

Article

Effect of Aging Temperature on Microstructure, Mechanical, and Wear Properties of 18Ni-300 Maraging Steel Produced by Powder Bed Fusion

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Abstract: Additive manufacturing technologies for metallic materials based on powder bed fusion have enormous industrial potential. In this study, we manufactured 18Ni-300 maraging steel using the powder bed fusion (PBF) process and investigated the effects of annealing temperatures of 430 °C, 490 °C, and 550 °C for 3 h on its microstructure, tensile fracture mechanism, and wear properties compared with the as-built specimen. The results show that annealing heat treatment effectively improved the dry sliding friction, wear properties, and room temperature tensile properties compared to the as-built specimen. Compared to other aging-treated samples, specimens that underwent heat treatment in optimal settings had superior properties. With optimal heat treatment, while melt pool boundaries remained, the cellular and columnar structures became finer compared to the un-treated specimens, and the number of dimples decreased. Consequently, the hardness and tensile strength improved by approximately 56.17% and 40.63%, respectively. The 18Ni-300 maraging steel sample that underwent heat treatment at optimal settings exhibited a coefficient of friction approximately 33.33% lower than the as-built alloy.



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Keywords: 18Ni-300 maraging steel; powder bed fusion; aging treatment; microstructure; wear behavior; tensile test; fracture mechanism

1. Introduction

18Ni-300 maraging steel is an iron-nickel-based alloy that contains 18% nickel. The term “maraging” was coined from a combination of “martensite” and “age hardening”. Its mechanical properties, including yield and tensile strength, ductility, toughness, fatigue limit, compressive strength, hardness, and wear resistance, are excellent. Maraging steel is used in aerospace components, extrusion, plastics injection molds, and metal casting dies where abrasion resistance is required. Currently, 18Ni-300 maraging steel parts are mainly produced through casting, forging, and welding processes, which have drawbacks such as prolonged production times and difficulties in fabricating complex-shaped parts [1–4]. The production of small, custom-made high-precision parts directly from metallic powder through additive manufacturing (AM) has become a primary focus of current research [5]. AM technologies offer engineers a new array of possibilities for designing and fabricating products that would not be feasible or cost-effective with conventional methods such as machining, injection molding, or casting [6]. Among the various additive manufacturing techniques, powder bed fusion (PBF) is one of the most commonly used methods in the industry. The powder bed fusion process has experienced significant growth in recent years due to its cost-effectiveness and high-quality output. Maraging steel powder is very compatible to be processed by the PBF process due to its low reflectivity and

high weldability. Parts can be manufactured with nearly full density (99.5%) and exhibit mechanical properties similar to those of conventionally produced metals.

During the PBF process, metal parts are built layer by layer. The orientation of grain texture is influenced by the various building directions of specimens, resulting in anisotropy in the microstructure. For instance, the grains are elongated and columnar in the plane along the building direction, while they are typically equiaxed in the plane perpendicular to the building direction. Therefore, the performance of the parts manufactured using PBF depends on their building direction [7]. Shamsdini et al. [8] examined the microstructure and mechanical properties of 18Ni-300 maraging steel produced by LPBF, and they investigated the effects of building direction (vertical and horizontal). They found that the porosity decreased after a 6 h aging treatment at 490 °C compared to the as-built state. Additionally, the horizontal directions exhibited higher strength and ductility compared to the vertical orientations. They also claimed that heat treatment affects the fracture mechanism, but the building direction does not affect fracture in both the as-built and heat-treated conditions. Song, Tang, et al. [9] fabricated 18Ni-300 maraging steel using PBF in both horizontal and vertical orientations. They conducted microhardness and tensile tests to analyze the mechanical properties and anisotropic behavior of the material. Under the same heat treatment conditions, no significant anisotropic properties were observed. However, the yield strength and ultimate tensile strength of the horizontally fabricated specimens were higher than those of the vertically fabricated ones. This was attributed to the formation of columnar grains along the build direction during PBF.

It is reported that in the 18Ni-300 maraging steel, the Ni_3Ti phase (or more generally, Ni_3X where $\text{X} = \text{Ti, Mo, V, and W}$) readily forms during short-term aging at low temperatures (400–450 °C), followed by the precipitation of Fe_2Mo or Fe_7Mo_6 . Aging at temperatures above 500 °C facilitates the simultaneous formation of austenite through a diffusion-controlled reaction. This process is also facilitated by the release of nickel into the matrix due to the decomposition of the Ni_3Ti phase [10]. In the PBF process, a previously deposited material may undergo cyclic reheating with a gradually decreasing laser intensity during the deposition of neighboring tracks and subsequent layers. This indicates that the material deposited in one layer undergoes in situ heat treatment by the subsequent tracks and layers, allowing the nuclei of the precipitates to form easily during the PBF process [11]. As a result, precipitation can occur easily without undergoing solution heat treatments. In this regard, some researchers have already considered omitting the solution heat treatment for the maraging steel produced by PBF [12]. Through aging treatment, the internal stress caused by quenching can be released, and evenly distributed nano precipitates would form in the substrate, thereby enhancing both strength and toughness [13]. Therefore, optimizing the aging temperature and time is crucial for achieving the desired properties. Aging treatment at an appropriate temperature leads to the hardening of the matrix as a result of the precipitation of Ni-Mo, Fe-Mo, and Fe-Ni intermetallic compounds within the martensitic structure. Casati et al. [10] conducted a study on the microstructural changes, hardness, and tensile properties of PBFed 18Ni-300 steel at different aging temperatures and durations. Using differential scanning calorimetry (DSC), isothermal aging curves were measured at temperatures of 460 °C, 490 °C, 540 °C, and 600 °C for durations ranging from 10 min to 14 days. During the aging treatment, it was discovered that austenite reversion initially occurred at the cell boundary, with austenite appearing at the intercellular level during over-aging. Kim, D, Kim, T, et al. [12] investigated the effect of heat treatment on the mechanical anisotropy of PBFed 18Ni-300 maraging steel. When heat-treated at 450 °C, similar hardness values to those of fully hardened martensitic steel were observed. As a result, it was claimed that the alloy can be hardened only through aging treatment, without the need for solution treatment. The specimens aged at 450 °C exhibited the highest yield strength when the building direction was horizontal. Yan et al. [14] studied the microstructure and mechanical properties of additively manufactured 18Ni-300 maraging steel through solution treatment and aging treatment. The aging-treated specimens had higher hardness than the solubilization-treated specimens. They also found that the

