Comparative Mechanical Improvement of Stainless Steel 304 Through Three Methods

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Abstract. Stainless Steel 304 (SS304) is one of stainless steel group widely used in industries for various purposes. In this paper, we compared the experimental process to enhance the mechanical properties of the surface SS304 through three different methods, cold rolled, annealed salt bath bronzing (ASB), and annealed salt bath boronizing-quench (ASB-Q). The phase change in SS304 due to the cold rolled process makes this method is to abandon. The increasing of the annealing time in the ASB method has a nonlinear relationship with the increases in hardness value. Comparing to the increases in hardness value of the ASB method, the hardness value of ASB-Q methods is still lower than that method.

Keywords. Annealing, ASB, and stainless Steel 304.

1. Introduction

Stainless Steel 304 (SS304) is one of stainless steel group which is widely used in industries for various purposes. Austenitic stainless steels have excellent corrosion resistance in many environments and good weldability as part of the high strength and good formability [1]. One use of SS304 is as a crude oil transport pipelines from an oil well to a processing plant in oil and gas industry. This transport method consumes less energy (compared to conveyer belt transportation). However, SS304 has many components fail in practice. Severe metallic wear frequently endures the SS304 as the effect of degradation of pipelines (erosion) by fine solids (clays), bitumen, and sands is a critical issue. In these situations, it is necessary to modify the surface properties while it is still important to maintain the nature of the SS304 itself [2]. For these purposes, various attempts have been made to engineer the surfaces of austenitic stainless steels. These efforts include cold rolled, carburizing and boronization methods. In the previous study, the deformation by the cold rolled process has transformed the austenite structure into martensitic structure [3]. Moreover, at in 20 % distortion, the value of magnetization is increased above 0.2 T with a small coercivity and caused the ability of corrosion resistant decreases by magnetic measurement [4,5]. However, Etessami et al. reported that the increase of annealing time of 80 % cold rolled 304 led to the formation of austenite at the edge of fine recrystallized ferrite grain boundaries [6]. Furthermore, the Enriching of the surface with boron had investigated by means of boron atoms diffuse into the metallic lattice of the surface of a workpiece, where provides wear and abrasion resistance [7].
In this paper, we compared the experimental process to enhance the mechanical properties of the surface SS304 through three different methods. The best achievements value of among that process is reported.

2. Materials and methods
The samples specimen is taken from the 11 mm thick SS304 pipe which has compound as in Table 1. The hardening process is carried out through three different methods: cold rolled, annealed salt bath boronizing (ASB), and annealed salt bath boronizing-quench (ASB-Q). The first method, cold rolled is used to deform the sample into 20 %, 40 %, and 60 %. Second methods, the boronizing is used by soaking the sample into borax (Na$_3$B$_4$O$_7$·10H$_2$O) and annealed at 750 °C dan 1000 °C for 6 h, 8 h, and 10 h. Third, the boronizing is used by annealed for 6 h of soaking sample into borax followed by quenched into the water at room temperature. The crystalline structure and the microstructure of the sample were examined using X-ray diffraction and scanning electron microscope (SEM). The enhancement of surface properties formed quantify through wear and hardness measurement.

| Table 1. Chemical composition of stainless steel 304 specimen |
|-----------------|-----|-----|-----|-----|-----|-----|-----|
|                | C   | Mn  | Si  | P   | S   | Ni  | Cr  | Fe  |
| Mill certificate | 0.05 | 1.08 | 0.35 | 0.038 | 0.001 | 8.50 | 18.65 | -   |
| AAS             | -   | 1.49 | -   | -   | -   | 10.6 | 21.20 | 66.44 |

3. Results and discussion

3.1. Cold rolled methods
The XRD result of mechanical treatment via cold rolled for 20 %, 40 % and 60 % deformation shows in Figure 1 wherein 0 % treatment is used as a reference. As in the figure, the structural changes can be seen from 0 % to 60 % by identified the reveal of new peak pattern or decreases and losses of some peaks pattern. At 20 % deformation, the new peak pattern reveals below 2θ = 30° which is correlated with martensitic phase present [8]. When the sample gets 40% of deformation, the presence of this peak followed by the reduction of the entire peak between 2θ = 35° to 55°. Moreover, at 60 % deformation, those peak patterns below 30° are disappeared followed by the decreases of the two peak at 2θ = 46° to 55° and the other peak. From these results, we see that the rise of the martensite phase occurs in 20 % to 40 % deformation. Furthermore at 60 % deformation increases the phase form has changed to another phase [8–10].

