Influence of Austenitizing Heat Treatment on the Properties of the Tempered Type 410-1Mo Stainless Steel

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Abstract. The modified 410-1Mo stainless steel has been developed with higher tensile strength and elongation compared to the standard 410 stainless steel. This paper reports the influence of austenitizing temperature on the microstructure, hardness, impact resistance and corrosion resistance of the modified 410-1Mo steel. The steel samples were prepared by a process sequence of induction melting, hot forging, annealing, hardening, and tempering. The microstructure of the tempered steels revealed additional phase of delta ferrite at pre-austenitizing temperatures of 950 to 1050 °C and disappeared at a temperature of 1100 °C. The steels which underwent pre-austenitizing at 1100 °C showed the largest sized lath martensite and the largest amount of retained austenite. The tempered steels maintained hardness at austenitizing temperatures of 950 °C to 1000 °C and showed an increasing hardness at austenitizing temperatures from 1000 to 1100 °C. At a range of austenitizing temperatures, it was investigated that the steels exhibited higher impact resistance at 1050 °C. The tempered steels that were pre-austenitized at 950 °C and 1100 °C showed the lowest pitting potential due to the existence of carbides and coarse-high carbon martensite, respectively.

Keywords: Austenitizing, tempered Type 410-1Mo, impact resistance.

1. Introduction
The martensitic stainless steels have been widely used for many applications such as power generation, building structure, vehicle construction and mining machine due to their high strength, high toughness and moderate corrosion resistance [1-2]. However, the martensitic stainless steels have been continuously modified to improve the mechanical and corrosion properties in order to accommodate the variably severe working conditions of the services, especially for power generation application [3-6]. The improvement of the mechanical and corrosion properties of the martensitic stainless steels can be optimized by chemical composition modification and heat treatments [7-8]. The 410-1Mo martensitic stainless steel is a modification of 410 type steel recently developed by incorporation about 1 wt.% Mo which exhibits higher tensile strength, higher elongation at breaks and higher pitting resistance compared to the standard 410 type steel for steam turbine blade application [9]. The heat treatment response properties of this steel is being investigated and parts of the results have been published elsewhere [10-11]. Although Mo are containing 4xx type stainless steel already
available such as 425Mod and 440 A, they contain higher carbon (> 0.5 wt.%) which are not suitable for turbine blade application. Thus, the modified 410-1Mo stainless steel with lower carbon content (<0.15 wt.%) has been developed aiming for better properties of the steam turbine blades.

It has already known that the heat treatment parameters of the martensitic stainless steels depend on their chemical composition. Once the chemical composition has been modified, the heat treatment of the martensitic stainless steels should be optimized to meet the technical requirements of the specific service. The heat treatment of the martensitic stainless steels for hardening is performed by quenching from high-temperature austenite phase providing solid martensite structure and subsequently tempering the as-quenched steel at suitable temperature and time to improve ductility and toughness. The austenitizing temperature determines the extent of carbide dissolution, dissolved alloying elements and grain growth influencing the final microstructure and properties of the tempered steels. Determining proper austenitizing temperature is required to ensure the desired microstructure and optimal properties of the tempered martensitic stainless steels. This paper presents the influence of the austenitizing temperature on the microstructure, hardness and impact energy of the tempered 410-1Mo martensitic stainless steel. The tempering of the as-quenched steels was performed at high temperature of 600 °C to facilitate the use of this martensitic stainless steel for turbine blades application operated up to such a high temperature.

2. Experimental Method

The induction melting furnace was used for fabricating the ingots of the investigated martensitic stainless steel of type 410-1Mo with a dimension of 5 x 5 x 10 cm. Hot forging of the steel ingots was carried out at a temperature of 900-1125 °C until the dimension of the ingots changed to about 3 x 3 x 27 cm. The square specimens with 1 cm in thickness were cut from forged steels for chemical composition testing using Optical Emission Spectrometer (OES). The chemical composition of the steels is shown in table 1. The other parts of the forged steels were annealed at a temperature of 800 °C for 20 h for softening to facilitate manufacturing the samples for testing. The steel samples were subjected to austenitizing at 1050 °C for 1 h followed by quenching in oil. The austenitized samples were then tempered at a temperature of 600 °C for 1 h followed by air cooling. The heat treated samples were surface ground to remove scales and processed through a machine to prepare samples for microstructure, impact test, hardness tests and cyclic polarization.

