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The solution treatment and precipitation kinetics of AISI 348 alloy for use in nuclear fuel cladding

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1. Introduction

AISI 348 austenitic stainless steel is a niobium-stabilized alloy widely used in high-responsibility components in the aerospace and nuclear industries due to its corrosion resistance. However, the mechanical forming history, such as cold rolling to obtain thin sheets, introduces severe deformations and microstructural heterogeneities, including the retention of delta ferrite out of thermodynamic equilibrium. Controlling the dissolution of this phase and properly quantifying secondary precipitates, such as carbonitrides after heat treatments are fundamental to assuring the mechanical properties of the steel.

2. Methodology

The starting material consisted of an as-received 1.2 mm thick sheet of AISI 348 austenitic stainless steel. The sheet was subjected to a solution heat treatment at 1100 °C for 1 hour in an air furnace, followed by immediate cooling in water at room temperature. To enable the analysis of minority phases and prevent peak masking by the material's bulk, the austenitic matrix was selectively dissolved via wet chemical extraction using Berzelius reagent (1). The resulting insoluble residue (composed of stable precipitates) was filtered through a sub-micrometric polytetrafluoroethylene (PTFE) membrane with a pore size of 3 μm, followed by rinsing with deionized water and drying in a desiccator for 24 hours. Crystallographic characterization of the residual powder from both the as-received and solution-treated conditions of the AISI 348 steel was performed by X-ray diffraction (XRD) using a Rigaku SmartLab diffractometer (2). The collected XRD pattern of the solution-treated condition was processed using specialized software, employing Pseudo-Voigt functions for peak profile fitting and linear background modeling.

3. Results and discussion

The microstructural characterization and integrated crystallochemical analysis of the insoluble precipitates obtained by wet dissolution using the Berzelius method in the Nb-stabilized AISI 348 austenitic stainless steel revealed a scenario of complex stoichiometric evolution governed by thermal history and element solubility constraints in the alloy. In the as-received condition, the microstructure characterized by scanning electron microscopy indicated the coexistence of two distinct populations of secondary phases. In this condition, both the secondary electron image and the X-ray diffraction pattern, see Figures 1(a and b) and 2a, revealed delta ferrite as the majority phase in the residue.

In the initial state (as-received sample), the delta ferrite is distributed as a continuous, interconnected, and massive network along the austenitic matrix (vermicular or banded morphology, typical of rolling). This common phenomenon for microstructurally austenitic steel is attributed to severe work hardening and the formation of segregated bands of alpha-stabilizing elements (such as Cr and Nb) generated during the industrial cold rolling process to achieve the 1.2 mm thickness (3). Compression deformation of the grains introduces a strong preferential orientation or crystallographic texture (4), which artificially multiplies the intensity of the body-centered cubic (BCC) structure peaks in the XRD due to the alignment of crystal planes parallel to the sheet surface (4). Figure 1a shows the presence of delta ferrite in the material's bulk, while Figure 1b details this phase.

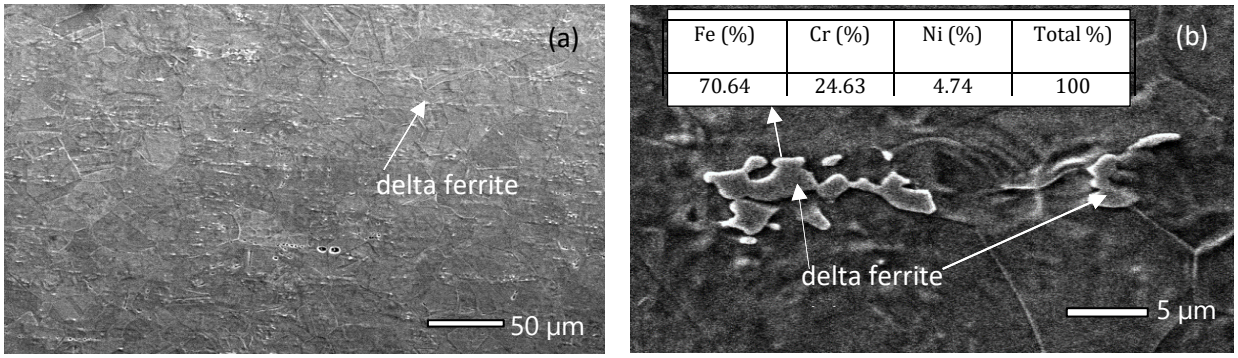
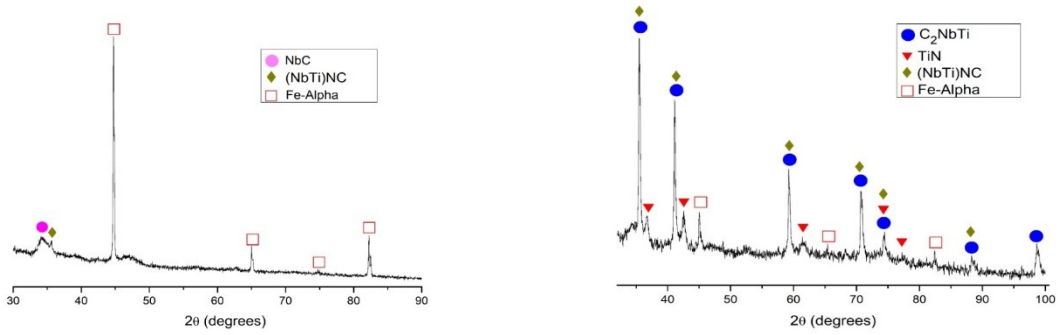


