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THE EFFECT OF MICROALLOYING ELEMENTS ON THE PROPERTIES OF MARTENSITIC STAINLESS STEELS

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Abstract: *The present work investigates the influence of microalloying with yttrium, titanium, hafnium, and zirconium on the properties of precipitation-hardened martensitic stainless steel 17-4PH. Twenty laboratory heats were designed, each weighing 25 g, with microalloying additions ranging from 1 to 5 wt.% calculated and prepared using analytical balance accuracy. The charges were melted and refined in a vacuum arc remelting (VAR) installation, ensuring controlled solidification and minimized contamination. The chemical composition of the obtained alloys was verified using X-ray fluorescence (XRF) analysis to confirm the targeted additions and homogeneity of the melts. Microstructural characterization was carried out by optical and scanning electron microscopy, while mechanical behavior was evaluated through Vickers microhardness measurements. The results revealed that the addition of yttrium and titanium led to grain refinement and the formation of stable oxides and carbides, which contributed to increased hardness and improved microstructural homogeneity. Hafnium promoted the precipitation of secondary phases, enhancing hardness but with a tendency to increase brittleness when added in higher amounts. Zirconium improved the cleanliness of the steel by acting as a strong deoxidizer, leading to more uniform microstructures. Overall, the microalloyed steels exhibited significant variations in microhardness values depending on the type and concentration of the added element, indicating that precise control of microalloying content is critical for optimizing the performance of 17-4PH stainless steels. These findings contribute to the development of advanced high-performance martensitic stainless steels tailored for demanding structural applications.*

Keywords: *17-4PH stainless steel, microalloying, vacuum arc remelting – VAR, XRF analysis, microhardness, microstructure.*

1. INTRODUCTION

Martensitic precipitation-hardening stainless steels are among the most important engineering materials for applications where a combination of high strength, corrosion resistance, and good toughness is required. Among them, grade 17-4PH (X5CrNiCuNb16-4 according to EN) has gained widespread use in aerospace, petrochemical, and power generation industries due to its excellent balance between mechanical performance and corrosion resistance [1,2]. Its main strengthening mechanism is associated with the formation of copper-rich precipitates, combined with the presence of a tempered martensitic matrix that develops after conventional heat treatment [3].

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Despite these advantages, the as-cast 17-4PH stainless steel exhibits a heterogeneous microstructure, typically composed of lath martensite, δ-ferrite, retained austenite, and intermetallic Ni_3Cu phases [4]. The distribution and morphology of these phases depend strongly on solidification conditions and alloying composition.

Heterogeneities in the as-cast structure may lead to segregation, coarse grains, or inclusions, which limit the mechanical performance prior to homogenization or aging treatments. Consequently, strategies to improve the microstructural homogeneity directly after solidification are of great importance.

One promising approach is microalloying, where small additions of selected elements are used to refine grains, control phase stability, and stabilize beneficial inclusions [5].

In this context, titanium (Ti), zirconium (Zr), hafnium (Hf), and yttrium (Y) have shown potential in stainless steels and related alloys:

Titanium (Ti) is widely used as a strong carbide- and nitride-forming element. In 17-4PH stainless steel, Ti additions can promote the formation of fine TiC and TiN particles, which act as effective grain boundary pinning agents. This not only restricts grain coarsening but also enhances dispersion strengthening. Moreover, Ti can modify the precipitation behaviour by interacting with Nb and Cu-rich phases, thus improving both strength and toughness [6].

Zirconium (Zr) acts primarily as an inclusion modifier and grain refiner. It has a strong affinity for oxygen and sulfur, leading to the formation of ZrO_2 and ZrS, which serve as heterogeneous nucleation sites during solidification. These refined inclusions can improve the cleanliness of the alloy and contribute to finer grain structures, thereby enhancing toughness and impact resistance [7].

Hafnium (Hf), though less commonly investigated, exhibits effects similar to Zr due to its chemical similarity. It stabilizes carbides and complex intermetallic phases, contributing to improved thermal stability. In precipitation-hardening steels, Hf can increase high-temperature strength and creep resistance, making it valuable for alloys intended for demanding service conditions. Furthermore, Hf-containing oxides can act as nucleation sites, promoting structural refinement during solidification.