The samples for microstructural observation were prepared by standard metallographic operations and etching was performed by using Vilela’s reagent. The microstructural images were taken by an optical microscopy and Scanning Electron Microscope (SEM). Rockwell C diamond indentor measured the hardness of the steel samples at a temperature of 120 °C with indentation load and time of 15 Kgf and 3s, respectively. The impact test was conducted at room temperature by Charpy impact tester using V-notch samples with a dimension of 10 × 10 × 55 mm according to ASTM E 23. For cyclic polarization measurement, the specimens with a surface area of 2.5 cm² were polished with SiC paper from grit 120 to grit 800. The measurement was conducted in the 3.5% Sodium Chloride solution at room temperature using a Gamry G750 system.

| Table 1. Chemical composition (wt. %) of the 410-1Mo martensitic stainless steels prepared in this work |
|-------------------------------|----|---|----|----|----|----|----|----|
| Steel                        | C  | S  | P  | Mn | Si | Cr | Mo | Ni |
|-------------------------------|----|---|----|----|----|----|----|----|
| 13Cr-1Mo                      | 0.13 | 0.003 | 0.02 | 0.94 | 0.31 | 13.13 | 0.80 | 0.29 |
|                               |    |    |    |    |    |    |    | Bal. |
3. Results and Discussion

The optical microscopy images of the microstructure of the 410-1Mo martensitic stainless steel after quenched at 950, 1000, 1050, and 1100 °C and tempered at 600 °C was presented in figure 1. In general, the tempered microstructure of the martensitic stainless steels consists of tempered martensite, carbides and retained austenite. However, in figure 1 (a)-(c), the tempered microstructure of 410-1Mo martensitic steel reveals an additional feature of phase called delta ferrite, the bright island-like phase seen in the images, which was formed due to a non-equilibrium solidification, particularly found in the steels containing high ferrite-stabilizing elements such as Cr and Mo [12]. It is worth to note that at the higher austenitizing temperature of 1100 °C, the delta ferrite disappeared in the tempered microstructure as shown in figure 1(d). It may be due to the increased amount of the alloying elements including Cr and Mo going to the solution at the higher austenitizing temperature. The formation of delta ferrite in the martensitic stainless steels was also reported in literature [12-13]. Meanwhile, the brighter phase appeared in the microstructure of figure 1(d) was not delta ferrite, but it was retained austenite. The austenite phase remained unchanged to martensite during quenching. The Increasing amount of retained austenite in the as-quenched microstructure was attributed to the increasing amount of carbon and carbide-forming elements in the solution of austenite phase which depressed the martensite transformation and lowering the Ms and Mf temperature (Martensite start and Martensite finish, respectively) at or even below room temperature [14].

The martensite and carbides appeared in the microstructure of the tempered martensitic stainless steels were also influenced by the austenitizing temperature. It can be observed from figure 1 that the martensite appeared in different size and density of lath, the higher the austenitizing temperature, the larger lath size. However, the density of the martensite lath seemed to be the most important in the steel austenitized at 1150 °C as displayed in figure 1(e). With respect to carbides, it is observed to appear in the tempered steels with pre-austenitizing at 1000 °C and lower (figure 1 (a) and 1 (b)) and to be absence in pre-austenitizing process at 1050 °C and higher. The carbides density in the tempered steel with pre-austenitizing at 950 °C is qualitatively larger compared to that of the one with pre-austenitizing at 1000 °C. From these findings, the carbides appeared in figure 1 (a) and (b) were from the non-dissolved carbides during the austenitizing heat treatment and the carbides dissolved completely in the solution of the austenite at the temperature of 1050 °C and higher. The existence of carbides in the steels with pre-austenitizing at 950 and 1000 °C was evidenced in the images taken from SEM as presented in figure 2 (a) and 2(b), respectively, where it is not observed in the SEM images in figure 2(c) and 2(d). Small white globular precipitates indicated the carbides in the respective images.

