

SESSION E: THERMAL TECHNIQUE AND ENVIRONMENTAL PROTECTION**Influence of overheating exposure time on the microstructural stability and mechanical properties of HP40-0.63Nb alloy**Milica Timotijević^{1*}, Olivera Erić Cekić^{2,3}, Dragan Rajnović⁴, Petar Janjatović⁴¹ University of Belgrade, The Academy of Applied Studies Polytechnic, Department of Mechanical Engineering, Belgrade, Serbia² University of Kragujevac, Faculty of Mechanical and Civil Engineering in Kraljevo, Kraljevo, Serbia³ University of Belgrade, Innovation Centre of the Faculty of Mechanical Engineering, Belgrade, Serbia⁴ University of Novi Sad, Faculty of Technical Science, Department of Production Engineering, Novi Sad, Serbia**ARTICLE INFO*** Correspondence: mtimotijevic@politehnika.edu.rsDOI: <https://doi.org/10.46793/ET26.E03T>

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ABSTRACT

HP40-Nb heat-resistant cast steels are widely used for reformer furnace tubes operating at elevated temperatures, where accidental overheating may induce microstructural degradation and alter mechanical properties. This study investigates the influence of overheating time on the microstructure and mechanical properties of centrifugally cast HP40-0.63Nb alloy. Samples were overheated at 950 and 1150 °C for 30, 120 and 480 min. Microstructural characterization was performed using light microscopy (LM), scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), while tensile testing and hardness measurements were used to evaluate mechanical performance. The as-cast microstructure consisted of an austenitic dendritic matrix and eutectic chromium-rich and niobium-rich carbides. Overheating promoted the precipitation of secondary chromium-rich carbides, causing significant changes in carbide morphology and distribution. The highest yield strength (346 MPa) was achieved after overheating at 950 °C for 120 min due to intensive precipitation hardening. Prolonged overheating led to carbide coarsening and partial dissolution, reducing the strengthening effect. The highest tensile strength (582 MPa) and favorable ductility (8.76 %) were obtained after overheating at 1150 °C for 30 min. The results demonstrate that overheating time strongly influences carbide evolution and mechanical behavior of HP40-0.63Nb alloy, with 950 °C/120 min providing maximum strengthening and 1150 °C/30 min the best strength–ductility balance.

KEYWORDS

HP40-Nb alloy, Overheating time, Secondary carbides, Mechanical properties.

1. INTRODUCTION

Heat-resistant centrifugally cast austenitic steels of the HP family are extensively used in petrochemical and chemical industries for the production of reformers, cracking and pyrolysis furnace tubes operating at high temperatures [1-4]. Their widespread application is primarily associated with excellent resistance to creep, oxidation and carburization at elevated temperatures [5-8]. These properties are achieved through a combination of austenitic matrix strengthening and the presence of primary and secondary carbide phases.

Among HP-type alloys, niobium-containing grades have attracted considerable attention because niobium promotes the formation of thermally stable MC carbides, improves high-temperature strength and enhances creep resistance [9]. The microstructure of HP-Nb alloys in the as-cast condition typically consists of an austenitic dendritic matrix and a continuous network of eutectic carbides distributed along grain boundaries and interdendritic regions [10,11]. Depending on alloy composition, and thermal history, chromium-rich carbides (mainly M_7C_3 in the as-cast condition and $M_{23}C_6$ after thermal exposure) and niobium-rich MC carbides may coexist in the microstructure.

During exposure to elevated temperatures, significant microstructural changes occur. Carbon diffusion from the super-saturated austenitic matrix promotes the precipitation of secondary chromium-rich carbides, while prolonged thermal

exposure may lead to carbide coarsening, partial dissolution and changes in carbide morphology. These microstructural processes directly influence mechanical properties, particularly yield strength, tensile strength and ductility [12-14].

Previous studies have shown that carbide precipitation, transformation and coarsening significantly influence the mechanical behavior of HP-Nb alloys during thermal exposure [15-18]. However, the influence of short-term overheating duration remains insufficiently investigated.

In industrial service conditions, accidental overheating events may occur due to process disturbances, burner malfunctions or local hot spots, resulting in temperatures significantly above the design operating range. Such events can induce rapid microstructural changes and consequently alter the mechanical performance of HP-Nb alloys.

