

ROLE OF HEAT TREATMENT IN MICROSTRUCTURAL MODIFICATION AND MECHANICAL RESPONSE OF ASTM A694 F65 STEEL

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Abstract

ASTM A694 F65 is a low-alloy steel valued for its high yield strength, good weldability, and excellent toughness at low temperatures. Owing to its balanced strength and impact resistance, it is extensively employed in the production of flanges and fittings for low-temperature, high-pressure service, where tensile strength and corrosion resistance are not the primary design requirements. This study examines the effect of heat treatment parameters, specifically the type of heat treatment and cooling conditions, on the microstructural and mechanical properties of an industrial hot-forged ASTM A694 F65 steel. Microstructural evolution was characterized using X-Ray Diffraction (XRD) and Scanning Electron Microscopy in Secondary Electron mode (SEM-SE), while mechanical behaviour was evaluated through microhardness measurements and tensile testing. Experimental results regarding phase composition, morphology and distribution, lattice strain, yield strength, ultimate tensile strength, elongation at fracture, and microhardness were systematically analysed in relation to the applied heat treatment and cooling regime. Based on the results obtained, double normalizing is recommended for hot-forged A694 F65 steel, with sand cooling for thin-walled components and air cooling for thick sections, to prevent excessively low cooling rates and the formation of undesirable banded microstructures.

Keywords: Heat treatment, microstructural evolution, mechanical properties, cooling conditions, steel

1. INTRODUCTION

ASTM A694 F65 is a high-strength low-alloy (HSLA) steel developed to withstand high-pressure environments. Due to its combination of mechanical strength, toughness, and good weldability, it is used for components and equipment operating in demanding and strategic industrial sectors, including oil and gas, petrochemical and power generation industries, as well as naval and submarine applications. Typical components manufactured from A694 F65 include pressure-containing equipment and pipeline components [1]. Following rolling or forging, the semi-finished products are subjected to normalizing, followed by quenching and tempering. Normalizing treatment applied to low-carbon steels is primarily intended to refine and homogenize the microstructure, improve ductility and toughness, and reduce residual stresses [2-4]. Although a single normalizing treatment should generally be sufficient to refine the grain size and restore the mechanical properties to a level that meets the tensile requirements of the lower-strength grades, particularly for thinner sections, this treatment may be inadequate for higher-strength grades, including F65 [2,3]. This is especially relevant for components with large section thicknesses, such as large-diameter valves. After a single normalizing treatment, both strength and toughness may remain below the maximum levels that can be achieved, making this treatment insufficient in some cases. Research and industrial experience showed that, when required, a double-normalizing heat treatment is generally sufficient to restore the mechanical properties of thick-section ASTM A694 F65 forgings [5]. It should be recognized that the presence of vanadium nitride or

niobium carbonitride precipitates restricts grain growth during normalizing. However, prolonged heating at forging temperature promotes the coarsening of these nitrides or carbonitride precipitates, and their distribution may change to the extent that they are no longer able to effectively restrict austenite grain growth. As a result, the ferrite grain size formed during cooling may become larger than the optimum grain size [6,7]. Consequently, after a subsequent normalizing treatment, both strength and toughness may remain below the maximum levels that can be achieved. The mechanical properties can, however, be restored by a solution treatment performed at a sufficiently high temperature to dissolve the vanadium nitride or niobium carbonitride precipitates. This requires a temperature of at least 1050 °C for vanadium nitride and 1200 °C for niobium carbonitride. The resulting austenite grains will nevertheless be relatively coarse, making a subsequent normalizing treatment necessary. Although research and industrial experience showed that such a double heat treatment [5-8], when required, is sufficient to restore the mechanical properties of thick-section ASTM A694 F65 forgings, the standard does not adequately emphasize, firstly, the need to select an appropriate micro alloyed steel and, secondly, the possibility that a high-temperature solution treatment followed by normalizing may be required to meet the mechanical properties specified for the grade.