hardness increased with the increase in aging time but decreased after exceeding 3 h due to coarsening of precipitates.

Several studies have been conducted on the microstructural evolution resulting from heat treatment. However, there is a lack of analysis regarding the tensile properties and wear behavior of 18Ni-300 maraging steel in relation to different heat treatment temperatures. This study investigates the effects of building direction and aging heat treatment conditions on the microstructure, tensile properties, fracture mechanisms, and wear properties of 18Ni-300 maraging steel. In particular, wear reduces the lifespan of components that operate at high speeds and high loads and requires in-depth analysis of wear behavior for industrial applications. The 18Ni-300 maraging steel produced by the PBF process underwent thermal treatment, including aging treatment at various temperatures followed by air cooling. Additionally, tensile tests, hardness evaluations, wear tests, and microstructure inspections were conducted on the thermally treated specimens to find the most optimal aging conditions.

2. Experimental Procedure

18Ni-300 maraging steel powder with a particle size of 10–90 μm was used, and the chemical composition is shown in Table 1. The Concept Laser M1 was utilized to manufacture wear and tensile specimens, as depicted in Figure 1, in an argon atmosphere. The machine specifications include a laser power of 180 W, scan speed of 1000 mm/s, hatch distance of 0.105 mm, beam diameter of 0.05 mm, layer thickness of 0.035 mm, focus move of -3 mm, rotating angle of 90° , and a laser pattern of continuous exposure. The specimens were aged at 430°C , 490°C , and 550°C for 3 h without solution treatment and then cooled in air, as shown in Figure 1c.

Table 1. Chemical composition of 18Ni-300 maraging steel powder.

Element	Ni	Co	Mo	Ti	Al	Si	C	P	S	Fe
Wt%	17.70	8.66	4.58	0.68	0.089	0.01	0.0043	<0.005	<0.003	Bal.

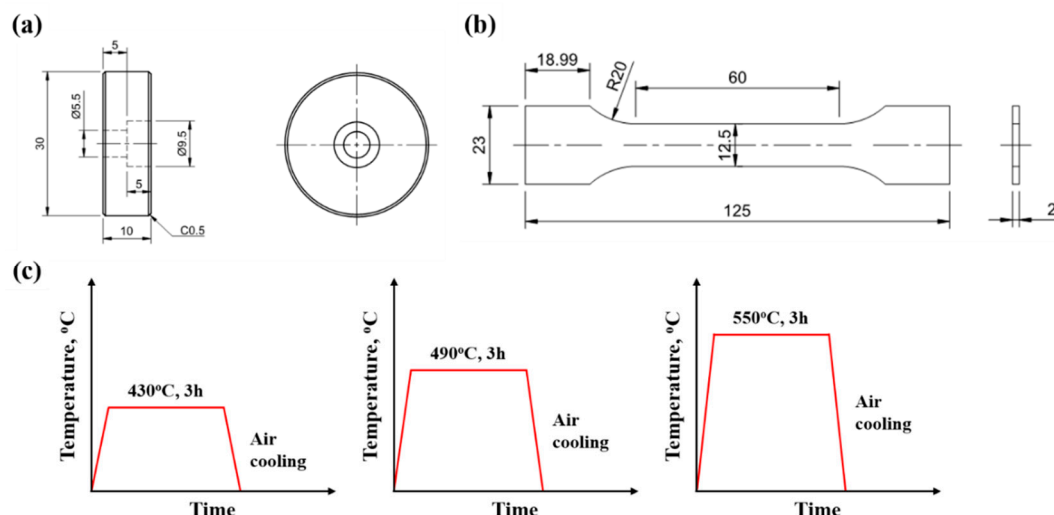


Figure 1. Schematics of (a) wear test specimen, (b) tensile test specimen (KS B 0801 13B, gauge length 50 mm), and (c) heat treatment conditions.

After heat treatment, the microstructure was observed by grinding with SiC paper (80–600 grit), polishing with a 3 μm diamond suspension, and further polishing with colloidal silica down to 0.04 μm before wear testing. The etching solution was Fry's reagent (25 mL of HNO_3 + 50 mL of HCl + 1 g of CuCl_2 + 150 mL of H_2O). Measurements were taken 12 times for each specimen with a 0.1 kgf load using a Vickers hardness tester (HM-210, Mitutoyo, Tokyo, Japan). XRD analysis (SC-XRD, D8 VENTURE, Bruker, Tokyo, Japan)

was conducted to examine the phases at various aging temperatures. It was measured at 40 kV and 40 mA within a 2θ range of 20° to 90° using $\text{CuK}\alpha$ radiation at a scan rate of $2^\circ/\text{min}$. The sample was then subjected to Rietveld analysis. The tribological properties were measured using the ball-on-disc method with an abrasion tester (RB-102PD, R&B Co., Ltd., Seoul, Republic of Korea) at room temperature. The test was performed for 99.645 s at 50 N, with a rotational speed of 1000 RPM and a sliding distance of 120 m using a tungsten carbide (WC) ball, which has higher hardness compared to the specimen. Tensile testing was conducted at room temperature using a tensile testing machine (KSU-20M, Kyoungsung, Seoul, Republic of Korea) with a load of 50 N and a deformation rate of 5 mm/min. The field emission scanning electron microscope (FE-SEM, Quanta 200 FEG, FEI Company, Amsterdam, The Netherlands) at the Converging Materials Core Facility of Dong-Eui University was used to observe the microstructure, wear tracks, mechanisms, and tensile fracture surface.

