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EFFECT OF HEAT TREATMENT ON THE HARDNESS, MICROSTRUCTURE
AND
WEAR RESISTANCE OF CAST FE-MN-AL-NI-C LIGHTWEIGHT STEEL

THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
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Approval of the Graduate School of Natural and Applied Sciences, Atılım University.

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ABSTRACT

EFFECT OF HEAT TREATMENT ON THE HARDNESS, MICROSTRUCTURE AND WEAR RESISTANCE OF CAST FE-MN-AL-NI-C LIGHTWEIGHT STEEL

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There is a growing interest in steels with low density. This thesis study investigated the effects of solutionizing and aging heat treatments on the microstructure and mechanical properties of cast Fe-Mn-Al-Ni-C lightweight steel with a density of 6.83 g/cm³. Solutionizing treatments were applied in the temperature range of 850-1150°C for 2, 4, and 16 hours. Subsequently, aging treatments were carried out at 550°C and 700°C for 1, 4, 8, and 16 hours. The effects of the heat treatments were evaluated by optical and electron microscopy, macro and nano hardness measurements, wear tests, EBSD, and XRD analysis. The as-cast hardness of 477 HB decreased to 258 HB after 2 hours of solution treatment at 1150°C, and an increase of 46%-88% was observed with subsequent aging. XRD analyses revealed the presence of austenite, B2, and κ -carbide phases in the as-cast samples while heat treatment caused significant differences both in the relative amounts and morphologies of these phases. Based on the results, solution treatment at 1150°C for 2 hours followed by aging at 700°C for 8 hours was determined to be the most suitable heat treatment procedure in terms of microstructural homogeneity and wear resistance.

Keywords: Fe-Mn-Al-Ni-C, Lightweight Steel, Solutionizing, Precipitation, Aging, B2, Wear, Low density

ÖZ

ISIL İŞLEMİN DÖKÜM FE-MN-AL-NI-C HAFİF ÇELİĞİNİN SERTLİK, İÇYAPI VE AŞINMA DİRENCİNE ETKİSİ

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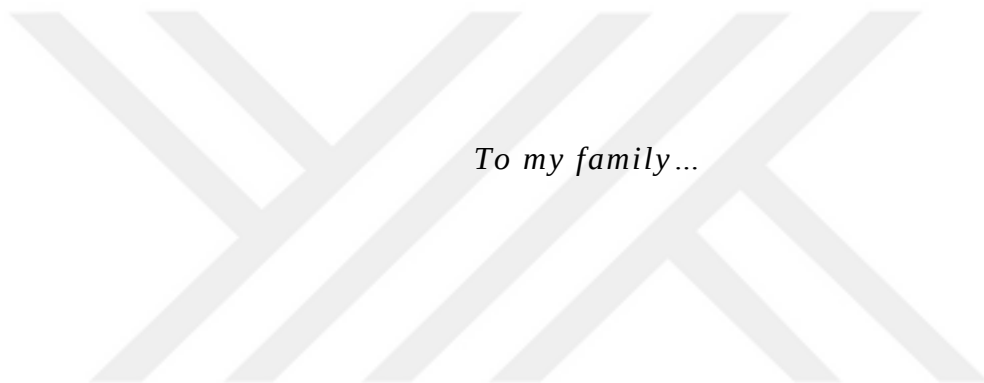
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Düşük yoğunluklu çeliklere olan ilgi giderek artmaktadır. Bu tez çalışmasında döküm Fe-Mn-Al-Ni-C hafif çeliğinde çözündürme ve yaşlandırma ısı işlemlerinin içyapı ve mekanik özellikler üzerindeki etkileri araştırılmıştır. Çözündürme işlemleri 850-1150°C sıcaklık aralığında, 2, 4 ve 16 saatlik sürelerle uygulanmıştır. Takiben, 550°C ve 700°C'de 1, 4, 8 ve 16 saatlik yaşlandırma işlemleri gerçekleştirilmiştir. Isıl işlemlerin etkileri; optik ve elektron mikroskopisi, makro ve nano sertlik ölçümleri, aşınma testleri, EBSD ve XRD analizleri ile değerlendirilmiştir. Döküldüğü haliyle 477 HB olan sertlik, 1150°C'de 2 saat çözeltiye alma sonrası 258 HB'ye düşmüş, yaşlandırma ile %46-%88 oranında artış gözlenmiştir. Faz analizi sonucunda, as-cast numunelerde östenit, B2 ve κ-karbür fazları tespit edilirken, ısıl işlem sonrasında östenit fazı ve B2 fazının farklı morfolojilerdeki çökeltilerinin oluştuğu görülmüştür. Elde edilen sonuçlar doğrultusunda, 1150°C'de 2 saat çözündürme ve ardından 700°C'de 8 saat yaşlandırma işleminin, içyapı homojenliği ve aşınma direnci açısından en uygun ısıl işlem parametreleri olduğu belirlenmiştir.

Anahtar Kelimeler: Fe-Mn-Al-Ni-C, Hafif Çelik, Çözündürme, Çökelme, Yaşlandırma, B2, Aşınma, Düşük yoğunluk



To my family...

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LIST OF ABBREVIATIONS

AHSS	Advanced High Strength Steel
BCC	Body Centered Cubic
CDRX	Continuous Dynamic Recrystallization
DDRX	Discontinuous Dynamic Recrystallization
DP	Dual Phase
EBSD	Electron Backscatter Diffraction
EDS	Energy Dispersive Spectroscopy
FCC	Face Centered Cubic
HB	Brinell Hardness
HV	Vickers Hardness
SEM	Scanning Electron Microscope
TRIP	Transformation-Induced Plasticity
TWIP	Twinning-Induced Plasticity
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Introduction to Lightweight (Low Density) Steels

The development of high manganese steels began with Robert Hadfield's 1882 publication on the Fe-13Mn-1.2C alloy and gained significant momentum. This momentum further accelerated with Hadfield's 1890 patent for Fe-Mn-Al-C steels, and significant advancements were made, particularly between 1950 and 1970. Around the same period, the concept of “transformation-induced plasticity (TRIP)” was introduced by V. F. Zackay in Fe-Ni-Cr steels, and due to this phenomenon, Fe-Mn-Al-C steels did not gain prominence until the early 1980s. However, in order to achieve similar mechanical properties, these steels gained renewed attention through the use of more economical alloying elements such as manganese instead of nickel and aluminum instead of chromium. As a result, these steels were referred to as “Ni-free,” “NiCr-free,” and even “poor man’s stainless steel” during that period [1].

With the development of material science and the increasing demands of industrial requirements, the need for steels that offer both high strength and ductility as well as steels that offer these properties with lower density values is increasing. In this context, Fe-Mn-Al-C steels emerge as the most important steel group that can fill this gap.

With this relatively new alloy, it is aimed to achieve higher performance in the “Elongation - Tensile Strength” graph, which is an important parameter in material selection (Figure 1.1).

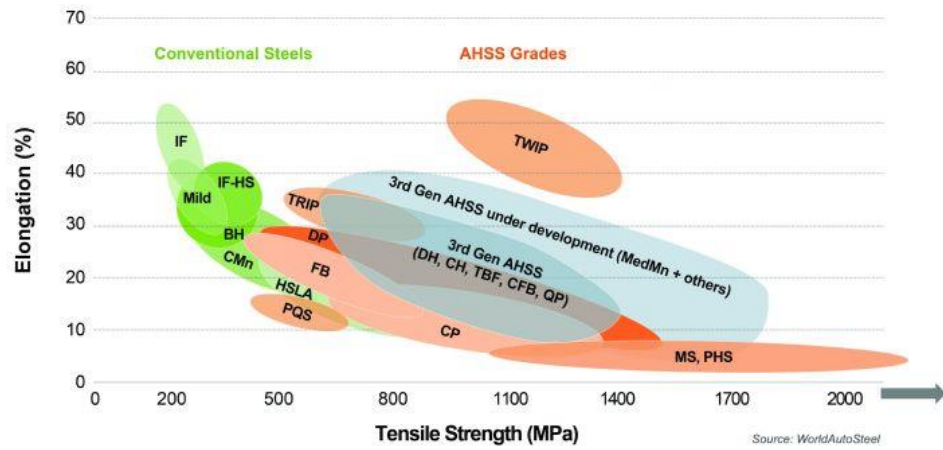


Figure 1.1 Elongation (%) vs Tensile Strength graph of different types of steels [2]

As shown in Figure 1.2, light steels with Fe-Mn-Al-C composition, which have gained research momentum in recent years and are seen as competitors to 3rd Generation AHSS steels with high strength and high ductility, emerge as a solution that aims to reach high specific strengths, i.e. higher strength values at lower densities [1].

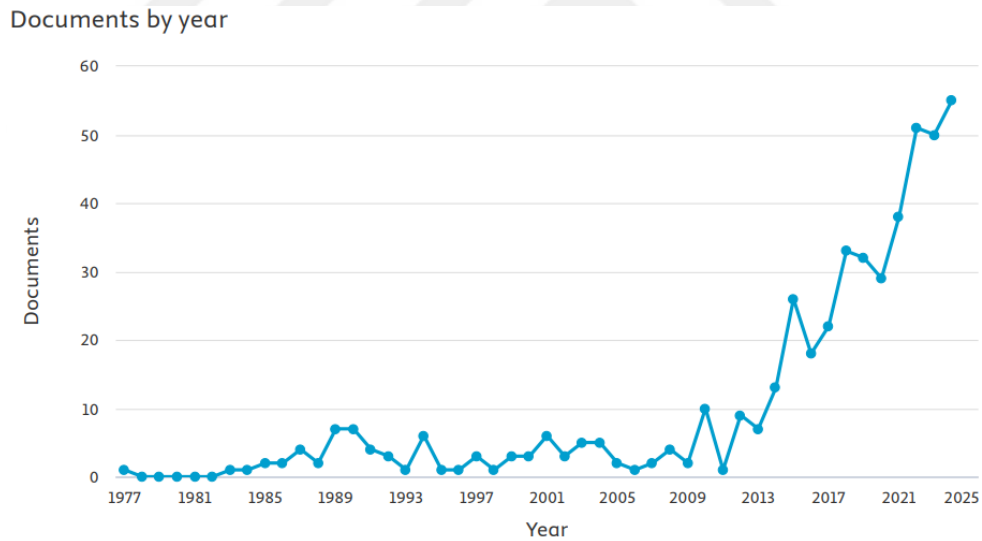


Figure 1.2 Number of documents about Fe-Mn-Al-C Steel 1960 to present in Scopus [3]

This way, especially in fields such as automotive, aerospace, and defense industries where being lightweight is critical, similar or better strength and ductility values can be achieved at lower weights, thereby reducing costs, fuel consumption in vehicles, and minimizing carbon emissions.

1.2 Types of Lightweight Steels

Lightweight steels can be classified into several categories based on their composition and microstructure (Table 1.1).

Table 1.1 Types of lightweight steels [4]

Matrix	Ferritic	Austenitic	Ferrite-based Duplex	Austenite-based Duplex
Possible Phases	δ , B2, D0 ₃ , κ	γ , α , δ , κ , β -Mn	α , δ , B2, D0 ₃ , γ , α' , κ	α , B2, D0 ₃ , γ , κ , β -Mn
Composition Range (wt%)	Mn < 5% Al: 5–9% C < 0.1%	Mn: 15–30% Al: 2–12% C: 0.5–2.0%	Mn: 3–10% Al: 3–9% C < 0.4%	Mn: 10–25% Al: 5–12% C: 0.6–1.0%

- **Ferritic Lightweight Steels:**

These steels are primarily composed of a ferritic matrix with aluminum additions to reduce density. They offer good ductility and corrosion resistance but have lower strength compared to other lightweight steel types [5].

- **Austenitic Lightweight Steels:**

These steels contain high levels of manganese and carbon, stabilizing the austenitic phase. They exhibit excellent ductility, work hardening, and toughness, making them suitable for impact-resistant applications [6].

- **Ferrite-Based Duplex Lightweight Steels:**

These steels have a balanced mixture of ferrite and austenite, providing a combination of strength, ductility, and reduced density. The presence of aluminum helps stabilize ferrite, while manganese and carbon contribute to austenite formation [7].

- **Austenite-Based Duplex Lightweight Steels:**

These steels are predominantly austenitic with a minor ferrite phase. They are strengthened by precipitation of intermetallic phases such as B2 (NiAl) and kappa carbide, leading to high strength and moderate ductility [8]. Possible phases in lightweight steels are described in Chapter 2.

1.3 Motivation and Objectives of the Thesis

Over the past fifty years, high-manganese steels have attracted significant attention due to their exceptional mechanical properties and increased use in engineering and structural applications, leading to extensive research on these alloys. Among these steels, Fe-Mn-Al-Ni-C alloys have held an important place in the category of lightweight steels, which continue to be studied today. While the effects of Mn and Al on these alloys have been widely investigated, the role of Ni and its interactions with other alloying elements, both in the as-cast condition and after heat treatment, have not yet been fully understood.

In this context, this study aimed to investigate the Fe-Mn-Al-Ni-C experimental steel in terms of microstructural evolution and phase transformations, both in the as-cast condition and after solutionizing and aging treatments applied at various temperatures and durations.

Ultimate goal is to create an optimal heat treatment recipe that would provide the Fe-Mn-Al-Ni-C experimental lightweight steel with mechanical and microstructural properties suitable for industrial applications, specifically with the highest possible combination of strength and ductility.

In this regard, Energy Dispersive Spectroscopy (EDS) analyses, microstructural examinations using scanning electron microscopy (SEM) and optical microscope, electron backscatter diffraction (EBSD) analyses, and phase analyses through X-ray diffraction (XRD) were carried out.

Additionally, to determine the mechanical properties, samples subjected to solutionizing and aging at different temperatures and durations were characterized using methods such as hardness testing, and wear testing. Through these analyses, the impact of the microstructure on the mechanical properties was thoroughly examined.

1.4 Outline of the Thesis

This thesis is structured into six main chapters. In Chapter 1, an overview of the concept of lightweight steels is presented, with a particular focus on lightweight steels alloyed with nickel (Fe-Mn-Al-Ni-C), highlighting their structural characteristics, advantages, and potential for engineering applications. Chapter 2 provides a comprehensive review of previous studies on Ni-containing lightweight steels, summarizing the knowledge accumulated in the literature regarding microstructure–property relationships, phase transformations, and mechanical performance. Chapter 3 gives the details of the experimental work employed in this study, including the design of the experimental plan based on insights from the literature and details of the characterization techniques used. This chapter also elaborates on the thermal and thermo-mechanical processing routes applied, along with the associated analytical instruments such as SEM, EBSD, XRD, tensile testing, hardness measurements, and wear testing. Chapter 4 presents experimental results, covering both microstructural characterization and mechanical test results. Chapter 5 is the chapter where the influence of various heat treatment parameters on material behavior is thoroughly analyzed and compared with existing literature to provide a scientific assessment. Finally, the thesis is concluded with Chapter 6 which also contains some suggestions for future work.

CHAPTER 2

BACKGROUND & LITERATURE SURVEY

2.1 Phase Equilibrium in Fe-Mn-Al-Ni-C System

In the Fe-Mn-Al-Ni-C system, various phases can form depending on the chemical composition and applied heat treatment. These phases are ferrite, austenite, B2, D0₃, and κ -carbide. The fractions of these phases and the temperatures at which they stabilize vary depending on the concentration of the respective alloying elements.

2.1.1 Ferrite

In Fe-Mn-Al-Ni-C lightweight steels, the ferrite phase plays a crucial role in determining the mechanical performance of the alloy. Ferrite is present as a body-centered cubic (BCC) matrix, which can host various strengthening precipitates, such as B2 intermetallic compounds and κ -carbides. The morphology, size, and distribution of these precipitates significantly influence the hardness and ductility of the material [9]. For instance, nano-sized NiAl-type B2 particles formed at higher annealing temperatures enhance hardness due to their strong atomic bonding [10]. Conversely, at lower temperatures, Mn-rich κ -carbide needles may form within ferrite, particularly in Ni-free alloys, which also contribute to strength but may reduce ductility [11]. The control of ferrite-phase precipitation through thermal processing is essential for tailoring the mechanical properties of these duplex steels [9].

2.1.2 Austenite

The austenite phase (γ) in Fe-Mn-Al-Ni-C steels, with its face-centered cubic (FCC) crystal structure, is stabilized by elements like Mn, Ni, and C, providing a balance between strength and ductility. Austenite can accommodate various precipitates, such as κ -carbides and B2 particles, depending on the thermal history [5]. At lower annealing temperatures, nano-sized κ -carbides tend to precipitate inside the austenite grains or along grain boundaries, enhancing strength but potentially reducing elongation [11]. In contrast, higher annealing temperatures lead to the dissolution of κ -carbides and formation of nano-dispersed B2 phases, especially in Ni-containing alloys, which support a better strength–ductility combination [10]. The interaction between γ -phase stability and the distribution of nano-precipitates is therefore critical in optimizing the mechanical performance of low-density steels [9, 12].

2.1.3 B2

The B2 phase is an intermetallic compound with an ordered BCC crystal structure. This phase is particularly stable in FeAl and NiAl compositions and plays a significant role in microstructural engineering and mechanical performance in Fe-Mn-Al-Ni-C based lightweight steels. The B2 structure is a CsCl-type lattice, where Fe and Al or Ni and Al atoms occupy specific lattice sites in an ordered structure as shown in Figure 2.1 [9].

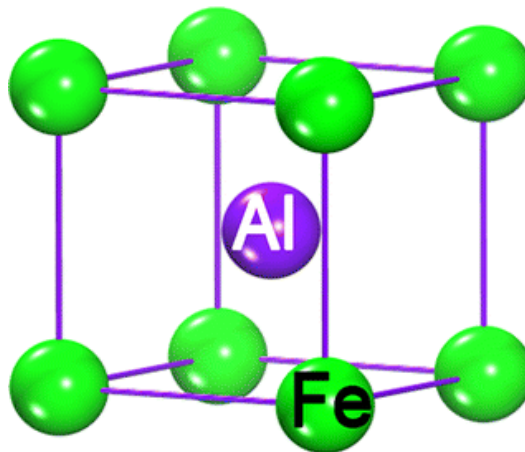


Figure 2.1 FeAl B2 Unit Cell, for NiAl Ni atoms are placed on the corners with the Al atom at the center [13]

Nickel addition plays a critical role in this process. In Ni-containing alloys, B2 precipitates tend to form earlier and exhibit greater stability. Thermodynamic studies supported by Thermo-Calc and CALPHAD simulations have shown that the B2 phase is stable across a wide composition range [12]. As the Ni content increases, NiAl-type B2 precipitates become dominant over the FeAl-type ones, enhancing the precipitation strengthening effect [14].

The B2 phase can appear in various morphologies within the microstructure (Figure 2.2). These include stringer-like, polygonal, and nano-sized disk-shaped precipitates. Stringer-type morphologies, especially when elongated in the rolling direction, tend to reduce ductility, whereas fine and uniformly distributed nano-precipitates offer a good balance between strength and ductility [10,15]. NiAl-based B2 precipitates typically form within the ferrite matrix and hinder dislocation motion, significantly contributing to hardness. These nano-sized particles exhibit localized hardness values in the range of 12–14 GPa, making them one of the key contributors to mechanical strength [9]. FeAl-type B2 precipitates, on the other hand, are more common in alloys with lower Ni content and are typically associated with the ferritic phase. While they also contribute to strengthening, they are generally more ductile than NiAl-based precipitates and exhibit lower brittleness during deformation [16]. The interaction between B2 precipitates and dislocations leads to the formation of planar slip bands and promotes strain hardening, both of which support the achievement of a high strength–ductility synergy [17].

From a mechanical perspective, B2 precipitates can form in both ferrite and austenite phases, exhibiting different effects depending on the matrix. Nano-sized NiAl-type B2 particles in ferrite enhance yield strength and hardness, while in austenite, B2 often co-precipitates with κ -carbides, contributing to strength while maintaining acceptable ductility [5]. However, excessive growth or stringer-type morphology of B2 precipitates may lead to poor processability. Such morphologies can promote microcracks, deformation localization, and low formability. Therefore, controlling the morphology of the B2 phase is essential via heat treatment or hot deformation.

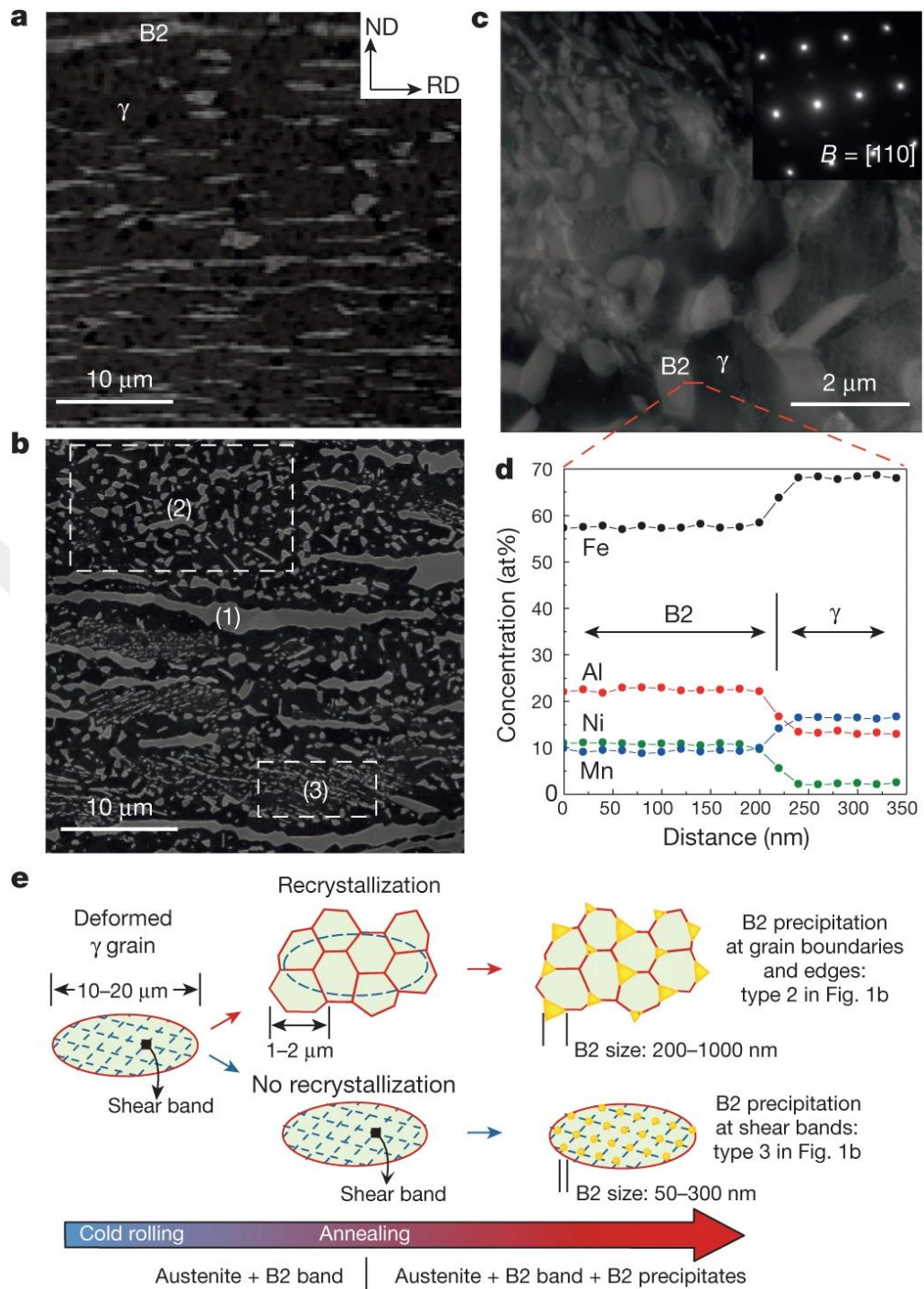


Figure 2.2 B2 Phase precipitation behavior in cold-rolled Fe–10Al–15Mn–5Ni 0.8C steel during thermal annealing, (a) cold-rolled microstructure (γ +B2), (b) annealed microstructure with B2 precipitates and B2 bands, (c) TEM image of annealed microstructure, (d) alloying elements between B2 and γ matrix, (e) schematic representation of formation of B2 precipitates of type 2 and type 3 (as shown in the panel (b) [17]

2.1.4 D0₃

The D0₃ phase is defined as a superlattice structure with a higher degree of atomic ordering compared to the B2 phase. Structurally, it represents an advanced form of the B2 phase, in which atoms are arranged periodically in three dimensions within a regular BCC lattice [10] (Figure 2.3). This phase is typically observed in FeAl or (Fe,Ni)Al-rich regions, and its formation is commonly associated with heat treatments such as fast aging, particularly in Ni-containing Fe-Mn-Al-C steels [10, 14]

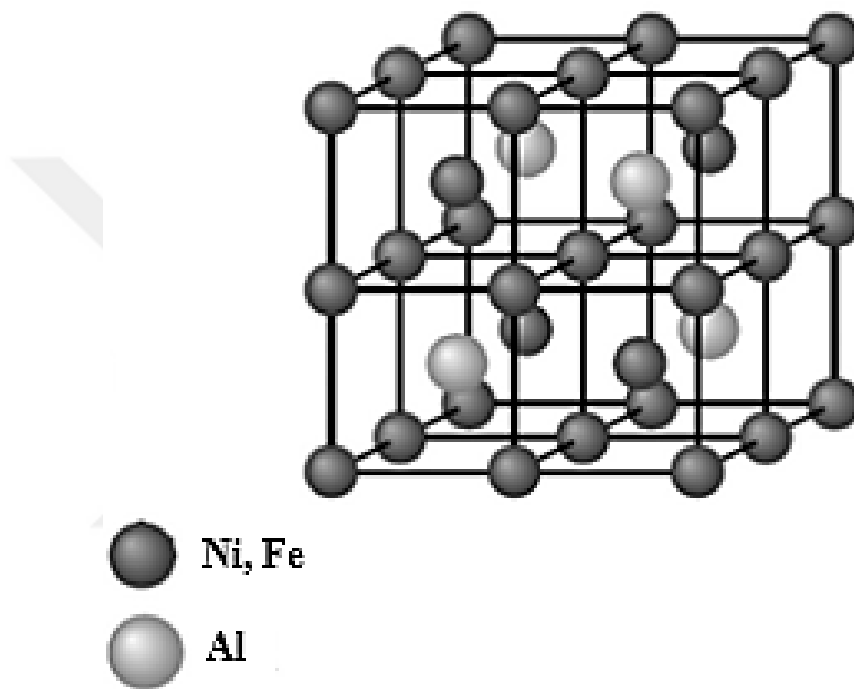


Figure 2.3 D0₃ structure [18]

In these alloys, the D0₃ phase generally forms through a local atomic rearrangement within the pre-existing B2 phase. This transformation occurs during post-solution treatment processes, especially under rapid aging conditions, and manifests as nano-sized D0₃ domains embedded within the B2 matrix [10]. The D0₃ phase plays a critical role in enhancing mechanical properties. By impeding dislocation motion, it contributes to a strengthening mechanism known as order strengthening. Experimental findings suggest that the formation of D0₃ increases the yield strength by approximately 178 MPa [10]. Furthermore, this phase can improve strength without significantly compromising ductility. However, if the volume fraction of D0₃ becomes

too high, it may lead to reduced formability and localized brittleness during deformation [15].

From a microstructural perspective, $D0_3$ precipitation occurs in three typical regions within the B2 matrix: (i) large B2 bands, (ii) B2-austenite interfaces (G-B2), and (iii) isolated B2 precipitates (I-B2). Each region offers distinct crystallographic orientations and kinetic conditions for $D0_3$ nucleation and growth [10]. Thermodynamically, the $D0_3$ phase is more stable at lower temperatures compared to B2 and exhibits slower growth kinetics. Therefore, it typically appears in localized zones within the B2 matrix or near grain boundaries. Controlling the B2-to- $D0_3$ transformation is essential to balancing strength and ductility in Fe-Mn-Al-Ni-C lightweight steels [19].

2.1.5 κ -carbide

The κ -carbide phase is a precipitate phase stabilized by high Al and Mn content in Fe-Mn-Al-C alloy systems, significantly affecting mechanical properties. This phase typically has a $(Fe,Mn)_3AlC$ chemical composition and exhibits an anti-perovskite crystal structure, often crystallizing in a pattern resembling an FCC structure [11] (Figure 2.4). κ -carbides can precipitate in both austenite (γ) and ferrite (α) phases. In ferrite, they often appear with a lamellar morphology, while in austenite they are usually seen as cuboidal and nano-sized precipitates.

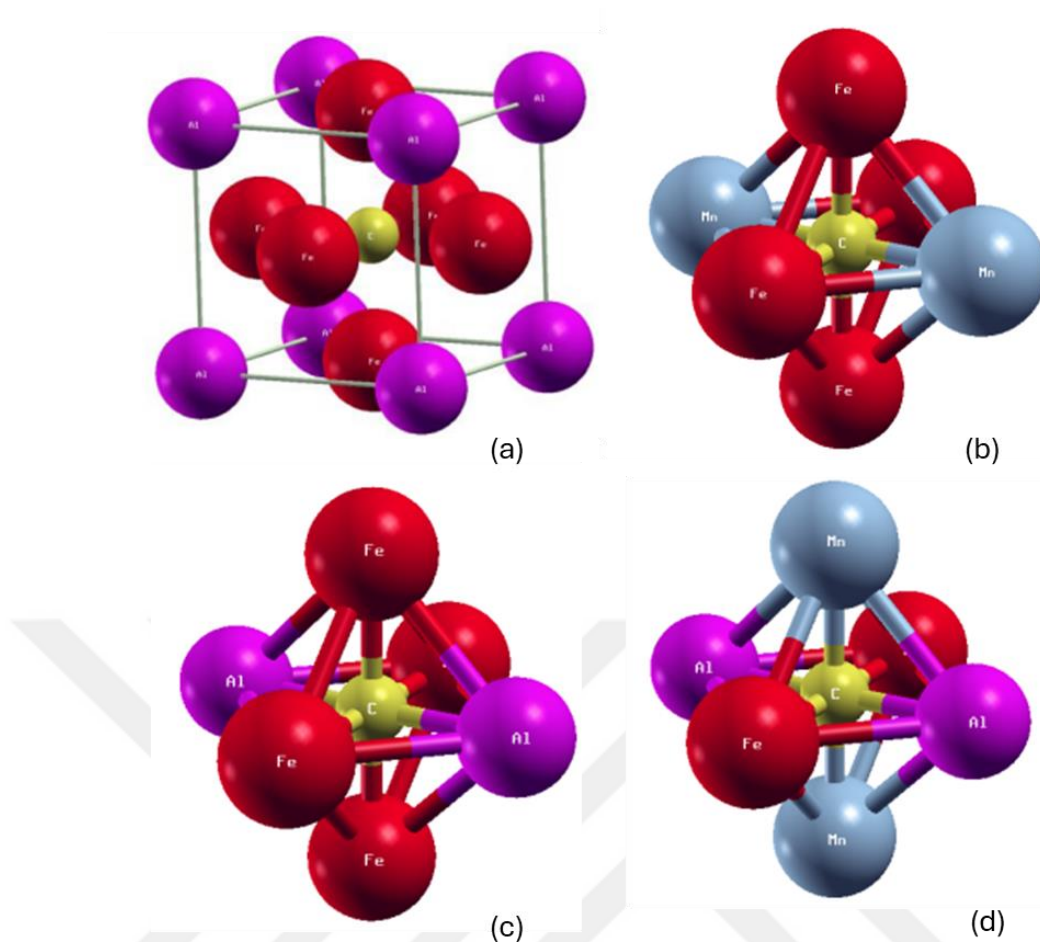


Figure 2.4 (a) κ -carbide unit cell, (b, c, d) possible configurations of octahedral sites [20]

Their formation in austenite is often driven by a spinodal decomposition mechanism, the impact of which on the crystal structure has been confirmed through EBSD analysis [11]. Precipitation of κ -carbides contributes to strain hardening by hindering dislocation movement, particularly during plastic deformation. These precipitates may activate mechanisms such as microband-induced plasticity (MBIP), shear banding, and glide plane softening. However, excessively large or continuous κ -carbide networks can negatively affect ductility [20]. The effect of Ni addition on κ -carbide is noteworthy. Thermodynamic calculations and experimental observations show that increasing Ni content lowers the precipitation temperature and reduces particle size. Furthermore, differences in bonding energies between Ni and Al atoms can lead to reverse partitioning of Al from κ -carbide into the γ -matrix under certain conditions [20]. Structurally, κ -carbides form semi-coherent interfaces with the austenite matrix

through a Kurdjumov–Sachs orientation relationship. Elastic strain fields at these interfaces significantly contribute to strengthening. Especially at aging temperatures around 550°C, nano-sized κ -carbide precipitates are beneficial for achieving a desirable balance of strength and ductility [11]. Thermodynamically, the κ -carbide phase becomes stable below approximately 864°C. Its precipitation kinetics depends on alloy composition, heat treatment time, and the presence of elements such as Ni and Si. While Si accelerates κ -carbide formation, elements like Mo and Cr tend to slow down the process [20].

2.2 Introduction to the Fe-Mn-Al-Ni-C System

Fe-Mn-Al-C alloys are among the most promising lightweight steel systems due to their high specific strength and low density. In this system, the density-reducing effect of Al and the austenite-stabilizing combination of Mn and C enable the production of low-density steels while preserving ductility [11]. Therefore, the base Fe-Mn-Al-C composition already provides a sufficient foundation for lightweight design.

However, the addition of Ni aims not only to maintain lightweight properties but also to enhance microstructural control and mechanical performance. Ni is known to promote the precipitation of B2-type intermetallic phases (e.g., NiAl-B2), which contribute to precipitation strengthening and increasing yield strength [10, 14]. Moreover, the formation of D0₃-type superlattice phases within B2 precipitates leads to a more stable microstructure and further improves the strength–ductility balance [10]. Ni also influences the behavior of κ -carbide phase in solution by altering the partitioning of Al and C. In some studies, Ni has been shown to induce reverse partitioning of Al from κ -carbide into the austenite matrix, thereby increasing matrix hardness [20]. From a microstructural perspective, Ni addition modifies the ferrite/austenite volume ratio, making it easier to control the duplex structure. This contributes not only to strength but also to ductility. Additionally, it leads to a finer and more isotropic morphology of B2 precipitates, reducing the risk of crack formation [15].

