

Investigation on impact and DWTT resistance at low temperatures in Nb and Ti micro alloy steels

Nb ve Ti mikro alaşımlı çeliklerde düşük sıcaklıklarda darbe ve DWTT dayanımının iyileştirilmesine yönelik inceleme

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Abstract

Depending on the climatic conditions in which they are used, new generation petroleum pipe steels come up with the need for high strength and excellent toughness. During the transportation of oil and natural gas, these steels should be at the highest level of impact resistance along with high hardness. In recent years, the general test temperatures, such as 0°C, tend to be lower minus degrees. In this study; (American Petroleum Institute X70 Quality) API X70, impact, (drop-weight tear test) DWTT, and strength were modeled on rolls with thicker than 10 mm. In addition, the necessary parameters for high impact strength at low temperatures were determined by modeling. After the steel production and rolling with the specified parameters, mechanical tests and microstructure examinations were carried out. In order to determine the effect of strength and toughness on mechanical properties, different test sets were prepared for chemical analysis and rolling parameters. The differences in the product microstructure and test results of the titanium level and different holding times and thermomechanical and conventional rolling were investigated. The impact energy values of the materials produced with different process parameters were examined using large data sets. As a result, it has been determined that the particles produced by the thermomechanical rolling method and the particles are the most minor and homogeneously dispersed in the microstructure meet the expectations in terms of material quality.

Keywords: Thermomechanical rolling, Grain refining, Micro structure, Coiling temperature, Niobium effect.

Öz

Yeni nesil petrol boru çelikleri, kullanıldıkları iklim koşullarına bağlı olarak yüksek mukavemet ve mükemmel tokluk ihtiyacı ile karşımıza çıkmaktadır. Petrol ve doğalgazın taşınması sırasında bu çeliklerin yüksek sertlik ile birlikte darbe dayanımının da en üst seviyede olması gerekmektedir. Son yıllarda, 0°C gibi genel test sıcaklıkları, eski derecelerden daha düşük olma eğilimindedir. Bu çalışmada (American Petroleum Institute X70 Quality) API X70, darbe, (düşürme-ağırlığı yırtılma testi) DWTT ve mukavemet; 10 mm'den kalın rulolar üzerinde modellenmiştir. Ayrıca düşük sıcaklıklarda yüksek darbe dayanımı için gerekli parametreler modelleme ile belirlenmiştir. Belirtilen parametrelerde çelik üretimi ve haddeleme sonrasında mekanik testler ve mikroyapı incelemeleri yapılmıştır. Mukavemet ve tokluğun mekanik özellikler üzerindeki etkisini belirlemek için kimyasal analiz ve haddeleme parametreleri için farklı test setleri hazırlanmıştır. Titanyum seviyesi ve farklı bekletme süreleri ile termomekanik ve konvansiyonel haddelemenin mamul mikro yapı ve test sonuçlarında yarattığı farklılıklar araştırıldı. Farklı proses parametreleri ile üretilen malzemelerin darbe enerji değerleri büyük veri setleri kullanılarak incelenmiştir. Sonuç olarak termomekanik haddeleme yöntemi ile üretilen ve tanelerin en küçük ve mikro yapı içerisinde homojen dağılmış olanların malzeme kalitesi olarak beklentileri karşıladığı tespit edilmiştir.

Anahtar kelimeler: Termomekanik haddeleme, Tane inceltme, Mikro yapı, Sarılma sıcaklığı, Niyobyum etkisi.

1 Introduction

Steels that comply with API 5L or (American Petroleum Institute Oil Pipe Standard) EN ISO 3183 are steels produced according to the criteria set by the American Petroleum Institute or European Standards and are used in oil and natural gas pipelines [1]. The characteristic of these steels can be expressed as being resistant to welding, shaping, and impacts due to their high strength and low alloy in accordance with the limits specified in the relevant quality descriptions. Spiral pipe is manufactured with hot rolled coil, and straight seam pipe is manufactured with sheetmetal and coil [2]. Today, steels that are produced with excellent (at maximum resistance to the environmental conditions in the place of use) hardness and the best toughness combination are preferred for oil pipelines. [3], [4].

The purpose of the study is to provide the excellent properties of steel properties needed in natural gas and oil transportation to be used in the hard climate of the northern regions. The desired toughness values at very low temperatures could not be achieved 100% with steel production practices so far. Although high strength has been attempted to be achieved with micro alloy support in previous productions, it is necessary to use new parameters today to support these properties with high toughness value [5],[6]. With the improvement to be made in these qualities, steel with high impact resistance even at low temperatures can be produced. The "petroleum pipe steels group," in which these qualities are represented (X52, X60, X65, X70), is a quality group with high added value, which is also frequently ordered from Iron and Steel factories because they are project-based works. In addition, since the final product is a pipe, it usually includes coil orders that are produced from

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large slabs with maximum weights, which positively affects the efficiency of the factories [7]-[9]. As a result, the process parameters that should be used for the optimum impact energy desired to be obtained with this scientific research are pointed out.

