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Research Article

Effect of Chemical Composition and Annealing Parameters for Advanced Packaging Steel Applications

Ramazan Uzun*, Ümran Başkaya, Yasemin Kılıç

Eregli Iron and Steel Works Co., R&D Center, Kdz. Eregli, Zonguldak, Türkiye, ruzun@erdemir.com.tr, uerten@erdemir.com.tr, ykilic@erdemir.com.tr
*Corresponding Author

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ABSTRACT

Packaging steels are widely used across a broad range of industries, from food and beverage to chemical products, for the protection, transportation and storage of goods. In this study, the effects of chemical composition and annealing parameters on the phase transformation behavior, microstructure, and mechanical properties of packaging steels were investigated. Two steel samples, Steel A and Steel B, with different chemical compositions (designed according to ASTM A623-22 standard limitations), were prepared using a vacuum induction melting (VIM) furnace. Within the scope of simulation studies, hot rolling, cold rolling, and annealing process simulators were utilized. Before the annealing simulations, the Gleeble 3500 thermal simulation device and JMatPro software were used to determine the process conditions. A light optical microscope (LOM) and a scanning electron microscope (SEM) were used for microstructural characterization studies. Mechanical properties were characterized with tensile tests. Steel A and Steel B samples with different alloying elements and cooling rates were compared to evaluate their suitability for advanced packaging applications. The results of this analysis show that the addition of Nb and Mn to Steel B enhances bainite formation, refines grain size, and improves mechanical properties compared to Steel A.

1. Introduction

Traditional steels often struggle to balance strength and ductility; as strength increases, ductility typically decreases, making it difficult to meet safety and performance standards [1, 2]. Advanced high-strength steels (AHSS) with multiphase microstructures, including ferrite, bainite, martensite, and retained austenite, are increasingly used in automotive applications [3-7]. These steels are favored over traditional high-strength low-alloy (HSLA) steels because of their exceptional strength-to-ductility ratio, which merges high strength, excellent ductility, continuous yielding and high initial work hardening rates [8, 9]. The packaging sector has seen significant advancements in steel grades over recent decades, focusing on both higher strength materials and the need for highly formable steels suitable for complex deep drawing applications [10]. A specific example of such applications is the forming of valve cups for aerosol tops, which requires a high level of deformation, thus placing substantial demands on the materials used [11]. Packaging steels are preferred for their compatibility with human health, non-toxicity, excellent formability, weldability, and resistance to corrosion [12].

Tinplate is a versatile material primarily used in the packaging industry, particularly for food and beverage containers packaging steel finds use across a wide range of applications [13, 14]. These include diverse types of containers for food, personal hygiene products, household and automotive care items, industrial products, and paints. Additionally, it is utilized in the creation of gifts or promotional products, as well as

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closures and aerosols [15, 16]. Tinplate materials are widely used in various industries due to their excellent properties such as corrosion resistance, high strength, and formability [17, 18].

Conventional tinplate materials primarily consist of a ferritic phase. Ferrite is characterized by its low carbon content, providing ductility and softness to the steel. This microstructure possesses high formability and low strength properties, enhancing the workability of tinplate materials and making them suitable for various packaging applications [19, 20]. Multiphase steels refer to materials that have a combination of different phases, such as ferrite, bainite, martensite and retained austenite. This combination enhances the mechanical properties of the tinplate, providing a balance of strength and ductility, which is beneficial for various packaging applications [21, 22]. In multiphase steels, high levels of alloying elements such as Mn, Si and Cr are required to achieve the desired microstructural and mechanical properties [23, 24]. However, for packaging steels that come into contact with food, the ASTM A623-22 standard imposes restrictions on the chemical composition of these elements [25]. Therefore, it is necessary to design and develop new concept packaging steels that go beyond the conventional approaches to multiphase steel production. This innovative approach aims to meet both the stringent chemical composition requirements for food contact and the mechanical performance demands of advanced packaging applications.

Recent literature reviews indicate a lack of comprehensive information on multiphase packaging steel. This is a significant gap considering the increasing demands of the packaging industry, which expects materials to maintain high strength and excellent formability even as thickness reductions become more stringent. Achieving these properties necessitates the presence of phases other than ferrite in the final microstructure. In traditional thin packaging steel production, a double cold reduction process is employed after annealing [26, 27]. This process not only reduces the material's thickness but also enhances hardness and strength, albeit at the expense of elongation percentage [14, 17, 18]. The desired mechanical properties and formability characteristics could potentially be achieved through a multiphase microstructure, thereby eliminating the need for a second cold reduction process. This approach would allow for direct utilization of the material post-annealing, meeting industry requirements for high performance without additional processing steps.