3. Results and Discussion

Figure 2 displays scanning electron microscope (SEM) observations of microstructure changes based on aging temperature. Figure 2a shows the microstructure of the as-built specimen without heat treatment. Melt pool boundaries are observed, and columnar and cellular structures appear. These fine cellular microstructures are unique to the PBF process. They form in response to the instantaneous melting and rapid solidification with an extremely high cooling rate of the powder alloy during laser irradiation. The refined cellular structure is a common microstructure of PBFed steels, which can enhance the hardness and strength of PBF-produced steels compared to conventionally manufactured steels [15]. Figure 2b–d shows the microstructure after annealing heat treatment. As shown in Figure 2b, the morphology was similar to the as-built specimen when annealed at 430°C . Melt pool boundaries were observed, and the cellular and columnar structure was dissolved by the heat treatment and not clearly visible. The black dots in Figure 2b,c are estimated to be pores, and the number of pores was higher when heat treated at 490°C . For heat treatments above 490°C , the melt pool boundaries disappeared, as shown in Figure 2c,d. Cellular and columnar structures disappeared, and an irregular, island-like microstructure was observed. Such sub-grain cells may disappear through diffusion during the post-heat treatment process. In addition, the thickness of the cells increased as the aging temperature increased, indicating an over-aging phenomenon. The microstructure exhibited very thick boundaries, as shown in the figure, due to the extremely prominent reversion of martensite to austenite. The retained austenite resulted from the microsegregation of solute elements, especially nickel, at cellular boundaries during solidification [15]. Retained austenite is easily distinguishable in the high magnification microstructure, where it appears as a bright phase that aggregates at cell boundaries. It arises in the as-built alloy from an incomplete transformation into martensite during rapid cooling or solidification. The presence of these tiny segregated solute-rich regions not only softens the as-built alloy but also enhances additional austenite reversion during the high temperature precipitation heat treatment cycle [16].

Figure 3 illustrates the XRD pattern changes depending on the aging temperature. The $(110)_\alpha$, $(200)_\alpha$, and $(211)_\alpha$ peaks, which are martensite phases, are observed in the as-built and all heat treatment conditions. However, no austenite phase appeared in the as-built specimen. When heat-treated at 430°C , the intensity of the martensite peaks decreased, and in the austenite phase, the $(111)_\gamma$ peak appeared. The $(111)_\gamma$ peak was also present at 490°C , and an additional $(002)_\gamma$ peak was formed. At 550°C , the $(022)_\gamma$ peak formed, and the intensity of the existing $(111)_\gamma$ and $(002)_\gamma$ peaks increased. The peak of the austenite phase was observed during the aging treatment. As the aging temperature increased, the peak intensity of martensite decreased, while the peak intensity of austenite increased. This indicates that the amount of austenite increased as the aging temperature increased. Austenite increases during aging due to the enrichment of the matrix containing Ni, which is called reverted austenite. Precipitates such as Ni_3X ($\text{X} = \text{Al}, \text{Ti}, \text{Mo}, \text{Fe}$) are

present at 20° to 40°, but they are difficult to distinguish because of their small size and low volume fraction [17–19]. Table 2 presents the quantitative analysis of the volume fraction of austenite and martensite as they vary with aging temperature, utilizing the Rietveld method. Previous literature has shown a low fraction of austenite in untreated specimens [20,21]. In this study, the austenite fraction was 0%, and only martensite was present. This is estimated to be due to the very low content of austenite, which did not appear in the XRD pattern. The austenite fraction was the lowest at 1.2% when aged at 430 °C and the highest at 21.8% when aged at 550 °C. The amount of austenite increased significantly to 12.9% during the heat treatment at 490 °C, indicating that overaging has occurred [22]. As the aging temperature increased, the fraction of austenite increased, but the fraction of martensite decreased.

