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Professional paper

EFFECT OF PRECIPITATION HARDENING ON MICROSTRUCTURE OF 17-7 PH STEEL WITH MODIFIED CHEMICAL COMPOSITION

Belma Fakić, Derviš Mujagić, Omer Beganović

University of Zenica, Institute "Kemal Kapetanović"

ABSTRACT

Steel 17-7PH is austenitic-martensitic steel with high strength, hardness, and resistance to creep, and corrosion. It is designed for aerospace components, but can also be used for other applications that require high strength and corrosion resistance, as well as leaf springs for operation at temperatures up to 316 °C. It can be used in a solution-treated or heat-treated state to obtain a wide range of property values. This paperwork shows that modification of the contents of alloying elements with a narrower interval of Cr, Ni, and Al can be obtained from austenitic-martensitic steel 17-7PH which by, a subsequent heat treatment, can have values of mechanical and chemical properties required for components of an automotive engine. Chromium is an alphagenic alloying element that stabilizes the ferrite region, nickel is a gammagenic alloying element that stabilizes austenite and gives these steels good strength and toughness, even at low temperatures and aluminum increases corrosion resistance in low-carbon corrosion-resistant steels Research has determined the most suitable interval of Cr, Ni, and Al, which in combination with the cryogenic heat treatment RH950 at -50 °C gives the mechanical and chemical properties that meet the requirements for steel with standard chemical composition.

Keywords: Heat treatment, mechanical properties, microstructure, precipitation-hardened semi-austenitic stainless steel

Corresponding Author:
Belma Fakić,
University of Zenica, Institute "Kemal Kapetanović" in Zenica
Travnička cesta 7, 72000 Zenica, B&H
Tel.: +387 61 588 883; fax: +387 32 247 980.
E-mail address: belma.fakic@unze.ba

1. INTRODUCTION

Thanks to the complex chemical composition (alloying with aluminum, molybdenum, etc.) and heat treatment, precipitation-hardened steels are a great challenge for metallurgists. Research into different combinations of the chemical composition of materials and heat treatment temperatures provides opportunities for the development of existing and creation of new types of these steels, but also for obtaining high-performance materials with lower production costs and a wider field of application.

2. PRECIPITATION-HARDENED STAINLESS STEELS

Designers of stainless steel products are faced with making tradeoffs between the properties needed for manufacturing and those required for its end use. Precipitation-hardened (PH) stainless steels are iron-chromium-nickel alloys with one or more precipitation-hardening elements such as aluminum, titanium, copper, niobium, and molybdenum. PH stainless steels were developed as a material for the aviation and space industry. But today they are gaining wider commercial importance because they are cost-effective and available in a wide

range of products (bars, wires, forgings, sheets, strips) [1]. These steels are characterized by a unique combination of high strength, toughness, good corrosion resistance, and ease of plastic processing [1].
PH stainless steels are available in one of two conditions – annealed (condition A) or tempered (condition C). The annealed alloys are relatively soft and formable. After forming parts can be age-hardened. Tempered alloys are passed through a rolling mill at room temperature to impart an element of cold work, usually 60 %. From this condition, the alloys can be heat treated to exceptionally high hardness levels and yield strengths in 1200 to 1790 N/mm^2 range. Condition C is the starting point for high-strength parts, but forming must be minimal and simple, with generous radii [2].
PH stainless steels are divided into three groups [1,3]:

- martensitic – 17-4 PH (AISI 630), 15-5 PH, PH 13-8Mo;
- austenitic – A-286 (AISI 600), 17-10 P, HNM;
- semi-austenitic – 17-7PH(AISI 631), PH 15-7Mo, PH 14-8.

The position of PH stainless steel in the Schaeffler-Delong diagram is given in Figure 1.

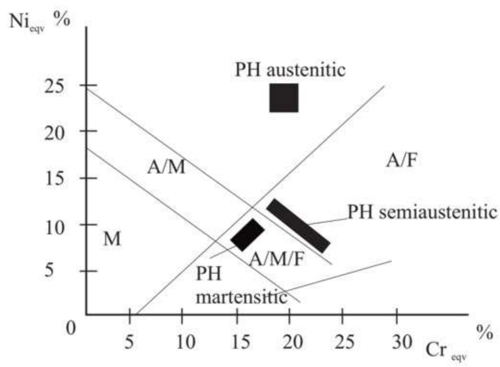


Figure 1. Position of PH steel in the Schaeffler-Delong diagram [3].

