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UTICAJ TERMIČKE OBRADE NAKON PROCESA IZRADE NA SVOJSTVA MARTENZITNOG NERĐAJUĆEG ČELIKA 15-5 PH PROIZVEDENOG POSTUPKOM ADITIVNE PROIZVODNJE ŽIČANIM ELEKTRIČNIM LUKOM (WAAM)

EFFECT OF POST-PROCESSING HEAT TREATMENT ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF WIRE ARC ADDITIVELY MANUFACTURED 15-5 PH MARTENSITIC STAINLESS STEEL

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Rezime: U radu je istraženo navarivanje nerđajućeg čelika 15-5 PH postupkom direktnog nanošenja energije (DED), nakon čega je sprovedena termička obrada u vidu rastvornog žarenja i precipitacionog ojačavanja (PH) radi poboljšanja homogenosti mikrostrukture i mehaničkih svojstava. Parametarska analiza izvršena je kako bi se ispitao uticaj rastvornog žarenja i starenja pri različitim kombinacijama temperature i vremena zadržavanja. Karakterizacija materijala obavljena je u stanju nakon navarivanja, nakon rastvornog žarenja i nakon precipitacionog ojačavanja primenom metalografske analize, ispitivanja tvrdoće, zateznih ispitivanja i ispitivanja udarne žilavosti. Mikrostruktura u stanju nakon navarivanja sastojala se pretežno od martenzitnih letvica sa zaostalim δ-feritom. Optimizacija režima rastvornog žarenja bila je neophodna za postizanje homogene austenitne mikrostrukture pre procesa starenja. Nakon precipitacionog

ojačavanja, materijal je pokazao mehanička svojstva uporediva sa svojstvima konvencionalno proizvedenog nerđajućeg čelika 15-5 PH.

Ključne reči: DED-Arc postupak, usmereno taloženje energijom pomoću električnog luka, termička obrada, mikrostruktura, mehanička svojstva, precipitaciono očvršćavajući martenzitni nerđajući čelik 15-5 PH.

Abstract: This paper investigates the directed energy deposition (DED) of 15-5 PH stainless steel followed by post-processing through solution annealing and precipitation hardening (PH) to enhance microstructural homogeneity and mechanical performance. A parametric study was conducted to evaluate the effects of solution annealing and aging under various temperature and time combinations. Material characterization was performed in the as-deposited, solution-annealed, and precipitation-hardened conditions using microscopy, hardness testing, tensile testing, and impact toughness measurements. The as-deposited microstructure consisted primarily of martensitic laths with retained δ-ferrite. Optimization of the solution annealing treatment was essential to achieve a homogeneous austenitic microstructure prior to aging. Following precipitation hardening, the material exhibited mechanical properties comparable to those of conventionally manufactured 15-5 PH stainless steel.

Key words: Direct Energy Deposition - Arc (DED-Arc), heat treatment, microstructure, mechanical properties, 15-5 PH martensitic stainless steel.

1. INTRODUCTION

Wire arc additive manufacturing (WAAM) is a directed energy deposition process in which an electric arc melts metallic wire to build components layer by layer. Its high deposition rates and suitability for producing large metal components have encouraged its application to stainless steels [1]. Repeated melting, solidification and reheating generate microstructures that differ from those of conventionally manufactured materials. Microsegregation, phase transformations and precipitation during deposition can therefore influence both the as-deposited properties and the response to subsequent heat treatment [2].

The precipitation-hardening martensitic stainless steel 15-5 PH offers a combination of strength and corrosion resistance, with mechanical properties adjustable through solution treatment and aging. Solution treatment promotes chemical homogenisation and dissolution of soluble precipitates, while subsequent cooling produces a predominantly martensitic matrix. Aging strengthens this matrix through the formation of fine Cu-rich precipitates. The resulting properties depend on aging temperature and duration, which govern precipitation and the evolution of the matrix. At higher temperatures or longer holding times, precipitate coarsening and austenite formation can alter the balance between strength and ductility [3].