Oil pipe steels (PSL; Product Specification levels) are labeled PSL1 or PSL2, 2 quality levels. Pipes are used in different types of environments, corrosive or non-corrosive. PSL2 quality level is preferred if the pipes are to be used in a severely corrosive environment that require material that can withstand such processing conditions. If the pipe is to be used in high pressure conditions, the weld seam must be able to withstand such a load. On the other hand, for normal services, a standard quality level pipe would be suitable for this purpose. For this reason, there are two product levels as discussed above. Depending on the environmental and economical conditions, the pipe can be selected as PSL1 or PSL2 [10].

In a study, the microstructure obtained in products rolled with different hot rolling parameters (such as different winding temperature, cooling speed, supply temperature) and their effects on mechanical properties were investigated. As a result of the investigation, it was understood that hard phases such as martensite and cementite have a negative effect on the notch impact energy of the material. [11]. In another study, similarly, the effects of the microstructure obtained with thermomechanical rolling method and different rolling temperatures, chemical analysis on the mechanic properties were investigated [12],[13]. It was investigated that the niobium alloy has a more uniform grain structure and has positive results in mechanical properties. It has been found that niobium alloy has more uniform and better mechanical properties. [14],[15]. The effect of rolling temperature on microstructure was investigated to see whether the material meets the DWTT specification. They concluded that there is no noticeable difference in the impact resistance of the products produced with two different winding temperatures such as 540 °C and 500 °C [8],[16]. In another study conducted in recent years, the behavior of winding temperature on mechanical properties of coils with different molybdenum alloy was investigated. In the examination, it was found that 0.15% molybdenum addition and decreasing the winding temperature from 560 °C to 420 °C had a positive effect on the mechanical properties [17]. The main purpose of thermomechanical rolling is to obtain the smallest grain size that can be obtained by controlling the recrystallization process. In thermomechanical rolling, the plates are first heated at about 1200 °C in annealing furnaces. The first rolling process is traditionally carried out as rolling roughly. Subsequent rolling operations are performed by selecting a temperature lower than that used in the

conventional method. The plastic deformation in this low temperature causes fine grain size and delayed sedimentation. Final rolling continues at temperatures below at which critical austenite begins to turn into ferrite during cooling (Ar3). In this way, it is necessary to apply a high amount of reducing force for low temperature rolling. The purpose of thermomechanical rolling is to control recrystallization and grain size and to improve the austenite grain structure. Due to the solubility changes in the austenite phase of titanium, the niobium and vanadium elements used as micro alloys play an important role in the thermomechanical rolling process [18]. Since the Nb-grain boundary interaction has a decelerating effect on grain growth, a decrease in grain size may occur with an increase in Nb [19]. They are known as micro alloyed elements as they are used below 0.1% by weight. These elements cause controlled sedimentation during the process, delaying recrystallization and grain size.

In this study, different thermomechanical rolling parameters were used together with different titanium and niobium alloy in order to see the effect on the structures to be obtained on the microstructure. The microstructure and mechanical properties to be obtained depending on the holding time in front of the strip mill entrance were examined. Differently from other studies, not only the parameters changed by chemical analysis or winding temperatures, but also the effects of holding time in thermomechanical rolling, conventional rolling, and thermomechanical rolling on mechanical properties were investigated. Therefore, a study in which steel analysis and hot rolling mill parameters were used together was recorded by using many parameters.

2 Material and method

The test runs were carried out in the rolling mill unit of the integrated iron and steel factory. The strength and impact resistance results of steels containing different alloying elements were examined. In experimental studies, attention has been paid to keeping the impact resistance of these two properties within the maximum limits at optimum conditions and low temperatures. Process flowchart is shown in Figure 1.

Chemical analyses for X70 material for API 5L standard is given in Table 1 and mechanical property limits are given in Table 2. Test runs cover data generated within standard chemical analysis ranges. Test runs were carried out on the samples taken after the steels poured from basic oxygen furnaces were turned into slab by continuous casting process and then produced by hot rolling method. Production was monitored with the determined trial practices and it was ensured that each line was realized within the targets.

