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Effect of microalloying with titanium on the properties of 17-4PH martensitic stainless steel

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Abstract. The 17-4PH (UNS S17400, 630) alloy is a martensitic stainless steel from the chromium-nickel-copper alloying system with the addition of niobium, in-situ strengthened by precipitation of intermetallic compounds. It is distinguished by superior mechanical properties and high corrosion resistance, being frequently used in applications requiring special functional performances in the aerospace domain, as result of its favorable ratio between mass and mechanical strength. The addition of elements such as Cu, Hf, Y, Ti, Zr to the standard chemical composition contributes to the microstructure refinement and the improvement of mechanical properties, with beneficial effects on the fulfillment of specific technological requirements and the increase of the service life.

In this work, Ti was added to the standard chemical composition in proportions of 1 to 5 wt%. The obtained batches were subjected to precipitation heat treatments, to study the effects on the microstructure, phase stability and on the mechanical strength characteristics. The experimental alloys were obtained by electric arc melting in the VAR ABJ 900 installation, using high-purity granular raw materials, under Argon protection. After melting, the micro-ingots were cut for sampling to perform precipitation heat treatments (annealing at 500 °C, holding for 4 hours and cooling in air followed by annealing at 600°C, holding for 4 hours and cooling in air), then microstructural analyses and microhardness measurements were performed. The effects of microalloying with Ti on the microstructure and the hardening effects resulting from the heat treatments were analyzed.

Keywords: martensitic stainless steel, microalloying, hardness, microstructure.

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1. Introduction

Martensitic precipitation-hardened stainless steels represent an important class of advanced alloys, widely used in critical applications that require high mechanical strength, structural durability and corrosion resistance [1, 2]. These alloys are notable for their ability to develop a hard martensitic structure combined with an efficient secondary strengthening mechanism, achieved through the controlled precipitation of dispersed intermetallic phases [3].

Among these alloys, 17-4PH stainless steel (UNS S17400) is one of the most used, due to its favorable balance of machinability, mechanical properties, and resistance to corrosive environments [4, 5]. The standard chemical composition includes approximately 17% Cr, 4% Ni, 4% Cu, along with Nb additions, an element that contributes to the formation of stable NbC-type phases, with direct effects on hardening and microstructural stabilization [6].

The final mechanical strength of this steel is obtained through a specific sequence of heat treatments, consisting of solution annealing followed by artificial aging, which enables the formation of fine copper-rich precipitates. As a result of these thermal treatments, a hardened martensitic microstructure is obtained, characterized by the uniform distribution of secondary phases, which significantly enhances yield strength, hardness and creep resistance [7, 8].

In recent decades, the mechanical and electrochemical performance of this type of steel has been further optimized through microalloying with small additions of various chemical elements. Titanium (Ti) is commonly used in stainless steels due to its ability to form stable interstitial compounds (TiC, TiN), which directly influence microstructural refinement and the stabilization of hard phases [9, 10]. Controlled addition of titanium can favorably influence the kinetics of martensitic transformation, reduce the amount of retained austenite and improving dimensional stability [11].

Recent studies indicate that titanium additions can influence the distribution and morphology of precipitates, leading to improvements in mechanical properties such as hardness and tensile strength [12, 13]. Titanium also acts as a fixative for interstitial impurities (carbon, nitrogen), thereby reducing susceptibility to intergranular corrosion by limiting the formation of unstable grain boundary phases [14].

However, the effects of titanium are not exclusively beneficial. At excessive concentrations, it may promote the formation of hard inclusions (TiN, Ti(C,N)), which can serve as initiation sites for stress-induced cracking, negatively affecting toughness and fatigue behavior [15]. Furthermore, non-uniform distribution of titanium-based compounds may result in structural heterogeneities, adversely impacting the alloy's functional properties [16].

Therefore, a detailed characterization of the effects of titanium microalloying on 17-4PH stainless steel is required, both from a microstructural perspective and in terms of mechanical and electrochemical performance. The analyzing of the relationship between titanium content, heat treatment parameters and the resulting

material properties is essential for establishing an optimized alloy composition tailored to industrial applications with specific performance demands [17–19].

