

Investigation of Static Strain Aging Behaviour of AISI 304 Austenitic Stainless Steel

Muhammet Anıl KAYA¹, Cengiz Görkem DENGİZ^{2*}, Kenan BÜYÜKKAYA³

Abstract

In this study, the change in the material's mechanical properties by the static strain ageing process after the pre-deformation applied to AISI 304 austenitic stainless steel was investigated and analysed. The samples were first applied to 5% pre-deformation and then annealed in the oven at 150°C, 250°C, 400°C and 550°C in separate groups for 30 and 60 minutes. The data obtained by performing hardness and tensile tests after static strain ageing were compared, and the interaction between test parameters and these parameters was investigated by performing Pareto analysis. As a result of the study, the highest tensile strength and hardness values were observed in the samples applied static strain ageing for 60 minutes at 150°C. The yield and tensile strengths of the as-received material increased by 10% with the ageing process. It was determined that these values decreased from 400°C. In addition, as a result of Pareto analysis, it was determined that the temperature value is the most critical parameter affecting the results.

Keywords: AISI 304, Static strain ageing, Pareto, Tensile test.

AISI 304 Östenitik Paslanmaz Çeliğin Statik Yaşlanma Davranışının İncelenmesi

Öz

Bu çalışmada AISI 304 östenitik paslanmaz çeliğe uygulanan ön deformasyonun ardından statik gerinim yaşlandırması işlemiyle malzemenin mekanik özelliklerindeki değişim incelenmiş ve analiz edilmiştir. Numunelere önce %5 ön deformasyon uygulanmış sonrasında 30 ve 60 dk boyunca ayrı gruplar halinde 150°C, 250°C, 400°C ve 550°C sıcaklıklarda fırında tavlansmıştır. Yapılan statik gerinim yaşlandırması sonrası sertlik ve çekme deneylerinin yapılmasıyla elde edilen veriler karşılaştırılmış, deney parametreleri ve bu parametreler arasındaki etkileşim Pareto analizi yapılarak araştırılmıştır. Çalışma sonucunda en yüksek çekme dayanımı ve sertlik değeri 150°C sıcaklıkta 60 dk boyunca statik gerinim yaşlandırması uygulanan numunelerde görülmüştür. Yaşlandırma işlemiyle, satın alınmış malzemeye göre akma ve çekme dayanım değerleri %10 artmıştır. Bu değerlerde 400°C sıcaklıktan sonra azalma tespit edilmiştir. Ayrıca Pareto analizi sonucunda sonuçları etkileyen en önemli parametrenin sıcaklık değeri olduğu belirlenmiştir.

Anahtar Kelimeler: AISI 304, Statik gerinim yaşlanması, Pareto, Çekme testi.

¹Giresun Üniversitesi, Teknik Bilimler Meslek Yüksekokulu, Giresun, Türkiye, anil.kaya@giresun.edu.tr

²Ondokuz Mayıs Üniversitesi, Makine Mühendisliği Bölümü, Samsun, Türkiye, gorkem.dengiz@omu.edu.tr

³Giresun Üniversitesi, Teknik Bilimler Meslek Yüksekokulu, Giresun, Türkiye, kenan.buyukkaya@giresun.edu.tr

¹<https://orcid.org/0000-0002-5717-0698> ²<https://orcid.org/0000-0003-1308-3223> ³<https://orcid.org/0000-0002-8263-0756>

1. Introduction

Stainless steels are frequently used in today's industrial applications due to their many superior properties. Austenitic stainless steels is most preferred group in the stainless steel class. Austenitic stainless steels are formed by the essential alloying elements of chromium and nickel also widely used due to their significant superiority over other stainless steel groups. These advantages can be said as corrosion resistance, high ductility, good formability, high strength and toughness (Altan Özbek et al., 2017). Austenitic stainless steels contain 12-25% Cr, 8-25% Ni and up to 20% Mn in their composition. These steels have a face-centred cubic lattice structure also cannot be hardened by heat treatment. They preserve their austenitic internal structure at room temperature and high temperatures. They are preferred in machine parts because of their advantages, such as increasing their strength and high corrosion resistance by cold forming methods. 304 stainless steel is a type of high alloy steel widely used in the austenitic stainless steel group. The chemical composition of 304 stainless steel includes nickel in addition to chromium. 304 stainless steel is used in various applications, including home and commercial kitchen equipment, aerospace and automotive equipment, heat exchanger, and electrical enclosures for sensitive electrical equipment (Wang and Li, 2003; Gupta et al., 2013; Tabin, 2021).

Strain ageing is among the strength-enhancing mechanisms and that a mechanical behaviour related to the concept of yield point that occurs in low carbon steels and appears in metals with a distinct yield point. Strain ageing is the process by which metallic materials are subjected to cold deformation, followed by annealing, usually at low temperatures or by holding them for a long time at room temperature. As a result of the realisation of this process at appropriate values, it is seen that the strength values of the material increase and its ductility decreases. (Dieter, 1961; Yurtışık, Batıgün and Gürbüz, 2010; Altuntaş, Özer and Gürel, 2020). Strain ageing is divided into static and dynamic ageing. Static ageing is defined as ageing after plastic deformation, and dynamic ageing is defined as ageing occurs during plastic deformation (Stewart and Jonas, 2004; Kaçar, Emre and Sinoplu, 2011).

It is known that the interstitial and foreign atoms in the crystal lattice structures of some materials tend to agglomerate in the dislocation regions (tension regions), locking and inhibiting the dislocation movement (A. H. Cottrell and B. A. Bilby, 1949). In the strain ageing mechanism, the dislocations in the material are released from the self-locking atoms with the applied plastic deformation. Dislocation motion also stops by removing the load on the material or stopping the deformation. If the deformed material is annealed at a low temperature for a specific time or kept at room temperature for a long time, the interstitial and foreign atoms in the crystal lattice structure begin to move the areas in the dislocation regions with an increasing number of repetitions with the effect of temperature and diffusion mechanism. As the increases in agglomeration, the dislocations

are recaptured, and their motion is locked. Locked dislocations provide increased material strength. (Lee, Choi and Nam, 2009; Mola et al., 2021; Çetin, 2022). The arrangement of interstitial and foreign atoms in the dislocation zones with the diffusion mechanism is called the Cottrell atmosphere (Zhao, De and De Cooman, 2001; Samek et al., 2020).