In the study conducted by Burja et al., the thermodynamically stable phases of alloys with different Ni compositions in the as-cast condition were investigated through CALPHAD calculations. The compositions of the as-cast samples used in this study are given in Table 2.1.

Table 2.1 Different Ni compositions in the study [21]

Composition (wt.%)	Mn	Al	Ni	C	Si	Cu	Fe
FeMnAlC	15.0	10.0	0.1	0.81	0.20	0.17	Bal.
FeMnAlNiC	14.9	10.2	1.7	0.81	0.21	0.17	Bal.
FeMnAlNiC	14.8	10.0	3.9	0.83	0.19	0.17	Bal.
FeMnAlNiC	14.8	10.1	5.6	0.82	0.20	0.17	Bal.
FeMnAlNiC	14.7	10.1	8.6	0.83	0.19	0.16	Bal.

In the compositions used for CALPHAD calculations, the wt% of C, Si, Cu, Mn, and Al were kept relatively constant, while the Ni content gradually increased [21]. This allowed the effect of Ni in the composition to be observed in Figure 2.5.

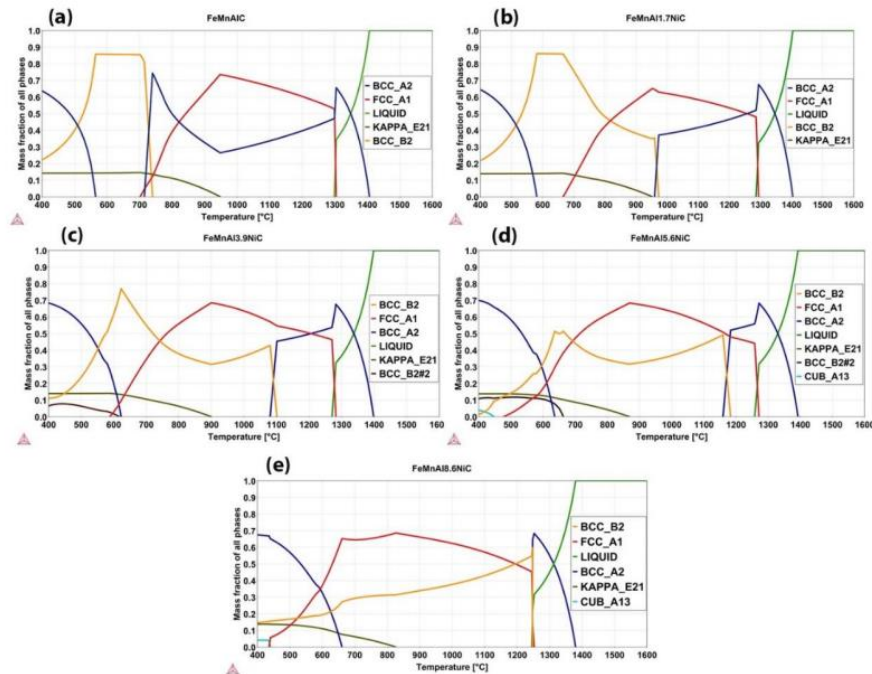


Figure 2.5 CALPHAD calculations of the different alloys studied by Burja et. al, eels studied, (a) FeMnAlC, (b) FeMnAl1.7NiC, (c) FeMnAl3.9NiC, (d) FeMnAl5.6NiC, and (e) FeMnAl8.6NiC [22]

As observed in the study, increasing Ni content affects the stability of the formed phases. Specifically, the δ -ferrite phase, referred to as BCC_A2 in the diagrams (at high temperatures), shows a decrease in stability with increasing Ni content. In contrast, the stability of the γ -austenite phase, referred to as FCC_A1, and the FeAl and NiAl phases, referred to as BCC_B2, increase even at lower temperatures. Similarly, the amount of the κ -carbide phase, referred to as KAPPA_E21, is observed to decrease [21].

As-cast samples of Fe-Mn-Al-C alloys exhibit a two-phase microstructure consisting of α -ferrite and γ -austenite, with the presence of κ -carbides. The α -ferrite phase appears as the lighter region, while the γ -austenite phase is darker in microstructural images. The dendritic morphology of α -ferrite is indicative of a typical as-cast structure [21].

The Ni content in the alloy significantly influences the morphology and distribution of κ -carbides. In the Ni-free Fe-Mn-Al-C sample, the carbides present at the ferrite/austenite grain boundaries are coarse and needle-shaped. The κ -carbides become finer and more rounded with lower Ni additions (FeMnAl1.7NiC and FeMnAl3.9NiC). At moderate Ni levels (FeMnAl5.6NiC), very fine carbides precipitate within the austenite grains. However, at higher Ni concentrations (FeMnAl8.6NiC), the carbide morphology resembles that of the alloys with lower Ni content (FeMnAl1.7NiC and FeMnAl3.9NiC), suggesting a reversion in carbide morphology with increasing Ni content [21].

These observations indicate that Ni plays a crucial role in controlling the size and distribution of κ -carbides in Fe-Mn-Al-C alloys. This variation in carbide morphology is expected to influence the mechanical properties of the alloys, particularly in terms of strength and toughness. Further quantitative analysis, such as carbide size distribution and volume fraction measurements, would be beneficial for a more comprehensive understanding of the microstructural evolution with Ni addition.

In conclusion, while the Fe-Mn-Al-C system alone provides sufficient lightness, the addition of Ni is aimed at achieving multiple improvements including precipitation strengthening, phase stability, microstructural control, and deformation behavior. This makes Ni-alloyed systems suitable not only for lightweight applications but also for high-performance structural use.

2.3 Hot Working and Age Hardening of Fe-Mn-Al-Ni-C Lightweight Steels

2.3.1 Hot Working of Fe-Mn-Al-Ni-C Steels

Hot working processes, involving plastic deformation at elevated temperatures, are fundamental to the control of microstructural evolution and the optimization of mechanical properties in metallic materials. Through dynamic recovery, recrystallization, and phase transformations, hot working allows the tailoring of grain size, texture, and precipitation behavior, thereby directly influencing the performance of the final product. In particular, lightweight steels based on the Fe-Mn-Al-C system have attracted extensive research attention due to their unique combination of low density, high strength, and reasonable ductility [17]. The addition of Ni to this system further enhances phase stability and enables novel strengthening mechanisms via B2-type intermetallic precipitation, making Fe-Mn-Al-Ni-C steels a prime subject for advanced hot working studies.

During hot deformation, the austenitic matrix of Fe-Mn-Al-Ni-C steels exhibits complex interactions between dislocation motion, dynamic recovery, and recrystallization. In compositions free of significant B2 intermetallic phases, hot working is predominantly governed by discontinuous dynamic recrystallization (DDRX), characterized by nucleation and growth of new strain-free grains [16]. However, when a significant amount of B2 phase is present, continuous dynamic recrystallization (CDRX) becomes more prominent, aided by the fragmentation and gradual rotation of subgrains within the hard B2 particles [16, 22].

As an example, in the study conducted by Mishra et al., the dynamic recovery and recrystallization mechanisms during hot working of Fe-Mn-Al-C-based low-density steels were systematically investigated. Two different alloys were produced by vacuum induction melting: a fully austenitic Fe-28Mn-9Al-0.9C alloy and a duplex Fe-15Mn-9Al-0.9C-5Ni alloy containing secondary B2 intermetallic and κ -carbide phases. Both alloys underwent a homogenization heat treatment to eliminate segregation and stabilize the initial microstructure after casting. Following this, hot compression tests were performed at temperatures ranging from 900°C to 1150°C and strain rates between 0.005 s⁻¹ and 5 s⁻¹. After deformation, samples were immediately water-quenched to preserve the deformation-induced microstructure [16].

Microstructural characterization revealed that in the B2-free alloy, deformation promoted discontinuous dynamic recrystallization (DDRX) within the austenite matrix. In contrast, the B2-containing alloy exhibited continuous dynamic recrystallization (CDRX) through subgrain rotation and fragmentation within the B2 particles as can be seen in Figure 2.6.

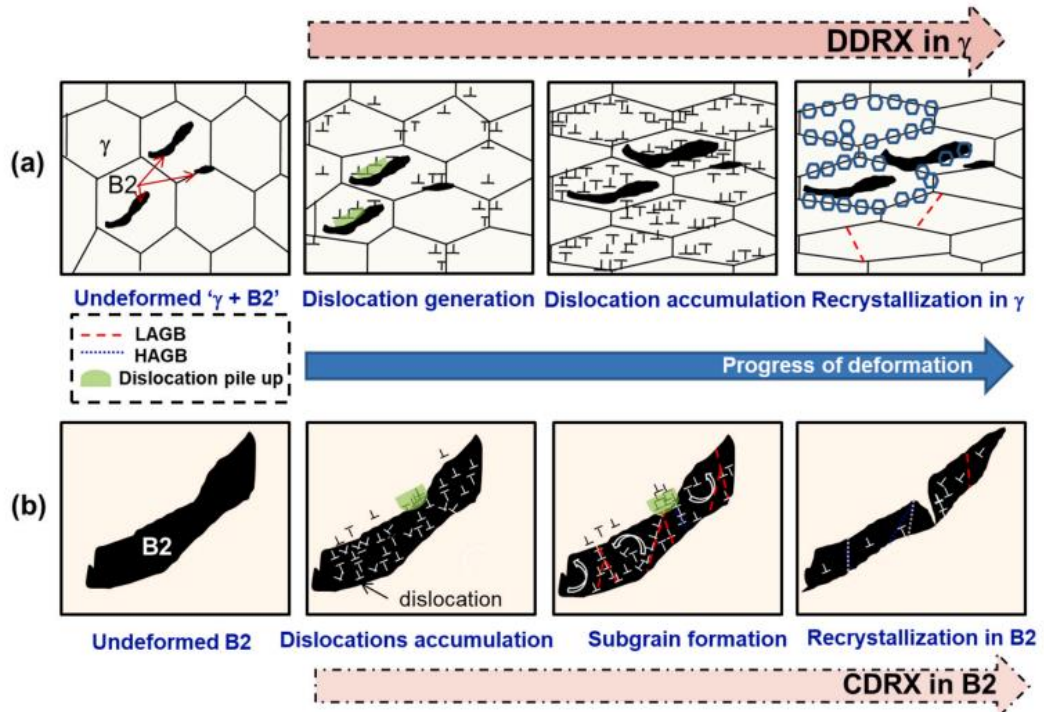


Figure 2.6 Schematic representation of hot deformation on (a) γ matrix, (b) B2 phase [16]

As evidenced by numerous studies in the literature, Fe-Mn-Al-Ni-C lightweight steels—despite variations in alloying element contents—are typically processed through a similar thermomechanical sequence. This general route involves homogenization, followed by hot working, cold working, and finally an aging or annealing treatment. The homogenization step is crucial to dissolve segregation-induced secondary phases such as δ -ferrite and coarse B2-NiAl, promoting a single-phase austenitic matrix suitable for deformation. For instance, solution treatments at 1200°C for 2 hours are commonly applied prior to hot deformation to dissolve as-cast precipitates and stabilize the matrix structure [6]. Similarly, in other works, homogenization is performed at 1100–1250°C to eliminate δ -ferrite and promote uniform grain growth before rolling operations [5].

The hot working stage typically includes hot rolling within the temperature range of 1000–1200°C, inducing dynamic recrystallization and initiating phase transformations. For example, in one study, two hot rolling procedures were performed between 900–1200°C, achieving up to 72% thickness reduction, followed by rapid water quenching to preserve the deformed microstructure and B2 phase morphology [6]. Hot deformation studies also show that careful control of temperature and strain rate is essential to avoid cracking and promote continuous recrystallization in duplex microstructures [16].

The cold working step, often conducted through cold rolling, introduces high dislocation densities and shear bands into the austenitic matrix. These defects act as nucleation sites for subsequent precipitation. In one example, sheets were cold rolled to 1 mm thickness and then annealed at 900°C for 15 minutes, providing a suitable condition for fine B2 and κ -carbide precipitation during fast aging [10].

Finally, the aging treatment, whether through isothermal holding or rapid cooling, is applied to induce controlled precipitation of strengthening phases. Precipitation hardening at 950°C for 10 minutes has been shown to produce nanoscale intragranular B2-NiAl precipitates, significantly enhancing mechanical strength while retaining reasonable ductility [6]. Alternative strategies involve annealing at 900–950°C

followed by fast air cooling to promote $D0_3$ and B2 precipitation without sacrificing ductility too severely [10].

Table 2.2 and Table 2.3 present the mechanical properties of Fe-Mn-Al-C steels alloyed with nickel, compiled from various studies in literature where hot and cold rolling processes were applied. This data provides a comparative basis for evaluating the influence of thermomechanical processing and heat treatment on the mechanical behavior of these alloys.



Table 2.2 Hot working parameters and mechanical properties of Ni added lightweight steels in the literature - Part 1

Compositions (wt.%)	UTS (MPa)	Total EL %	Process										Ref.
			Homogenization (°C)	Time (min)	Hot Rolling (°C)	Solution Treatment (°C)	Time (min)	Cold Rolling	Annealing (°C)	Time (min)	Aging (°C)	Time (min)	
Fe-16Mn-9Al-0.8C-3Ni	1147	43,2	-					+	900	15			[23]
Fe-16Mn-9Al-0.8C-3Ni	866	34,7	as-cast										
Fe-16Mn-9.6Al-0.86C-4.9Ni	1548	20,3	1150	120	1050			+	900	2			[24]
Fe-16Mn-9.6Al-0.86C-4.9Ni	1486	24,7	1150	120	1050			+	900	8			
Fe-16Mn-9.6Al-0.86C-4.9Ni	1350	31,8	1150	120	1050			+	900	15			
Fe-16Mn-9.6Al-0.86C-4.9Ni	1473	25,6	1150	120	1050			+	870	15			
Fe-20.7Mn9.5Al-1.0C-4.7Ni	1507 ± 12	24,3 ± 0,1	1150	120	1050			+	900	2			[25]
Fe-20.7Mn9.5Al-1.0C-4.7Ni	1372 ± 4	35 ± 2,1	1150	120	1050			+	900	15			
Fe-20.7Mn9.5Al-1.0C-4.7Ni (Aged)	1622 ± 12	20,2 ± 1,7	1150	120	1050			+	900	2	550	10	
Fe-20.7Mn9.5Al-1.0C-4.7Ni (Aged)	1485 ± 8	30,2 ± 0,5	1150	120	1050			+	900	15	550	10	

Table 2.3 Hot working parameters and mechanical properties of Ni added lightweight steels in the literature - Part 2

Compositions (wt.%)	UTS (MPa)	Total EL %	Process										Ref.
			Homogenization (°C)	Time (min)	Hot Rolling (°C)	Solution Treatment (°C)	Time (min)	Cold Rolling	Annealing (°C)	Time (min)	Aging (°C)	Time (min)	
Fe-15Mn-10Al-0.8C-5Ni	630	22,4			+	1250	35						[26]
Fe-15Mn-10Al-0.8C-5Ni	980	10			+	1250	35				500		
Fe-15Mn-10Al-0.8C-5Ni	~1050	~8,9			+	1250	35				700		
Fe-15Mn-10Al-0.8C-5Ni	~1300	~9,5			+	1250	35				900		
Fe-15Mn-10Al-0.8C-5Ni	1350	22			+	1250	35				1050		
Fe-19.7Mn-9.12Al-0.80C-4.48Ni	1410	19,1	1200	120	1200			+	900	2			[27]
Fe-19.7Mn-9.12Al-0.80C-4.48Ni	1200	38,6	1200	120	1200			+	900	15			
Fe-15Mn-9Al-0.9C-5Ni	1800 ± 15	7 ± 0.5	1150	150	1050			+					[28]
Fe-15Mn-9Al-0.9C-5Ni	1303 ± 12	36 ± 1	1150	150	1050			+	1000	60			
Fe-30Mn-10Al-0.9C-1.55Mo-1.5Ni	1044	31	1050	120							550	1200	[29]
Fe-30Mn-10Al-0.9C-1.55Mo-1.5Ni	1049	30	1050	120							550	6000	
Fe-30Mn-10Al-0.9C-1.53Mo-3Ni	1146	10	1050	120							550	1200	
Fe-30Mn-10Al-0.9C-1.53Mo-3Ni	Premature Fracture	-	1050	120							550	6000	
Fe-20Mn-8Al-5Ni-0.8C	1174	31				1280	60	+	850	120			[30]

* +: The process done, but there is no information.

According to the study by Rahnama et al., in Fe-Mn-Al-C based duplex mild steels, Ni addition plays an important role in phase evolution, precipitation behavior and mechanical performance during thermomechanical processing. Comparative analysis of two alloys one containing Ni (Fe-15Mn-10Al-0.8C-5Ni, referred to as S1) and another not containing Ni (Fe-15Mn-10Al-0.8C, referred to as S2) reveals significant differences in microstructural development and resulting mechanical behavior [22].

The most notable microstructural divergence between the two compositions occurs in the formation and stability of B2 intermetallics and κ -carbides during annealing. In the Ni-containing alloy (S1), B2-type NiAl intermetallic precipitates form preferentially, driven by strong Ni–Al chemical affinity. These precipitates are thermodynamically more stable and exhibit a disk-like morphology, particularly when annealed at temperatures between 900–1050°C, due to the high affinity between Ni and Al atoms leading to stable NiAl-type B2 formation (Figure 2.7). In contrast, the Ni-free alloy (S2) tends to favor the formation of FeAl-based B2 phases, which are comparatively softer and less stable under identical heat treatment conditions [22].

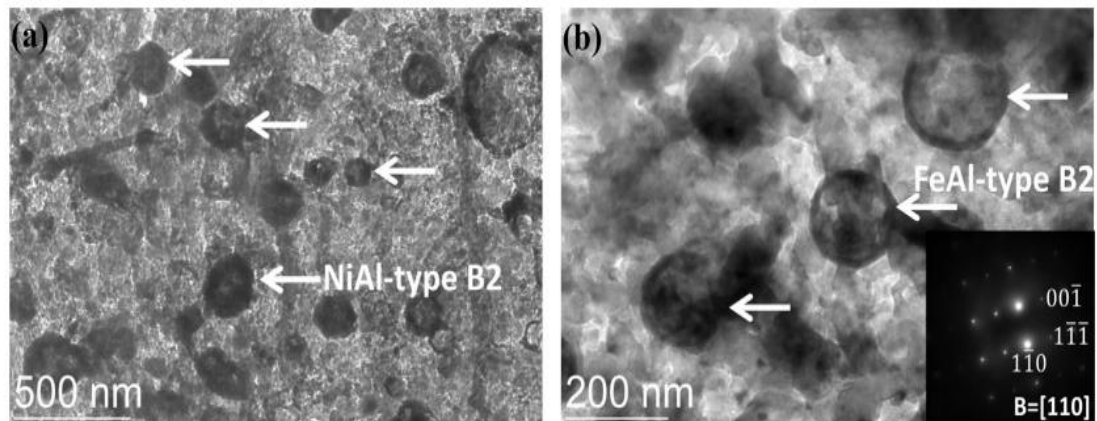


Figure 2.7 TEM bright-field images showing disk-like B2 precipitates formed in the ferrite matrix after heat treatment at 1050 °C; (a) NiAl-type B2 precipitates in Fe-15Mn-10Al-0.8C-5Ni alloy, (b) FeAl-type B2 precipitates in Fe-15Mn-10Al-0.8C alloy [22]

Another critical effect of Ni addition is observed in the suppression of κ -carbide precipitation. In S1, the formation of NiAl-type B2 precipitates reduces the chemical availability of Al for κ -carbide nucleation and results in a lower κ -phase volume fraction, mostly confined to the γ -austenite matrix. In contrast, in S2, where Ni is absent, Al is more readily available for κ -carbide formation. As a result, κ -carbides precipitate more extensively in both γ and α matrices, especially during intermediate annealing treatments at 900 °C [22].

These compositional and phase differences translate into significant changes in mechanical properties. As shown in Figure 2.8 and 2.9, the Ni-containing alloy (S1) exhibits significantly higher tensile strength (UTS $\approx$ 1350 MPa) due to the presence of uniformly distributed, coherent B2 precipitates that act as strong barriers to dislocation motion. However, this comes at a small cost to ductility, with a total elongation of approximately 22%. In contrast, the Ni-free alloy (S2) exhibits a lower UTS ($\approx$ 1040 MPa) but improved elongation ($\sim$ 25%); this is attributed to the presence of κ -carbides, which provide moderate strengthening while preserving plasticity [22].

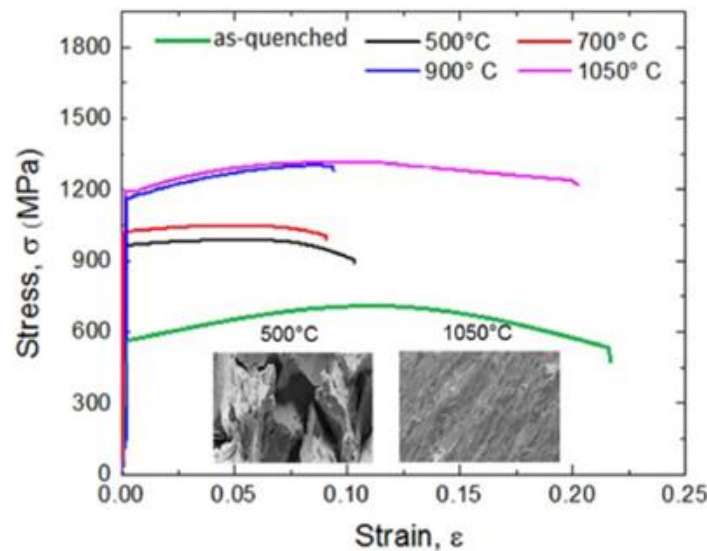


Figure 2.8 Room-temperature tensile properties of Fe-Mn-Al-C-based duplex steels after isothermal annealing at various temperatures, (a) Fe-15Mn-10Al-5Ni-0.8C (S1) annealed at 500°C, 700°C, 900°C, and 1050°C corresponding fracture surfaces at 500 °C and 1050°C are included [22]

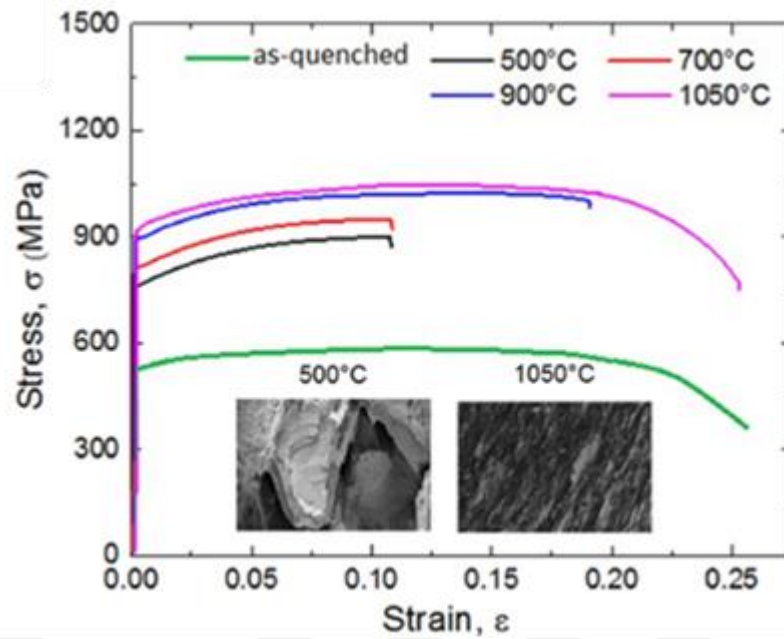


Figure 2.9 Room-temperature tensile properties of Fe-Mn-Al-C-based duplex steels after isothermal annealing at various temperatures (b) Fe-15Mn-10Al-0.8C (S2) annealed at 500°C, 700°C, 900°C, and 1050°C corresponding fracture surfaces at 500°C and 1050°C are included [22]

In summary, the addition of Ni to Fe-Mn-Al-C steels significantly alters the precipitation sequence and phase stability, promoting the formation of thermally stable NiAl-type B2 precipitates while reducing κ -carbide content. This shift in microstructure results in a marked improvement in strength, with a minor reduction in ductility. These findings suggest that controlled Ni addition, combined with optimized annealing, offers an effective route to optimize the balance between strength and formability in low-density duplex steels [22].

2.3.2 Age Hardening of Fe-Mn-Al-Ni-C Steels

Age hardening is a key strengthening mechanism in Fe-Mn-Al-Ni-C-based lightweight steels. This mechanism involves the formation of fine intermetallic phases and carbides during aging treatments following prior thermomechanical processing [22].

In alloys containing nickel, aging promotes the precipitation of NiAl-type B2 intermetallics, which form within the ferrite matrix. These particles are typically coherent and thermodynamically stable, offering significant resistance to dislocation motion and contributing to increased strength [8]. In the absence of nickel, the dominant precipitates are κ -carbides, which can nucleate both within the austenite and ferrite matrices. Although they also contribute to strengthening, their effect is generally less pronounced compared to the NiAl-type B2 phase [22].

The precipitation behavior is highly dependent on the alloy composition and prior deformation history. As shown in the typical process diagram in Figure 2.10, cold working prior to aging increases defect density, which in turn facilitates nucleation of precipitates, leading to a more refined and uniform distribution [10]. Aging treatments can be tailored to control the type, morphology, and distribution of precipitates. The balance between B2 and κ -carbide formation is closely linked to the presence or absence of nickel, which alters the chemical driving forces during phase separation [6].

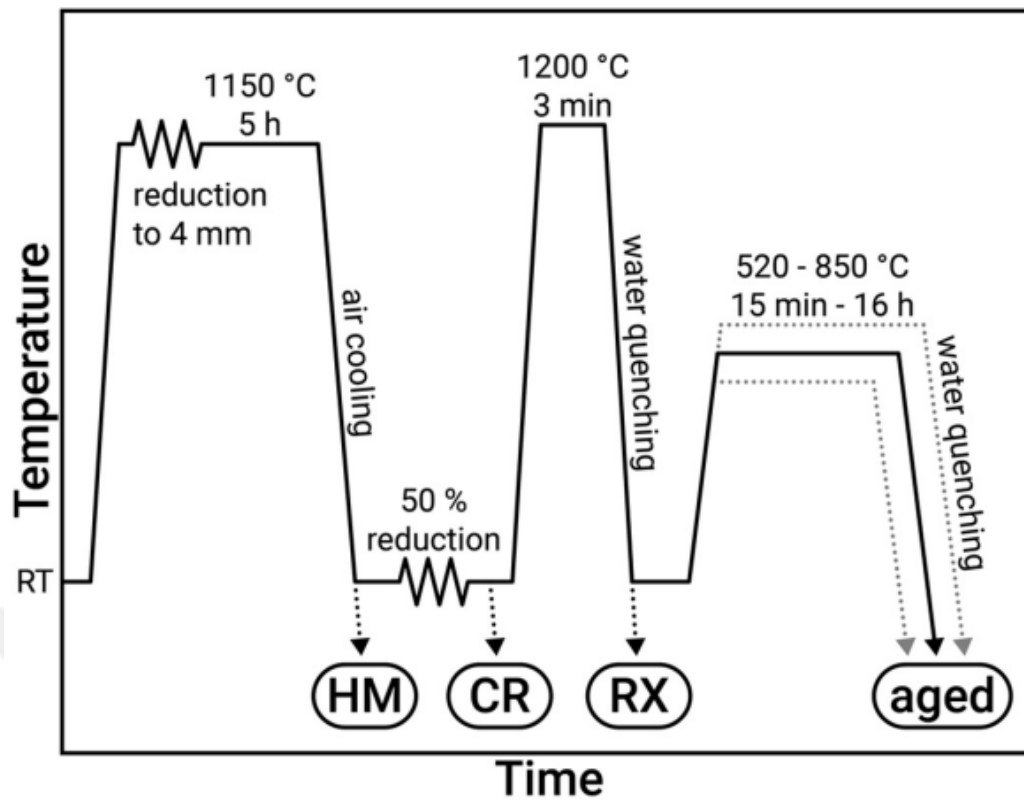


Figure 2.10 Typical process chart for Fe-Mn-Al-Ni-C Steel [14]

Overall, precipitation hardening through aging is a reliable method for improving the strength of lightweight steels without severely compromising ductility, especially when the phase evolution is properly controlled [7].

CHAPTER 3

EXPERIMENTAL

3.1 Casting and Preparation of the Samples

The casting process for the composition determined within the scope of the light steel project was carried out using an induction furnace at Akdaş Döküm (Ankara, Türkiye). In order to facilitate the extraction of samples from the casting, the alloy was cast as a keel block with the dimensions of 340x330x110 in mm as shown in Figure 3.1. This method ensures that the sample extraction process can be performed efficiently.

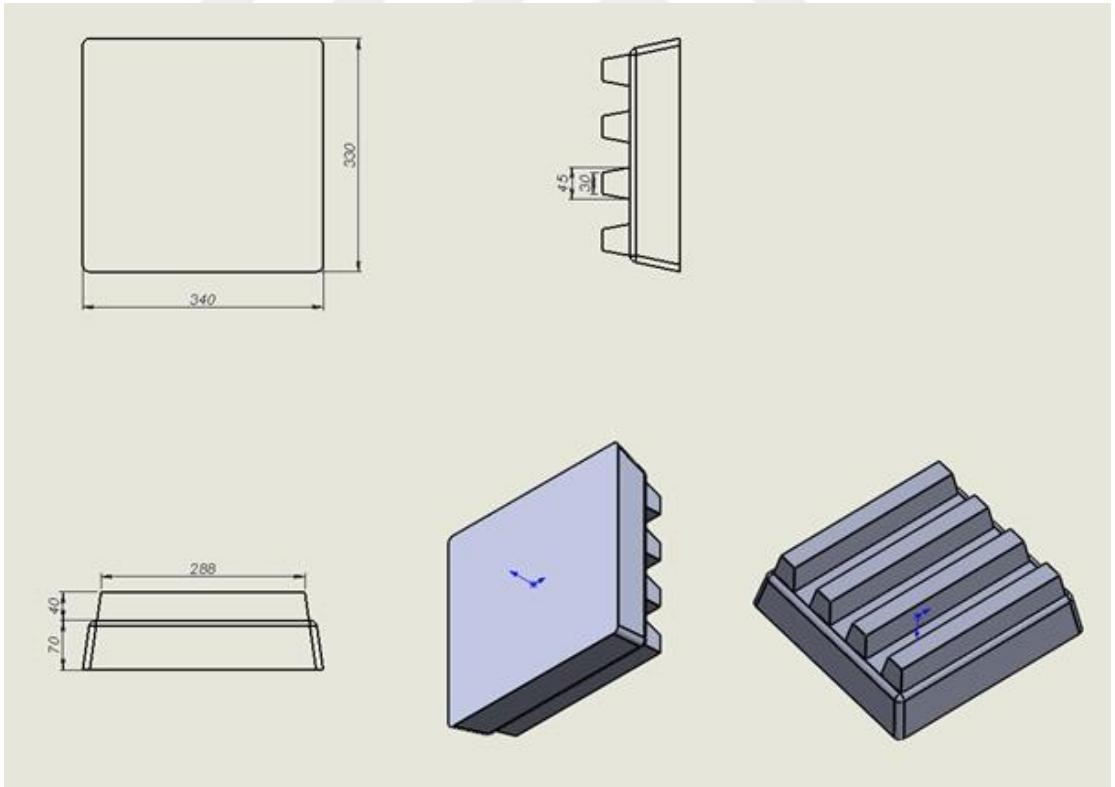


Figure 3.1 Schematic representation of cast keel block

The chemical composition of the cast material (Table 3.1) was determined using Thermo Scientific Element GD Plus GD-MS and Leco CS 744 Series instruments (Figure 3.2).

Table 3.1 Chemical composition of the casting

Element	Mn	Al	Ni	C	Si	Fe
wt%	16.75	9.29	9.25	0.98	0.20	Bal.



Figure 3.2 Leco CS 744 combustion analysis for carbon and sulfur [31]

After the chemical composition verification process, disk samples with a diameter of 25 mm and a thickness of 11 mm were cut from the keel block, for the first stage of experiments (Figure 3.3).

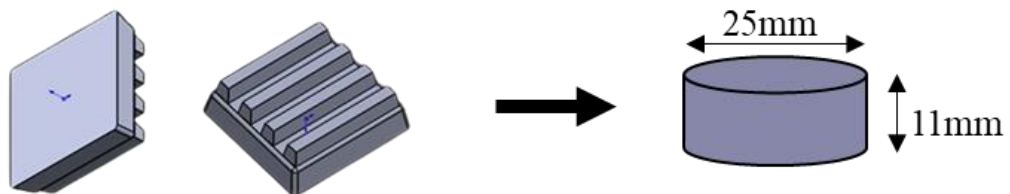


Figure 3.3 Preparation of disk samples

The selection of these dimensions was made to maximize the sample yield from the keel block and to ensure that there is sufficient sample volume for material characterization procedures which include microstructural examinations using optical microscope and SEM, XRD analysis, wear testing, macro and nano hardness testing.

As the second stage of the experimental process, samples were wire cut from the threads on the keel block to examine the effect of the thermal treatment parameters, deemed optimal based on mechanical properties such as hardness, microstructure, and wear resistance obtained from the heat treatment process, on mechanical properties. These samples were used to evaluate how the changes induced by the heat treatment affect the material's mechanical behavior and to determine the optimum thermal treatment parameters.

3.2 Density Measurement

The density of the cast material was calculated using the Equation (3.1), the weight and volume of the samples. The sample weight was determined using a TP-214 (S/N:21460049) precision balance from Denver Instrument (Figure 3.4), while the sample dimensions were measured using a precision caliper. Based on the values obtained, the material density was calculated using the mass-volume ratio formula.

$$\rho = \frac{m}{V} \quad (3.1)$$

Where:

- **ρ** : Density (measured in g/cm³),
- **m** : Mass (measured in g),
- **V** : Volume (measured in cm³).