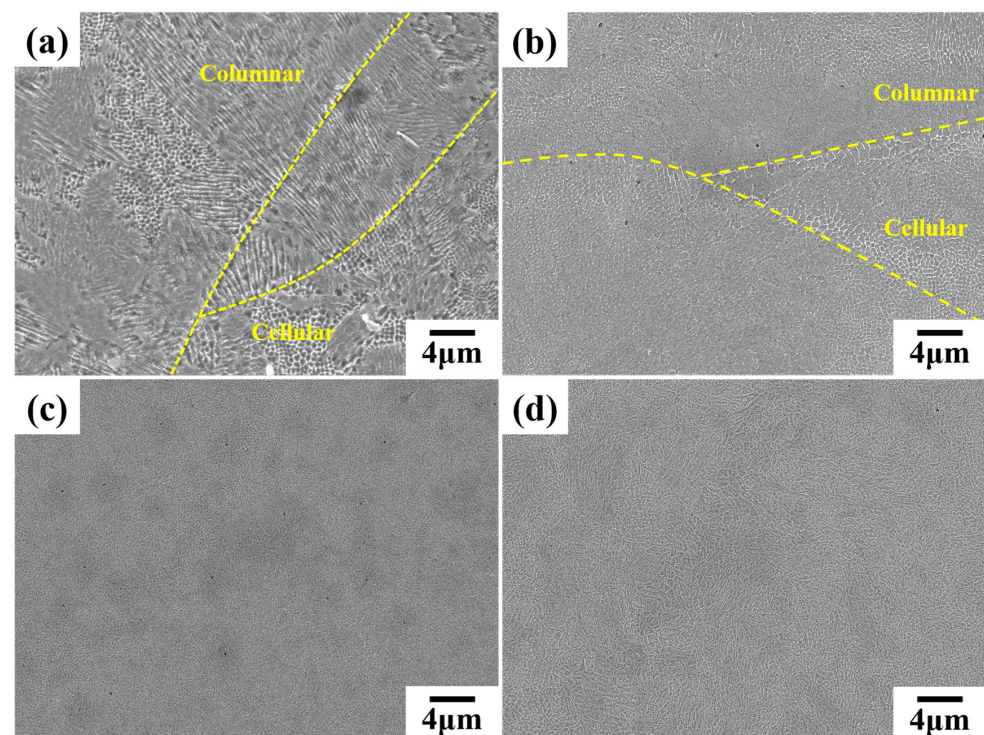


Figure 2. Microstructure of 18Ni-300 maraging steel with different annealing heat treatment temperature of (a) as-built, (b) 430 °C, (c) 490 °C, and (d) 550 °C.

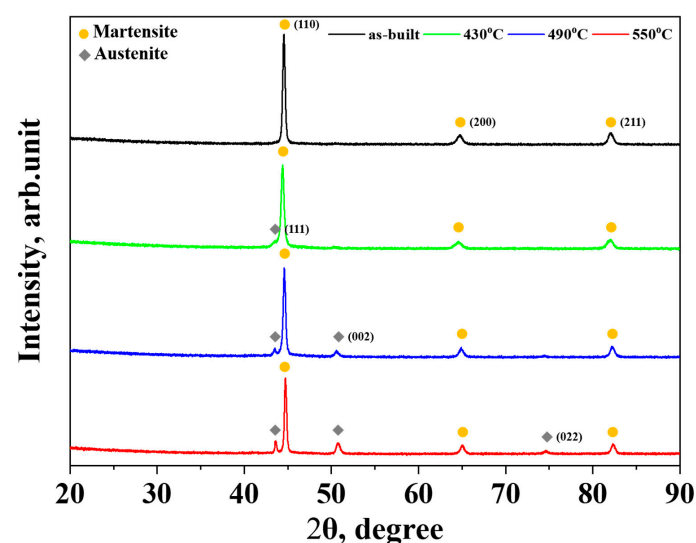


Figure 3. XRD pattern of PBFed 18Ni-300 maraging steel with varying aging heat treatments.

Table 2. Rietveld analysis of different aging temperatures.

	Martensite (%)	Austenite (%)
as-built	100	0
430 °C	98.8	1.2
490 °C	87.1	12.9
550 °C	78.2	21.8

Figure 4 shows the results of Vickers hardness measurements varying with heat treatment temperature. The differences between the heat treatment conditions were minimal, but the hardness of the specimens subjected to heat treatment increased significantly compared to the as-built specimens. The as-built specimens, which were not subjected to heat treatment, have the lowest hardness at 380.3 ± 10.8 HV due to the absence of precipitate formation [23]. In the heat treatment process, the highest hardness value of 593.9 ± 12.5 HV was obtained at 430 °C. Hardness values of 575.6 ± 7.6 HV and 558.7 ± 8.9 HV were measured for specimens heat-treated at 490 °C and 550 °C, respectively. The hardness increased with heat treatment and decreased with higher aging temperatures. The hardness decreased with the increasing thickness of the bright phase (austenite), as shown in Figure 2d. The Rietveld results show that the highest hardness values were obtained when the amount of austenite was 1.2%, with a sharp decrease in hardness above this amount. Therefore, the presence of austenite affects the hardness, which decreases as it dissolves intermetallic precipitates [24].

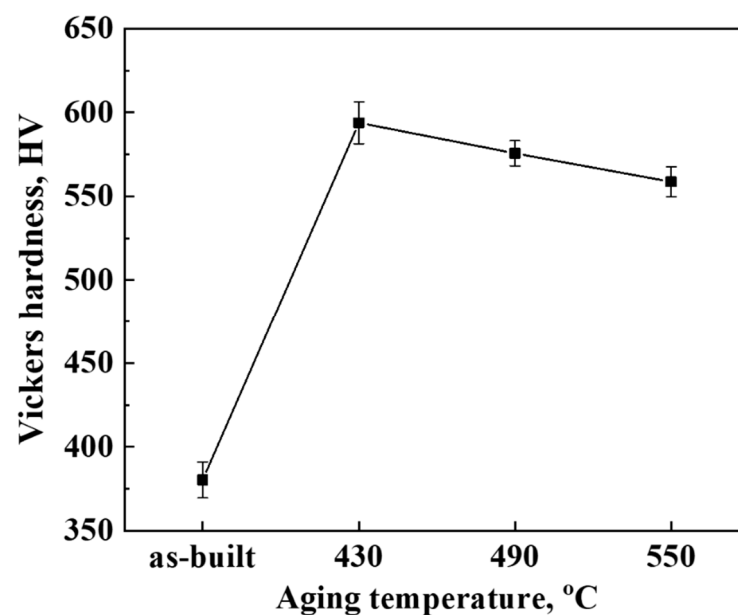
**Figure 4.** Vickers hardness at different heat treatment temperatures.