Figure 1. Morphological and chemical characterization of the AISI 348 steel bulks in the as-received condition: (a) Electron micrograph showing the presence of delta ferrite in the matrix; (b) magnification of interest highlights the delta ferrite and the elemental chemical composition of the delta ferrite phase, mass %.



(a) **(b)**

Figure 2. (a) X-ray diffraction (XRD) pattern of the residues extracted by chemical dissolution (Berzelius) of the 348 steel in the as-received condition, presenting the phase identifications of NbC, (NbC,Ti)NC, and delta ferrite**;** (b) X-ray diffraction (XRD) pattern of the residues extracted by chemical dissolution (Berzelius method) of the 348 steel solution-treated at 1100 °C for 1 h, presenting the phase identifications of C_2NbTi, TiN, (Nb,Ti)NC, and delta ferrite.

The second population of secondary phases, as shown in Figure 2a, was identified as NbC and (Nb,Ti)NC. The NbC phase was observed via transmission electron microscopy (TEM), see Figures 3a and 3b. Due to its reduced (nanometric) dimensions, it comprises a widespread distribution of secondary, nanometer-sized niobium carbides that nucleated homogeneously from the remaining solute enriched in niobium and carbon during the subsequent cooling of the material. Although the X-ray diffraction was performed directly on the concentrated residue

obtained by Berzelius extraction, the diffractogram corresponding to the as-received state (Figure 2a) presented a flat profile, lacking well-defined peaks. From the perspective of X-ray diffraction physics, this phenomenon stems from the combination of the reduced crystallite size and the volume fraction detection limit (4).

The widespread population of nanometric secondary carbides induced a severe geometric broadening of the reflections (2θ) around 34.8°. Since this nanometric mass overwhelmingly dominates the volume fraction of the initial residue, the rare primary micrometric carbonitrides dispersed in the powder fall below the detection limit of the X-ray diffractometry technique, resulting in the absence of several sharp lines.

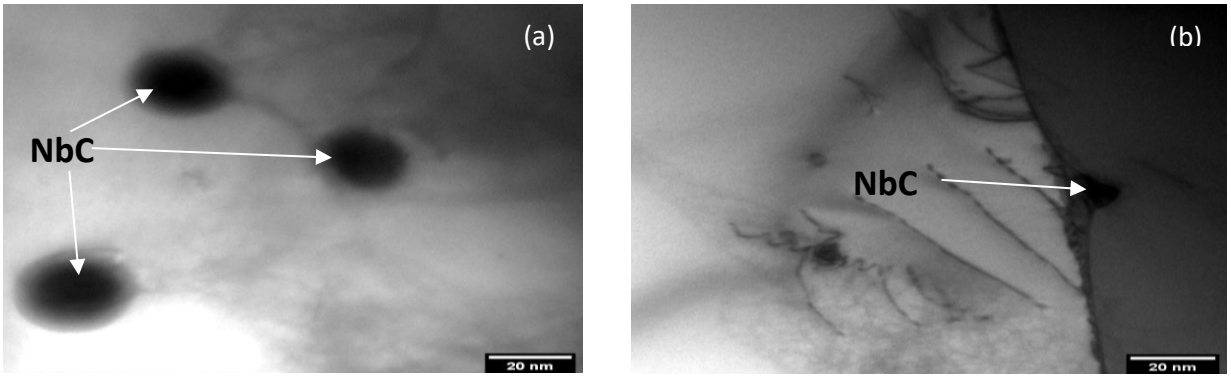


Figure 3. Transmission electron microscopy (TEM) images of the AISI 348 steel in the as-received condition: (a) NbC precipitates in the matrix; (b) NbC precipitate at a grain boundary.