The mechanical properties of the tempered 410-1Mo with various pre-austenitizing temperature are presented in figure 3 and figure 4. It is seen in figure 2 that the tempered steels maintained hardness at pre-austenitizing temperatures of 950 °C to 1000 °C and showed increasing hardness at a pre-austenitizing temperature from 1000 to 1100 °C. It is known that the hardness of the martensitic stainless steels strongly depends on the martensite phase involved in the microstructure. As all the steels in this work were tempered at the same temperature, the difference in hardness of these steels was attributed to the nature of the martensite resulted from different austenitizing temperatures. The lowered hardness of the steel with pre-austenitizing at 950 and 1000 °C corresponded to the tempered martensite with lower lath density and size. Whereas, the higher value of hardness in the steels with pre-austenitizing at 1100 °C corresponded to the higher carbon content of the martensite. As the austenitizing temperature increased, the amount of the carbon dissolved in the solution of austenite also increased resulting in the higher carbon content of the martensite after quenching. The higher the carbon content of the martensite, the higher the hardness of the martensite.

The correlation of pre-austenitizing temperature with an impact energy of the tempered 410-1Mo martensitic steel is shown in figure 4. The impact energy of the steels increased with the increasing pre-austenitizing temperature up to 1050 °C and decreased at the temperature of 1100 °C. The increasing impact energy as pre-austenitizing temperature increased may be due to the increasing retained austenite. Additionally, the decrease in the impact energy at the temperature 1100 °C might correspond to the brittleness of the hard high content carbon martensite and growth of the pre-austenite grains due to high-temperature austenitization [15-16].
Figure 1. Optical microscopy images of the microstructure of the 410-1Mo martensitic stainless steels austenitized at various temperature of (a) 950, (b) 1000, (c) 1050, and (d) 1100 °C and subsequently tempered at 600 °C.

Figure 2. SEM images of the microstructure of the tempered 410-1Mo martensitic stainless steel austenitized at various temperatures of (a) 950, (b) 1000, (c) 1050, and (d) 1100 °C and subsequently tempered at 600 °C.
Table 2. Corrosion parameters of 410-1Mo martensitic stainless steel austenitized at different temperatures and tempered at 600 °C

| Heat Treatment (°C) | $E_{cor}$ (mV) | $I_{cor}$ ($\times 10^{-6}$ A/cm$^2$) | $E_{pit}$ (mV) |
|---------------------|----------------|-------------------------------------|----------------|
| 950-600             | -622           | 74                                  | -540.4         |
| 1000-600            | -436           | 12.9                                | -134.5         |
| 1050-600            | -506           | 19.5                                | -291.5         |
| 1100-600            | -899           | 23.2                                | -542.6         |

Figure 3. The hardness of the 410-1Mo martensitic stainless steels austenitized at various temperatures and subsequently tempered at 600 °C

Figure 4. The impact energy of the 410-1Mo martensitic stainless steels austenitized at various temperatures and subsequently tempered at 600 °C

Figure 5. Cyclic polarization curves of the 410-1Mo martensitic stainless steel austenitized at various temperatures and subsequently tempered at 600 °C