Figure 3.4 Denver Instrument brand TP-214 precision balance

3.3 Thermo-Calc Analysis

Prior to experimental planning, Thermo-Calc analysis was conducted to gain more information about the cast steel and to assist in determining the heat treatment parameters. This analysis was used to calculate the temperature-dependent equilibrium phase fractions between room temperature and 1700°C based on Gibbs energy minimization.

The resultant diagram given in Figure 3.5 shows the phase fractions and critical transformation temperatures. The analysis revealed that at the solidification start temperature of 1355°C, delta ferrite forms in the relevant composition. At approximately 1244°C, a peritectic transformation occurs, and the primary precipitate of austenite and NiAl (B2) forms. When the steel solidifies at 1237°C, it was observed that the Kappa phase forms at 800°C, and upon examining the lattice structure, it was

identified as an AlFe_3C precipitate. Alpha ferrite forms at 610°C . Additionally, it was observed that the austenite phase completely disappears below 436°C .

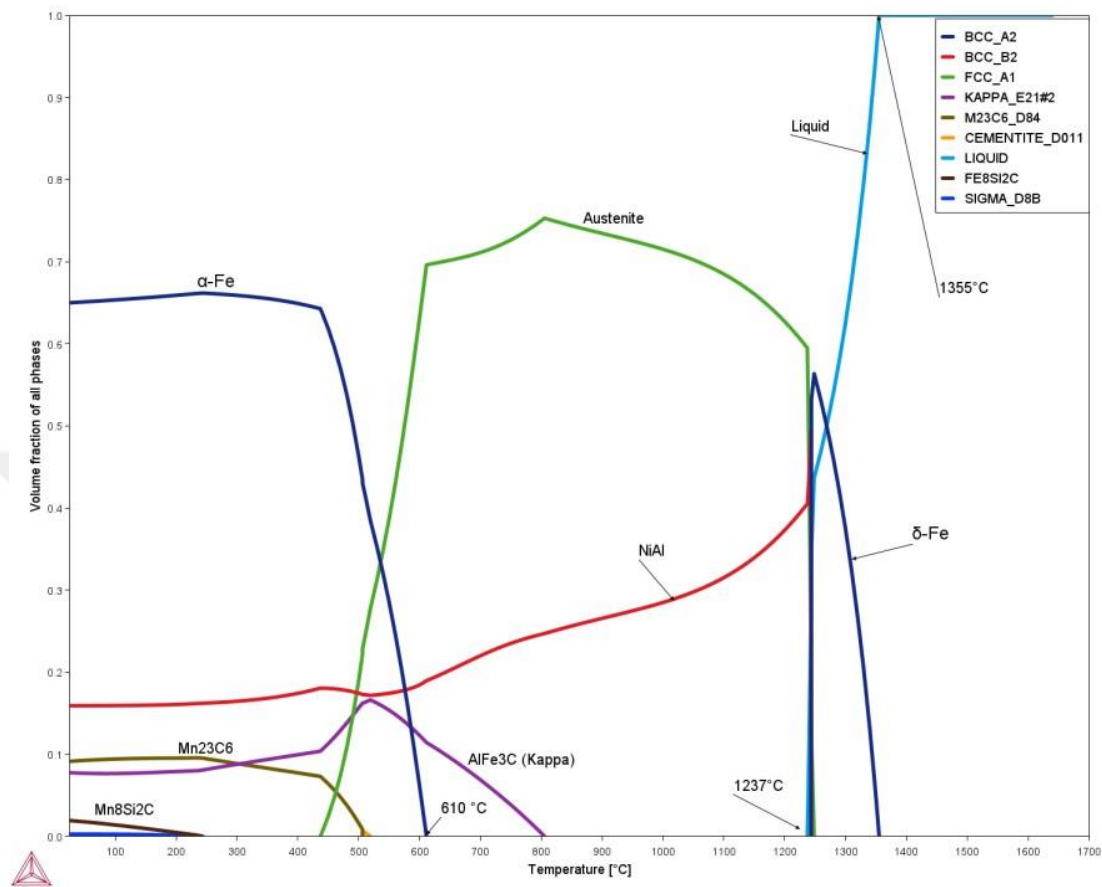


Figure 3.5 Thermo-Calc analysis of the studied composition

3.4 Heat Treatment Conditions

The initial stage of the project, involving heat treatment, was carried out in a Protherm MOS 170/8 furnace (Figure 3.6). To ensure the accuracy of the applied heat treatment, temperature deviations of the furnace were monitored and evaluated in real-time using an ORDEL UDL100 Universal Data Logger and a thermocouple.



Figure 3.6 Protherm furnace and ORDEL thermocouple system

The heat treatment process is also referred to as age (precipitation) hardening within the scope of the project. The parameters used in this process were determined based on data obtained from literature research and Thermo-Calc analyses. The precipitation hardening process consists of two main stages. The first stage focuses on the solutionizing treatment (ST). In this stage, various trials were conducted to determine the optimal ST parameters, while the aging parameters were kept constant throughout the process. Following the ST process, the samples were subjected to quenching to rapidly cool down to room temperature and retain the supersaturated state. This approach allowed for a detailed investigation of the effects of the ST process on the material's microstructural development and mechanical properties.

In the second stage, aging treatments were performed at different temperatures and durations while keeping the previously determined ST parameters constant. Following each aging process, the material was subjected to quenching to preserve the desired microstructural characteristics. Within this scope, the effects of the aging process on mechanical properties and microstructure were evaluated, aiming to determine the

optimal precipitation hardening conditions. Throughout both stages, experiments and analyses were conducted to identify the ideal parameters that would ensure the material achieves its highest performance. The parameters of all heat treatments performed within the scope of Stage 1 and Stage 2 are shown in Table 3.2.

Table 3.2 Age hardening conditions of the samples

Sample	Solutionizing		Cooling	Aging		Cooling
	Temperature (°C)	Time (min)		Temperature (°C)	Time (min)	
1	850	120	Quench into water at 25°C	700	480	Quench into water at 25°C
2	850	240		700	480	
3	950	960		550	960	
4	950	960		-	-	
5	1050	120		700	480	
6	1050	240		700	480	
7	1150	120		700	480	
8	1150	240		700	480	
9	1150	120		700	60	
10	1150	120		700	240	
11	1150	120		700	960	
12	1150	120		550	60	
13	1150	120		550	240	
14	1150	120		550	480	
15	1150	120		550	960	
16	1150	120		700	480	Air Cool
17	1150	120		550	480	
18	1150	120		550	960	

3.5 Characterization of the Samples

In order to observe and evaluate the effects of applied heat treatments, a comprehensive series of analyses and characterization techniques were conducted. These analyses aimed to assess microstructural transformations, mechanical properties, and overall material performance, providing a detailed understanding of the impact of the applied heat treatment parameters.

3.5.1 Microstructural Examination

Before the microstructural analyses conducted using optical microscopy and SEM, the materials were subjected to the metallographic sample preparation procedure. This procedure includes grinding, polishing, and etching steps. For the grinding process, SiC grinding papers with grit sizes of 180, 320, 600, 1200, and 2500 were used. Afterwards, the samples were polished with diamond suspensions of 6 μm , 3 μm , and 1 μm particle sizes, respectively. These operations were carried out using the automatic grinding and polishing device, Metkon FORCIPOL 202 (Figure 3.7). For the etching process, Vilella, 3% nital, and 5% nital etchants were tried. Observations showed that the best results were obtained using the 5% nital, hence the etching process was continued with the 5% nital etchant.



Figure 3.7 Metkon FORCIPOL 202 automatic grinding and polishing device [31]

High magnification images obtained using SEM and elemental analysis performed with Energy-Dispersive X-ray Spectroscopy (EDS) were acquired using the ZEISS MERLIN model (Figure 3.8). For these processes, the samples underwent grinding, polishing and etching steps. However, for advanced analyses such as phase mapping, dislocation density mapping, and grain size analysis, Electron Backscatter Diffraction (EBSD) techniques were employed. In these analyses, the samples were not subjected to etching; instead, after polishing with a 1 μm diamond suspension, a final polishing step using colloidal silica was performed.



Figure 3.8 ZEISS MERLIN Scanning Electron Microscopy [32]

3.5.2 X-Ray Diffraction (XRD)

X-ray Diffraction (XRD) patterns of the samples were obtained using the Rigaku SMARTLAB model (Figure 3.9). The analyses were performed with a Cu x-ray tube at 40 kV and 40 mA, covering the 2θ range from 30° to 100° . The data was collected with a step size of 0.01° and a scanning rate of 1.5° per minute.



Figure 3.9 Rigaku Smartlab XRD machine

The data obtained were plotted and analyzed using Origin Pro 2025 software to ensure accurate visualization and facilitate comparative evaluation of the experimental results [33].

3.5.3 Hardness Measurements

Hardness tests were conducted using the EMCOTEST Duravision 30 G5 hardness test machine in accordance with ASTM E10 standard (Figure 3.10). A 10 mm diameter ball and a load of 3000 kgf were applied during the measurements. The tests were performed with five repetitions.

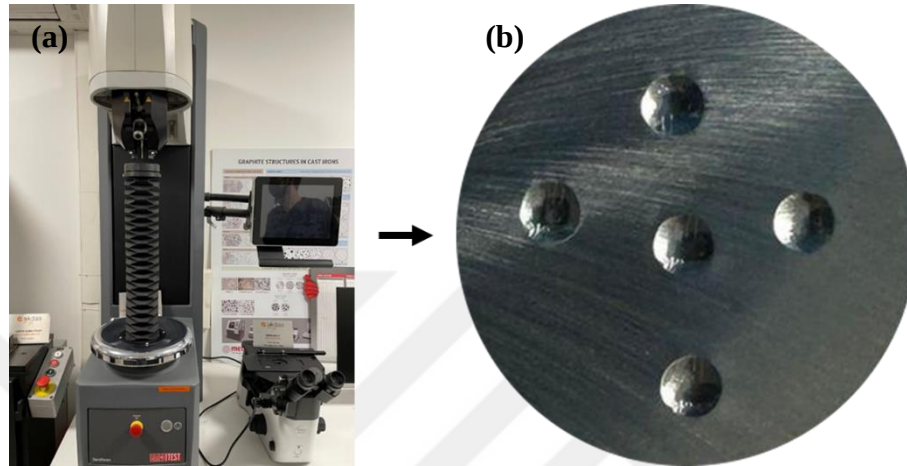


Figure 3.10 (a) EMCOTEST Duravision 30 G5 hardness test machine and (b) sample after hardness test

In addition to macro hardness testing, in order to investigate the hardness differences between phases within the material in more detail in the as-cast condition and after heat treatment, measurements were carried out using a CSM Ultra Nanoindentation Tester (UNHT) with an applied load of 25 mN (Figure 3.11).

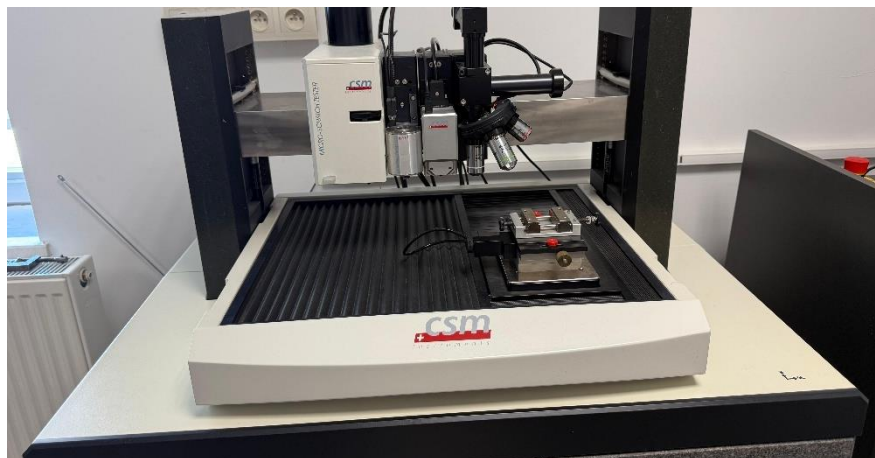


Figure 3.11 CSM Ultra Nanoindentation Tester (UNHT)

3.5.4 Wear Tests

The wear tests were conducted using a custom-made wear test machine in the Mechanical Test Laboratory of the Department of Metallurgical and Materials Engineering Department of Atılım University (Figure 3.12.a). The samples were weighed before and after the test using a precision balance of Denver Instrument. The wear tests were performed against a 6 mm zirconia ball as a counterface under a constant load of 84.3N (8600.3 g). The average diameter of the wear tracks was 14 mm. Rotational speed during the wear tests was 150 rpm which produced a linear sliding speed of 11 mm/s. Duration of the wear tests were 2 hours which correspond to 18,000 cycles and 791.3 m of total sliding distance. The tests were carried out at room temperature and under a relative humidity of 40%. After the tests, the wear rate of the sample was calculated using the Equation (3.2).

$$\text{Wear Rate: } \frac{\Delta m}{S \cdot N} \left(\text{in } \frac{g}{N \cdot m} \right) \quad (3.2)$$

Where:

- Δm : Mass change of the sample (in grams),
- S : Total sliding distance (in m),
- N : Total load applied during the test (in Newtons).

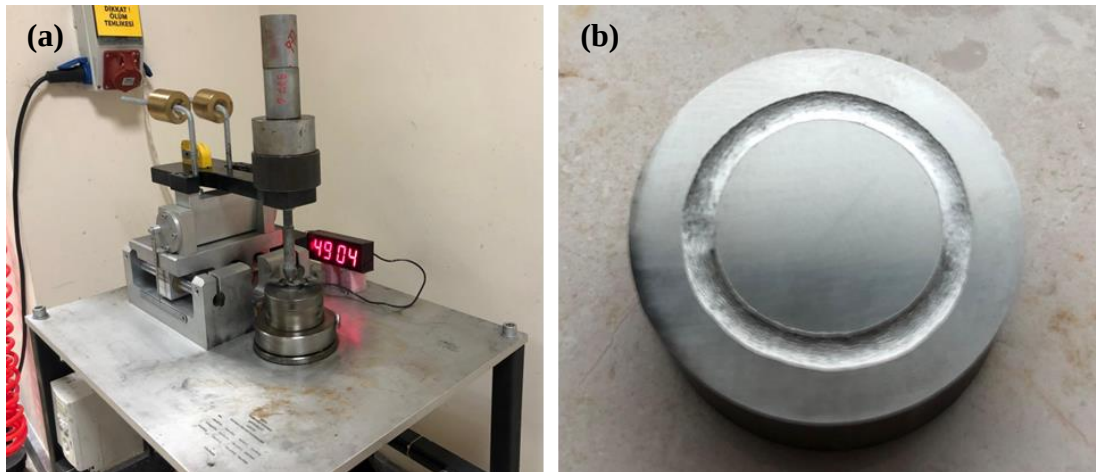


Figure 3.12 (a) Pin-on-Disk wear test machine and (b) a sample after the wear test

3.5.5 Surface Metrology

To further examine the effects of wear rate tests on the surface, surface measurements were performed on selected samples using the Alicona InfiniteFocus device (Figure 3.13). To increase measurement precision, parameters were set for a vertical resolution of 400 nm and a lateral resolution of 7 μm .

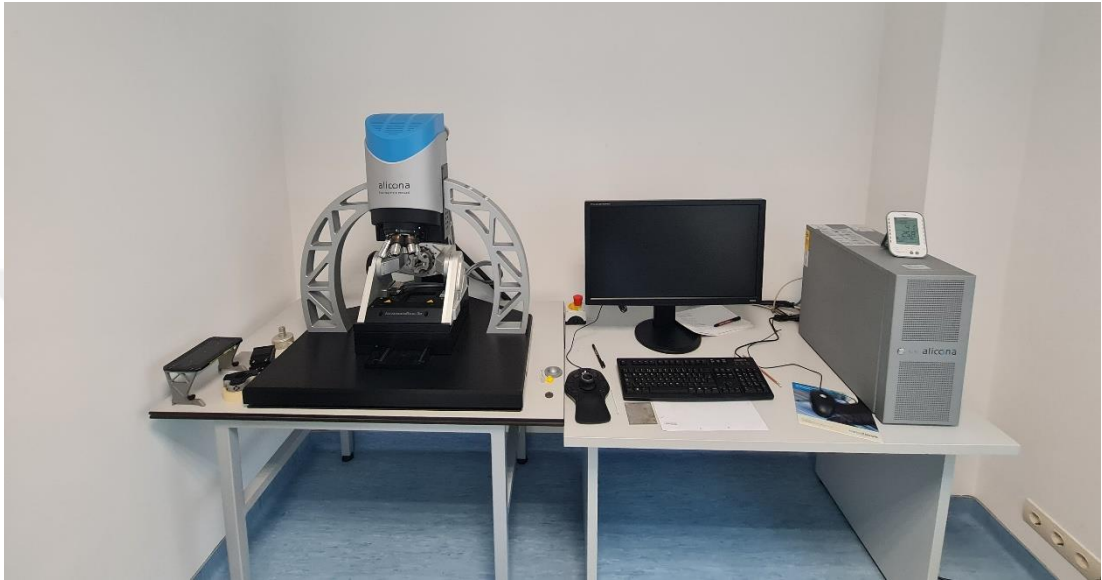


Figure 3.13 Alicona InfiniteFocus surface metrology instrument

CHAPTER 4

RESULTS

4.1 Density Measurement

Based on the mass and the volume values of the disk sample, the material density was calculated using the mass-to-volume ratio formula (Equation 3.1).

$$\rho = \frac{m}{V} = \frac{m}{\pi * r^2 * h} \quad (3.1)$$

Dimensional and weight measurements were performed on three different samples, and the corresponding calculated density values are presented in Table 4.1. The measured densities were 6.78 g/cm³, 6.81 g/cm³, and 6.91 g/cm³, respectively. The average of these measurements was determined to be 6.83 g/cm³.

Table 4.1 Density calculations for different samples

Sample	Diameter (mm)	Thickness (mm)	Mass (g)	Density (g/cm ³)
1	24.8	10.46	34.24	6.78
2	24.8	10.80	35.55	6.81
3	25.0	9.61	32.54	6.91
Average Density		6.83 g/cm ³		
Standard Dev.		0.068		

4.2 Hardness Measurements

4.2.1 Macro Hardness

Hardness measurements were conducted at each stage of the heat treatment process, starting from the as-cast samples. To ensure accuracy, five measurements were taken from each sample, and the average of these values was considered as the hardness of the corresponding sample.

The hardness values of the as-cast and solutionized samples are presented in Figure 4.1. The average hardness of the as-cast sample was found to be 476.8 ± 3.9 HB. For the solutionizing treatments carried out at different temperatures and durations, the hardness values were measured as 377 ± 4.3 HB after 2 hours and 363.9 ± 8.3 HB after 4 hours at 850°C . Similarly, for the solutionizing conducted at 1050°C for 2 and 4 hours, the average hardness values were determined to be 284 ± 4.8 HB and 289.4 ± 7.1 HB, respectively. Following the 16-hour solutionizing treatment at 950°C , the average hardness was observed to be 306 ± 4.7 HB. For the highest solutionizing temperature of 1150°C , the average hardness values after 2 and 4 hours of heat treatment were 258 ± 21.6 HB and 267.6 ± 14.3 HB, respectively.

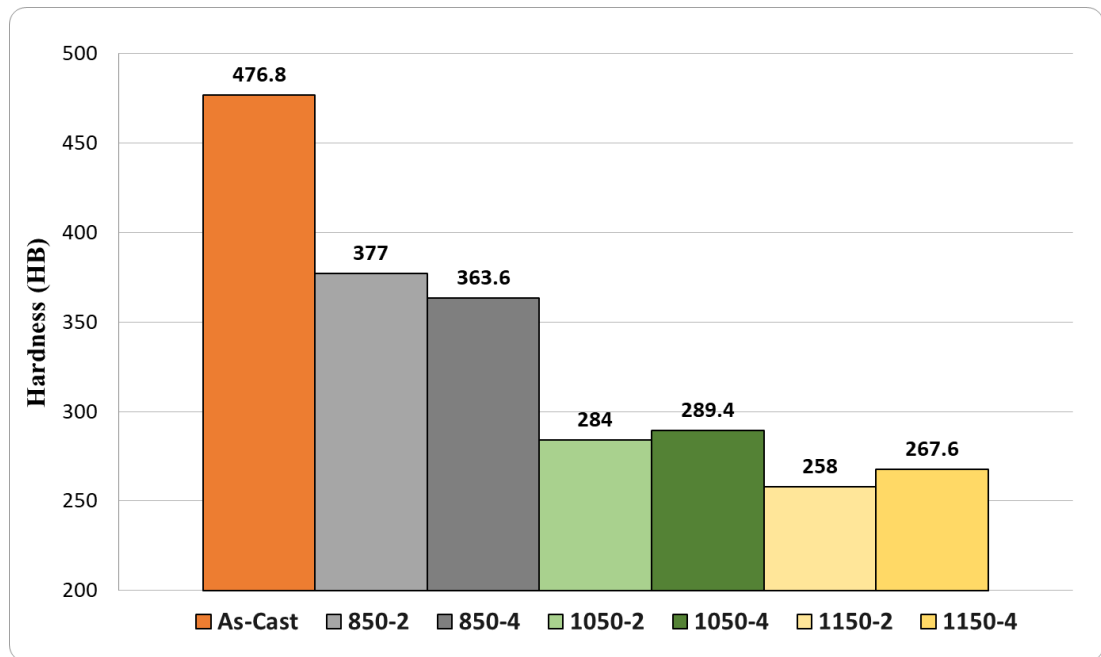


Figure 4.1 Average hardness values of as-cast and solutionized samples

For a more comprehensive interpretation of the results, the hardness tests and the five measurements recorded for each sample are presented in detail in Table 4.2.

Table 4.2 Average hardness values of as-cast and ST conditions

	Temperature (°C) – Time (Hours)							
#	As-cast	850-2	850-4	950-16	1050-2	1050-4	1150-2	1150-4
1	471	382	372	303	292	289	272	276
2	478	381	363	302	281	290	286	286
3	481	375	350	306	280	278	251	269
4	475	375	367	314	282	293	251	256
5	481	372	366	305	285	297	230	251
Average	476.8	377.0	363.6	306.0	284.0	289.4	258.0	267.6
Standard D.	3.9	4.3	8.3	4.7	4.8	7.1	21.6	14.3

As discussed in Chapter 3, in the initial stage of the heat treatment process, the aging parameters were fixed at 700°C for 8 hours in order to observe the effect of solutionizing on the material. Additionally, to investigate the impact of prolonged aging, the sample that underwent solutionizing at 950°C for 16 hours was subsequently aged at 700°C for 8 hours. The average hardness values obtained after the aging treatments are presented in Table 4.3.

Table 4.3 Hardness values for the same aging parameters with different ST conditions

	Temperature (°C) – Time (Hours)					
ST	850-2	850-4	1050-2	1050-4	1150-2	1150-4
AGING	700°C – 8 hours (Cooling by Quenching)					
1	395	404	386	404	379	393
2	390	377	376	389	382	404
3	384	365	367	393	393	392
4	375	390	351	390	391	407
5	399	378	382	389	354	402
Average	388.6	382.8	372.4	393.0	379.8	399.6
Standard D.	9.4	14.8	13.9	6.4	6.7	15.6

Following all these processes, the solutionizing temperature and duration were determined to be 1150°C for 2 hours. In the next phase of the project, aging parameter trials were conducted at various temperatures and durations while keeping the solutionizing conditions constant. Based on the data obtained from Thermo-Calc analysis, the aging temperatures were selected as 700°C to investigate the effects of the B2 phase, and 550°C to observe the influence of κ -carbides. The aging durations were set as 1, 4, 8, and 16 hours.

The hardness values obtained from the aging treatments conducted at 700°C and 550°C under constant solutionizing temperature and duration are presented in Table 4.4 and Table 4.5, respectively.

Table 4.4 Hardness values of samples aged at 550°C for 1h, 4h, 8h, 8h (air cooled), 16h, and 16h (air cooled)

	Temperature(°C) – Time (Hours)					
ST	1150°C - 2 hours					
AGING	550-1	550-4	550-8	550-8 (Air Cooled)	550-16	550-16 (Air Cooled)
1	392	446	464	446	497	465
2	387	455	385	440	486	456
3	397	450	404	446	478	468
4	395	456	-	431	-	486
5	389	452	-	431	-	462
Average	392.0	451.8	412.7	438.8	487.0	467.4
Standard D.	4.1	4.0	41.2	7.5	9.5	11.3

Table 4.5 Hardness of samples aged at 700°C; 1h, 4h, 8h, 8h (air cooled), and 16h

	Temperature (°C) – Time (hours)				
ST	1150°C - 2 hours				
AGING	700-1	700-4	700-8	700-8 (Air Cooled)	700-16
1	382	390	393	392	390
2	377	368	404	384	387
3	375	378	392	397	381
4	370	377	407	387	384
5	383	384	402	392	392
Average	377.4	379.4	399.6	390.4	386.8
Standard D.	5.3	8.2	6.7	5.0	4.4

As shown in the tables, following the solutionizing treatment conducted with the ST-1150-2 parameter, the aging processes performed at 550°C for 1, 4, 8, 8 (air cooled), 16 and 16 hours (air cooled) resulted in hardness values of 392±4.1 HB, 451.8±4.0 HB, 412.7±41.2 HB, 438±7.5 HB, 487±9.5 HB, and 467.4±11.3 HB respectively. Due to cracking of the material during the hardness test after aging with the 550-16 and 550-8 parameters, only three hardness values could be obtained for that condition. In addition, the hardness test data for condition 550-8 was not taken into consideration due to inconsistency.

Following the aging treatments at the other designated temperature, 700°C, the hardness values were observed to be 377.4±5.3 HB after 1 hour, 379.4±8.2 HB after 4 hours, 399.6±6.7 HB after 8 hours, and 386.8±4.4 HB after 16 hours. An overall increase in hardness with aging time was observed, followed by a slight decrease at the longest duration.

In addition to the hardness tests, a three-point bending test was also performed on the casting specimen to provide a more comprehensive evaluation of the mechanical properties. The results of this test are presented in Appendix A.

4.2.2 Nano Hardness

Nanoindentation tests were performed in two rows with 15 indents per row in order to obtain hardness values from both the austenitic matrix and the B2 phase on the sample surface (Figure 4.2). To prevent interaction between the indents, a spacing equal to 30 times the indentation depth was maintained between each indent. The average hardness obtained from the upper row is 9.3 GPa with the standard deviation of 0.67 GPa and for the lower row 9.1 GPa with the standard deviation of 0.47 GPa.

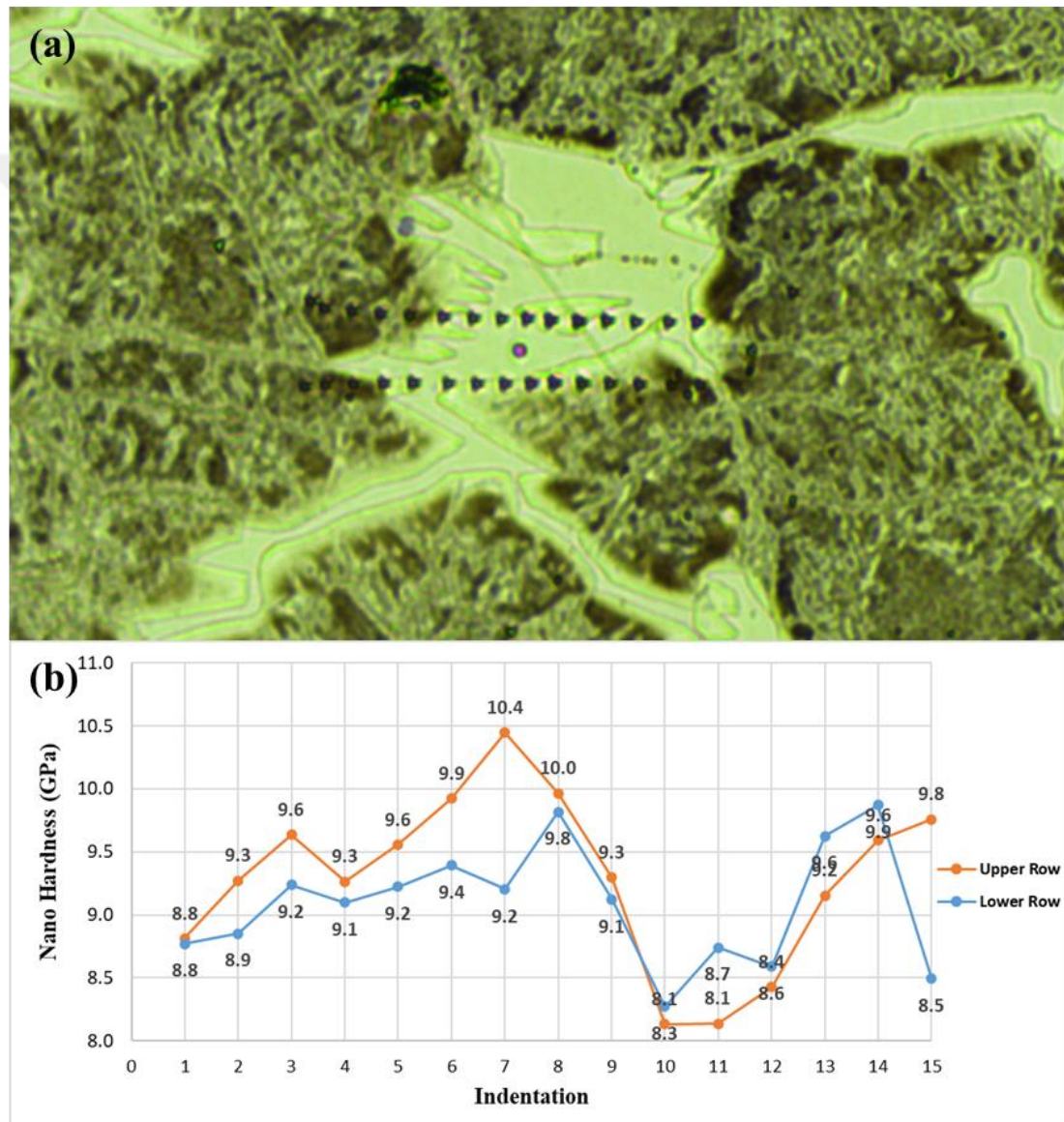


Figure 4.2 (a) Nanoindentation applied sample, (b) test results

4.3 Microstructural Examination of the Samples

4.3.1 As-Cast Microstructure

The microstructural analysis of the samples was conducted using the parameters described under Section 3.5.1. The microstructural images obtained for the as-cast sample are presented in Figure 4.3. Within the as-cast microstructure, B2 phase with various morphologies and the austenitic matrix can be distinctly observed.

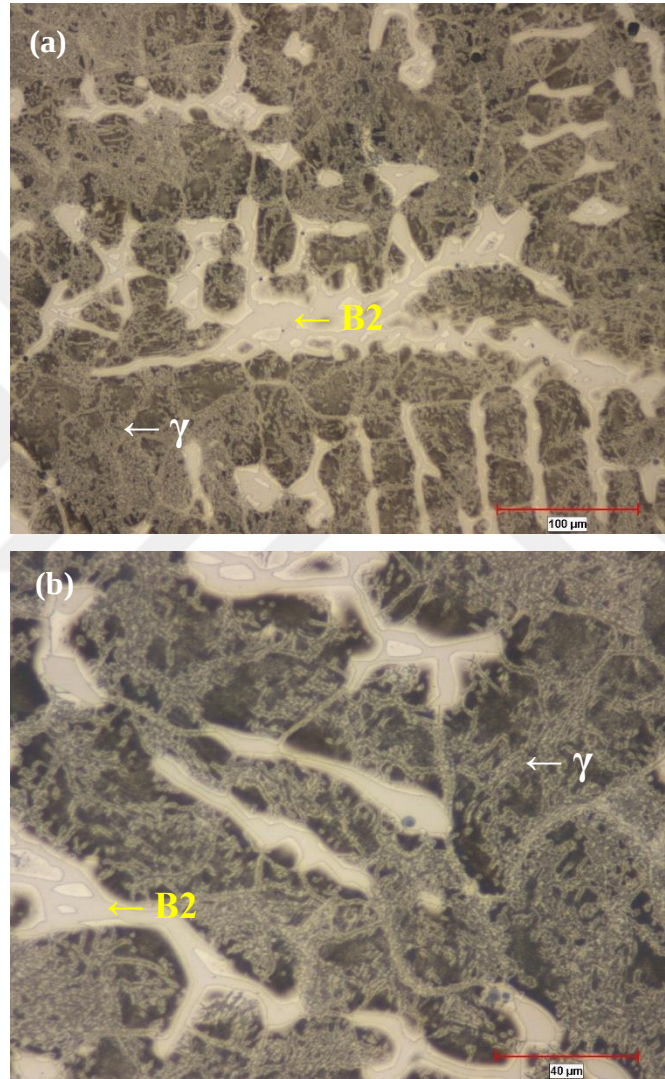


Figure 4.3 Optical microstructural images of the as-cast sample: a) 200X and b) 500X. (5% Nital etched.)

4.3.2 Microstructural Examination of the Solutionized Samples

The microstructural images obtained as a result of solutionizing treatments performed at four different temperatures and durations are presented as follows:

The effect of solutionizing at 850°C for 2 and 4 hours on the microstructure is shown in Figure 4.4. In the microstructure, the light-colored regions correspond to the B2 phase, while the dark-colored regions indicate the austenite phase. The B2 phase islands embedded within the austenitic matrix can be clearly observed in the microstructures.