Therefore, the aim of this work was to investigate the influence of overheating time on the microstructure and mechanical properties of HP40-0.63Nb alloy. Special attention was paid to microstructural evolution, particularly changes in carbide morphology and microstructural features, and their effect on the mechanical properties of the alloy. The influence of overheating at 950 °C and 1150 °C for different exposure times on yield strength, tensile strength, elongation and hardness was systematically evaluated and interpreted in relation to the observed microstructural changes.

2. EXPERIMENTAL PROCEDURE

In this study, specimens were sectioned from a centrifugally cast HP40-Nb austenitic heat-resistant cast steel alloy in the as-cast condition. The chemical composition of the alloy was determined using an Optical Emission Spectrometer (OES, I Spark 8860, Thermo Scientific™, USA). The specimens were subjected to thermal exposure at 950 and 1150 °C for 30, 120 and 480 min, followed by cooling to room temperature. Heat treatments were carried out in a chamber-type laboratory furnace equipped with a PID temperature controller. The furnace temperature was continuously monitored using Pt-Pt13%Rh thermocouples positioned in close proximity to the specimens.

Following thermal exposure, the specimens were prepared for metallographic examination using standard grinding and polishing procedures. Grinding was performed with SiC abrasive papers ranging from 180 to 2400 grit, followed by polishing with diamond suspensions of 6, 3, 1, and 0.25 µm particle size. The polished surfaces were etched using a solution containing 15 mL HCl, 10 mL glycerol, and 5 mL HNO₃.

Microstructural characterization was performed using light microscopy (LM), scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). For LM a Leitz-Orthoplan microscope was used, while for SEM a JEOL JSM-6460LV was used. Phase identification and chemical characterization of the microstructural constituents were conducted by energy-dispersive X-ray spectroscopy (EDS), INCA, Oxford Instruments attached to the SEM.

Vickers hardness (HV10) was measured in accordance with SRPS EN ISO 6507-1:2023 using a load of 98.07 N (10kgf) and a dwell time of 15 s on an HPO 250 hardness tester (WPM, Markkleeberg, Germany). Five measurements were performed for each specimen, and the mean hardness value was calculated after excluding the highest and lowest readings.

Mechanical properties were evaluated by tensile testing at room temperature in accordance with SRPS EN ISO 6892-1:2020 on three specimens. Tests were carried out on a Schenck Trebel universal testing machine (F_{max} = 250 kN) at a crosshead speed of 2 mm/min. The yield strength, ultimate tensile strength, and total elongation were determined from the resulting engineering stress-strain curves for specimens in the as-cast and heat-treated conditions.

3. RESULTS AND DISCUSSION

3.1. Microstructure

3.1.1. As-Cast Microstructure

The microstructure of the HP40-0.63Nb alloy in the as-cast condition is presented in Fig. 1. The typical as-cast microstructure consists of a γ -austenitic dendritic matrix surrounded by a network of primary eutectic carbides concentrated in the interdendritic regions and along grain boundaries. Figure 1b shows two morphologically distinct carbide phases within the interdendritic regions. The dark grey phase was identified as a Cr-rich M₇C₃ carbide, whereas the bright phase corresponded to Nb-rich MC-type carbides containing minor amounts of Ti and Cr.

The contrast differences observed in the BSE images (Fig. 1b) arise from variations in the average atomic number of the constituent phases, with phases containing heavier elements appearing brighter. In the present alloy, Nb-rich carbides exhibit a brighter contrast than Cr-rich carbides due to the significantly higher atomic number of niobium compared with chromium. The applicability of SEM-BSE imaging for distinguishing carbide phases in heat-resistant cast steels has been widely reported in the literature [19–21].

Results of further EDS analyses of carbides are given in Table 1. The Spectrum 1 and 2 revealed high chromium contents, confirming the presence of chromium M₇C₃ carbides. In contrast, Spectrum 3 and 4 were enriched in Nb, indicating the formation of MC-type carbides. Spectrum 5 corresponded to the γ -austenitic matrix.