3. SEMI-AUSTENITIC PRECIPITATION-HARDENED STAINLESS STEELS

Semi-austenitic stainless steels are precipitation-hardened alloys in which heat treatment can achieve a wide range of required property values. These alloys are used for their combination of high strength, high toughness, and corrosion resistance. Semi-austenitic stainless steels, however, have low fracture toughness in high-strength conditions and a low (subzero) temperature.
The semi-austenitic stainless steels are variants of common austenitic stainless steels with additions of alloying elements such as aluminum and molybdenum. Table 1 lists the approximate composition specification ranges for several common semi-austenitic stainless steels [4].

Table 1. Compositions (wt %) of several common semi-austenitic (precipitation-hardened) stainless steels [4]

Steel		C	Si	Mn	Cr	Ni	Mo	Al
S 17700	17-7PH	max 0.09	max 1.00	max 1.00	16.00-18.00	6.50-7.75	---	0.75-1.50
S 15700	15-7PH	max 0.09	max 1.00	max 1.00	14.00-16.00	6.50-7.75	2.00-3.00	0.75-1.50
S 14800	14-8PH	max 0.05	max 1.00	max 1.00	13.75-15.00	7.75-8.75	2.00-3.00	0.75-1.50
S 35000	AM-350	0.07-0.11	max 0.50	0.50-1.25	16.00-17.00	4.00-5.00	2.50-3.25	---
S 35500	AM-355	0.10-0.15	max 0.50	0.50 - 1.25	15.00-16.00	4.00-5.00	2.50-3.25	---

4. HEAT TREATMENT OF SEMI-AUSTENITIC PH STAINLESS STEELS

Heat treatment of precipitation-hardened (PH) stainless steel is carried out to achieve different levels of mechanical properties related to achieved microstructure. The first step in the heat treatment of PH stainless steels is solution annealing. The aim of solution annealing is to dissolve all secondary phases that may be present in the matrix phase to obtain γ–solid solution. Rapid cooling from the solution annealing temperature suppresses the phase transformation of the high-temperature phase into phases stable at low temperature, i.e. homogeneous compressed solid solution is obtained at room temperature [1,5].

The precipitation-hardened stainless steel can attain high mechanical properties and good resistance [6].

Semi-austenitic precipitation hardened stainless steel 17-7PH is austenitic in the solution annealed state, while after the heat treatment of precipitation strengthening it is martensitic. Precipitation takes place from indirectly achieved martensite. The prefix "semi" means that the austenite in these steels is metastable rather than stable at room temperature [7]. Semi-austenitic steels can contain up to 20% delta ferrite after annealing in their predominantly austenitic microstructure [1].

4.1 Heat treatment of semi-austenitic steel 17-7PH

The influence of the solution annealing temperature on the formation of martensite during quenching is shown in Figure 2. In the case of 17-7PH steel, after quenching at a temperature of 1020 °C, an austenite-ferrite microstructure (60 – 90 % austenite) remains, which enables easier machining. The lower part of the picture shows that by cooling to temperatures of 50 °C and more degrees below zero, the residual austenite turns into martensite. Figure 2 shows that in the case of a higher temperature of solution annealing, the temperature of the beginning of martensite formation is lower. The austenite transformation into martensite is the first stage of strengthening of semi-austenitic stainless steel [8].

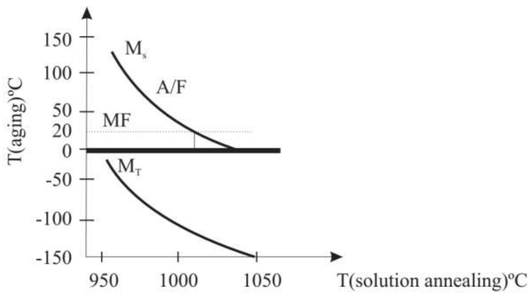


Figure 2. Effect of solution annealing temperature on the martensite formation during quenching [8]

The high strength of precipitation-hardened stainless steel 17-7PH is achieved in three steps: austenite conditioning, cooling below the critical temperature, which can be below zero, transforming austenite into martensite, and precipitation hardening.

Standard heat treatment steps for conditions TH1050 and RH950 are given in Table 2.