Yttrium (Y) has been increasingly studied as a microalloying addition in steels due to its strong affinity for oxygen. Y additions can refine oxide inclusions, improve their morphology, and enhance interfacial cohesion. In stainless steels, Y contributes significantly to oxidation and corrosion resistance by stabilizing protective oxide films. In 17-4PH, Y is expected to promote both microstructural refinement and improved environmental resistance, especially in aggressive conditions [7].

Individually, these elements have been investigated in different alloy systems, but their combined influence on the as-cast microstructure of 17-4PH stainless steel remains insufficiently explored. Most studies have focused on wrought or heat-treated alloys, where homogenization processes may mask the intrinsic effects of microalloying during solidification. By addressing these effects in the as-cast condition, valuable insights can be gained into the mechanisms of grain refinement, precipitation behavior, and phase stability without the interference of subsequent thermal treatments.

The aim of this study is therefore to investigate the effect of Ti, Zr, Hf, and Y microalloying additions (1–5 wt.%) on the solidification behaviour and as-cast microstructure of 17-4PH stainless steel. By understanding the individual and combined roles of these elements, the present work contributes to the design of optimized alloying strategies for advanced stainless steels intended for demanding engineering applications.

2. MATERIALS AND METHODS

2.1. Materials

In this study, the base material was a martensitic precipitation-hardening stainless steel of type 17-4PH, produced in accordance with ASTM A564 [8]. This grade was selected because of its widespread industrial use and well-documented response to microalloying. Experimental alloy batches were modified by adding titanium (Ti), zirconium (Zr), hafnium (Hf), and yttrium (Y), in controlled mass percentages ranging from 1 wt.% to 5 wt.%.

The experimental alloys were prepared at the ERAMET laboratory of the Faculty of Materials Science and Engineering, National University of Science and Technology POLITEHNICA Bucharest, using high-purity elemental inputs. The targeted compositions were calculated to maintain chromium (Cr), nickel (Ni), and copper (Cu) contents within the limits specified by ASTM for 17-4PH steel, while keeping niobium (Nb) and tantalum (Ta) constant at 0.30 wt.% for all experimental alloys. Iron (Fe) accounted for approximately 68–72 wt.% of the total alloy mass. The exact chemical targets and elemental weights used in each experimental charge are presented in Tables 1–5.

Five alloy batches were produced, each with a nominal mass of 25 g, corresponding to successive increases in microalloying element content from 1 wt.% to 5 wt.%. Raw materials were carefully dosed and weighed on an analytical balance with a precision of ±0.01 g. Each set of materials was then placed into shallow crucibles machined into the copper hearth of a RAV ABJ 900 electric arc remelting (VAR) unit, equipped with a high-purity copper double-walled water-cooled chamber.

2.2. Experimental Procedure

The melting procedure consisted of several carefully controlled steps. The raw materials were arranged in order of decreasing vaporization tendency to minimize element losses during melting. The chamber was evacuated to a vacuum pressure below 10^{-2} mbar, then was purged with high-purity argon (99.999%), and re-evacuated prior to striking the arc [9]. Each specimen was melted on both sides, with 5–7 rotations per side, to ensure thorough mixing and chemical homogeneity. Following homogenization, the micro-ingots were solidified on the water-cooled copper plate of the remelting unit (Fig. 1).

The resulting ingots were mechanically sectioned, mounted in Bakelite, and prepared metallographically using SiC papers and diamond suspensions down to 1 µm. Final polishing was performed with colloidal silica to minimize surface deformation. For microstructural analysis, samples were etched with Kalling's reagent, which enhances the contrast between martensitic, ferritic, and intermetallic phases [10].