2. MATERIALS AND METHODS

ASTM A694 F65 steel is characterized by high mechanical strength, with a minimum tensile strength of 530 MPa and a minimum yield strength of 450 MPa, together with a minimum elongation of 20 %. Its specific chemical composition (in wt%), including C 0.10 - 0.14, Mn 1.10 - 1.50, P/S ≤ 0.025 and Si 0.15 - 0.30, contributes to its suitability for high-pressure applications. According to the ASTM A694/A694M-16 standard, the mechanical properties specified for low-carbon A694 F65 steel in the normalized and tempered condition include a tensile strength (σ_{UTS}) in the range of 550 - 660 MPa, a minimum yield strength ($\sigma_{0.2}$) of 450 MPa, an elongation to fracture (ϵ_f) of at least 20 %, and a hardness of 152 - 235 HB [1]. Based on the theoretical considerations [2-8] and the specific requirements associated with the components manufactured from the investigated alloy, a heat treatment cycle was designed and tested (**Figure 1**). The treatment consisted of one or two normalizing steps, with heating to a temperature range of 915 – 925 °C, corresponding to $T = A_{c3} + (30 - 50 \text{ °C})$, followed by a holding time of 14 min., calculated according to $t = 1 \text{ h/in.}$ of section thickness. After each normalizing treatment, the specimens were slowly cooled under three different conditions: air cooling (AC), sand cooling (SC), and furnace cooling (FC). A694 F65 steel was investigated in the as-received hot-forged condition (AR), without subsequent heat treatment, and after each normalizing treatment variants. The material was characterized from both microstructural and mechanical perspectives. Microstructural characterization was performed using XRD and SEM-SE, while the mechanical behaviour was evaluated by tensile testing and microhardness measurements.

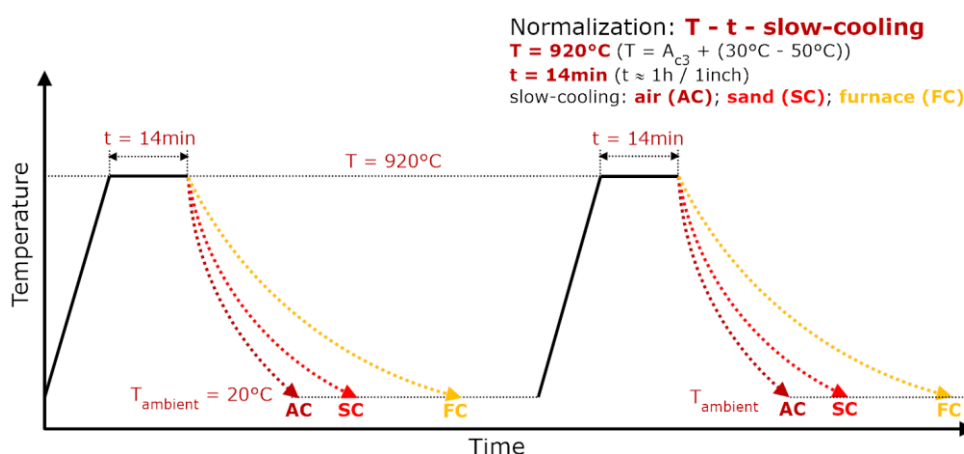


Figure 1 Schematic representation of the normalizing heat treatment applied to ASTM A694 F65 steel