For WAAM materials, the thermal history during deposition must also be considered when selecting heat-treatment conditions. Xu et al. [4] demonstrated partial aging and spatially non-uniform precipitation in wire arc additively manufactured maraging steel, illustrating how repeated thermal cycling can produce heterogeneous properties. Although its precipitation chemistry differs from that of 15-5 PH, this finding supports evaluating heat treatments specifically for the deposited material. For WAAM 15-5 PH, Guo et al. [5] investigated the microstructure and mechanical properties before and after treatment at 1050 °C for 1 h. Their study provides a basis for examining the additional effects of aging on the balance between strength, ductility and impact resistance.

The present study examines the influence of post-deposition heat treatment on WAAM 15-5 PH stainless steel. Following solution treatment at 1105 °C for 1.5 h, aging at 400–600 °C is assessed through microstructural observations and hardness measurements. Tensile properties and Charpy absorbed energy are compared for the as-deposited condition and specimens aged for 2.5 h, establishing how aging temperature affects the resulting mechanical-property relationship.

2. MATERIALS AND METHODS

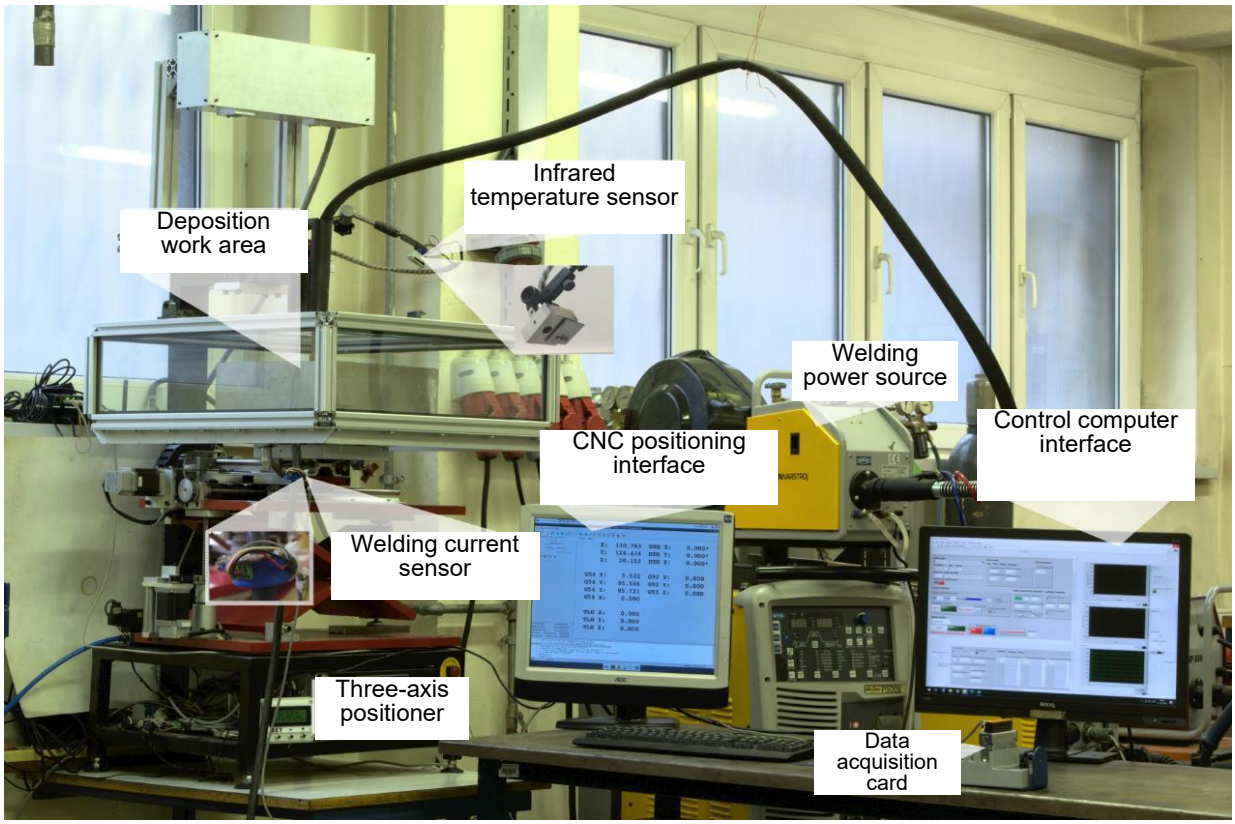
Wall specimens were manufactured by wire arc additive manufacturing (WAAM) using a commercial 3Dprint AM 15-5 PH stainless steel wire (1.4545) with a diameter of 1.2 mm. The nominal wire composition was 0.02 C, 0.50 Si, 0.50 Mn, 14.8 Cr, 4.5 Ni, 3.3 Cu and 0.28 Nb, with Fe as the balance, in wt.%. Deposition was done on 304 stainless steel (1.4301) substrates measuring 200 × 120 × 10 mm. The WAAM system comprised a Daihen Welbee P500L welding power source, a three-axis CNC positioning table and a stationary welding torch oriented perpendicular to the substrate (Figure 1a). Each wall consisted of 31 layers, with five overlapping passes per layer at a pass overlap of 55%. The arc start and stop positions were alternated between successive layers. The deposition strategy, a representative wall and the specimen extraction locations are shown in Figures 1b–d.

