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Effect of Austenitizing Temperature and Holding Time on Microstructure, Hardness, and Retained Austenite Amount of HSLA Steel as Automotive Components Material

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Abstract. Retained Austenite is a metastable phase that can cause isothermal transformation at room temperature, resulting in cracks occurring long after producing components made from HSLA steel. This research focuses on the heat treatment process's austenitizing temperature and holding time. For the study on the effect of temperature, the austenitizing stage was carried out at 850ºC, 870ºC, 900ºC, and 926ºC for 28 minutes. In contrast, for the study of holding time, austenitizing was carried out for 28 minutes, 43 minutes, 58 minutes, and 73 minutes on 926ºC. The characterization conducted was metallography using optical microscopy (OM) and scanning electron microscopy (SEM), micro and macro hardness testing (using Vickers and Rockwell C method), and retained austenite phase quantification using Image-J image analyzer. The microstructure produced was tempered martensite, lower bainite and a small amount of retained Austenite were also found. All the variables produce the same microstructure but have different hardness values and retained austenite percentages. It was found from this research that the optimum variable with the highest hardness value (500 HV) and the lowest amount of retained Austenite (1.50%) is at the austenitizing temperature of 900°C and the holding time of 58 minutes. With a low amount of retained Austenite, the possibility of isothermal transformation at room temperature of the retained Austenite to other phases becomes less, reducing the likelihood of cracking some time after the production process of components.

Keywords: HSLA steel, austenitizing, microstructure, hardness, retained Austenite

1. Introduction

High strength low alloy (HSLA) steel is a type of steel with 0.05%-0.25% weight carbon content and better mechanical properties and corrosion resistance than carbon steel. HSLA steel is widely used in various applications[1,2] such as automotive components. The component, which after manufactured and heat treated has no defects, had cracks on it after some time.

With medium carbon content and the presence of a alloys of Mn, Cr and Mo, in small quantities, the HSLA material has quite high strength and hardness, but tends to contain retained austenite after the hardening process, with decreasing temperature Ms (Start Martensite).

The cause of cracks that appear after some periode of time after hardening process was suspected to be an isothermal transformation of the retained Austenite, which is metastable at room temperature, producing residual stress and initiating a crack after a period of time[3,4]

Cracks in components will be the starting point for cracks to propagate quickly and cause product fracture, which poses a risk to the safety of automotive product users.

The retained austenite will transform at room temperature into another phase, for example into martensite, with different unit cell dimensions from the austenite, so that after the transformation the dimensions changes in certain parts of the material, which causes high stress concentrations. , exceeds the strength of the material, causing cracks[4]. .Previous study has been done to predict the Austenite grain growth in alloy steel[5]. This study focuses on the effect of the heat treatment process, especially in the austenitizing stage with varying temperatures and holding times. The effects of austenitizing temperature and holding time can be observed with varying variables on the microstructure, hardness, and amount of retained Austenite or other phases[6,7]. Therefore the purpose of this research is to obtain the temperature and holding time produce the least amount of retained Austenite, therefore it could be predicted that the delayed crack could be reduced while still maintaining the hardness of the material[8,9]. The hardness characteristics of the material are critical for its tribological property, especially in application that requires wear resistance[10,11]. Other than heat treatment, to increase the hardness of the material, additional alloying can be utilized[12,13].

2. Experimental Procedure

The material used in this study is a high-strength low alloy (HSLA) steel produced by a casting process. The detailed chemical composition of steel is given in Table 1. The material received was as-normalized tempered. The detailed heat treatment schematic diagram is shown in Fig. 2. The specimens cut into 40x10x40 mm were heated to 850ºC, 870ºC, 900ºC, and 926ºC at 10ºC/min. 926°C was used as the last temperature variable, which is the austenitizing temperature used in the automotive components industries. Then, the samples were held for 28 minutes, followed by quenching in 1% Volume of PAG (polyalkylene glycol) in a water-quenching medium. The usage of PAG as quenchant is quite common in the steel heat treatment industries[14,15]. The quenching process is essential to improve the characteristics of the steel[16,17], especially its hardness. 210 ml of polyalkylene glycol was added to the water until the volume was 21 litres, the volume needed for the quenching bath.