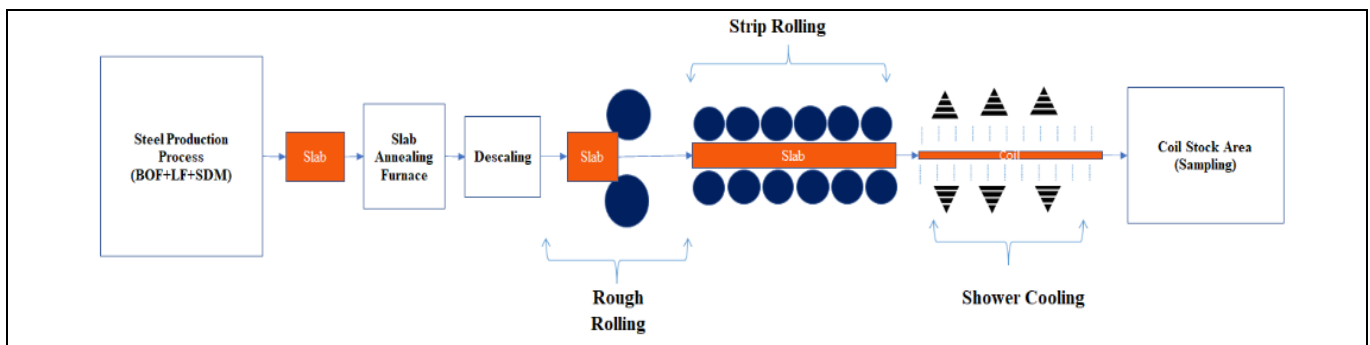


Figure 1. Process flowchart.

Table 1. Chemical analysis ranges (API 5L X70).

C (Max %)	Mn (Max %)	Si (Max %)	P(Max %)	S(Max %)	Ceq* (Max %)	V+Nb+Ti (Max %)
0.12*	1.70*	0.45	0.025	0.015	0.25	0.15

Table 2. Mechanical property limits (API 5L X70).

Re (N/mm ²)	Rm (N/mm ²)	Rt0,5/Rm (Max.)	A50 (Min. %)	Darbe (Joule) (Min.)
485-635	570-760	Max 0.93	22	0 °C'de 68 joule

2.1 Mechanical tests used

Along with high post-production strengths of X70 quality, the most appropriate chemical analysis design and rolling parameters are required for optimum impact and DWTT resistance. For this reason, obtaining a homogeneous microstructure with mechanical properties constitutes the main purpose of the study. In order for the post-rolling microstructure properties to meet the desired conditions, the nitrogen level should be kept low together with impurity elements such as low sulfur and phosphorus in steel production practices. For X70 qualities in liquid steel production process, calcium injection should be made in ladle furnaces and low pressure flotation for at least 5 minutes should be carried out for low sulfur value and inclusion modification. For the targeted low temperature toughness and microstructure, experimental designs were made on eight different (Table 3) slabs in order to see the effects such as thermomechanical rolling, conventional rolling, and winding temperature difference. The flowchart of the tests is given in Figure 2. The trial plan of the tests carried out in the study is shown in Table 3.

The process data and chemical analysis values used in this study are given in Table 4. Within the scope of the study, 8 slabs of the same thickness and width with similar chemical analyses except titanium alloy were rolled. Tensile test, impact test, DWTT and metallographic examination were performed on the samples taken from the rolled coils. During the rolling of 8 different slabs, different winding temperatures were used along with thermomechanical (T) and conventional type rolling. Thermomechanical rolling process is one of the microstructural control techniques that combine controlled

rolling and controlled cooling in order to achieve the superior steel properties such as high strength, excellent toughness, and weldability. The mechanical properties of steel as a result of this process are similar to those of traditionally rolled or forged steels after heat treatment. Temperature controlled rolling and micro alloying elements are important criteria of the process.

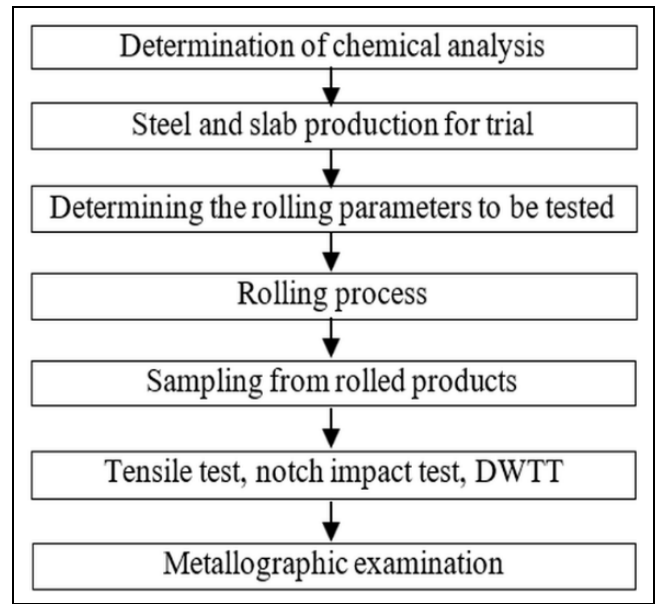


Figure 2. Test flowchart.

Table 3. Trial plan.