Deposition used pulsed gas metal arc welding (GMAW) with a deposition current of 120 A, an arc voltage of 19.9 V, a travel speed of 500 mm/min and a wire feed speed of 5.1 m/min. Shielding was provided by a mixture of 98% Ar and 2% CO_2 at a flow rate of 12 L/min. The shielding arrangement is shown in Figure 2. The substrate was preheated to 150 °C, and the interpass temperature was controlled at 120 °C using a Melexis MLX90614 infrared temperature sensor integrated into the process control system.

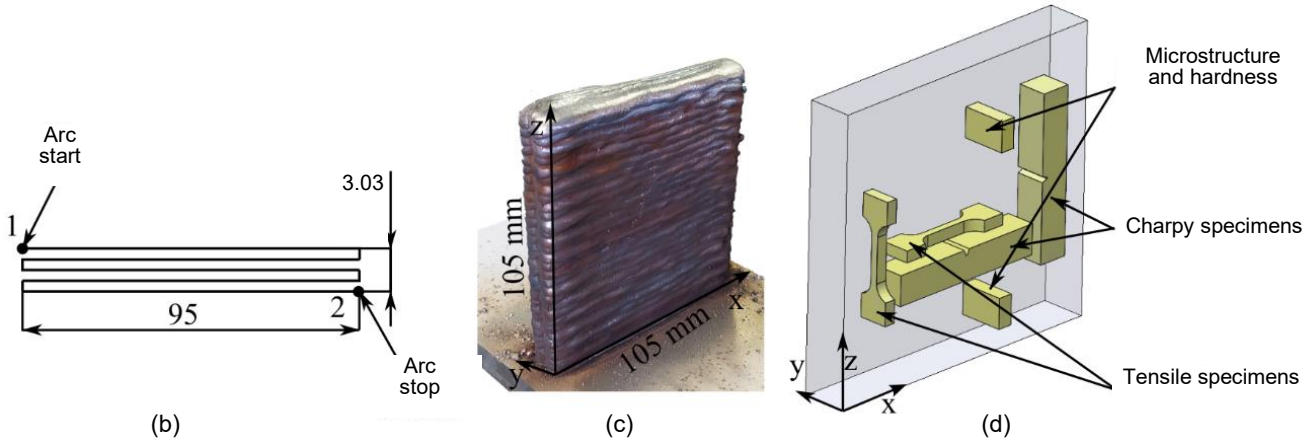
The material was examined in the as-deposited (AD), solution-treated (S) and solution-treated and aged (S+A) conditions. Solution treatment was done at 1105 °C for 1.5 h in a Pika tube furnace under an air atmosphere, followed by water quenching to approximately 20 °C. The furnace was heated at 35 °C/min and stabilised at the target temperature for 1.5 h before specimen insertion. Subsequent aging was done in an Inko EM2 chamber furnace under an air atmosphere at 400, 500 and 600 °C for 2.5 h, followed by air cooling to approximately 20 °C. The heat-treatment sequence is illustrated in Figure 3. Hardness measurements covered all nine aging conditions, whereas the microstructural comparison and tensile and Charpy results presented here focus on specimens aged for 2.5 h.