This study aims to examine the change in the strength of AISI 304 austenitic stainless steel with 5% pre-strain applied at different temperatures and times. The changes between the obtained data were determined, and the experimental parameters and the interaction between these parameters were revealed by performing Pareto analysis. A Pareto chart is a bar chart where the plotted values are arranged from largest to smallest (Minitab 18 Support, 2010). The most frequently occurring defects, the most common causes of defects, or the most frequent causes of customer complaints can be identified with the Pareto chart.

2. Material and Method

2.1. Material

In the study, a 0.5 mm thick AISI 304 (1.4301 or X5CrNi18-10) austenitic stainless steel sheet plate produced by the cold rolling method was purchased. Samples with 60 x 60 mm dimensions given in Figure 1 were taken from AISI 304 quality stainless steel sheet plate by laser cutting, and chemical analysis was carried out.

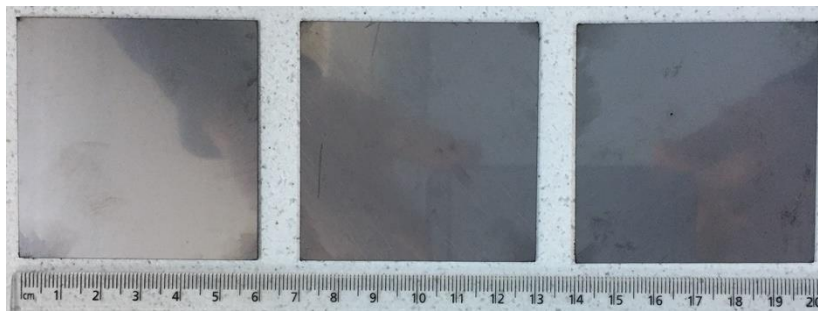


Figure 1. AISI 304 sheet samples for chemical analysis

According to standard and chemical analysis, the chemical composition (wt. %) of the AISI 304 stainless steel are given in Table 1. It is seen that the analysis results are compatible with the component values given in the standards.

Table 1. Chemical analysis results and standard composition of AISI 304 stainless steels

Material		C	Mn	P	wt. %		Cr	Ni
					S	Si		
AISI 304	Standard composition	Max. 0.08	Max. 2.0	Max. 0.045	Max. 0.03	Max. 0.75	18.0-20.0	8.0-10.5
	Analysis results	0.0069	1.14	0.037	0.0095	0.423	18.249	8.012

2.2. Pre-deformations and Tensile Tests

Pre-deformation should be applied to the samples before the static strain ageing process. After the pre-deformation process, the tensile test was performed to determine the change in mechanical properties. For this reason, the samples were prepared under ASTM E8 tensile test standards (ASTM E8, 2010). Sample dimensions determined according to the standard are given in Figure 2a. The sample cut on sheet metal plates is shown in Figure 2b.

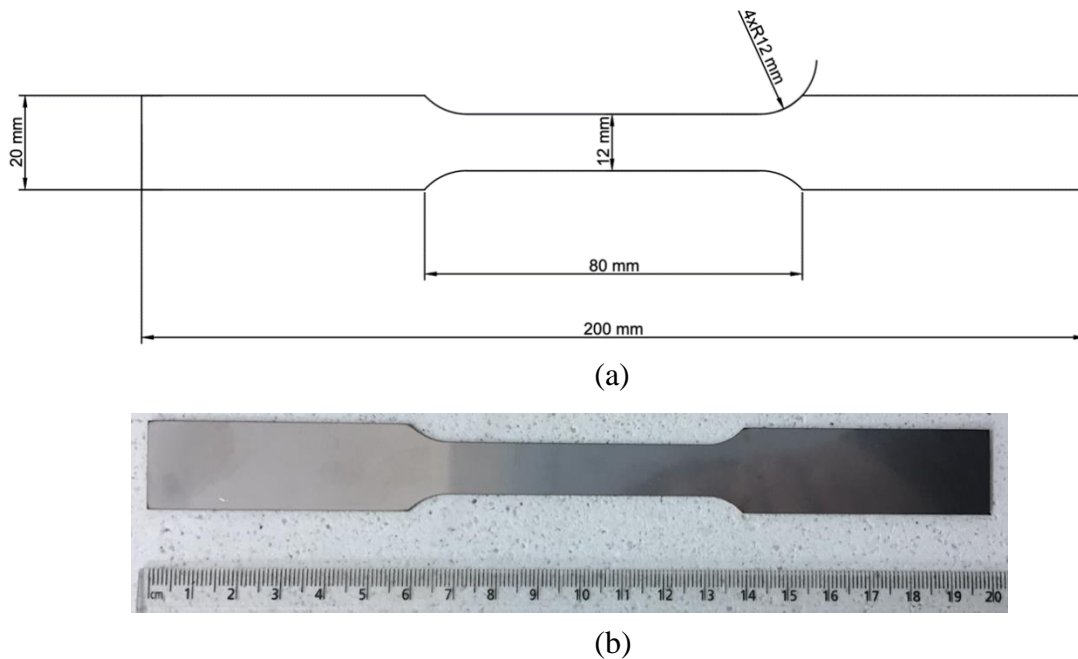


Figure 2. Sample dimensions (a), test sample (b) according to ASTM E8 standard

The prepared tensile specimens were pre-deformed by stretching 5% before annealing at different temperatures. The number of α' -martensites is minimal when the pre-strain is below 5%. Pre-deformation processes and tensile tests were carried out on Instron 5982 model 100 kN loading capacity tensile-compression test device located in Karadeniz Advanced Technology Research Center (KITAM). In the pre-deformation processes, the sample gripped to the device was stretched until it deformed 5%. The amount of deformation was measured precisely with an extensometer attached to the sample. The pre-deformation process was carried out with a 10 mm/min crosshead speed. After

pre-deformation, the samples removed from the device were heat treated. Heat treatment details are given in the next section.