Sample	Coil Thickness (mm)	C (%)	Ti (%)	Furnace Discharge Temperature (°C)	Number of passes (RM)	Rough Rolling Mill Thickness (mm)	Waiting Time in Front of Strip Mill (sn.)	Rolling Type	Shower Table Cooling Mode	Run Out Table Cooling Speed
Slab 1	17.8	0.074	Low	1200	Same	54.9	-	Conventional	Same	Same
Slab 2	17.8	0.064	Low	1200	Same	47.8	100	Thermomechanics	Same	Same
Slab 3	17.9	0.067	Low	1200	Same	54.9	60	Thermomechanics	Same	Same
Slab 4	17.5	0.073	Low	1200	Same	54.9	-	Conventional	Same	Same
Slab 5	17.5	0.080	Low	1200	Same	54.9	-	Conventional	Same	Same
Slab 6	18.0	0.079	High	1200	Same	54.9	40	Thermomechanics	Same	Same
Slab 7	17.5	0.072	High	1200	Same	54.9	-	Conventional	Same	Same
Slab 8	17.5	0.079	High	1200	Same	54.9	-	Conventional	Same	Same

Table 4. Process parameters and chemical analysis of trial production.

Sample	Finishing Mill Exit Temperature (°C)	Coiling Temperature (°C)	C (%)	Mn (%)	Si (%)	Cr (%)	Ti (%)	Nb (%)
Slab 1		819	0,074	1,63	0,27	0,11	0,029	0,056
Slab 2 (T*)		833	0,064	1,62	0,27	0,12	0,023	0,055
Slab 3 (T*)		810	0,067	1,68	0,29	0,16	0,032	0,054
Slab 4		822	0,073	1,61	0,25	0,15	0,024	0,065
Slab 5		818	0,080	1,63	0,27	0,19	0,029	0,064
Slab 6 (T*)		823	0,079	1,63	0,28	0,15	0,045	0,063
Slab 7		827	0,072	1,61	0,24	0,15	0,039	0,058
Slab 8		861	0,079	1,63	0,28	0,15	0,045	0,063

*(T): Thermomechanical Rolling.

During the controlled rolling of 8 slabs, thermomechanical rolling was used for slabs 2, 3, and 6. Conventional rolling method was used for the other 5 slabs. As can be seen from the parameters given in Table 3, besides the rolling type, low and high winding temperature and titanium value were also used. At the end of all this process, tensile test at room temperature, notch impact test at -30 °C, and DWTT test at -20 °C were performed on the samples taken from the bobbins. The tensile tests were carried out on the Zwick/Roell Z600E test device, and the yield, tensile, and elongation values within the standards were measured for all the bobbins. Notch impact tests were carried out on Zwick/Roell RKP450 device with 450 joule capacity. The results measured at -20 °C in all coils except coil number 8 are within standard limits. After the microstructure examination of all coils, interpretations were made about the reason for the differences. DWT tests were carried out on the Pragma brand test device with a capacity of 55000 joules and the % ductile area value did not meet the targets except for samples numbered 2 and 4. As seen in Table 5, test results of coils numbered 2 and 4 seem to have reached the targeted strength and toughness. Although thermomechanical rolled, some coils had a brittle morphology in response to the desired tear resistance.

3 Findings and discussion

In order to see the differences in test results corresponding to process parameters, microstructure examination of all samples was performed with Nikon brand X1000 magnification optical microscope. After pickling coil samples with 2% nital solution, microstructure analysis was performed. Microstructure distribution and particle size measurements according to ASTM E112 were performed at X100, X200, and X500 magnifications in the examinations performed with Nikon optical microscope.

Figure 3 shows the examination points of the samples taken from each coil. Microstructure examination of the samples taken from the coils was carried out throughout the thickness such that the coil top surface (a), the upper edge of the center (b), the center region (c), the center bottom edge (d), and the bottom surface were 18 mm thick.

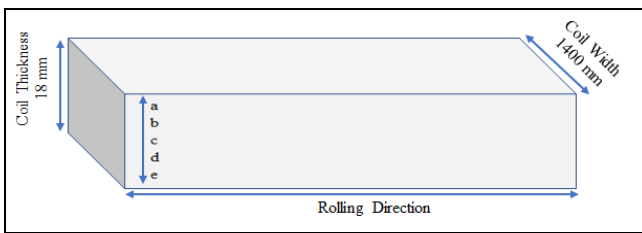


Figure 3. The image of samples defined as (a), (b), (c), (d), (e) extracted from the coil.

The microstructure images of the trial number 1 are given in Figure 3 at X500 magnification. Throughout the thickness of the coil, from top to bottom Figure 3(a) to Figure 3(e), the microstructure has polygonal ferrite as seen in the figures.

Grain size of 4 microns was measured according to ASTM E112 standard. Considering the mechanical test results given in Table 1, it is seen that the experiment was successful in general, the brittle area was measured at 70% in only 1 DWTT sample. In the light of these values, it can be said that the experiment is very close to better impact values and requirements of the standards [20]. Microstructure images also contain an almost homogeneous and uniform grain distribution and a slight segregation band parallel to the result. Generally, it is seen that polygonal ferrite and acicular Figure 4(b) and Figure (d) ferrite structures are prevalent. There is some difference in the micro-image of the upper and lower edges Figure 4(a) and Figure (b), and it can be said that the upper edge grain size is relatively smaller. This may be caused by differences in the upper and lower surface cooling degree during rolling [21]. In the central region, there is a slight level of segregation Figure 4(c).