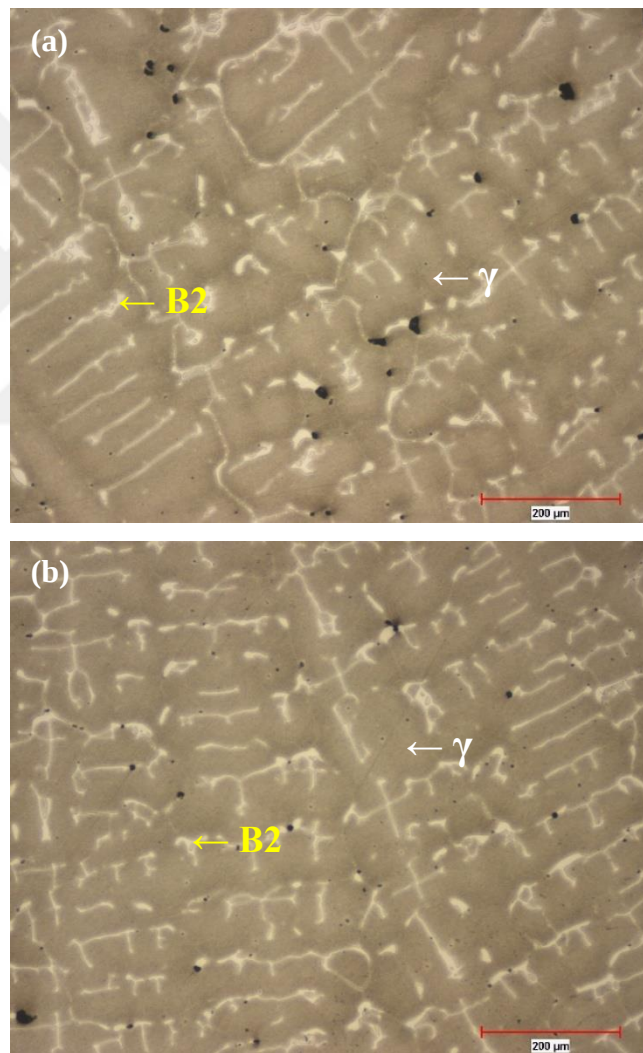


Figure 4.4 Optical microstructural images of the sample solutionized at 850°C for (a) 2h, (b) 4h. (5% Nital etched, 100X)

The microstructure of another solutionized sample, which was held at 950°C for 16 hours followed by quenching, is shown in Figure 4.5. In the image, the dark-colored regions represent the austenitic matrix, while the light-colored areas correspond to the B2 phase, both of which can be clearly distinguished. At higher magnifications, needle-like B2 phases within the matrix are also observable.

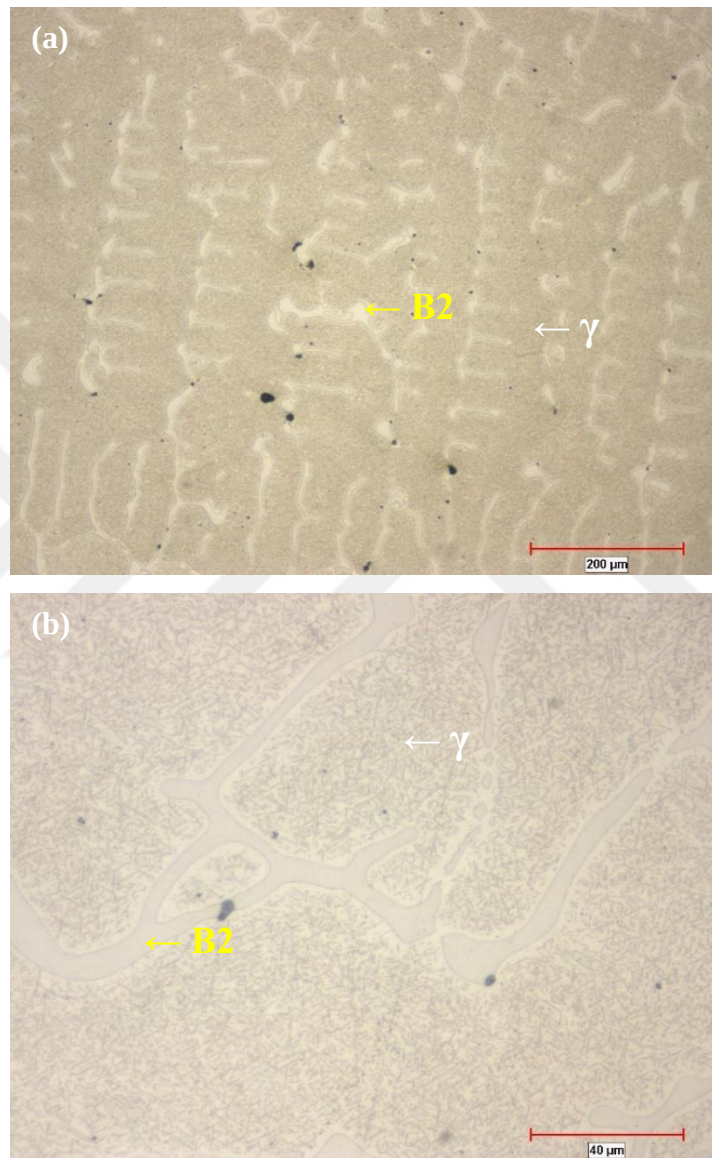


Figure 4.5 Solutionized microstructure at 950°C for 16h (a) 100X, (b) 500X (Samples etched with %5 Nital)

The effect of solutionizing at 1050°C for 2 and 4 hours on the microstructure is shown in Figure 4.6. B2 phase islands within the austenitic matrix are clearly observed in both conditions. In addition, needle-like B2 phases dispersed throughout the matrix can be distinguished at higher magnifications.

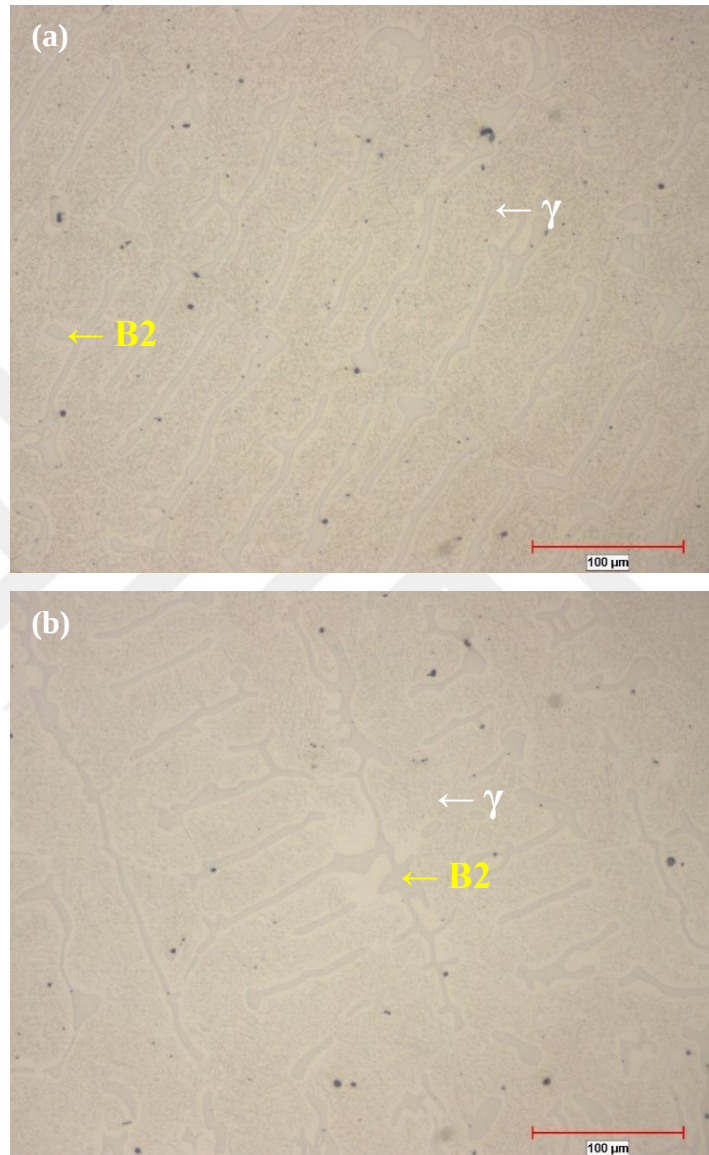


Figure 4.6 Optical microstructural images of the sample solutionized at 1050°C for (a) 2h, (b) 4h. (5% Nital etched, 200X)

The final solutionizing treatment was carried out at 1150°C. The microstructural images obtained from samples solutionized at 1150°C for 2 and 4 hours are presented in Figure 4.7.

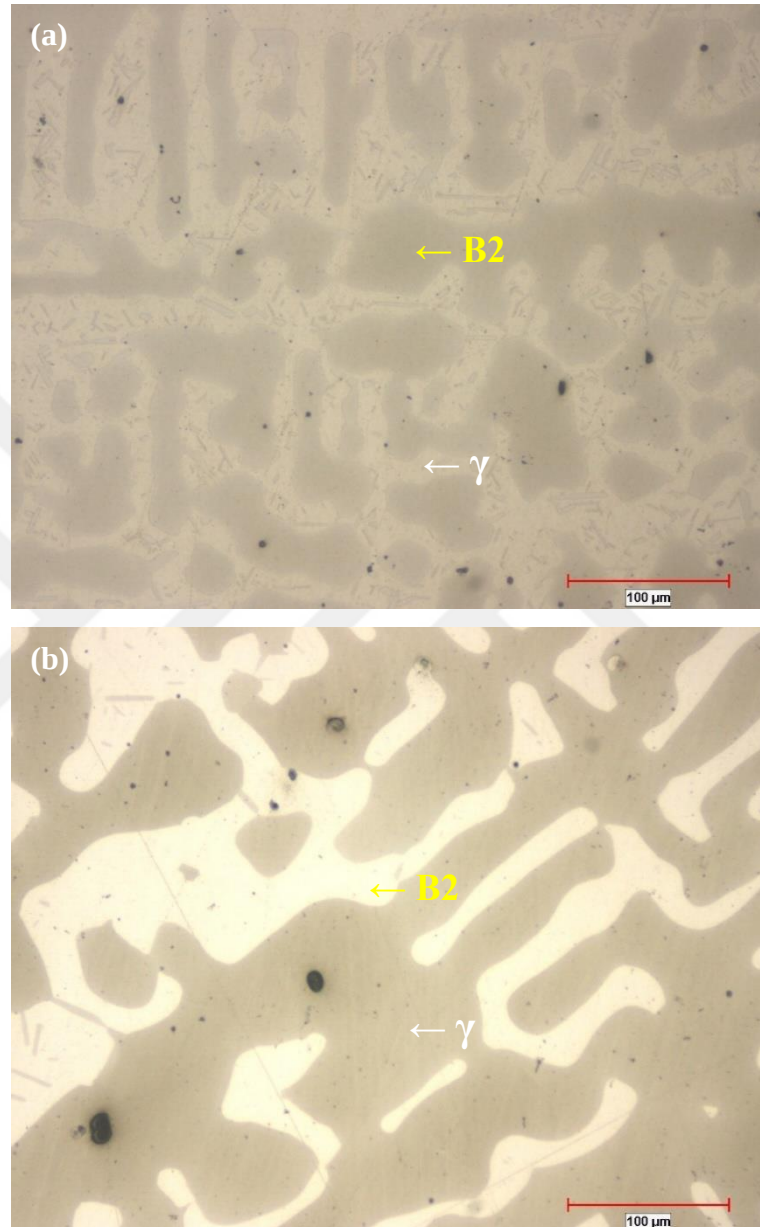


Figure 4.7 Optical microstructural images of the sample solutionized at 1150°C for (a) 2h, (b) 4h. (5% Nital etched, 200X)

4.3.3 Microstructural Examination of the Aged Samples

Within the scope of this thesis, the effect of aging temperature is studied under two different aging temperatures of 700°C and 550°C.

4.3.3.1 Samples Aged at 700°C

The microstructural images corresponding to the aging treatment conducted at 700°C for 8 hours, after solutionizing at 850°C for 2 and 4 hours, are presented in Figure 4.8. The dark-colored austenitic matrix and the light-colored B2 phase within the microstructure can be observed.

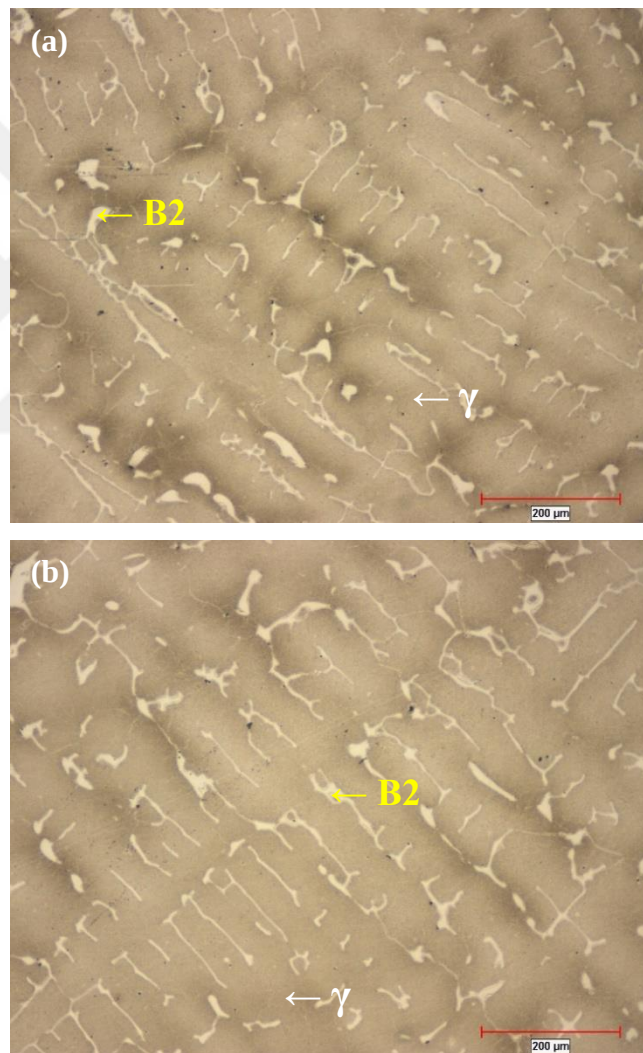


Figure 4.8 Optical microstructural images of the samples aged at 700°C for 8h after solutionizing at 850°C for (a) 2h, (b) 4h. (5% Nital etched, 100X)

The microstructural images corresponding to the aging treatment conducted at 700°C for 8 hours, following solutionizing at 1050°C for 2 and 4 hours, are shown in Figure 4.9. The needle-like B2 structures that were present within the matrix after solutionizing were not observed in the microstructure following the aging treatment.

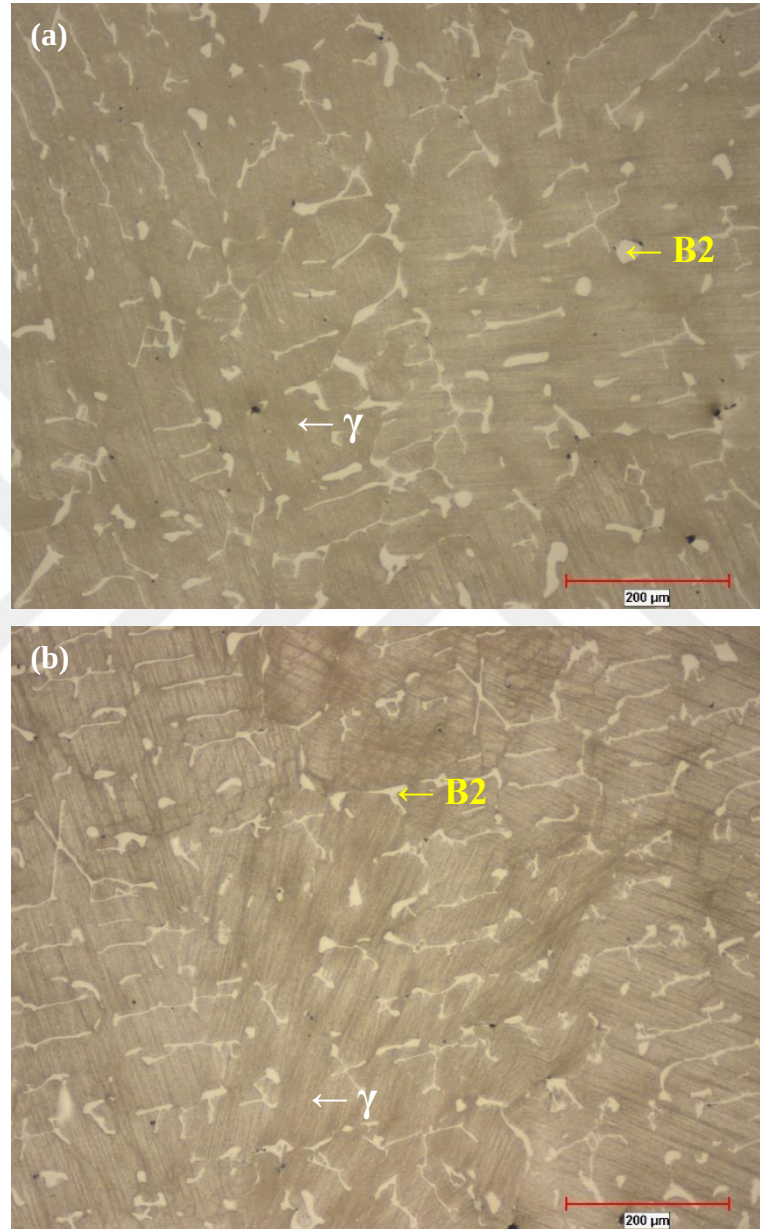


Figure 4.9 Optical microstructural images of the samples aged at 700°C for 8h after solutionizing at 1050°C for (a) 2h, (b) 4h. (Viella etched, 100X)

The microstructure images obtained after the aging treatment at 700°C for 8 hours, following solutionizing at 1150°C for 2 and 4 hours, are presented in Figure 4.10. Notably, in the sample that underwent solutionizing at 1150°C for 4 hours, needle-like B2 structures were observed within the microstructure in clustered formations.

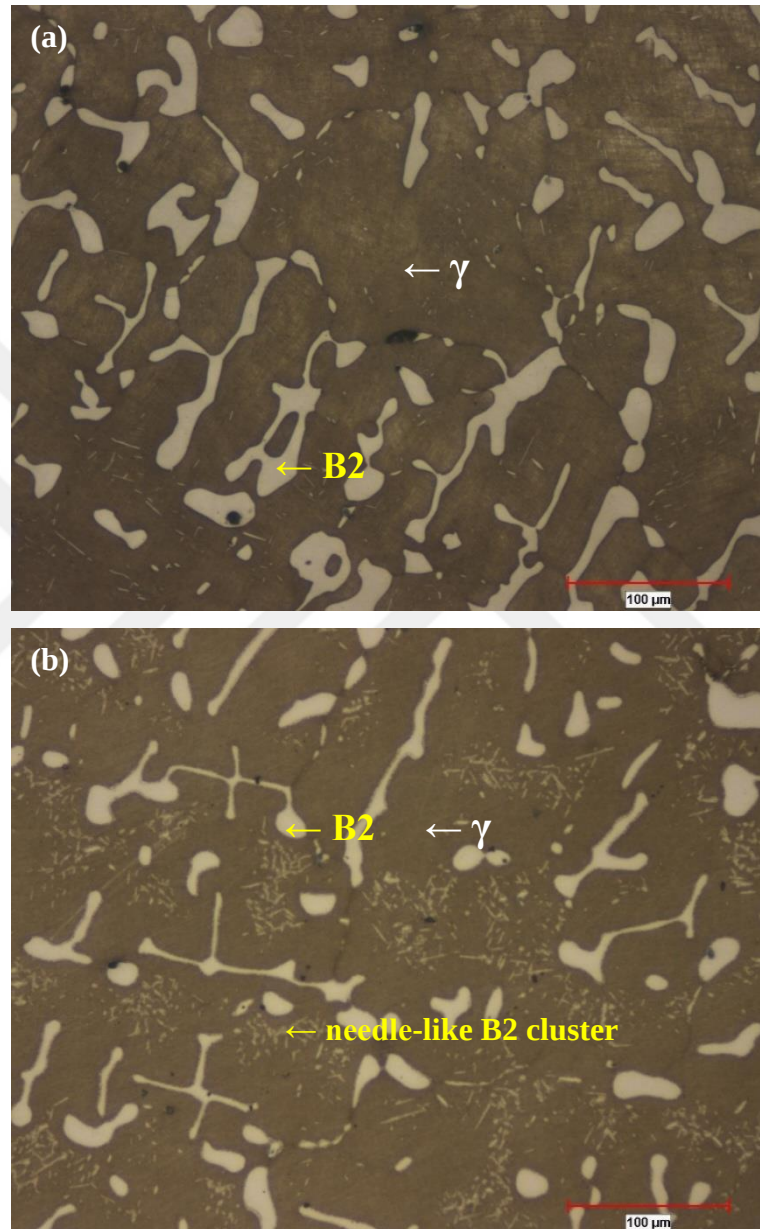


Figure 4.10 Optical microstructural images of the samples aged at 700°C for 8h after solutionizing at 1150°C for (a) 2h, (b) 4h. (Viella etched, 100X)

In the first stage of the project, an aging treatment at 700°C for 8 hours was applied to all samples in order to evaluate the effects of the solutionizing parameters, and the resulting microstructures were presented above. In the second stage of the project, after determining the solutionizing parameters as 1150°C for 2 hours, aging treatments at 700°C for 1, 4, 8 (air cooled) and 16 hours were carried out to observe the effects of different aging durations and different cooling methods on the microstructure. The resulting microstructures obtained after these treatments are presented in Figure 4.11.

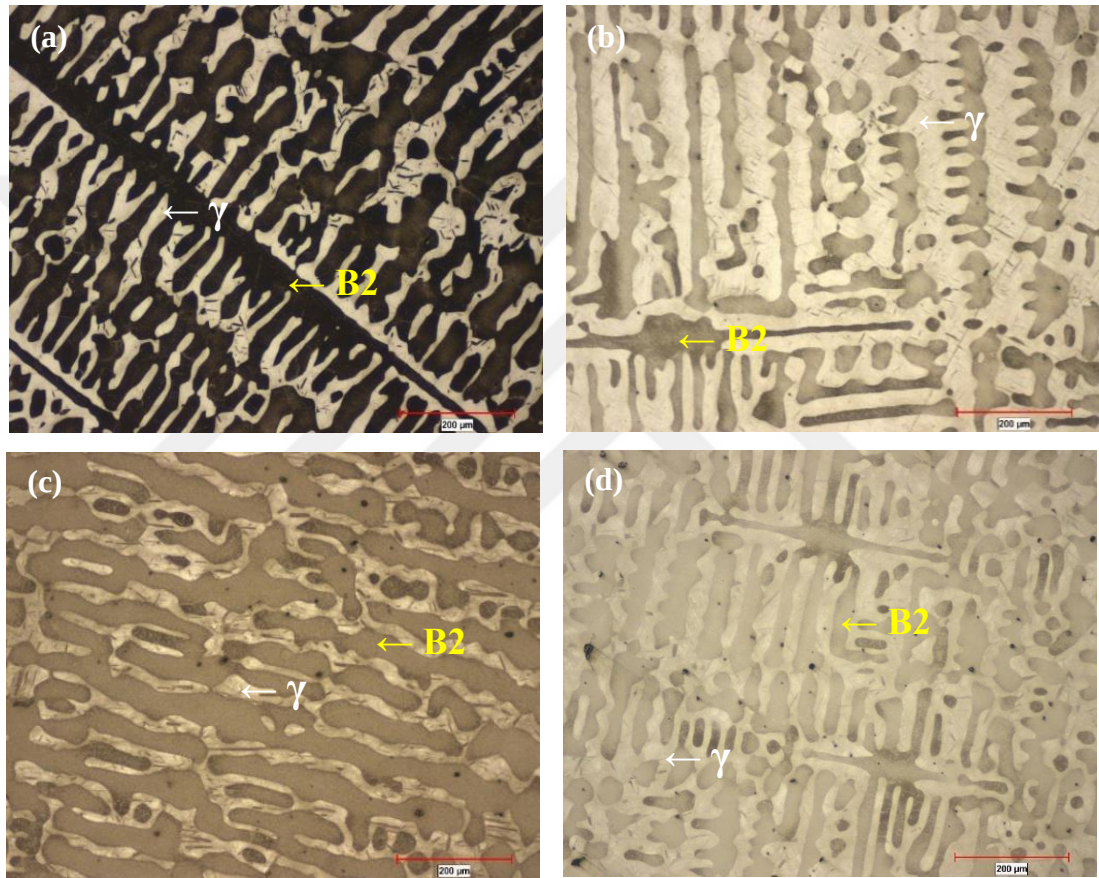


Figure 4.11 Optical microstructural images of the samples aged at 700°C for (a) 1h, (b) 4h, (c) 16h, and (d) 8h (air cooled) after solutionizing at 1150°C for 2h. (5% Nital etched, 100X)

4.3.3.2 Samples Aged at 550°C

The second aging temperature selected after the solution treatment is 550°C. Similar to 700°C, the microstructure images obtained in this aging process, which was carried out for 1, 4, 8, 8 (air cooled), 16, and 16h (air cooled) in order to see the effects of κ -carbide on the mechanical properties and microstructure, are shown in Figure 4.12.

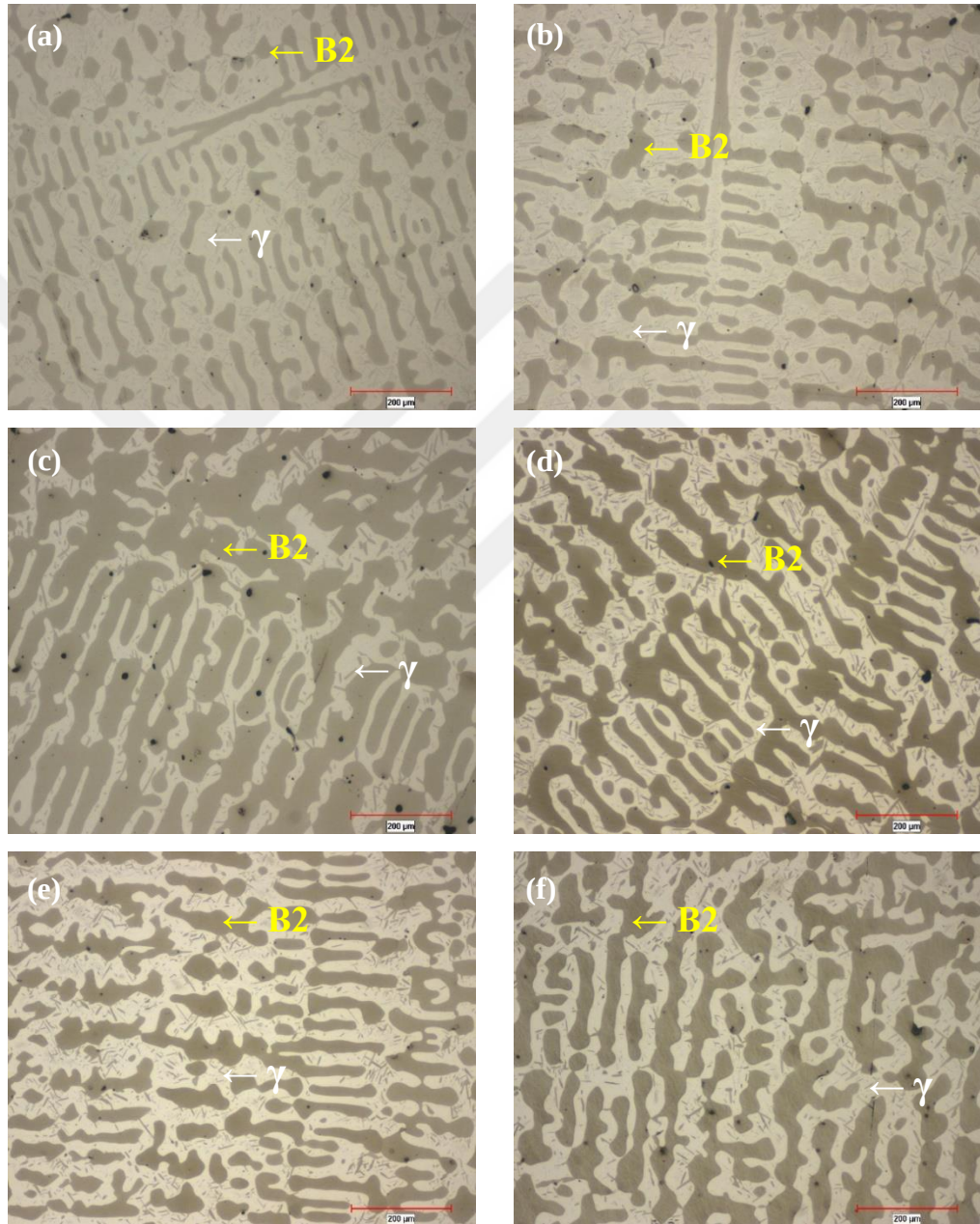


Figure 4.12 Optical microstructural images of the samples aged at 550°C for (a) 1h, (b) 4h, (c) 8h, d) 16h, (e) 8h (air cooled), and (f) 16h (air cooled) after solutionizing at 1150°C for 2h. (5% Nital etched, 100X)

4.4 SEM Investigation

SEM investigation was conducted in order to examine the microstructures formed in the as-cast, solutionized, and aged samples at high magnifications to obtain further information.

4.4.1 As-cast

From the as-cast sample, images were taken from two different areas, and EDS analyses were performed on various points and along lines on different regions within these images. In Figure 4.13, the austenitic matrix and the B2 islands within this matrix can be clearly observed.

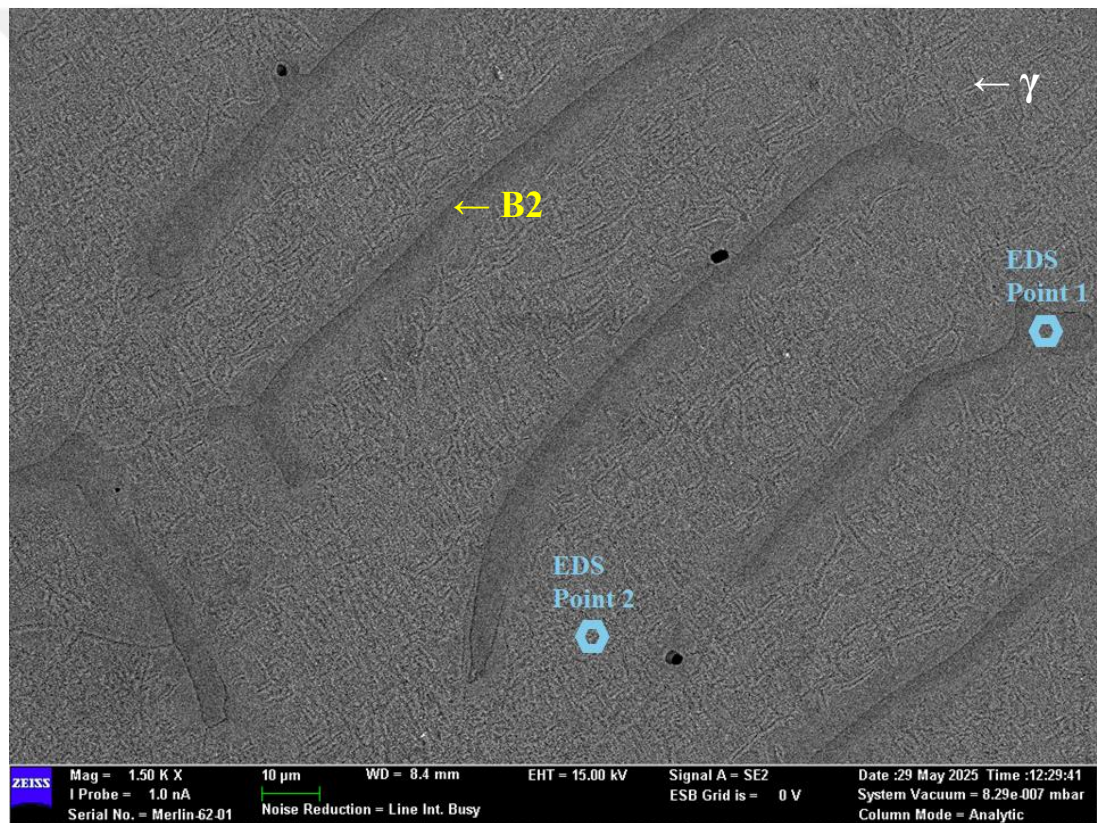


Figure 4.13 As-cast EDS point analysis at austenite matrix and B2 phase (5% Nital etched, 1500X)

The results of EDS analysis performed at the points shown in Figure 4.14 are shown in Table 4.6.

Table 4.6 Results of EDS points 1 and 2

Element (wt%)	Mn	Al	Ni	Fe
EDS Point 1	8.25	17.9	25.27	48.57
EDS Point 2	16.43	9.74	7.83	66.00

The EDS line analysis shown in Figure 4.14, performed across the austenitic matrix and B2 phase over 500 points, yielded a composition of 64.96 wt.% Fe, 14.96 wt.% Mn, 9.692 wt.% Al, and 10.38 wt.% Ni.