The specimens heated to 926ºC were held for 28, 43, 58, and 73 minutes and quenched using the same medium. The respective holding times were obtained by comparing the actual component to the specimen used, as shown in Fig. 1.

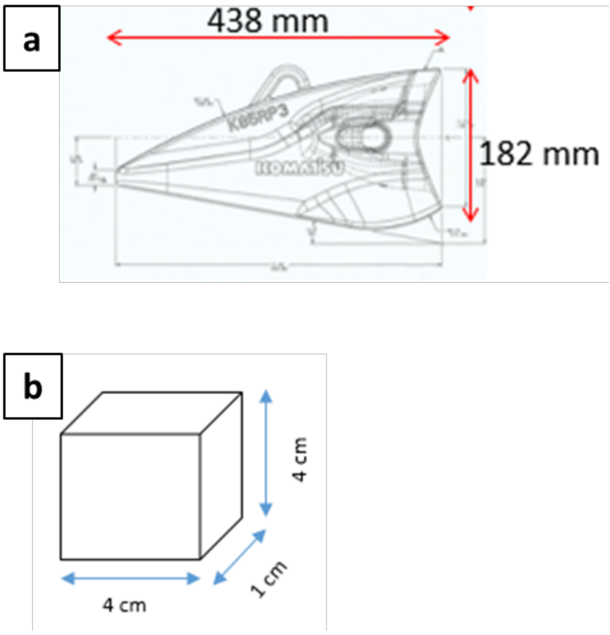


Fig.1: Dimension of (a) actual product and (b) specimen

Comparison of the distance from the surface to the core of the actual component with the specimens (which was half the height of the actual component, 91 mm, and half the height of the specimens, 20 mm) was made into a proportional relationship to the actual product holding time and specimen holding time. The actual austenitizing holding time in the automotive components industry was 60 minutes, and after calculation, the austenitizing holding time obtained for the sample was 28 minutes. The following variables were the initial 28 minutes added by 15, 30, and 45 minutes, respectively.

After quenching, the specimens were double-tempered at 205ºC for 80 minutes each, followed by air cooling. Double tempering was conducted to reduce the amount of retained austenite[18]. The heat treatment cycles are shown in Fig. 2. The samples were then mounted, ground, polished, and etched using 5% Nital for 5 seconds, and then the microstructures were observed by OM and SEM. Hardness testing using Micro Vickers with a load of 300 gf, dwell time 10 seconds, and Rockwell C with 150 kgf, dwell time 5 seconds were conducted on these samples. The amount of retained Austenite was measured by using image analyzing software. The Image Analyzer software used in this research was Image-J. The retained austenite phase was given a different color, so by using the phase calculation menu with a certain color, this software will calculate the percentage of the phase with a certain color.

Table 1. Chemical Composition of HSLA Steel (weight%)

	C	**Si**	**Mn**	**P**	**S**	**Ni**
Range	0.27 – 0.30	1.30 – 1.60	0.85 – 1.05	<0.03	0.005 – 0.015	<1.10
Target	0.28	1.40	0.95	0.025	0.009	1.60
	Cr	**Mo**	**Co**	**Cu**	**Al**	
Range	1.70 – 2.00	0.25 – 0.45	0.002 – 0.005	<0.5	0.02 – 0.05	
Target	0.27	0.27	0.003	0.02	0.035	

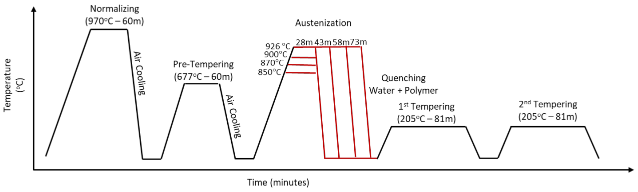


Fig. 2: Schematic diagram of austenization at various austenitizing temperatures and holding times.

3. Results and Discussion

3.1 Effect of Austenitizing Temperature on Microstructure

The microstructure produced from various temperatures, as shown in Fig. 3, is lath martensite, as it contains less than 0.6% of carbon. Phases such as lower bainite, retained Austenite, and the primary phase, tempered martensite, can be seen. The lower bainite phase is shown by the dark, small needle shape, which is formed by transformation at low temperatures.