It is thought that the segregation formation occurs during the slab casting stage depending on the casting speed and temperature parameters. [22].

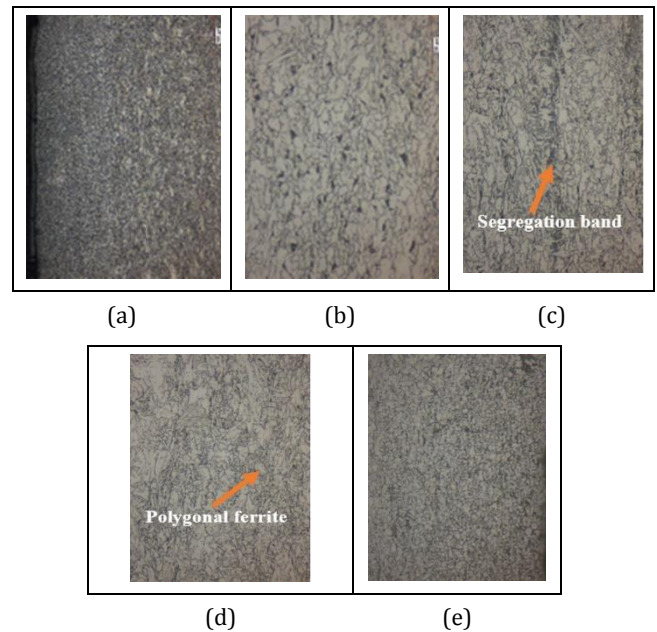


Figure 4. Slab 1 sample X500 magnified microstructure image (Grain size 12.9 (G)).

Table 5. Mechanical properties achieved as a result of the trials.

Sample	Coil Thickness (mm)	Yield Strength (485-635) (MPa)	Tensile Strength (570-760) (MPa)	% Elongation (Min 22)	Notch Impact 1 (Min 68 joule)**	Notch Impact 2 (Min 68 joule)	Notch Impact 3 (Min 68 joule)	Notch I mpact Average (joule)	DWTT 1 (Min %80)**	DWTT 2 (Min %80)**	DWTT Average (%)
Slab 1	17.8	574	676	21	254	238	232	241	91	70**	81
Slab 2 (T)	17.8	529	638	22	228	209	200	212	84	89	87
Slab 3 (T)	17.9	582	711	35	148	178	165	164	67**	62**	65**
Slab 4	17.5	579	672	36	212	216	209	212	91	93	92
Slab 5	17.5	563	698	36	198	153	181	177	83	77**	80
Slab 6 (T)	18.0	541	663	40	101	129	102	111	47**	43**	45**
Slab 7	17.5	574	701	36	153	177	169	166	50**	52**	51**
Slab 8	17.5	566	714	34	51	68	86	68	39**	42**	41**

** : Mark represent values that below the desired limit.

It is seen that the result of trial number 2 given in Figure 5 has achieved the targeted mechanical values in Table 2. In the micro structure shown in Figure 5, 5.6 micron was measured with a larger grain size than the number 1 coil micro structure. The microstructure has a ferritic microstructure from top to bottom Figure 5(a) and downward Figure 5(e) along the coil thickness as seen in figures. (As shown in the schematic image in Figure 3 It has a finer grain structure than the general structure of coil. This is due to the faster cooling of the upper and lower edges. In addition, the segregation band becomes clearer and thicker Figure 5(c). Within the scope of this information, it can be interpreted that the speed and temperatures are selected at the optimum level from the time of casting [22]. When the test process parameters were examined, it is observed that by performing thermomechanical rolling, it had the highest holding time (100 sec) (trial slab Number 2) different from other trials and consequently the coil had the lowest temperature at the strip mill entrance. It can be said that these values are beneficial in obtaining high strength and toughness value in test results.