Following the solution heat treatment and Berzelius extraction, the Rietveld refinement yielded statistical indices of excellent numerical reliability. The goodness-of-fit indicator (GoF) was 1.1117, mathematically validating the proposed structural model. The weighted profile residual factor ($R_{\omega p}$ = 8.26%) and the expected residual factor (R_{exp} = 7.43%) demonstrated high proximity (a difference of less than 1%), indicating that the background noise was perfectly controlled and that the photon count (exceeding 800 counts at the main peak) was fully sufficient for the refinement (2). The phases quantified by the refinement software are described as follows.

Titanium and niobium carbide (Nb,Ti)C with a mass percentage (%) in the residue of 55.00%: the majority phase of the powder, with a lattice parameter (a = 0.44377 nm); **titanium nitride (TiN)** with a mass percentage (%) in the residue of 28.40%: presented a lattice parameter (a = 0.42841 nm); **titanium and niobium carbonitride (Nb,Ti)NC** with a mass percentage (%) in the residue of 8.50%: presented a lattice parameter (a = 0.4414 nm) and **delta ferrite** with a mass percentage (%) in the residue of 8.10%: presented a lattice parameter (a = 0.28741 nm).

The lattice parameter values obtained for the complex Nb and Ti precipitates lie within an intermediate range between pure TiC (approximately (a = 0.432 nm) and pure NbC (approximately (a = 0.447 nm) reported in the literature (6). This confirms the formation of a mixed substitutional solid solution of the (Ti, Nb) (C,N) type. The absence of asymmetry in the gray difference line at the bottom of the spectrum proves that the peak profile is narrow, indicating grown crystalline grains free of micro strains from internal stress (4). The reduced thickness of the AISI 348 steel sheet (1.2 mm) contributes to the reduction of thermal gradients and shortens atomic diffusion distances during the solution heat treatment at 1100 °C for 1 hour. This enabled accelerated phase transformation kinetics, where nearly all of the original delta ferrite was dissolved and consumed to stabilize the austenitic matrix (5). The drastic quenching in water at room temperature acted by metastable freezing the small remaining delta

ferrite islands, preventing deleterious secondary transformations (such as sigma phase formation). Due to the selective Berzelius etching—which eliminated more than 98% of the less resistant austenitic matrix—a meticulous profile analysis of the extracted residue revealed the persistence of a low-intensity integrated reflection fixed at $2\theta = 44.7°$, exactly coinciding with the position of the strong intensity peak recorded in the as-received bulk condition. This crystallographic feature is attributed to the (110) plane of the residual delta ferrite (BCC structure). The presence of this minor peak in the isolated residue evidences a local chemical passivation phenomenon during the chemical dissolution process (Berzelius). Due to the strong enrichment in ferrite-stabilizing elements (such as Cr and Nb) intrinsic to these residual ferritic islands in the AISI 348 steel, small fragments of this phase exhibited a corrosion resistance superior to that of the austenitic matrix in the Berzelius solution. Consequently, these insoluble fractions were mechanically retained on the PTFE filter membrane, exerting a residual X-ray diffraction contribution that indicates the coexistence of stabilized delta ferrite after the solution heat treatment at 1100 °C for 1 hour.

4. Conclusion

Delta ferrite, which appeared as the residue majority phase in the as-received condition due to work hardening and industrial segregation, was drastically reduced to microstructural traces in the solution-treated condition.

Quenching in water at room temperature proved to be mandatory for retaining the residual delta ferrite in a metastable state, preventing the precipitation of brittle intermetallic phases.

The **isolation method via Berzelius reagent** associated with Rietveld refinement proved to be a robust scientific route, providing excellent statistical quality ($\chi^2 = 1.1117$) in the identification and quantification of complex Nb and Ti carbonitride solid solutions in the solution-treated condition of AISI 348 steel.

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