After the heat treatments, the samples were again attached to the tensile device. As in the pre-deformation process, the tensile tests were carried out at a 10 mm/min crosshead speed. The tensile test continued until the samples were damaged. The mechanical properties of the samples were determined from the stress-strain diagrams after the test. Experiments were performed in 3 repetitions to minimise experimental errors.

2.3. Heat Treatments

AISI 304 stainless steel tensile specimens with 5% pre-deformation were applied to heat treatment ageing at different temperatures and times. Considering the recrystallisation temperature of the material, temperatures of 150°C, 250°C, 400°C and 550°C were selected and annealed in the furnace in separate groups for 30 and 60 minutes. In order to obtain the values with the least error, 3 samples were annealed in the furnace for each temperature and time value. The tensile samples were put into the furnace when the furnace reached the determined temperature, removed from the furnace when the applied time was reached, and allowed the samples to cool in the air. Annealing heat treatments were carried out in a programmable Protherm laboratory type furnace with a maximum temperature of 1150°C and an internal volume of 45 litres.

2.4. Hardness Tests

Vickers hardness measurement method was used in hardness tests. The Mitutoyo HM brand device made hardness measurements under 0.5 kg load (HV 0.5). Since the samples are thin, hardness indentation at higher loads causes deformation on the sheet to be seen on the backside. For this reason, a load that will not create deformation on the other side of the sheet has been selected. Vickers hardness measurement method was used in hardness tests. Hardness was determined by taking the average of 5 hardness measurements for each sample.

3. Results and Discussion

3.1. Pre-Deformation Processes

Before annealing at different times and temperatures, pre-deformation was applied to all tensile specimens by lengthening 5% in the tensile device. 5% pre-deformed specimen is shown in Figure 3.

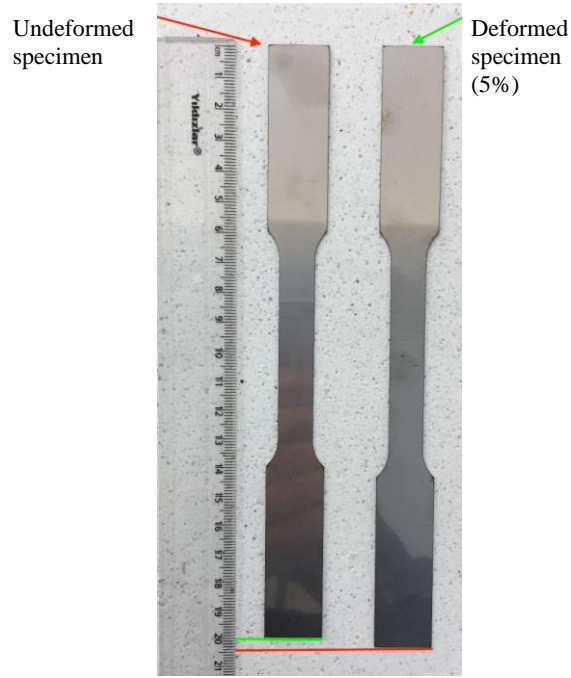


Figure 3. The pre-deformed tensile test specimen

3.2. Hardness Tests

In the measurements made in the micro-hardness device, the average hardness value of the AISI 304 quality stainless steel sheet without pre-deformation and heat treatment was found 175 HV. The image of the hardness indentation on the surface of the AISI 304 quality stainless steel is given in Figure 4.

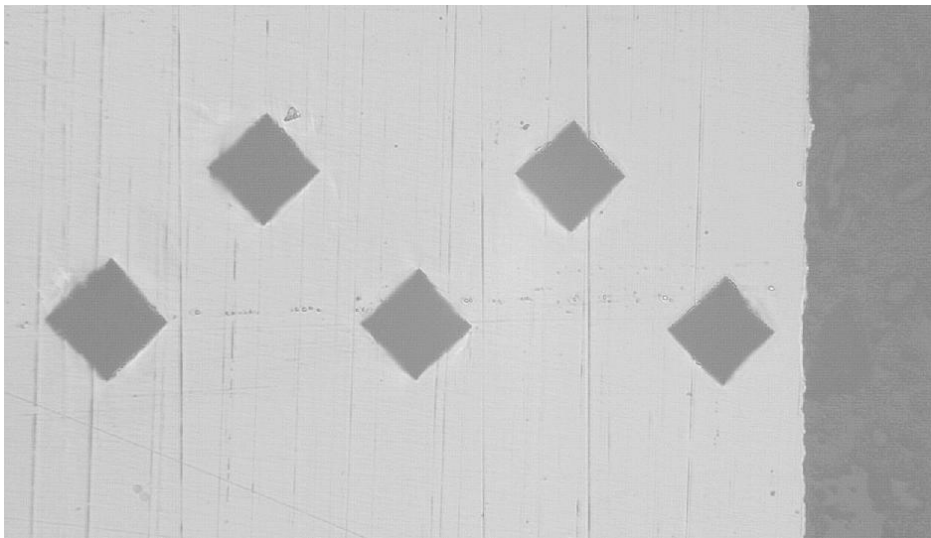


Figure 4. Hardness indentation of AISI 304 quality steel

The average hardness taken from the samples, which were subjected to 5% pre-deformation and heat treatment at different times and temperatures, is given in Figure 5. An increase in hardness is observed in the samples that are heat-treated up to 400°C compared to the sample as received. This increase occurs when free interstitial atoms and hard structures such as carbide in the material are directed to the dislocation regions with increased temperature. These structures prevent dislocation movement and cause an increase in dislocation density. The decrease in hardness after 400°C can be stated as the inability of the gradually increasing precipitate sizes in the material structure to sufficiently cover the dislocations due to excessive ageing (Lee and Zuidema, 1994). It is expected that the hardness of the steel will also increase by increasing the amount of cold deformation applied to the material.