Figure 5 shows the cyclic polarization curves of the 410-1Mo martensitic stainless steels austenitized at various temperatures and subsequently tempered at 600 °C. The curves are typical for
stainless steels, displaying passivation region and breakdown potential (pitting) clearly. It was observed that the different austenitizing temperatures applied to the steels resulted in various positions of the polarization curves in the E-I plot, affecting different corrosion parameters of the tempered steels. The corrosion parameters extracted from the curves are shown in table 2. The corrosion potential and pitting potential are observed to follow the similar trends, they decreased on the increasing austenitizing temperature from 950 °C to 1000 °C, and they increased again from 1000 °C to 1100 °C, whereas the corrosion current density followed the opposite trend. Both the tempered steels that were pre-austenitized at the temperatures of 950 °C and 1100 °C exhibited high susceptibility to pitting corrosion with almost similar low potential pitting values of -540.4 and -542.6 mV, respectively. These might be due to the higher content of carbides in the tempered steels that were pre-austenitized at 950 °C and higher carbon content of the martensite in the tempered steels that were pre-austenitized at 1100 °C. However, the steels that were pre-austenitized at 1100 °C exhibited the repassivation potential on the reverse polarization scan. The repassivation process occurred in this steel might be attributed to a higher content of retained austenite with high content of carbide-forming Cr and Mo elements which were dissolved in the solid solution during a high austenitizing temperature of 1100 °C.

4. Conclusion
The influence of austenitizing temperature on microstructure, hardness and impact resistance of the modified 410-1Mo stainless steel has been investigated. The microstructure of the tempered steels revealed additional phase of delta ferrite at pre-austenitizing temperatures of 950-1050 °C and disappeared at 1100 °C. The steels with pre-austenitizing at 1100 °C showed the largest sized lath martensite and largest amount of retained austenite. The tempered steels maintained hardness at pre-austenitizing temperatures of 950 °C to 1000 °C and showed increasing hardness at the austenitizing temperatures from 1000 to 1100 °C. At a range of austenitizing temperature, it was investigated that the steels exhibited higher impact resistance at 1050 °C. The tempered steels that were pre-austenitized at 950 °C and 1100 °C showed the lowest pitting potential due to the existence of carbides and coarse-high carbon martensite, respectively.

5. References
[1] Mesa D H, Toro A and Sinatora A 2003 Wear 255(1) 139
[2] Zhu X, Dai O and Zhou O 2009 Transactions of Materials and Heat Treatment 30(1) 11
[3] Krishna S C, Gangwar N K, Jha A K, Pant B and George KM 2015 Steel. Res. Int. 86 (1) 51-57
[4] Fan R, Gao M, Ma Y, Zha X, Hao X and Liu K 2012 J. Mater. Sci. Technol. 28(11) 1059–66
[5] Kumara B R, Sharma S, Mundaa P and Minz R K 2013 Mater. Des. 50 392–398
[6] Cuevas, Rodriguez J A, Clemente C M, Rodriguez J M and Mariaca Y 2014 Am. J. Mech. Eng. 2(6)164-168
[7] Lua S Y, Yaoa K F, Chenb Y B, Wang M H, Liua X and Ge X 2015 Electrochim. Acta. 165 45–55
[8] Singh S and Nanda T 2013 Int. J. Eng. Technol. Sci. Res. 1(1)
[9] Mabruri E, Anwar M S, Prifiharni S, Romijarso T B and Adjiangtoro B 2016 AIP Con. Proc. 1725 020039
[10] Prifiharni S, Anwar M S and Mabruri E 2016 Widyariset 2(1) 9-16
[11] Mabruri E, Syahlan Z A, Sahlan, Anwar M S, Romijarso T B and Adjiangtoro B 2017 Int. J. of Eng. and Tech. (in review)
[12] Wang P, Lu S P, Li D Z, Kang X H and Li Y Y 2008 Acta Metall. Sin. 44 681–685
[13] Wang P, Lu S P, Xiao N M, Li D Z, Li Y Y 2010 Mat. Sci. and Eng. A. 527 3210–16
[14] Barlow I D and Toit M D 2012 J. of Mat. Eng. and Perf. 21(7) 1327.
[15] Balan K P, Venugopal R A and Sarma D S 1998 Scripta Mater. 39 901–905
[16] Yu R, Ye D, Yong Q, Su J, Zhao K and Jiang W 2011 J. Iron and Steel Res. Int. 18(11) 60-66
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