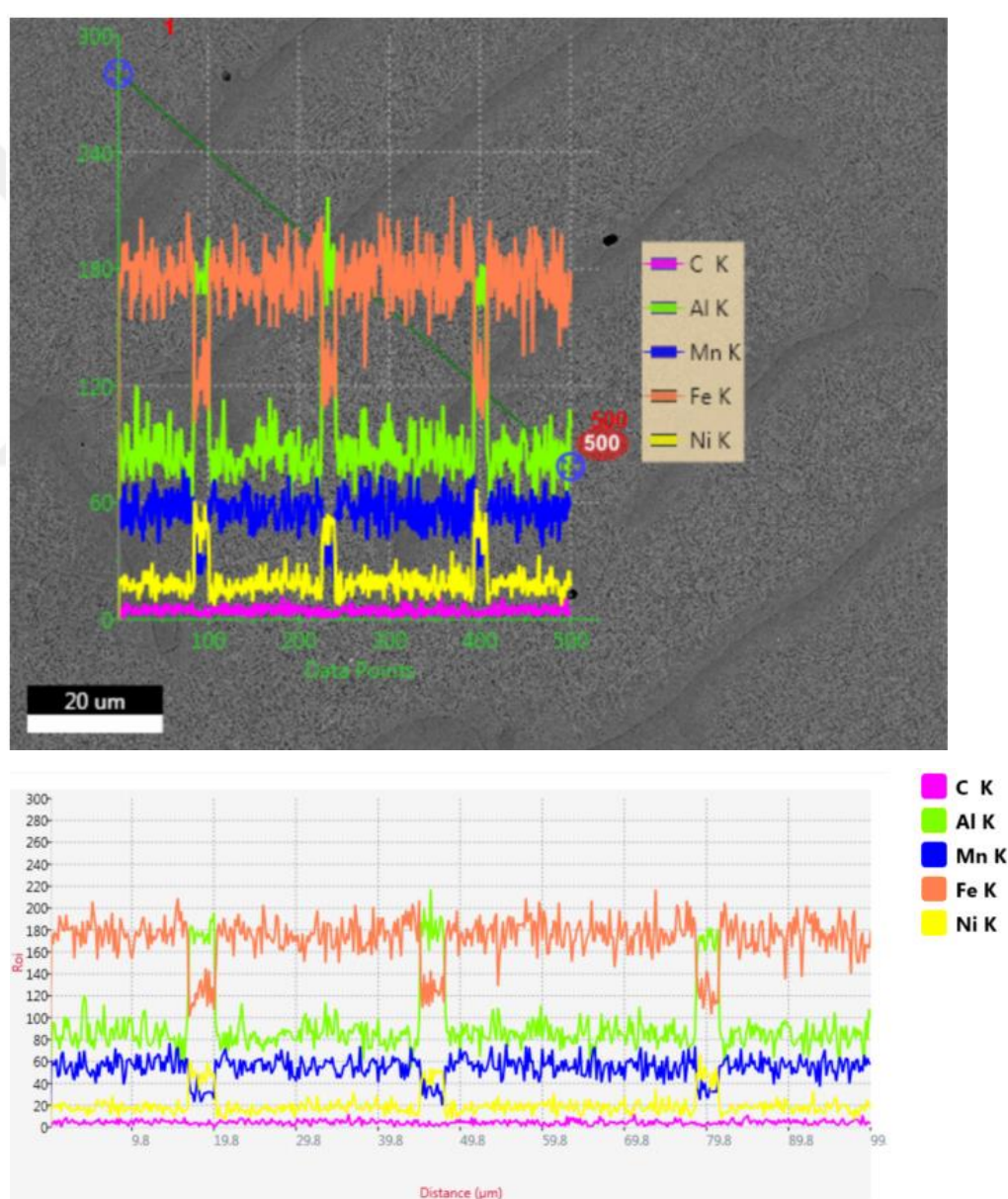


Figure 4.14 EDS line analysis for austenite matrix and B2 phase

Figure 4.15 clearly illustrates the distinction between the matrix and the B2 islands and also contains images of three point-based EDS analyses performed in this region. In this image, the points were selected from within the B2 phase and from the austenitic matrix, respectively.

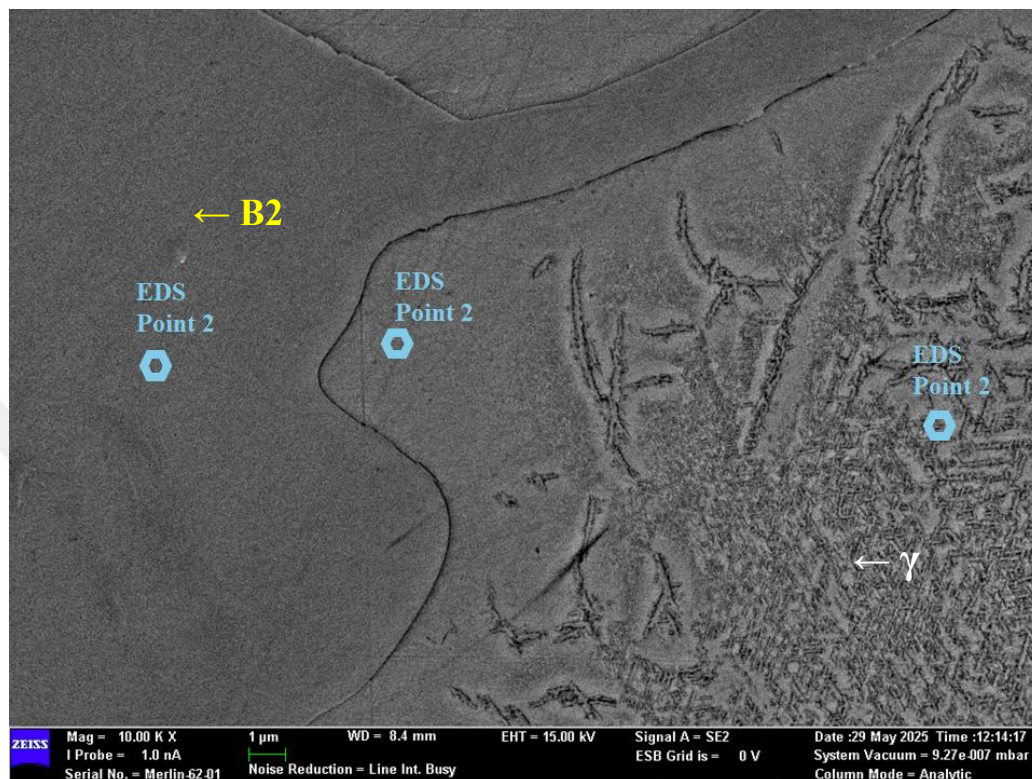


Figure 4.15 As-cast SEM image and EDS point analysis at austenite matrix and B2 phase (etched with %5 Nital, 10,000X)

The results of EDS analysis performed at the points shown in Figure 4.15 are shown in Table 4.7.

Table 4.7 Results of EDS points 1, 2 and 3

Element (wt%)	Mn	Al	Ni	Fe
EDS Point 1	9.25	17.5	25.1	48.15
EDS Point 2	17.25	7.59	6.22	68.94
EDS Point 3	15.87	9.57	11.14	63.42

The EDS line analysis shown in Figure 4.16, performed across the austenitic matrix and B2 phase over 90 points, yielded a composition of 58.83 wt.% Fe, 14.16 wt.% Mn, 11.95 wt.% Al, and 15.06 wt.% Ni.

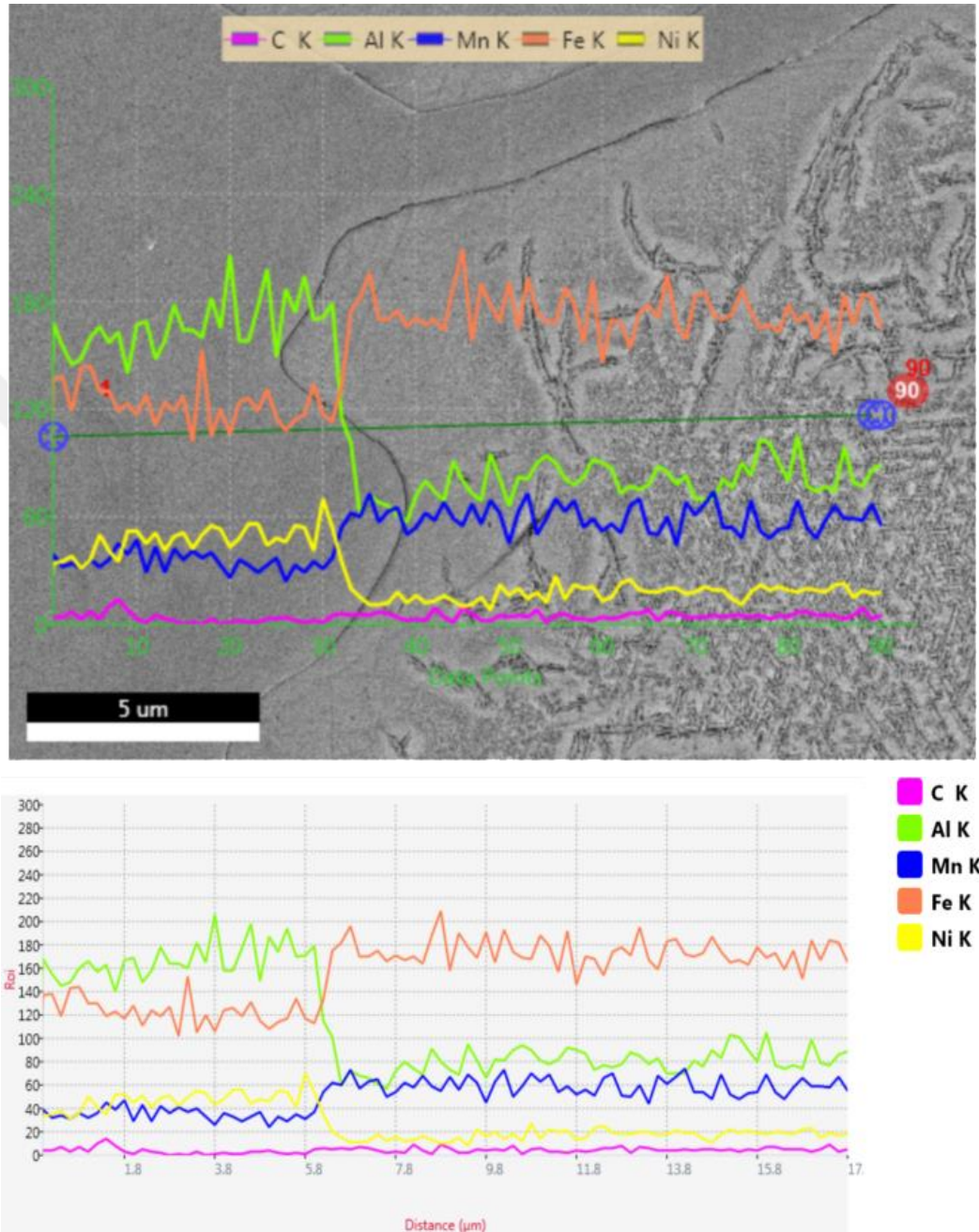


Figure 4.16 EDS line analysis for austenite matrix and B2 phase

4.5 EBSD Analysis

The phase maps obtained from the EBSD analyses conducted on the as-cast, ST-1150-2, and ST-1150-2 / A-700-8 samples are presented in Figure 4.17, 4.19, and 4.20, respectively. The EBSD analysis, performed at 250X magnification, revealed that the scanned area of the as-cast sample consists of approximately 91.9% austenite and 8.1% ferrite. Furthermore, the grain size distribution analysis indicated that 90% of the grains have a size of 7.6 μm , 6% have a size of 22.8 μm , and the average grain area was calculated to be 231 μm^2 .

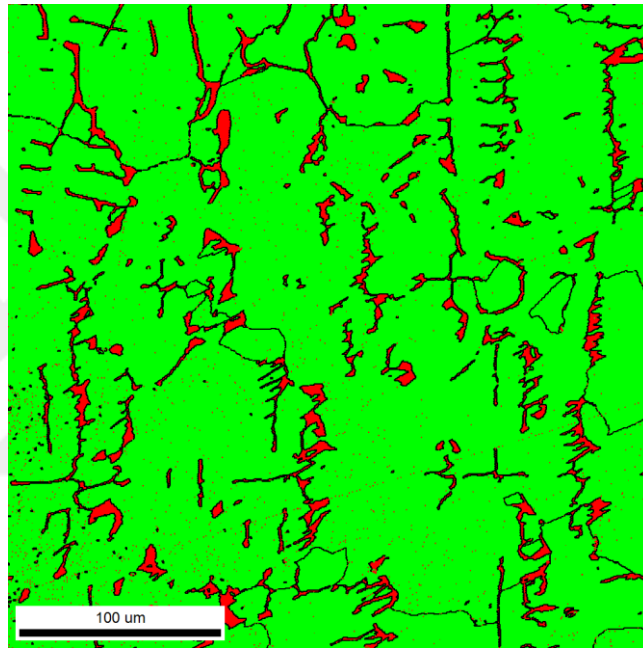


Figure 4.17 EBSD phase map of the as-cast sample. (Green areas indicate austenite, while red areas correspond to ferrite)

For the sample solutionized at 1150°C for 2 hours, EBSD analysis revealed that the scanned area of the sample consists of approximately 90% austenite and 10% ferrite (Figure 4.18). Also, analysis showed that 93% of the grain size distribution corresponds to 7.3 μm , 5% to 20.1 μm , and the average grain area was determined to be 87.8 μm^2 .

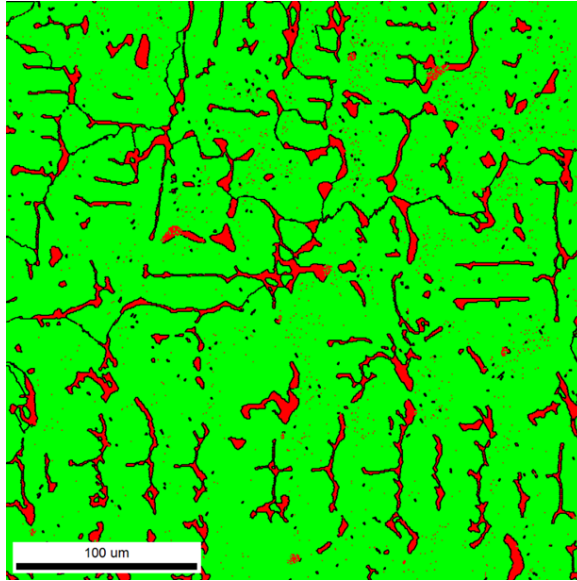


Figure 4.18 EBSD phase map of the sample solutionized at 1150°C for 2 h. (Green areas indicate austenite, while red areas correspond to ferrite)

For the sample solutionized at 1150°C for 2 hours and the aged at 700°C for 8 hours, EBSD analysis showed that the scanned area of the sample consists of approximately 89.5% austenite and 10.5% ferrite (Figure 4.19). Also, analysis showed that 99% of the grain size distribution corresponds to 9.03 μm , 0.1% to 25.2 μm , and the average grain area was determined to be 36.2 μm^2 .

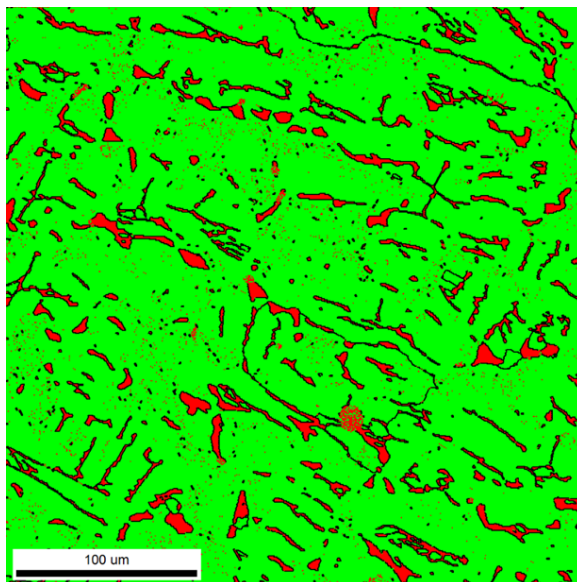


Figure 4.19 EBSD phase map of the solutionized and aged sample. (Green areas indicate austenite, while red areas correspond to ferrite)

4.6 ImageJ Phase Analysis

Following a 2-hour solution treatment at 1150°C and subsequent aging at 700°C, phase analyses were performed using ImageJ image processing software [34]. Phase ratios were determined from the microstructure images obtained using this software, and the relevant phase distribution and microstructure images are presented below. In the ImageJ analyses, areas shown in white represent the matrix, or austenite, while areas shown in black represent the B2 phase.

It was found that the austenite ratio in the sample aged at 700°C for 1 hour after solution treatment at 1150°C for 2 hours was 57.6% and the B2 phase ratio was 42.4% (Figure 4.20).

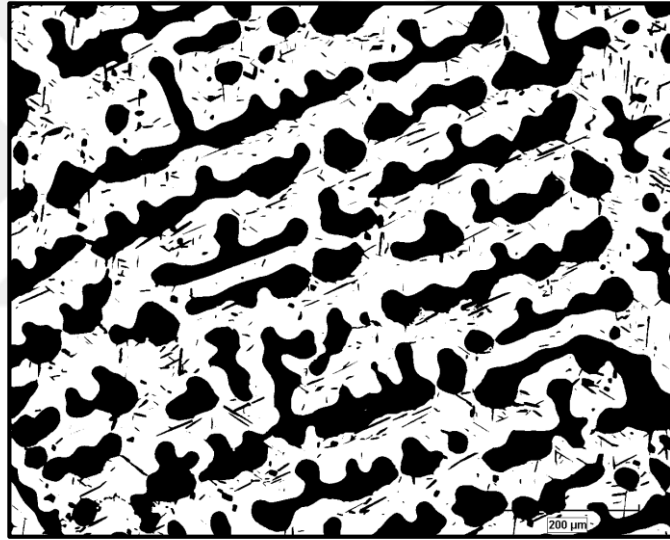


Figure 4.20 ImageJ phase analysis image of the sample ST-1150 / A-700-1

It was found that the austenite ratio in the sample aged at 700°C for 4 hours after solution treatment at 1150°C for 2 hours was 49.4% and the B2 phase ratio was 50.6% (Figure 4.21).

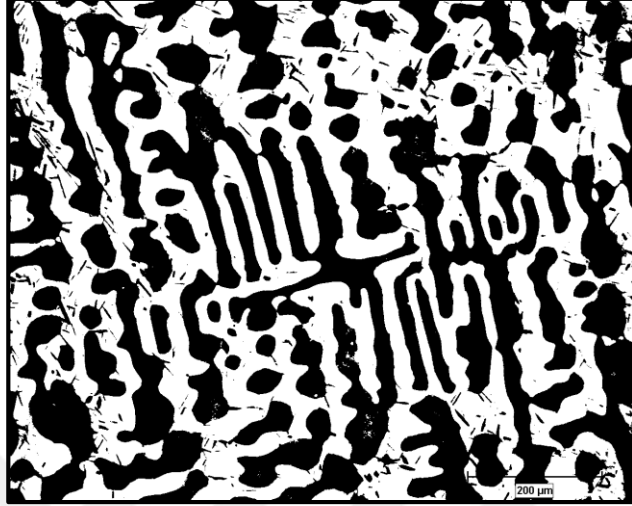


Figure 4.21 ImageJ phase analysis image of the sample ST-1150 / A-700-4

In the sample aged for 8 hours at 700°C after 2 hours of solution treatment at 1150°C, the austenite ratio was found to be 83.3% and the B2 phase ratio was found to be 16.7% (Figure 4.22). The closeness of the obtained value to the EBSD analysis increases the accuracy of the analysis.

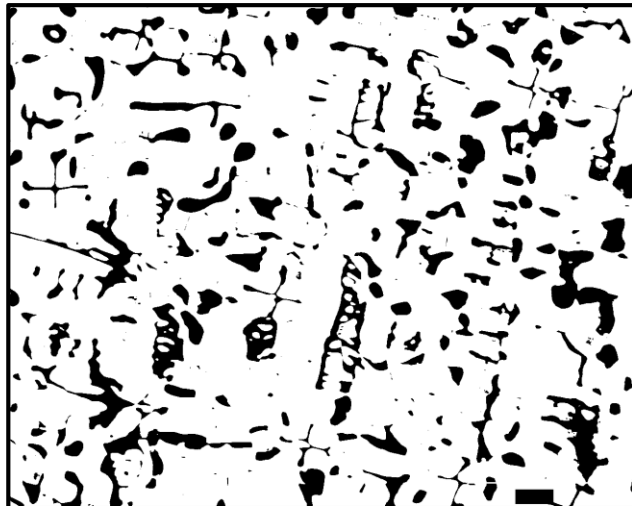


Figure 4.22 ImageJ phase analysis image of the sample ST-1150 / A-700-8

After solution treatment at 1150°C for 2 hours and aging at 700°C for 8 hours, the austenite ratio in the air-cooled sample was found to be 57.8% and the B2 phase ratio was found to be 42.2% (Figure 4.23).



Figure 4.23 ImageJ phase analysis image of the sample ST-1150 / A-700-8 (Air cooled)

It was found that the austenite ratio in the sample aged at 700°C for 16 hours after solution treatment at 1150°C for 2 hours was 29.4% and the B2 phase ratio was 70.6%. (Figure 4.24)



Figure 4.24 ImageJ phase analysis image of the sample ST-1150 / A-700-16

4.7 XRD Analysis

XRD analyses conducted to observe the phases present in the material in the as-cast, solutionized, and aged conditions are presented below.

4.7.1 XRD Pattern of the As-cast Sample

The XRD analysis performed on the as-cast sample is presented in Figure 4.25. In the analysis, diffraction peaks corresponding to the austenite (111), (200), (220), and (311) planes were detected, with the (200) peak exhibiting the highest intensity among them. In addition to these peaks, the structure also exhibited peaks corresponding to the B2 (110) and κ -carbide (200) planes.

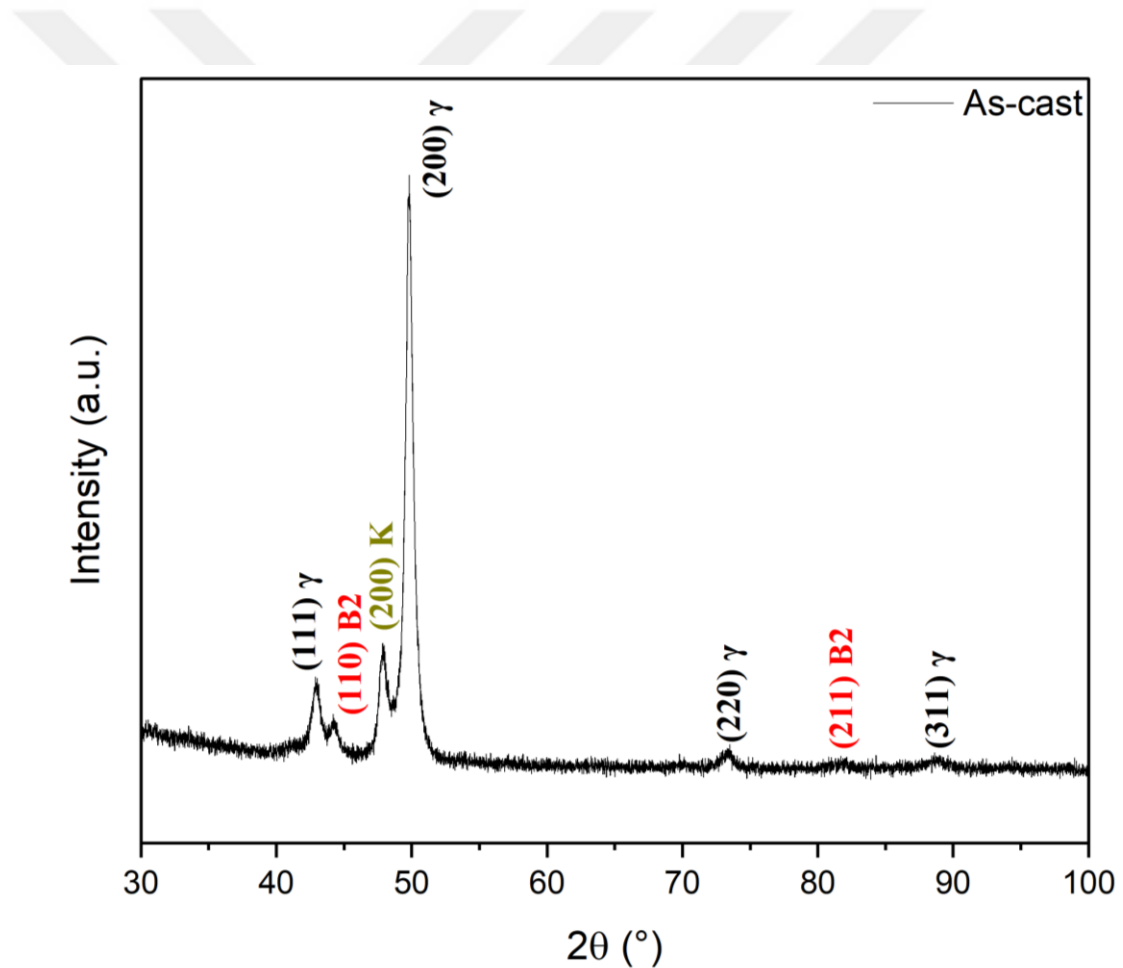


Figure 4.25 XRD pattern of the as-cast sample

4.7.2 XRD of Solutionized Sample

The XRD analysis shown in Figure 4.26, performed after solutionizing at 1150°C for 2 hours, revealed peaks corresponding to the austenite (111), (200), (220), and (311) planes. In addition to the austenite peaks, B2 phase peaks were observed at the (110), (200), and (211) planes, with the (110) peak showing the highest intensity. No κ -carbide peak was detected in the XRD analysis following the solutionizing treatment at 1150°C for 2 hours.

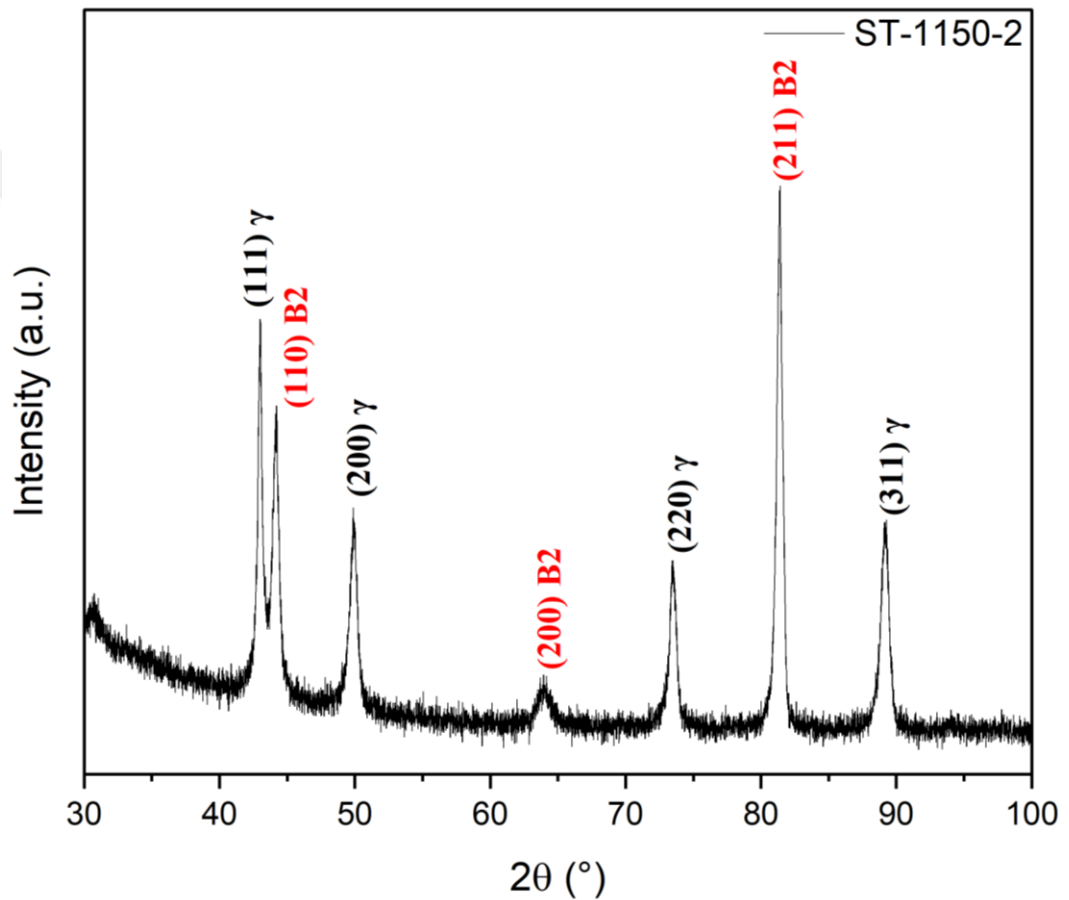


Figure 4.26 XRD pattern of the sample solutionized at 1150°C for 2h

4.7.3 XRD of Aged Sample

In the analysis shown in Figure 4.27 performed on the sample solutionized at 850°C for 2 hours and subsequently aged at 700°C for 8 hours, diffraction peaks corresponding to the (111), (200), (220), and (311) planes of austenite were detected. Additionally, peaks were detected from the B2 phase at the (110), (200), and (211) planes, as well as from the κ -carbide phase at the (110) and (311) planes.

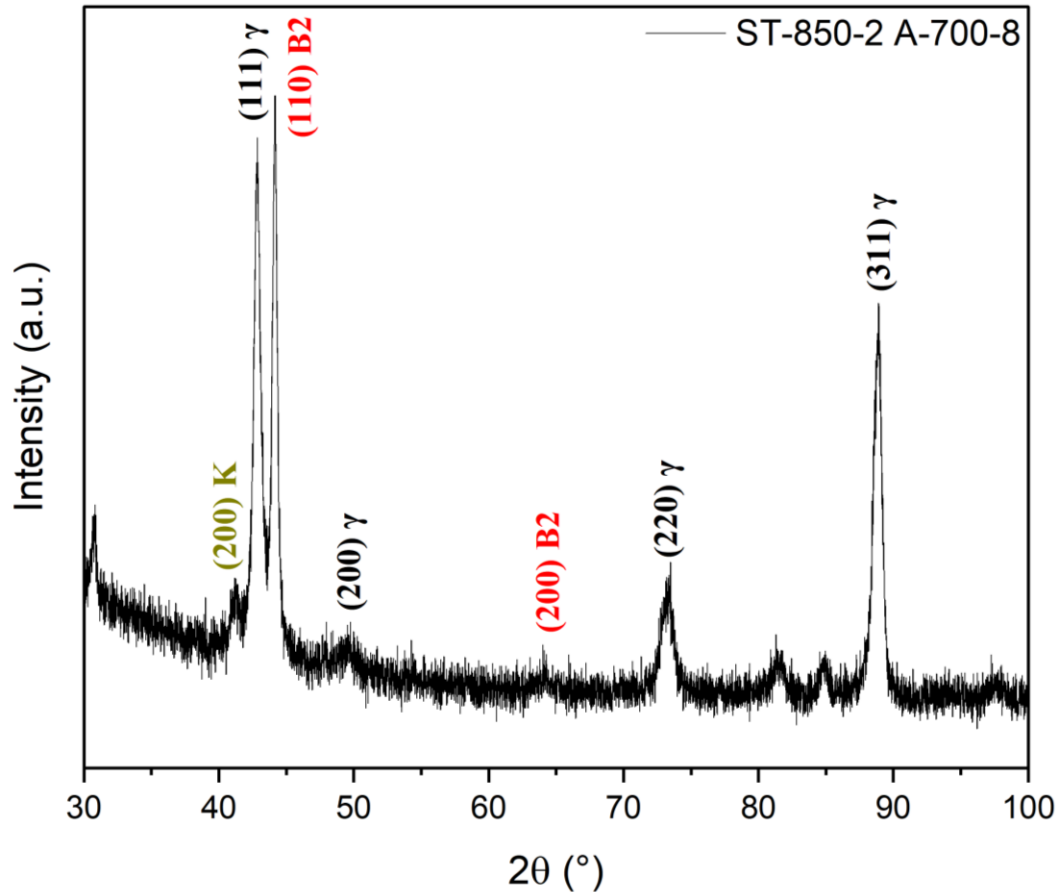


Figure 4.27 XRD patterns of the samples aged at 700°C for 8h after being solutionized at 850°C for 4h

In the analysis shown in Figure 4.28 conducted on the sample solutionized at 950°C for 16 hours and subsequently aged at 550°C for 16 hours followed by air cooling, four diffraction peaks corresponding to the austenite phase were observed. These peaks belong to the (111), (200), (220), and (311) planes of austenite. In addition to the austenite, diffraction peaks from the B2 phase were also detected, corresponding to the (110), (200), and (211) planes.

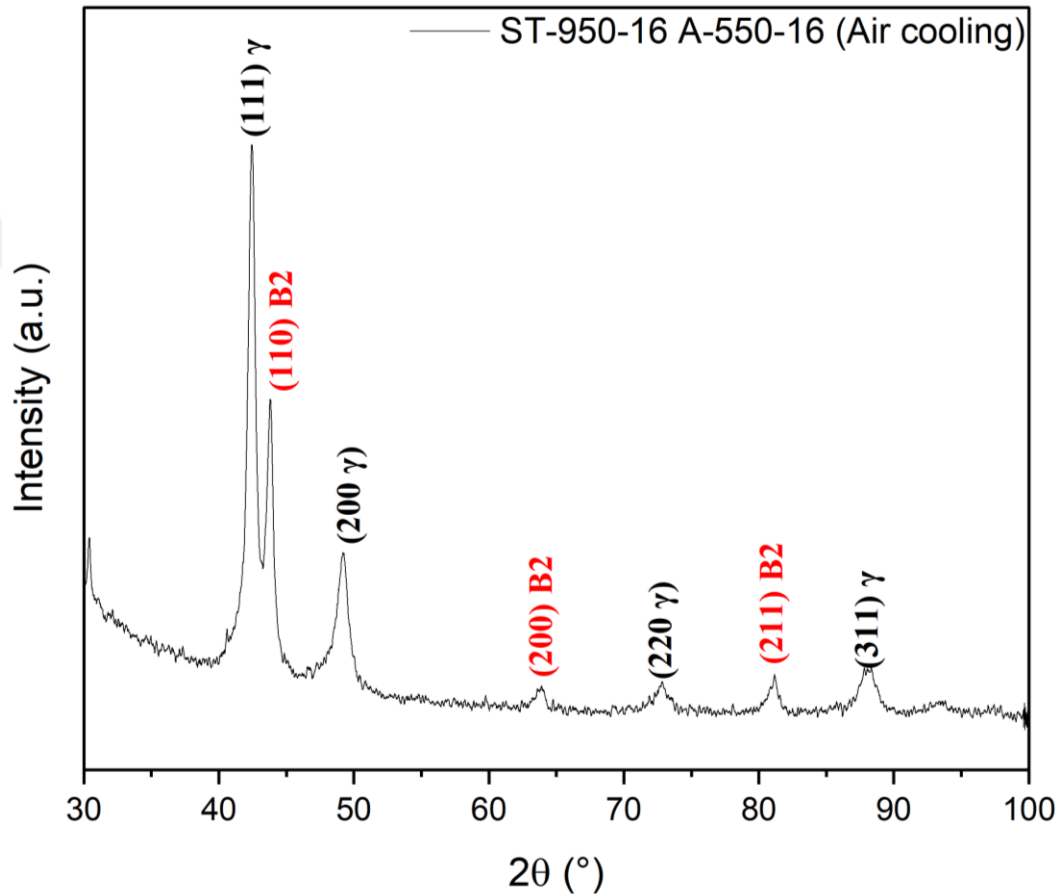


Figure 4.28 XRD pattern of the sample, solutionized at 950 for 16h and aged 550°C for 16h

Figure 4.29 presents the XRD analysis of two samples with different solutionizing parameters under the same aging conditions (700°C for 8 hours). In both conditions, diffraction peaks corresponding to the austenite (111), (200), (220), and (311) planes were observed. Similarly, peaks from the B2 (110) plane were detected in both samples. Additionally, the sample solutionized at 1050°C for 2h showed a B2 peak at the (211) plane, while the sample solutionized at 1050°C for 4h exhibited a B2 peak at the (220) plane. Distinctively, in the sample solutionized at 1150°C for 2h, peaks corresponding to the κ -carbide (111) and (200) planes were observed.