The main objective of this study is to investigate the influence of controlled titanium microalloying on martensitic stainless steel 17-4PH. The research focuses on characterization of microstructural changes, assessing mechanical properties and evaluation of corrosion behavior using advanced analytical techniques. The results obtained may contribute to the development of new compositional variants of this alloy, featuring enhanced performance and improved stability under demanding operating conditions.

2. Materials and methods

2.1 Materials

In this study, the metal matrix used was a martensitic stainless steel of type 17-4PH, produced in accordance with the ASTM A564 standard. Experimental alloy batches microalloyed with titanium in mass percentages of 1%, 2%, 3%, 4%, and 5% were prepared in the ERAMET laboratory of the Faculty of Materials Science and Engineering, National University of Science and Technology POLITEHNICA Bucharest, with the aim to evaluate the influence of titanium content on the steel's microstructure and mechanical properties.

The target chemical composition of the steel and the mass of each element used are presented in Tables 1–5. The contents of Cr, Ni and Cu were maintained within the limits specified by the standard for 17-4PH steel, while titanium (Ti) was added gradually. The proportions of niobium (Nb) and tantalum (Ta) were kept constant at 0.3% for all experimental batches. Iron (Fe) was the base element, accounting for approximately 68–72 wt.% of the total alloy mass [19].

For this research, five steel batches were prepared and weighed, each corresponding to a different titanium addition level, ranging from 1% to 5% by weight, with each batch having a total mass of 25 grams. After dosing and weighing (with the chemical composition of each batch validated through charge calculation based on total mass), the raw materials were placed individually into crucibles machined into the base plate of the VAR ABJ 900 electric arc remelting unit, made of high-purity copper and equipped with a double-walled, water-cooled chamber.

Figure 1 shows the stages of the experimental process used to obtain metallic specimens in the form of buttons produced using the crucible plate. The procedure included: weighing the raw materials using an analytical balance with a precision of 0.01 g, arranging the chemical elements based on their behavior during rapid electric arc melting (to prevent vaporization losses), sealing the remelting chamber and performing vacuum evacuation, purging the working chamber with argon, and initiating the electric arc. The melting process was then carried out, with melting performed on both sides of each specimen, rotating them 5–7 times per side to

ensure alloy homogenization, followed by final solidification of the micro-ingots on the copper plate.

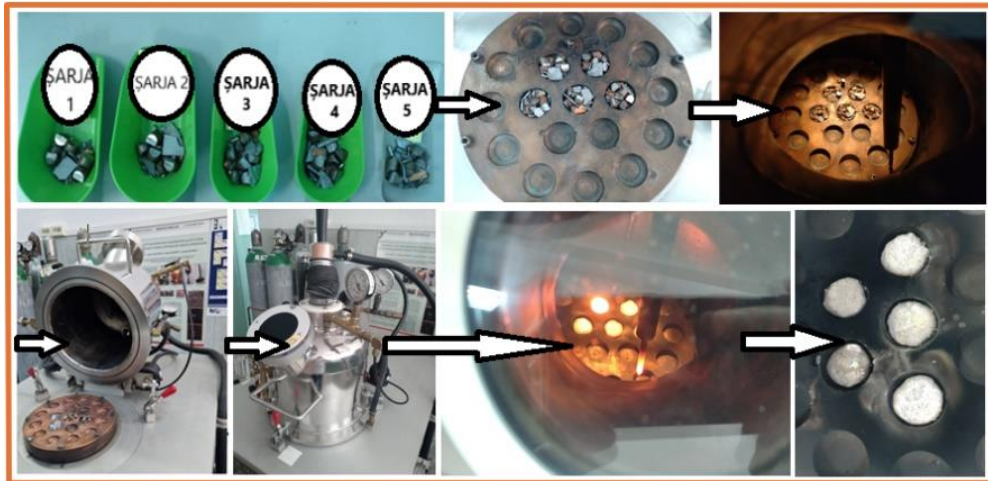


Fig. 1. Stages of the obtainment of experimental alloys by vacuum arc remelting using the VAR ABJ 900 system.

2.2 Methods

After obtainment, samples were weighed to evaluate the efficiency of the melting process. The flat surfaces of the samples were then prepared for chemical composition analysis using spectroscopy. Analyses were performed using a SpectroMaxx optical emission spectrometer in the ERAMET laboratory, as well as a BRUKER X-ray fluorescence (XRF) spectrometer.