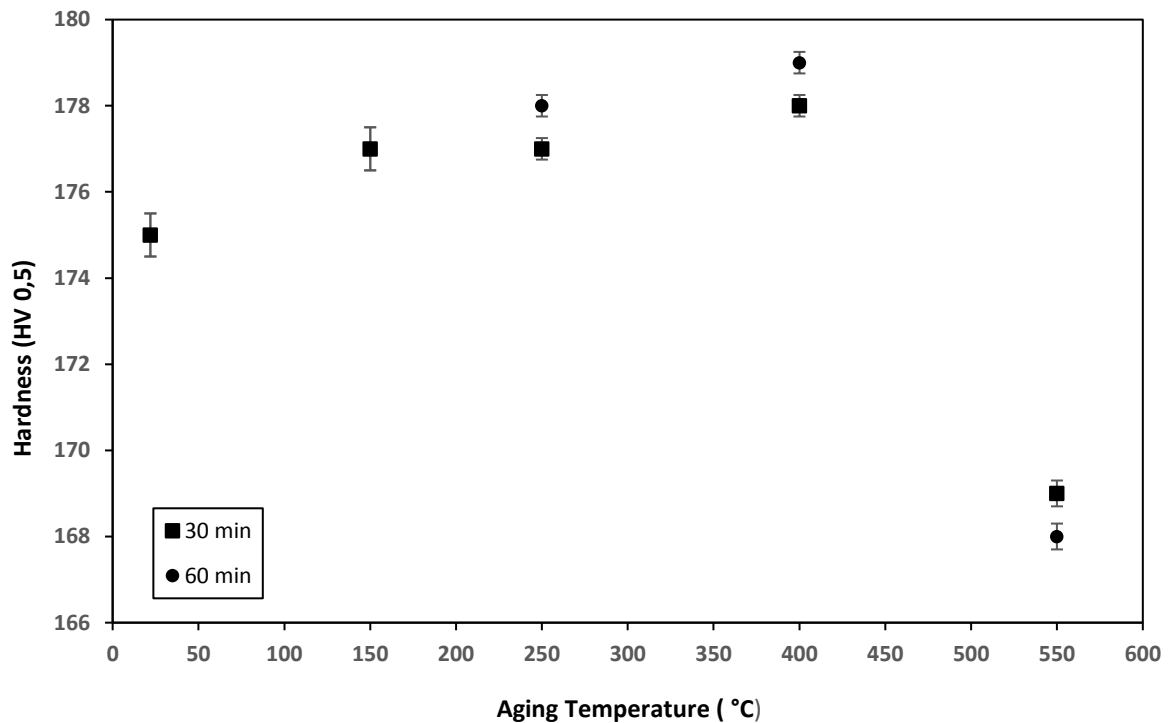


Figure 5. The hardness of the specimens depends on the same processing temperature and different ageing times

3.3. Tensile Test Results

The changes in the yield and tensile strength of the material by ageing the 5% pre-deformed AISI 304 stainless sheet specimen at different temperatures and times are shown with stress-strain diagrams in Figure 6 for 30 minutes ageing time and Figure 7 for 60 minutes. In Figure 6, the tensile strength (UTS) is 716 MPa for the undeformed specimen, 788 MPa for the specimen aged at 150°C for 30 minutes after 5% pre-deformation, 782 MPa for the specimen aged at 250°C for 30 minutes, 791 MPa for the specimen aged at 400°C for 30 minutes and 776 MPa for the specimen aged at 550°C

for 30 minutes. According to the test results of all specimens, the highest tensile strength was found in the specimens that were statically aged for 30 minutes at 400°C.