3. RESULTS AND DISCUSSION

In normalizing heat treatment, the steel is heated above the temperature at which ferrite begins to form from austenite, usually by 30-50 °C, the steel being subsequently cooled in still air which results in transformation within the upper range of subcritical temperatures, leading to the formation of a finer pearlitic structure than that obtained after annealing [2,3]. The microstructural characteristics of ASTM A694 F65 steel were investigated by X-Ray Diffraction (XRD) analysis (**Figure 2.1**) and Scanning Electron Microscopy in Secondary Electron mode (SEM-SE) (**Figure 2.2**). XRD analysis of the investigated alloy revealed a phase composition consisting of approximately 99 % ferrite and 1% cementite in all analysed conditions, both in the initial and normalized states. SEM-SE examination of the alloy in the initial condition (hot-forged, without subsequent heat treatment) revealed a microstructure consisting of ferrite and pearlite, with the two constituents present in approximately equal proportions. The average grain size was approximately 25 µm. For the single- or double-normalized steel followed by air cooling, the microstructure also consisted of ferrite and pearlite, but with a higher pearlite fraction than ferrite. In the case of single- or double-normalized ASTM A694 F65 steel followed by sand cooling, the microstructure consisted of the same two constituents, although the ferrite fraction was higher than that of pearlite. For the single- or double-normalized steel followed by furnace cooling, the microstructure consisted of ferrite and pearlite, with a relatively high ferrite fraction and a pronounced banded appearance, characterized by alternating ferrite-rich and pearlite-rich bands. This morphology is promoted by the lower cooling rate associated with furnace cooling and is considered undesirable, as it can increase the susceptibility of the material to cracking and crack initiation [9-12]. A decrease in the cooling rate (AC > SC > FC) promotes the development of a banded microstructure.

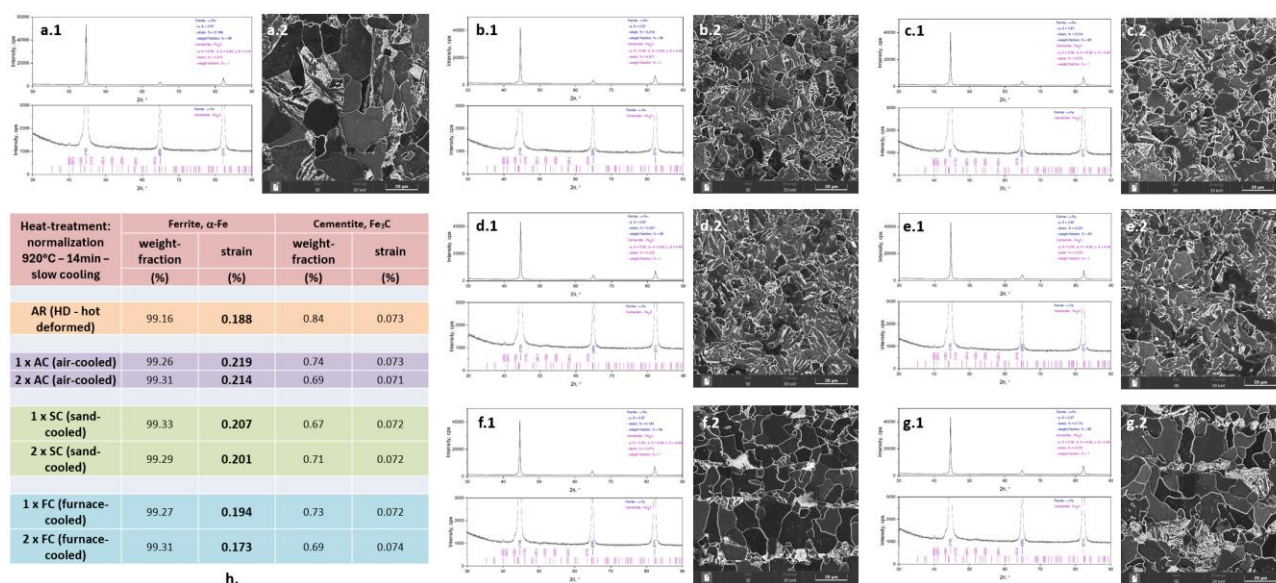


Figure 2 Microstructural evolution of ASTM A694 F65 steel: a. AR (HD hot-deformed); b. 1 x AC (air-cooled); c. 2 x AC (air-cooled); d. 1 x SC (sand-cooled); e. 2 x SC (sand-cooled); f. 1 x FC (furnace-cooled); g. 2 x FC (furnace-cooled); h. phase composition and lattice strain; 1. XRD analysis; 2. SEM-SE analysis

The mechanical behaviour of ASTM A694 F65 steel was evaluated by analysing the stress-strain curves obtained from tensile testing (**Figure 3**), together with microhardness measurements (**Figure 4**). The results indicate that the recommended post-deformation heat treatment for hot-forged A694 F65 steel is a double-normalizing treatment followed by sand cooling for components with relatively thin walls, and air cooling for components with greater wall thicknesses. For thick-section components, sand cooling, in addition to being difficult to implement in practice, may result in an excessively low cooling rate, particularly in the central regions of the components. This may promote the formation of an undesirable banded microstructure [9].