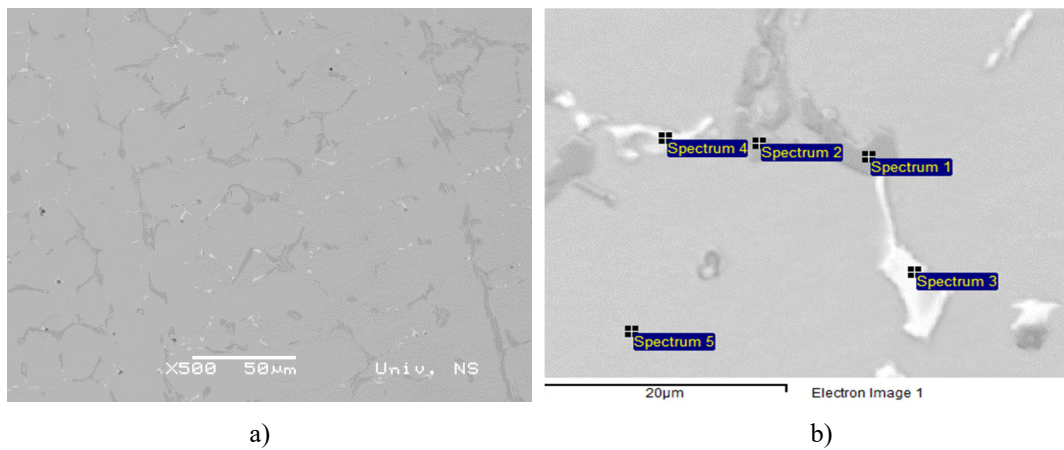


Figure 1: The microstructure of the HP40-0.63Nb alloy in the as-cast condition: a) SEM-BSE, b) SEM-BSE [10]

Table 1: Chemical composition (mass. %) of phases in HP40 Nb alloy, according to Fig.1b

Spectrum	C	Si	Ti	Cr	Mn	Fe	Ni	Nb
Spect. 1	36.97			54.39		6.92	1.72	
Spect. 2	15.83	3.41		28.73		27.11	24.93	
Spect. 3	44.98		4.30	4.62		2.56	1.80	41.73
Spect. 4	38.99	1.52	1.02	11.78		10.03	8.49	28.16
Spect. 5	10.76	2.56		22.12	0.94	34.03	29.58	

The presence of thermally stable MC-type carbides is particularly important for the high-temperature performance of HP40-Nb alloys. Unlike chromium-rich carbides, Nb-rich MC carbides exhibit superior thermal stability and resistance to coarsening and dissolution during long-term exposure at elevated temperatures. Consequently, these carbides play a crucial role in maintaining creep strength and microstructural stability during service [22-23].

3.1.2. Effect of Overheating Time at 950 °C on the Microstructure

Representative microstructural features observed in the HP40–0.63Nb alloy after overheating at 950 °C for different exposure times (30, 120, and 480 min) are shown in Figure 2.