In the present work, the effects of chemical composition and annealing parameters on the phase transformation behavior, microstructure and mechanical properties of multiphase packaging steels were investigated.

2. Experimental Study

A schematic diagram illustrating the steps of the experimental procedure is shown in Figure 1.

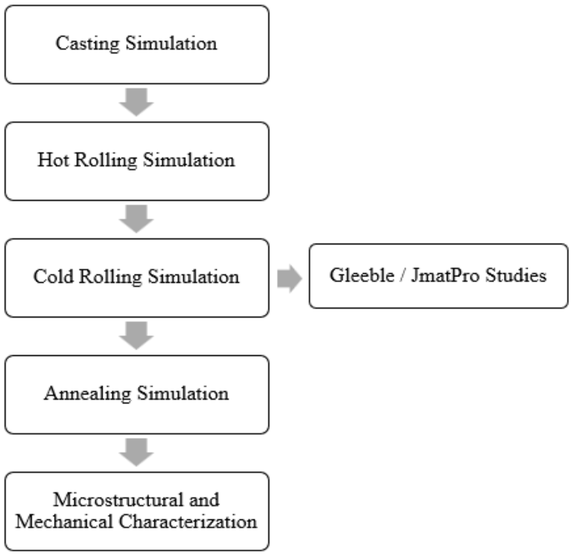


Figure 1. Schematic diagram of experimental process

2.1. Material

In the experimental studies, cold-rolled steel has been used. The chemical compositions of the materials used in the experimental studies are given in Table 1. The percentage values of the elements used in Table 1 have been designed considering the limit values specified in the ASTM A623-22 standard.

Table 1. Chemical composition of samples (wt.%)

Elements	Steel A	Steel B
C	0.110	0.110
Mn	0.350	0.530
Si	0.015	0.015
P	0.011	0.012
Cr	0.060	0.060
Nb	-	0.017

2.2. Simulation tests

Two different chemical compositions were used to produce ingots with dimensions of 500×150×225 mm in a vacuum induction melting (VIM) furnace. VIM furnace process is a technique used to melt metals under controlled vacuum conditions, typically for producing high-quality alloys. The process takes place under a vacuum environment to minimize contamination from gases such as oxygen or nitrogen, which could negatively affect the quality of the molten metal. This method is particularly useful for producing high-purity metals and alloys, as it reduces the presence of impurities that can occur in more conventional melting methods. The casting of materials with the compositions of Steel A and Steel B was completed, followed by hot rolling processes. During the hot rolling process, the finishing temperature was set at 900°C and the coiling temperature was applied at 550°C. In the cold rolling simulator, a reduction of 80% was applied to the samples. The rolled samples were subjected to annealing simulations to achieve the desired mechanical properties and to develop a multiphase microstructure. Prior to the annealing process, transformation temperatures can be predicted through physical simulations and computational software.

The production of multiphase steel involves determining the ferrite-austenite phase transition temperatures (A1 and A3), heating the material to the specified process temperature within these critical temperature ranges and maintaining this temperature to achieve the two-phase region. The material is then quenched to room temperature in order to achieve the desired microstructural features [28-29].

Computer simulations of intercritical continuous cooling transformation (CCT) diagrams, which are calculated based on chemical composition, have been utilized as a strategy to develop new chemistries for producing multiphase steels [30, 31]. The JmatPro software was performed to predict phase transformations.

Determining the transformation temperatures, Continuous Cooling Transformation (CCT) tests were conducted using a Gleeble 3500 thermal simulation device. The test involved heating solid flat specimens (Figure 2), 17.5 cm in length and 5 cm in width, to 920°C at a heating rate of 1°C/s. The specimens were held at this temperature for 30 seconds before being cooled to room temperature. It was cooled from this temperature to room temperature at a cooling rate of 1°C/s.