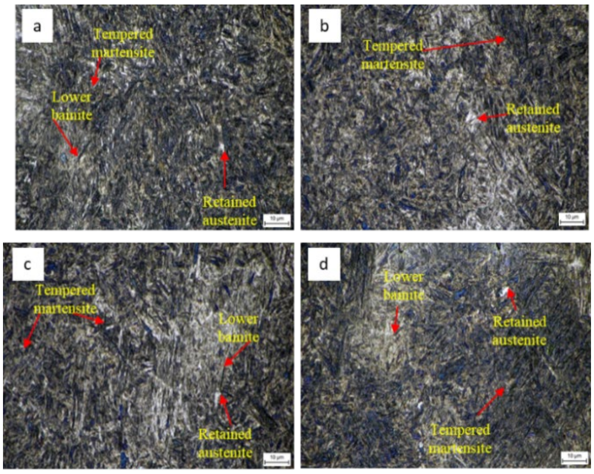


Fig. 3: Microstructure of HSLA steel austenitized for 28 minutes at (a) 850°C, (b) 870°C, (c) 900°C and (d) 926°C. Nital 5%. Magnification 1000X.

The SEM results in Fig. 4 show that the dominant phase is tempered martensite as the similarity is seen with previous work by De Knijf et al.[19], whereas other constituents, such as M/A, which is shaped like a small island[20] can also be seen along with retained Austenite. Well-tempered martensite (Well-TM), which is darker in color, and less-tempered martensite (less-TM), which is lighter, could also be identified by comparison to previous work by Matsuda et al.[21]. Well-TM is the area of martensite which has transformed well into tempered martensite. In contrast, Less-TM is the area of martensite that has not transformed fully into tempered martensite.

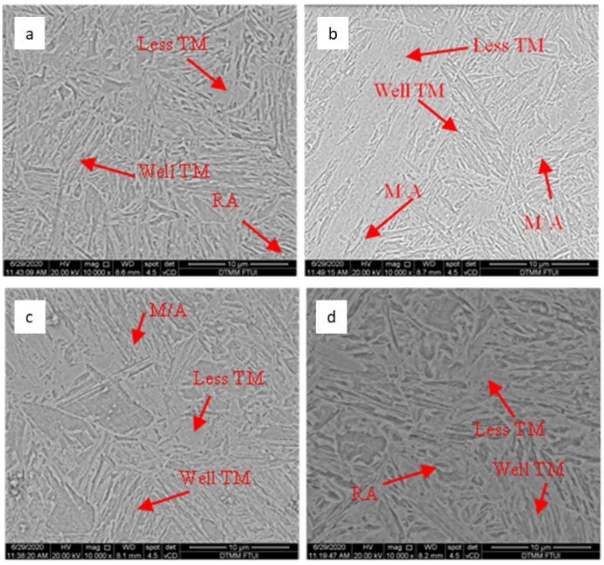


Fig. 4: SEM of HSLA steel austenitized for 28 minutes at (a) 850°C, (b) 870°C, (c) 900°C and (d) 926°C. Nital 5%. Magnification 10000X.

3.2 Effect of Austenitizing Time on Microstructure

Figure 5 shows the microstructure of HSLA steel at different austenitizing times. It can be seen that the microstructure of HSLA steel is lath martensite, which has a dark, needle-shaped phase that is tempered martensite (TM) or lower bainite (LB) and a light phase, which is a transformation zone that is thought to consist of fresh martensite and retained Austenite (RA) surrounded by bainite[22,23].

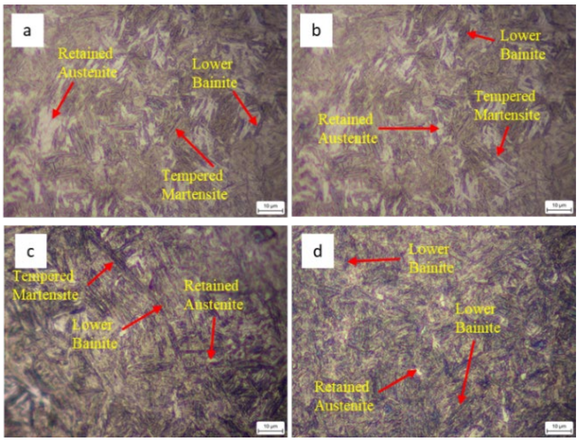


Fig. 5: Microstructure of HSLA steel austenitized at 926°C for (a) 28 min, (b) 43 min, (c) 58 min, and (d) 73 min. Nital 5%. Magnification 1000X.