The samples underwent standard metallographic preparation, which included grinding with progressively finer abrasive papers and polishing with alumina suspensions.

To reveal microstructural features, the polished surfaces were etched using Kalling's reagent, followed by examination under an Olympus GX 52 optical microscope equipped with AnalySIS image processing software.

The microhardness of the alloys was assessed using the Vickers method, with five successive measurements performed on each alloy type, using a Shimadzu HMV-2T microhardness tester.

Table 1. Chemical composition of 25g steel sample micro-alloyed with 1% Ti

Grade	SAMPLE 1												
17-4PH	Chemical composition, wt.%												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,07	15,0 - 17,5	3,0 - 5,0	3,0 - 5,0	1,0	1,0	0,15 - 0,45	0,15 - 0,45	0,03	0,04	1,0	balance	100
Steel / crucible	Mass, g												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,0175	4,0625	1,0	1,0	0,25	0,25	0,075	0,075	0,0075	0,01	0,25	balance	25

Table 2. Chemical composition of 25g steel sample micro-alloyed with 2 % Ti

Grade	SAMPLE 2												
17-4PH	Chemical composition, wt.%												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,07	15,0 - 17,5	3,0 - 5,0	3,0 - 5,0	1,0	1,0	0,15 - 0,45	0,15 - 0,45	0,03	0,04	2,0	balance	100
Steel / crucible	Mass, g												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,0175	4,0625	1,0	1,0	0,25	0,25	0,075	0,075	0,0075	0,01	0,5	balance	25

Table 3. Chemical composition of 25g steel sample micro-alloyed with 3 % Ti

Grade	SAMPLE 3												
17-4PH	Chemical composition, wt.%												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,07	15,0 - 17,5	3,0 - 5,0	3,0 - 5,0	1,0	1,0	0,15 – 0,45	0,15 – 0,45	0,03	0,04	3,0	balance	100
	Mass, g												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
Steel / crucible	0,0175	4,0625	1,0	1,0	0,25	0,25	0,075	0,075	0,0075	0,01	0,75	balance	25

Table 4. Chemical composition of 25g steel sample micro-alloyed with 4 % Ti

Grade	SAMPLE 4												
17-4PH	Chemical composition, wt.%												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,07	15,0 - 17,5	3,0 - 5,0	3,0 - 5,0	1,0	1,0	0,15 – 0,45	0,15 – 0,45	0,03	0,04	4,0	balance	100
	Mass, g												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL

Table 5. Chemical composition of 25g steel sample micro-alloyed with 5 % Ti

Grade	SAMPLE 5												
17-4PH	Chemical composition, wt.%												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
	0,07	15,0 - 17,5	3,0 - 5,0	3,0 - 5,0	1,0	1,0	0,15 – 0,45	0,15 – 0,45	0,03	0,04	5,0	balance	100
	Mass, g												
	C	Cr	Ni	Cu	Mn	Si	Nb	Ta	S	P	Ti	Fe	TOTAL
Steel / crucible	0,0175	4,0625	1,0	1,0	0,25	0,25	0,075	0,075	0,0075	0,01	1,25	balance	25

3. Results and discussion

3.1. Analysis of the role of main alloying elements in 17-4PH stainless steel

Carbon, maintained at a constant level of approximately 0.07% in all alloy batches, contributes to the formation of the martensitic structure and directly influences the final hardness of the steel [3].

Chromium is the primary ferrite-forming element, alongside iron and is present in a standard concentration of around 16.25%. It ensures excellent corrosion resistance and microstructural stability in various environments [4].

The austenite-stabilizing elements, which also contribute to precipitation hardening - nickel and copper - were both present at approximately 4.0%. Nickel stabilizes the austenitic phase during thermal processing, while copper promotes alloy hardening by intermetallic precipitates forming, particularly copper-rich phases that develop during aging treatments [5].

Microstructural stabilizers and hardening elements such as niobium and tantalum, each added at approximately 0.30%, promote the formation of stable intermetallic carbides (NbC, TaC). These compounds play a key role in dislocation pinning and grain size stabilization, thus contributing to increased mechanical strength and alloy durability [6].