Figure 5 compares the tensile strength and elongation at different annealing temperatures of 18Ni-300 maraging steel produced by PBF. The as-built specimen without heat treatment had the lowest tensile and yield strengths of 960 MPa and 740 MPa, respectively, but the highest elongation at break of 10.88%. The tensile strength at 430 °C was 1350 MPa, which was similar to the value obtained when heat-treated at 550 °C (1340 MPa). However, the yield strength after heat treatment at 430 °C was 870 MPa, which was 120 MPa higher than the specimen heat-treated at 550 °C. Heat treatment at 490 °C resulted in a tensile strength of 1400 MPa, which was a 45.83% increase over the as-built condition and represented the highest tensile strength among the heat-treated conditions. However, the yield strength was lower than that of the specimen heat-treated at 430 °C. After the heat treatment, the tensile and yield strength increased, but the elongation decreased. The differ-

ence in tensile strength with aging temperature was not significant. This is likely due to the small variation in heat treatment temperature. However, the yield strength decreased with increasing aging temperature. The intensity of the austenite peak increased as the aging temperature increased, as shown in Figure 3. Enhancing the austenite content can lead to improved elongation but may result in a decrease in strength [25,26]. In addition, the yield strength of the metal was inversely proportional to the size of the cellular structure, and these structures contribute to improving the yield strength of AM parts [27,28]. Therefore, the highest elongation and lowest tensile strength were obtained at 550 °C, where the amount of austenite was the highest at 21.8%. At 430 °C, the finest cellular structure was observed, with an optimal combination of martensite and austenite structures, resulting in the highest yield strength. Heat treatment was performed at similar temperatures as in previous studies, but the tensile and yield strengths were lower [29]. However, high mechanical properties were achieved at lower temperatures compared to the original study.

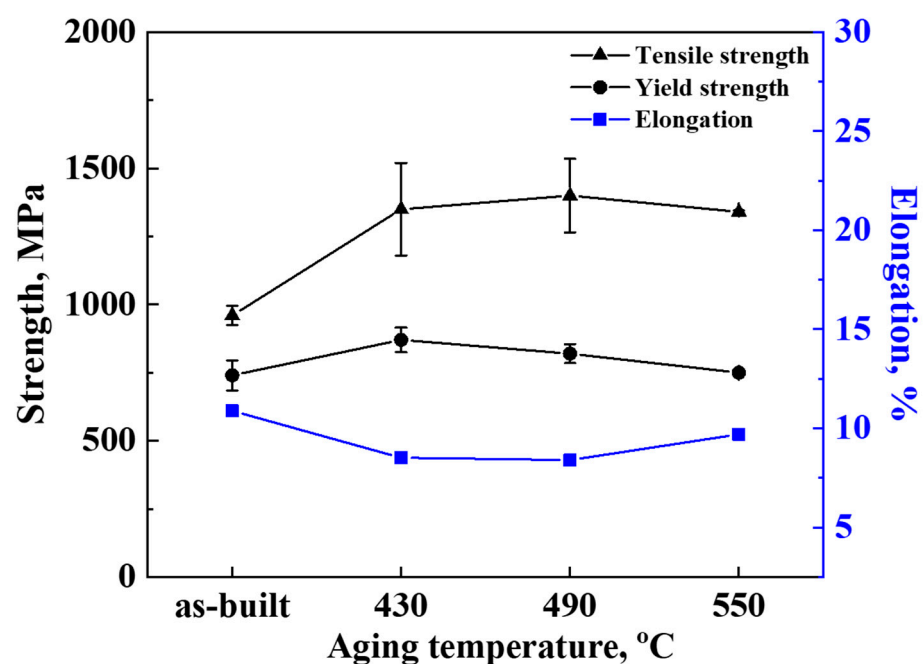


Figure 5. Tensile strength, yield strength, and elongation with variations at different aging temperatures.

In Figure 6, the fracture surface after tensile testing was observed using SEM. All specimens in Figure 6 exhibit mixed fracture behavior, with both ductile and brittle fractures observed. In ductile fracture, dimples are observed, which are created by the coalescence of micropores. The smaller and more numerous the dimples, the less ductile and more brittle they become when broken. In brittle fracture, cracks propagate rapidly in a specific direction, leading to cleavages and river patterns [30,31]. The as-built specimen (Figure 6a) has the highest elongation due to the highest number of dimples and larger dimples with a river pattern compared to other heat treatments. Figure 6b shows fewer dimples compared to the as-built sample, and transgranular fracture occurred with crack propagation across the grain. Figure 6c, which was heat-treated at 490 °C, exhibited more brittle fracture with increased transgranular fractures compared to 430 °C, and cracks were observed. In addition, the river patterns became more numerous and distinct. Figure 6d shows that the number of dimples is the highest among the heat-treated specimens, and large dimples are observed. Therefore, the ductility is better than that of the specimens heat-treated at 430 °C and 490 °C, resulting in a more ductile fracture. In addition, a small number of transgranular fractures occurred. Similar to the other specimens, cracks and river patterns were observed, although the river patterns were faintly visible. Transgranular fracture

was not observed in the as-built specimen, but it occurred in the heat-treated specimen. Additionally, the dimple size increased as the aging temperature rose.