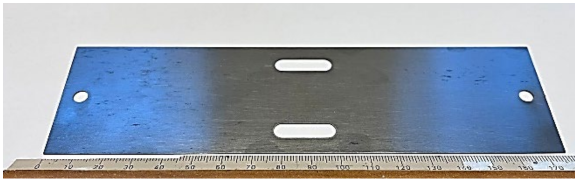


Figure 2. The sample of Gleeble 3500 thermal simulation device

2.3. Microstructural and mechanical characterization

Samples for microstructural analysis were cut along the transverse direction (TD). These sectioned specimens hot-mounted, grounded and polished in sequence. After polishing, the samples were etched with 2% nital and picral solutions for characterization. Samples were characterized using a light optical microscope (Nikon Eclipse MA200) and a scanning electron microscope (SEM, Zeiss EVO10).

Tensile tests were performed using a Zwick Z250 testing machine. The tests were conducted in accordance with the ISO 6892-1 standard [32].

3. Results and Discussion

3.1. Evaluation of simulation tests

Before annealing operation, dilatometric analyzes were carried out on the Gleeble Thermal Simulation device to determine the Ac_1 and Ac_3 transformation temperatures of the Steel A composition. The dilatation curves featured two key points corresponding to the intercritical temperatures. These points, marked by the first and second peaks on the dilatation curves, represent the Ac_1 and Ac_3 temperatures, respectively. The CCT diagrams created and calculated with the usage of JMatPro software were used to examine the phase transformation and transition temperatures. It has been determined that the Ac_1 (725°C) and Ac_3 (870°C) temperatures obtained from CCT tests conducted on the Gleeble device are compatible with the temperatures predicted using JmatPro. The temperature values set on the Gleeble device were consistent with the measured values. The dilatometric curve of Steel A is given in Figure 3 and CCT diagram of Steel A is shown in Figure 4. According to the CCT diagram, the possible phases in the Steel A sample are ferrite and pearlite, with no additional phase transformations such as bainite and/or martensite observed as the cooling rate increases.

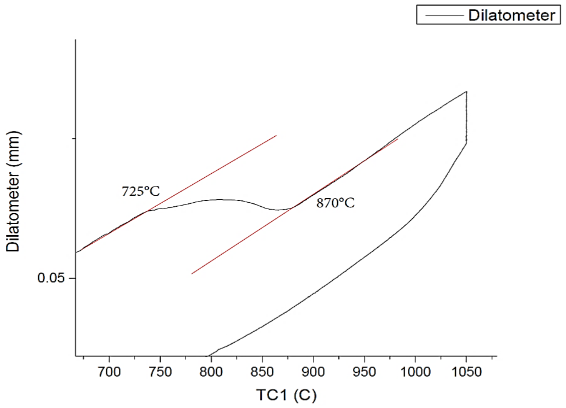


Figure 3. Gleeble dilatometry curve for Steel A

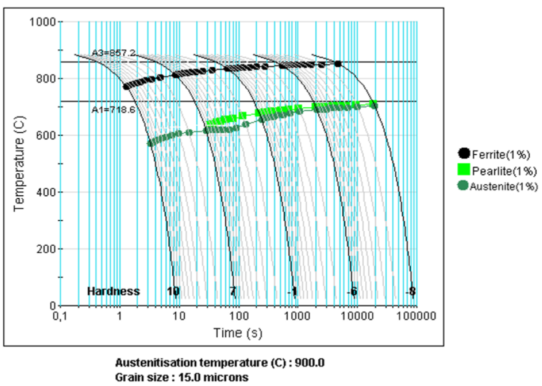


Figure 4. Continuous cooling transformation diagram for Steel A

The CCT diagram calculated for the composition of Steel B using JMatPro is presented in Figure 5. When comparing the CCT diagrams of Steel A and Steel B, it is observed that the Ac_1 and Ac_3 temperature values are close. The expected phase structures in the microstructure vary depending on the cooling rates applied in the CCT diagrams. In terms of microstructure, it is predicted that bainite and ferrite may form depending on the applied cooling rate.