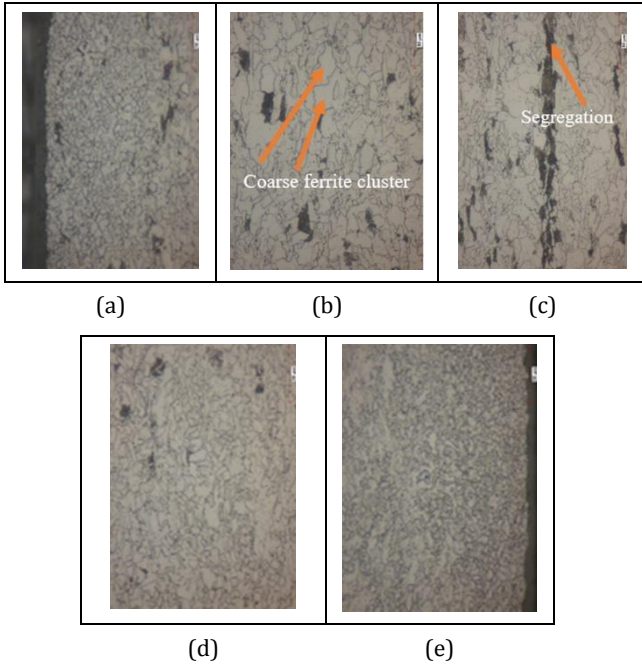


Figure 5. Slab 2 sample X500 magnified microstructure image (Grain size 12.2 (G)).

In the trial number 3 shown in Figure 6, the grain size is 4.7 microns and the grain structure consists of acicular ferrite and polygonal ferrite. In a thick area in the center, the segregation band and the difference in upper and lower edge structure draws attention Figure 6(a), Figure (b). It is seen that a finer grain structure was obtained in the lower surface Figure 6(e). Considering the results in Table 4, it is understood that its yield, tensile strength, and impact energy are suitable except for DWTT results, but it does not have sufficient tear resistance. The unsuitable DWTT results are thought to be due to the thick segregation band in microstructure Figure 6(c), the presence of coarse ferrite islets, and heterogeneous structure Figure 6(b) and Figure (d). The presence of bulging grains again reminds the importance of the presence of elements used for precipitation hardening such as titanium and niobium in these steels [20],[23].

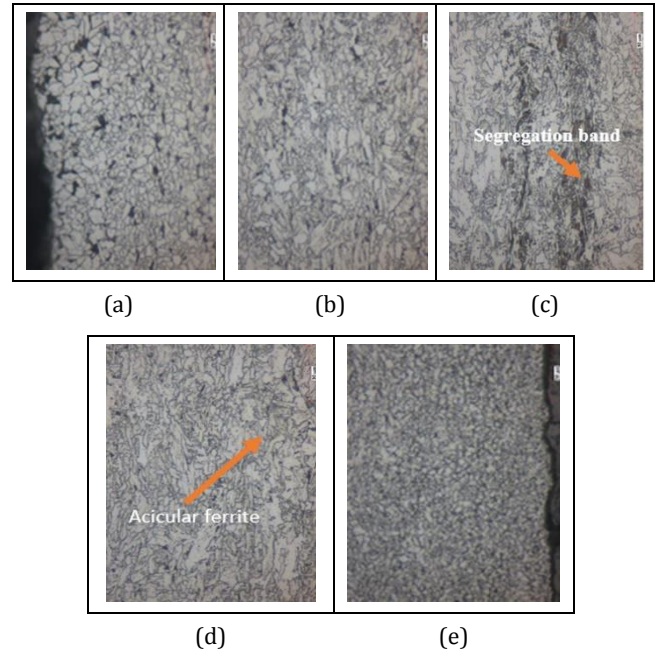


Figure 6. Slab 3 sample X500 magnified microstructure image (Grain size 12.4 (G)).

At the same time, temperature changes during deformation at recrystallization temperatures should prevent the growth of grains. As a result of the lack of these properties, coarse grains can form in the microstructure [23],[24].

The trial coil Number 4 shown in Figure 7 seems to have provided all mechanical properties (in Table 5). Throughout the thickness of the coil, from top (Figure 7-a-upper surface) to bottom Figure 7(e) lower surface), the microstructure is as seen in the figures (shown in the schematic image in Figure 4). The particle size being very fine (4.5 micron) and the effect of low winding temperature parallels the occurrence of this result. Acicular and polygonal ferrite formations are in the foreground in the structure and there is a slight level of segregation Figure 7(c) band. The top surface of the coil Figure 7(a) and the bottom surface Figure 7(b) are in fine-grained ferritic structure, and the area between the center edge Figure 7(b) and Figure (d) has a larger grain structure and in places needle-like ferrite structure Figure 7(b).

In Figure 8, there are microstructure images of the trial coil numbered 5, and although the structure is similar to the micro structure numbered 4, the coarse ferrite islets Figure 8(b) and Figure (d) draw attention. Through the thickness of the coil, the microstructure is as seen in figures from top Figure 8(a) to bottom Figure 8(e). There is a slight level of segregation band in the central region. In experiment number 5, DWTT had good results, but did not achieve the targeted value. However, it can be interpreted that the results are close to the targets with the advantage of small microstructure particle size of the trial coil numbered 5.

The thick segregation band in the central region and the perlite formation around it Figure 9(c) and Figure (d) draw attention in the microstructure image of the sample number 6 seen in Figure 9. The upper Figure 9(a) and lower Figure 9(b) surface micro view of the coil has a low grain structure and the microstructure is as seen in the figures from top Figure 9(a) to

bottom Figure 9(e) along the coil thickness. It can be seen in the hardness measurement made in the regions where the hardness is high within the segregation band Figure 9(c). The presence of large ferrite islands in the structure Figure 9(b) and (d) shows that DWTT results in the formation of brittle structure.