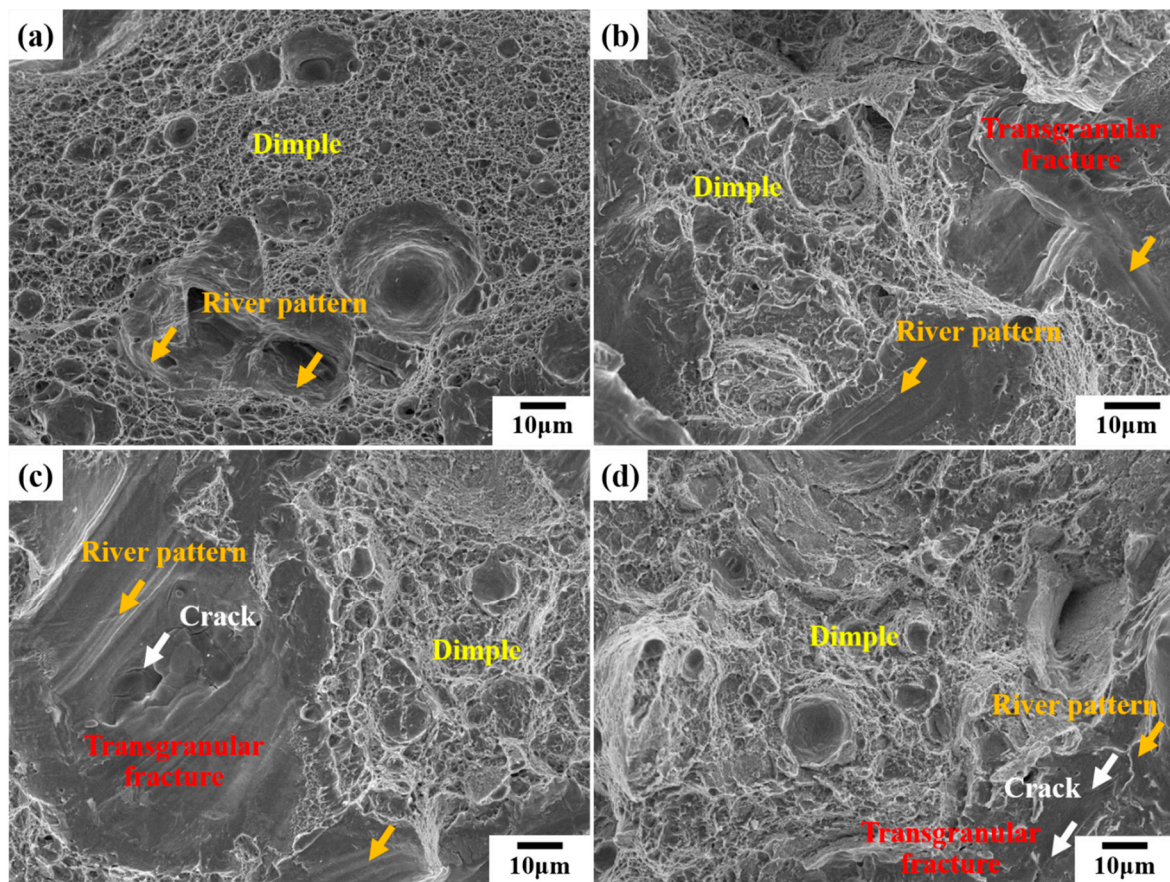


Figure 6. Tensile fracture surface SEM images with different aging temperature of (a) as-built, (b) 430 °C, (c) 490 °C, and (d) 550 °C.

Figure 7 shows SEM images of the width of the wear track after the wear test. Debris was observed around the wear tracks of all specimens. Under a certain load, abrasives move across the surface of a material, producing wear debris. Material was removed from the wear track and pushed to the sides of the track due to the plowing effect. Successive layers of materials were squeezed out with each pass of the counterpart movement to accommodate the wear. It can be observed that material from the wear track is extruded, forming plate-like debris due to repeated sliding. The plates then fracture and break away as debris [32,33]. In Figure 7a, numerous large and small debris are observed at the edge of the wear track, with the widest wear width measuring 1190 μm from the as-built specimen. However, specimens subjected to annealing heat treatment at 430 °C (Figure 7b) show that only a few small debris are observed around the wear track, and the wear width is the narrowest at 854 μm . It can be seen from Figure 7c that the specimens annealed at 490 °C and 550 °C have an average wear width of 1010 μm , and Figure 7d is 1050 μm . The degree of specimen adhesion around the wear track is similar, but the specimens heat-treated at 490 °C and 550 °C exhibit wider wear tracks. The heat-treated specimens tended to have a narrower wear width compared to the untreated specimens. In addition, the amount of debris and the width of wear around the wear track decreased with increasing hardness.