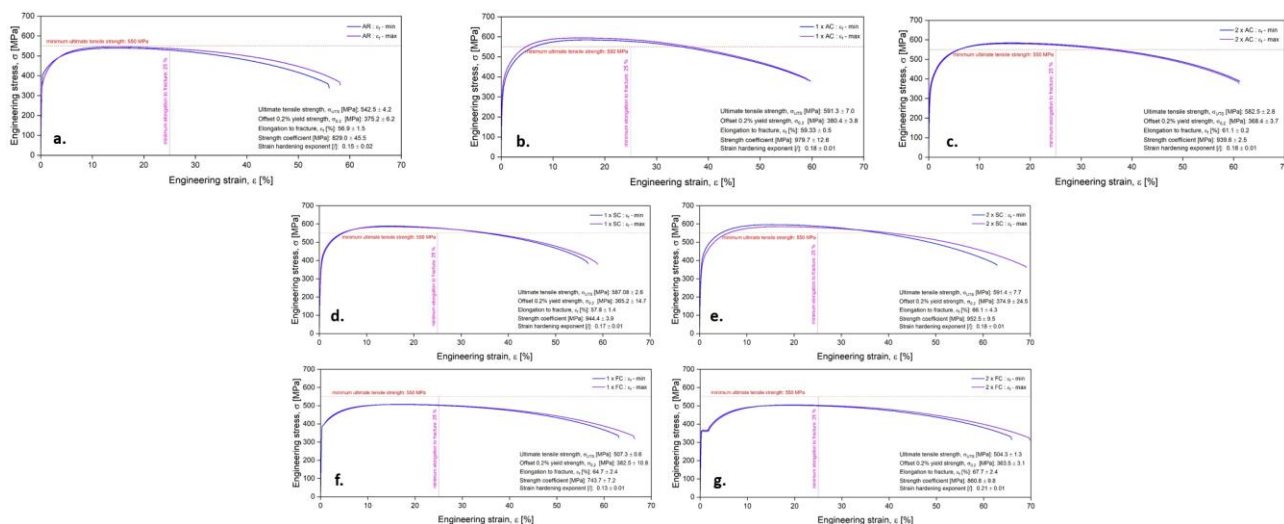


Figure 3 Tensile testing of ASTM A694 F65 steel: a. AR (HD hot-deformed); b. 1 x AC (air-cooled); c. 2 x AC (air-cooled); d. 1 x SC (sand-cooled); e. 2 x SC (sand-cooled); f. 1 x FC (furnace-cooled); g. 2 x FC (furnace-cooled)

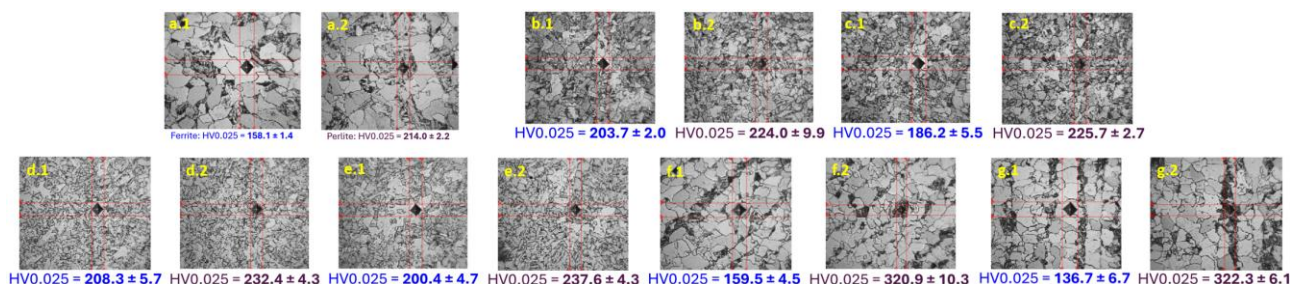


Figure 4 Microhardness evolution of ASTM A694 F65 steel: a. AR (HD hot-deformed); b. 1 x AC (air-cooled); c. 2 x AC (air-cooled); d. 1 x SC (sand-cooled); e. 2 x SC (sand-cooled); f. 1 x FC (furnace-cooled); g. 2 x FC (furnace-cooled); 1. Ferrite; 2. Pearlite