Table 2. Heat treatment steps for conditions TH1050 and RH950 [9]

Treatment	State RH950	State TH1050
Solution annealing State A	Austenite conditioning Heating at 955 °C, holding for 10 minutes, cooling at the air to room temperature	Austenite conditioning Heating at 760 °C, holding for 90 minutes, cooling at the air to room temperature
	Transformation Within one hour, start cooling to -75 °C, hold for 8 hours, and heat in air to room temperature	Transformation Within one hour, start cooling to 15±3 °C, hold for at least 30 minutes
	Aging Heating at 510 °C, holding for 90 minutes, cooling at the air to room temperature	Aging Heating at 565 °C, holding for 90 minutes, cooling at the air to room temperature
	Tensile strength after aging	Tensile strength after aging

1450-1650 N/mm^2	1240-1450 N/mm^2

Solution annealing involves heating the material to a temperature at which all elements are dissolved in a solid solution [1]. In 17-7PH steel, by solution annealing at temperatures around 1050 °C, all the carbon goes into solid solution (giving the austenite stability as in type 301 steel). During solution annealing, aluminum dissolves. After cooling, aluminum remains in solution. Since the steel at this stage has an austenitic microstructure, it means that it is relatively soft, ductile and easily formed into the required shape and dimension. Annealing results in the precipitation of $M_{23}C_6$ carbide, which is a deliberate increase in the sensitivity of the alloy. The increase in sensitivity is the consequence of a decrease in the chromium content around the precipitated carbides, which causes an increase in the M_s temperature due to a lower carbon and chromium content in the austenite solution. Depending on the temperature at which the annealing was carried out, the M_s temperature can be controlled so that the transformation to martensite can start at temperatures close to room temperature or at temperatures below zero. The lower level of strength achieved in the TH condition reflects the lower carbon content of the martensite.

4.2 Aging

Aging is a relatively low-temperature heat treatment that hardens the material by precipitation of secondary phases from a supersaturated solid solution [1]. The first stage of hardening of steel 17-7PH is the transformation of austenite into martensite. At solution annealing temperatures, the steel is in the austenitic region. The solubility of carbon in γ-iron is significantly higher than in α-iron. During rapid cooling, γ phase transforms into the phase. Due to the high cooling rate, carbon is unable to leave the BCC lattice (Body Centered Cubic Lattice) of the α phase, which is why the BCC lattice is deformed into a BCT lattice (Body Centered Tetragonal Lattice). This tetragonal α phase is martensite, a supersaturated carbon solid solution in α-Fe, which has greater hardness and strength than austenite. Steel 17-7PH has a low carbon content, so the martensite formed by hardening is soft.

Martensite hardening is the result of the following effects:

- very pronounced solvent hardening,
- high density of dislocations or doubles,
- fewer sliding systems in the BCT lattice compared to the BCC lattice,
- crushing of martensite tiles and
- formation of appropriate precipitates.

4.2.1 State of TH1050

During austenite conditioning at 760 °C, chromium carbides precipitate at grain boundaries or in other high-energy areas, e.g. on slip planes. By reducing the effective content of carbon and chromium in austenite, precipitation leads to transformation during cooling. After treatment at 760 °C, only 0.016 % C remains in the solid solution [1].

Austenite transformation into martensite starts at around 95 °C, and the reaction continues by lowering the temperature and ends by holding for 30 minutes at 15 °C. It is important to note that the cooling from 760 °C to 15 °C should be carried out within one hour, to complete the transformation.

Aging is an additional increase in hardness and strength and is achieved by aging the transformed material by precipitation of secondary phases and additional precipitation of carbides. Hardening during aging reaches a maximum at 510 °C, but is accompanied by minimal ductility. Thus, the heating goes up to 565 °C, where the strength value is slightly lower, but the ratio of strength and ductility values is improved..

4.2.2 State of RH950

After austenite conditioning at 955 °C, 0.034 % of carbon remains in a solid solution. The result of this treatment is an increase in temperature of M_s close to room temperature. Thus, the material conditioned at 955°C retains the austenite microstructure by cooling to room temperature, so it must be transformed with cooling to -75 °C. If the conditioning temperature is higher than 955 °C, martensitic transformation is suppressed,

so cooling to -75 °C will not lead to complete transformation into martensite.
Austenite transformation after conditioning at 955 °C, cooling to -75 °C, and holding for eight hours at this temperature, starts the transformation of austenite into martensite. Most of the transformation occurs during cooling to -75 °C and the first hour at this temperature.
Aging is an additional increase in hardness and strength and occurs at 510 °C for one hour. Higher or lower aging temperatures give lower strength, but at higher aging temperatures, better ductility is obtained [1].