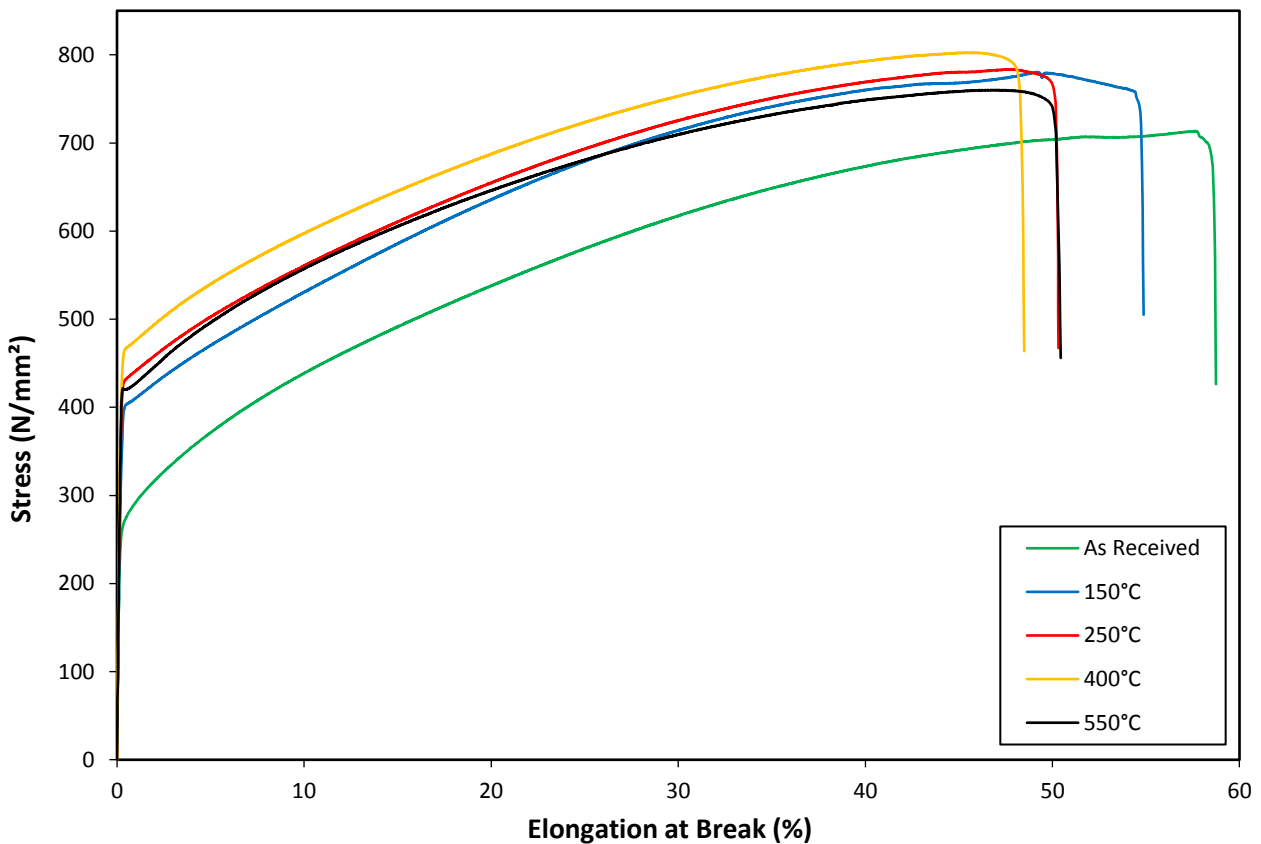


Figure 6. Stress-strain diagram of specimens aged for 30 minutes at different temperatures after 5% pre-deformation

In Figure 7, the tensile strength (UTS) is 716 MPa for the undeformed specimen, 796 MPa for the specimen aged at 150°C for 60 minutes after 5% pre-deformation, 785 MPa for the specimen aged at 250°C for 60 minutes, 782 MPa for the specimen aged at 400°C for 60 minutes and 779 MPa for the specimen aged at 550°C for 60 minutes. According to the test results of all specimens, the highest tensile strength was found in the specimens that were statically aged for 60 minutes at 150°C.

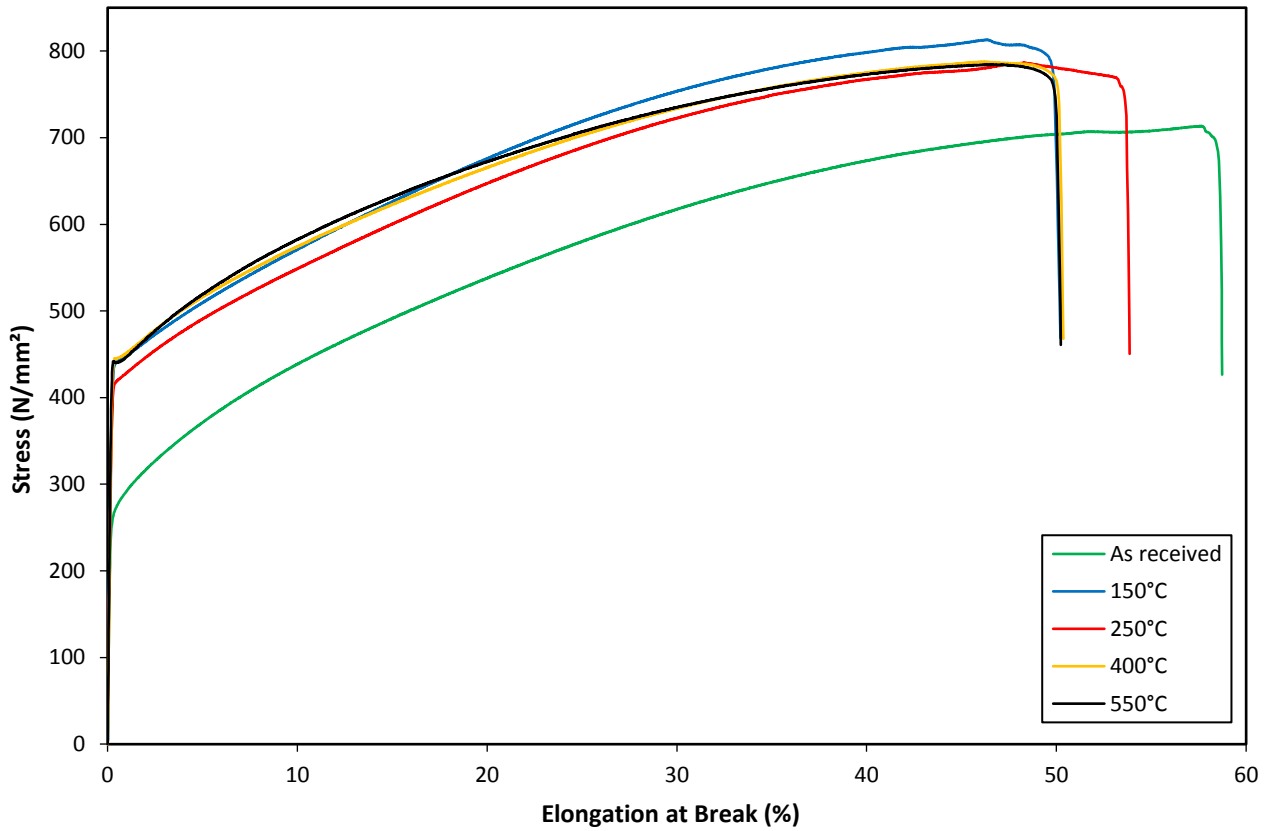


Figure 7. Stress-strain diagram of specimens aged for 60 minutes at different temperatures after 5% pre-deformation

In Figure 8, the variation of the maximum tensile strengths of the samples according to different ageing temperatures and times is given. According to different processing times, the highest tensile strength value was obtained as 796 MPa in the statically aged sample at 150°C for 60 minutes. In addition, it was observed that the tensile strength of the sample aged at 400°C for 30 minutes was close to the highest tensile strength.

