of the workpiece must be done at a rate that ensures the expected structure of the steel. In the case of martensitic quenching, the cooling process must be carried out at speed higher than the critical speed for a given material so that no other phase transformations take place to the martensitic transformation temperature and that the supercooled austenite can transform into martensite only at a temperature below the martensitic transformation temperature. Therefore, proper quenching requires taking into account a number of process parameters because each of them – the type and properties of the cooling media, surface, shape, and weight of the cooled elements – has a significant impact on the final effect [77].

3. Geometric deformations

According to the International Federation for Heat Treatment and Surface Engineering and the American Society for Metals, the global issue of heat treatment at the beginning of the 21st century was defined by four points [12, 78-80]:

geometric deformations after heat treatment, methods of their control and reduction, and quality control of the product after heat treatment;
optimal selection and use of cooling media;
development and implementation of modelling and simulation technologies supporting heat treatment, in particular, thermo-chemical;
reduction of energy costs of heat and thermo-chemical treatment.

Regarding *case hardening*, trends 1-3, in particular, have been reflected in the literature of the last two decades.

Dimensional and shape changes that occur during the manufacturing of metallic components cause high additional costs because they give rise to reworking or even scrap. According to a 1995 survey of the Verband Deutscher Maschinen- und Anlagenbau, in the area of power-transmission technology alone, the costs for removing distortion totalled 850 million Euros/year in Germany. To minimise these costs, controlling distortion is one of the greatest challenges in modern economic production. It is gaining importance in current trends of downsizing or lightweight construction [81]. Many researchers have thoroughly analysed the factors affecting the internal stresses in the element after heat treatment. A comprehensive dependency diagram was presented by T. Lubben et al. [82].

The complexity of the issue of stresses and strains has meant that in the last twenty years, a broad literature has been devoted to deformations while preventing deformations is inextricably linked to their prediction. This became possible at a satisfactory level after the dissemination of numerical simulations. The main problem in this area is the shortage of materials databases and their properties on which simulations are based. Still, where these data are available, numerical calculations well illustrate the phenomenon of hardening geometric deformations [81, 83-86]. Because deformations are an element that cannot be eliminated in the production of equipment and machine parts, the work on understanding and predicting deformation phenomena is expected to be a permanent trend in heat treatment research. An interesting direction may appear in the single-piece flow method (Fig. 2), focused on a single part, which often improves the precision and repeatability of heat treatment results and reduces hardening deformations [7,83, 87-89].

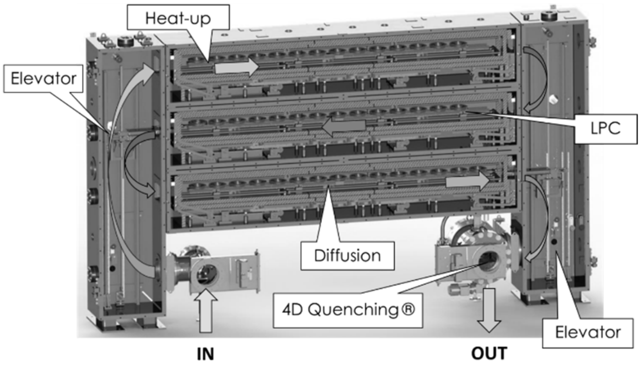

Fig. 2. Single-piece flow heat treatment furnace concept [7]

4. Cooling media role

The exact beginning of humanity's interest in cooling media and their proper selection is difficult to determine. Nevertheless, it is undoubtedly associated with the beginnings of heat treatment of iron alloys around 4th century BC.

The first petroleum-based quenching oils were developed around 1880. Previously, many types of oils of natural origin were used, including vegetable, fish, and animal oils, especially sperm whale oil. However, since then, many advances have been made in developing quench oils to provide highly specialised products for specific applications. High-quality quenching oils are formulated from refined base oils with high thermal stability. Selected wetting agents and accelerators are added to achieve specific toughening properties. Additions of antioxidants are also used to maintain performance over long periods of continuous use, and emulsifiers for easy cleaning after quenching [90]. Petroleum-based quenching oils are divided into several categories depending on the performance requirements (quenching rates, operating temperatures, and ease of removal) [91,92]. Normal-speed quench oils have a relatively low heat transfer coefficient and are used in applications where the material being quenched has a high hardenability (high alloy steels or tool steels are typical examples of steels hardened in normal-speed oils). Medium-speed quench oils provide intermediate quench characteristics and are widely used in medium to high-quench applications where reliable, consistent metallurgical properties are required. Rapid quench oils are used in applications such as low hardenability alloys, carburised and carbonitrided components, or large sections of medium hardenable steels with high cooling rates to ensure maximum mechanical properties. Between 2001 and 2020, extensive literature on oil quenching was developed [18, 90-99].

However, with increasing environmental, disposal, safety, and toxicological concerns, there is an increasing interest in using alternative quenching technologies. One of the most commonly considered alternatives to quench oils is aqueous solutions of water-soluble polymers. In addition to providing substantially greater safety with respect to fire and disposal, polymer quenchants have been shown to provide more uniform heat removal during quenching resulting in reduced thermal gradients and reduced distortion. The potential of polymer hardenings was already recognised at the beginning of the 21st century, and they are increasingly used in the heat treatment industry, displacing classic oil hardening [100-104]. There are also reports of the development of water-cooling methods that could result in cooling rates several times faster than conventional quenching. While this is an exciting curing option, information on the industrial use of this method in 2001-20 is scarce [105-112].