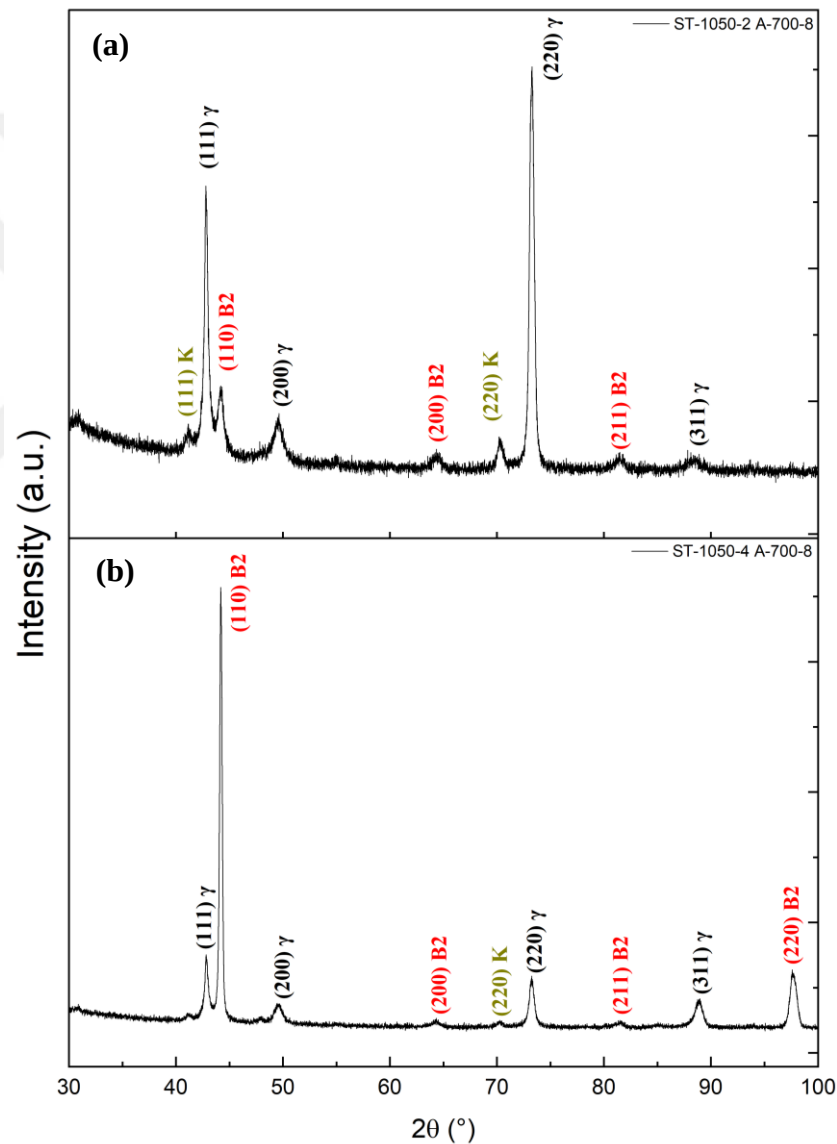


Figure 4.29 XRD patterns of the samples, (a) solutionized at 1050 for 2h and aged 700°C for 8h (b) solutionized at 1050 for 4h and aged 700°C for 8h

Figure 4.30 shows the XRD analysis of samples that were solutionized at 1150°C for 2 and 4 hours and subsequently aged at 700°C for 8 hours. In both conditions, austenite peaks were observed at 2θ angles corresponding to the (111), (200), (220), and (311) planes. For both conditions, B2 phase peaks were detected at the (110), (200), and (211) planes, while an additional peak from the B2 (220) plane was observed in the sample solutionized at 1050°C.

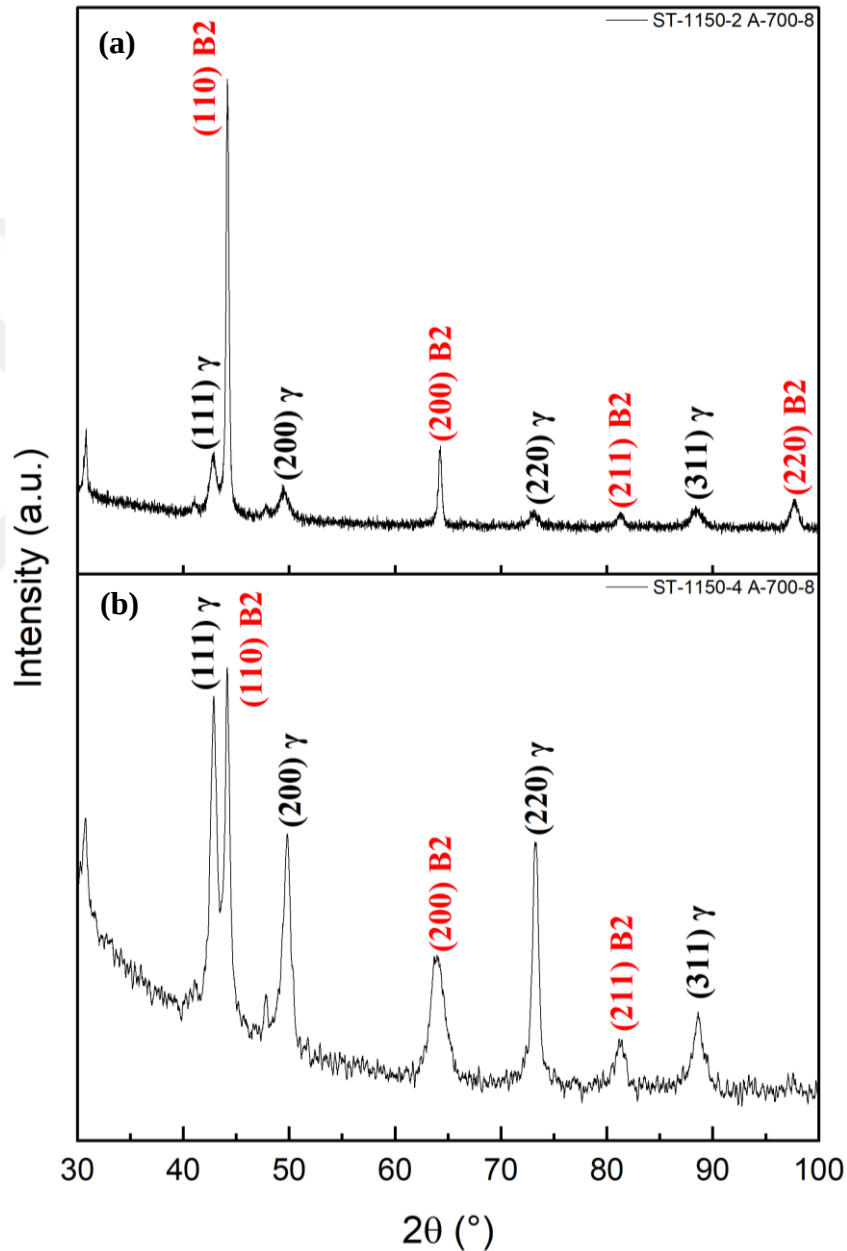


Figure 4.30 XRD patterns of the samples, (a) solutionized at 1150 for 2h and aged 700°C for 8h (b) solutionized at 1150 for 4h and aged 700°C for 8h

Figure 4.31 presents the XRD analyses of samples aged at 700°C for 1, 4, 8, 8 air-cooled and 16 hours under fixed solutionizing conditions of 1150°C for 2 hours. To better understand the effect of aging duration, the analysis result for 8 hours of aging was included again. In all conditions, austenite peaks at (110), (200), and (220) planes were observed, while the (311) peak was not detected after 1 hour of aging. The B2 phase showed common peaks at the (110) and (211) planes across all conditions; however, the (200) peak was absent only in the 4-hour aging condition, and the (220) peak appeared exclusively after 8 hours of aging. No κ -carbide peaks were detected in the analysis.

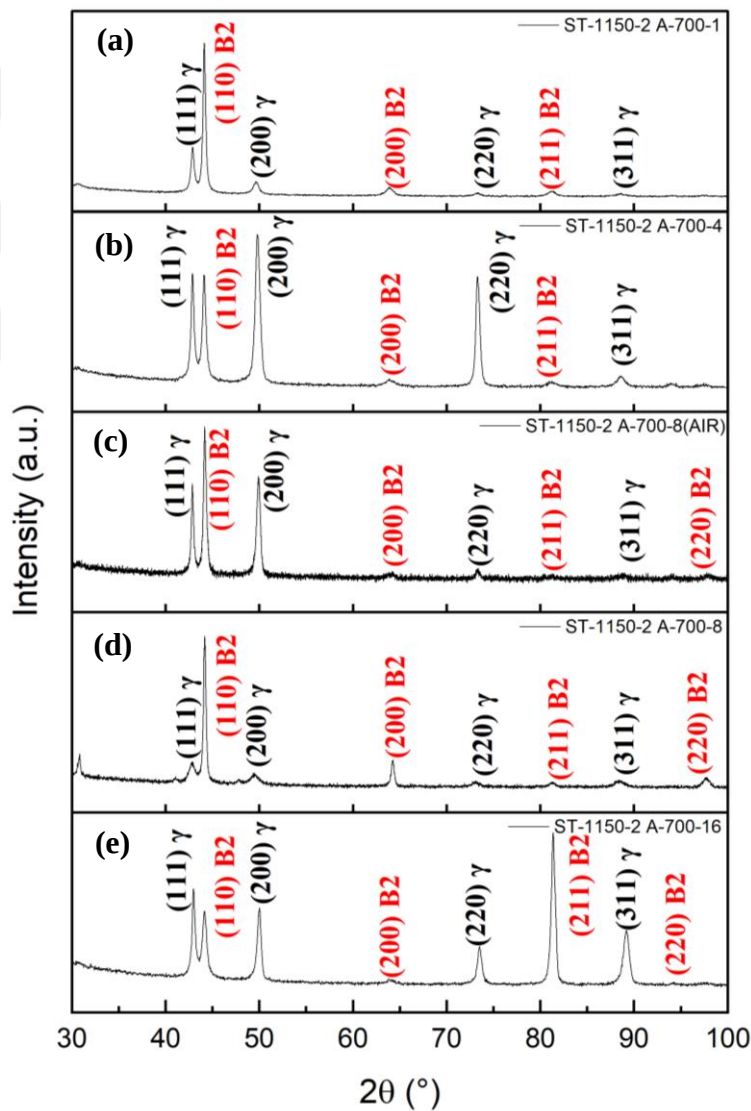


Figure 4.31 XRD pattern of the samples, solutionized at 1150 for 2h and aged at 700°C for (a) 1h, (b) 4h, (c) 8h (air cooled), (d) 8h and (e) 16h

Figure 4.32 shows the XRD analysis of the samples aged at 550°C for 1, 4, 8, and 16 hours following solutionizing at 1150°C for 2 hours. The analyses revealed that the austenite (111) and (200) peaks were present in all samples, while the (220) peak appeared only in the 1-hour and 8-hour aging conditions, the (311) peak was observed in all conditions except for the 8-hour aging, and the (222) peak was detected only in the 4-hour aging condition. The B2 (110) peak was present in all samples, whereas the B2 (200) peak was detected only in the sample aged for 16 hours. No peaks corresponding to κ -carbide were observed in any of the analyses.

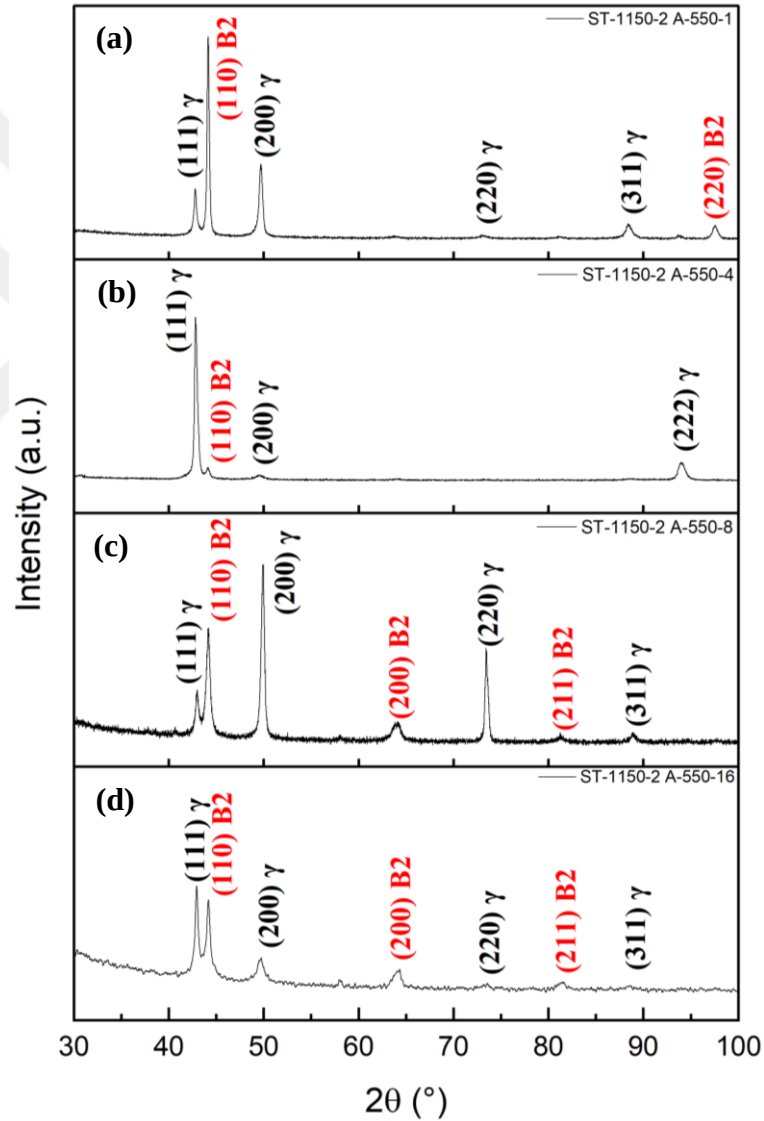


Figure 4.32 XRD patterns of the samples solutionized at 1150 for 2h and aged at 550°C for (a) 1h, (b) 4h, (c) 8h, (d) 16h

Figure 4.33 shows the XRD analyses of samples that were solution treated at 1150°C for 2 hours and then aged at 550°C for 8 and 16 hours followed by cooling in air. In both conditions, austenite peaks were observed at 2θ angles corresponding to the (111), (200), (220), and (311) planes. For both conditions, B2 phase peaks were detected at the (110), and (211) planes, while additional peaks from the austenite (220) plane and B2 (220) plane were observed in the sample aged at 550°C for 8 hours.

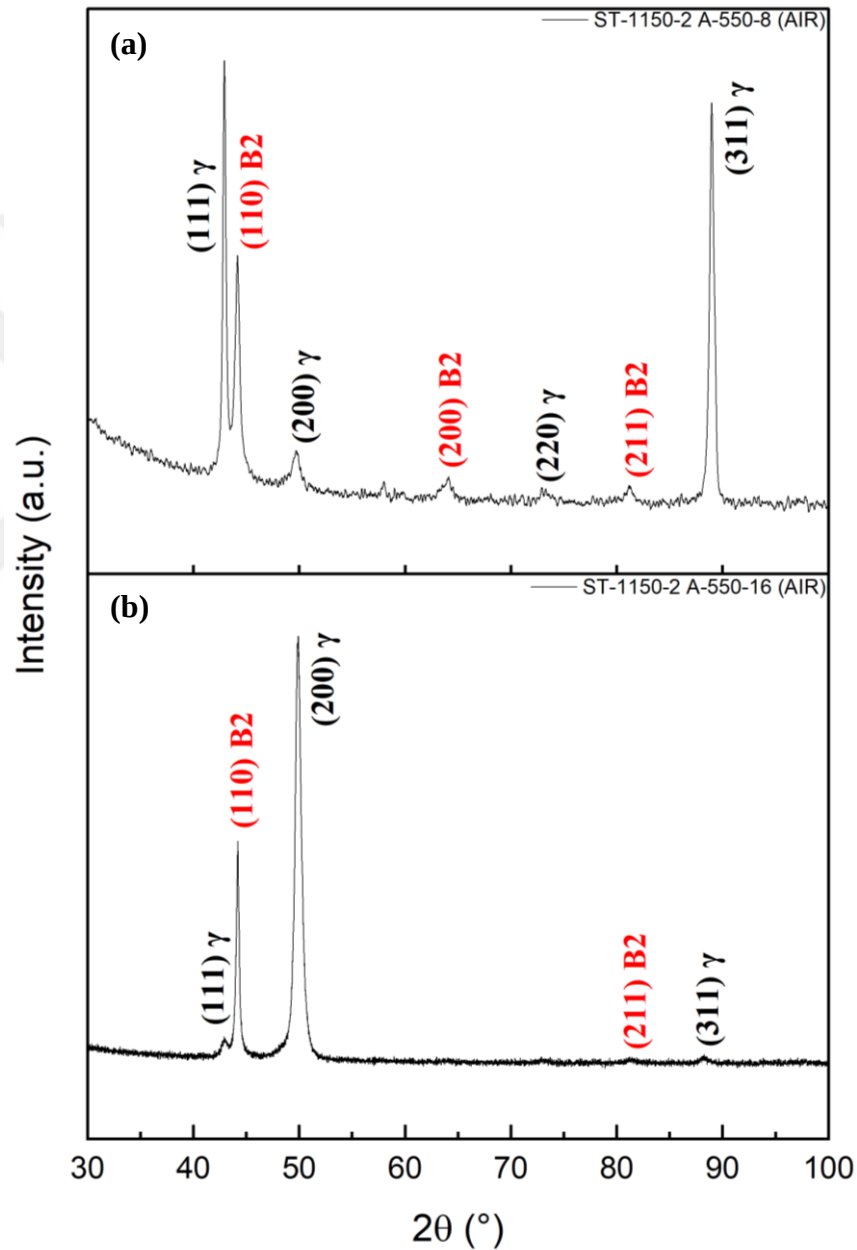


Figure 4.33 XRD pattern of the samples, (a) aged 550°C for 8h and air cooled, (b) aged 550°C for 16h and air cooled

4.8 Wear Test Results and Examination of the Worn Samples

The wear tests of the samples were conducted using the parameters described in Section 3.6.5, and the wear rates were calculated using the formula provided in the same section. The wear rate results for all the tests performed are shown in Table 4.8.

Table 4.8 Wear rate results of the samples

Heat Treatments	Samples	Wear Rate ($10^{-7} \cdot \text{g/Nm}$)
-	As-cast	5.92
-	AISI 4140	12.80
ST	ST-950-16	7.92
ST	ST-1050-2	6.35
ST	ST-1050-4	7.60
ST	ST-1150-2	9.57
ST+AGED	ST-850-2 / A-700-8	6.60
ST+AGED	ST-850-4 / A-700-8	3.62
ST+AGED	ST-1050-2 / A-700-8	8.42
ST+AGED	ST-1050-4 / A-700-8	7.75
ST+AGED	ST-1150-4 / A-700-8	4.58
ST+AGED	ST-1150-2 / A-700-1	6.28
ST+AGED	ST-1150-2 / A-700-4	7.90
ST+AGED	ST-1150-2 / A-700-8	5.65
ST+AGED (AIR COOLED)	ST-1150-2 / A-700-8	7.00
ST+AGED	ST-1150-2 / A-700-16	6.52
ST+AGED	ST-1150-2 / A-550-1	10.70
ST+AGED	ST-1150-2 / A-550-4	12.85
ST+AGED	ST-1150-2 / A-550-8	9.12
ST+AGED (AIR COOLED)	ST-1150-2 / A-550-8	7.30
ST+AGED	ST-1150-2 / A-550-16	10.11
ST+AGED (AIR COOLED)	ST-1150-2 / A-550-16	7.20
ST+AGED	ST-1150-2 / A-400-168	6.15

The wear rate values for the as-cast sample and those subjected to different solutionizing parameters are presented in the figure. Additionally, to compare wear test results with different types of steel, AISI 4140 steel sample with a hardness of 385.6 HB was subjected to wear tests with the same test parameters as shown in Figure 4.34.

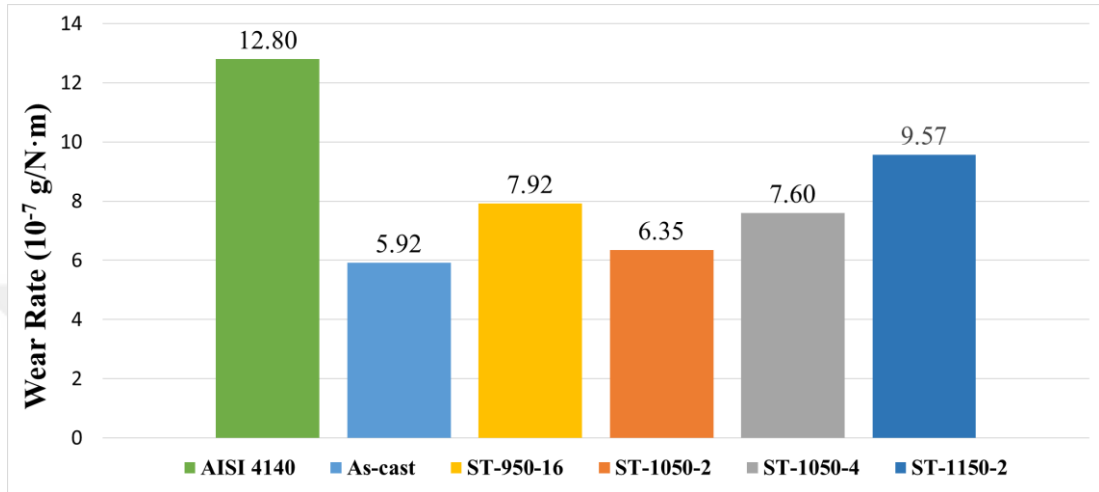


Figure 4.34 Wear rates of the AISI 4140, as-cast, and solutionized samples

As shown in Figure 4.34, the wear rate for the as-cast sample was measured to be $5.92 \times 10^{-7} \text{ g/N}\cdot\text{m}$, while the sample solutionized at 950°C for 16 hours exhibited a wear rate of $7.92 \times 10^{-7} \text{ g/N}\cdot\text{m}$. For the samples solutionized at 1050°C for 2 and 4 hours, the wear rates were calculated as $6.35 \times 10^{-7} \text{ g/N}\cdot\text{m}$ and $7.60 \times 10^{-7} \text{ g/N}\cdot\text{m}$, respectively. For another solutionizing condition, which was set as 1150°C for 2 hours prior to aging, the wear rate was calculated as $9.57 \times 10^{-7} \text{ g/N}\cdot\text{m}$. The wear rate of 4140 steel sample was calculated $12.80 \times 10^{-7} \text{ g/N}\cdot\text{m}$.

The results of the wear tests conducted for different solutionizing temperatures and aging treatments in the first stage of the project are presented in Figure 4.35. For samples aged at 700°C for 8 hours, the wear rate was measured as 6.60×10^{-7} g/N·m for the condition solutionized at 850°C for 2 hours. When the solutionizing time for the same temperature was increased to 4 hours, the wear rate decreased to 3.62×10^{-7} g/N·m. Similarly, for the samples solutionized 1050°C for 2 hours, the wear rate was 8.42×10^{-7} g/N·m, and it decreased to 7.75×10^{-7} g/N·m for 4 hours of solutionizing. In addition, for the condition solutionized at 1150°C for 2 hours, the wear rate was 5.65×10^{-7} g/N·m, and similarly it decreased to 4.58×10^{-7} g/N·m when the duration was extended to 4 hours.

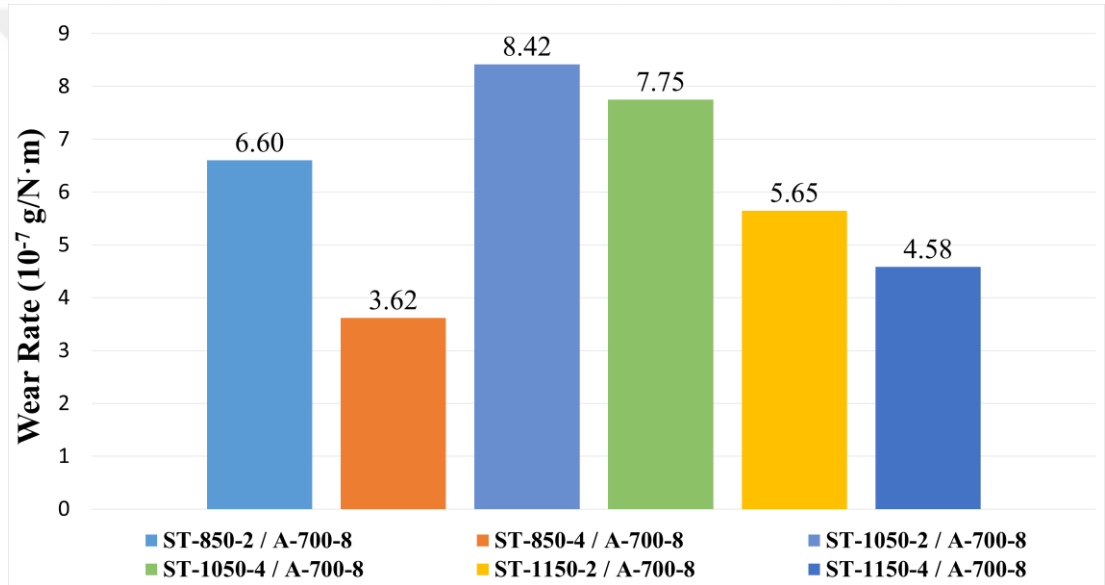


Figure 4.35 Wear rates of samples with different solutionizing parameters

The wear test results for samples aged at different durations and cooling methods at 700°C, following the fixed solutionizing parameter of 1150°C for 2 hours — representing the first step of the second stage of the project— are presented in Figure 4.36. The wear rate of the sample aged for 1 hour at 700°C was measured as 6.28×10^{-7} g/N·m, while the result for 4 hours of aging was calculated as 7.90×10^{-7} g/N·m. When the aging time was increased to 8 hours, the wear rate in the quenched sample was calculated as 5.65×10^{-7} g/N·m and in the air-cooled sample as 7.00×10^{-7} g/N·m. For the longest aging duration, the wear rate was determined to be 6.52×10^{-7} g/N·m.

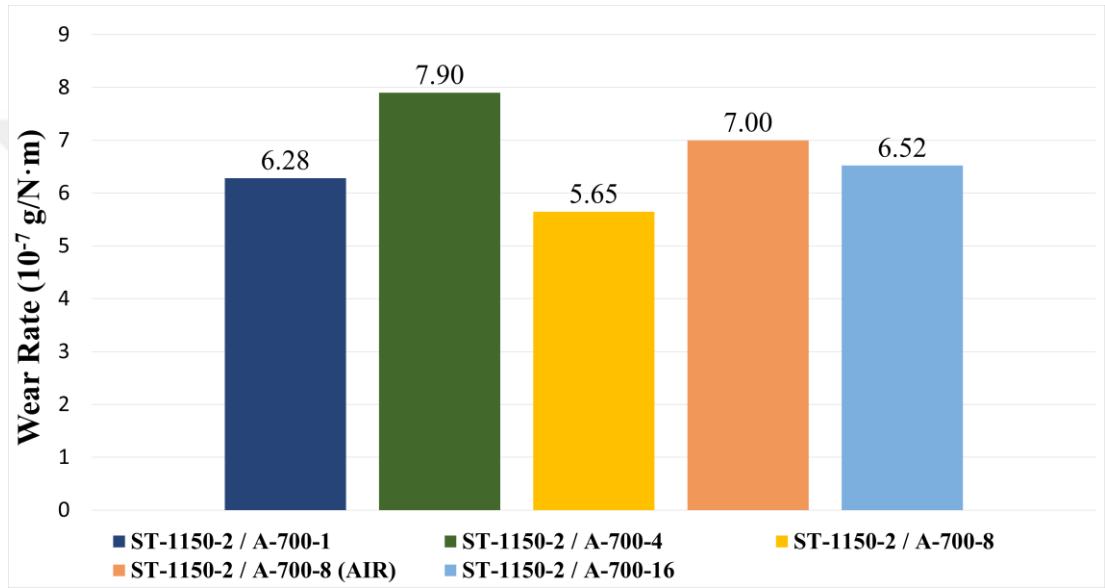


Figure 4.36 Wear rates of the samples solutionized at 1150 for 2h and aged at 700°C for 1h, 4h, 8h, 8h (Air cooled), 16h

The wear test results for samples aged at different durations at 550°C, following the fixed solutionizing condition of 1150°C for 2 hours representing the second step of the second stage of the project are presented in Figure 4.37. In the tests, the sample aged for 1 hour at 550°C exhibited a wear rate of 10.70×10^{-7} g/N·m, while the result for 4 hours of aging was calculated as 12.85×10^{-7} g/N·m. When the aging duration was doubled to 8 hours, the wear rate in the quenched sample was determined to be 9.12×10^{-7} g/N·m, in the air-cooled sample as 7.30×10^{-7} g/N·m. The longest aging duration of 16 hours, the wear rate was calculated as 10.11×10^{-7} g/N·m, and for the air-cooled sample, it was determined 7.20×10^{-7} g/N·m.

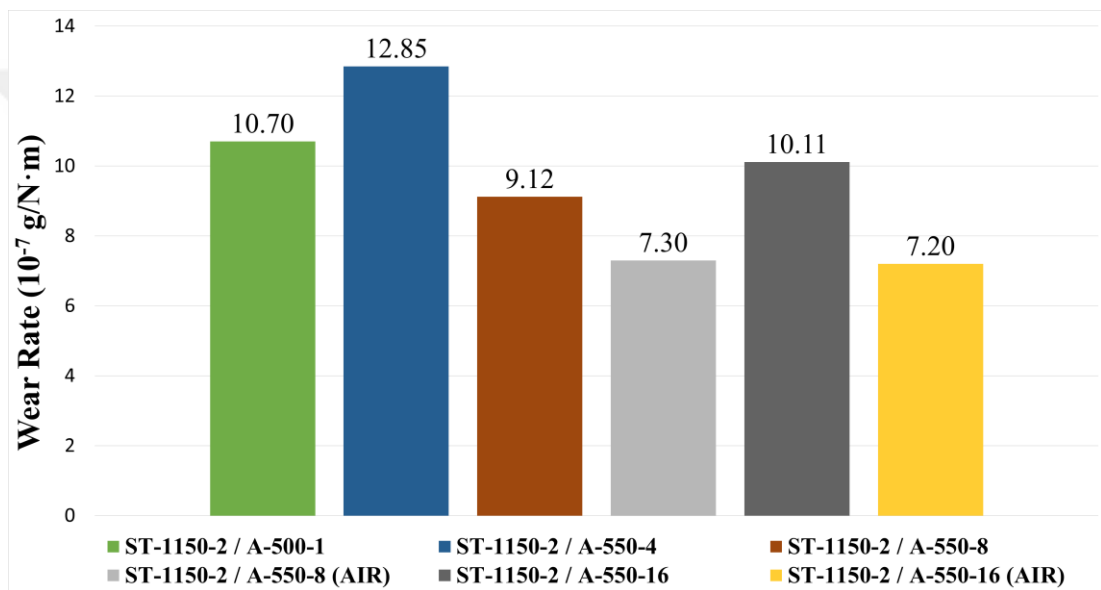


Figure 4.37 Wear rates of samples, solutionized at 1150 for 2h and aged at 550°C for 1h, 4h, 8h, 8h (Air cooled), 16h and 16h (Air cooled)

Among all tests, the highest wear rate, 12.9×10^{-7} g/N·m, was observed in the sample with the ST-1150-2 / A-550-4 condition, while the lowest wear rate, 3.6×10^{-7} g/N·m, was recorded for the sample with the ST-850-4 / A-700-8 condition.

In addition to the wear tests, in order to evaluate the wear mechanisms of the material more comprehensively after the tests on as-cast, ST-1150-2, and ST-1150-2 / A-700-8 samples, the microstructure and surface morphology of the samples were examined in detail. In this context, the samples were subjected to microstructure analysis using SEM and 3D surface scans were performed with the Alicona InfiniteFocus device for surface topography examination. The results obtained are given below.

The surface analysis performed on the as-cast sample is shown in Figure 4.38. After the wear test, it was observed that the depth of the worn surface was 168.311 μm .