Metallographic specimens measuring 15 × 10 × 5 mm were extracted from the upper and lower regions of the walls and examined on sections parallel to the build direction. Specimens were ground sequentially using abrasive grades 220, 320, 500, 800 and 1000, followed by polishing with 3 and 1 μm diamond suspensions. The AD and S specimens were etched with Vilella's reagent, comprising 100 mL ethanol, 5 mL hydrochloric acid and 1 g picric acid. Aged specimens were etched with aqua regia, prepared using nitric and hydrochloric acids in a volume ratio of 1:3. Microstructures were examined using a Keyence VHX-6000 digital optical microscope.

Vickers hardness was measured using a Zwick ZHU Z2.5 hardness tester at a load of 98.1 N (HV10) and a dwell time of 11 s. Three to five indentations were made at each measurement location or material condition. In the AD condition, hardness was measured along the wall height to assess variation in the build direction. For the S and S+A conditions, measurements were made on the specimens used for metallographic examination.



(a) WAAM system and its principal components.



(b) (c) (d)

Figure 1. (a) WAAM system used to deposit 15-5 PH stainless steel; (b) deposition toolpath; (c) representative deposited wall; and (d) specimen extraction locations. Dimensions are in millimetres.

Tensile and Charpy V-notch tests were done on the AD material and specimens solution-treated and subsequently aged for 2.5 h at each selected temperature. Tensile testing used an Instron 8802 servohydraulic testing system equipped with an extensometer to determine the 0.2% proof stress, ultimate tensile strength and elongation after fracture. Charpy specimens were sectioned from the walls, milled and ground to their final dimensions, and V-notches were produced by broaching. Impact testing was done using an Amsler RPK 300 pendulum in accordance with EN ISO 148-1, with absorbed energy, KV, reported in joules. Three specimens were tested for each material condition in both tensile and impact testing.

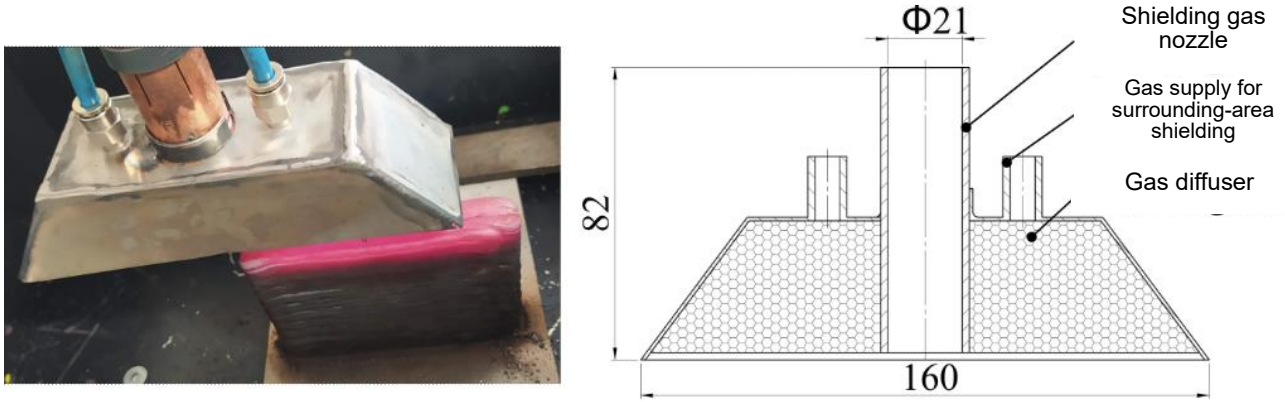


Figure 2. Shielding nozzle used to protect the melt pool and surrounding deposited material from oxidation: photograph and cross-sectional schematic. Dimensions are in millimetres.

