

Development of SA-508 Grade 4N low-alloy steel with modified Mn-Ni-Cr content

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Received 25 September 2025, received in revised form 14 July 2026, accepted 15 July 2026

Abstract

The development of advanced reactor pressure vessel (RPV) steels with improved strength and toughness is essential for ensuring the safety and reliability of nuclear power plants. In this study, a modified SA508 Grade 4N low-alloy steel with reduced Mn-Ni-Cr content was developed through induction melting, followed by austenitization, water quenching, and tempering. Microstructural characterization using optical microscopy, scanning electron microscopy, and X-ray diffraction confirmed the formation of a predominantly tempered lath martensitic microstructure without detectable retained austenite. XRD analysis further indicated recovery of the martensitic lattice after tempering, as evidenced by reduced peak broadening. The developed alloy exhibited a yield strength of 585 MPa, an ultimate tensile strength of 635 MPa, 24% elongation, and 55% reduction in area, satisfying the minimum mechanical property requirements for SA508 Grade 4N Class 3 steel. Charpy impact testing demonstrated the characteristic ductile-to-brittle transition behaviour, with an absorbed energy of 41 J at -82°C and an upper-shelf energy of approximately 115 J at room temperature. Although the upper-shelf impact energy was lower than that reported for conventional SA508 Grade 4N steels, the developed alloy satisfied the specified toughness requirements. It achieved the required combination of strength and ductility. The results demonstrate that an induction-melted, cast SA508 Grade 4N model alloy with reduced Mn-Ni-Cr content can achieve the required mechanical performance after quenching and tempering. In contrast, further optimization of the nickel content may enhance impact toughness.

Key words: martensite, reactor pressure vessel (RPV), synergistic effect, lath martensite, irradiation embrittlement, dislocation.

1. Introduction:

Global energy resources are on the verge of decline; the obsessive use of fossil fuels like coal, oil, and natural gas is driving an energy shortage. The world relies on coal-fired power plants to meet its energy needs, but the widespread use of fossil fuels depletes overall energy resources and drives up prices [1]. To tackle this crisis, the world has been shifting to nuclear energy, a clean, renewable, and economical source of energy. However, some safety issues are associated with nuclear power plants and their components. Therefore, research has been underway to develop a suitable material for the Nuclear reactor pressure vessel (RPV). This important component deter-

mines the safety and lifespan of a nuclear power plant [2]. At the start, the first-generation reactor pressure vessel was fabricated from plates of SA-212B, a C-Si-based steel in the normalized and tempered condition, along with forgings of SA-105 Class 2 and SA-182 F1 modified in the quenched and tempered condition for flanges and valves; these steels were used between 1950 and 1960 because of their high strength and good weldability. Still, its poor impact toughness, reduced strength at high temperatures, and low hardenability created a reason to replace it with steel having better properties. So, it was replaced by Mn-Mo base high-strength steel, specified as SA-302B plate, with improved strength and toughness for RPV, called the second-generation RPV. Later on, the SA-533B

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was developed by alloying SA-302B with Ni ranging from 0.4–1 % [3]. Irradiation embrittlement of RPV material during service makes the first and second generations less reliable, as there are more longitudinal and circumferential welds in the plates, creating more susceptible areas to irradiation embrittlement. To tackle this problem, the world decided to take a shift toward fabricating RPV by forging materials instead of rolled ones to reduce the weld areas, but due to expensive thermal treatment to minimize Hydrogen blistering, it was enforced to use Ni-Cr-Mo steel, eventually, this steel turned into SA-508 class 2 forging material, has been used in the RPV ring forgings, flanges and nozzle [4]. Presently, to meet the power demand, there has been an increasing trend toward constructing nuclear power plants on a large scale. Nonetheless, employing larger pressure vessels necessitates increasing the thickness of these components required by the prescribed endurance strength criteria. However, increasing thickness can result in reduced weldability and toughness. To address these challenges, particularly irradiation-induced embrittlement and the associated loss of toughness, the alloy composition was modified by increasing the nickel (Ni) and chromium (Cr) contents, as both elements play a vital role in enhancing fracture toughness and improving the irradiation resistance of reactor pressure vessel steels [5]. SA508 Grade 4N is a modified version of SA508 Grade 3 with increased nickel (Ni) and chromium (Cr) contents of up to 3.9 and 2.0 wt.%, respectively. These alloying additions enhance hardenability and promote the formation of a predominantly martensitic and bainitic microstructure during quenching. The resulting microstructure provides superior mechanical properties, including higher strength and improved fracture toughness, compared with SA508 Grade 3 steel. Despite these advantages, SA508 Grade 4N has not yet been widely adopted in commercial nuclear power plants because of its relatively high nickel content [6, 7]. The reason for this is the synergistic effect of Ni on irradiation embrittlement, due to the higher Mn content, which is involved in the formation of cluster embryos leading to precipitation in these high-Ni steels. This results in a significant loss of impact toughness. However, decreasing the Mn content reduces the synergistic effect of Ni [8]. In this study, SA-508 Grade 4N low-alloy steel has been developed with modified Mn-Ni-Cr content to determine whether the developed model alloy meets the minimum criteria specified by ASTM A508.