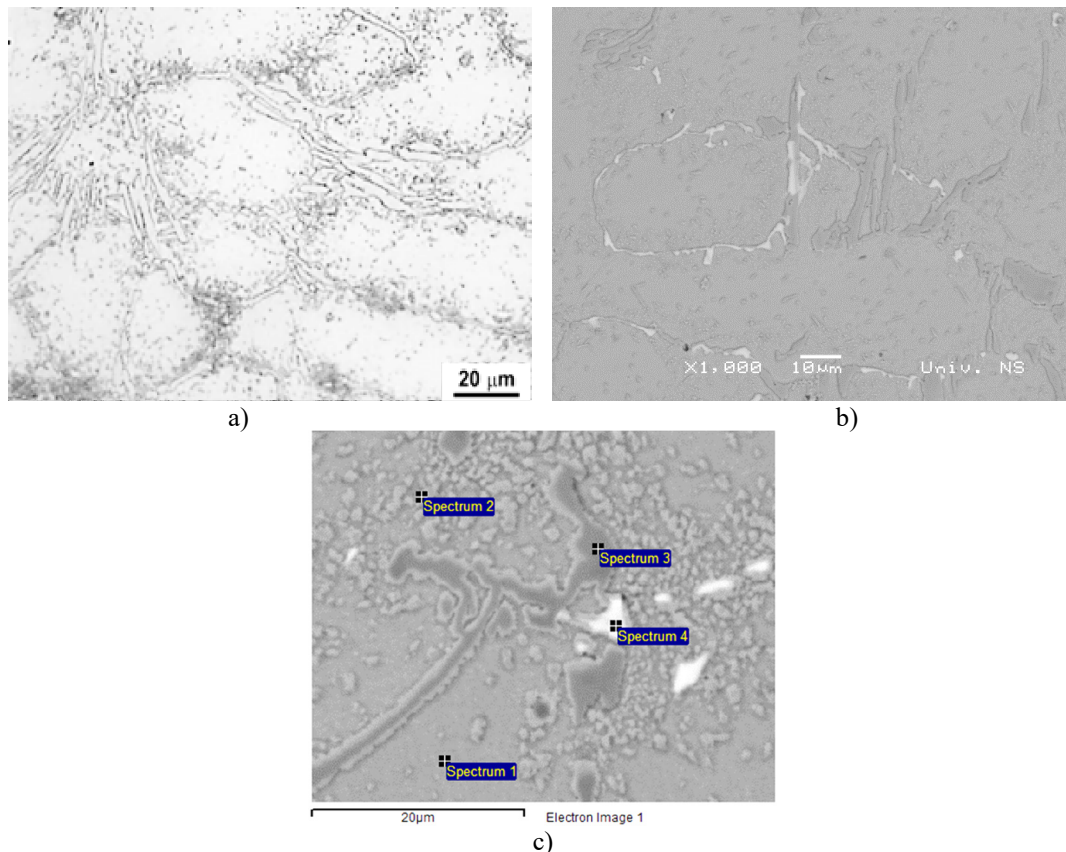


Figure 2: Microstructure after overheating at 950 °C: a) for 30 min (LM), b) for 120 min (SEM-BSE), c) for 480 min (SEM-BSE)

Table 2: Chemical composition (mass. %) of participating phases in HP40 Nb alloy, corresponding to Fig.2c

Spectrum	C	O	Si	Ti	V	Cr	Mn	Fe	Ni	Nb
Spectrum 1	16.17	2.62	2.09			19.26	1.13	30.71	28.01	
Spectrum 2	21.61		3.15			20.84	1.34	26.61	26.45	
Spectrum 3	26.77				0.41	61.06	1.57	7.84	2.35	
Spectrum 4	54.13		0.69	0.53	0.36	6.94		4.39	4.71	28.24

Overheating at 950 °C for 30 min resulted in the precipitation of numerous fine secondary chromium-rich carbides within the austenitic matrix as carbon diffused from the supersaturated solid solution. Thermal exposure promoted the transformation of metastable M_7C_3 carbides into the more stable $M_{23}C_6$ phase, accompanied by the precipitation of secondary chromium-rich carbides, thereby strengthening the matrix [26]. Increasing the overheating duration to 120 min led to a substantial increase in the density of secondary carbides. EDS analysis confirmed the presence of chromium-rich $M_{23}C_6$ carbides and thermally stable niobium-rich MC carbides. The extensive precipitation of fine $M_{23}C_6$ carbides corresponded to the highest yield strength (346 MPa), while elongation decreased to 5.06%, indicating significant precipitation hardening. Further extension of the overheating time to 480 min promoted carbide coarsening and partial dissolution. As a result, the strengthening effect diminished slightly, as evidenced by a small reduction in yield strength and a lower fraction of chromium-rich carbides. These findings suggest that carbide precipitation reached its maximum at approximately 120 min, after which coarsening became the dominant microstructural process.

3.1.3. Effect of Overheating Time at 1150 °C on the Microstructure

The microstructural changes induced by overheating at 1150 °C are shown in Figure 3, where representative micrographs corresponding to 30, 120, and 480 min of exposure are presented.