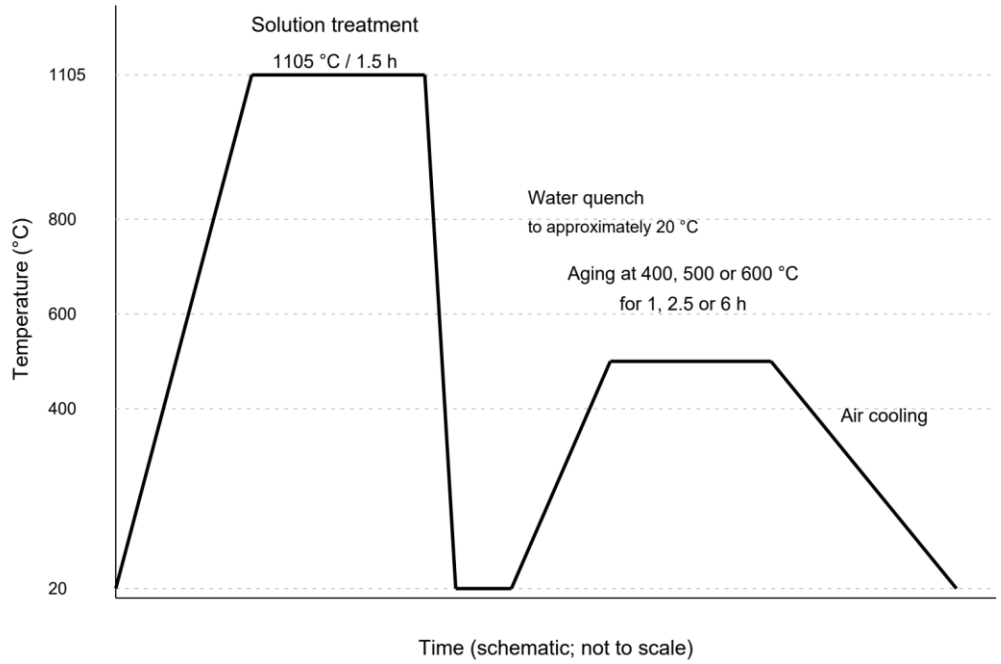


Figure 3. Heat-treatment sequence used for the reported aging experiments: solution treatment at 1105 °C for 1.5 h, water quenching to approximately 20 °C, aging at 400, 500 or 600 °C for 1, 2.5 or 6 h, and air cooling. The time axis is schematic.

3. RESULTS AND DISCUSSION

The optical micrographs in Figure 4 show the influence of aging temperature on the etched microstructure after solution treatment at 1105 °C for 1.5 h and aging for 2.5 h. At 400 and 500 °C, the microstructure retains the elongated features associated with the martensitic matrix. At 600 °C, these features appear more distinctly acicular, indicating changes in the matrix during aging. Optical contrast alone does not establish a transformation from lath to needle-shaped martensite or identify the phases responsible for these changes. Peng et al. [6], using transmission electron microscopy and X-ray diffraction, associated aging of conventionally produced 15-5 PH steel with progressive changes in the martensitic substructure, Cu-precipitate coarsening and austenite reversion. These mechanisms provide a basis for interpreting the present mechanical-property trends, although nanoscale precipitates and reverted austenite cannot be confirmed from the optical micrographs.

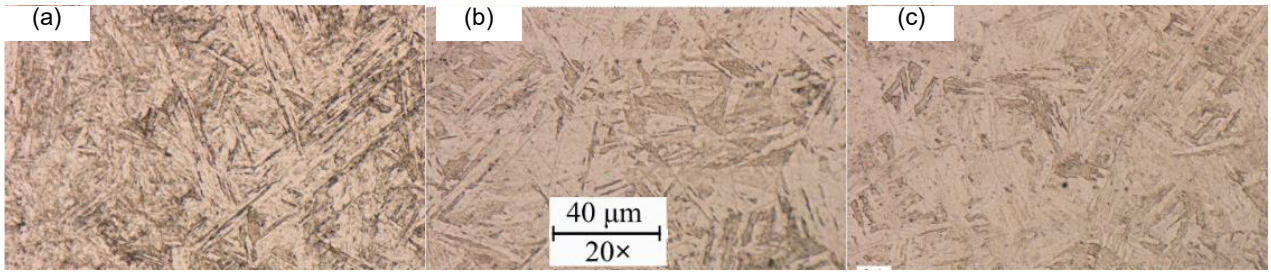


Figure 4. Optical micrographs of WAAM 15-5 PH stainless steel after solution treatment at 1105 °C for 1.5 h and aging for 2.5 h at (a) 600 °C, (b) 500 °C and (c) 400 °C. The images show the etched martensitic morphology; nanoscale Cu precipitates cannot be resolved by optical microscopy. Original magnification: 20×.