Austenitizing holding time affects the grain size of the Austenite, where increasing holding time will cause grain growth so that the Prior Austenite Grain Size (PAGS) becomes larger. With increasing austenitizing holding time, the PAGS increases so that the block and package size of martensite lath becomes larger[24,25].

Figure 6 shows the results of the SEM test microstructure image of HSLA steel variables 43 minutes and 73 minutes of holding time. The microstructure is dominated by tempered martensite (as a matrix), which is in the form of dark fine needles, and there is a small amount of residual Austenite (RA) in the form of light-colored islands. The longer the holding time, the larger the PAGS size, which causes the size of the martensite lath to be larger, as shown by the red box in the image.

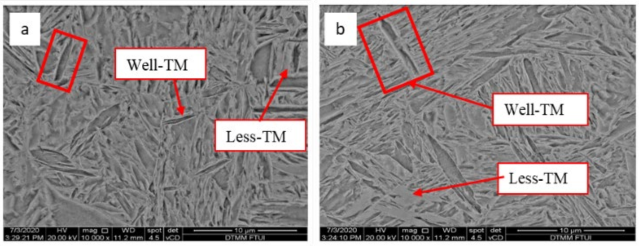


Fig. 6: SEM of HSLA steel austenitized at 926°C for (a) 43 min and (b) 73 min. Nital 5%. Magnification 10000X.

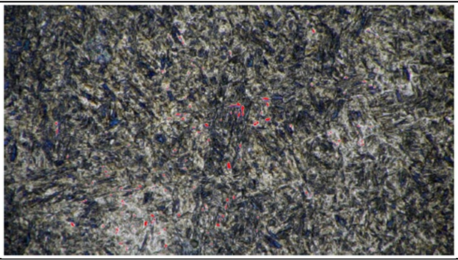
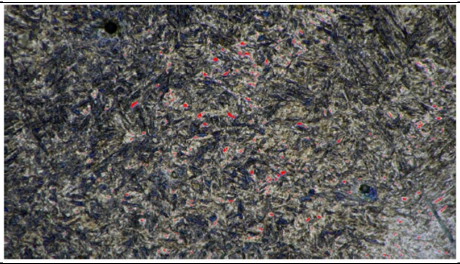
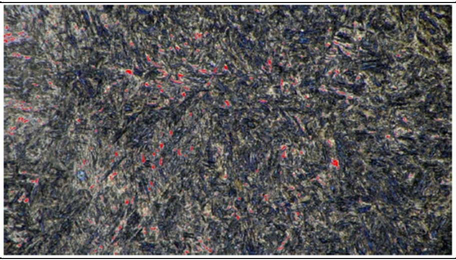
3.3 Hardness and Amount of Retained Austenite

The samples were conducted a microhardness test, which gives hardness results of the phases in which the HSLA phase hardness corresponds with the hardness of lower bainite + martensite (425-525 HV)[26,27]. The macro hardness tests' initial results were in HRC, which were then converted to HV[28], and the results are shown in Fig. 7(a).

The results of the OM microstructural images of the HSLA samples were used to calculate the retained Austenite. The amount of retained Austenite is calculated using ImageJ software based on the darkness of the phase in the image. The retained Austenite in the microstructure is red/light in color, so calculations are carried out on the bright phases of the image. Calculations were carried out 2-3 times at the different austenitizing temperatures, namely 850°C, 870°C, 900°C, and 926°C at 28 minutes holding time. Examples of the calculation are shown in Table 2 – 4. With the same methods, the calculation was conducted with variable austenization holding time.

The austenitizing temperature of 850°C-900°C makes the grain size bigger and the grain boundary fewer, which makes Austenite decompose to martensite easier, followed by the rise of carbon content in the austenitic matrix so when quenched, martensite with high carbon is formed and the hardness increases. However, at 926°C, the grain becomes more coarse, the carbon content and alloying element will further increase in the austenitic solution, and the transformation temperature range to martensite becomes lower as well[29,30], hence the decrease in hardness as it becomes more difficult for Austenite to transform into martensite.

Table 2. Retained austenite calculation by Image J Software for austenizing temperature at 850°C.