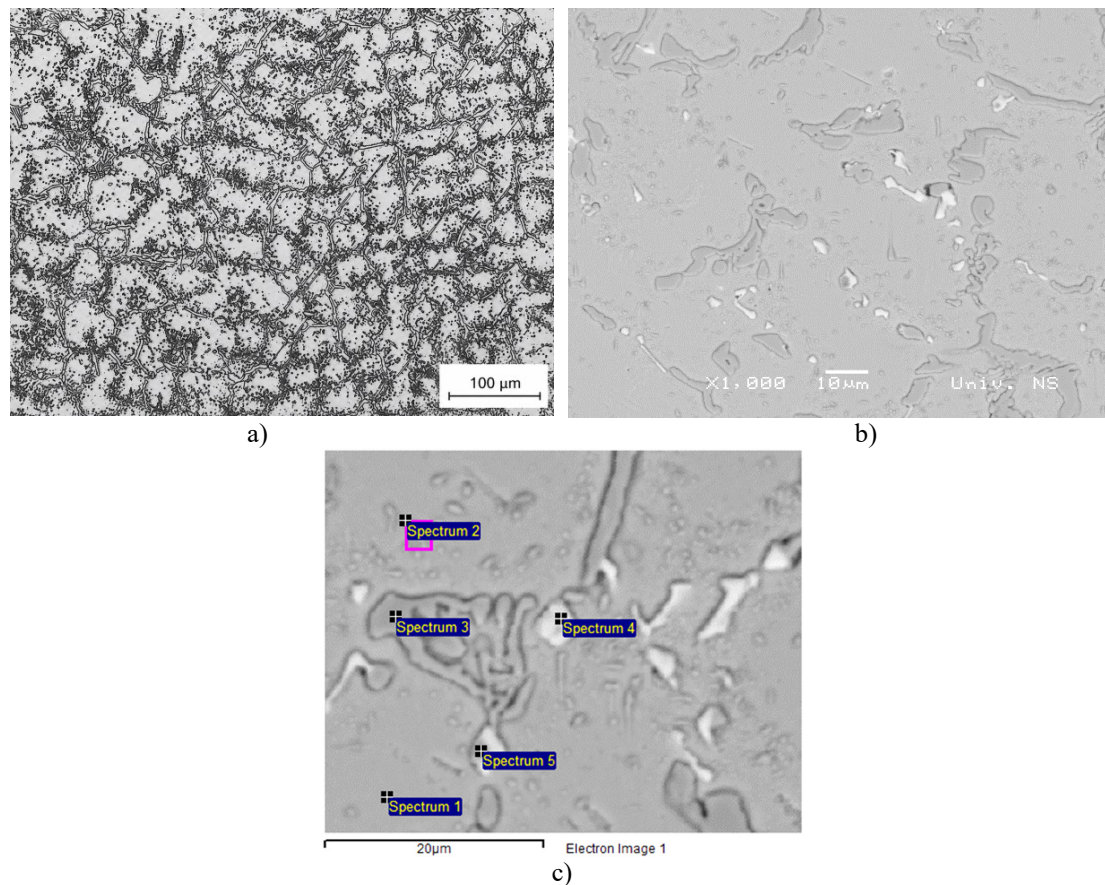


Figure 3: Microstructure after overheating at 1150 °C: a) for 30 min (LM), b) for 120 min (SEM-BSE), c) for 480 min (SEM-BSE)

Table 3: Chemical composition (mass. %) of participating phases in HP40 Nb alloy, corresponding to Fig.3c

Spectrum	C	Si	Ti	V	Cr	Mn	Fe	Ni	Nb
Spectrum 1	21.47	2.81			18.69	1.11	28.13	27.79	
Spectrum 2	31.43	2.28			20.46	0.94	22.91	21.98	
Spectrum 3	36.64				48.06	0.90	9.76	4.63	
Spectrum 4	70.21		2.31	0.26	2.65		1.58	1.26	21.72
Spectrum 5	54.73		16.44	0.63	3.67		1.09	0.93	22.51

After 30 min of overheating at 1150 °C, the density of secondary carbides in the austenitic matrix decreased, while chromium-rich grain-boundary carbides became coarser and developed a blocky morphology. Although $M_{23}C_6$ carbides are considered thermodynamically stable, they undergo morphological changes during exposure to elevated temperatures [27]. At the same time, globular secondary carbides appeared in interdendritic regions. These microstructural changes resulted in the highest tensile strength recorded in this investigation (582 MPa), while elongation remained relatively high (8.76 %).

Increasing the overheating time to 120 min promoted further coarsening of primary and secondary carbides. EDS analysis confirmed the presence of chromium-rich $M_{23}C_6$ carbides and thermally stable niobium-rich MC carbides, indicating that overheating affected carbide morphology rather than carbide type.