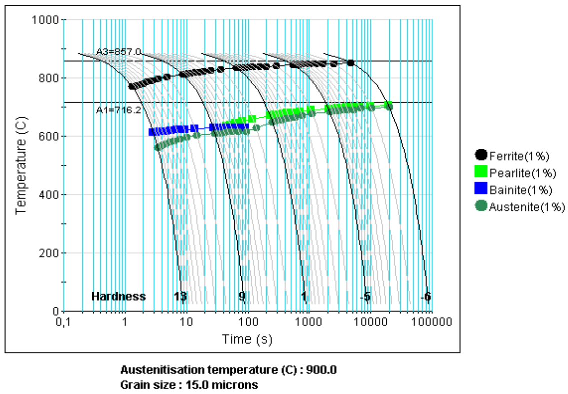


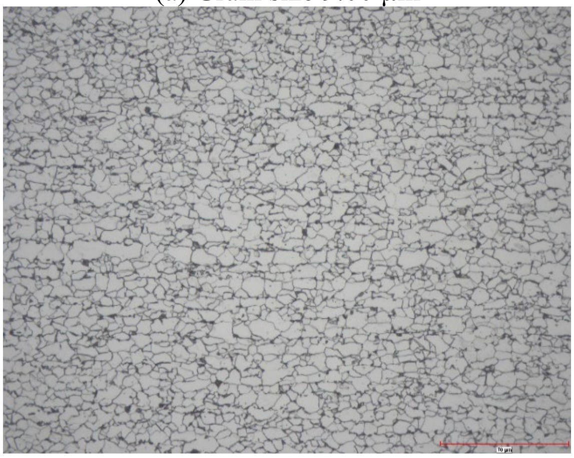
Figure 5. Continuous cooling transformation diagram for Steel B

Generally, achieving multiphase steel requires a high alloy content. However, as previously mentioned, due to the alloying limitations specified in the ASTM A623-22 standard for packaging steels, alloying can not be utilized. Therefore, process parameters were optimized to achieve the desired final properties. For cold-rolled steels with low alloy content in their chemical composition, it is necessary to apply high cooling rates during the annealing process. In the annealing process, a temperature of 810°C for 100 seconds within the A1 and A3 temperature range, was applied. Trials were performed at different cooling rates (30°C/s and 150°C/s).

3.2. Characterization of microstructure and mechanical properties

Niobium increases material strength with solid solution, grain refinement and precipitation hardening [33]. Nb is precipitated in the microstructure and delays recrystallization and forms an effective barrier to grain growth. Grain refinement effect also contributes positively to material strength and toughness [33, 34]. Microstructure images of samples etched with nital solution are given in Figure 6. Grain size of the Steel B was finer than the Steel A sample. It has been observed that niobium has a grain refining effect.

(a) Grain size 9.00 µm



(b) Grain size 6.00 µm

Figure 6. Optical microscope micrographs of nital etched Steel A (a) and Steel B (b) samples

In the micrographs etched with nital, it was observed that the microstructure consisted of a two-phase structure. The light-toned regions represent the ferrite phase while the dark black areas are likely indicative of pearlite or bainite. The secondary phase content has been calculated using Clemex software. According to the calculation, the Steel A contains 8% secondary phase while the Steel B contains 9% secondary phase. However, this calculation does not differentiate between bainite and pearlite. In order to determine the second phase, picral etched samples were characterized by using SEM.

In the Steel A, the microstructure consists of ferrite and pearlite (Figure 7). This situation is also consistent with the CCT data in the simulation studies. Since the Steel A sample had a lean analysis, the experimental studies were continued with the Steel B sample in order to obtain the targeted microstructure and mechanical properties.

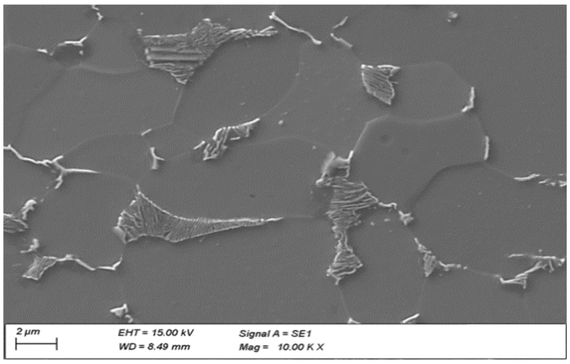


Figure 7. SEM images of steel A sample annealed at low cooling rate

In order to support the bainite transformation and strength increase, the Mn alloy element was increased and the Nb alloy element was added to the Steel B sample. In addition to alloy design within standard limits, cooling rates in the process were also revised. With the increasing cooling rate, bainite formation occurred and the microstructure consists of ferrite and bainite (Figure 8 and Figure 9).