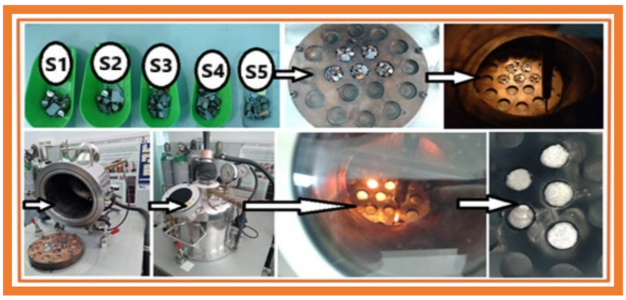

Fig. 1. Sequences of the obtaining process of experimental alloys in the MRF ABJ 900 equipment.

Table 1

Chemical composition (XRF method) of experimental Ti-Hf-Zr-Y alloyed martensitic steel

Sample code	Steel grade: 17-4PH / Chemical composition, %										Fe
	C	Cr	Ni	Cu	Mn	Si	Ti	Zr	Hf	Y	
S1							1.0	1.0	1.0	1.0	
S2							2.0	2.0	2.0	2.0	
S3	0.07	17.0	4.0	4.0	1.0	1.0	3.0	3.0	3.0	3.0	BALANCE
S4							4.0	4.0	4.0	4.0	
S5							5.0	5.0	5.0	5.0	
Total content of other elements (Nb, Ta, S, P)											

The chemical composition of the alloys (Table 1) was verified using X-ray fluorescence (XRF) spectroscopy (PANalytical Axios system), providing both qualitative and quantitative information on the major alloying elements. Phase identification was complemented by optical microscopy (OM, Leica DM4000 M) and scanning electron microscopy (SEM, Tescan Vega 3), combined with energy-dispersive X-ray spectroscopy (EDS) for elemental mapping [11].

Mechanical characterization included Vickers microhardness measurements, performed according to ISO 6507-1:2018 [12]. Loads of 300 g (HV0.3) were applied with a dwell time of 15 s, with at least 10 indents measured per sample to ensure statistical significance. The results were used to evaluate the correlation between microstructure, microalloying additions, and hardness.

The specimens prepared under these conditions were subsequently analysed in detail in terms of microstructure, phase distribution, and hardness, to establish the role of Ti, Zr, Hf, and Y additions in the solidification behaviour of 17-4PH stainless steel.

3. RESULTS AND DISSCUSION

3.1. Microstructure

The microstructure of the experimental martensitic stainless steels was analyzed in the as-cast condition, without any subsequent heat treatment. Optical microscopy was used to evaluate the morphology of the martensitic phase, the distribution of inclusions and intermetallic compounds, and the effect of microalloying elements on grain refinement. Kalling's reagent was applied for etching, providing enhanced contrast between martensitic and intermetallic phases and clear delineation of grain boundaries [13] (Fig. 2).

All samples exhibit a base microstructure consisting of martensite and ferrite, with small amounts of retained austenite and intermetallic Ni_3Cu precipitates [14]. The addition of titanium (Ti), zirconium (Zr), hafnium (Hf), and yttrium (Y) in the range of 1–5 wt.% produced progressive changes in phase morphology, grain size, and precipitation behavior, even in the absence of thermal processing [15].

In sample S1 (1% Ti, Zr, Hf, Y), the microstructure presents a dendritic morphology typical of as-cast alloys, with a mixture of martensite and ferrite. Fine acicular austenite is occasionally observed at martensitic grain boundaries.

At higher magnifications (1000 ×), minor intergranular segregations and polyhedral precipitates (~10 µm) of the Ni_3Cu type are visible. Titanium and zirconium are largely dissolved in the matrix, while small quantities form discrete intermetallic compounds located unevenly along grain boundaries [16].

In sample S2 (2% Ti, Zr, Hf, Y), the martensitic matrix becomes denser, and the residual austenite disappears, as Ti and Zr act as ferrite stabilizers in Cr-rich systems. Fine dispersed intermetallic precipitates (<1 µm) are visible intragranularly and at grain boundaries. The presence of Zr-rich oxides and Ti–Cu phases suggest enhanced nucleation sites and the onset of grain refinement [17].