After 480 min, the amount of secondary carbides decreased further due to coarsening and partial dissolution. Dissolution occurred both within dendritic regions and near primary grain-boundary carbides, increasing the fraction of the austenitic matrix. Consequently, the tensile strength decreased to 553 MPa, while elongation remained relatively high (7.93 %). The results indicate that prolonged exposure at 1150 °C reduces precipitation strengthening because carbide coarsening and dissolution become dominant microstructural processes.

3.2. Mechanical Properties

The effects of overheating temperature and holding time on the mechanical properties of the HP40-0.63Nb alloy are shown in Table 4 and Figures 4-6.

3.2.1. Hardness

The hardness results are presented in Table 4. The hardness of the as-cast alloy was 215 HV10. After overheating at 1150 °C, the hardness increased from 201 HV10 after 30 min to 232 HV10 480 min, which was the highest value measured. At 950 °C, the hardness increased from 197 HV10 after 30 min to 226 HV10 after 480 min. Overall, increasing the overheating time from 30 to 480 min resulted in a progressive increase in hardness at both temperatures. The increase in hardness is attributed to the precipitation of secondary carbides within the austenitic matrix and the associated strengthening effect, which becomes more pronounced with increasing overheating time.

The hardness values obtained in this study are in good agreement with those reported in the literature [18]. Furthermore, Table 4 shows that the highest hardness values were achieved after 480 min of overheating, regardless of the treatment temperature.

Table 4: The hardness values of the alloy HP40-0.63Nb in its initial state and after being overheated at 950 and 1150°C for 30, 120 and 480 min, respectively.

Temperature, [°C]	Hold Time, [min]	Hardness HV10					Average	Standard Deviation
		1	2	3	4	5		
As-cast	-	228	199	222	230	197	215	16
950	30	184	205	197	198	199	197	8
	120	210	205	206	206	206	207	2
	480	224	224	230	226	228	226	3
1150	30	204	196	204	199	203	201	4
	120	204	208	208	209	208	207	2
	480	233	230	230	233	232	232	2

3.2.2. Tensile Characteristics

The influence of overheating time on the tensile strength of the alloy is presented in Fig. 4. Compared with the as-cast condition (450 MPa), all overheated specimens exhibited substantially higher tensile strength.

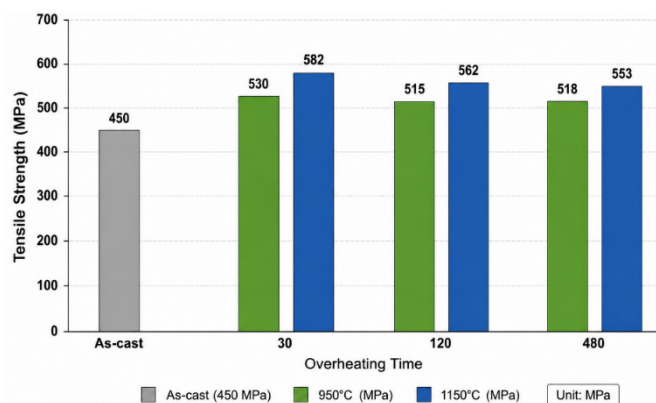


Figure 4: Influence of overheating treatment on the tensile strength of the HP40–0.63Nb alloy

The maximum value of 582 MPa was achieved after 30 min at 1150 °C, whereas overheating at 950 °C resulted in a tensile strength of 530 MPa. Increasing the overheating duration led to a gradual decline in tensile strength; however, even after 480 min, the measured values remained above that of the as-cast alloy. These results suggest that short-term overheating promotes strengthening, while prolonged exposure contributes to microstructural coarsening and a moderate reduction in strength.

The effect of overheating time on yield strength is shown in Figure 5. Relative to the as-cast alloy (240 MPa), overheating at 950 °C produced a marked increase in yield strength, reaching 346 MPa after 120 min, followed by a slight decrease to 342 MPa after 480 min. In contrast, specimens treated at 1150 °C exhibited lower yield strength values, which gradually decreased from 308 MPa to 303 MPa with increasing exposure time. Overall, overheating at 950 °C proved more effective in enhancing yield strength, whereas prolonged exposure at 1150 °C promoted softening.