2. Experimental detail

To develop an alloy, steel scrap was used as a primary material, along with Granular carbon and ferroalloys were also utilized. The Charge calculations

Table 1. Summary of the charge calculation for 70 kg heat

Total	70 kg
Scrap	63.75 kg
Carbon	105 gm
Fe Si	317 gm
Fe Mn	200 gm
Fe Mo	640 gm
Fe Cr	1.78 kg
Fe Ni	3.3 kg

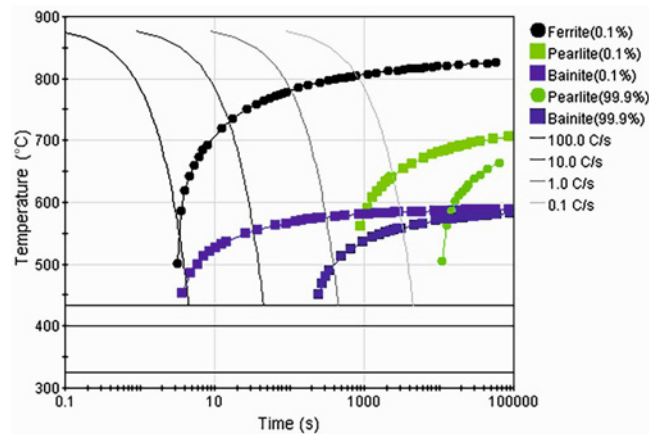


Fig. 1. Simulated continuous cooling transformation (CCT) diagram of the developed SA508 Grade 4N model alloy generated using *JMatPro*.

were performed before the melting process to ensure the correct quantities of materials were used for a 70 kg heat. The quantities of each material were calculated based on the target composition and expected yield during the melting process; the approximate values are listed in Table 1. For melting, a high-frequency induction furnace with a frequency of 1000 Hz and a capacity of 70 kg, lined with silica refractory, was employed.

At the start, the scrap was added, and power was applied. As the scrap melted, granular carbon was added to adjust the carbon content. Ferrosilicon was introduced to deoxidize the bath before the addition of ferroalloys. After deoxidation, ferromanganese, ferromolybdenum, ferrochrome, and ferronickel were added, and the mixture was held for approximately 5 minutes before tapping into a ladle, where a further deoxidizer was added to fully deoxidize the bath. Following tapping, teeming was carried out, where molten metal was poured into a mold. The final product, a slab measuring $5 \times 30 \times 30 \text{ cm}^3$, was obtained. The resulting chemical composition is shown in Table 2, along with the chemical composition of SA-508 Grade 4N. SA-508 Grade 4N [9]. The slab was then cut to produce samples for heat treatment. Before perform-

Table 2. Chemical composition of the developed alloy and SA 508 Gr. 4N

	C	Mn	Si	Ni	Cr	Mo	S	P
SA508 Gr. 4N	0.23 max	0.2–0.4	0.40 max	2.8–3.9	1.5–2.00	0.40–0.60	0.020 max	0.020 max
Model alloy	0.10	0.1	0.316	1.308	1.18	0.4	0.012	0.015

ing the heat treatment, a CCT (Continuous Cooling Transformation) diagram, illustrated in Fig. 1, was generated using *JMatPro software* to identify the austenitizing temperature of the developed alloy. The samples were austenitized at 900 °C for 1 hour to be within a safe margin compared to the temperature indicated by the simulated CCT diagram, followed by water quenching, and then tempered at 655 °C for 60 minutes.