Silicon and manganese, present with about 1.0%, primarily aid the steelmaking process by facilitating deoxidation and desulphurization, ultimately affecting the quality and final properties of the steel. In contrast, sulfur and phosphorus are undesirable elements in this type of steel; their content was kept at low levels ($\leq 0.04\%$) to prevent the formation of brittle inclusions and to avoid embrittlement phenomena in the material. Titanium is the key alloying element investigated in this study. It was introduced in varying concentrations (1–5 wt.%) to assess its influence on the microstructure and mechanical properties of the alloy. Titanium contributes at the formation of fine carbides and nitrides, which enhance hardness and provide high-temperature stability.

Titanium microalloying has the potential to reduce the fraction of retained austenite by promoting martensitic transformation during cooling, thereby improving dimensional stability and ensuring predictable in-service behavior of components. Regarding corrosion performance, the literature suggests that titanium may have a positive indirect effect by reducing the amount of carbon and nitrogen in solid solutions and contributing to the stabilization of the passive film [13, 14]. However, this aspect requires confirmation through specific electrochemical testing.

At higher concentrations ($>4\%$), titanium can lead to the formation of hard inclusions such as Ti (C, N) or massive TiN particles, which may act as crack initiation sites under load, negatively affecting toughness and fatigue resistance. Nevertheless, the use of titanium as a microalloying element remains an effective approach for improving the mechanical performance of 17-4PH steel, enabling

microstructural refinement, enhanced precipitation strengthening and superior hardness.

3.2. X-ray fluorescence - XRF analysis

Following the melting and solidification process, the chemical composition of each experimental steel sample was analyzed to evaluate the melting efficiency and assess the degree of alloy homogenization. The results obtained through spectrometric measurements are presented in Tables 6–10.

From the data shown in Tables 6–10, it can be observed that the titanium concentrations achieved in the samples are generally in good agreement with the target values proposed in this study, except for the Ti-5 sample, where the desired 5 wt.% Ti concentration was not reached. This deviation will be addressed and corrected in future experimental tests.

Table 6. Chemical composition determined by XRF spectrometry for sample 1, microalloyed with 1% Ti

Element	Concentration, %	Standard Deviation %
Fe	72,797	0,459
Cr	16,655	0,196
Cu	3,805	0,109
Ti	1,077	0,095
Ni	3,796	0,133
Mn	0,916	0,092
Si	0,266	0,060
Al	0,200	0,013
V	0,162	0,098
Ta	0,124	0,012
Nb	0,062	0,013
S	0,061	0,010
P	0,055	0,009

Table 7. Chemical composition determined by XRF spectrometry for sample 2, microalloyed with 2% Ti

Element	Concentration, %	Standard Deviation %
Fe	71,719	0,494
Cr	15,348	0,169
Cu	4,356	0,139
Ti	1,777	0,017
Ni	4,330	0,220
Mn	1,072	0,098

Element	Concentration, %	Standard Deviation %
Si	0,404	0,053
Al	0,245	0,050
V	0,219	0,015
Ta	0,151	0,013
Nb	0,106	0,013
S	0,051	0,009
P	0,049	0,034

Table 8. Chemical composition determined by XRF spectrometry for sample 3, microalloyed with 3% Ti

Element	Concentration, %	Standard Deviation %
Fe	71,222	0,496
Cr	14,724	0,167
Cu	4,322	0,122
Ti	3,162	0,022
Ni	4,098	0,136
Mn	0,973	0,096
Si	0,435	0,054
Al	0,254	0,050
V	0,243	0,162
Ta	0,230	0,016
Nb	0,136	0,013
S	0,100	0,013
P	0,054	0,009

Table 9. Chemical composition determined by XRF spectrometry for sample 4, microalloyed with 4% Ti

Element	Concentration, %	Standard Deviation %
Fe	70,352	0,490
Cr	14,667	0,166
Cu	4,404	0,121
Ti	3,523	0,022
Ni	4,253	0,135
Mn	1,025	0,095
Si	0,519	0,055
Al	0,301	0,055
V	0,241	0,048

Element	Concentration, %	Standard Deviation %
Ta	0,219	0,015
Nb	0,198	0,159
S	0,137	0,013
P	0,100	0,013

