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Influences of Heat Treatment Parameters on Microstructure And Mechanical Behavior of TWIP Steel

Şahlı Başkurt¹, Fatih Hayat¹, Cihangir Tevfik Sezgin^{2,*}, Sadettin Şahin³

¹Karabük University, Faculty of Engineering, Karabük, Türkiye

²Kastamonu University, Kastamonu Vocational School, Kastamonu, Türkiye

³Kırıkkale University, Faculty of Engineering and Natural Sciences, Kırıkkale, Türkiye

the untreated sample.

ARTICLE INFORMATION	ABSTRACT
Received: 21.08.2024 Accepted: 25.10.2024	In this study, the effects of different heat treatment temperatures and times on the mechanical properties and microstructure of Twinning Induced Plasticity (TWIP) steel
Keywords: TWIP steel Heat treatment Microstructure Mechanical properties	were examined. TWIP steel slabs produced by casting were shaped into plates by hot and cold rolling processes, respectively. The heat treatments were carried out at 600, 700, 800, and 900 °C for 20, 60, and 150 min. As a result of the experiments, M_3C carbide precipitates were formed instead of twinning in the tempered sheets at 600 °C and 700 °C, and twinning occurred at 800 °C and 900 °C. The microstructure analysis and mechanical test results demonstrate that the carbide precipitates prevent twinning plane formation. The Vickers hardness and tensile test results showed the intense presence of carbides at 600 °C and 700 °C and twinning at 800 °C and 900 °C. As the annealing temperature and time increased, a decrease in hardness and tensile strength

Isıl İşlem Parametrelerinin TWIP Çeliğinin Mikro Yapısı Ve Mekanik Davranışı Üzerindeki Etkileri

was observed. Elongation increased. However, as a result of annealing at 600 °C for 20 minutes, an increase in elongation and tensile strength was observed compared to

MAKALE BİLGİSİ	ÖZET
Alınma: 21.08.2024 Kabul: 25.10.2024	Bu çalışmada, farklı ısıl işlem sıcaklıkları ve sürelerinin ikizlenme kaynaklı plastisite (Twinning Induced Plasticity-TWIP) celiğinin mekanik özellikleri ve mikro yapısı
Anahtar Kelimeler: TWIP çeliği Isıl işlem Mikroyapı Mekanik özellikler	üzerindeki etkileri incelenmiştir. Dökümle üretilen TWIP çelik levhalar sırasıyla sıcak ve soğuk haddeleme işlemleriyle levha haline getirilmiştir. Isıl işlemler 600, 700, 800 ve 900 °C 'de 20, 60 ve 150 dakika süreyle gerçekleştirilmiştir. Yapılan deneyler sonucunda, 600 °C ve 700 °C'de temperlenmiş saclarda ikizlenme yerine M ₃ C karbür çökeltileri oluşmuş, 800 °C ve 900 °C'de ise ikizlenme meydana gelmiştir. Mikroyapı analizleri ve mekanik test sonuçları ayrıca karbür çökeltilerinin ikiz düzlemlerinin oluşumunu engellediğini göstermiştir. Yapılan Vickers cinsinden sertlik ve çekme testleri sonuçları 600 °C ve 700 °C'de karbürlerin varlığının, 800 °C ve 900 °C'de ise ikizlenme plakalarının yoğun olduğunu göstermiştir. Tavlama sıcaklığı ve süresi arttıkça sertlik ve çekme mukavemetinde azalma gözlemlenmiştir. Uzama ise artmıştır. Fakat 600 °C' de 20 dakika yapılan tavlama sonucunda yüzde uzama miktarı ve çekme mukavemeti değeri ısıl işlemsiz numuneye göre artış göstermiştir.

1. INTRODUCTION (GİRİŞ)

Automotive companies are under pressure from drivers and governments due to increasing demands in terms of passenger safety, CO_2 emissions, and fuel consumption. This necessitates the search for solutions that will enable the realization of these goals. For this reason, it has become a necessity for car manufacturers to both reduce fuel consumption and CO_2 emissions by reducing vehicle weight and to ensure passenger safety by increasing material strength. This innovative thinking led to the emergence of advanced high-strength steels (AHSS). One of the most important

*Corresponding author, e-mail: ctsezgin@kastamonu.edu.tr

groups of AHSS is TWIP (Twinning Induced Plasticity) steels. The high manganese content causes the structure in TWIP steels to be fully austenitic at room temperature. TWIP steels show a perfect combination of plasticity and strength with twinning which is its basic deformation mechanism [1-4]. As a result of plastic deformation, twins form within the austenite grains and these cause high mechanical behaviors such as high strength and elongation [5-9].