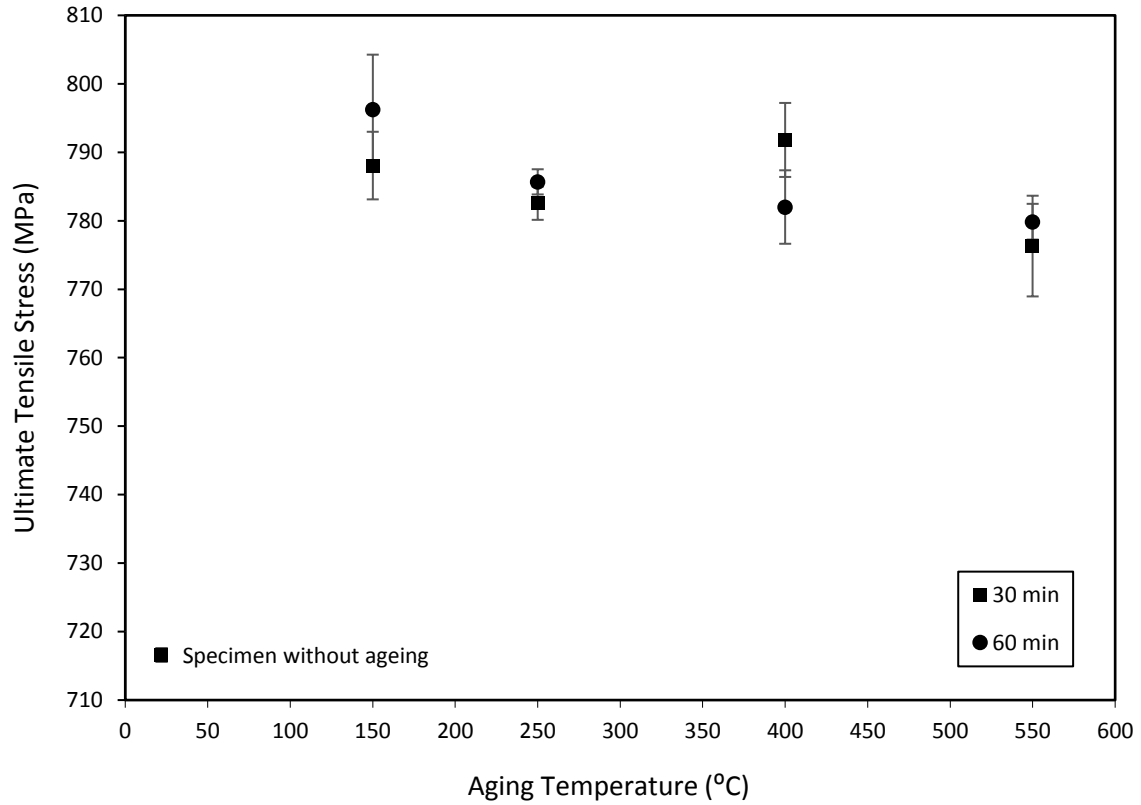


Figure 8. Tensile strength values of samples depending on different ageing times

In Figure 9, the % elongation at break of the aged samples was compared. When the graph is examined, it is observed that the % elongation at break decreases with the increase in the ageing temperature, although the % elongation at break values are close to each other according to different ageing temperatures and times.

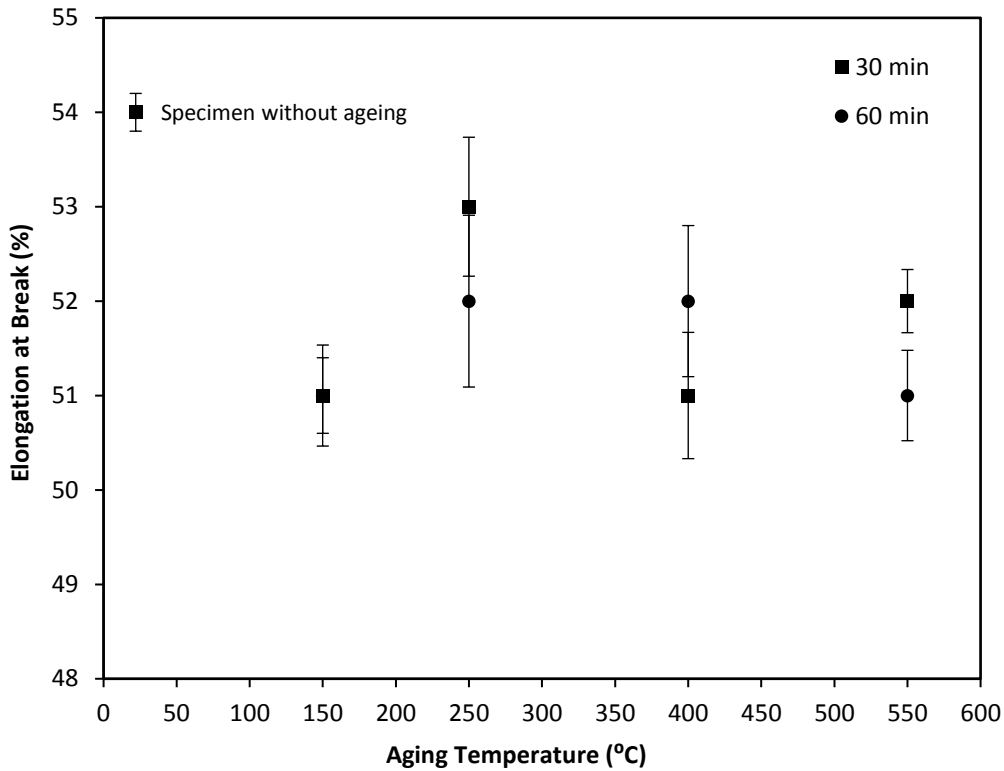


Figure 9. % elongation at break depending on the different aging times of the specimens

According to the stress-strain diagrams, an increase in yield and tensile strengths and a decrease in % elongation at break were observed in the samples with static ageing heat treatment after 5% pre-deformation in general, compared to the as-received sample. This shows that the static strain ageing heat treatment effectively affects AISI 304 stainless steel. Interstitial atoms in the microstructure and precipitates formed by the effect of temperature prevent dislocations by ageing heat treatment. In addition, the α' -martensite structure, which gradually increases in the microstructure after cold rolling and pre-deformation, significantly affects the strength increase of the material (Lee et al., 2009).