Optical microscopy and scanning electron microscopy (SEM) were used for microstructural observations. The samples were prepared by sectioning, mounting, grinding, and polishing, followed by etching with a 3% nital solution. X-ray diffraction (XRD) analysis was performed using a PANalytical X'Pert PRO MPD diffractometer with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$). A tensile test was conducted using a computerized tensile testing machine with a crosshead speed of 5 mm min^{-1} to examine the tensile properties. The samples were prepared according to ASTM E8 [10]. The tensile samples had a gauge length of 24 mm and a diameter of 6 mm, respectively. Charpy V-notched testing was performed on 300-joule impact testing machines. Samples were prepared according to the ASTM standard E23 for 25, 0, –23, –50 and –88 °C [11].

3. Results and discussion

3.1. Microstructural characterization

Optical microscopy and scanning electron microscopy (SEM) in Figs. 2a,b and 3a revealed that the developed SA508 Grade 4N model alloy exhibits a primarily tempered lath martensitic microstructure following austenitization, water quenching, and tempering. The absence of visible ferritic or bainitic constituents indicates that the selected heat treatment provided sufficient cooling to suppress diffusional transformations, allowing the austenite to transform predominantly into martensite. This transformation behaviour is mainly attributed to the combined addition of Ni, Cr, and Mo, which significantly increases the hardenability of low-alloy steels by delaying ferrite and bainite formation during cooling. Consequently, martensitic transformation can occur throughout the section, producing a relatively homogeneous microstructure suitable for reactor pressure vessel applications. Similar microstructural evolution

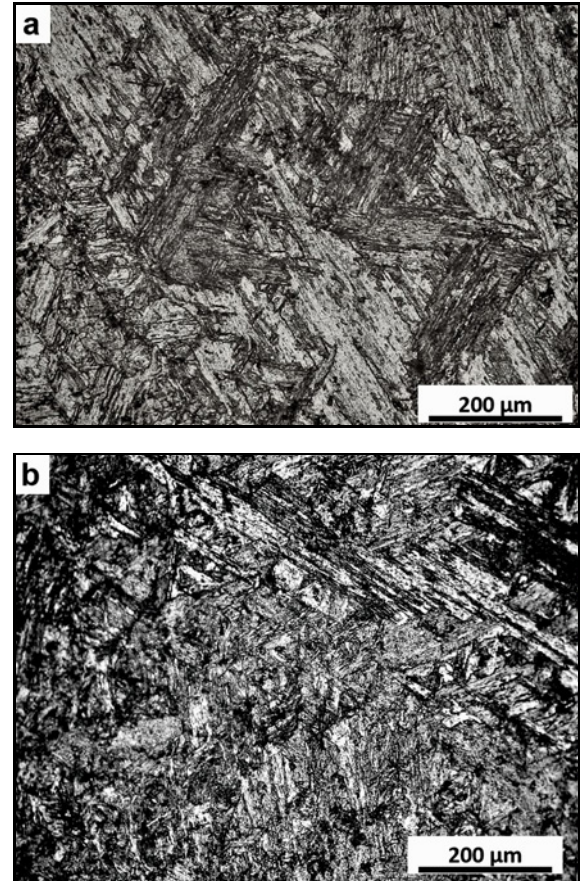


Fig. 2. Optical micrographs of the developed alloy: (a) as quenched condition and (b) quenched-and-tempered condition.

has been widely reported for SA508 Grade 4N steels, where the optimized Ni-Cr-Mo composition promotes the formation of tempered lath martensite [1, 5, 6].

The SEM observations further revealed that the martensitic microstructure demonstrates the characteristic hierarchical arrangement of low-carbon lath martensite. Parallel martensitic laths are grouped into packets with different crystallographic orientations, which originate from the transformation of prior austenite grains. Although the prior austenite grain boundaries cannot be directly identified from the present SEM images, the observed packet morphology clearly indicates the formation of multiple martensitic variants during the diffusionless transformation. This hierarchical arrangement is a characteristic feature of quenched low-carbon martensitic steels and plays an important role in controlling both strength