Figure 5 provides a comparative overview of the evolution of mechanical and structural characteristics of ASTM A694 F65 steel following the applied normalizing treatments. The measured values of ultimate tensile strength (σ_{UTS}), yield strength ($\sigma_{0.2}$), elongation to fracture (ϵ_f), ferrite and pearlite microhardness (HV0.025), and lattice strain in ferrite are presented for all investigated structural conditions. These include the initial hot-forged as-received condition (AR), as well as the normalized and double-normalized conditions followed by air cooling (1 x AC, 2 x AC), sand cooling (1 x SC, 2 x SC), or furnace cooling (1 x FC, 2 x FC). The dispersion of measured data is also indicated. The data presented in **Figure 5** shows that, in the initial AR condition, the ultimate tensile strength σ_{UTS} does not meet the product specification requirement of a minimum of 550 MPa, highlighting the need for post-deformation heat treatment. Following the normalizing treatments, σ_{UTS} values satisfying the specified requirement are obtained for both air cooling (AC) and sand cooling (SC), with comparable values measured under the two cooling conditions. In contrast, furnace cooling (FC), which involves a considerably lower cooling rate, results in a significant decrease in σ_{UTS} , with values falling below the minimum level specified for the product. This behaviour can be correlated with the development of a banded microstructure, characterized by alternating ferrite-rich and pearlite-rich bands, which has a detrimental effect on mechanical behaviour and may increase the susceptibility to crack initiation and crack propagation [9-12]. However, the applied normalizing heat treatments are not showing a substantial effect on the yield strength $\sigma_{0.2}$ for the investigated material. The recorded $\sigma_{0.2}$ values are relatively similar for all structural considered states. Measurement uncertainty is also taken into account.