3.3. Pareto Analyse Results

The results obtained from the tensile tests and hardness measurements were analysed in a statistical program. In Figure 10, the experimental parameters and the interaction between these parameters are presented graphically by using Pareto analysis. Pareto analysis can be defined as the separation of high importance from unimportant factors affecting a result. Pareto analysis was performed in this study at a 95% confidence interval ($\alpha=0.05$). Parameters crossing the red dashed line in the given graphs show the factor that has the most significant impact on the relevant result. It is seen that the results obtained from the hardness and tensile tests are affected mainly by the temperature. The interaction of the time factor and temperature time does not significantly affect the

experimental results. Although it is seen in Figures 10b and 10d that the temperature-time interaction affects the results more than the annealing time, this is not a statistically significant effect.

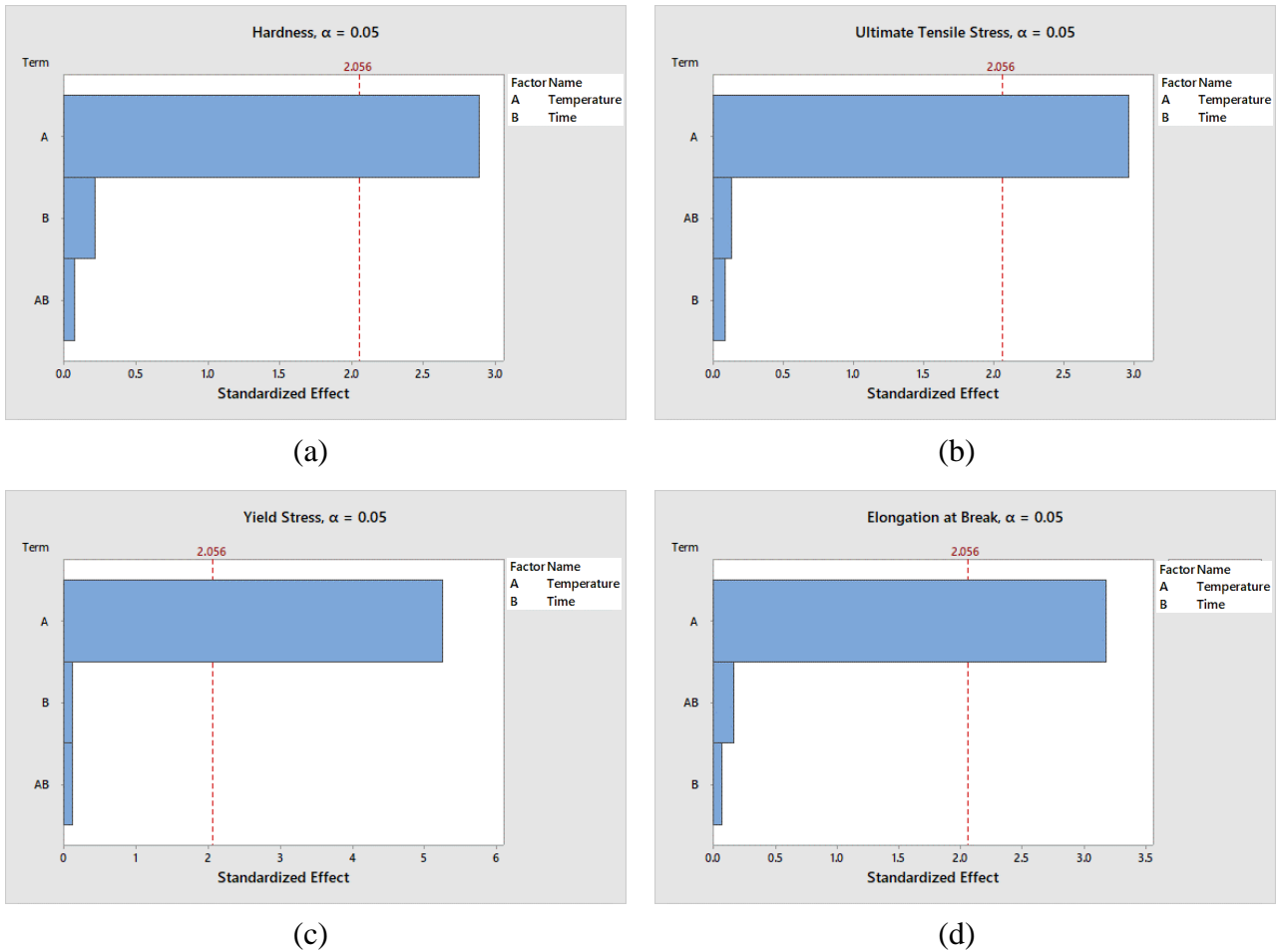


Figure 10. Pareto charts created for the test results (a) hardness (b) tensile stress (c) yield stress (d) elongation at break

In Figure 11, changes in hardness, tensile stress, yield stress and elongation at break depending on temperature and time are given. As indicated in the Pareto charts, the duration of the heat treatment did not make any difference in the test results. On the other hand, the yield and tensile strengths increase with increasing heat treatment temperature. According to Figures 11a and 11d, it is seen that the hardness and elongation at break values decrease with increasing temperature.