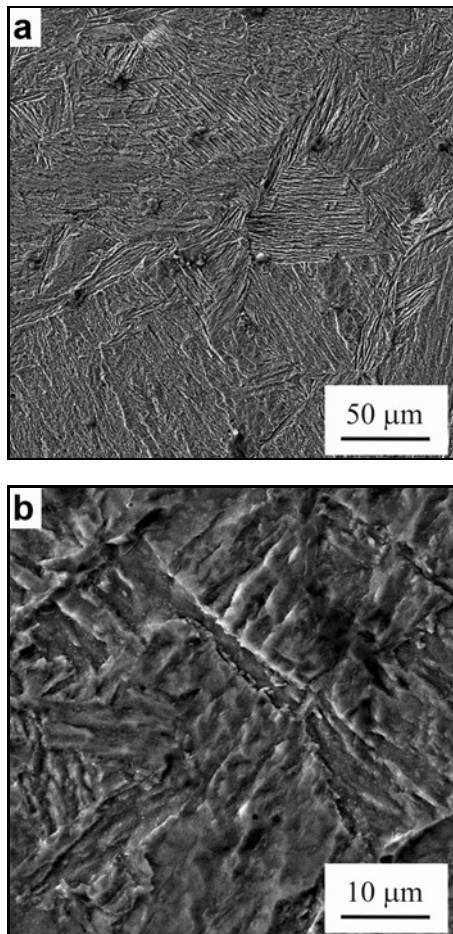


Fig. 3. SEM micrographs of the developed alloy: (a) as quenched condition and (b) quenched-and-tempered condition showing fine carbide precipitates.

and fracture behaviour. Previous investigations have demonstrated that packet and block boundaries generally possess high misorientation angles and therefore act as effective barriers to dislocation motion and cleavage crack propagation. Consequently, the refinement of the martensitic hierarchy improves both the strength and toughness of reactor pressure vessel steels by increasing the effective boundary density within the microstructure [12, 13]. Following tempering at 655°C in Fig. 3b, the overall lath morphology was retained, indicating that the selected tempering treatment promoted recovery rather than recrystallization of the martensitic matrix. During tempering, carbon atoms gradually diffuse from the supersaturated martensite and the high dislocation density is reduced through recovery processes, thereby relieving the residual stresses generated during quenching. Nevertheless, the hierarchical lath structure remains largely preserved [14–16]. Similar behaviour has been reported for SA508 Grade 4N steels tempered between approximately 630 and 660°C, where recovery of martensite without significant microstruc-

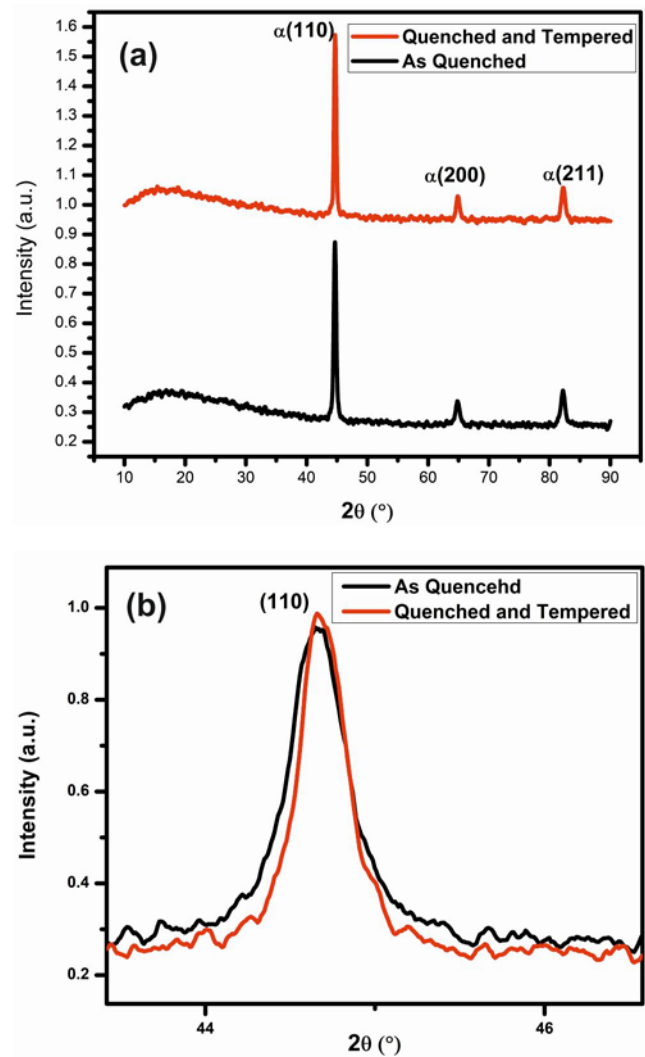


Fig. 4. XRD patterns of the developed alloy: (a) as quenched and quenched-and-tempered conditions and (b) enlarged view of the α -Fe (110) diffraction peak showing the peak shift and reduction in peak broadening after tempering at 655°C for 1 h.