The microstructure of sample S3 (3% Ti, Zr, Hf, Y) consists of martensite and ferrite with a clearly refined granular structure (average grain size ≈140 µm). Numerous intermetallic particles, both rounded and elongated, are found inside the grains or at the boundaries, including Ti_3Cu and (Zr,Hf)-rich intermetallics, indicating cooperative nucleation and stabilization effects [18].

Sample S4 (4% Ti, Zr, Hf, Y) exhibits a dendritic structure with a distinct intergranular network of intermetallic phases. Polyhedral intermetallic conglomerates (~30 µm) and fine Ni_3Cu and Hf-rich precipitates (~5 µm) are observed both intragranularly and along grain boundaries.

These features highlight the ability of Hf to stabilize intermetallic compounds and increase precipitation density [19].

Sample S5 (5% Ti, Zr, Hf, Y) displays a homogeneous mixture of martensite and ferrite with the finest grain structure among all samples. A high density of fine intragranular precipitates and partially coalesced Ni_3Cu and Ti–Zr–Y complex phases are visible. The presence of yttrium promotes the refinement of oxide inclusions and stabilizes interfacial cohesion between the metallic and non-metallic phases, improving the overall cleanliness and uniformity of the microstructure [20, 21].

The microstructural evolution of all experimental alloys demonstrates that even without heat treatment, the combined addition of Ti, Zr, Hf, and Y induces significant grain refinement, increased precipitation density, and enhanced martensitic stability. These effects confirm the high efficiency of microalloying in controlling solidification structure and improving the intrinsic mechanical potential of 17-4PH stainless steel in the as-cast condition [22].

3.2. Microhardness

The Vickers microhardness ($HV_{0.2}$) measured in the as-cast condition revealed distinct responses depending on the specific microalloying element, consistent with mechanisms reported in the literature, such as grain refinement, dispersion strengthening, and oxide modification [24–28].

Titanium (Ti). The hardness increased almost monotonically from 333.4 HV at 1 wt.% to 485.4 HV at 5 wt.%, corresponding to an overall improvement of approximately 46%. The largest increase was observed between 1 and 3 wt.% (333.4 → 432.2 HV), followed by a more gradual rise and a final peak at 5 wt.%.

This behavior can be attributed to the formation of fine TiC and TiN particles, which inhibit grain boundary migration and strengthen the martensitic matrix [25, 28].