Microstructure Image	**Retained Austenite (%)**
	0.2
	0.6
	0.9
Average	**0.57**

Based on the image analyzing program, the percentage of retained Austenite (%RA) shows that the %RA initially decreases from 850°C-900°C, while inversely to the hardness, because the grain size becomes bigger. Hence, the grain boundaries decrease, and it becomes easy for Austenite to decompose as lath martensite, and the %RA becomes less. In 926°C, the %RA rises due to grain coarsening, carbon content, and alloying element increase, causing the martensitic transformation temperature range to lower. It will be more difficult for Austenite to transform to other phases, such as martensite or bainite. However, as the %RA is still <1%, the HSLA product is still safe from defect and delayed crack[31], for the alarming amount of %RA that causes a decrease in dimension stability is >12-15%[31,32].

Table 3. Retained austenite calculation by Image J Software for austenizing temperature at 870°C.

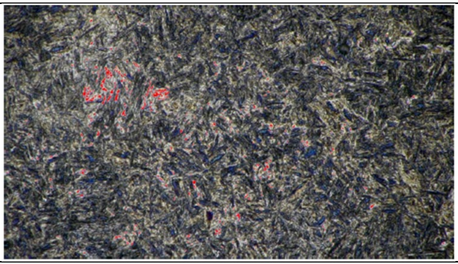
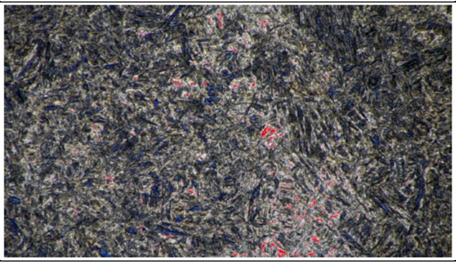
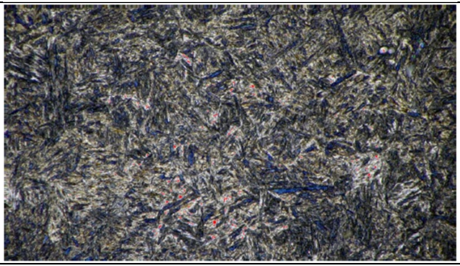
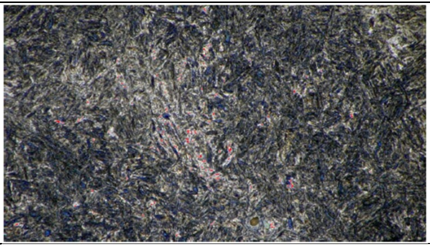
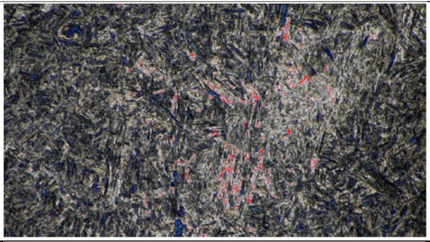
Microstructure Image	**Retained Austenite (%)**
	0.7
	0.6
	0.2
Average	**0.50**

Table 4. Retained austenite calculation by Image J Software for austenizing temperature at 900°C.

Microstructure Image	**Retained Austenite (%)**
	0.3
	0.6
	0.2
Average	**0.37**

The Retained Austenite calculation could be summarized in Table 5 for the calculation at various austenization temperatures and Table 6 for the different holding times.

Table 5. Retained austenite calculation at various Austenization temperature

Austenization Temperature (°C)	**Average Retained Austenite (%)**
850	0.57
870	0.50
900	0.37
926	1.70

Table 6. Retained austenite calculation at different holding times at temp 926°C of Austenization.

Austenization Holding Time (minutes)	**Average Retained Austenite (%)**
28	1.70
43	1.70
58	1.50
73	1.90

Figures 7(a) and 7(b) show the value of Retained Austenite and hardness as a function of austenization temperature and holding time, corresponding to the summary of the macrohardness test as shown in Tables 7 and 8.

Table 7. Effect of austenitization temperature on microhardness of materials

Austenization Temperature (°C)	**Average Hardness (HV)**
850	490
870	493
900	494
926	486

Table 8. Effect of austenization holding time to microhardness of materials.