5. EXPERIMENTAL PART

The main subject is research on the effect of precipitation hardening on the microstructural characteristics of 17-7 PH stainless steel.

5.1 Chemical analysis

The prescribed chemical composition according to BAS EN 10088-5 standards and the achieved chemical composition of the steel batch are given in Table 3.

Table 3. Chemical composition [1]

Standard/Batch	Chemical composition, wt %							
	C, max	Mn, max	Si, max	P, max	S, max	Cr	Ni	Al
BAS EN 10088-5	0.04	0.52	0.53	0.009	0.023	14.5	7.8	1.53
V1783	0.05	0.55	0.51	0.011	0.029	15.6	7.4	1.18

5.2 Solution annealing

After finished plastic processing, the ϕ 16 mm bars were heat treated as follows:

- heating to a temperature of 1050°C for 115 minutes,
- heating to a temperature of 1050 °C for 115 minutes,
- hold to a temperature of 1050 °C for 30 minutes and cooling in the air.

5.3 Precipitation heat treatment

Precipitation hardening of bars was done according to the standard condition TH1050, and condition RH950 was performed by modifying the standard heat treatment as shown in Table 4.

5.4 Mechanical properties

The testing of mechanical properties at room and elevated temperatures was carried out on the Universal hydraulic machine for static tests - AMSLER, in the measurement range from 0 to 200 kN. The literature values of the mechanical properties for the conditions TH1050 and RH950 and the results of the mechanical properties test at room and elevated temperature are given in Table 5.

5.5. Metallography testing

As part of the metallographic tests, an analysis of the microstructure of the samples was carried out on an optical microscope OLYMPUS PMG3. After metallographic preparation (grinding and polishing), samples were etched in reagents for stainless steels.
The best results were shown by the Kalling reagent [10] with composition:

- 100 cm^3 of hydrochloric acid HCI,
- 5 g of copper II chloride $CuCI_2$ and
- 100 cm^3 of ethanol C_2H_5OH.

5.6. Microstructure evaluation

After etching all the samples are ready for microstructure examination. Microstructures are observed using an optical microscope. Figures 3 and 4 show the observed microstructure.

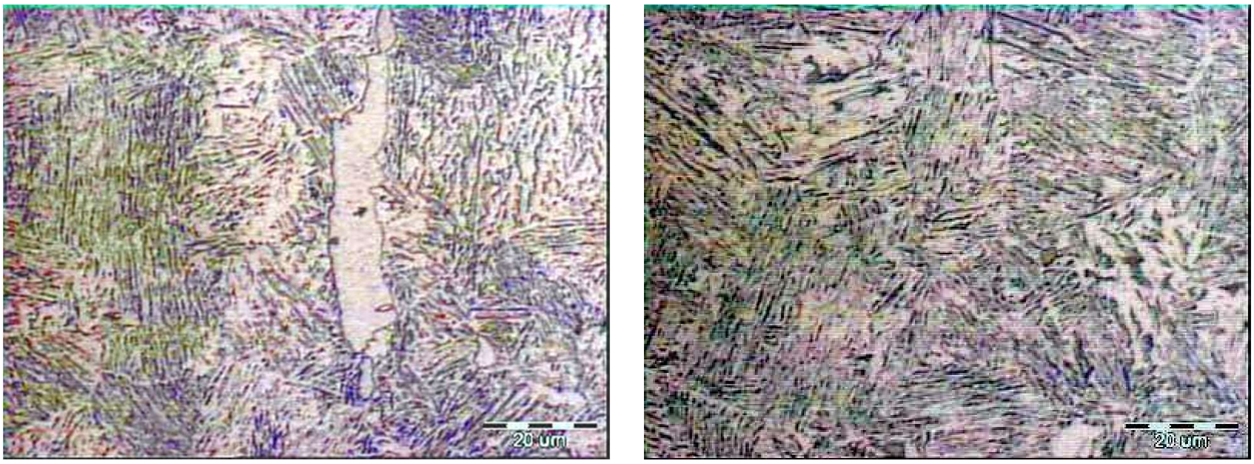
Table 4. Precipitation hardening [1]