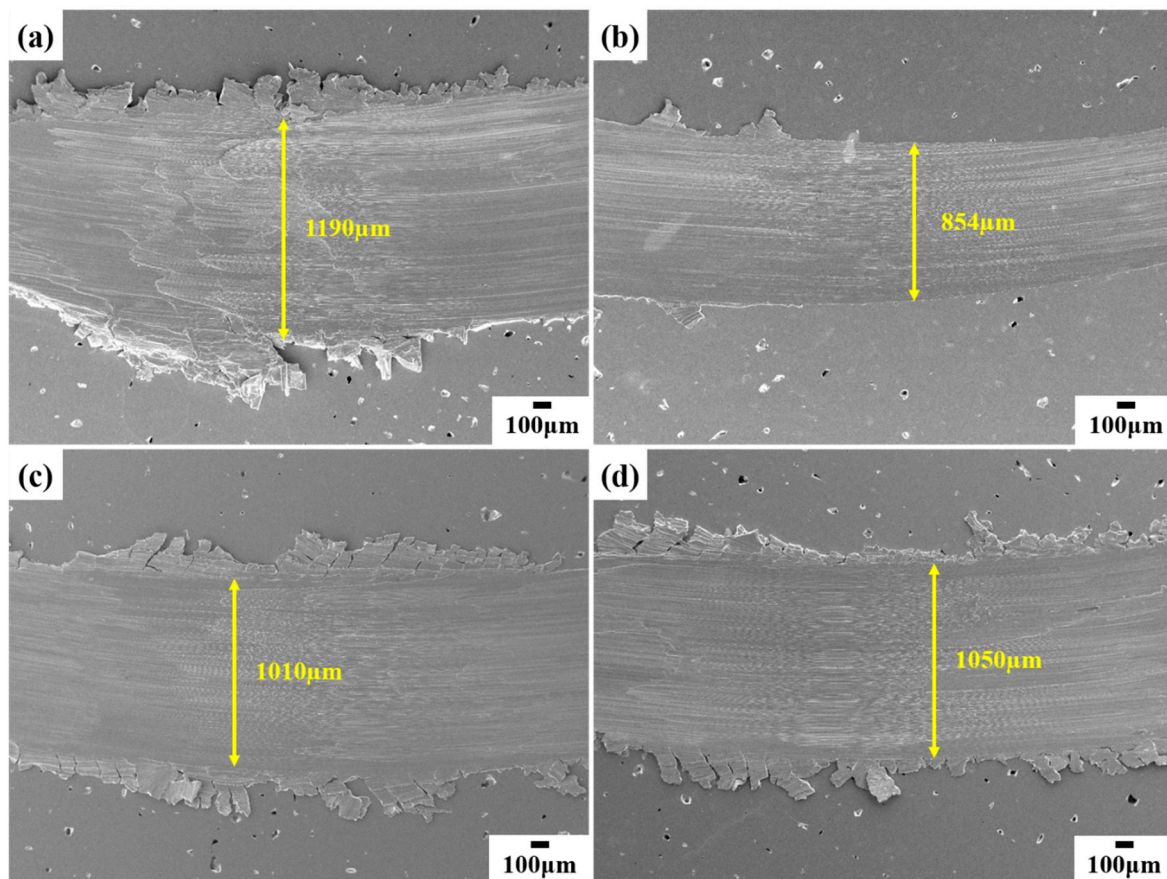


Figure 7. Wear surface analysis on the specimen with aging treatment temperature of (a) as-built, (b) 430 °C, (c) 490 °C, (d) 550 °C.

In Figure 8, observations were made at high magnification to analyze the wear mechanism, and the average friction coefficients are shown in Figure 9. There are wear mechanisms including abrasive wear, adhesive wear, fatigue wear, and fretting wear. This is influenced by factors such as the applied load, sliding velocity, surface hardness, roughness, lubrication, and other related variables [34,35]. In Figure 8a, more adhesive wear is observed compared to abrasive wear. The average friction coefficient is the highest at 0.453 μ because the ball does not slide well due to the adhered part. On the other hand, in Figure 8b, which was heat-treated at 430 °C, a significant amount of grinding wear with grooves was observed. The width of the groove was the narrowest among the specimens, and delamination occurred. Delamination was more prevalent in specimens with heat treatment than in those without heat treatment. The EDS results showed a high content of oxygen and tungsten in the area affected by adhesive wear. The presence of W is believed to be caused by the WC ball counterpart detaching during the wear test. In addition, the formation of these oxides is associated with reduced wear and friction. Therefore, the oxide present on the wear surface in Figure 8b has the lowest average friction coefficient of 0.302 μ as the friction force is reduced due to the oxide on the wear surface. Figure 8c,d exhibits similar mechanisms involving a combination of abrasive and adhesive wear. Abrasive wear typically occurs when softer surfaces come into contact with rough, hard protrusions, while adhesive wear takes place when two essentially flat solid surfaces are in sliding contact, with or without lubrication [36,37]. The average coefficient of friction in Figure 8c is 0.349 μ , while in Figure 8d it is 0.375 μ , which is the highest among the heat-treated specimens. Figure 8c exhibits higher hardness compared to Figure 8d, leading to increased abrasive wear and observed galling. Figure 8d also shows significant galling due to adhesive wear, with the widest groove width and the least delamination. As the heat treatment temperature decreased, abrasive wear increased, leading to more delamination and a reduction in

groove width. In addition, the average coefficient of friction (Figure 9) exhibited a sharp decrease with heat treatment and increased with higher heat treatment temperatures due to the presence of adhesive wear.