Heat treatment is one of the effective methods to improve the strength and elongation of TWIP steels. Escobar et al. found that the hardness of Fe-22Mn-0.45C TWIP steel, which was heat-treated at different tempering temperatures, increased with increasing cold deformation and decreasing tempering temperature [10]. Akinay and Hayat observed that the tensile and yield strength of annealed TWIP steel reduced, but its elongation increased with increasing annealing temperature [11]. Zhang et al. improved the tensile strength of TWIP steel to 1457 MPa and its elongation to 46.1% by annealing at 800 °C. In addition to the advantages of annealing, it also has the effect of forming carbide precipitates, which affect the mechanical properties of TWIP steel [8]. Although carbide precipitation increased the strength, it reduced the elongation ratio [9]. On the other hand, carbide formation is not desirable in Hadfield steels, another high manganese steel.

Hadfield steels, which are high manganese steels, contain more than 1% carbon, and therefore, carbides are formed. Combined with the influence of non-metallic inclusions, these carbides clearly reduce the ductility of cast Hadfield steel [12]. According to Stradomski, the precipitation of carbides at grain boundaries significantly reduces the impact strength of cast high manganese steel [13]. Metal carbides form at the boundaries of austenite grains and block the movement of the grains. This generally results in an unusable and brittle product [14, 15]. The air quenching provides sufficient time for carbide formation in Hadfield steels. Therefore, by quenching Hadfield steels in water after heat treatment, carbide precipitation is prevented, and a completely austenitic microstructure is formed [12]. The aim of this study is to determine the mechanical behavior and microstructure changes of this new TWIP steel depending on the heat treatment temperature and time.

2. MATERIAL AND METHOD (MATERYAL VE YÖNTEM)

2.1. Experimental Setup (Deney Düzeneği)

Table 1 shows the chemical composition of TWIP steel. The test samples were melted in a vacuum induction furnace and cast into ingots of 70 mm x 95 mm x 400 mm dimensions. Firstly, the ingots were carried out the homogenization heat treatment at 1200 °C for 6 h and hot-rolled to a plate 6 mm thick at 1100 °C. After hot rolling, the thickness of specimens was decreased by 3 mm by cold-rolled. The annealing was carried out at 600, 700, 800, and 900 °C for 20, 60, and 150 min, followed by air-quenching. The specimens were ground and polished for optical and scanning electron microscope (SEM) Carl Zeiss Ultra Plus. The samples were etched in 4% Nital solution. Optical images of the test pieces were obtained with the Nikon ECLIPSE L150 device after etching. The fracture zone of samples was detected with a field emission scanning electron microscope (FESEM). The SEM observations and multi-point EDX analyses of specimens were carried out by FESEM with an energy-dispersive X-ray spectroscopy (EDX) analysis system. XRD analysis was carried out on the Rigaku Ultima IV Brand XRD Device in the 20-90 °C temperature range with a scanning speed of 5 degrees/min. The tensile tests were applied at room temperature with 2 mm/min crosshead speed by an MTS Servohydraulic test machine (100kN). Vickers hardness measurements were carried out by Shimadzu HMV hardness (HV 0.2). The sample nomenclatures are shown in Table 2.

Table 1. Chemical composition of the TWIP steel (wt.%)

С	Si	Mn	Р	S	Cr	Al	Fe
0.582	0.626	24	< 0.03	< 0.005	0.1	0.002	balance

Temperature (°C)	Time (min.)	Sample
600	20	1
600	60	2
600	150	3
700	20	4
700	60	5
700	150	6
800	20	7
800	60	8
800	150	9
900	20	10
900	60	11
900	150	12

 Table 2. Sample list of heat-treated TWIP (Isıl işlemli TWIP çeliğinin numune listesi)