Table 10. Chemical composition determined by XRF spectrometry for sample 5, microalloyed with 5% Ti

Element	Concentration, %	Standard Deviation %
Fe	70,156	0,491
Cr	14,323	0,165
Cu	4,332	0,120
Ti	4,152	0,024
Ni	4,070	0,132
Mn	1,067	0,095
Si	0,418	0,053
Al	0,328	0,166
V	0,269	0,050
Ta	0,269	0,051
Nb	0,220	0,015
S	0,132	0,013
P	0,112	0,013

3.3. Microstructure

After solidification, the mini-ingots were subjected to a metallographic preparation process, which involved grinding with progressively finer abrasive papers (240, 400, 600, 800, and 1000 μm), followed by final polishing using an alpha-alumina suspension with a grain size of 0.8 μm for 4–6 minutes, in order to obtain surfaces suitable for subsequent microstructural analyses (Fig. 2, a).

The morphology of the martensitic phase, the presence and distribution of inclusions, as well as the potential effects of titanium microalloying on structural homogeneity were evaluated using optical microscopy. The samples were coded Ti-S x , with $x = 1$ to 5, corresponding to the titanium content.

The Kalling's reagent used for etching enabled the contrast enhancement between intermetallic phases and the clear delineation of grain boundaries [11]. The microstructures of the cast alloys are shown in Fig. 3.

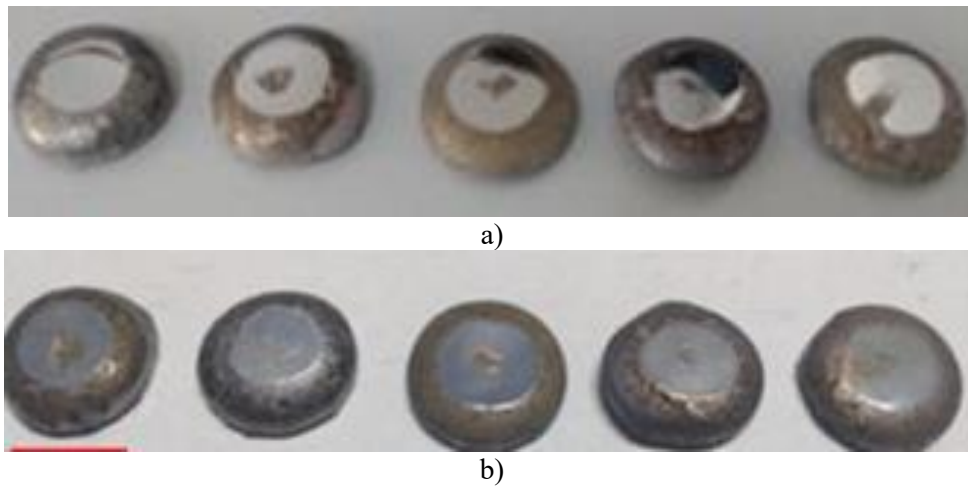


Fig. 2. Experimental 17-4PH alloy samples microalloyed with Ti. a) Samples prepared for optical microscopy analysis; b) Samples after etching with Kalling's reagent.