tural coarsening provides an optimum balance between strength and fracture resistance. Although the present investigation did not include chemical characterisation of the precipitates, tempering of SA508 Grade 4N steels is generally accompanied by the precipitation of fine chromium-rich carbides, predominantly $M_{23}C_6$ together with M_7C_3 . Consequently, the retained lath morphology observed in the present investigation is consistent with carbide precipitation reported in previous investigations of tempered SA508 Grade 4N steels, indicating that the selected tempering treatment was appropriate for developing the desired tempered martensitic microstructure [5, 15, 17].

3.2. XRD analysis

To investigate the phase constitution and mi-

Table 3. Quantitative analysis of the α -Fe (110) diffraction peak before and after tempering

Parameter	As-quenched	Quenched & Tempered
Peak position, 2θ ($^\circ$)	44.664	44.692
FWHM ($^\circ$)	0.516	0.456
Apparent crystallite size (nm)	17.0	19.2
Estimated lattice microstrain ($\times 10^{-3}$)*	5.48	4.85

*Note: Apparent crystallite size was calculated using the Scherrer equation from the α -Fe (110) reflection.

crostructural evolution after tempering, X-ray diffraction (XRD) analysis was performed. The results are shown in Fig. 4a; both the as-quenched and quenched-and-tempered specimens exhibit three dominant diffraction peaks corresponding to the α -Fe (110), (200), and (211) planes, confirming that the matrix remained predominantly BCC lath martensite after tempering. Phase identification using *X'Pert High-Score* revealed no detectable retained austenite. Furthermore, no additional diffraction peaks corresponding to carbide phases were observed after tempering, indicating that the precipitated carbides were either nanoscale or present in a volume fraction below the detection limit of conventional laboratory XRD. Similar observations have been reported by Bellot et al. [18], who demonstrated that fine alloy carbides formed during tempering are often undetectable by conventional XRD despite significant carbon redistribution within the martensitic matrix.

A detailed comparison of the α -Fe (110) reflection, shown in Fig. 4b, reveals a slight shift toward higher diffraction angles together with a noticeable reduction in peak broadening after tempering. Quantitative analysis of the (110) peak, as shown in Table 3, indicates that the FWHM decreased from 0.516° in the as-quenched condition to 0.456° after tempering, corresponding to an approximately 11.5 % reduction in peak broadening. The apparent crystallite size, estimated using the Scherrer equation, increased from 17.0 to 19.2 nm, while the estimated microstrain decreased from 5.48×10^{-3} to 4.85×10^{-3} . These results indicate that tempering promoted recovery of the supersaturated martensitic structure through carbon redistribution and partial annihilation of dislocations, thereby relieving lattice distortion without altering the dominant BCC phase. It should be noted that the reported crystallite sizes represent apparent sizes estimated from X-ray peak broadening and should not be interpreted as the actual martensite lath dimensions. Similar reductions in XRD peak broadening and lattice strain during tempering of martensitic steels have been reported by Sun et al. and Zheng et al. [19, 20], who attributed these changes to recovery of lath martensite and relaxation of transformation-induced residual stresses.