Condition	Austenite conditioning	Transformation	Aging
TH1050	Heating up to 760 °C, 70 minutes Holding at 760 °C,	Within one hour, cooling to 15±3 °C, holding 30 minutes	Heating to 565 °C for 50 minutes holding at that temperature for 90 minutes and cooling in air

Condition	Austenite conditioning	Transformation	Aging
	90 minutes		
Modified RH950	Heating up to 955 °C, 90 minutes Holding at 955 °C, 10 minutes	Within one hour, start cooling to - - 50 °C in dry ice holding 8 hours	Heating to room temperature, heating to 510 °C for 45 minutes and holding at that temperature for 60 minutes, and cooling in air

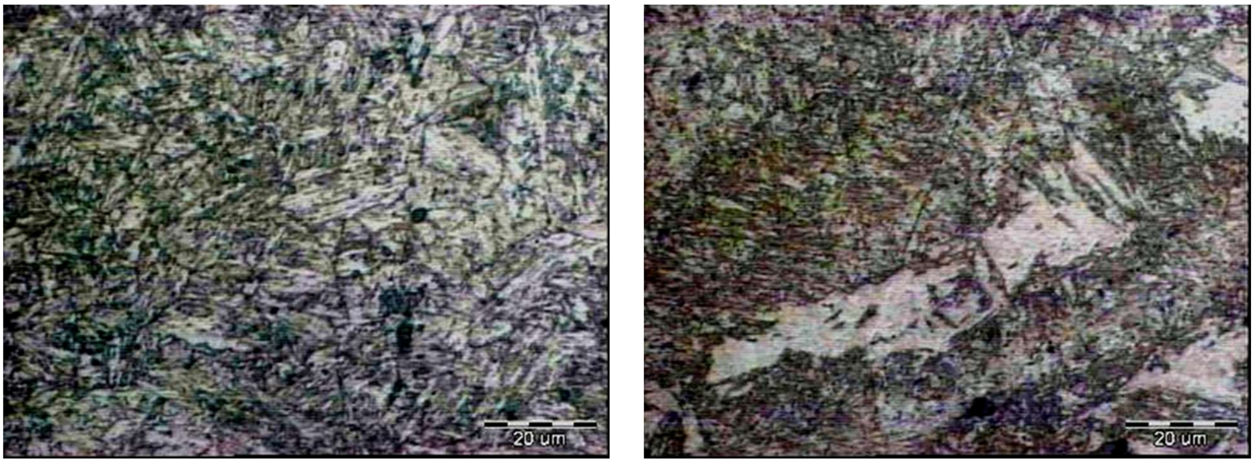
Table 5. Mechanical properties [1]

Literature/Batch	R_m [N/mm^2] Room temperature	R_m [N/mm^2] Elevated temperature 425 °C	Hardness HV 10
Metals handbook [10]	1170	-	255-361
AK Steel bulletin [11]	-	986	-
V1783 State TH1050	1419	923	437
Metals handbook [10]	1378	-	438
AK Steel bulletin [11]	-	1103	-
V1783 State RH950	1562	1100	541



Kalling x750 Longitudinal section
Kalling x750 Cross section

Figure 3. State TH1050, batch V1783; martensite, austenite and delta ferrite [1]



Kalling x750 Longitudinal section
Kalling x750 Cross section

Figure 4. State RH950, batch V1783; martensite, austenite, and delta ferrite [1]

6. CONCLUSIONS

The research aimed to determine the effects of precipitation hardening on the microstructure of 17-7PH steel with modified chemical composition. Steel 17-7PH with a modified chemical composition, with a lower chromium content and a narrower range of nickel and aluminum content, shows higher values of mechanical and chemical properties in the RH950 state compared to the TH1050 state. The modified heat treatment of precipitation hardening RH950 carried out at -50 °C achieved very good mechanical properties of steel with modified chemical composition. The amount of delta ferrite in the TH1050 state is about 5 % and retained austenite is about 1 5%. The amount of delta ferrite in the RH950 state is less than 5% and retained austenite about 19 %.

Conflicts of Interest

The authors declare no conflict of interest.

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