Gas as a cooling medium appeared together with the technology of vacuum heat treatment. At first, it accelerated the cooling of the charge through natural convection and thermal conductivity. With the development of technology, cooling forced by gas circulation at increased pressure in a closed system began to be used, which multiplied the speed of gas cooling. However, it was still slower than oil quenching until about 2000 [113]. Gas quenching rates currently achieved are comparable to medium-speed quench oils. The essential advantage of gas quenching in relation to oil quenching is the possibility of reducing quenching strains; hence much attention is devoted to research on increasing the rate of gas cooling [114-121].

5. Modelling and simulation technologies

Modelling and simulation publications are a particularly dynamic trend in the 21st-century heat treatment literature. This is related to the systematic increase in the computing power of computers and the development of software for modelling thermal and thermochemical phenomena, as well as the measurable economic benefit of the transition from experimental research to research using computer calculation methods. There is extensive literature on modelling carburizing [34,66, 122-129] and nitriding [128, 130-136]. In many cases, its added value was not the modelling itself but the optimization of the course of phenomena, which was challenging to achieve through manual iterations.

In the case of quenching, from the point of view of calculation precision, the most critical parameters of the quenching model are the coefficients determining the heat capacity of the cooled element, which result from its physical properties, and then the ability to effectively dissipate heat (resulting from the development of the element surface) and the conditions hardening. The above coefficients make it possible to calculate the cooling rate of the charge, which in combination with the carbon concentration in the material, makes it possible to determine the hardness of the surface layer. Numerous works have been devoted to all these coefficients [99, 136-141]. Because the analytical mathematical formulas of such complex physical phenomena are difficult to solve in an acceptable time, many researchers report that in the study of the properties of steel after thermo-chemical treatment, they stopped using analytical calculations in favour of FEM software, such as HEARTS [142], ABAQUS [143], SYSWELD [144,145], DANTE [91,139,140,146,147], COSMAP [148,149] and DEFORM-HT [84, 150-154]. Emphasising the usefulness of numerical methods [122,123,148, 153-161], researchers also point to the growing use of artificial intelligence methods in this issue [126,131,132, 162-176]. Probably the greatest attention in heat treatment modelling is devoted to the modelling of hardening stresses [83,84,144, 177-184] and strains [134,148,181, 185-193]. However, even in commercialised computational programs, some problems still remain without a satisfactory solution [148].

6. Future challenges

The last twenty years have been necessary for developing *case-hardening* technologies. The technologies developed in this period are now more ecological and economical. This is an essential step in today's economy, where the anthropological carbon footprint and energy and consumables savings are significant when undertaking new economic investments [194-196]. Implementing various heat treatment technologies is expected to be modified towards increasing the share of automation and robotization (Industry 4.0, Industry 5.0) [197-200]. It is also expected that the share of numerical calculations will increase toward creating digital copies of machine parts and devices at the design stage (so-called digital twins) [197]. At the same time, however, it seems that all the problems of *case hardening* identified at the beginning of the 21st century have remained valid, and none of them has been completely eliminated; in particular, the issues of stresses and strains after hardening remains at the centre of interest of researchers and industry. It is reasonable to expect that work on these problems will continue, although the methodology of solutions will be different.

Patents

Table 1.
Patents of the last twenty years related to case hardening

Year	Authors	No and year	Country	Ref.
2000	Williams et al.	US 6,093,303	USA	
2001	Waka et al.	US 6,187,111	USA	[201]
2001	Garg et al.	US 6,287.393	USA	
2002	Yamaguchi et al.	US 6,431,761	USA	
2002	Pelissier	US 6,451,137	USA	
2002	Williams et al.	US 6,461,448	USA	
2003	Williams et al.	US 6,547,888	USA	
2005	Kawata et al.	US 6,846,366	USA	
2006	Poor et al.	US 6,991,687	USA	
2006	Poor et al.	US 7,033,446	USA	[202]
2006	Kula et al.	EU 1,558,781	EU	[203]
2006	Fett	US 2006/0266436	USA	[204]
2007	Chin et al.	US 7,186,304	USA	

Table 1. *cont.*

Year	Authors	No and year	Country	Ref.
2007	Poor et al.	US 7,204,952	USA	[205]
2007	Eiraku et al.	US 7,217,327	USA	
2007	Kula et al.	EU 1,558,780	EU	[206]
2007	Poor et al.	US 7,267,793	USA	[207]
2008	Ishii et al.	US 7,326,306	USA	
2008	Somers et al.	US 7,431,778	USA	
2008	Tipps and Byrnes	US 7,468,107	USA	[208]
2009	Kuwabara et al.	US 7,622,009	USA	
2009	Obhayashi and Okada	US 2009/0266449	USA	
2009	Kula et al.	US 7,550,049	USA	[209]
2009	Kula et al.	US 7,513,958	USA	[210]
2010	Hammond et al.	US 7,648,588	USA	
2010	Kuwabara	US 7,655,100	USA	
2010	Imbrogno et al.	US 7,794,551	USA	
2010	Ishii et al.	US 7,811,390	USA	
2010	Collins and Williams	US 2010/0116377	USA	[211]
2011	Iwasaki et al.	US 7,887,747	USA	
2011	Jo and Kang	US 8,137,482	USA	[212]
2013	Moyer	US 6,425,691	USA	[213]
2014	Foerster et al.	US 8,828,150	USA	[214]
2014	Obayashi et al.	US 8,733,199	USA	[215]
2014	Somers and Christiansen	US 8,784,576	USA	[216]
2016	Korecki et al.	EP 16000164.0	EU	[217]
2018	Korecki et al.	US 10,072,315	USA	[218]
2018	Korecki et al.	US 9,989,311	USA	[219]
2020	Korecki et al.	EP 3006576	EU	[220]

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