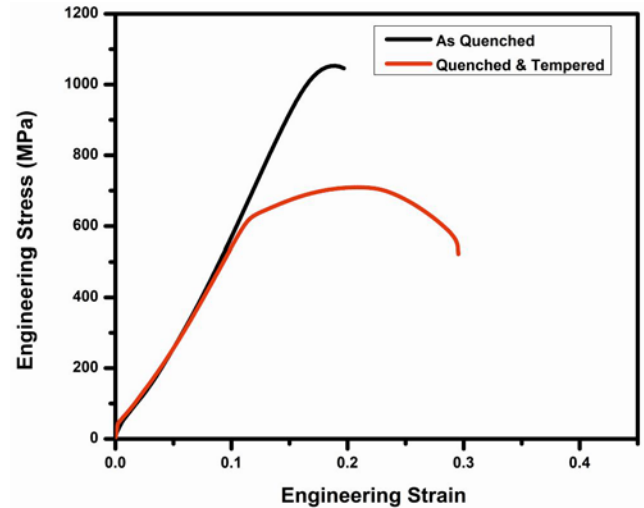


Fig. 5. Engineering stress-strain curves of the developed alloy in the as quenched and quenched-and-tempered conditions.

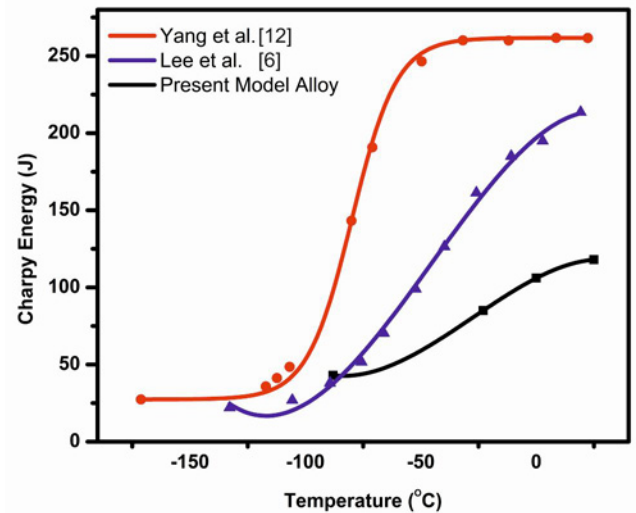


Fig. 6. Comparison of the Charpy V-notch impact energy temperature curves of the present alloy with published SA508 Grade 4N steels reported by Lee et al. and Yang et al. [6, 12]. Literature data were digitized.

3.3. Mechanical properties

A tensile test was performed at room temperature

Table 4. Comparison of the tensile properties of the as quenched and quenched-and-tempered model alloy with the minimum requirements for SA508 Grade 4N Class 3 steel

Mechanical properties	SA508 Grade 4N (ASME)	As quenched	Quenched & Tempered
0.2% Proof stress (MPa)	≥ 485	1041	585
Ultimate tensile strength (MPa)	620–795	1050	635
Elongation (%)	≥ 20	5	24
Reduction of area (%)	≥ 48	8	55

to evaluate the mechanical properties of the developed SA508 Grade 4N model alloy. The tensile properties of the as quenched and quenched-and-tempered conditions are summarized in Fig. 5 and Table 4, while the Charpy impact results are presented in Fig. 6. The Charpy impact tests were performed only on the quenched-and-tempered specimens because the tempered condition represents the final service condition specified for SA508 Grade 4N reactor pressure vessel steels. As expected, the as quenched specimen exhibited significantly higher tensile strength but only 5% elongation, lower than the quenched-and-tempered specimen because of the presence of supersaturated lath martensite with a high dislocation density and substantial residual stresses. Following tempering, the alloy exhibited a 0.2% proof stress of 585 MPa, an ultimate tensile strength of 635 MPa, an elongation of 24%, and a reduction in area of 55%, satisfying the minimum mechanical property requirements specified for SA508 Grade 4N Class 3 steel. The decrease in strength after tempering is attributed to the recovery of the martensitic matrix, relaxation of residual stresses, and precipitation of fine alloy carbides, whereas the improvement in ductility results from the reduction in lattice strain and redistribution of carbon from supersaturated martensite. Such a strength-ductility trade-off is characteristic of tempered martensitic steels and has been widely reported for SA508 Grade 4N alloys. Similar behaviour was reported by Lee et al. [6], who demonstrated that SA508 Grade 4N model alloys possess superior mechanical properties compared with conventional Grade 3 steels because of the combined effects of increased Ni and Cr contents and the resulting tempered martensitic microstructure. Furthermore, they reported that increasing Ni and Mo contents increases the yield strength, whereas Cr contributes primarily to improved fracture toughness through carbide refinement.