3. EXPERIMENT AND OPTIMIZATION RESULTS (DENEY VE OPTIMIZASYON SONUÇLARI)

Figures 1a-c show the optical views of samples 1,2 and 3, respectively. As can be seen, while the grain boundaries were not clearly visible after cold rolling in the heat treatment lasting 20 minutes at 600 °C, the grain structures became clear in the 60-minute heat treatment, and the grain sizes increased when the heat treatment time was increased to 150 minutes. Increasing the heat treatment time made the austenite grains more distinct and larger. However, a clear austenite structure could not be determined as in the heat treatments performed at 800 and 900 °C. Figures 2a-d show samples 3, 8, 5, and 12 optical views, respectively. While the austenite grain boundaries were unclear in the heat treatments carried out at 600 and 700 °C, the austenite grain boundaries became clear at 800 °C and above, and thermal twin bands appeared. When Figures 2b and 2d are compared, it is seen that austenite grains increase with increasing temperature and time.



Figure 1. Microstructure views of sample; a) 1, b) 2, c) 3 (Numunelerin mikroyapı görüntüleri; a)1, b)2,c)3)

Figure 3 shows the SEM images of the microstructure obtained from 60 and 150 min of heat treatment for each temperature. In Figure 1a, it was mentioned above that austenite grain structures were not clear as a result of heat treatment at 600 °C for 20 minutes. In Figure 3, austenite grain structures became more clear in heat treatment at 600 °C for 60 minutes, while austenite structures were fully formed in heat treatment at the same temperature for 150 minutes. While the austenite grain structure was not clear in the 60-minute heat treatment at 700 °C, the grain structures became clear when the time was increased to 150 minutes. It was also determined that the carbide structure was formed in the form of white dots. While the austenite grain structure and twinnings could not be determined from the SEM photographs in the 20-minute heat treatments at 800 and 900 degrees, as can be seen in Figure 3, as a result of the 60 and 150-minute heat treatments for both temperatures, both the austenite grain structures became clear and twinnings occurred within the austenites.



Figure 2. Microstructure views of sample; a) 3, b) 8, c) 5, d) 12 (Numunelerin mikroyapı görüntüleri; a)3, b)8,c)5, d)12)





Figure 3. SEM images of samples resulting from 60 and 150 minutes of heat treatment at each temperature

Figures 4a-d show samples 2, 6, 8, and 12 SEM micrographs, respectively. The microstructure of the sample annealed at 600 (sample 2) and 700 °C (sample 6) not only contains carbide precipitates but also contains a few mechanical twins in the austenite grains (Fig. 4a-b).



Figure 4. SEM micrographs of sample; a) 2, b) 6, c) 8, d) 12 (Numunelerin SEM görüntüleri; a)2, b)6, c)8, d)12)

As seen in Figure 5, twinning is observed when the image is zoomed in. Usually, twinning mechanisms obtained due to deformation reach a more distinct and regular orientation due to heat treatment. However, carbide formation prevented the orientation of these twinning mechanisms, and twinning formation was not obtained as desired. On the other hand, in Fig. 4c-d, twin bands were observed in TWIP steels annealed at 800 (sample 8) and 900 °C (sample 12). Singon Kang et al. observed that carbides were formed in TWIP steel (Fe–18Mn–0.6C–1.5Al) between 700 °C and 800 °C. They reported that these carbides were dissolved and dispersed at 800 °C, and twinning planes were formed in austenites [16].



Figure 5. SEM view of sample 6 (Numune 6'nın SEM görüntüsü)

Figure 6a and 6b show the XRD results of samples 6 and 12, respectively. In the XRD results, the presence of carbides seen in the SEM images of TWIP steels annealed at 600 and 700 degrees was detected. Although carbide formation was not clearly visible in SEM images it was also

detected in TWIP steels annealed at 800 and 900 °C. Carbide formation may have occurred due to quenching in air after annealing.



Figure 6. XRD results of sample; a) 6, b) 12 (Numunelerin XRD sonuçları; a) 6, b) 12)

In addition, when the elemental analysis of the white circular structures in the picture is examined in the EDX analysis shown in Figure 7, it is seen that they are metal carbides.