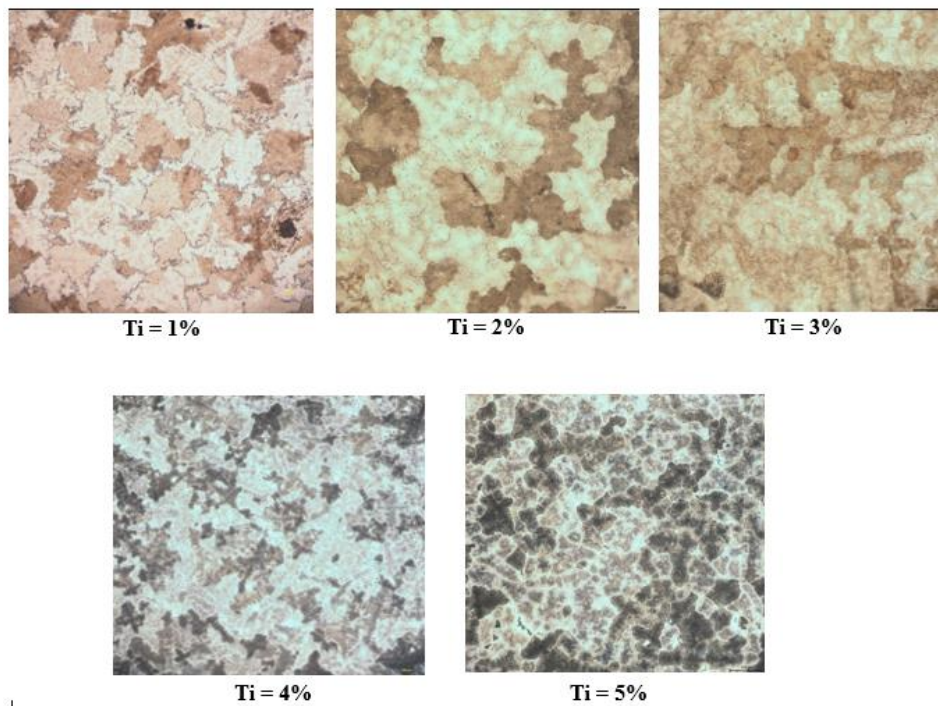


Fig. 3. As-cast microstructures of 17-4PH steels with different Ti concentrations (from 1 to 5%).

The microstructure of **Ti-S1** (1% Ti) sample shows a mixture of homogeneous martensite and ferrite, with fine grain size and no evident segregation (magnification 500X). A clear dendritic structure, typical of the solidification

process, is observed, with grain growth oriented along the most favorable cooling directions in the copper plate cooled by forced water circulation. Minor intergranular segregations and some larger precipitates, possibly nonmelted Nb particles, are visible. Titanium is largely dissolved in the matrix, while small amounts precipitated as carbides/nitro-carbides, which are unevenly dispersed.

The microstructure of **Ti-S2** (2% Ti) sample highlights the presence of small precipitates intragranular dispersed as well as along grain boundaries (500X). Relatively large dendritic grains with irregular margins, typical of as-cast microstructures, are observed.

In **Ti-S3** (3% Ti) sample, well-defined grains with visible internal nucleation are highlighted (500X). Additionally, the precipitation effect of secondary phases is more pronounced both inside the grains and along their boundaries.

Ti-S4 (4% Ti) sample shows a distinct intergranular network of intermetallic phases (500X). The dendritic structure and grain orientation along the cooling directions are typical of the as-cast condition.

The microstructure of **Ti-S5** (5% Ti) sample reveals a high density of fine precipitates with a tendency toward local coalescence (500X). The granular structure is much finer but more distinctly defined.

3.4. Microhardness

To assess the microhardness of the samples, Vickers microhardness measurements ($HV_{0.2}$) were performed using a load of 200 gf applied for 10 seconds. Each sample was tested at five distinct points, and the results are summarized in Table 11. The measured hardness values ranged from 333.4 $HV_{0.2}$ (Ti-S1) to 485.4 $HV_{0.2}$ (Ti-S5), indicates a significant increase in hardness with increasing of titanium content.

Table 11. Vickers microhardness ($HV_{0.2}$) measurements of experimental 17-4PH steel samples microalloyed with titanium, prior to heat treatment.

S1	S2	S3	S4	S5	Mean value
329	401	473	412	466	333,4 $HV_{0.2}$
291	371	429	490	487	388 $HV_{0.2}$
353	396	418	453	463	432,2 $HV_{0.2}$
351	412	415	423	487	439,2 $HV_{0.2}$
343	360	426	418	524	485,4 $HV_{0.2}$

For each experimental batch, the microhardness data were statistically analyzed by determining the following indicators: arithmetic mean, minimum and maximum values, range, standard deviation, and coefficient of variation.

These parameters enable the assessment of data dispersion and material homogeneity.

The distribution of values and the calculated statistical parameters (standard deviation, coefficient of variation) used to evaluate the homogeneity of each sample are presented in Table 12. The results indicate that samples with higher Ti content exhibit both increased average hardness and a slight rise in hardness value

dispersion, suggesting a combined influence of titanium on material strengthening and structural homogeneity.

Table 12. Statistical analysis of Vickers microhardness ($HV_{0.2}$) values for titanium microalloyed 17-4PH steel prior to heat treatment.