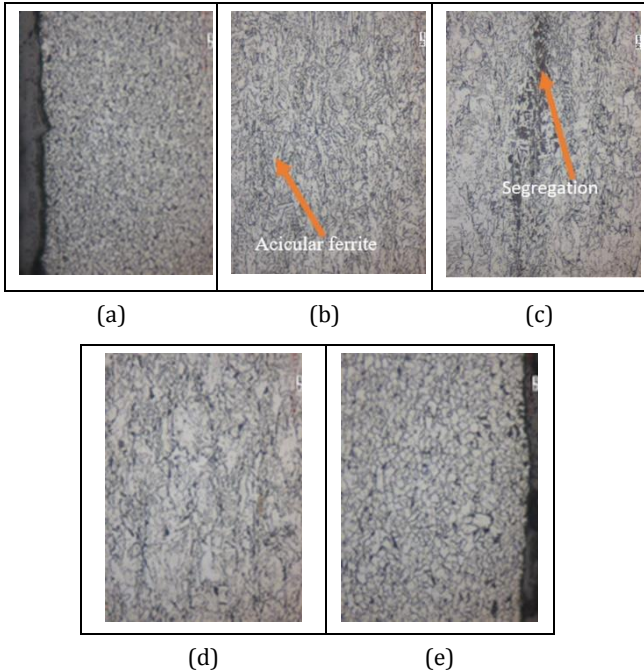


Figure 7. Slab 4 sample X500 Magnified microstructure image (Grain size 12.7 (G)).

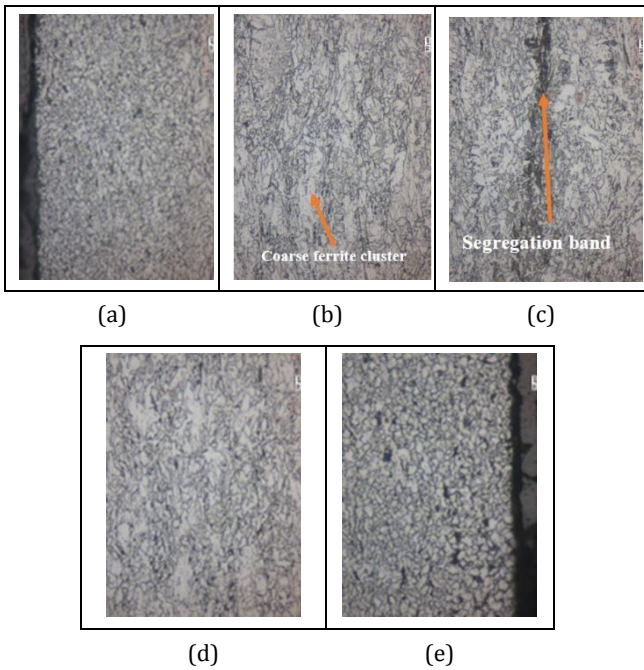


Figure 8. Slab 5 sample X500 Magnified Microstructure Image (Grain size 12.7 (G)).

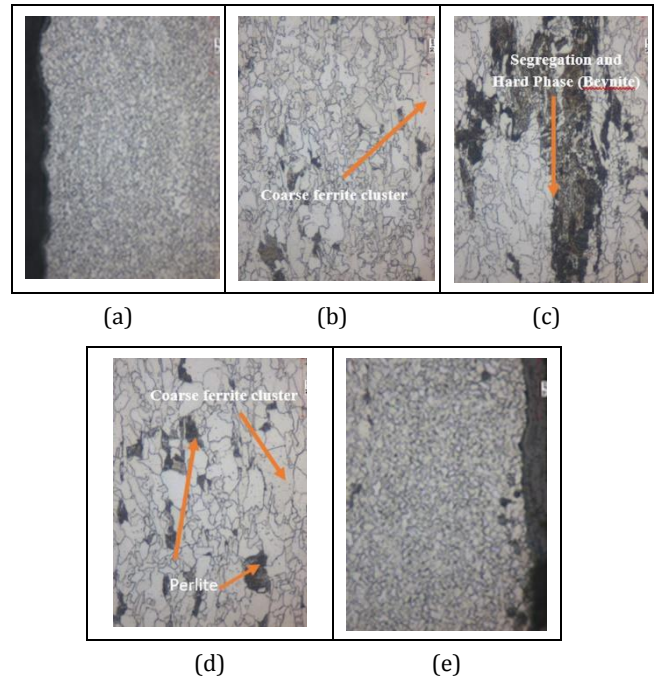


Figure 9. Slab 6 sample X500 Magnified Microstructure Image (Grain size 12.3 (G)).