Figure 2 shows the SEM result of the sample surface of every deformation level. At 0 % deformation, it appears that the SS304 alloy consists of an austenite phase. This phase can be identified by its grain shape. The structural changes occurring in the SS304 alloy resulted in morphological changes of the grain shape. This begins with the form of wrinkles that occur on the surface of grains that have 20 % deformation. Furthermore, at 40 % deformation shows the occurrence of shift between the grains marked by the difference between the surface heights of the grains accompanied by the emergence of the needle-shaped pattern on the surface of the sample. Then at 60 % deformation, it appears that the grain shape is increasingly elongated followed by the impingement of the spacing between the grains. This form is reminiscent of the martensitic phase in steel.
Figure 1. XRD of a) 0% deformation (black line), b) 20% deformation (pink line), c) 40% deformation (green line), d) 60% deformation (blue line). The red line arrows show the reveal of new peak appears at 20% dan 40% deformation.

Table 2. Hardness and wear test value of SS304

| Deformation (%) | Hardness (HVc) | Wear Resistance |
|-----------------|---------------|----------------|
| 0               | 145           | 1.45           |
| 20              | 344           | 4.42           |
| 40              | 374           | 2.28           |
| 60              | 395           | 2.81           |

As seen in Table 2, the highest value of enhancement hardness is obtained when deformation occurs 20% of the initial condition. Meanwhile, the highest value of enhancement friction resistance is also obtained at 20% deformation. Furthermore, the optimum value of hardness and frictional resistance met in the 20% of deformation. However, the emergence of the martensite phase form which indicates a phase change in SS304 due to the cold rolled process makes this method is to be abandoned.
Figure 2. SEM of a) 0% deformation, b) 20% deformation, c) 40% deformation, d) 60% deformation. The cold rolled methods have changed their grain shape.

3.2. Annealed salt baht boronizing (ASB) and annealed salt baht boronizing-quench (ASB-Q) methods.

Figure 3 shows morphological cross-section used scanning electron microscope from sample ASB and ASB-Q methods which represented in Figure 3a, Figure 3b and Figure 3c. The borax content had a notorious influence on the presence of boronization layer was observed in all methods. Simple scratch test for this cross-section sample is using 1200 - type sandpaper. The streaks line formed on the surface between the substrate layer and boronized layer have a correlation to the wear resistance properties of those material surface.

| Method | Temperature (°C) | Holding time (Hours) | Hardness (HVC) |
|--------|------------------|----------------------|----------------|
| 0      | 0                | 0                    | 145            |
|        |                  | 6                    | 223            |
| 750    | 8                | 226                  |
| ASB    | 10               | 218                  |
| 1000   | 6                | 227                  |
|        | 8                | 215                  |
|        | 10               | 602                  |

Table 3. The result of hardness test of ASB and ASB-Q methods

The ASB method is represented by the annealed sample at 750 °C and 1000 °C for 10 h. The result of all the ASB method shows the formation of a thin layer with a ~5 μm of thick. In Figure 3a and Figure 3b it appears that a streak line is clearly formed in both the surface area with the deepest streak occurring in the substrate area. Refer to Table 3, the hardness value formed of 750 °C annealed is higher than of 1000 °C annealed in ASB method. We can see that the increase of the annealing time has a non-linear relationship with the increases in hardness value. This condition occurs either at 750 °C or at 1000 °C of annealing temperature. From both annealed temperatures, the optimum hardness value is obtained at 750 °C for 8 h of annealing time.

The ASB-Q method is represented by the annealed sample at 1000 for 6 h which follows by a quench in water. Figure 3c shows that the streaks in the boronization area look vague compared to the streaks formed in the substrate area. Two distinct regions were identified on the boronized SS304 surface: these are (i) substrate region and (ii) boronized region which have globular matrix shape [11–13]. Refer to Table 3, the hardness value formed of this methods is 602 HVC. On the face of hardness values obtained from this method looks much higher than the previous method. Comparing to the increases in hardness value of ASB at 750 °C for 8 h anealling, this value is still lower than that.
method. Therefore, the characterization of ASB-Q method at 750 °C for 8 h needs to be done to obtain more relevant benchmark results.

![Image](attachment:image_url)

(a) Boronization at 750 °C within 10 h with 4.5 μm layer thick, b) Boronization at 1000 °C within 10 h with 17.7 μm layer thick, c) Boronization 1000 °C within 6 h continued Quench in water, the red line shows the boundary between metal and boronized region.

**Figure 3.**

4. **Conclusions**

Microstructure and microhardness properties of stainless steel 304 with different treatment by three methods were investigated. Methods are compared the experimental process to enhance the mechanical properties of the surface stainless steel 304. Characterization by XRD shows that cold rolled affected to decreased on structural peaks materials as long as high deformation. It can happen because structural transformation from austenite to martensitic which can be seen with increasing hardness until 390 H Vc at 60 % deformation. The highest value of increment hardness is achieved by ASB-Q method 602 H Vc, and the lowest value of hardness is achieved by the based material of stainless steel 304 145 H Vc.

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