The as-deposited hardness profile in Figure 5 is relatively uniform within the deposited wall, with local mean values of approximately 337–353 HV10 and a reported average of 348 ± 4 HV10. No systematic decrease in hardness with increasing build height is apparent. The substantially lower value of 210 ± 14 HV10 corresponds to the measurement below the substrate–deposit interface and should therefore be distinguished from the deposited material. The limited variation within the wall indicates a relatively consistent indentation response despite the different thermal histories experienced along the build direction. This does not necessarily imply uniform phase fractions or precipitation states. In comparison, Xu et al. [4] observed a build-height-dependent hardness variation in WAAM maraging steel arising from partial aging during deposition. The difference illustrates that the effect of repeated thermal cycling depends on alloy chemistry and deposition conditions.

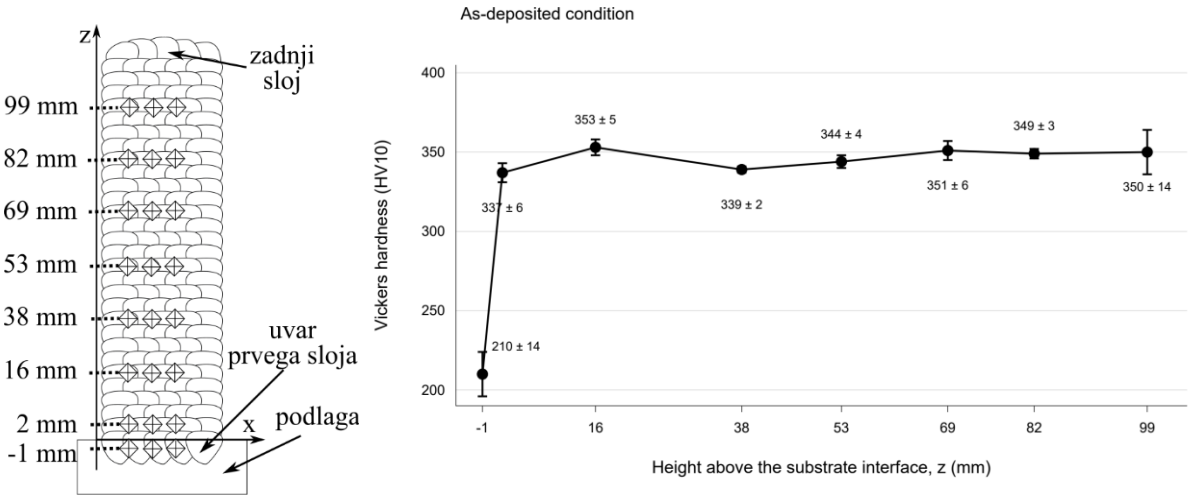


Figure 5. Vickers hardness profile along the build direction in the as-deposited wall. The position z = 0 denotes the substrate–deposit interface; the measurement at z = −1 mm is below the interface. Labels and error bars reproduce the values and reported uncertainties in the source figure.

Solution treatment reduced hardness to 326 ± 9 HV10 (Figure 6), approximately 6% below the reported as-deposited average. This decrease is consistent with dissolution of strengthening precipitates that may have formed during deposition and modification of the matrix during solution treatment and subsequent quenching. Nevertheless, hardness alone cannot establish

complete chemical homogenisation or quantify the contribution of individual microstructural constituents.