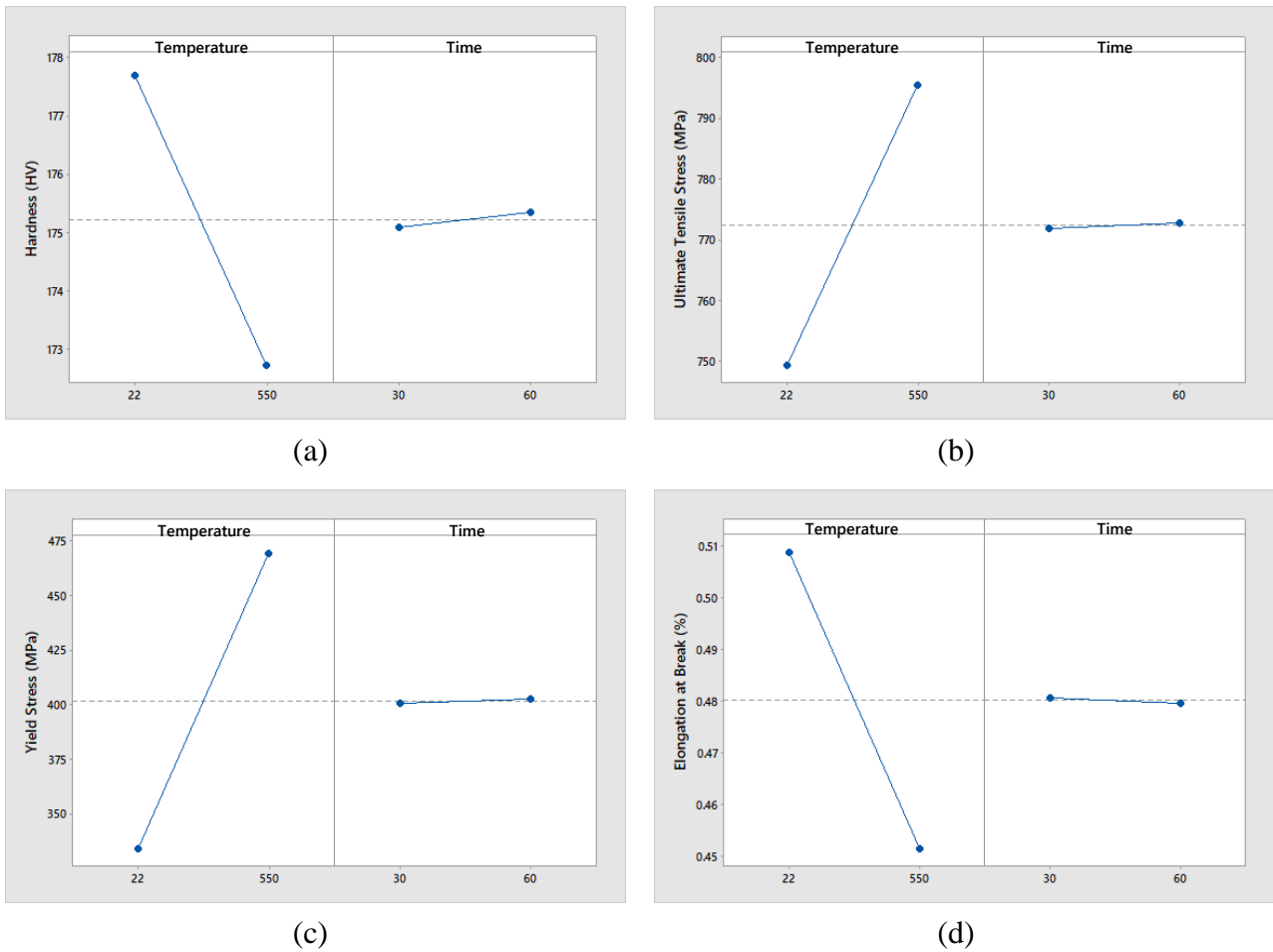


Figure 11. Variation of (a) hardness (b) tensile stress (c) yield stress (d) elongation at break with temperature and time

4. Conclusions

This study investigated static strain ageing conditions of 0.5 mm thick AISI 304 stainless steel sheets at different temperatures and annealing times. After 5% pre-deformation, the prepared tensile samples were annealed at 150, 250, 400 and 500°C temperatures for 30 and 60 minutes. Then, the samples were subjected to the tensile test, and the changes in the mechanical properties were examined. The results obtained according to the test results compared with the reference sample are below.

- It has been observed that the static strain ageing heat treatment of AISI 304 stainless steel causes an increase in the maximum tensile and yield strengths. The highest tensile strength was obtained as 791 MPa in the sample aged at 400 °C for 30 minutes and 796 MPa in the sample aged at 150 °C for 60 minutes. According to these results, the yield and tensile strengths of the as-received material increased by 10% with the ageing process. It was determined that these values decreased after 400 °C.

- As a result of the applied static strain ageing heat treatment, the elongation at the break of the samples decreased by 4.6% compared to the as-received condition.
- As a result of the static strain ageing heat treatment, a gradual increase of up to 2% was observed in the hardness of the samples up to 400°C compared to the as-received condition. With the temperature rising to 550°C, the hardness decreased by 4% compared to the as-received condition.
- Pareto analyses show that the temperature is the most influential parameter effect on hardness, ultimate tensile stress, yield stress, and elongation at break.

As a result of the study, it was seen that the heat treatment temperature had a significant effect on the results in the static strain ageing process. In contrast, the heat treatment time did not significantly differ in the test results. In future studies, the effects of the heat treatment time can be revealed more clearly by investigating the heat treatment times with more levels.

Acknowledgements

This work is supported by Giresun University Scientific Research Project Office. (Project Number: FEN-BAP-A-230218-17)

Authors' Contributions

All authors contributed equally to the study.

Statement of Conflicts of Interest

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The author declares that this study complies with Research and Publication Ethics.

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