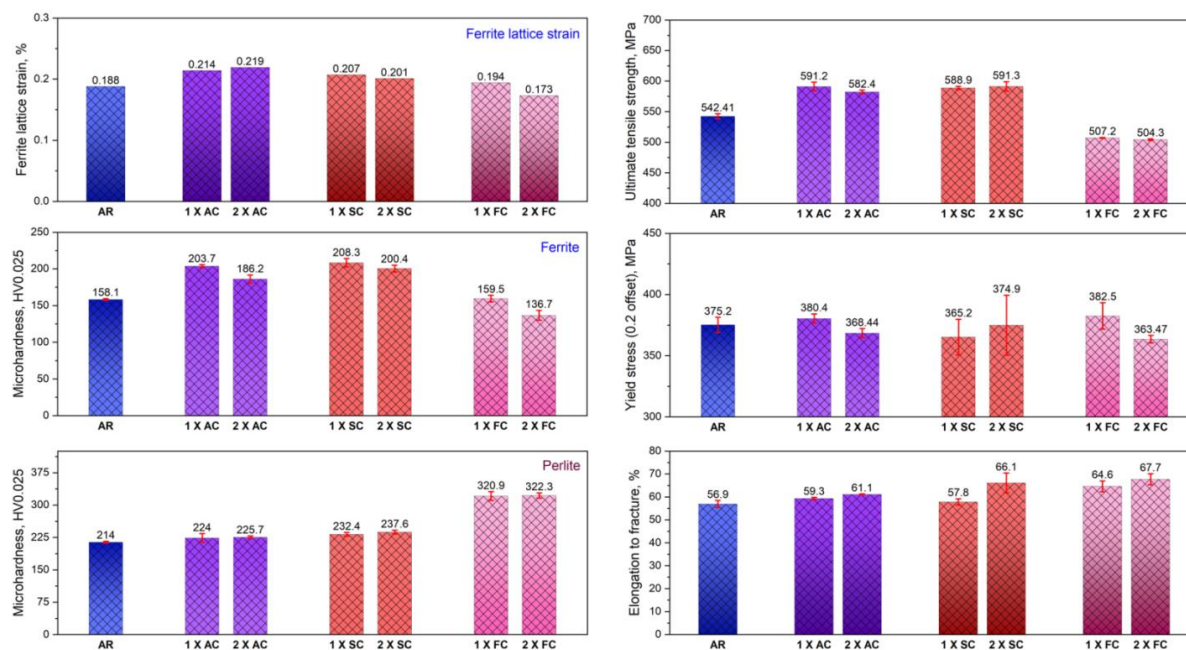


Figure 5 Mechanical and structural characteristics evolution of ASTM A694 F65 steel after normalizing heat treatments

A similar trend can also be observed for the elongation to fracture ϵ_f , although a slight increase in ϵ_f is recorded after the normalizing treatments. This increase becomes more pronounced as the cooling rate decreases. It should also be noted that elongation to fracture ϵ_f meets product specification requirement of a minimum of 25 % in all investigated structural conditions. Regarding ferrite microhardness, a similar trend to that observed for ultimate tensile strength σ_{UTS} can be identified. An increase in ferrite microhardness is observed after normalizing followed by air cooling and sand cooling, with slightly higher values obtained for sand cooling (SC). In contrast, furnace cooling (FC), characterized by a lower cooling rate, results in lower ferrite microhardness values, which are comparable to those measured in the initial AR condition. This variation in ferrite microhardness is consistent with the changes in ferrite lattice strain. On the other hand, normalizing followed by air cooling (AC) or sand cooling (SC) has only a minor effect on pearlite microhardness. The measured values are slightly higher than those recorded in the initial AR condition, while remaining comparable to the ferrite microhardness values measured for the corresponding structural conditions. This relatively balanced hardness between the two structural constituents has a favourable effect on mechanical behaviour for the investigated material. For normalizing followed by slow furnace cooling (FC), a considerable increase in pearlite microhardness is observed. Combined with the lower ferrite microhardness values, this pronounced difference between the hardness of the two phases has a detrimental effect on mechanical behaviour for the investigated steel. The large difference in microhardness between ferrite and pearlite, together with their banded distribution within the microstructure, may promote crack initiation and propagation [9-12], as discussed previously.

4. CONCLUSIONS

The results of present study show that, in the initial hot-forged condition, the ultimate tensile strength of ASTM A694 F65 steel does not meet the minimum requirements specified for the product, highlighting the need for a post-deformation heat treatment. Accordingly, a series of normalizing and double-normalizing treatments followed by air, sand, and furnace cooling were investigated. The microstructure of A694 F65 steel consisted mainly of ferrite and pearlite, with the average grain size and ferrite-to-pearlite ratio being affected by the applied heat treatment and cooling conditions. The most favourable microstructural features were obtained

after normalizing followed by air or sand cooling, whereas the decrease in cooling rate, particularly under slow furnace cooling, promoted the development of an undesirable banded microstructure consisting of ferrite-rich and pearlite-rich regions. This morphology was associated with a deterioration in mechanical behaviour and may increase the susceptibility to crack initiation and propagation. The best overall mechanical properties were obtained after normalizing followed by sand cooling, while the application of a second normalizing step resulted in a further improvement in both mechanical properties and microstructural characteristics for the ASTM A694 F65 investigated steel. Based on the combined microstructural and mechanical results, a double-normalizing treatment followed by sand cooling is recommended for hot-forged components with relatively thin wall sections, whereas double normalizing followed by air cooling is recommended for components with greater wall thickness. For thick-section components, sand cooling is not only difficult to implement in practice but may also result in an excessively low cooling rate, particularly in the central regions of the components, thereby promoting the formation of undesirable banded microstructures.

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