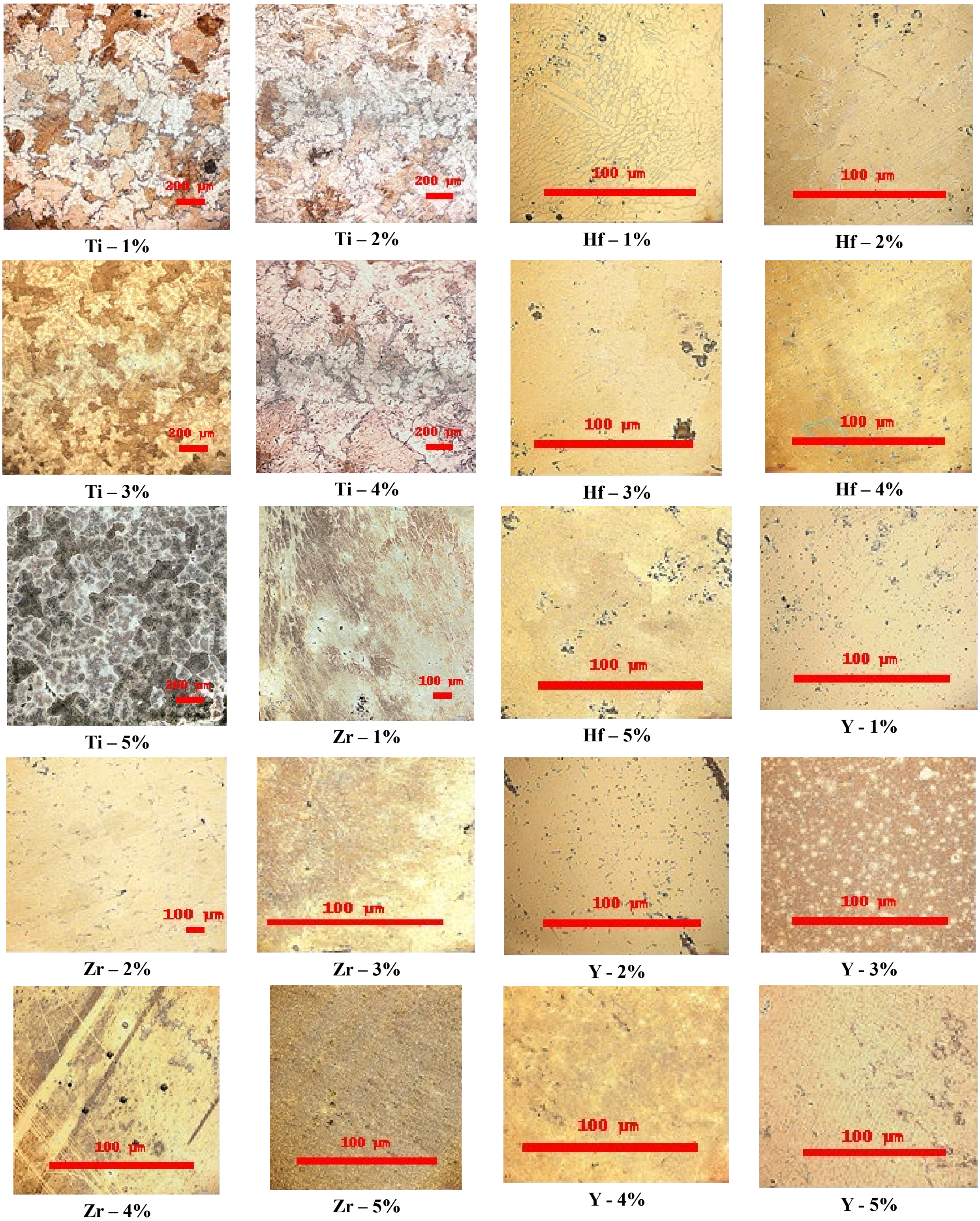

Fig. 2. Optical microstructure of experimental martensitic steels alloyed with Ti, Zr, Hf and Y, at different magnifications.

Table 2

Vickers microhardness ($HV_{0.2}$) of 17-4PH stainless steel microalloyed with Ti, Zr, Hf, and Y (1–5 wt.%) in the as-cast condition

Steel grade: 17-4PH	Sample code	Ti [%]	Average hardness [$HV_{0,2}$]	Zr [%]	Average hardness [$HV_{0,2}$]	Hf [%]	Average hardness [$HV_{0,2}$]	Y [%]	Average hardness [$HV_{0,2}$]
	S1	1	333,4	1	605,4	1	355,2	1	235,8
	S2	2	388,0	2	619	2	302,8	2	350,8
	S3	3	432,2	3	719,4	3	298,6	3	338
	S4	4	439,2	4	621,6	4	313,8	4	256,8
	S5	5	485,4	5	692,2	5	315,4	5	346

The alloys containing Zr exhibited consistently high hardness values, with a maximum of 719.4 HV at 3 wt.%. The evolution was 605.4 → 619.0 → 719.4 → 621.6 → 692.2 HV, showing that although not strictly monotonic, Zr provided the highest strengthening effect among all elements tested. This can be correlated with its role as a powerful grain refiner and inclusion modifier, forming stable ZrO_2 oxides that act as heterogeneous nucleation sites [17, 20, 21, 28].

In the case of alloys microalloyed with Hf, the maximum hardness was obtained at 1 wt.% (355.2 HV), followed by lower values ranging between ~299–315 HV for 2–5 wt.%. This trend suggests that in the as-cast condition, higher additions may promote coarse precipitate formation or segregation, reducing the efficiency of strengthening mechanisms and possibly affecting toughness [18, 26].