Sample	Mean	Minimum	Maximum	Range	Standard deviation	Coefficient of variation (%)
S1	333,40	291	353	62	25.54	7.66
S2	388,00	360	412	52	21.70	5.59
S3	432,20	415	473	58	23.55	5.45
S4	439,20	412	490	78	32.43	7.39
S5	485,40	463	524	61	24.31	5.01

4. Conclusions

The conducted research aimed to investigate the influence of titanium microalloying on the structural and mechanical properties of martensitic stainless steel 17-4PH. The chemical composition of the microalloyed steel was precisely controlled, with all batches accurately weighed (± 0.01 g) and validated through charge calculations. Based on the experimentally produced samples and the performed analyses, the following conclusions can be drawn:

Titanium was introduced successively in concentrations ranging from 1% to 5% to observe changes in microstructure and microhardness. It was found that titanium microalloying significantly influenced the microstructural features and hardness properties of 17-4PH steel, even at a low concentration. The presence of titanium caused modifications in grain size and the precipitation of secondary phases, which contributed to hardening.

The Vickers microhardness ($HV_{0.2}$) measurements revealed a clear increasing trend with rising titanium content. Specifically, the average hardness values increased from approximately 333 $HV_{0.2}$ for the sample with 1% Ti (Ti-S1) to about 485 $HV_{0.2}$ for the sample with 5% Ti (Ti-S5). This outcome demonstrates the efficiency of the dispersion strengthening mechanism associated with the formation of hard TiC and TiN phases. The results indicate that titanium, due to its high affinity for carbon and nitrogen, contributes to the formation of stable interstitial compounds that act as nucleation sites for precipitates and inhibit dislocation movement, thereby enhancing stiffness and resistance to plastic deformation.

The microstructure analyzed by optical microscopy revealed a reduction in the size of martensitic grains with increasing titanium content, which further contributes to the material's hardening according to the Hall-Petch mechanism. Additionally, a more homogeneous distribution of hard phases was observed in samples with moderate titanium content (2 to 3%), suggesting an optimized microalloying effect within this compositional range. Higher titanium contents (4 to 5%) were associated with a slight increase in microstructural heterogeneity. The hardness variation coefficient values indicate good mechanical property stability for batches with 2% and 3% Ti, whereas samples with 5% Ti exhibit greater dispersion in

values, potentially linked to the localized formation of hard phase agglomerates or inclusions.

The study confirms that microalloying with Ti is a viable alternative for the development of martensitic stainless steels with enhanced properties, without major changes to the processing or heat treatment routes. The results obtained in this research can serve as a basis for the development of customized alloys tailored to specific applications, particularly in fields requiring high hardness, microstructural stability, and wear resistance.

Overall conclusions support the idea that titanium, when used in moderate concentrations and properly integrated into the alloying system of 17-4PH steel, can improve the overall performance of the material, strengthening its position in critical and demanding mechanical and thermal applications.

It is important to note that the samples analyzed in this study have not yet undergone the specific heat treatments for 17-4PH steel, such as solution annealing and artificial aging, followed by quenching and tempering. All structural observations and microhardness measurements presented were performed on the as-cast condition of the alloy, immediately after processing. Therefore, the results directly reflect the compositional effect of titanium on the microstructure and hardness, in the absence of subsequent thermal treatment.

The results confirm that titanium can partially or completely replace niobium in the hardening system of 17-4PH steel without compromising mechanical properties, and potentially improving them, provided the dosing is carefully controlled. The optimal titanium content, from the perspective of balancing increased hardness and structural homogeneity, appears to be in the range of 2 - 3% Ti, where both high hardness values and low data dispersion were recorded.

The next phase of the research will involve applying the full sequence of heat treatments according to the standard H900, H1025, or H1150 regimes to analyze the effect of titanium on:

- Precipitation behavior (including the formation of Cu and TiC phases);
- Martensitic stability after aging;
- Changes in mechanical properties in the heat-treated state;
- Corrosion behavior in various environments.

Performing these heat treatments and correlating the results obtained before and after will enable a comprehensive characterization of the microalloying effect of titanium, providing an overview of the structural and functional evolution of 17-4PH steel under simulated industrial conditions.

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