Figure 7. EDX analysis of sample 6 (Numune 6'nın EDX analizi)

Table 3 shows the tensile test results of TWIP steel. As the table shows, the yield and tensile strength of TWIP steel decrease as temperature and time increase. On the other hand, the elongation increases. In sample 1, there was an improvement in yield, tensile strength, and elongation compared to the sample without heat treatment. In samples 6 and 9, an increase in tensile strength values was observed with increasing heat treatment time. The reason for this increase may be due to a more refined carbide distribution during the 150-minute heat treatment of the samples compared to the 20 and 60-minute heat treatments. Dagoberto B. Santos et al. found the highest yield strength as 1081.0 ± 15.3 MPa in the cold-rolled and unheated-treated 24 Mn TWIP steel sample. The highest elongation value was 58.4 ± 2.3 in the sample annealed at 850 °C. The lowest tensile value was 662.5 ± 8.0 MPa in the sample annealed at 850 °C [17]. The increase in the size of the austenite grains and the dissolution of the carbides at high-temperature values causes decreasing in tensile

and yield strength [18]. Table 3 also shows the Vickers hardness test results of TWIP steel. In the applied Vickers hardness test, it was observed that increasing temperature and time decreased the hardness of the steel. Dagoberto B. Santos et al. investigated the hardness values of TWIP steel with a composition of 24Mn–3Al–2Si–1Ni–0.06C wt.% between 100 °C and 850 °C. They reported that the values varied between 180 HV and 360 HV. It was observed that the cold-rolled sample reached the highest value. The lowest hardness value was measured in the heat-treated sample at 850 °C with a value of 180 HV [17]. In the study conducted by Singong Kang et al. [16], it was stated that grain growth and hardness decrease in the samples were directly related to the dissolution of M_3C carbide precipitates.

Sample number	Heat treatment temperature (°C/min)		Yield Strength (MPa)	Tensile strength (MPa)	Elongation (%)	Hardness (HV 0.2)
	No treatment		972 ± 10	1324 ± 10	2	476
1	600	20	955 ± 10	1342 ± 10	12 ± 2	485
2	600	60	588 ± 10	992 ± 10	14.1 ± 2	442
3	600	150	534 ± 10	946 ± 10	24 ± 2	421
4	700	20	423 ± 10	903 ± 10	34 ± 2	414
5	700	60	453 ± 10	870 ± 10	24 ± 2	410
6	700	150	494 ± 10	946 ± 10	27 ± 2	375
7	800	20	368 ± 10	893 ± 10	47 ± 2	392
8	800	60	320 ± 10	838 ± 10	56 ± 2	388
9	800	150	395 ± 10	911 ± 10	57 ± 2	327
10	900	20	324 ± 10	826 ± 10	55 ± 2	379
11	900	60	276 ± 10	773 ± 10	58 ± 2	309
12	900	150	246 ± 10	695 ± 10	60 ± 2	301

Table 3. Tensile test results (Çekme testi sonuçları)

As can be seen from the SEM images of the fracture surface, the heat-treated sample at 600 °C and 700 °C showed a more brittle fracture. However, while wider and deeper cavities (dimples) are seen in the fracture surface images of the heat-treated sample at 600 °C and 700 °C, a completely ductile fracture morphology is seen at temperatures of 800 °C and 900 °C. This proves that the samples exhibited more ductile behavior as the heat treatment temperature increased. SEM images of the fracture surface resulting from the tensile test are given in Figure 8.



Figure 8. SEM images of the fractured surfaces of the samples; a) 3, b) 6, c) 9, d) 12

4. CONCLUSIONS (SONUÇLAR)

The following results were observed in this study:

Carbide precipitations were observed in the structure after heat treatment at 600 °C and 700°C. Carbide precipitations dissolved with increasing temperature, and very few carbide precipitations were observed in samples heat treated at 800 °C and 900 °C. The highest values in the tensile test and hardness test results were observed in sample 1 (samples heat treated at 600 °C for 20 minutes). The lowest values were observed in sample 12 (samples heat treated at 900 °C for 150 minutes). It was observed that hardness and tensile values decreased and elongation values increased with increasing temperature and time. Increasing temperature and time cause the dissolution of metal carbides. As found in this study, Zhang et al. observed that the tensile strength decreased as the temperature increased in TWIP steel, which they annealed at 750, 800 and 850 °C for 10 minutes [8]. Tewary et al. observed that the tensile strength and hardness of TWIP steel decreased as the annealing temperature and time increased. They attributed this to grain coarsening [19].

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