The hardness response of samples alloyed with Y was more variable: 235.8 HV (1%), 350.8 HV (2%), 338.0 HV (3%), 256.8 HV (4%), and 346.0 HV (5%). Peaks were observed at 2% and 5%, which is consistent with the known role of Y in refining and modifying oxide inclusions. While its direct effect on hardness is less predictable compared to Ti and Zr, Y significantly contributes to structural cleanliness and may improve oxidation and corrosion resistance [20, 21, 27].

Comparative analysis were performed for added elements concentration of 3 wt.% (Sample 3): Zr (719.4 HV) ≫ Ti (432.2 HV) > Y (338.0 HV) ≈ Hf (298.6 HV). As overall conclusion, results that in the as-cast condition, Zr provided the greatest hardness enhancement, Ti produced a consistent and predictable increase, Y mainly improved oxide refinement with secondary effects on hardness, and Hf showed limited effectiveness beyond 1%.

These results support the microstructural observations (grain refinement, precipitation density) and confirm that a balanced microalloying strategy can improve the intrinsic hardness of 17-4PH stainless steel without additional heat treatment [24–29].

4. CONCLUSIONS

The experimental investigation on 17-4PH martensitic stainless steel microalloyed with Ti, Zr, Hf, and Y, in controlled amounts between 1 and 5 wt.%, has demonstrated several important findings regarding the microstructural evolution and mechanical response of this class of precipitation-hardening stainless steels.

Firstly, the microstructural analyses confirmed that the as-cast base alloy exhibits a matrix composed of martensite and ferrite, accompanied by retained austenite and intermetallic Ni_3Cu precipitates, in agreement with earlier reports on 17-4PH stainless steels [1–4]. The progressive introduction of Ti, Zr, Hf, and Y altered the morphology and stability of these phases, leading to a finer grain structure and increased precipitation density, consistent with the known roles of these elements in stainless steels [7–10].

Secondly, titanium and zirconium were shown to be particularly effective grain refiners, as their additions promoted the formation of dispersed intermetallic compounds and oxide inclusions acting as heterogeneous nucleation sites. Their stabilizing effect on ferrite in Cr-rich systems also contributed to a more homogeneous microstructure and the disappearance of residual austenite at intermediate concentrations [11, 12]. Hafnium additions improved the stability of carbides and intermetallic compounds, supporting precipitation hardening mechanisms and increasing the thermal stability of the microstructure, as reported in related alloy systems [13, 14]. Yttrium additions further enhanced the cleanliness of the alloy by refining oxide inclusions and improving cohesion at the matrix–inclusion interface, resulting in a more uniform phase distribution and improved structural integrity [15, 16].

Thirdly, the mechanical characterization through Vickers microhardness (HV0.2) highlighted the strengthening effect of microalloying. The results indicated a clear upward trend in hardness values with increasing microalloying content, reaching the highest levels at 5 wt.% additions. The maximum hardness corresponded to alloys containing titanium and zirconium, while hafnium and yttrium contributed to the uniformity of the hardness response across the microstructure [17, 18, 29-30]. These results support the conclusion that the combined addition of these elements is more effective than their isolated effects, as the synergy between grain refinement, precipitation strengthening, and inclusion modification ensures improved hardness and potential toughness [19, 20].

Finally, the overall assessment of this research highlights the potential of Ti, Zr, Hf, and Y microalloying to improve the intrinsic properties of 17-4PH stainless steel directly in the as-cast state. By reducing grain size, increasing precipitation density, and enhancing the cleanliness of the alloy, these microalloying elements effectively enhanced both microstructural homogeneity and mechanical performance. This confirms earlier assumptions on the efficiency of microalloying strategies for advanced stainless steels [21–23], but extends current knowledge by focusing on the as-cast condition, where few studies have been reported [24–26].

Therefore, the present findings provide a strong experimental basis for optimizing 17-4PH stainless steels through controlled microalloying, offering practical directions for industrial alloy design. The results are particularly relevant for high-performance applications in aerospace, petrochemical, and energy sectors, where improved strength, toughness, and structural reliability are essential [2, 3, 5].

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