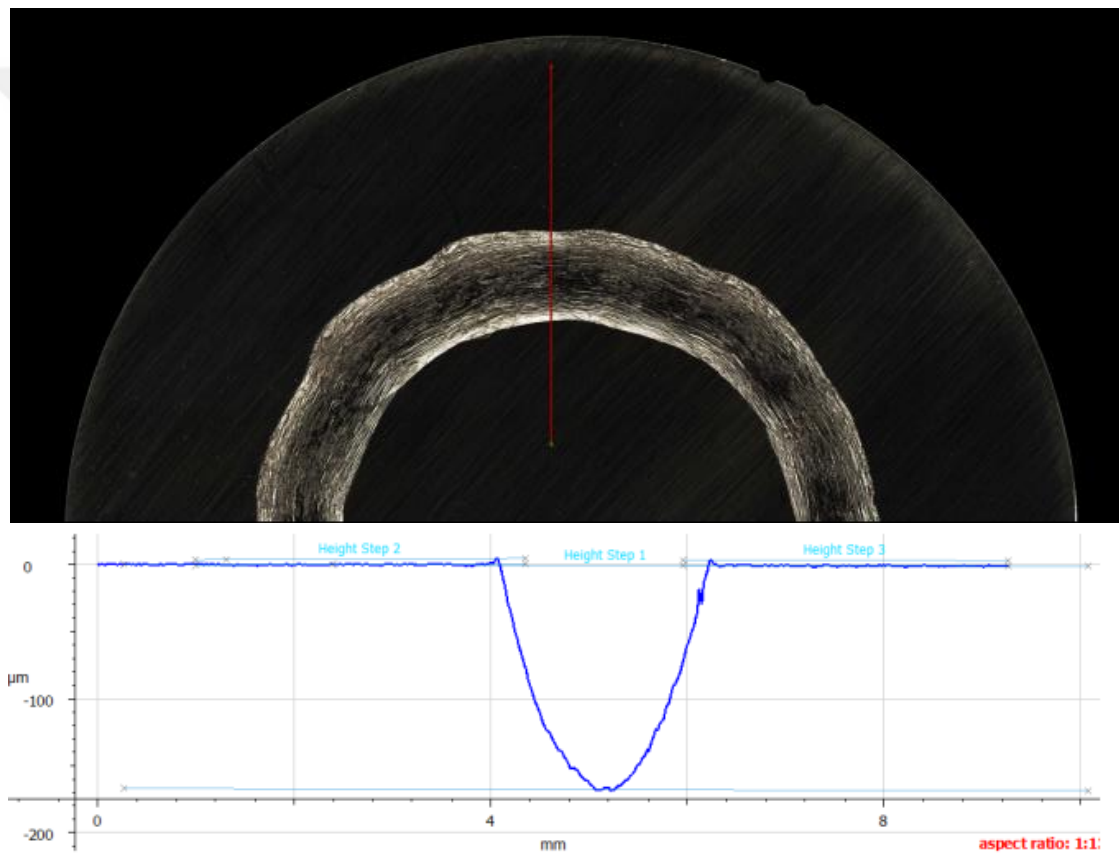


Figure 4.38 Surface analysis of the worn as-cast sample

The surface analysis performed on the sample taken into solution at 1150°C for 2 hours is shown in Figure 4.39. After the wear test, it was observed that the depth of subsurface deformation was 152.801 μm .

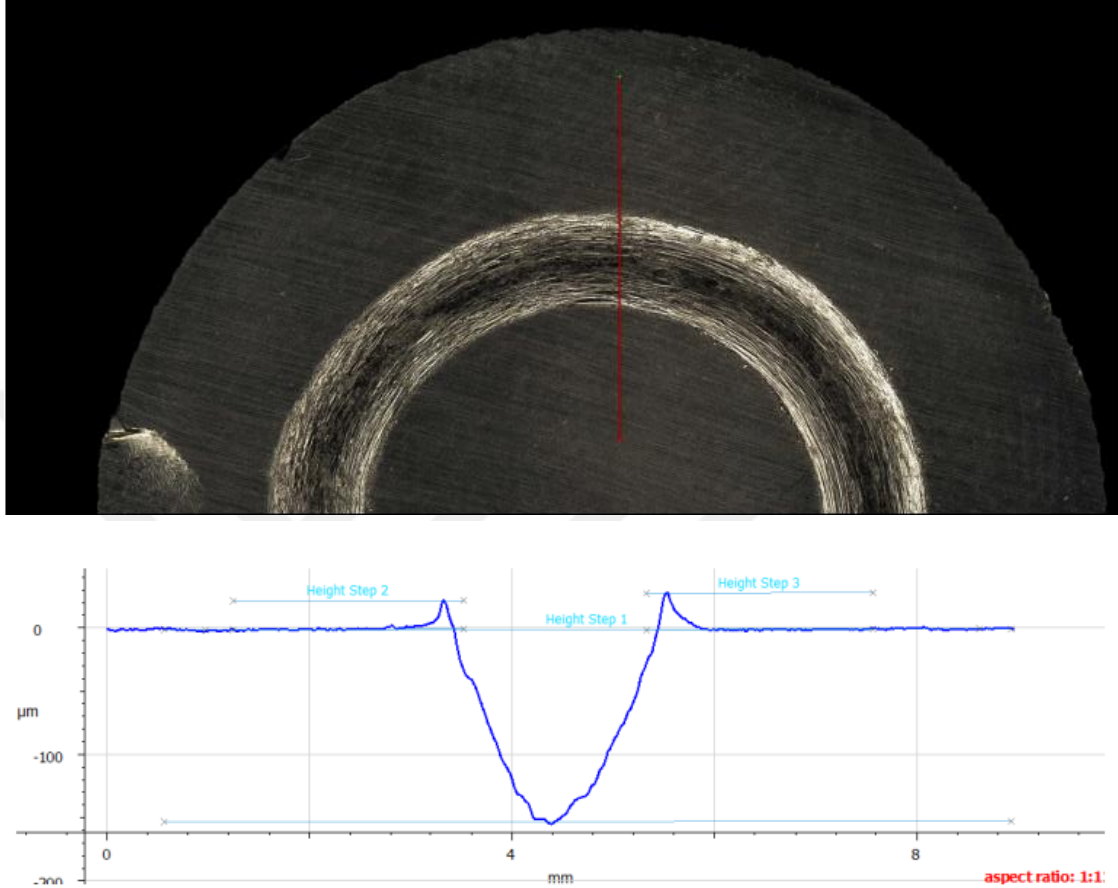


Figure 4.39 Surface analysis of the wear tested sample that solutionized at 1150°C for 2h

The surface analysis performed on the sample that was taken into solution at 1150°C for 2 hours and then aged at 700°C for 8 hours is shown in Figure 4.40. After the wear test, it was observed that the depth of the worn surface was 95.530 μm .

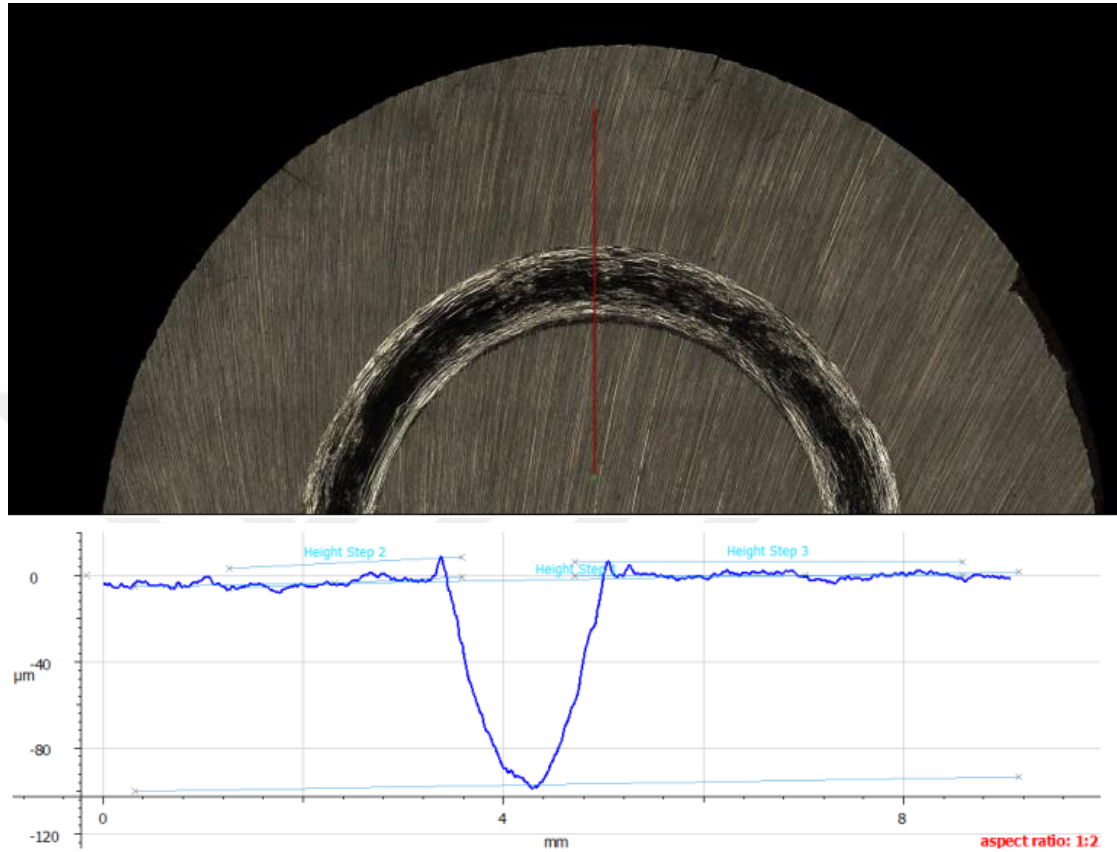


Figure 4.40 Surface analysis of the sample taken into solution at 1150°C for 2h and aged at 700°C for 8h

In the SEM analysis, the area to be analyzed was cut with an EDM device and then subjected to metallographic sample preparation. The cut area and the area within the cut area where the analysis was performed are shown in Figure 4.41.

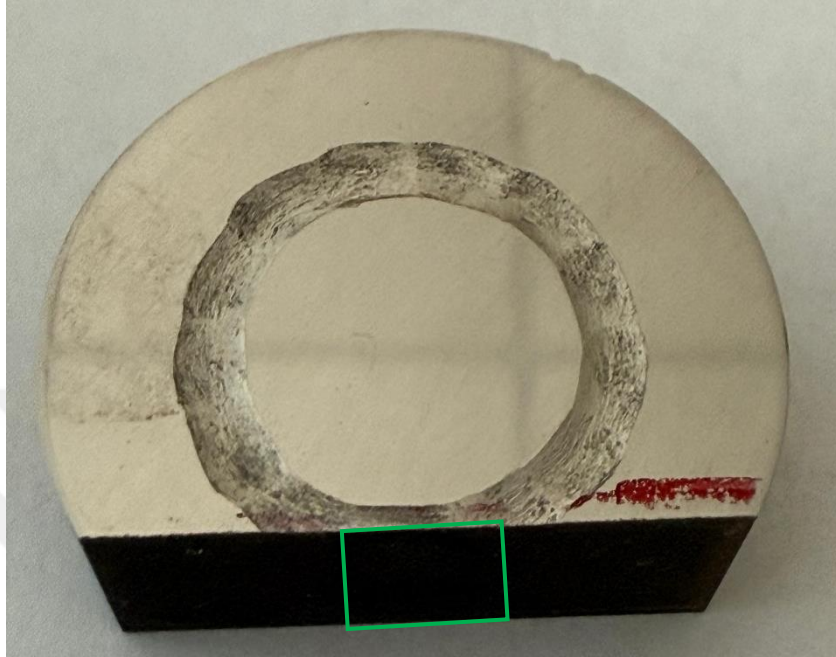


Figure 4.41 Sample used for SEM analysis and the area of analysis (Green area)

Additionally, the image in Figure 4.42 includes a schematic representation of the image expected to be seen in the SEM analysis.

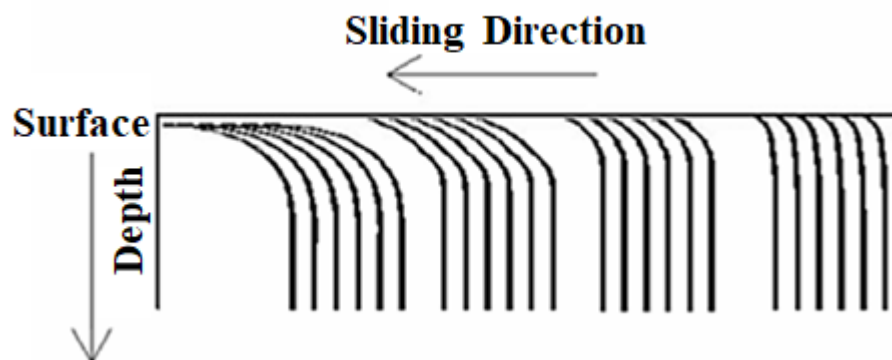


Figure 4.42 Schematic representation of the cross-sectional worn area

In the analysis shown in Figure 4.43, which was first performed on the as-cast sample, it is seen that the material flows from the surface to a depth of 16.52 μm .

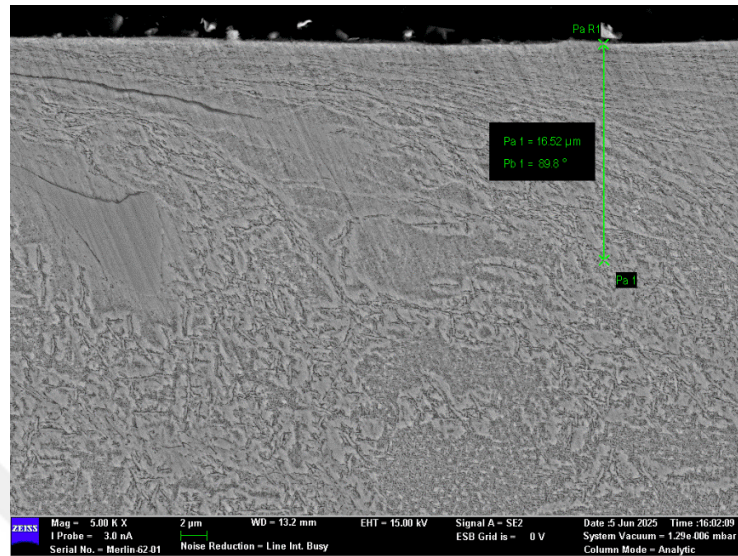


Figure 4.43 SEM analysis of the cross-sectional surface of as-cast worn sample (5% Nital etched, 5000X)

The SEM analysis image performed for the solution treatment at 1150°C for 2 hours is shown in Figure 4.44. In the analysis, it was observed that the material flowed from the surface to a depth of 32.59 μm .

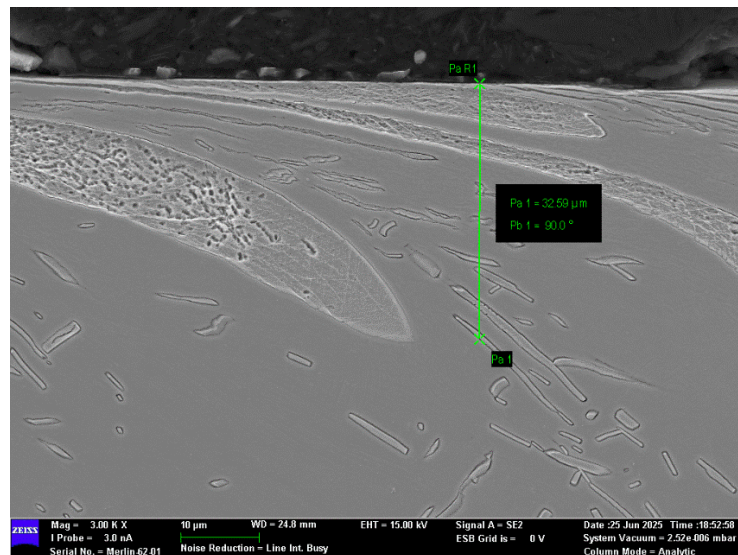


Figure 4.44 SEM analysis of the cross-sectional surface of solutionized worn sample (5% Nital etched, 3000X)

Figure 4.45 shows the SEM analysis image of the sample aged 8 hours at 700°C after 2 hours of solution treatment at 1150°C. In the analysis, it was observed that the material flowed from the surface to a depth of 46.27 μm .

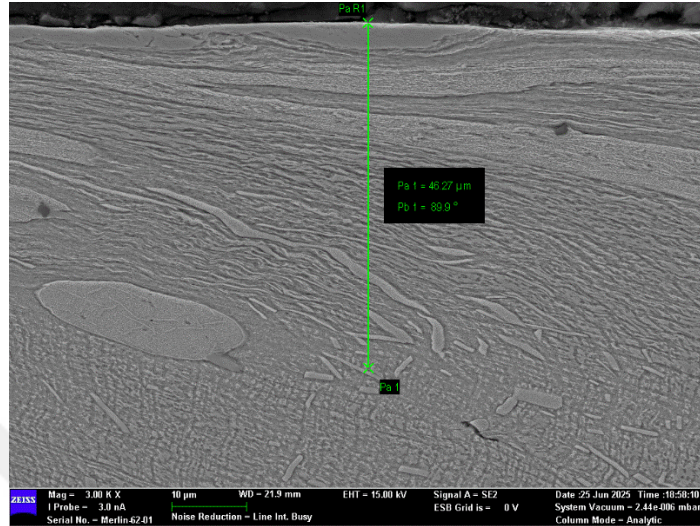


Figure 4.45 SEM analysis of the cross-sectional surface of solutionized and aged worn sample (5% Nital etched, 3000X)

Images of the zirconia ball, used as the counterface material in the wear tests and examined in detail with an optical microscope after all tests were completed, are presented in Figure 4.46. There was no wear of the counterface and some material transfer from the tested disk to the surface of the counterface was detected.

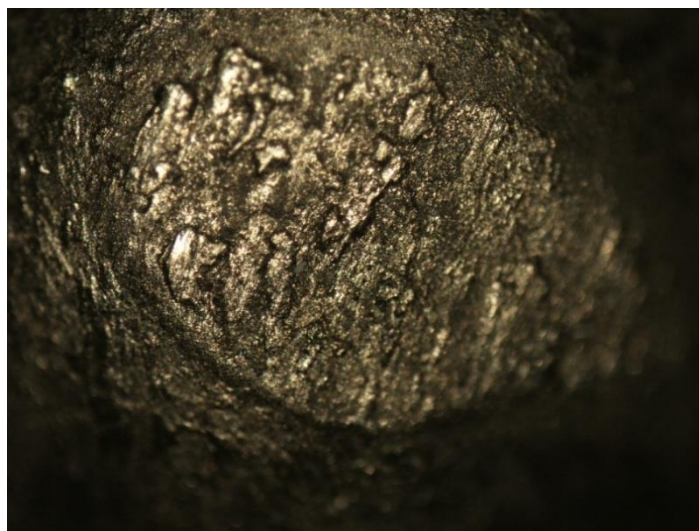


Figure 4.46 Image of contact surface of the zirconia ball used for wear tests (50X)

CHAPTER 5

DISCUSSION

5.1 Effect of Age Hardening Conditions

5.1.1 As-cast Condition

In the tests carried out on the as-cast sample at the beginning of the project, it was observed that the sample hardness was 477HB and in the XRD analysis, there were different phases during solidification and subsequent cooling. As seen in the XRD analysis in Figure 4.26, the κ -carbide phase is clearly observed in the sample, in addition to the expected austenite and B2 phases. It is known that in order for the κ -carbide phase to be clearly visible in the XRD analysis, it must be present in grain boundaries or as clusters in the matrix, not as nano-sized dispersions in the matrix. For this reason, it is thought that the κ -carbide peak is visible in the XRD analysis of the as-cast sample.

In the microstructure analysis performed with an optical microscope, shown in Figure 5.1, it was observed that the B2 phase was relatively large in size and heterogeneously distributed within the microstructure. In the EDS analysis performed on the same sample, the elemental differences for the austenite matrix and the B2 phase can be seen more clearly.

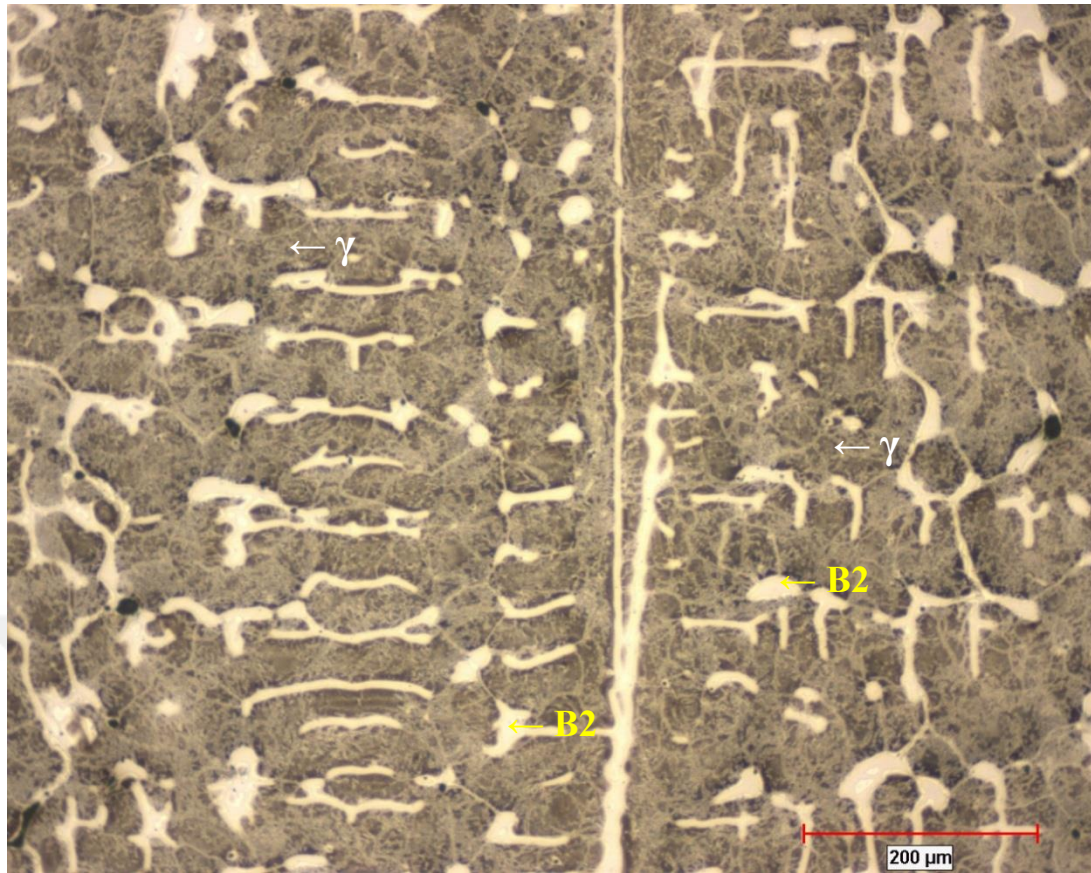


Figure 5.1 Optical microstructural images of as-cast sample 100X (5% Nital etched.)

As seen in Figure 5.2, EDS line analysis clearly shows that the Ni and Al ratios decrease drastically, and the Fe ratio increases as we move from the B2 phase to the matrix. The numerical values of this increase and decrease are shown in Table 5.1.

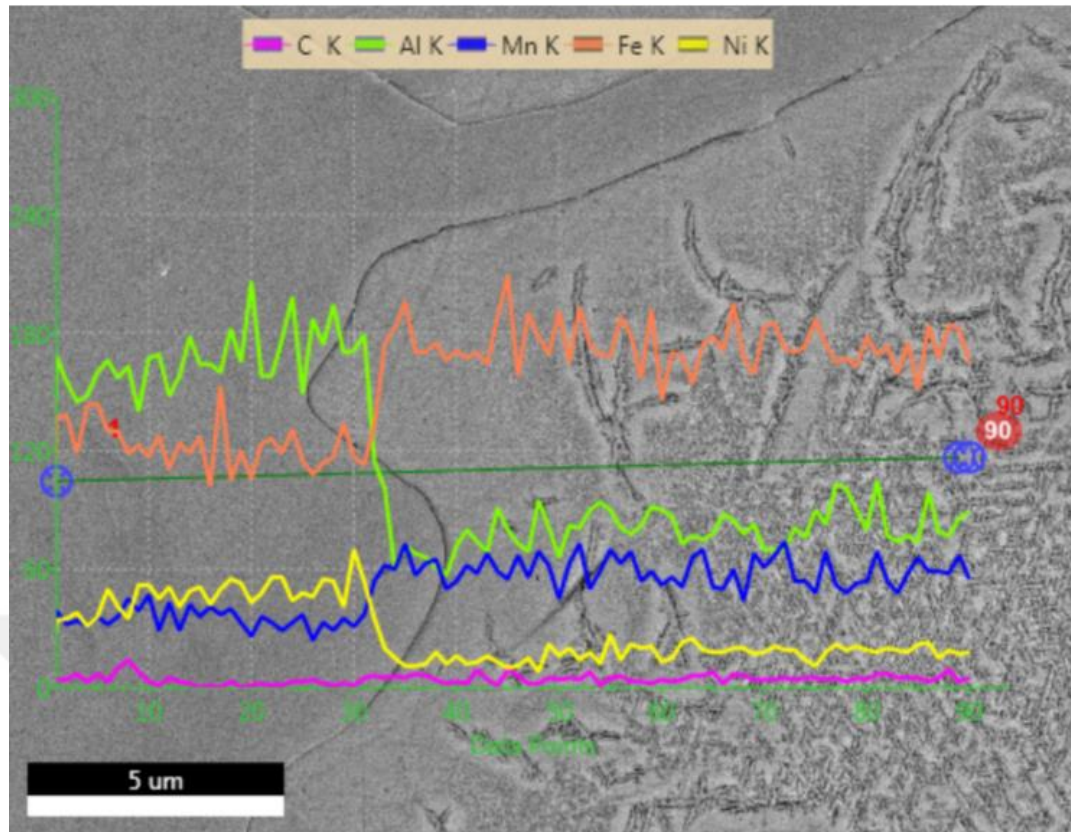


Figure 5.2 EDS line analysis of as-cast sample B2 to matrix

Table 5.1 Numerical values of EDS line analysis

Element (wt%)	Mn	Al	Ni	Fe
B2	9.25	17.5	25.1	48.15
Matrix	17.31	9.79	8.34	64.55

The homogenization of the microstructure and the development of hardness properties were aimed with the solutionizing (ST) and aging processes applied within the scope of the project. The experimental data obtained, and the evaluations made regarding these processes are given below.

5.1.2 Effect of Solutionizing

As stated under the Experimental chapter, 4 different temperatures (850°C, 950°C, 1050°C, 1150°C) and 3 different times (2, 4 and 16 hours) were tested for the solution process. The hardness values obtained after the heat treatments are given in Figure 5.3.

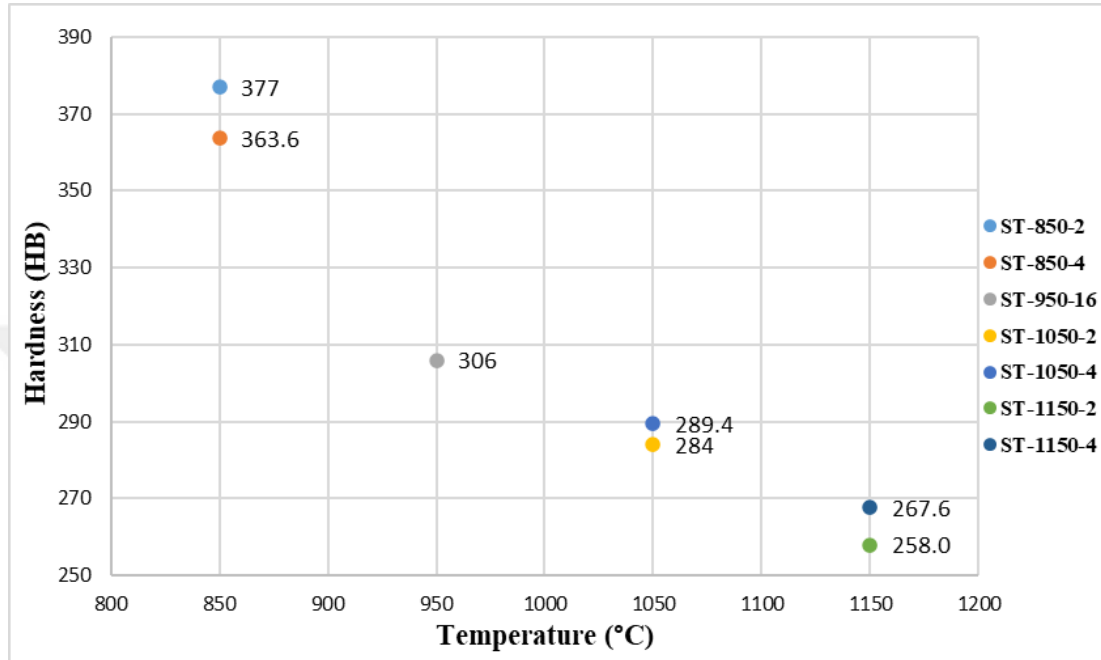


Figure 5.3 Hardness values of different solutionizing conditions of samples

As seen in Figure 5.3, the hardness values obtained as 377 HB and 363.6 HB after 2 and 4 hours of solution treatment at 850°C, 306HB after 16 hours of solution treatment at 950°C, and 284 HB and 289.4 HB after 2 and 4 hours of solution treatment at 1050°C, respectively, were obtained. Finally, the hardness values measured as 258 HB and 267.6 HB after 2 and 4 hours of solution treatment at 1150°C were determined. These results show that there was a significant decrease in the hardness values due to the increase in temperature. In fact, the hardness, which was 470 HB in the as-cast case, decreased significantly after the applied solution treatment.

It is thought that this hardness decrease is related to the dissolution, redistribution and homogenization of the intermetallic phases formed in the structure during the slow cooling after casting at high temperatures. According to the Thermo-Calc phase diagram analysis in Figure 3.5, the structure transforms predominantly into austenite + B2 phases at these temperatures. It is evaluated that this microstructural transformation has a direct effect on the mechanical properties of the material.

Under normal conditions, an increase in solution treatment duration is expected to lead to a decrease in hardness and a more homogenized microstructure. However, in this lightweight steel alloyed with 9 wt.% Ni, it has been observed that solution treatments conducted at elevated temperatures such as 1050°C and 1150°C promote the formation of the B2 phase over time. This phenomenon is considered to have an adverse effect on the mechanical properties by altering the microstructure in an undesirable manner.

For this reason, 1150°C temperature and 2 hours were preferred for the solution process in the later stages of the project. As shown in Figure 5.4, when the applied solution heat treatment and the as-cast microstructure are compared, it is observed that the homogeneity within the matrix increases after the solution process, but needle-like B2 structures are formed.

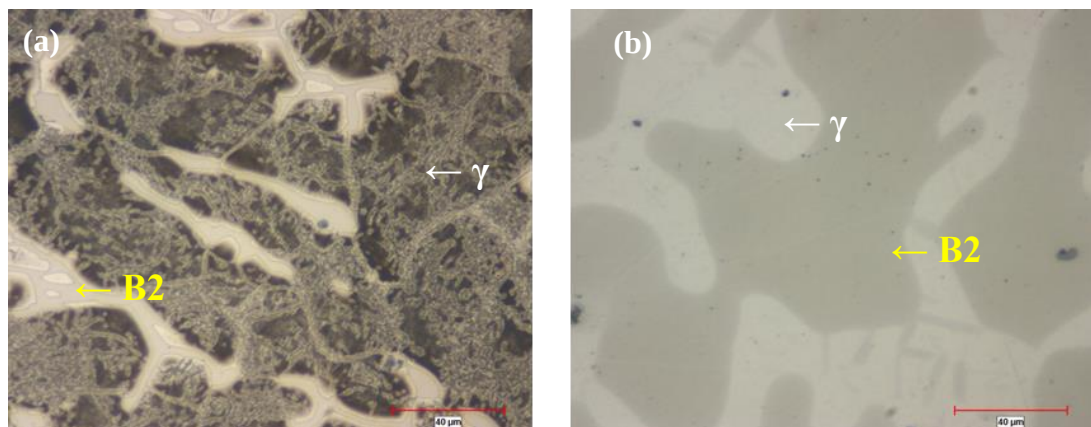


Figure 5.4 (a) As-cast and (b) ST-1150-2 microstructure 100X, (5% Nital etched.)

Similarly, when the XRD analyses of the relevant samples in Figure 5.5 are examined, it is evaluated that the κ -carbide in the as-cast microstructure after the solution treatment are dissolved at high temperatures and transformed into austenite. In addition, it is thought that the ratio of the B2 phase increases during the 2-hour waiting period and therefore the peaks obtained in the XRD analysis mainly belong to the B2 and austenite phases.