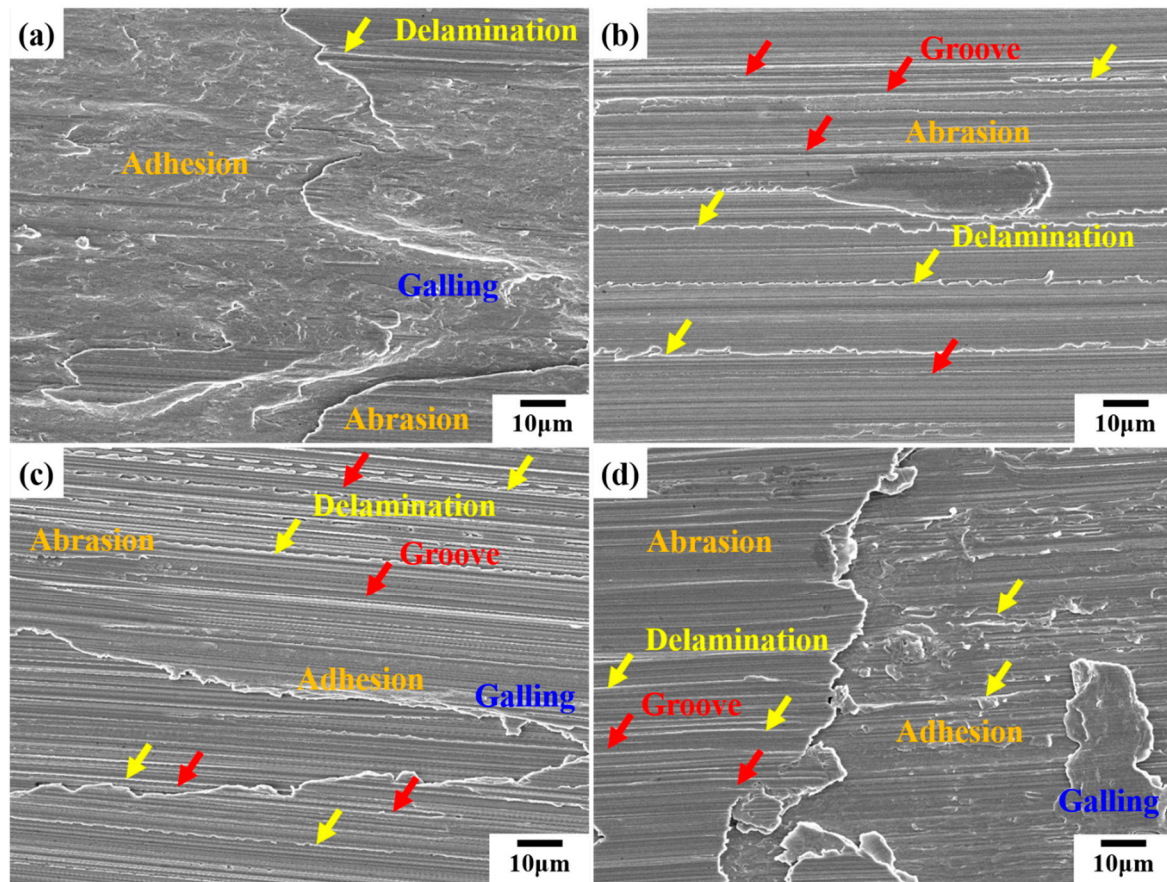


Figure 8. Wear mechanism on the specimen with aging treatment temperature of (a) as-built, (b) 430 °C, (c) 490 °C, (d) 550 °C (high magnification).

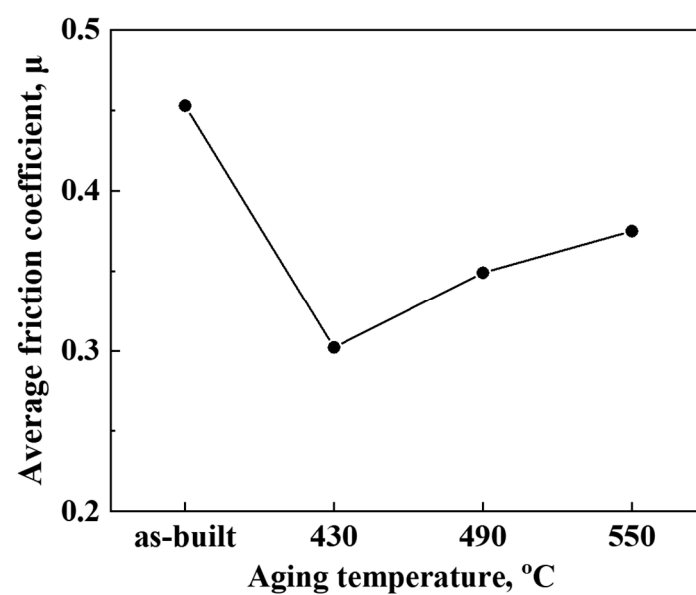


Figure 9. Average friction coefficient of aging treatment temperature.

4. Conclusions

In this study, 18Ni-300 maraging steel was processed using powder bed fusion (PBF) with optimized processing parameters. The effect of various heat treatment conditions on the microstructure, mechanical properties, and wear behavior of 18Ni-300 maraging steel was analyzed. The microstructure of the as-built sample exhibits columnar and cellular structures within the boundaries of the melt pool. When annealed at temperatures above 490 °C, the boundaries of the melt pool disappeared. The average microhardness of the samples in the as-built state and after aging at 430 °C, 490 °C, and 550 °C are 380.3, 593.9, 575.6, and 558.7 HV, respectively. The austenite present at the grain boundaries precipitated during the aging treatment, leading to an increase in cell thickness and an irregular microstructure. Although the microhardness could be improved by aging, the hardness decreased due to excessive precipitation of austenite when heat-treated above 490 °C. Aging also affects the transformation of martensite to austenite. The austenite phase fraction analyzed by the Rietveld method was 1.2%, 12.9%, and 21.8% at 430 °C, 490 °C, and 550 °C. The austenite fraction increased with the rise in aging temperature. Specimens aged at 430 °C exhibited UTS of 1350 MPa, which is nearly 40.63% higher than that of the as-built specimen and 0.75% higher than the specimen aged at 550 °C, attributed to the finer microstructure and moderate amount of austenite. The as-built specimens exhibited the highest elongation because of the distribution of large dimples. The heat-treated specimens had both ductile and brittle fractures, with a higher occurrence of brittle fracture observed at 490 °C. The average coefficient of friction (COF) of the sample, heat-treated at 430 °C (COF = 0.302 μ), is reduced by 33.33% compared to the as-built state. The cause of the reduced friction was the high oxygen content in the area affected by adhesive wear. This results in excellent wear resistance when aged at 430 °C. Adhesive wear is the dominant wear mechanism in the as-built samples, while the heat-treated specimen exhibits a mixed mode of abrasive and adhesive wear. The optimal heat treatment conditions we found (430 °C, 3 h) are expected to enhance mechanical and wear properties when implemented in the industry.

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