Austenization Holding Time (minutes)	**Average Hardness (HV)**
28	486
43	486
58	500
73	480

The value of macro hardness tends to be constant from the holding time of 28 minutes to 43 minutes, equal to 486 HV. Then, the hardness of the steel increased at 58 minutes of holding time to 500 HV and decreased at 73 minutes of holding time, where the hardness of the steel reached 480 HV. From these data, it can be seen that the macro hardness of HSLA steel has increased with increasing austenitizing holding time. However, after reaching a certain optimal point, the hardness decreases again. With the increase of the austenitizing holding time, the grain size of the Austenite will increase so that the grain boundary (which is the nucleating site for pearlite) area will decrease, causing the Austenite to be more easily decomposed into martensite. The longer the austenitizing time, the greater the integration of the alloy elements, and the higher the hardness of the martensite matrix as well$^{(33,34)}$.

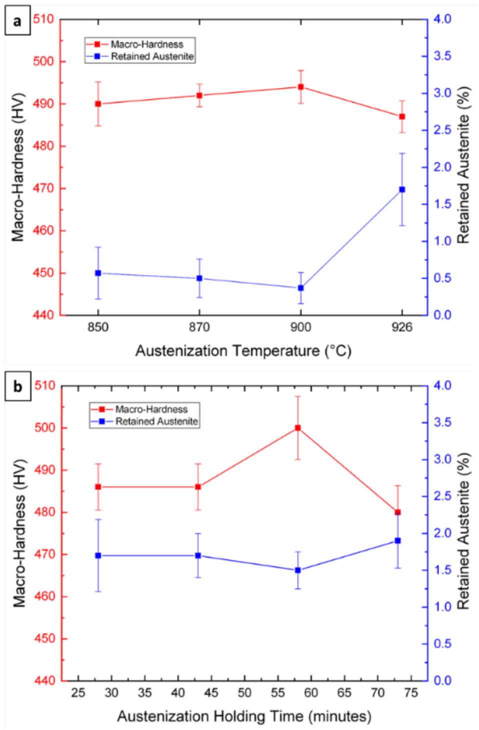


Fig. 7: Macro-hardness and retained austenite percentage at (a) various austenitizing temperatures and (b) various austenitizing holding times.

However , when the holding time is more than 58 minutes, the austenite grains will experience grain growth, which causes the grain size to be non-uniform, reducing the steel's hardness. In addition, longer than 58 minutes holding time will increase the amount of carbon diffusing into the austenite matrix so that the amount of high-carbon austenite increases. This increases the stability of the Austenite, which makes it more difficult for the Austenite to transform into martensite so that the hardness value is reduced, and retained austenites are increased, and the hardness be reduced[35,36]. The amount of retained austenite grains of HSLA steel at different heating times is shown in Fig. 7 (b). It can be seen from the figure that the amount of retained Austenite gradually decreases from 1.7% to 1.5% as the austenitizing holding time increases from 43 minutes to 58 minutes. When the holding time exceeded 58 minutes, the amount of retained Austenite increased again. When the holding time reached 73 minutes, the amount of retained Austenite was 1.9%.

4. Conclusion

The microstructure produced from the varying austenitizing temperatures and holding times is lath martensite; the phases present are tempered martensite, lower bainite, and retained Austenite. With the increase of austenization temperature and holding time, the grain becomes coarser; therefore, the size of the martensite lath becomes larger. The SEM results show the presence of M/A islands and well-tempered martensite and less-tempered martensite; the increase in austenization and holding time at 926° result in bigger martensite needles, and higher the percentage of retained austenite, because the transformation temperature range to martensite becomes lower as well, and more difficult for Austenite to Martensite.

The hardness value of both varying austenitizing temperatures and holding times shows an initial increase until 900°C and 58 minutes, and then it decreases at 926°C and 73 minutes. The percentage of retained Austenite initially decreases with the rise of austenitizing temperature and holding time (until 900°C and 58 minutes) but increases at 926°C and 73 minutes. The optimum variable with the highest hardness value (500 HV) and the lowest amount of retained Austenite (1.50%) is at the austenitizing temperature of 900°C and the holding time of 58 minutes.

The appropriate austenitizing temperature and holding time can potentially reduce the retained austenite, The lower the amount of retained Austenite, the less the isothermal transformation, and less dimensional change that caused the residual stress and the feasibility of crack initiation. Hence, it will lessen the likelihood of delayed crack.

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