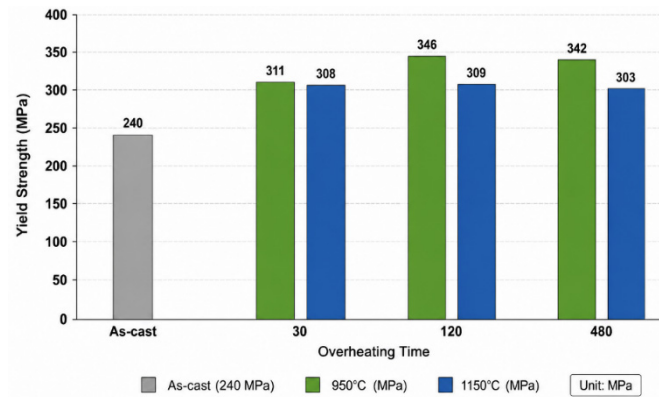


Figure 5: Influence of overheating treatment on the yield strength of the HP40–0.63Nb alloy

Changes in elongation as a function of overheating time are presented in Fig. 6. The highest ductility was recorded in the as-cast condition (9.67%). Exposure to elevated temperatures reduced elongation at both overheating temperatures, with a more pronounced decrease observed at 950 °C.

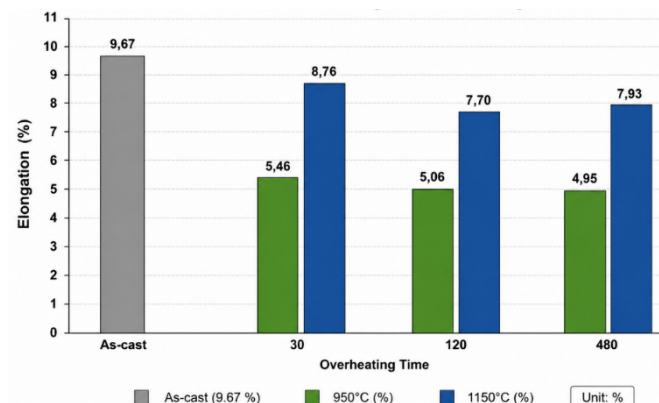


Figure 6: Effect of overheating treatment on the elongation of the HP40–0.63Nb alloy

Throughout the investigated period, specimens overheated at 1150 °C retained higher elongation values, decreasing only slightly from 8.76% after 30 min to 7.93% after 480 min. Although all heat-treated samples exhibited lower ductility than the as-cast alloy, the results indicate that overheating at 1150 °C provides a more favorable balance between strength and plasticity than overheating at 950 °C.

3.3. Correlation Between Carbide Evolution and Mechanical Properties

The evolution of mechanical properties was closely related to time-dependent changes in carbide morphology and distribution. Short-term overheating promoted the precipitation of fine secondary chromium-rich carbides within the austenitic matrix, leading to precipitation strengthening [26,28]. With increasing exposure time, carbide coarsening and partial dissolution reduced the effectiveness of precipitation strengthening, resulting in a gradual decrease in tensile strength. In contrast, niobium-rich MC carbides remained stable throughout all overheating treatments, confirming their superior thermal stability and their contribution to maintaining microstructural stability during prolonged exposure at elevated temperatures [22,23,25].

4. CONCLUSIONS

1. Analysis of variance revealed that overheating exposure time is the dominant factor affecting the mechanical properties of the HP40-0.63Nb alloy, whereas overheating temperature was found to be statistically significant only for tensile strength.

2. Overheating at 950 °C for 120 min exhibited the most favorable effect on the mechanical properties of the alloy, resulting in an increase in yield strength without a statistically significant reduction in elongation.
3. Increasing overheating exposure time promoted the evolution of the carbide phase, characterized by a reduction in the amount of secondary carbides within the austenitic matrix and the growth of $M_{23}C_6$ carbides. The highest tensile strength, 582 MPa, was achieved after overheating at 1150 °C for 30 min, which was associated with the presence of coarser and uniformly distributed secondary carbides.
4. Overheating at 1150 °C for 480 min led to the dissolution of secondary carbides and a reduction in the amount of primary carbides due to their transformation and dissolution, indicating reduced microstructural stability of the alloy at elevated temperatures.
5. The obtained results demonstrate that the changes in mechanical properties are directly related to the evolution of the carbide phase during overheating, with exposure time being the key parameter controlling the microstructural stability and performance of the HP40-0.63Nb alloy.

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