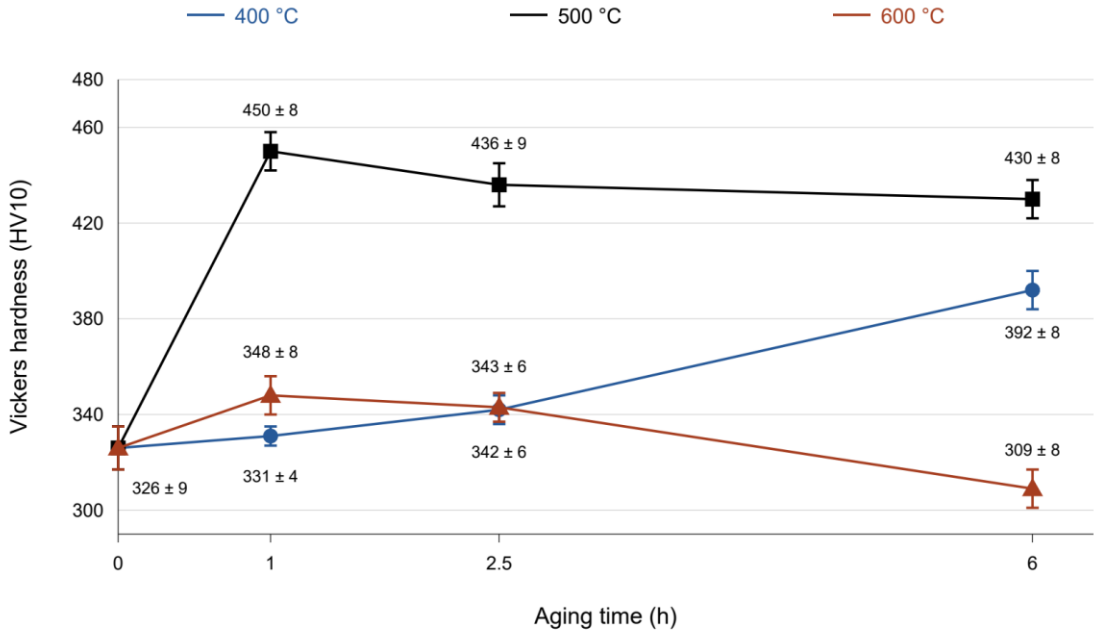


Figure 6. Effect of aging temperature and time on Vickers hardness after solution treatment at 1105 °C for 1.5 h. The value at zero aging time represents the solution-treated condition. Labels and error bars reproduce the source values and reported uncertainties.

The subsequent aging response depended strongly on temperature and holding time. At 400 °C, hardness increased from 331 ± 4 HV10 after 1 h to 342 ± 6 HV10 after 2.5 h and 392 ± 8 HV10 after 6 h. The continued increase indicates that the maximum hardening response had not been reached within the investigated time range. At 500 °C, the highest measured hardness, 450 ± 8 HV10, was obtained after 1 h, followed by a gradual decrease to 436 ± 9 and 430 ± 8 HV10 after 2.5 and 6 h, respectively. This behaviour is consistent with accelerated precipitation of strengthening Cu-rich particles, followed by the onset of overaging [3,6]. The 1 h condition represents the highest hardness among the tested conditions rather than a precisely resolved kinetic maximum.

At 600 °C, hardness decreased from 348 ± 8 HV10 after 1 h to 343 ± 6 HV10 after 2.5 h and 309 ± 8 HV10 after 6 h. This trend is consistent with an overaging regime in which precipitate coarsening and matrix softening outweigh further precipitation strengthening. Austenite reversion may also contribute: Peng et al. [6] detected reverted austenite in 15-5 PH steel aged at 550 °C and above. Its contribution to the present hardness decrease remains an interpretation, since phase fractions were not measured in the results presented here.

The tensile and Charpy results in Table 1 demonstrate that similar hardness values can accompany different mechanical responses. The as-deposited material exhibited a proof stress of 640 ± 28 MPa, an ultimate tensile strength of 1016 ± 6 MPa, an elongation of 17.4 ± 1% and a Charpy absorbed energy of 42.5 ± 2.5 J. After solution treatment and aging at 400 °C for 2.5 h, proof stress increased to 941 ± 51 MPa, whereas ultimate tensile strength increased more moderately to 1105 ± 7 MPa. Elongation remained comparable at 18.2 ± 2%, and absorbed energy was essentially unchanged at 42.5 ± 2.4 J. Thus, this treatment increased resistance to the onset of

plastic deformation without a discernible loss of elongation or absorbed energy within the reported scatter, despite hardness remaining close to the as-deposited value.