The engineering stress-strain curve exhibits continuous strain hardening followed by stable plastic deformation before fracture, showing that the tempered martensitic microstructure retained sufficient work-hardening capability. The Charpy V-notch impact results exhibited the characteristic ductile-to-brittle transition behaviour of low-alloy reactor pressure vessel steels, with the absorbed impact energy increasing from approximately 41 J at -82°C to 115 J

at room temperature. The alloy therefore satisfies the SA508 Grade 4N toughness requirement while maintaining an excellent combination of strength and ductility [9]. Figure 6 compares the impact transition behaviour of the present model alloy with the published results of Lee et al. [6] and Yang et al. [12]. Although all three materials exhibit the characteristic ductile-to-brittle transition of SA508 Grade 4N steels, the present alloy shows a lower upper-shelf impact energy. This difference is likely associated with differences in manufacturing route and alloy chemistry. Lee et al. and Yang et al. [6, 12] investigated vacuum-melted and forged SA508 Grade 4N steels with conventional Ni-Cr-Mo compositions, whereas the present alloy was produced by induction melting and casting with a modified Mn-Ni-Cr composition. Vacuum melting and forging generally produce cleaner and more homogeneous steels with fewer casting defects and finer microstructures, resulting in improved impact toughness. In addition, the reduced Ni content in the present alloy decreases hardenability and limits refinement of the effective martensite packet structure, contributing to the comparatively lower upper-shelf energy. Nevertheless, the developed alloy satisfied the minimum impact toughness requirement specified for SA508 Grade 4N Class 3 steel.

Previous studies have shown that increasing the Ni content enhances hardenability, increases the martensite fraction, and refines the effective packet size, thereby lowering the ductile-to-brittle transition temperature and improving impact toughness. Chromium further improves toughness by promoting a finer and more homogeneous distribution of Cr-rich carbides, which reduces local stress concentrations during crack propagation. Furthermore, recent work by Yang et al. [12] further demonstrated that the superior fracture toughness of SA508 Grade 4N steel originates from its lath martensitic microstructure, where the high density of high-angle interfaces effectively suppresses cleavage crack propagation compared with the ferrite bainite microstructure of SA508 Grade 3 steel. Moreover, irradiation studies conducted by Kim et al. [21] showed that SA508 Grade 4N retains excellent strength and toughness even after neutron exposure, confirming the suitability of the Ni-Cr-Mo alloy design for advanced reactor pressure vessel applications.

4. Conclusions

A modified SA508 Grade 4N low-alloy steel with reduced Mn-Ni-Cr content was successfully developed through induction melting followed by quenching and tempering. Based on the present investigation, the following conclusions can be drawn:

1. The selected heat treatment produced a predominantly tempered lath martensitic microstructure consisting of martensite laths and packets. The retained lath morphology after tempering indicates recovery of the martensitic structure without significant microstructural coarsening.

2. X-ray diffraction analysis confirmed a predominantly body-centred cubic martensitic structure with no detectable retained austenite. Tempering reduced the α -Fe (110) peak broadening by approximately 11.5%, increased the apparent crystallite size from 17.0 to 19.2 nm, and reduced the estimated microstrain, indicating recovery of the martensitic lattice and relaxation of residual stresses.

3. The developed alloy exhibited a yield strength of 585 MPa, an ultimate tensile strength of 635 MPa, 24 % elongation, and 55 % reduction in area, satisfying the minimum mechanical property requirements specified for ASTM SA508 Grade 4N steel.

4. Charpy impact testing exhibited the characteristic ductile-to-brittle transition behavior of reactor pressure vessel steels. Comparison with published SA508 Grade 4N steels showed that the present alloy exhibited a lower upper-shelf impact energy, which is likely associated with differences in alloy chemistry and manufacturing route. Nevertheless, the developed alloy satisfied the minimum toughness requirement specified for ASTM SA508 Grade 4N steel.

5. The present study demonstrates that an induction-melted and cast SA508 Grade 4N model alloy with reduced Mn-Ni-Cr content can achieve the required combination of strength, ductility, and impact toughness after quenching and tempering. Further optimization of the nickel content within the ASTM SA508 Grade 4N specification may further improve impact toughness while preserving the desirable tempered martensitic microstructure.

Acknowledgements

This study was supported by the Pakistan Institute of Engineering and Applied Sciences (PIEAS) and the National Institute of Laser and Optics (NILOP).

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