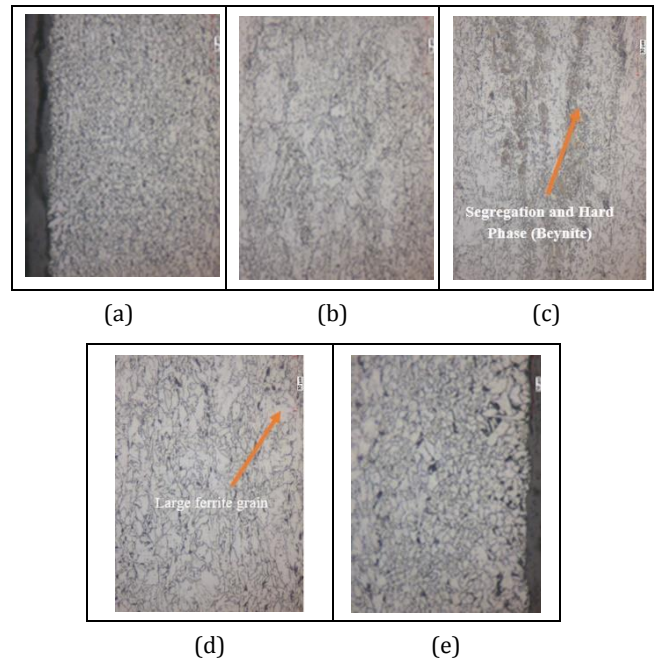


Figure 10. Slab 6 sample X500 Magnified Microstructure Image (Grain size 12.3 (G)).

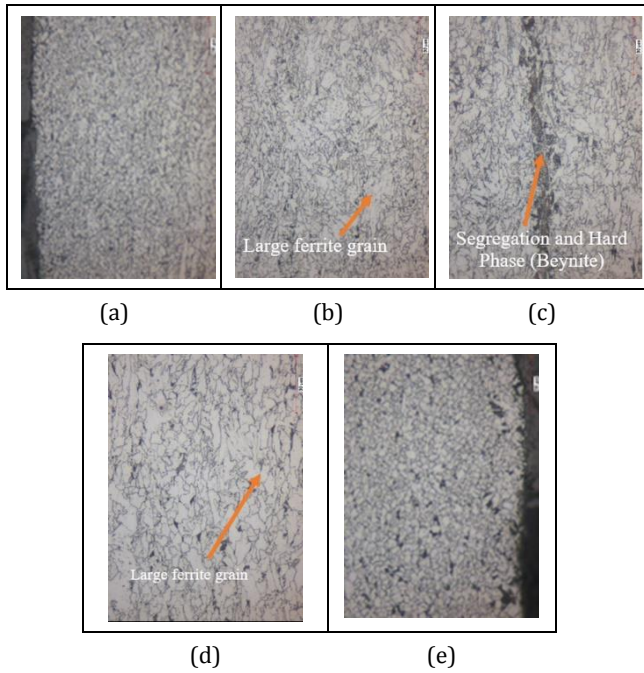


Figure 11. Slab 8 sample X500 Magnified Microstructure Image (Grain size 12.4 (G)).

The large ferrite islets in the samples in Figure 9 and Figure 10 draw attention with Figure 10(d) and Figure 11(b) and (d). The microstructure is as seen in figures from top Figure 10(a) to bottom Figure 10(e) along the thickness of the coil. The big size of the grains Figure 11(d) and the segregation formed into a hard structure in the center Figure 10(c), Figure 11(c) confirm the brittle structure as a result of DWTT.

4 Conclusion

Quality improvement studies can be carried out using different rolling and process variations between Tnr-Ar3 temperatures. In conclusion, much smaller grains were obtained with this process and the mechanical properties can be improved. With the mechanical properties obtained after thermomechanical rolling, results that meet API 5L standard requirements were obtained.

Due to the impact and DWTT results, ductile fracture surfaces were observed in the samples even at low temperatures. As understood from the microstructure analyses, it is seen that the ductile area amount after DWTT can meet the targets when the grain size is at the level of 4.5 and 4.7 microns according to ASTM E-112. In addition, the grain size is 5.6 microns and the holding time, which is a thermomechanical feature, was at the highest level in the trial production meeting the test results. On the other hand, polygonal ferrite phase distribution and slight segregation band seen in microstructure are interpreted negatively. At this stage, it will be useful to evaluate the grain structure characterization by examining the intergranular sliding systems and the angle factor between the grains in future studies.

5 Author contributions declaration

In the study carried out, Ömer Saltuk BÖLÜKBAŞI under the titles of forming the idea, designing the experiment, evaluating the results obtained, literature review; Cemre KEÇECİ contributed to the procurement of the materials used and the realization of experimental studies.

6 Ethics committee approval and conflict of interest statement

Ethics committee approval is not required for the prepared article. There is no conflict of interest with any person/institution in the prepared article.

7 References

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