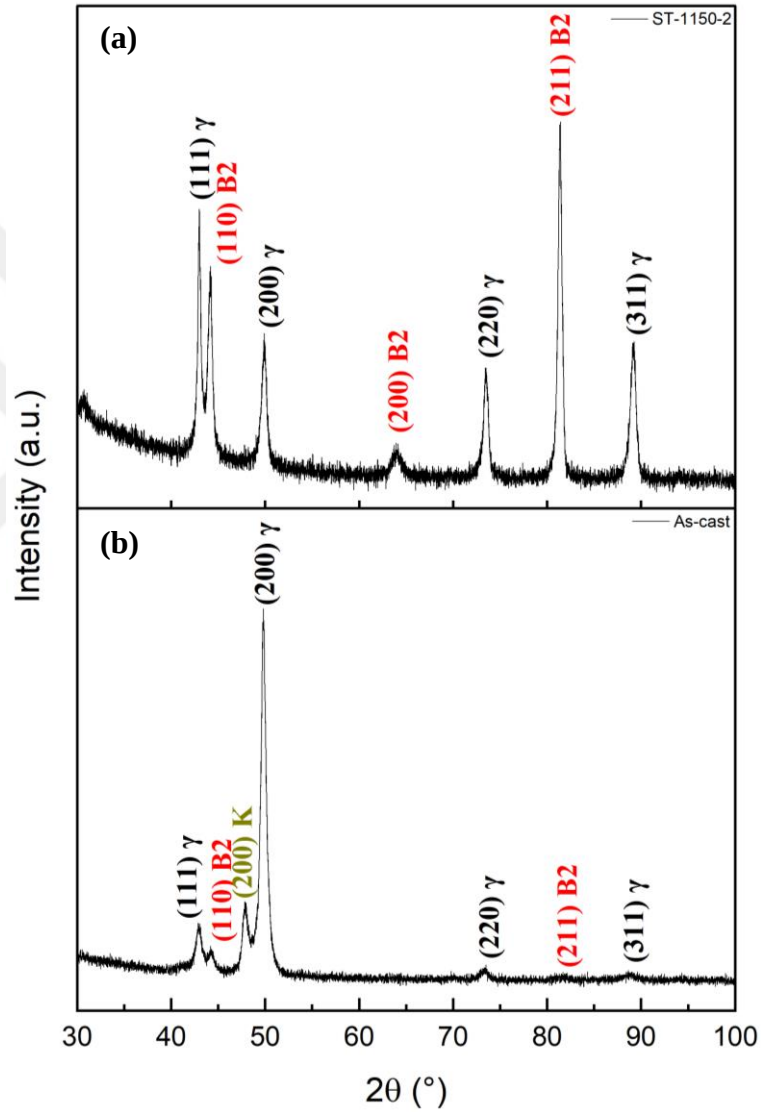


Figure 5.5 XRD pattern of the samples, (a) solutionized at 1150 for 2h, (b) as-cast

5.1.3 Effect of Aging

In the aging treatments conducted following the solution treatment (ST), the Thermo-Calc simulation results and data reported in the literature were taken as references. The hardness values obtained from the aging treatments performed at 700°C are presented in Figure 5.6.

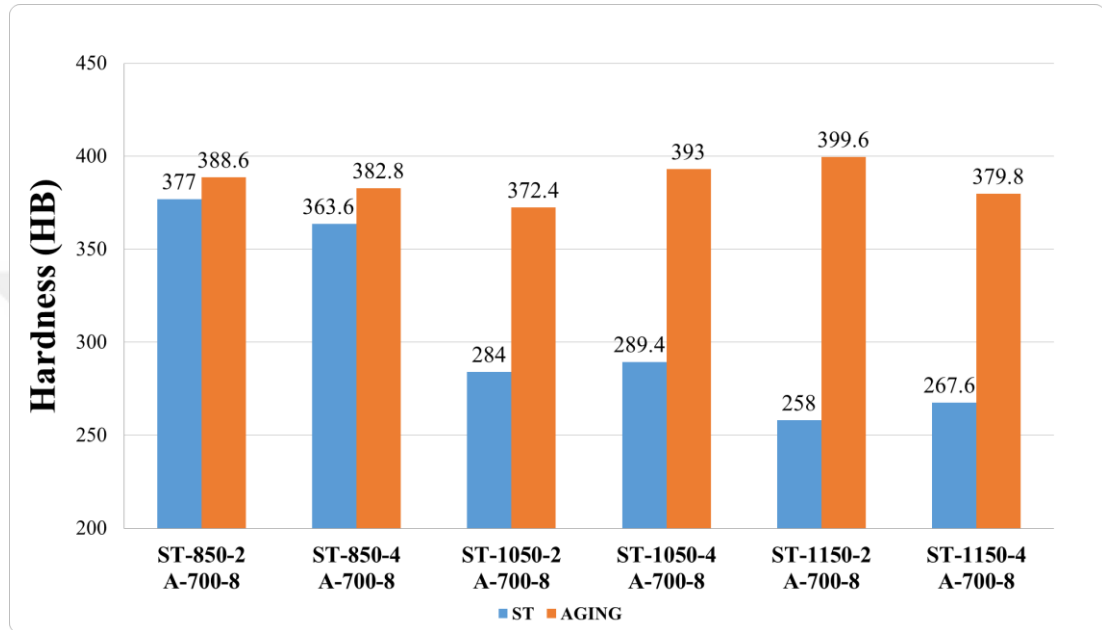


Figure 5.6 Hardness values of Aging at 700°C for 8 hours with different ST conditions

Following the aging treatment conducted at 700°C for 8 hours, the increase in hardness varied between 3% and 36% for solution treatment temperatures below 1150°C. However, after solution treatment at 1150°C, this increase ranged from 40% to 55%. This significant enhancement is believed to be associated with an increased volume fraction of the B2 phase within the microstructure.

Detailed results of aging treatments performed at different temperatures, using the ST-1150-2 condition where the highest increase in hardness was observed as the reference, are presented below.

5.1.3.1 Effect of Aging at 700°C

To evaluate the effect of aging duration at a constant temperature, the results of time-dependent aging experiments are presented in Figure 5.7. Aging treatments were carried out for 1, 4, 8, and 16 hours. The maximum hardness was achieved after 8 hours of aging, followed by a significant decline in hardness beyond this point.

This decline is attributed to overaging during which the prolonged exposure promotes excessive B2 phase precipitation. The increased volume fraction (coarsening) of the B2 phase beyond the optimal aging time is believed to impact the mechanical properties of the alloy negatively.

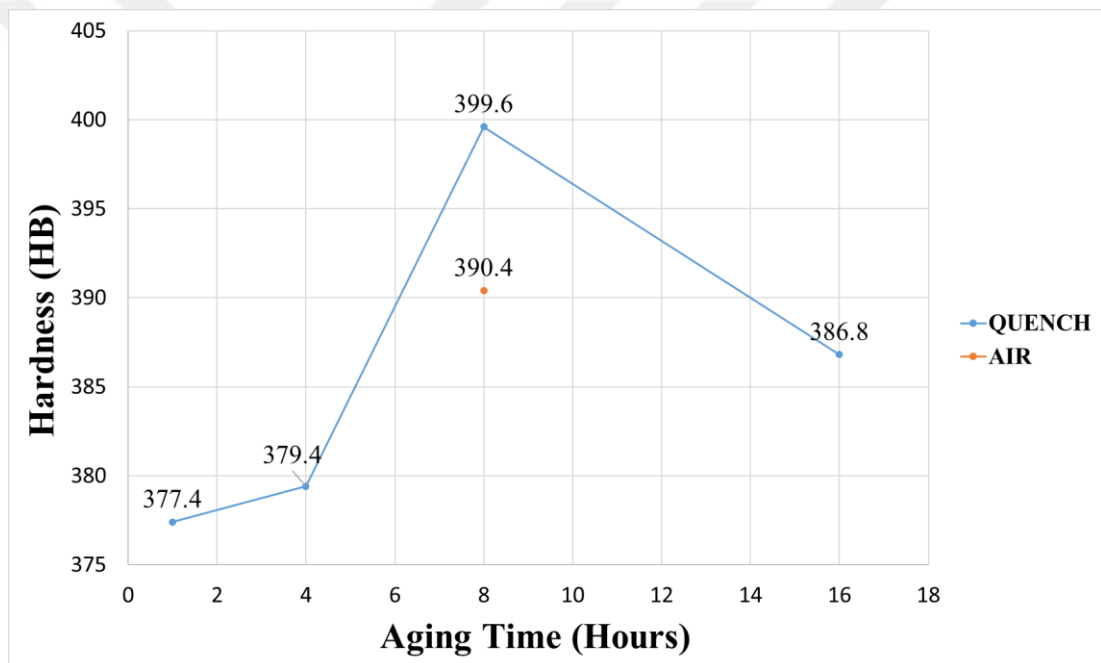


Figure 5.7 Hardness values of aging at 700°C for 1, 4, 8, 8 (air cooled) and 16 h

As a result of the XRD analysis, it was determined that the B2 and austenite phases were clearly observed throughout all aging periods (Figure 5.8). An increase in the intensity of the peaks belonging to the B2 phase was observed with the increase in aging time. Especially in the samples aged for 8 and 16 hours, more distinct peaks were obtained from the (220) plane belonging to the B2 phase compared to the samples aged for 1 and 4 hours. This situation is thought to be related to the coarsening of the B2 phase.

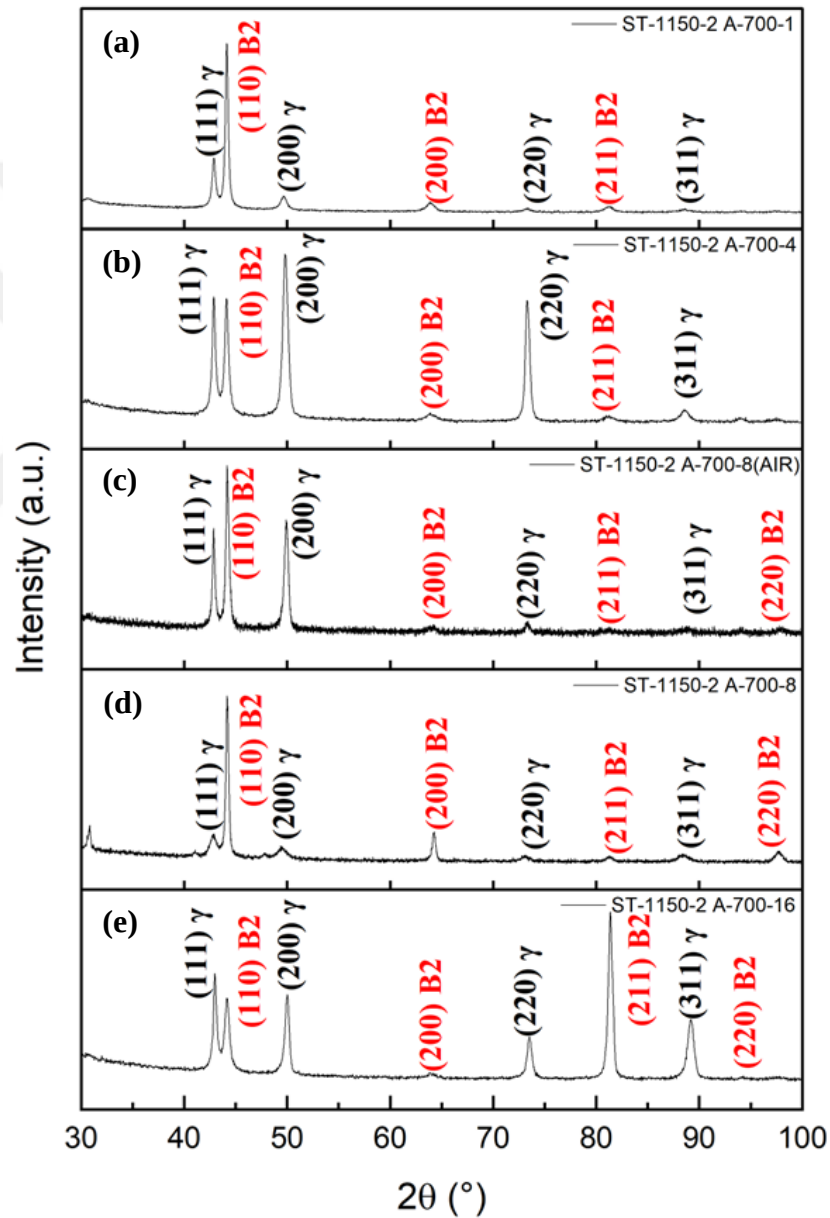


Figure 5.8 XRD Analysis of the samples, solutionized at 1150°C for 2h and aged at 700°C for (a) 1h, (b) 4h, (c) 8h (air cooled), (d) 8h, and (e) 16h

5.1.3.2 Effect of Aging at 550°C

The hardness values obtained after aging at 550°C for 1h, 4h, 8h, and 16 hours are presented in Figure 5.9. Due to the inconsistency of the data obtained from the sample aged for 8 hours at 550°C, this condition was excluded from the discussion section.

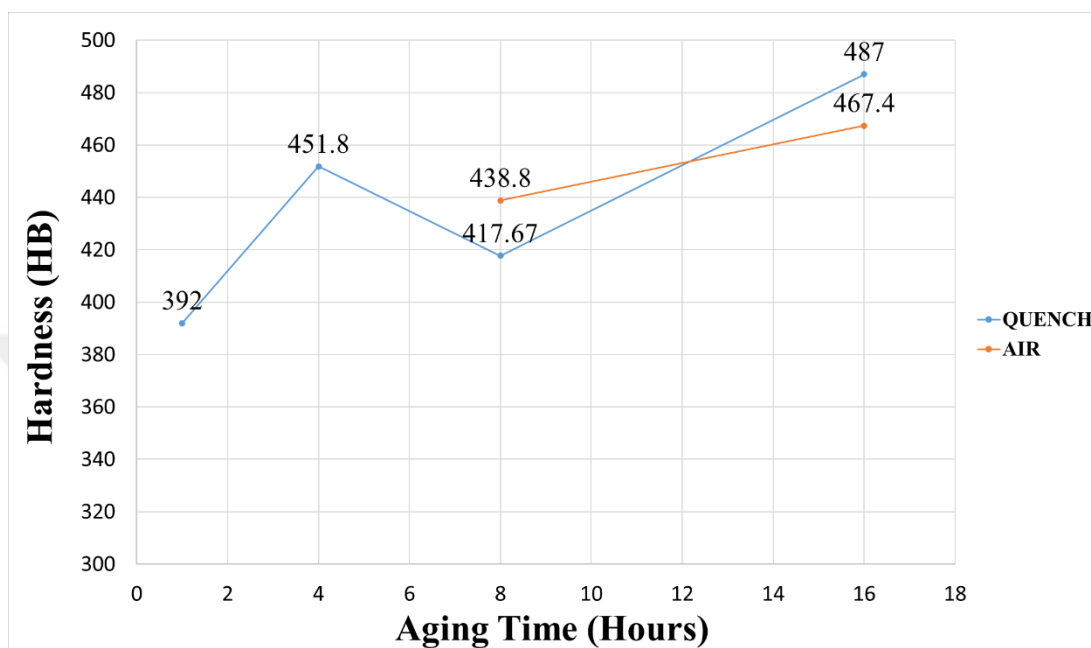


Figure 5.9 Hardness values of samples aged at 550°C for 1h, 4h, 8h, 8h (air cooled), 16 h, and 16h (air cooled)

Based on the data obtained, it is considered that the κ -carbide, which forms between approximately 7 to 10 hours of aging, leads to maximum embrittlement of the material. At longer aging durations, this embrittlement appears to decrease. The data obtained from the samples aged for 8 and 16 hours supports this hypothesis.

5.2 Wear Behavior of the Samples

5.2.1 Wear Behavior of As-cast and Solutionized Samples

In Figure 5.10, the wear test results of AISI 4140, as-cast, ST-1050-4, ST-950-16 and ST-1150-2 samples are given together with the hardness values of the samples. Although it was observed that the wear values of as-cast, ST-1050-4 and ST-1150-2 samples were directly proportional, it was observed that the wear value of ST-950-16 and ST-1050-2 sample did not have a direct connection with the other relevant samples and the measured hardness value.

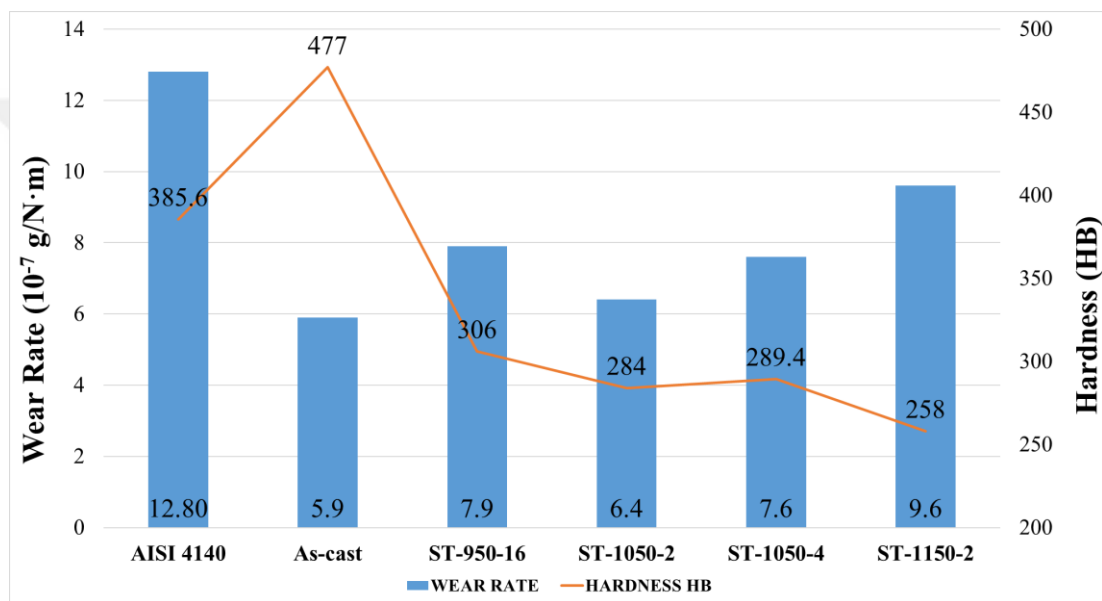


Figure 5.10 Wear rate and hardness values as-cast and solutionized samples

When the calculated wear rate values are compared with the wear rate of the AISI 4140 sample, it is seen that the solutionized Ni-added lightweight steels have lower wear rate values despite having lower hardness values.

5.2.2 Wear Behavior of Aged Samples

The wear behavior of the samples subjected to the aging process can be examined in three different cases. In the first case, the aging temperature was kept constant at 700°C for 8 hours and the effect of different solution parameters was evaluated under this condition. In the second case, the solution parameters were fixed, and the aging process was applied at 700°C for 1, 4, 8 and 16 hours, respectively, and the data of the wear tests performed after these processes were compared. In the third case, after the solution parameters were fixed again, the aging process was performed at 550°C for 1, 4, 8 and 16 hours and the wear data obtained were evaluated.

In the first case, the results obtained from the wear tests applied to samples with different solution parameters are presented in Figure 5.11. When the relevant graph is examined, it is observed that there is an inversely proportional relationship between the hardness values of the samples and the wear rates. However, in the experiments carried out at 850°C, 1050°C and 1150°C, it was observed that the increase in solutionizing time caused an increase in the wear rate, contrary to expectations.

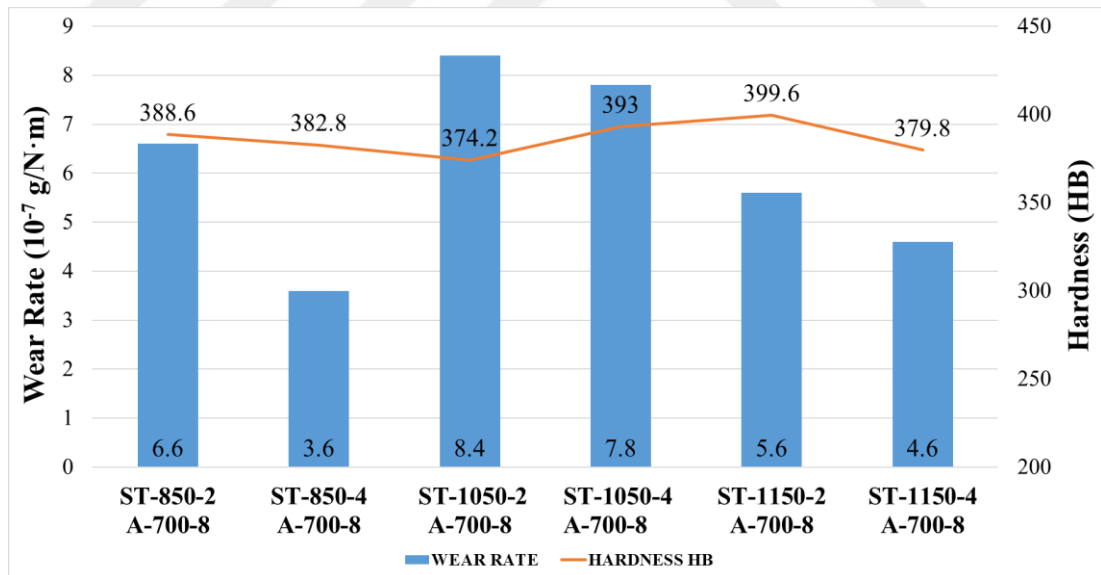


Figure 5.11 Wear rate and hardness values of the samples aged 700°C for 8h after being solutionized at 850°C, 1050°C and 1150°C for 2 and 4h

In the second case, the wear rate obtained after aging processes carried out at 700°C for different durations at constant solution treatment parameters are presented in Figure 5.12. Wear rates corresponding to 1, 4, 8 and 16 h aging times were measured as 6.3, 7.9, 5.6, 7.0 (air cooled) and $6.5 \times 10^{-7} \text{ g/N}\cdot\text{m}$, respectively. The lowest wear rate was obtained in the 8 h aging condition, which had the highest hardness value. On the other hand, no significant correlation was observed between the wear data for 1, 4 and 16 h aging times.

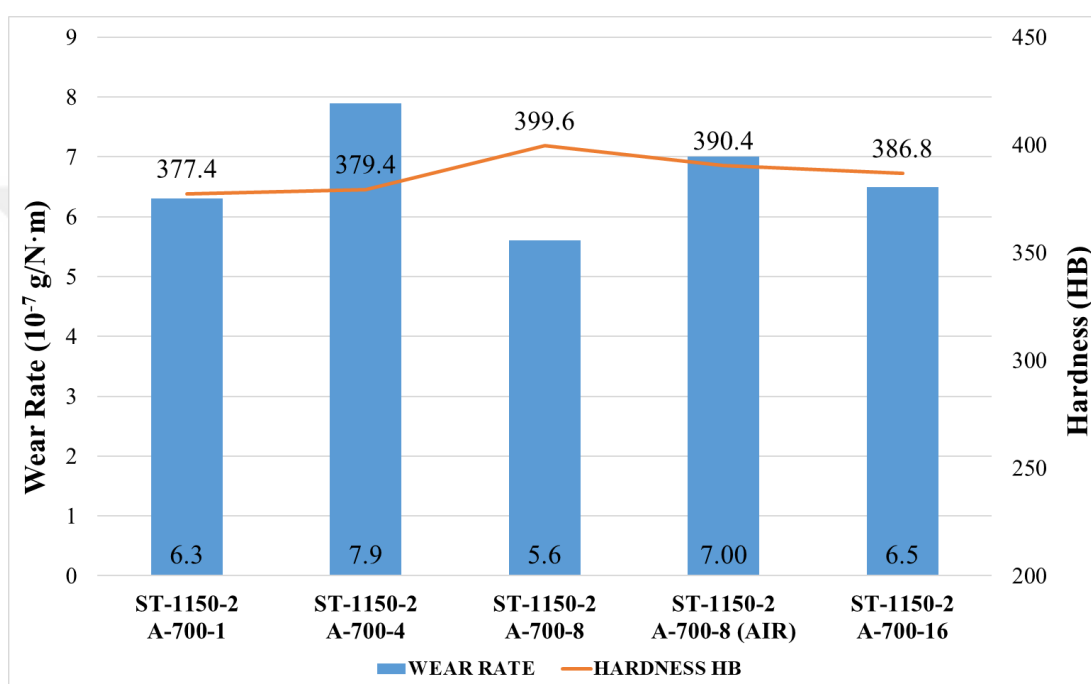


Figure 5.12 Wear rate and hardness values of the samples aged at 700°C for 1, 4, 8 and 16h after being solutionized at 1150°C for 2h

In addition, when comparing the wear test results with AISI 4140 steel, it was observed that the wear rate of lightweight steel was lower for similar hardness values.

In the third case, the results of the wear tests conducted after aging at 550°C for different durations are presented in Figure 5.13. The lowest wear rate was observed under the 550-8 condition. This result is believed to be associated with the extreme brittleness of the sample, which was so severe that a hardness test could not be performed.

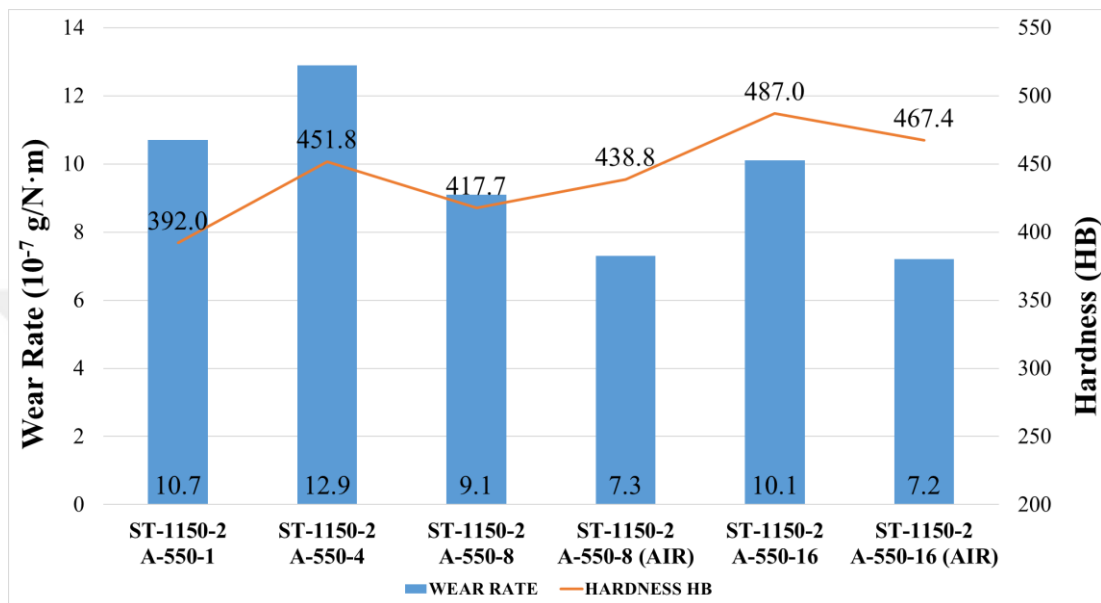


Figure 5.13 Wear rate and hardness values of the samples aged at 550°C for 1, 4, 8 and 16h after being solutionized at 1150°C for 2h

In addition, when the wear rates of the samples that underwent solutionizing at constant temperature and time followed by aging at 550°C for different durations were compared with AISI 4140 steel, it was observed that the wear rates of the lightweight steels with higher hardness values after aging were lower than that of the tested AISI 4140 steel (except for A-550-4).

5.3 General Discussion

The tests and analyses performed have revealed important findings that are limited in the literature about Fe-Mn-Al-Ni-C light steel with 9% Ni addition. Since the current information on the relevant chemical composition is very limited, the originality and importance of the experimental data and conclusions obtained in this study increases.

In lightweight steels with Ni addition, in order to benefit from the contribution of the B2 phase in the microstructure on the mechanical properties at the maximum level, hot and cold rolling, solution (or annealing) and aging processes are generally applied after casting. Thanks to this process sequence, both strength is increased by ensuring the homogeneous distribution of the B2 bands formed after casting and the negative effects of these bands on the mechanical properties are minimized.

In this study, the aim was to develop a heat treatment recipe suitable for industrial use with only solutionizing and aging processes without applying plastic deformation. Solutionizing processes were carried out between 850°C and 1150°C for 2, 4 and 16 hours. The aging processes were conducted for 1, 4, 8 and 16 hours at 550°C and 700°C, covering a wider range compared to literature.

The data obtained from the tests showed that solution treatments conducted at high temperatures and for extended periods increased the size of the B2 phase, which increased material hardness. Additionally, it was observed that material hardness decreased after a certain time during prolonged aging at 700°C, while at 550°C, the same decrease in hardness increased after a certain time, leading to brittleness.

Furthermore, the results obtained in this thesis may function as a guide for future studies to be conducted with alloys of similar chemical composition and fill gaps in literature. In addition, it is shown that Fe-Mn-Al-Ni-C lightweight steels can be a strong alternative to AHSS in advanced engineering applications such as automotive, aerospace and defense industries that require high specific strength.

CHAPTER 6

CONCLUSION

6.1 Main Conclusions

In this study, an experimental Fe-Mn-Al-Ni-C lightweight steel containing 9 wt.% Ni was cast and the effects of various solutionizing (between 850°C and 1150°C for 2, 4, and 16 hours) and aging (at 550°C and 700°C for 1, 4, 8, and 16 hours) treatments on the microstructure, hardness, and wear resistance were systematically investigated. The key findings can be summarized as follows:

- The density of the cast alloy was measured as 6.83 g/cm³, which is approximately 12% lower than that of conventional steels, highlighting its potential as a lightweight alternative for structural applications.
- The lowest hardness value was obtained after the solutionizing treatment conducted at 1150 °C for 2 hours, where the hardness decreased by almost 46%, from 476 HB in the as-cast condition to 258 HB. Also, prolonged solution treatment at elevated temperatures resulted in B2 phase coarsening, which caused a decrease in hardness and microstructural uniformity.
- Aging at 550°C after solution treatment at 1150°C for 2 hours resulted in κ -carbide formation; however, excessive aging times (≥ 8 hours) increased hardness but resulted in deterioration of microstructural properties (coarsening) and embrittlement.

- Since recrystallization cannot be achieved by heat treatment alone, the coarse and heterogeneous microstructure formed during solidification mostly remains even after heat treatment.
- The lowest wear rate value of 3.6×10^{-7} g/N·m was measured in the samples solutionized at 850°C for 4 hours and then aged at 700°C for 8 hours. On the other hand, the highest wear rate value of 12.9×10^{-7} g/N·m was observed in the samples aged at 550°C for 4 hours after 2 hours of solutionizing at 1150°C.
- It is concluded that due to its poor ductility Ni-containing cast Fe-Mn-Al-Ni-C lightweight steels exhibit limited applicability when only heat treatment is employed. For these materials to be industrially viable and meet the demands of advanced structural applications, it is necessary to incorporate hot and/or cold deformation steps in the processing route to induce recrystallization and enhanced precipitation so that microstructures producing both high strength and ductility at the same time could be obtained.

6.2 Suggestions for Future Work

- In addition to hardness measurements, it is recommended to perform tensile tests with extensometer support to accurately determine further mechanical properties such as % elongation, yield strength, and ultimate tensile strength (UTS).
- A comprehensive study should be conducted on Fe-Mn-Al-Ni-C lightweight steels with varying Ni contents between 0wt% to 8wt%, including detailed phase analysis, microstructural characterization, and mechanical property evaluation for both the as-cast condition and under the same heat treatment conditions applied in this study.

- In addition to heat treatment-based investigations, future studies should also focus on the effects of high-temperature thermo-mechanical processing. Specifically, samples subjected to solutionizing at 1200°C for 2 hours and then hot deformation at temperature of 1050°C with a thickness reduction of approximately 70–80% should be aged at 700°C for varying durations.



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APPENDIX

3-Point Bending Test Results

In order to obtain more data in the evaluation of mechanical properties of as-cast material, a 3-point bending test was performed with the 300DX device of the Instron brand, located in the laboratory of the Metallurgical and Materials Engineering Department of Atılım University. Samples measuring 20 mm wide, 120 mm long, and 2 mm thick were cut and used for the tests. The distance between supports in the test setup was set at 60 mm. Images of the test setup are shown in Figure 1.

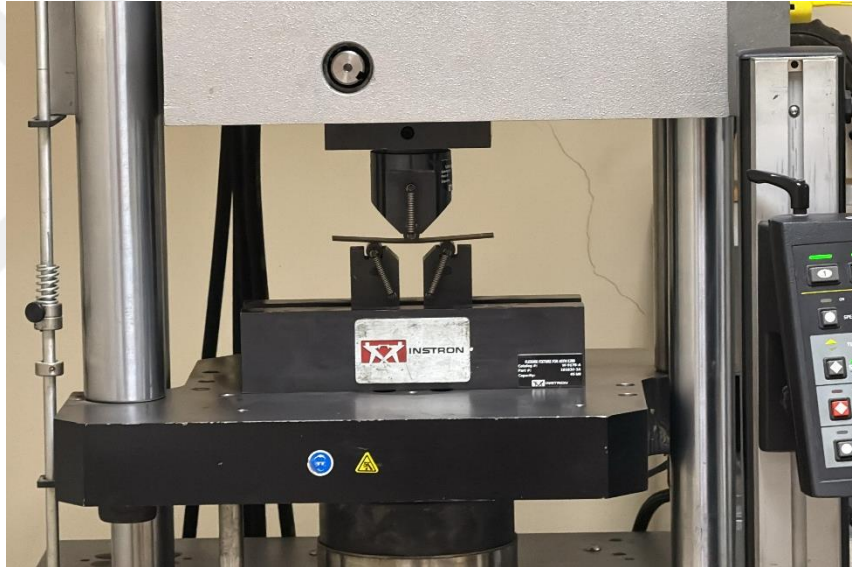


Figure 1. Image during the 3-point bending test

The results of the tests performed on four as-cast samples are shown in Table 1.

Table 1. 3-Point bending test results

Compressive Load (kN)	Maximum Compressive Displacement (mm)	Maximum Compressive Strain (%)	Fracture Displacement (mm)	Fracture Load (kN)	Fracture Strain (%)	Offset Yield Strain (%)
0.35	2.41	3.12	3.19	-0.18	4.12	2.37
0.49	3.19	4.14	4.49	-0.22	5.83	4.14
0.55	3.38	4.38	4.19	-0.25	5.43	4.40
0.47	2.94	3.84	3.61	-0.29	4.71	3.84

As a result of the tests, it was observed that all tested samples fractured in a manner indicating brittle behavior. In other words, there was no sign of plastic deformation in the fractured samples. Images of the samples obtained after the tests are presented in Figure 2.



Figure 2. Images of as-cast samples after tests

Additionally, the values obtained from the three-point bending test conducted on the as-cast samples, along with the flexural strength values calculated using the formula presented in Equation (1), are provided in Table 2.

$$\sigma = \frac{3FL}{2bd^2} \quad (1)$$

where:

- F: Force at fracture point (in N)
- L: Support span length (in mm)
- b: Width of the sample (in mm)
- d: Thickness of the sample (in mm)

Table 2. Calculated flexural strength values of the as-cast samples

Sample	Fracture Load (kN)	Flexural Strength (MPa)
1	0.18	202.50
2	0.22	247.50
3	0.25	281.25
4	0.29	326.25
Mean	0.24	264.38
Standard D.	0.05	52.37