Table 3. Mechanical properties of WAAM 15-5 PH stainless steel in the as-deposited condition and after solution treatment at 1105 °C for 1.5 h followed by aging for 2.5 h.

Sample	Condition	0.2% proof stress, $Rp_{0.2}$ (MPa)	Ultimate tensile strength, Rm (MPa)	Elongation after fracture, A (%)	Charpy absorbed energy, KV (J)
AD	As-deposited	640 ± 28	1016 ± 6	17.4 ± 1	42.5 ± 2.5
S+A2	400 °C / 2.5 h	941 ± 51	1105 ± 7	18.2 ± 2	42.5 ± 2.4
S+A5	500 °C / 2.5 h	1130 ± 55	1350 ± 2	10.7 ± 1	20.7 ± 2.7
S+A8	600 °C / 2.5 h	907 ± 49	1012 ± 10	27.2 ± 2	56.4 ± 2.6

Aging at 500 °C for 2.5 h produced the highest strength among the conditions included in Table 3, with a proof stress of 1130 ± 55 MPa and an ultimate tensile strength of 1350 ± 2 MPa. These increases were accompanied by reductions in elongation to 10.7 ± 1% and Charpy absorbed energy to 20.7 ± 2.7 J. Relative to the as-deposited condition, proof stress increased by approximately 77%, while absorbed energy decreased by approximately 51%. The combination of high hardness and strength with reduced elongation and impact energy is consistent with strong precipitation hardening and a reduced capacity for plastic accommodation. However, the absorbed-energy measurements alone cannot distinguish changes in crack initiation resistance from changes in crack propagation resistance or establish the fracture mechanism.

Aging at 600 °C for 2.5 h shifted the property balance towards greater ductility and impact energy. Ultimate tensile strength was 1012 ± 10 MPa, essentially matching the as-deposited value, while proof stress remained higher at 907 ± 49 MPa. Elongation increased to 27.2 ± 2%, and Charpy absorbed energy increased to 56.4 ± 2.6 J. Compared with aging at 500 °C, this condition therefore sacrificed strength while allowing greater deformation and energy absorption before fracture. Its hardness of 343 ± 6 HV10 was almost identical to that obtained after aging at 400 °C for 2.5 h, yet its elongation and absorbed energy were higher. This comparison shows that hardness alone is insufficient for selecting the aging condition: the evolution of the matrix and strengthening precipitates must also be considered alongside tensile and impact properties.

4. CONCLUSIONS

Post-deposition solution treatment and aging enabled adjustment of the mechanical properties of WAAM 15-5 PH stainless steel. The as-deposited wall exhibited relatively uniform hardness, averaging 348 ± 4 HV10, which decreased to 326 ± 9 HV10 after solution treatment at 1105 °C for 1.5 h. Subsequent aging produced a temperature- and time-dependent response, with the highest measured hardness of 450 ± 8 HV10 obtained at 500 °C for 1 h. Longer holding times at this temperature progressively reduced hardness, consistent with the onset of overaging.

Among the conditions evaluated by tensile and Charpy testing, aging at 500 °C for 2.5 h produced the highest proof stress and ultimate tensile strength, reaching 1130 ± 55 and 1350 ± 2 MPa, respectively. However, elongation decreased to 10.7 ± 1% and Charpy absorbed energy to 20.7 ± 2.7 J. Aging at 400 °C for 2.5 h increased proof stress to 941 ± 51 MPa while retaining elongation and absorbed energy comparable to the as-deposited condition.

Aging at 600 °C for 2.5 h provided the highest elongation and absorbed energy among the tested conditions, reaching 27.2 ± 2% and 56.4 ± 2.6 J, respectively. Ultimate tensile strength remained comparable to the as-deposited value, while proof stress increased to 907 ± 49 MPa. The different tensile and impact responses of conditions with similar hardness demonstrate that heat-treatment selection should consider the required balance between strength, ductility and impact resistance.

The observed trends are consistent with precipitation strengthening followed by overaging and matrix softening, although the individual contributions of Cu-precipitate evolution and austenite reversion require further microstructural verification.

Acknowledgements

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