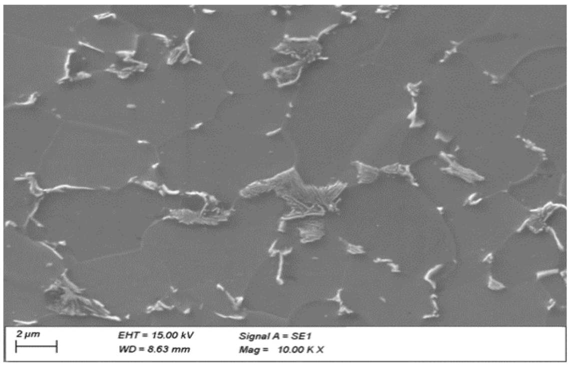


Figure 8. SEM images of Steel B sample annealed at low cooling rate

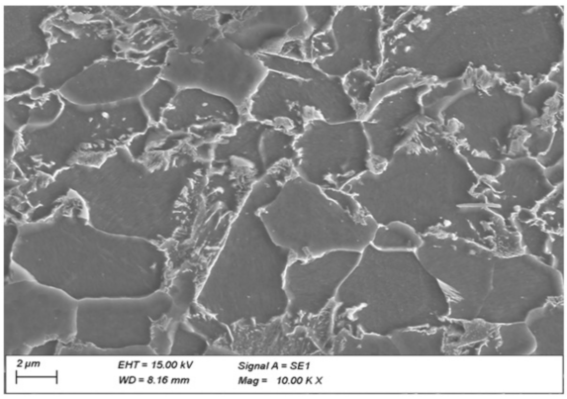


Figure 9. SEM images of Steel B sample annealed at high cooling rate

The mechanical test results observing the effects of process and alloy design for Steel A and Steel B samples are presented in Table 2. The mechanical tests were performed two times.

When simulations performed at low cooling rates are compared, it is determined that the mechanical properties of the Steel B sample are better. Although both steels contain ferrite and pearlite phases, this difference in mechanical properties can be explained by variations in grain size (Steel A: 9 µm / Steel B: 6 µm) and alloy composition. Different mechanisms are used to enhance the strength of steels. These include solid solution strengthening, precipitation hardening, grain size refinement, phase transformation and work hardening [35-37].

As the grain size decreases, the strength and toughness of the steel improve. The relationship between grain size and strength is explained by the Hall-Petch equation [35]. The precipitates present in the microstructure prevent grain boundary coarsening and contribute to achieving a fine-grained structure [35]. As the grain size decreases, grain boundaries act as barriers to dislocation motion, thereby enhancing strength. In this study, the higher strength observed in the Steel B sample compared to Steel A is attributed to the contributions of the Nb alloying element to fine grain size and precipitation strengthening, as well as the Mn alloying element to solid solution strengthening. In addition to annealing simulation studies performed at low cooling rates, the Steel B sample was carried out to both low and high cooling rates.

The microstructure of the simulation sample subjected to a low cooling rate consisted of ferrite and pearlite whereas the sample subjected to a high cooling rate exhibited the formation of ferrite and bainite (Figure 7, Figure 8 and Figure 9). In the Steel B sample, the increase in cooling rate promoted bainite formation, which positively contributed to the improvement of tensile strength.

Table 2. Mechanical values of samples

Mechanical Values	Steel A	Steel B	
	LCR	LCR	HCR
Yield Strength, MPa	351±6	370± 5	347±5
Tensile Strength, MPa	402±7	430± 7	502±6
% Elongation	21 ±1	21± 1	23± 1

LCR: low cooling rate (30°C/s)
HCR: high cooling rate (150°C/s)

3. Conclusion

This study investigated the effects of chemical composition and annealing parameters on the phase transformation, microstructure, and mechanical properties of multiphase packaging steels. The results are summarized below.

- The alloying values were used in the production of conventional multiphase steels can not be applied for packaging steel due to the alloying elements limit specified in the ASTM A623-22 standard. In the production of standard high strength packaging steel, the double reduction process enables an increase in strength. If the production of multiphase packaging steel becomes feasible, the double reduction process may not be necessary. Within this scope, a new alloy and process design has been developed.

- Mn contributed significantly to solid solution strengthening while Nb promoted grain refinement and precipitation strengthening. These modifications improved the tensile strength of Steel B compared to Steel A.

- High cooling rates facilitated the formation of bainite which was directly affected to tensile strength in Steel B. Conversely, lower cooling rates resulted in a microstructure dominated by ferrite and pearlite.

Article Information Form

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Authors Contribution
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The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by authors.

The Declaration of Ethics Committee Approval

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